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Interlaboratory Validation of EPA 1600 Series Methods

Draft EPA Method 1638 for Analysis of Matels in Water by InductivelyCoupled Plasma - Mass Spectrometry (ICP-MS)

Technical Report

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REPORT SUMMARY

Federal and state permits are requiring wastewater dischargers to monitor for ever lower concentrations of trace metals, in some cases at levels that may preclude reliable measurement. In this joint EPA-EPRI interlaboratory data collection effort, eight laboratories evaluated draft EPA Method 1638: Determination of Trace Elements in Ambient Water by Inductively Coupled Plasma-Mass Spectrometry. This method is intended for the analysis of low levels (parts per trillion) of antimony, cadmium, copper, lead, nickel, selenium, silver, thallium, and zinc in ambient water. Data generated on the method's performance should help in evaluation of its applicability and reliability for assessing ambient water quality conditions and for use in the National Pollutant Discharge Elimination System (NPDES) permit program.

Background

The EPA has proposed a series of trace metals methods, referred to as EPA 1600 Series Methods, which are potentially capable of producing measurements at very low concentrations. The EPA and EPRI jointly funded the data collection effort for the interlaboratory validation of Method 1638 but evaluated the results independently. EPRI provided important input to the study design based on earlier Institute studies that validated methods for the analysis of metals in water.

Objective

To evaluate the performance of EPA Draft Method 1638 for measurement of metals in water by determining the relation of interlaboratory standard deviation to analyte concentration as well as recovery, detection, and quantification levels.

Approach

The EPA loosely based its study design for Draft Method 1638 on ASTM D-2777, Standard Practice for the Determination of Precision and Bias of Applicable Test Methods of Committee D-19 on Water. Investigators tested four matrix types: reagent grade water, freshwater, and filtered and unfiltered effluent from a municipal wastewater treatment plant. Eight laboratories completed the round-robin study using the specified analytical method. EPRI investigators calculated analyte recoveries (measured amount versus true concentration) and determined single operator (within laboratory) standard deviation and overall (among laboratories) standard deviation data for each element, concentration level, and matrix. Based on this information, they derived interlaboratory detection estimates (IDEs) and interlaboratory quantitation

estimates (IQEs) for each study analyte in the reagent grade water and the freshwater matrices. They next compared these values with federal water quality criteria (WQC) as well as method detection limits (MDLs) and interim minimum levels (MLs) previously published by the EPA for the method.

Results

EPA Draft Method 1638 was evaluated in reagent water and in filtered lake water. Filtered and unfiltered secondary treated, municipal wastewater was also included in the study; however, since only a single pair of samples from each effluent matrix was analyzed, the method remains unvalidated in those matrices. The method has not been validated in other matrices.

Recoveries of the study elements from a standard reference material (SRM) ranged from 93-102%. Although these recoveries tend to indicate that the method is free from bias, the concentrations of the study elements in the SRM ranged from 3-50 times higher than the highest spiked concentrations in the freshwater samples. Thus, recoveries from this SRM are not a good predictor of the method's performance with real-world samples.

In the evaluation of detection and quantitation limits for elements other than silver—which was not stable in the test matrices—IDEs were generally higher than the corresponding EPA MDLs. Likewise, the IQEs for a relative standard deviation of 20% were generally higher than the EPA MLs. In a freshwater matrix, only nickel had an IQE 20% less than or equal to the corresponding EPA ML. The detection and quantification estimates for all study analytes were less than their respective federal WQC. These findings indicate that EPA Draft Method 1638 is suitable for the analysis of the tested elements at the lowest federal WQC in freshwater but is generally not appropriate for compliance monitoring at or near the EPA MDLs and MLs listed in the method documentation.

EPRI Perspective

This report is the second in the EPA 1600 Series Method Validation Studies. The first—an evaluation of Draft EPA Method 1631 for Analysis of Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectroscopy (CVAFS)—is available as EPRI Report TR-111424. EPRI's work has been used by the EPA, the Utility Water Act Group, the Inter-Industry Analytical Group (IIAG), and the ASTM in their efforts to develop scientific detection and quantification levels for trace metals in water.

Keywords

Chemical analysis
Measurement/accuracy
Trace metals
Compliance monitoring
Detection/quantification

ABSTRACT

This report presents the results of a joint EPA/EPRI Interlaboratory Validation Study of Draft EPA Method 1638 for Determination of Trace Elements in Ambient Water by Inductively Coupled Plasma-Mass Spectrometry. The objective of this study was to evaluate the method's performance by evaluating precision and bias for the method in qualified analytical laboratories. Full validation data were obtained for reagent grade water and a fresh (lake) water sample. Limited data were obtained for filtered and unfiltered municipal wastewater treatment plant effluent. Eight laboratories completed the study.

Performance data were derived using STATCALC, a statistical analysis program developed by EPRI based on ASTM D 2777. The recovery and interlaboratory standard deviation data were used to calculate estimates of detection and quantification for antimony, cadmium, copper, lead, nickel, selenium, thallium and zinc in reagent grade water and freshwater. The ninth study element, silver, was not stable in the study matrices.

Mean recoveries of the target elements in fortified filtered and unfiltered effluent were generally greater than 90% for all analytes except silver, which was not stable. Although reported recoveries of study analytes from a Standard Reference Material (SRM) were greater than 90% for all data reported by the participants, these recoveries were based on a SRM that has much higher element concentrations than the study samples. Thus, these results may not reflect real-world performance.

The interlaboratory detection estimates (IDEs) and interlaboratory quantitation estimates (IQEs) calculated by EPRI were generally higher than the corresponding EPA method detection limits (MDLs) and minimum levels (MLs). In the freshwater matrix, only nickel had an IQE with a relative standard deviation of 20% that was less than the corresponding EPA ML. The detection and quantification estimates for all of the study analytes were less than their respective water quality criteria. These findings indicate that EPA Draft Method 1638 is suitable for the analysis of the tested analytes at the lowest Federal WQC in freshwater, but generally is not appropriate for compliance monitoring at or near the EPA MDLs and MLs quoted in the method. Performance in other matrices must be validated by collaborative studies designed in accordance with ASTM D 2777.

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INTRODUCTION

1.1 Background and Objectives of the EPA/EPRI Method 1638 Validation Study

The electric power industry is required under the Federal Water Pollution Control Act Amendments of 1972 (Clean Water Act), as amended, to monitor their discharges into the nation's waters for numerous chemical substances. Individual facilities are issued permits under the National Pollutant Discharge Elimination System (NPDES) that impose limitations on the type and amount of certain pollutants they may legally discharge. The discharge limits vary based on the applicable water quality standards, the way the permitting authority implements the standards, and the nature of the receiving waterbody. Since the initial issuance of the Clean Water Act, amendments have resulted in increasingly lower permissible discharge concentration limits, requiring more sensitive techniques for measuring the analytes and more stringent protocols for sample collection to prevent contamination.

On October 1, 1993, the United States Environmental Protection Agency (EPA) Office of Water issued a memorandum entitled "Office of Water Policy and Technical Guidance on Interpretation and Implementation of Aquatic Life Metals Criteria." In the memorandum, EPA outlined their plans to develop "clean" (ppb range) and "ultraclean" (ppt range) techniques and protocols for the collection and analysis of trace level metals.

The EPA trace metals draft methods proposed thus far include:

- Method 1631: Mercury in Water by Oxidation, Purge and Trap, and CVAFS
- Method 1632: Inorganic Arsenic in Water by Hydride Generation Quartz Furnace AA
- Method 1636: Determination of Hexavalent Chromium by Ion Chromatography
- Method 1637: Determination of Trace Elements in Ambient Water by Chelation Preconcentration with Graphite Furnace Atomic Absorption
- Method 1638: Determination of Trace Elements in Ambient Water by Inductively Coupled Plasma-Mass Spectrometry
- Method 1639: Determination of Trace Elements in Ambient Waters by Stabilized Temperature Graphite Furnace Atomic Absorption
- Method 1640: Determination of Trace Elements in Ambient Waters by On-Line Chelation Preconcentration and Inductively Coupled Plasma-Mass Spectrometry

All of the 1600 Series trace metals methods reference the sampling protocol, Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria.

In the spring of 1997, the EPA began circulating study plans for interlaboratory validation studies of the 1600 series trace metals methods. EPA and EPRI completed a joint validation study of the draft EPA Method 1631: Mercury in Water by Oxidation, Purge and Trap, and CVAFS, July 1996 version (EPA-821-R-96-012). EPRI's evaluation of the mercury study results (TR-111424) was published in November 1998.

The EPA conducted large, multi-method validation studies of the other proposed methods in 1999. The EPA and EPRI jointly funded two of the method evaluation studies: (1) EPA Draft Method 1638: Determination of Trace Elements in Ambient Water by Inductively Coupled Plasma-Mass Spectrometry and (2) EPA Draft Method 1632: Inorganic Arsenic in Water by Hydride Generation Quartz Furnace AA. This report summarizes the Method 1638 study. EPRI funded the preparation and analysis of additional sample sets to the study to conform to the experimental design for interlaboratory round-robin studies recommended by the American Society for Testing and Materials (ASTM) "Standard Practice for Determination of Precision and Bias of Applicable Methods of Committee D19 on Water," D 2777. Both the EPA and EPRI agreed to share all study data. The study design is described in detail in Chapter 2.

EPRI's primary objective was to collect interlaboratory precision (standard deviation) and bias (observed value minus true value) data at multiple concentration levels for each analyte. Such data would describe how Method 1638 could be expected to perform and would enable the calculation of detection and quantification levels. EPRI built on the techniques and experience developed in EPRI's earlier Analytical Methods Qualification (AMQ) studies, which were interlaboratory validation studies of methods for the analysis of trace metals in water. These studies were published as EPRI reports CS-5910 (Vols. 1, 2 and 3), TR-105910, TR-108989 and TR-11424.

1.2 Overview of the Study Design

The joint EPA/EPRI Method 1638 validation study for trace elements by ICP-MS was a round-robin study. All participant laboratories received identical sample sets and instructions and were directed to follow the Method using the same procedures. The study design is described in Chapter 2.

1.3 Objectives Of This Report

The objective of this report is to provide validated standard deviation and bias data as a function of true concentration for antimony (Sb), cadmium(Cd), copper (Cu), lead (Pb), nickel (Ni), silver (Ag), selenium (Se), thallium (Tl) and zinc (Zn) by EPA Draft Method 1638: Determination of Trace Elements in Ambient Water by Inductively Coupled Plasma-Mass Spectrometry (January 1996). In addition, we have provided estimates of the ASTM Interlaboratory Detection Estimate (IDE) and the ASTM Interlaboratory Quantitation Estimate (IQE) for each analyte in reagent grade water and freshwater based on the data collected. The IDE and IQE represent the lowest

level of reliable detection and quantification, respectively, for samples analyzed in multiple laboratories.

1.4 Use Of This Report

When an analyst produces a result that falls below the Interlaboratory Quantitation Estimate, the result needs to be evaluated carefully in terms of its potential use. By definition, measurements below the IQE cannot be used reliably to ascertain the concentration of a pollutant in a sample. Thus, the use of such values in a regulatory setting generally is inappropriate. The intent of this report is to provide detection and quantification levels based on measurements made in qualified laboratories using common matrices. The estimates of detection and quantification levels derived from these interlaboratory data provide a framework for evaluating data to be used in assessing ambient water quality conditions and in permit negotiation and compliance monitoring situations.

1.5 Organization of this Report

The study design is discussed in Chapter 2. A discussion of the statistical methods applied to the data is presented in Chapter 3. The complete data evaluation process is described in Chapter 4.The development of detection and quantification definitions is discussed in Chapter 5. The results of the data evaluation are presented in Chapter 6, including detection and quantification estimates calculated from the validation study data. The report's conclusions are given in Chapter 7. The appendices contain nomenclature used in the report, the analytical results and the standard reference material (SRM) data reported by the laboratories, and the input/output files from statistical processing of the data.

2
STUDY DESIGN

The interlaboratory validation study of EPA Method 1638 was a joint EPA/EPRI project designed by the EPA. The EPA specified the criteria for laboratory participation in the study, selected the laboratories, sample matrices and concentrations and specified the sample collection, preparation and analysis procedures. EPRI contributed funding to include additional samples to the EPA's sample sets. The goal of EPRI's participation was to ensure that sufficient validation data were collected to enable validation of the method in reagent grade water and one additional matrix, freshwater.

The study was divided into three phases: (1) Laboratory Qualification and Selection, (2) Sample Collection and Preparation and (3) Method Performance Evaluation.

2.1 Phase 1 – Laboratory Qualification and Selection

The objective of Phase 1 of the validation study was to select laboratories that met the EPA's requirements for participation in the study. The EPA selected Frontier Geosciences, Inc. as the referee laboratory. DynCorp was selected as the study coordinator and to act as the Sample Control Center (SCC) for the study. Prospective laboratories were asked to submit to the study coordinator Method Detection Limits (MDLs) determined in accordance with 40 CFR Part 136, Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," revision 1.11, and the results of a method blank analysis. The eight laboratories selected by the EPA based on the Phase 1 test results are shown in Table 2-1.

Table 2-1
Laboratories Participating in the Method 1638 Validation Study

Laboratory
Battelle Marine Sciences Laboratory
Commonwealth of Virginia, Department of General Services, Division of Consolidated Laboratory Services
* Hampton Roads Sanitation District
Southwest Research Institute
* Texas A&M, Trace Element Research Laboratory
University of Iowa Hygienic Laboratory
University of Notre Dame, Department of Civil Engineering and Geological Sciences
*USEPA Region 10 Laboratory

* = volunteer laboratory

The EPA MDL procedure requires analysis of *n* replicates (minimum seven) of a sample containing 1-5 times the estimated method detection level of the analyte of interest. The standard deviation of the replicate measurements is multiplied by the appropriate t-value for the degrees of freedom (*n*-1) to obtain the MDL. The calculated MDL is then compared to the total concentration of the analyte of interest. The ratio of the total analyte concentration to the MDL must be in the range of 1 to 5. If it is not, the MDL obtained is not considered valid and the procedure must be repeated.

Table 2-2 summarizes the range of MDLs reported by the participants in comparison to the EPA MDLs listed in the method. Tables 2-3 through 2-11 show the MDLs calculated from the raw data submitted by the participant laboratories. The laboratories reported MDLs based on 7 to 10 replicates. The Hald (1) test for equality of variance was applied to the data to determine if the values could be pooled. In most cases, the range of values obtained was great enough that an excessive number of data sets would have to be removed to meet the Hald criteria for pooling the data. A pooled MDL could only be calculated for the antimony data.

Table 2-2
Summary of MDL Data

Element	MDL Range, µg/L	Pooled MDL, µg/L	EPA MDL, µg/L
Sb	0.005 - 0.048	0.008	0.0097
Cd	0.002 - 0.077	---	0.025
Cu	0.008 - 0.148	---	0.087
Pb	0.0017 - 0.0129	---	0.015
Ni	0.009 - 0.109	---	0.33
Se	0.152 - 0.927	---	0.45
Ag	0.006 - 0.028	---	0.029
Tl	0.001 - 0.015	---	0.0079
Zn	0.043 - 0.187	---	0.14

--- = unable to pool MDL, required removal of excessive number of laboratories to pass Hald test for equality of variances.

Despite the instructions in the study statement of work that in accordance with 40 CFR Part 136, reported MDL to concentration ratios must lie within the range of 1 to 5, many of the laboratories reported MDLs where the ratio was outside this range. Such data are flagged in the tables. Laboratory 4 elected not to submit MDL data and the study coordinator waived the MDL requirement for this laboratory. Laboratory 7 submitted MDLs calculated using two different internal standards, rhodium and bismuth. The MDL data reported using rhodium as the internal standard are shown for all analytes except lead, where the data are reported using bismuth as the internal standard.

Table 2-3
Antimony Method Detection Limits

Lab	Results, µg/L										Mean, µg/L	s µg/L	n	MDL, µg/L	Ratio conc./ MDL
	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10					
1	0.0135	0.0173	0.0112	0.0141	0.0218	0.0177	0.0171				0.0161	0.0035	*7*	**0.011**	1.5
2	0.0284	0.0366	0.0371	0.0306	0.0267	0.0404	0.0319				0.0331	0.0050	*7*	**0.016**	2.1
3	0.0329	0.0329	0.0274	0.0273	0.0328	0.0288	0.0274				0.0299	0.0028	*7*	**0.009**	3.4
5	0.0265	0.0248	0.0229	0.0252	0.0274	0.0247	0.0248				0.0252	0.0014	*7*	**0.005**	5.6
6	0.0481	0.0429	0.0407	0.0456	0.0385	0.0379	0.0429	0.0308	0.0438	0.0370	0.0424	0.0037	*10*	**0.010**	4.1
7	0.0065	0.0018	0.0033	0.0045	0.0048	0.0003	0.0021				0.0033	0.0021	*7*	**0.007**	0.5
8	0.0889	0.0630	0.0568	0.0507	0.0434	0.0458	0.0561				0.0578	0.0153	*7*	**0.048**	1.2

Data from laboratory 8 were removed using Hald test for equality of variances. Pooled MDL for remaining data is 0.008 µg/L.

EPA MDL = 0.0097 µg/L

Values in shaded boxes did not meet the 40 CFR Part 136, Appendix B criteria of a ratio of between 1 and 5 for measured concentration to MDL.

Table 2-4
Cadmium Method Detection Limits

Lab	Results, µg/L										Mean, µg/L	s µg/L	n	MDL, µg/L	Ratio conc./ MDL
	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10					
1	0.0307	0.0349	0.0281	0.0351	0.0361	0.0290	0.0251				0.0313	0.0042	7	**0.013**	2.4
2	0.3105	0.3265	0.3409	0.3230	0.2907	0.3686	0.3188				0.3256	0.0244	7	**0.077**	4.2
3	0.0211	0.0168	0.0202	0.0217	0.0248	0.0203	0.0236				0.0212	0.0026	7	**0.008**	2.6
5	0.0535	0.0699	0.0730	0.0621	0.0627	0.0650	0.0646				0.0644	0.0062	7	**0.020**	3.3
6	0.0638	0.0410	0.0528	0.0454	0.0352	0.0432	0.0425	0.0322	0.0476	0.0542	0.0463	0.0093	10	**0.026**	1.8
7	0.0121	0.0122	0.0114	0.0132	0.0121	0.0107	0.0118				0.0119	0.0008	7	**0.002**	4.9
8	0.0132	0.0107	0.0132	0.0078	0.0143	0.0127	0.0238				0.0137	0.0050	7	**0.016**	0.9

Data set required removal of excessive number of laboratory data sets (labs 2, 6 & 7) to pass Hald test for equality of variances. Unable to calculate pooled MDL.

EPA MDL = 0.025 µg/L

Values in shaded boxes did not meet the 40 CFR Part 136, Appendix B criteria of a ratio of between 1 and 5 for measured concentration to MDL.

Table 2-5
Copper Method Detection Limits

Lab	Results, µg/L										Mean, µg/L	s µg/L	n	MDL, µg/L	Ratio conc./ MDL
	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10					
1	0.0386	0.0481	0.0472	0.0468	0.0464	0.0382	0.0577				0.0461	0.0066	7	**0.021**	2.2
2	0.3607	0.3419	0.3738	0.3462	0.3418	0.3493	0.4137				0.3611	0.0259	7	**0.081**	4.4
3	0.0355	0.0315	0.0248	0.0288	0.0301	0.0301	0.0328				0.0305	0.0033	7	**0.011**	2.9
5	0.2670	0.2570	0.2570	0.2500	0.2650	0.2650	0.2650				0.2609	0.0063	7	**0.020**	13.2
6	1.0342	1.0125	1.0164	0.9061	0.9665	1.0351	1.0629	1.0034	1.0601	1.0282	1.0048	0.0525	10	**0.148**	6.8
7	0.0729	0.0676	0.0689	0.0693	0.0676	0.0703	0.0649				0.0688	0.0025	7	**0.008**	8.8
8	0.0553	0.0495	0.0426	0.0540	0.0466	0.0483	0.0536				0.0500	0.0046	7	**0.014**	3.5

Data set required removal of excessive number of laboratory data sets (labs 2 & 6) to pass Hald test for equality of variances. Unable to calculate pooled MDL.

EPA MDL = 0.087 µg/L

Values in shaded boxes did not meet the 40 CFR Part 136, Appendix B criteria of a ratio of between 1 and 5 for measured concentration to MDL.

Table 2-6
Lead Method Detection Limits

Lab	Results, µg/L										Mean, µg/L	s µg/L	n	MDL, µg/L	Ratio conc./ MDL
	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10					
1	0.0234	0.0217	0.0235	0.0194	0.0220	0.0240	0.0211				0.0222	0.0016	7	**0.0051**	4.4
2	0.0674	0.0610	0.0704	0.0651	0.0648	0.0644	0.0646				0.0654	0.0029	7	**0.0091**	7.2
3	0.0215	0.0211	0.0192	0.0206	0.0204	0.0207	0.0216				0.0207	0.0008	7	**0.0025**	8.2
5	0.0608	0.0648	0.0685	0.0679	0.0612	0.0678	0.0661				0.0653	0.0032	7	**0.0100**	6.5
7	0.0164	0.0156	0.0156	0.0163	0.0168	0.0153	0.0160				0.0160	0.0005	7	**0.0017**	9.6
8	0.0873	0.0921	0.0951	0.0984	0.0980	0.0980	0.0971				0.0952	0.0041	7	**0.0129**	7.4

Laboratory 6 did not submit MDL data for lead.

Data set failed Hald test for equality of variances. Unable to calculate pooled MDL.

EPA MDL = 0.015 µg/L

Values in shaded boxes did not meet the 40 CFR Part 136, Appendix B criteria of a ratio of between 1 and 5 for measured concentration to MDL.

Table 2-7
Nickel Method Detection Limits

Lab	Results, µg/L										Mean, µg/L	s µg/L	n	MDL, µg/L	Ratio conc./ MDL
	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10					
1	0.0843	0.0835	0.0935	0.0905	0.0649	0.0687	0.0924				0.0825	0.0115	7	**0.036**	2.3
2	0.2330	0.2147	0.2076	0.2243	0.2110	0.2030	0.2049				0.2141	0.0110	7	**0.034**	6.2
3	0.0217	0.0217	0.0268	0.0233	0.0288	0.0263	0.0215				0.0243	0.0030	7	**0.009**	2.6
5	1.3350	1.3420	1.3660	1.3770	1.3490	1.3220	1.3450				1.3480	0.0185	7	**0.058**	23.2
6	0.9760	0.9316	0.9347	0.9863	0.9919	1.0141	1.0363	1.0033	1.0089	1.0249	0.9816	0.0385	10	**0.109**	9.0
7	0.4646	0.4637	0.4611	0.4685	0.4650	0.4533	0.4636				0.4628	0.0047	7	**0.015**	31.0
8	0.0345	0.0377	0.0371	0.0424	0.0348	0.0387	0.0415				0.0381	0.0030	7	**0.010**	4.0

Data set required removal of excessive number of laboratory data sets (labs 1,2, 5 & 6) to pass Hald test for equality of variances. Unable to calculate pooled MDL.

EPA MDL = 0.33 µg/L

Values in shaded boxes did not meet the 40 CFR Part 136, Appendix B criteria of a ratio of between 1 and 5 for measured concentration to MDL.

Table 2-8
Selenium Method Detection Limits

Lab	Results, µg/L										Mean, µg/L	s µg/L	n	MDL, µg/L	Ratio conc./ MDL
	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10					
1	0.3100	0.5160	0.5030	0.5780	0.9680	0.5450	0.4030				0.5461	0.2074	7	**0.652**	0.8
3	0.7030	0.8050	0.8210	0.8880	0.9560	0.7790	0.7680				0.8171	0.0830	7	**0.261**	3.1
5	1.3270	1.2600	1.3140	1.2880	1.5380	1.2060	1.3900				1.3319	0.1074	7	**0.337**	3.9
6	2.2374	2.1433	1.8820	2.0597	1.4324	1.4951	1.5892	2.1329	2.2270	1.6519	1.8342	0.3286	10	**0.927**	2.0
7	2.3601	2.3761	2.2401	2.2920	2.3283	2.2720	2.2945				2.3090	0.0485	7	**0.152**	15.2
8	0.4657	0.6169	0.4296	0.4670	0.5218	0.6048	0.6014				0.5296	0.0780	7	**0.245**	2.2

Laboratory 2 did not submit selenium MDL data.

Data set required removal of excessive number of laboratory data sets (labs 1 & 6) to pass Hald test for equality of variances. Unable to calculate pooled MDL.

EPA MDL = 0.45 µg/L

Values in shaded boxes did not meet the 40 CFR Part 136, Appendix B criteria of a ratio of between 1 and 5 for measured concentration to MDL.

Table 2-9
Silver Method Detection Limits

Lab	Results, µg/L										Mean, µg/L	s µg/L	n	MDL, µg/L	Ratio conc./ MDL
	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10					
1	0.0248	0.0271	0.0316	0.0283	0.0239	0.0264	0.0266				0.0270	0.0025	7	**0.008**	3.4
2	0.0646	0.0557	0.0673	0.0512	0.0619	0.0594	0.0523				0.0589	0.0061	7	**0.019**	3.1
3	0.0194	0.0188	0.0144	0.0224	0.0181	0.0174	0.0198				0.0186	0.0024	7	**0.008**	2.4
5	0.0315	0.0305	0.0277	0.0283	0.0314	0.0333	0.0298				0.0304	0.0020	7	**0.006**	4.9
6	0.0978	0.0870	0.0815	0.0669	0.0890	0.0760	0.0790	0.0696	0.0768	0.0796	0.0825	0.0100	10	**0.028**	2.9
7	0.0483	0.0447	0.0457	0.0467	0.0454	0.0423	0.0440				0.0453	0.0019	7	**0.006**	7.5
8	0.0947	0.1059	0.1101	0.1095	0.1134	0.1156	0.1100				0.1085	0.0068	7	**0.021**	5.1

Data set required removal of excessive number of laboratory data sets (labs 2, 6 & 8) to pass Hald test for equality of variances. Unable to calculate pooled MDL.

EPA MDL = 0.029 µg/L

Values in shaded boxes did not meet the 40 CFR Part 136, Appendix B criteria of a ratio of between 1 and 5 for measured concentration to MDL.

Table 2-10
Thallium Method Detection Limits

Lab	Results, µg/L										Mean, µg/L	s µg/L	n	MDL, µg/L	Ratio conc./ MDL
	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10					
1	0.0254	0.0238	0.0239	0.0228	0.0226	0.0234	0.0213				0.0233	0.0013	7	**0.004**	5.8
2	0.0173	0.0178	0.0179	0.0284	0.0260	0.0261	0.0255				0.0227	0.0048	7	**0.015**	1.5
3	0.0034	0.0027	0.0031	0.0028	0.0025	0.0034	0.0032				0.0030	0.0004	7	**0.001**	2.7
5	0.0292	0.0243	0.0251	0.0246	0.0256	0.0260	0.0275				0.0260	0.0017	7	**0.005**	4.7
6	0.0423	0.0451	0.0422	0.0422	0.0450	0.0436	0.0420	0.0397	0.0423	0.0418	0.0432	0.0014	10	**0.004**	11.2
7	0.0097	0.0099	0.0101	0.0104	0.0101	0.0095	0.0093				0.0099	0.0003	7	**0.001**	9.8
8	0.0398	0.0397	0.0401	0.0381	0.0407	0.0418	0.0422				0.0403	0.0014	7	**0.004**	9.4

Data set required removal of excessive number of laboratory data sets (labs 2, 3 & 7) to pass Hald test for equality of variances. Unable to calculate pooled MDL.

EPA MDL = 0.0079 µg/L

Values in shaded boxes did not meet the 40 CFR Part 136, Appendix B criteria of a ratio of between 1 and 5 for measured concentration to MDL.

Table 2-11
Zinc Method Detection Limits

Lab	Results, µg/L										Mean, µg/L	s µg/L	n	MDL, µg/L	Ratio conc./ MDL
	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10					
1	0.1240	0.1390	0.1550	0.1540	0.1210	0.1720	0.1490				0.1449	0.0182	7	**0.057**	2.5
2	0.5475	0.5205	0.5266	0.5098	0.5100	0.5801	0.5334				0.5326	0.0248	7	**0.078**	6.8
3	0.1000	0.1080	0.1030	0.1140	0.1540	0.1450	0.1540				0.1254	0.0245	7	**0.077**	1.6
5	1.0850	1.0950	1.1110	1.1250	1.1140	1.0730	1.2500				1.1219	0.0593	7	**0.186**	6.0
6	1.0418	1.0558	0.9119	0.9936	1.0473	1.0393	1.1299	1.0168	1.0875	1.0839	1.0314	0.0664	10	**0.187**	5.5
7	0.2314	0.2166	0.2191	0.2261	0.2466	0.2143	0.2478				0.2288	0.0138	7	**0.043**	5.3
8	0.5231	0.5268	0.5569	0.5295	0.5444	0.5375	0.4940				0.5303	0.0198	7	**0.062**	8.5

Data set required removal of excessive number of laboratory data sets (labs 5 & 6) to pass Hald test for equality of variances. Unable to calculate pooled MDL.

EPA MDL = 0.14 µg/L

Values in shaded boxes did not meet the 40 CFR Part 136, Appendix B criteria of a ratio of between 1 and 5 for measured concentration to MDL

2.2 Phase 2 – Sample Collection and Preparation

In Phase 2 of the study , EPA's referee laboratory, Frontier Geosciences Inc., collected, prepared and distributed the study samples. The referee laboratory also performed background metals analyses and stability analyses of the samples. The sample matrices were collected in accordance with the procedures outlined in "Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels" (EPA 821-R-96-008, July 1996). The background and stability analyses were performed according to EPA Method 1638.

2.2.1 Sample Collection and Preparation

According to Frontier Geosciences (FGS), the sample matrices were collected in Teflon carboys, the samples were spiked and split in high density polyethylene (HDPE) carboys and the samples were shipped to the participants in HDPE bottles. FGS reported that each of the carboys and bottles used in the study was cleaned in the following manner:

1. Soaked for 24 hours in 25% nitric acid.

2. Emptied and rinsed copiously with reagent water.

3. Filled with 0.5% nitric acid and allowed to stand 24 hours in a clean hood.

4. Emptied and rinsed copiously with reagent water.

5. Filled with 0.5% nitric acid and placed in an oven overnight.

6. Emptied and rinsed three times with reagent water.

7. Dried in a clean hood and stored.

The sample matrices collected in the field included filtered freshwater and filtered and unfiltered secondary treated effluent from a municipal wastewater treatment plant. The reagent grade water sample matrix was collected in the referee laboratory's facility. After preparation of each set of individual samples of a particular matrix/concentration level, the samples were labeled with their identification and tracking numbers and stored together in plastic ice chests until they were distributed to the study participant laboratories.

The reagent grade water matrix was dispensed directly from the referee laboratory's Milli-Q® reagent water system which triple deionized the water, passed it through activated carbon and then through a 0.2 micron (μm) filter. The required volume of water for a particular spiking level was dispensed into a high density polyethylene (HDPE) carboy, preserved with nitric acid, spiked with the required amounts of standards, mixed by shaking, and then dispensed into individual HDPE sample bottles.

The reagent grade water sample sets consisted of four pairs of duplicate samples and one pair of Youden paired samples. A Youden pair (2) is a pair of samples of similar but not identical

concentration. ASTM D 2777 "Standard Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D-19 on Water," which is the basis of all of EPRI's previous method validation studies, utilizes a Youden-pair design. The purpose of this design is to eliminate bias due to the analyst's tendency to make known replicates match. For convenience, ASTM D 2777 permits up to half of the pairs in a study to be duplicates. Eighty percent of the sample pairs in the reagent grade water sample sets were duplicates: thus the reagent grade water sample sets were not in compliance with ASTM D 2777.

The freshwater sample was collected from Lake Washington near Seattle, Washington in December 1998. The sample was collected into Teflon carboys by pumping lake water through an acid cleaned, 0.45 μm in-line filter and preserved with nitric acid. Individual samples were prepared in a manner similar to that described for reagent grade water. The freshwater sample set consisted of one duplicate pair of samples and three Youden pairs of samples, thus fulfilling the requirements of ASTM D 2777.

Plant personnel provided the referee laboratory with unfiltered, secondary treated, municipal wastewater effluent collected into Teflon bottles. The referee laboratory filtered half of the collected matrix through 0.45 μm filters to prepare the filtered effluent samples. The remainder was not filtered and was used to generate the unfiltered effluent samples. The bulk samples were preserved with nitric acid. The individual samples were prepared in a manner similar to that described for the reagent grade water and spiked with amounts of the study analytes selected by the EPA. The filtered and unfiltered effluent samples were deemed to represent two separate matrices since the filtration step altered the matrix. Each filtered effluent sample set and each unfiltered effluent sample set consisted of one pair of duplicate samples; thus, neither sample set was in compliance with ASTM D 2777 for validation of the effluent matrix.

2.2.2 Test Matrix Characterization

The EPA did not perform general background characterization of the study matrices. Instead, the referee laboratory was instructed to analyze the matrices only for the study analytes. General water quality information for Lake Washington was found at a website hosted by the Water and Land Division of the King County, Washington Department of Natural Resources. Table 2-12 summarizes data from the website for December 1998 at the site closest to the collection site. Values are estimates as only plots are presented on the site, not raw data.

Table 2-12
Background Constituents of Lake Washington Water

Analyte	Units	Freshwater Concentration
Dissolved Oxygen	mg/L	11
pH	---	7.5
Conductivity	μmhos/cm	90
Total Nitrogen	mg/L	0.4
Total Phosphorus	mg/L	0.01

2.2.3 Verification of Initial Sample Concentration and Stability

Tables 2-13 through 2-16 summarize the background concentrations, spike concentrations and the results of the verification and stability analyses of the Method 1638 study analytes reported by the referee laboratory. The "true" concentrations listed in the tables are based on the referee's initial background analyses plus the added spikes. The referee laboratory did not provide analysis results for the unspiked background reagent grade water. For purposes of verification and stability, the reagent grade water background is assumed to be zero. Calculation of the true concentrations of the samples from the study data is described in Section 4.2.1.

The two lowest spikes of antimony into reagent grade water exhibited poor stability when analyzed by the referee laboratory approximately two months after preparation, as did the 0.2 and 0.24 μg/L spikes of silver into reagent grade water and the 0.07, 0.08, 0.3 and 0.36 μg/L silver spikes into freshwater. For the unfiltered effluent, the silver recovery during both the verification and stability analyses was only 52.1% of that originally measured by the referee laboratory. All other sample spikes showed suitable stability.

Table 2-13
Verification and Stability Data for Reagent Grade Water

Element	Sample ID	Background	Spike	"True"	Verification	% Recovery	Stability	% Recovery
Sb	Bkg	0						
Sb	TRW A		0.1	0.1	0.082	82.0%	0.008	8.0%
Sb	TRW B		0.12	0.12	0.106	88.3%	0.068	56.7%
Sb	EPA-Low		1	1	0.968	96.8%	0.908	90.8%
Sb	EPA-Med		5	5	4.88	97.6%	4.73	94.6%
Sb	EPA-High		20	20	19.7	98.5%	19.1	95.5%
Cd	Bkg	0						
Cd	TRW A		0.05	0.05	0.043	86.0%	0.050	100.0%
Cd	TRW B		0.06	0.06	0.050	83.3%	0.058	96.7%
Cd	EPA-Low		0.1	0.1	0.095	95.0%	0.096	96.0%
Cd	EPA-Med		1	1	0.979	97.9%	0.964	96.4%
Cd	EPA-High		10	10	9.58	95.8%	9.57	95.7%
Cu	Bkg	0						
Cu	TRW A		0.2	0.2	0.202	101.0%	0.21	106.5%
Cu	TRW B		0.24	0.24	0.268	111.7%	0.26	106.3%
Cu	EPA-Low		0.5	0.5	0.538	107.6%	0.512	102.4%
Cu	EPA-Med		2	2	2.14	107.0%	2.05	102.5%
Cu	EPA-High		20	20	20.5	102.5%	20.2	101.0%
Pb	Bkg	0						
Pb	TRW A		0.03	0.03	0.023	76.7%	0.033	110.0%
Pb	TRW B		0.036	0.036	0.034	94.4%	0.037	102.8%
Pb	EPA-Low		0.1	0.1	0.086	86.0%	0.090	90.0%
Pb	EPA-Med		0.5	0.5	0.402	80.4%	0.480	96.0%
Pb	EPA-High		5	5	4.96	99.2%	4.91	98.2%
Ni	Bkg	0						
Ni	TRW A		0.5	0.5	0.481	96.2%	0.49	98.2%
Ni	TRW B		0.6	0.6	0.581	96.8%	0.60	100.7%
Ni	EPA-Low		1	1	1.08	108.0%	1.01	101.0%
Ni	EPA-Med		10	10	10.5	105.0%	10.2	102.0%
Ni	EPA-High		100	100	99.6	99.6%	100	100.0%
Se	Bkg	0						
Se	TRW A		1	1	0.754	75.4%	1.280	128.0%
Se	TRW B		1.2	1.2	0.850	70.8%	1.64	136.7%

Table 2-13 (continued)
Verification and Stability Data for Reagent Grade Water

Element	Sample ID	Background	Spike	"True"	Verification	% Recovery	Stability	% Recovery
Se	EPA-Low		5	5	4.70	94.0%	5.10	102.0%
Se	EPA-Med		20	20	20.2	101.0%	20.5	102.5%
Se	EPA-High		100	100	98.8	98.8%	104	104.0%
Ag	Bkg	0						
Ag	EPA-Low		0.1	0.1	0.107	107.0%	0.094	94.0%
Ag	TRW A		0.2	0.2	0.213	106.5%	-0.052	-26.0%
Ag	TRW B		0.24	0.24	0.253	105.4%	-0.009	-3.8%
Ag	EPA-Med		1	1	1.01	101.0%	1.100	110.0%
Ag	EPA-High		10	10	10.3	103.0%	11.300	113.0%
Tl	Bkg	0						
Tl	TRW A		0.04	0.04	0.040	100.0%	0.043	107.5%
Tl	TRW B		0.05	0.05	0.050	100.0%	0.052	104.0%
Tl	EPA-Low		0.1	0.1	0.1050	105.0%	0.097	97.0%
Tl	EPA-Med		0.5	0.5	0.5070	101.4%	0.490	98.0%
Tl	EPA-High		2	2	2.050	102.5%	2.00	100.0%
Zn	Bkg	0						
Zn	EPA-Low		0.5	0.5	0.447	89.4%	0.348	69.6%
Zn	TRW A		2	2	1.93	96.5%	2.39	119.5%
Zn	TRW B		2.4	2.4	2.39	99.6%	2.64	110.0%
Zn	EPA-Med		5	5	4.96	99.2%	4.82	96.4%
Zn	EPA-High		50	50	49.3	98.6%	49.7	99.4%

All units μg/L unless otherwise specified
Analysis Dates:
Verification: EPA samples (4/29/99), TRW samples (5/11/99)
Stability: EPA & TRW (6/29/99)

Table 2-14
Verification and Stability Data for Freshwater

Element	Sample ID	Background	Spike	"True"	Verification	% Recovery	Stability	% Recovery
Sb	Bkg	0.126						
Sb	EPA		0	0.126	0.153	121.4%	0.101	80.2%
Sb	1A		0.08	0.206	0.197	95.7%	0.168	81.6%
Sb	1B		0.12	0.246	0.231	93.9%	0.203	82.5%
Sb	2A		0.28	0.406	0.381	93.8%	0.355	87.4%
Sb	2B		0.38	0.506	0.463	91.4%	0.446	88.2%
Sb	3A		0.68	0.806	0.765	94.9%	0.739	91.7%
Sb	3B		0.88	1.006	0.947	94.1%	0.922	91.7%
Cd	Bkg	0						
Cd	1A		0	0	-0.008	---	0.002	---
Cd	1B		0	0	-0.007	---	0.001	---
Cd	2A		0.04	0.04	0.032	79.0%	0.040	99.5%
Cd	2B		0.05	0.05	0.041	81.2%	0.049	98.4%
Cd	3A		0.12	0.12	0.105	87.8%	0.116	96.9%
Cd	3B		0.14	0.14	0.131	93.2%	0.137	97.8%
Cd	EPA		0.25	0.25	0.244	97.6%	0.242	96.8%
Cu	Bkg	1.03						
Cu	EPA		0	1.03	1.21	117.5%	1.12	108.7%
Cu	1A		0.5	1.53	1.57	102.8%	1.56	102.0%
Cu	1B		0.7	1.73	1.73	100.2%	1.71	98.8%
Cu	2A		1.5	2.53	2.51	99.2%	2.52	99.6%
Cu	2B		2	3.03	3.04	100.5%	3.09	102.0%
Cu	3A		3.5	4.53	4.44	98.0%	4.45	98.2%
Cu	3B		4	5.03	4.91	97.5%	4.94	98.2%
Pb	Bkg	0.0128						
Pb	1A		0	0.0128	0.020	157.8%	0.023	178.9%
Pb	1B		0	0.0128	0.020	158.6%	0.021	167.2%
Pb	2A		0.04	0.0528	0.055	104.4%	0.058	109.3%
Pb	2B		0.05	0.0628	0.067	106.5%	0.068	107.6%
Pb	EPA		0.19	0.2028	0.197	97.1%	0.205	101.1%
Pb	3A		0.3	0.3128	0.306	97.8%	0.313	100.0%
Pb	3B		0.35	0.3628	0.361	99.4%	0.363	100.0%
Ni	Bkg	0.31						
Ni	1A		0	0.31	0.229	74.0%	0.291	93.7%
Ni	1B		0	0.31	0.234	75.5%	0.290	93.5%
Ni	2A		1.3	1.61	1.46	90.8%	1.58	98.0%

Table 2-14 (continued) Verification and Stability Data for Freshwater

Element	Sample ID	Background	Spike	"True"	Verification	% Recovery	Stability	% Recovery
Ni	2B		1.5	1.81	1.70	93.7%	1.71	94.4%
Ni	3A		3	3.31	3.10	93.8%	3.17	95.8%
Ni	3B		3.7	4.01	3.83	95.6%	3.94	98.2%
Ni	EPA		5.7	6.01	6.14	102.2%	6.05	100.7%
Se	Bkg	0.43						
Se	1A		0	0.43	0.063	14.7%	0.322	75.0%
Se	1B		0	0.43	0.086	20.1%	0.299	69.4%
Se	2A		0.6	1.03	0.669	64.9%	0.940	91.3%
Se	2B		0.8	1.23	0.945	76.8%	1.245	101.2%
Se	3A		1.6	2.03	1.69	83.1%	2.16	106.4%
Se	3B		2	2.43	2.04	83.9%	2.67	109.8%
Se	EPA		3.6	4.03	3.71	92.1%	3.93	97.5%
Ag	Bkg	0						
Ag	1A		0	0	0.045	---	-0.057	---
Ag	1B		0	0	0.034	---	-0.060	---
Ag	2A		0.07	0.07	0.089	127.6%	-0.030	-43.0%
Ag	2B		0.08	0.08	0.101	125.9%	-0.020	-24.6%
Ag	EPA		0.20	0.20	0.245	122.5%	0.211	105.5%
Ag	3A		0.3	0.3	0.301	100.4%	0.080	26.6%
Ag	3B		0.36	0.36	0.378	105.1%	0.079	21.8%
Tl	Bkg	0.001						
Tl	1A		0	0.001	0.0006	60.0%	0.0073	730.0%
Tl	1B		0	0.001	-0.0005	-50.0%	0.0067	670.0%
Tl	2A		0.02	0.021	0.017	79.5%	0.024	113.3%
Tl	2B		0.023	0.024	0.022	89.6%	0.028	115.8%
Tl	EPA		0.06	0.061	0.063	103.3%	0.059	96.7%
Tl	3A		0.1	0.101	0.098	96.8%	0.099	97.5%
Tl	3B		0.12	0.121	0.117	97.0%	0.115	95.1%
Zn	Bkg	0.75						
Zn	1A		0	0.75	0.826	110.1%	0.878	117.1%
Zn	1B		0	0.75	0.867	115.6%	0.891	118.8%
Zn	2A		0.13	0.88	0.932	105.9%	0.899	102.2%
Zn	2B		0.33	1.08	1.15	106.5%	1.12	103.4%
Zn	EPA		0.53	1.28	1.41	110.2%	1.49	116.4%
Zn	3A		0.82	1.57	1.62	103.4%	1.60	101.7%
Zn	3B		1.22	1.97	2.03	102.8%	2.10	106.8%

All units µg/L unless otherwise specified. --- = unable to calculate percent recovery when the background is zero.
Analysis Dates: Verification: EPA samples (4/29/99) TRW samples (5/11/99). Stability: EPA & TRW (6/29/99)

Table 2-15
Verification and Stability Data for Filtered Effluent (FEF)

Element	Sample ID	Background	Spike	"True"	Verification	% Recovery	Stability	% Recovery
Sb	FEF	0.956	0.03	0.986	1.02	103.4%	0.981	99.5%
Cd	FEF	0.108	0.2	0.308	0.315	102.3%	0.321	104.2%
Cu	FEF	13.8	0	13.8	14.6	105.8%	13.3	96.4%
Pb	FEF	0.481	0.05	0.531	0.443	83.4%	0.519	97.7%
Ni	FEF	2.6	4.4	7	6.56	93.7%	6.51	93.0%
Se	FEF	0.3	4.7	5	4.49	89.8%	4.95	99.0%
Ag	FEF	0.478	0	0.478	0.468	97.9%	0.452	94.6%
Tl	FEF	<0.002	0.2	0.2	0.186	93.0%	0.180	90.0%
Zn	FEF	43.25	0	43.25	46.8	108.2%	47.0	108.7%

All units μg/L unless otherwise specified.
Analysis Dates:
Verification: 4/29/99
Stability: 6/29/99

Table 2-16
Verification and Stability Data for Unfiltered Effluent (UEF)

Element	Sample ID	Background	Spike	"True"	Verification	% Recovery	Stability	% Recovery
Sb	UEF	0.411	9.6	10.011	9.50	94.9%	9.40	93.9%
Cd	UEF	0.125	0.38	0.505	0.505	100.0%	0.495	98.0%
Cu	UEF	15.1	0	15.1	15.9	105.3%	14.6	96.7%
Pb	UEF	1.11	0	1.11	1.00	90.1%	1.03	92.8%
Ni	UEF	2.71	5.3	8.01	7.20	89.9%	7.31	91.3%
Se	UEF	0.27	5.7	5 .97	5.22	87.4%	6.02	100.8%
Ag	UEF	2.88	0	2.88	1.50	52.1%	1.50	52.1%
Tl	UEF	<0.002	0.9	0.9	0.887	98.6%	0.835	92.8%
Zn	UEF	48.47	0	48.47	52.5	108.3%	52.9	109.1%

All units μg/L unless otherwise specified.
Analysis Dates:
Verification: 4/29/99
Stability: 6/29/99

2.3 Phase 3 – Sample Analysis

The samples were shipped to the laboratories around the end of October 1997. Participants were instructed to analyze the reagent grade water samples for total recoverable metals and all other samples for dissolved metals. Analyses were completed by the first week of December 1997. All eight laboratories that agreed to participate in the study completed the analyses and submitted their results to the sample control center (SCC) in electronic spreadsheets. After data review and

resolution of discrepancies, the SCC forwarded the compiled raw data to EPRI. The data analysis is described in Chapter 4.

2.4 References

1. Hald, A., *Statistical Theory with Engineering Applications*, John Wiley & Sons, Inc., pp. 290-299, 1952.

2. American Society for Testing and Materials, "Standard Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water", D 2777-96.

3
STATISTICAL ANALYSIS PROGRAM

Data for this method performance study were analyzed by STATCALC, a data preparation and statistical analysis program developed by EPRI. STATCALC calculates precision and bias according to the procedures contained in the ASTM D 2777 method. Figures 3-1, 3-2 and 3-3 provide an overview of the program.

3.1 STATCALC Collaborative Statistical Analysis Program

STATCALC is a set of software programs designed to operate in the personal or desktop computer environment, commonly known as a “PC”. STATCALC was designed to analyze data resulting from collaborative analytical method validation studies. Such studies are conducted, both by government regulatory bodies and by private industry, to assess the precision and bias of analytical methods. The program uses procedures based on and developed using sound scientific principles, procedures that have been demonstrated and verified with actual field studies.

STATCALC was conceived and developed as a computer-assisted implementation of the preparatory and statistical information processing required by a well-known and successful standard for collaborative laboratory studies, ASTM D 2777-86 (1). This standard, while providing detailed examples for collaborative studies with replicate experimental design, also provides for studies based upon Youden pairs. The experimental design for this method performance study uses the STATCALC subprogram "YOUDENPR", can handle datasets of up to 100 laboratories, with five Youden pair observations per laboratory for each analyte and matrix.

Over the course of the EPRI Analytical Methods Qualification (AMQ) studies, the STATCALC program was modified to conform to the latest version of ASTM D 2777. The version used to reduce the data for the EPA Method 1638 validation study is compliant with ASTM D 2777-96 (2).

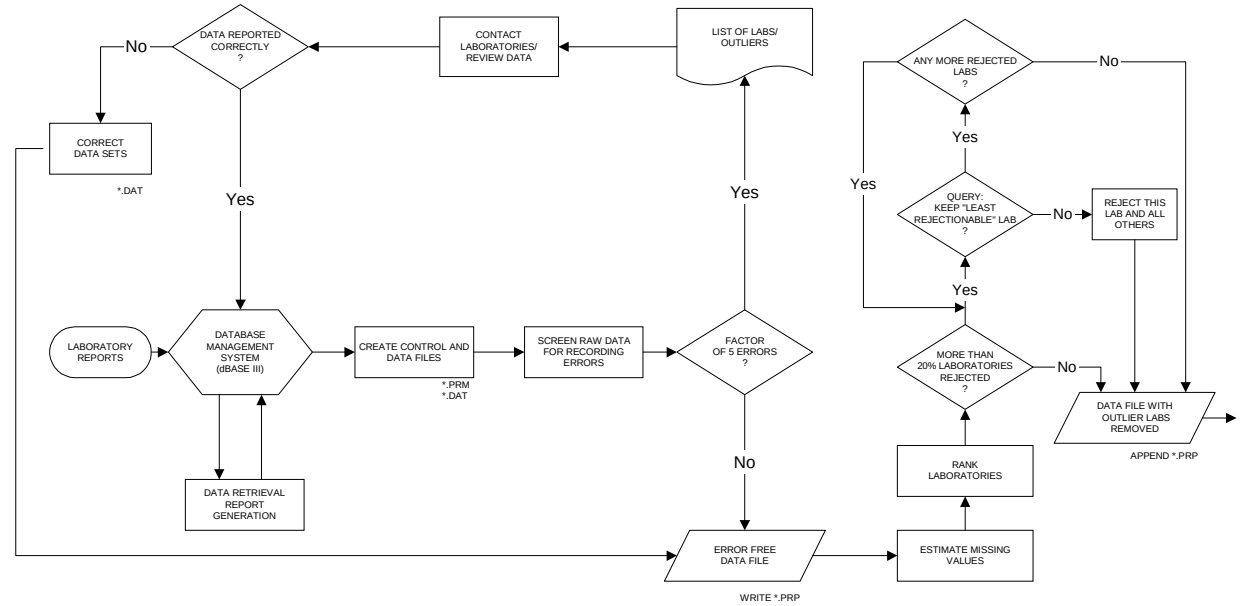

Figure 3-1
STATCALC data screening/lab ranking

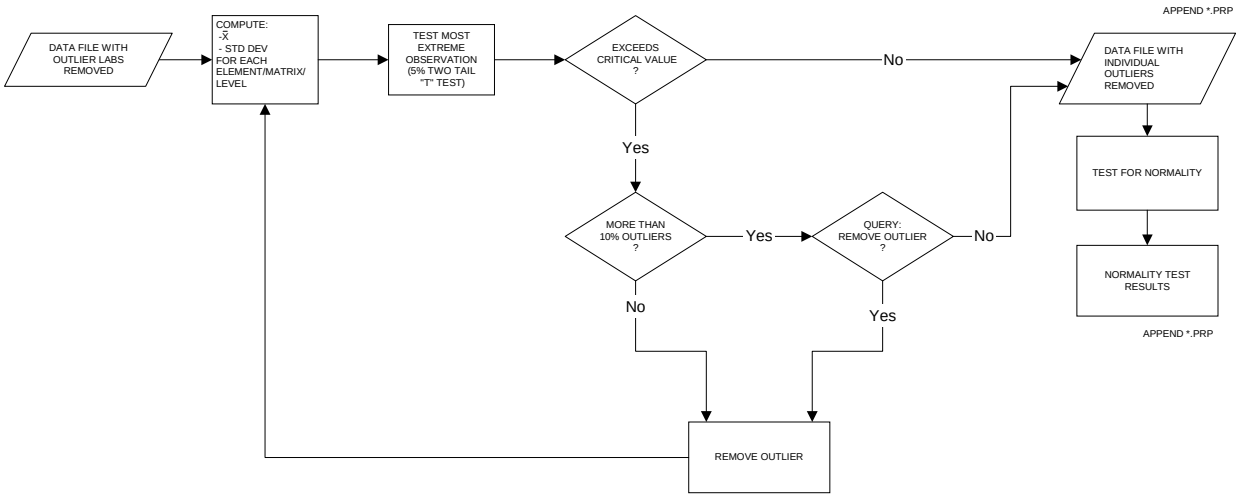

Figure 3-2
STATCALC outlier testing

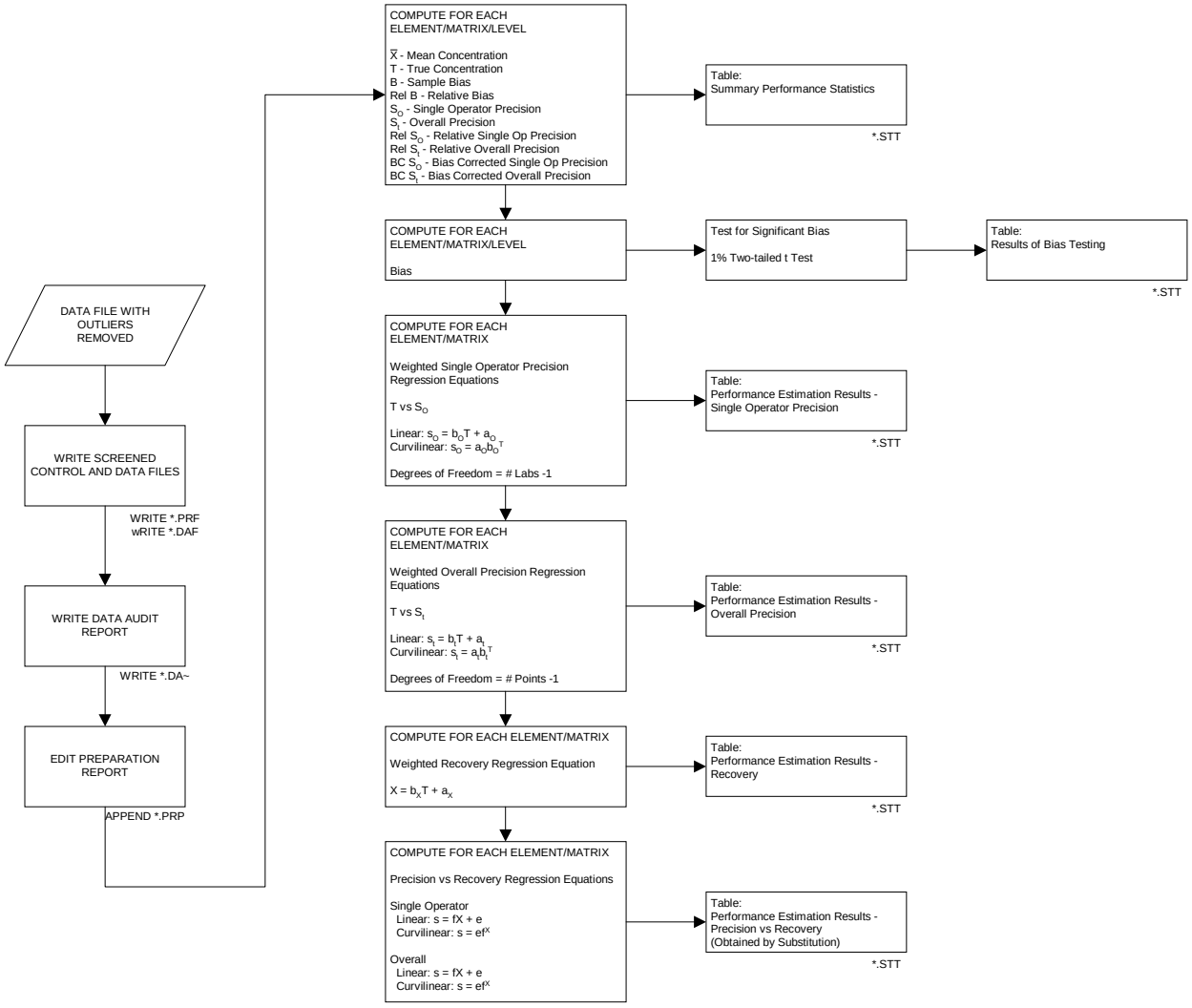

Figure 3-3
STATCALC statistical processing

Program Capacities:

Number of Elements	1
Number of Laboratories	100
Number of Youden Pairs	5
Number of Replicate Concentration Levels	9
Number of Replicates per Level	7
Number of Matrixes per Run	1
Number of Data Sets	1

3.1.1 Notation for STATCALC

Users may freely specify, under the limitations of the Personal Computer operating system, a file name "root" of up to eight characters which precedes the "." which separates the file name root from its three letter "extension." STATCALC uses this user specified root, referred to as "*" in the following discussions, to systematically name all of the intermediate and final input and output files created during processing with this same root name. The following files, with the exception of the data (*.DAT) and parameter (*.PRM) files which the user must create for each analyte/matrix, are created during STATCALC data processing. They are discussed in more detail in the following sections.

*.PRM	Of the two files that STATCALC must have, the first is a control file called the parameter (*.PRM) file. This file contains information about the type of analyte under study, the analytical method being used, the name of the matrix, the spike levels, units of measurement, and so forth.
*.DAT	The second file is the data (*.DAT) file which contains the laboratories' reported results and associated identification indexes for the analyte/matrix described in the parameter file.
*.DA~	Data that are read initially from the *.DAT file are echoed to an intermediate file (*.DA~), which provides an audit record of the actual data the program analyzes.
*.PRF	This file is a copy of the parameter file used for actual final processing, and amounts to a reconciliation of the *.PRM and *.DAT files.
*.DAF	After all checking has been accomplished, a file containing only non-rejected data is created (*.DAF). This file represents the final data set. It is used in the performance evaluation section of the program where precision and bias are estimated and reported.

*.PRP	The data preparation file, *.PRP, tracks the data preparation steps. After reading the *.PRM and *.DAT files, STATCALC first screens the data and reports possible transcription or calculation errors in the raw data file, *.DAT. Results of the lab ranking test as well as results of individual outlier testing are reported in the *.PRP file. The individual outlier test assumes that the underlying statistical population follows a normal probability model, and the results of normality testing are recorded in this file.
*.STT	The results of the summary statistic calculations are written in the statistical processing output file, *.STT, as are results of bias testing, estimation and performance regressions.
*.RGC	A file of regression coefficients is produced for use as input to other programs.
*.MEF *.MDT	Matrix effects testing (optional for pairs of matrices) produces two files, *.MEF for results of the test, and *.MDT for internal program use.

3.1.2 Data Notation

An observation (also called a result, recovery, measurement, observed value, data value, or data point) is the actual numerical value of the analyte concentration as reported by the laboratory. The letter "X" is used as generic notation for these reported values.

When appropriate, or necessary, the subscript "a" will be used to denote measurements for different analytes, the subscript "m" for different matrixes, the subscript "c" for different concentration levels, the subscript "l" for different laboratories, the subscript "r" for different replicates. In all cases, unless noted otherwise, the lower limits are 1 and the upper limits are the corresponding upper case letters of the respective categories of subscripts, that is:

Observation Hierarchy
Analyte: (a = 1,2,...,A)
Matrix: (m = 1,2,...,M)
Concentration Level: (c = 1.,2,...,C)
Laboratory: (l = 1,2,...,L)
Replicates: (r = 1,2,...,R)
Youden Pairs: r=1

The notation for a completely specified observation would be:

Observation (Recovery)
X_{amclr}

The letter "T" is used as generic notation for the true concentration of the analyte. Since there are "C" different concentration levels, the set of concentration levels (T) and incremental spikes (ΔT_c) would be denoted:

True Analyte Spikes and Concentrations
ΔT_c, c=1,2,...,C
T_c, c=1,2,...,C

Subscripts used in the text following are suppressed when not needed, as in (X_{clr}). This is usually sufficient since with one analyte, a and A equal 1, and with a single matrix, m and M equal 1.

3.2 Data Validation

Data are screened and examined in various ways before being placed in a data bank and used for estimating population parameters or making decisions. To this end, data that are read initially from the DAT file are echoed to an intermediate file (*.DA~) and, optionally, to the screen. This operation provides an audit record of the actual data the program analyzed. It also serves to isolate the user provided data file (*.DAT) from program alteration and provide data to the next step in the analysis.

3.2.1 "Zero" Data

"Zero" data are not noted by the program as unusual and are treated in the same way as other nonzero measurements.

3.2.2 Censored or "Less Than" Data

Measurements which were reported as "less than" a particular value are handled by STATCALC as follows. A single character "flag field" is provided in the *.DAT (input data) file for each reported value. The program checks for a "<" symbol in the flag field and, optionally, ignores the flag (treats the value as if it were measured at the censoring level) or substitutes a 0.0, depending on the result of a query to the user. The program, as written, treats such values as being reported at the upper limit or lower limit (0.0) of the possible range.

3.2.3 "Negative" Data

"Negative" observations are values reported as negative by a laboratory. They are handled by the program as valid data values. That is to say, the program does not preclude processing negative reported values.

3.3 "Factor of 5" Data Screening

Mistranscriptions of data such as transposition errors, misplaced decimal points, or inadvertently misreported units will interfere with the statistical estimates of precision and bias produced by STATCALC. To guard against this, the data are initially screened for patently obvious inconsistencies. STATCALC does not remove any data during this screening procedure but flags it for the user's review.

3.3.1 The Screening Procedure

After reading the *.PRM (processing control file) and *.DAT files, the program screens each measurement of a given analyte in a given matrix for possible transcription or clerical errors by two methods. If a value is more than five times the mean result of values from all laboratories at that concentration level, or less than one-fifth that mean, it is reported as a questionable observation.

Since the above method will not work with data sets having negative values, the program also computes the Mean Absolute Deviation, *MAD*, for each level, i.e.,

$$MAD = \frac{\sum \left| X - \overline{X} \right|}{N} \qquad \text{(eq. 3-1)}$$

where $\overline{X}$ is the sample mean of all N results at a given level. It then calculates the individual deviation

$$\left| X - \overline{X} \right| \qquad \text{(eq. 3-2)}$$

for each value and checks if

$$\frac{\left| X - \overline{X} \right|}{MAD} \qquad \text{(eq. 3-3)}$$

is greater than 5.0.

The program reports all questionable observations and their associated identification information to the user in the data preparation result file, *.PRP. If no such observations are encountered, that fact is also recorded in the data preparation result file. No data are removed by the screening tests. The user must evaluate each questionable observation and correct any errors in the data in the *.DAT file. If any such corrections are necessary, the data processing must be initiated again using the corrected *.DAT file. After all individual values have been screened, processing of the data in the *.DAT file proceeds to the lab ranking routine.

3.4 Lab Ranking

After the data set is screened initially for transcription errors and the like, it is subjected to a statistical procedure designed to detect laboratories whose results as a whole are either so consistently high or so consistently low, compared to the other laboratories, as to be considered unreasonable. Results from such a laboratory should be rejected for a given analyte and matrix. If the laboratory's data are allowed to remain in the analysis, it will materially affect the precision and bias estimates, and the results of the analysis will not be representative.

3.4.1 Rationale for the Test

If a particular laboratory is producing exceedingly biased results that are consistently lower or higher than the other laboratories, and if the results from that laboratory are ranked among the other laboratories' results, the suspect laboratory will tend to get either consistently high or consistently low ranks. The measurements for a laboratory which has no such large systematic bias should rank randomly among the other laboratories' results, receiving some high ranks and some low ones. The statistical procedure which is used to test for outlying laboratories uses the sums of the several laboratories' ranks over several concentrations as the test statistic. If any given laboratory's sum is too large or too small, that laboratory may be excluded from the analysis.

STATCALC operates with a limit of no more than 20 percent of the laboratories removed by laboratory ranking. If the program detects that more than 20 percent of the laboratories need to be considered for rejection from the study, the operator is given a choice to keep the data from a laboratory that has failed the laboratory ranking test.

3.4.2 Discussion of the Test

The results of each laboratory at each concentration level are ranked in ascending order with the lowest value assigned “1” and the highest value the rank of “L”. If one or more observations are tied, each laboratory is given the average of the sum of the ranks of the equal observations. Rank sums are computed by summing across all concentrations for each laboratory. If the minimum rank sum is less than the lower critical value, or the maximum rank sum is larger than the upper critical value, then laboratories with those extreme sums are rejected. If no more than 20 percent of the laboratories are rejected, and if the operator does not retain data from a laboratory that failed the laboratory ranking test, their data are excluded. Their data are not used in any further analysis and are flagged with an "r" in the data validation file (*.DA~). The quantities needed for the lab ranking test and the test's results are reported in *.PRP in the laboratory ranking section of the file.

As a general rule, the analyst must have a complete set of data to perform the laboratory ranking procedure. That is, every laboratory must have at least one observation at each concentration level for replicate data or both members of each pair for the Youden pair analytical design. In the case of missing data, an estimation procedure is used. If there are measurements reported for at least three different concentration levels (one member of each of three pairs for Youden paired data) for a given laboratory, the ranking subroutine fits a simple linear regression to the actual

values reported for that laboratory. The estimation procedure returns estimated recoveries from its regression line to be used in place of the missing observations. Values substituted for missing observations are used only in the laboratory ranking routine are discarded before subsequent data processing.

If there are fewer than 15 laboratories involved, the critical values for the rank sums are those found in ASTM D 2777-96 (2). If there are more than 15 laboratories, the critical values for the laboratory rank sums are computed using an approximating uniform probability distribution to calculate the quantity Q below. The data for a laboratory are rejected and not used in subsequent calculations if the laboratory's rank sum satisfies either of the inequalities below:

$$Rank\,Sum < C + Q \qquad \text{(eq. 3-4)}$$

or

$$Rank\,Sum < C \times L - Q \qquad \text{(eq. 3-5)}$$

where

C = Number of Concentration Levels in the study
L = Number of Laboratories

and

$$Q = L\left(\frac{\alpha C!}{2L}\right)^{1/C} - \left(\frac{C+1}{2}\right) \qquad \text{(eq. 3-6)}$$

where

α = Significance Level of the Test = 0.05 (Default)

3.4.3 Justification for Use of the Test

This laboratory ranking procedure in ASTM Standard D 2777 is the standard in general use for evaluating data from interlaboratory studies .

3.5 Testing for Outliers

The next step in the data analysis is to check those data remaining following rejection of laboratories for individual outlying observations, that is, individual recoveries for a given concentration that differ markedly from the other observations for that concentration, i.e., by an amount that cannot be explained solely by sampling variability. Such observations occur when a major disturbance (such as a radical deviation from standard procedure or an error in calculation

or recording of the value) impacts the measuring process so as to grossly affect the accuracy of the measurement. These observations should be excluded from the subsequent analysis provided that either a) it is known that a substantial departure from prescribed experimental procedure has occurred, or b) a valid statistical criterion shows them to be outlying.

Because the program may exclude too many values from small data sets, a limit of 10 percent is imposed on the number of the values automatically excluded by this test. After 10 percent of the outliers have been removed, the operator is queried for the choice of retaining or discarding data that failed the outlier test.

3.5.1 Rationale for the Test

It is reasonable to expect variability from one measurement to another for a given concentration because of a certain amount of imprecision in the measurement process. However, when a major discrepancy between measurements occurs, the extreme value is no longer representative of the regular measurement process and should be eliminated from the collaborative analysis. The judgment as to whether a given observed recovery is so disturbed is based on determining how far that result lies from the average of all measured results for that concentration. A two-sided T test is used to determine whether the measurement lies too far from the average and should be rejected.

3.5.2 Discussion of the Test

As mentioned above, the statistical procedure which is used in STATCALC for individual outlier testing is based on comparing each individual recovery to the average of all laboratories' recoveries at a given concentration level in a given matrix.

Individual Outlier Detection Procedure.

Step 1 - The distance of each individual recovery from the overall average is compared to the average distance of all recoveries as measured by the overall (also called "total", or "multiple laboratory operational") standard deviation, s_t.

For a given concentration level, with L laboratories and one replicate (for Youden pairs, the test is done for each pair level), the average recovery is computed as

$$\overline{X} = \frac{\sum_{l=1}^{L} X_l}{L} \qquad \text{(eq. 3-7)}$$

and the overall standard deviation as

$$s_t = \sqrt{\frac{SS_t}{L-1}} \qquad \text{(eq. 3-8)}$$

where

$$SS_t = \sum_{l=1}^{L} X_l^2 - \frac{\left(\sum_{l=1}^{L} X_l\right)^2}{l} \qquad \text{(eq. 3-9)}$$

Step 2 - To run the test, the distance of the most extreme observation from the overall average is computed, and divided by the overall standard deviation. The test statistic is

$$T = \max \frac{\left|X_l - \overline{X}\right|}{s_t} \qquad \text{(eq. 3-10)}$$

and the value associated with this maximum is discarded if T exceeds the critical value of *T* for a given level of significance, alpha, (Default: 5%) and number of observations equal to the current number of retained data for the given concentration level and matrix. Whenever such a rejection takes place, the pertinent information is recorded in *.PRP.

Step 3 - The test is conducted in the same manner for each and every concentration level, until all the data have been examined. The results of the outlier testing procedure are reported in *.PRP, in the outlier testing section of the file. After all checking has been accomplished, a file containing only nonrejected data is created (*.DAF). This file represents the final data set. It is used in the performance evaluation section of the program where precision and bias are estimated and reported. In all there are four files created by STATCALC's data preparation phase:

*.PRP	Results of Data Preparation
*.DA~	Data Echo File for Audit Trail
*.DAF	Final Data for Analysis
*.PRF	Parameter File for Final Processing

3.5.3 Justification for the Procedure

The outlier detection method described above and employed in STATCALC is the standard in general use for such purposes. It is presented in ASTM Standard D 2777-96, Section 10.4, (2) The procedure also appears in Grubbs (3), along with a general and informative discussion of the subject of statistical determination of outliers.

3.6 Testing for Normality

The test for individual outliers assumes that the underlying statistical population, that is, the population from which the observed measurements are drawn, follows a normal probability

model. Much of the regression analysis performed later in the program also assumes normality (at least approximate) as well. The YOUDENPR subprogram checks this assumption for each set of data consisting of all nonrejected measurements of all labs within each analyte, matrix, concentration level. To check the assumption, one of two test procedures is used, depending on the sample size. If there are 50 or fewer observations remaining within an analyte, matrix, concentration level, the data are checked using the Shapiro-Wilk (W) test (4, 5). When more than 50 observations remain, D'Agostino's (D) test (6, 7) is used.

In both cases, the statistical procedure tests the null hypothesis that all data in the sample come from the same normally distributed population versus the alternative hypothesis that the population's probability distribution is not normal. While a decision not to reject the null hypothesis lends credence to the assumption that the underlying distribution is normal, it cannot be viewed as proof.

3.6.1 The Shapiro-Wilk (W) Test (Sample Size <= 50)

The theory used in the development of the Shapiro-Wilk test is complex, and the test statistic does not lend itself easily to intuitive interpretation. A complete technical discussion of the theory and development of the test is given in Shapiro and Wilk (4).

Discussion of the Test. The computation of the test statistic for the W test is accomplished in several stages.

Shapiro-Wilk Test Procedure -

Step 1 - First the number of observations to be tested is determined. There must be at least 3 observations in the data set in order for the test to be run.

Step 2 - Next, when N is 50 or less, the data for the analyte-matrix-concentration under analysis is sorted into ascending order using a Shell-Metzner sort routine. The "sum of squares" (sum of squared deviations from the mean) of the data set being tested is then computed from the formulas below. Let R_l, l=1,2,...,L be the number of retained values for the L laboratories, for the selected analyte-matrix-concentration level. Then renumber the observations

$$X_{(i)}, i = 1,2,\ldots,N \qquad \text{(eq. 3-11)}$$

where the use of parentheses (1), (2), ... indicates that the array has been sorted into ascending order (so that $X_{(1)}$ is the smallest observation, $X_{(2)}$ the second smallest, ...), and

$$N = \sum_{l=1}^{L} R_l \qquad \text{(eq. 3-12)}$$

The sum of squares, SS_{XX}, is, then,

$$SS_{XX} = \sum_{i=1}^{N}\left(X_{(i)} - \overline{X}\right)^2 = \sum_{i=1}^{N} X_{(i)}{}^2 - \frac{\left(\sum_{i=1}^{N} X_{(i)}\right)^2}{N} \qquad \text{(eq. 3-13)}$$

Step 3 - Next the appropriate set of k coefficients,

$a_1, a_2, ..., a_k$

are taken from Shapiro and Wilk (4), where

$$k = \begin{cases} \dfrac{N}{2} \\ \dfrac{N-1}{2} \end{cases} \qquad \text{(eq. 3-14)}$$

Step 4 - Then the test statistic, W, is computed from the above quantities,

SS_{XX} $a_1, a_2, ..., a_k$, and the ordered X values, $X_{(1)}, X_{(2)}, ..., X_{(N)}$

from the formula

$$W = \frac{1}{SS_{XX}}\left[\sum_{i=1}^{k} a_1\left(X_{(N-i+1)} - X_{(i)}\right)\right]^2 \qquad \text{(eq. 3-15)}$$

Step 5 - The test is run at the 5% significance level. It rejects the normality assumption if the value of W is too small, that is, less than the critical value for N data points.

Step 6 - The results of the normality test ("A" = Accept normality, the data pass the test; "R" = Reject normality, the data do not pass the test.) are reported to the normality testing section of the *.PRP output file.

Justification for the Procedure. The Shapiro-Wilk procedure is a standard method of testing for departures from normality. It is one of the most powerful methods available for such purposes. The original development work for the test was done by S. S. Shapiro and M B. Wilk, (4).

3.6.2 D'Agostino's (D) Test (Sample Size > 50)

For larger samples, when there are more than 50 data points involved, tables for the critical values of the Shapiro-Wilk statistic are not generally available. In that case, a test developed by R. B. D'Agostino (6, 7) is used. The program is currently restricted to no more than 500 data points for a single element-matrix-concentration level.

Discussion of the Test. The preliminaries in the calculation of D, the test statistic for D'Agostino's test, are similar to those for Shapiro-Wilk.

D'Agostino's Test Procedure -

Step 1 - The data are sorted into ascending order as with the Shapiro-Wilk test.

Step 2 - Next the maximum likelihood estimate of the standard deviation, s′, of the data set is computed from the formulas below. (Note: The "maximum likelihood estimate of the standard deviation" is similar to the "sample standard deviation", the difference being that where the latter divides by (N-1) the former divides by N.) Let R_l, l =1,2,..., L be the number of retained results for the L laboratories, for the selected analyte-matrix-concentration level. Then renumber the observations, as with the Shapiro-Wilk test,

$$X_{(i)}, I = 1,2,\ldots,N \qquad \text{(eq. 3-16)}$$

where the use of parentheses (1), (2), ... indicates that the array has been sorted into ascending order (so that $X_{(1)}$ is the smallest observation, $X_{(2)}$ the second smallest, ...), and

$$N = \sum_{l=1}^{L} R_l \qquad \text{(eq. 3-17)}$$

As before, the sum of squares, SS_{XX}, is,

$$SS_{XX} = \sum_{i=1}^{N} \left(X_{(i)} - \overline{X} \right)^2 \qquad \text{(eq. 3-18)}$$

$$SS_{XX} = \sum X_{(i)}{}^2 - \frac{\left(\sum X_i \right)^2}{N} \qquad \text{(eq. 3-19)}$$

Where $\overline{X}$ is the sample mean

$$\overline{X} = \frac{\sum_{i=1}^{N} X_{(i)}}{X} \qquad \text{(eq. 3-20)}$$

The maximum likelihood estimate of the standard deviation is then,

$$s' = \sqrt{\frac{SS_{XX}}{N}} \qquad \text{(eq. 3-21)}$$

Step 3 - Next, D, the preliminary form of the test statistic is computed:

$$D = \frac{N\sum_{i=1}\left[i - \frac{(N-1)}{2}\right]X_{(i)}}{N^2 s'} \qquad \text{(eq. 3-22)}$$

Step 4 - Then the actual test statistic, Y, is computed from the formula

$$Y = \frac{D - 0.28909479}{0.02998598/\sqrt{N}} \qquad \text{(eq. 3-23)}$$

Step 5 - The test is run at the 5% significance level, it rejects the normality assumption if the value of Y is too small, i.e., less than the 2.5 percentage point, or if Y is too large, greater than the 97.5 percentage point.

Step 6 - The results of the normality test ("A" = accept normality, the data pass the normality test", "R" = Reject normality, the data do not pass the normality test.) are reported to the normality testing section of *.PRP.

Justification for the Procedure. D'Agostino's development of the test for normality can be found in D'Agostino, (7).

3.7 Calculation of Analytical Method Performance Statistics

In the next step of the analysis, STATCALC calculates the sample summary statistics for precision, recovery, and bias as specified by D 2777.

In addition, because they will be used in subsequent analyses, the program calculates bias-corrected, i.e., statistically unbiased, estimates of single operator and overall precision and includes these estimates in the output.

At this point in the analysis, the final data set consists of observations:

$$X_{cl}, = 1,2,\ldots C; l = 1,2,\ldots, L \qquad \text{(eq. 3-24)}$$

where

X_{cl} = measurement for the l [th] laboratory, at the c [th] concentration level

L = the number of retained laboratories after lab ranking

C = the number of concentration levels

R_{cl} = the number of retained measurements for laboratory l, at level c

These values are used in the calculations.

3.7.1 Calculation of Method Mean Recovery and Bias

Statistics are calculated for average recovery and bias.

Recovery. The mean recovery, $\overline{X}_c$, which is calculated for each concentration level, is found by averaging over the L sets of laboratory measurements at that level, i.e.,

$$\overline{X}_c = \frac{\sum_{l=1}^{L} X_{cl}}{N_{tc}} \qquad \text{(eq. 3-25)}$$

where

$$N_{tc} = \sum_{l=1}^{L} R_{cl} \qquad \text{(eq. 3-26)}$$

N_{tc} is the total number of retained observations among the L laboratories at concentration level c. With Youden pairs, R_{cl} is either 0 or 1. The notation "tc" is used here to be consistent with later notation requirements.

True Concentration. To calculate the true concentrations, the program accumulates the incremental spikes, ΔT_c, c = 1,2,...,C, given in the *.PRF file.

The value of the true concentration, T_c, for level c is given by

$$T_c = \sum_{i=1}^{C} \Delta T_i \qquad \text{(eq. 3-27)}$$

Bias. The sample bias, B_c, for concentration level c is defined as the difference between the true concentration and the mean recovery (D 2777), i.e.,

$$B_c = \left(\overline{X}_c - T_c\right) \quad \text{(eq. 3-28)}$$

The relative bias is then given by

$$\left(Relative\,Bias\right)_c = \frac{\left(\overline{X}_c - T_c\right)}{T_c} \times 100\% \quad \text{(eq. 3-29)}$$

$$\left(Relative\,Bias\right)_c = \frac{B_c}{T_c} \times 100\% \quad \text{(eq. 3-30)}$$

3.7.2 Calculation of Method Precision Statistics (Standard Deviations)

STATCALC computes estimates of the parameters of regression relating operator precision (s_o), overall precision (s_t), and method recovery (result), (X) to true concentration, (T). These regressions are:

Single Operator Precision regressions:

$$s_o = b_o T + a_o \quad \text{(eq. 3-31)}$$

Overall Precision regressions:

$$s_t = b_t T + a_t \quad \text{(eq. 3-32)}$$

Recovery regressions:

$$X = b_X T + a_X \quad \text{(eq. 3-33)}$$

Single Operator Precision with Youden Pairs. The objective of the Youden pair design is to produce within laboratory measurements which are free from the analyst's tendency to make known replicates match. By selecting two concentrations that are nearly, but not exactly the same, and observing the difference between the uncorrelated measurements at the two concentration levels, the variance of this difference should be twice the variance of the individual measurements, i.e. twice the single operator precision which the analyst is attempting to estimate. If one assumes that single operator precision is equal for all laboratories, then the information (differences) for the several laboratories may be pooled to give a single operator precision estimate for each pair of observations.

Observations for the l^{th} laboratory (in a given data set, i.e., a given matrix/analyte combination) for the P pairs are

$$X_{lp1}, X_{lp2}; \quad p = 1,\ldots,P \qquad \text{(eq. 3-34)}$$

and the differences between the observations within pairs are

$$D_{lp} = \left(X_{lp1} - X_{lp2}\right); \quad p = 1,\ldots,P \qquad \text{(eq. 3-35)}$$

The estimate of the variance of D_{lp} is

$$\hat{\sigma}_p^2 = \frac{\sum_{l=1}^{L_p}\left(D_{lp} - \overline{D}_p\right)}{\left(L_p - 1\right)} \qquad \text{(eq. 3-36)}$$

where

$$\overline{D}_p = \frac{\left(\sum_{l=1}^{L_p} D_{lp}\right)}{L_p} \qquad \text{(eq. 3-37)}$$

and L_p is the number of retained laboratories with both observations present (i.e. no outliers or missing values) for concentrations of pair p. D_{lp} is the difference between two observations, its variance is twice the single operator variance (assuming that the two observations in the pair are independent) so that the estimate of the single operator standard deviation for the p^{th} pair is

$$s_{op} = \sqrt{\frac{\sum_{l=1}^{L_p}\left(D_{lp} - \overline{D}_p\right)^2}{2\left(L_p - 1\right)}} \qquad \text{(eq. 3-38)}$$

Overall (Total) Precision for Youden Pairs. Overall precision (S_{tc}) represents the total combined variability, i.e., both within and between laboratories. Let N_{tc} be the total number of retained observations among the L laboratories at concentration level c. Then

$$s_{tc} = \sqrt{\frac{\sum_{l=1}^{N_{tc}}\left(X_{cl} - \overline{X}_c\right)^2}{N_{tc} - 1}} \qquad \text{(eq. 3-39)}$$

Weights for the Precision Regressions. The STATCALC program uses explicitly weighted linear regression techniques which incorporate weights based upon 1) the number of retained data at a

given concentration level and 2) the change in precision with concentration. The selected weights compensate for changing precision and rejected data to give approximately constant variances for the dependent variable at each concentration. The weights also compensate for "statistical bias" in the sample standard deviation, s, as an estimate of the true standard deviation.

Bias Factor (bf). Although a sample variance, s^2, is statistically unbiased for a true "population" single operator variance, σ^2, its square root, *s*, is well known to be biased for σ and can be corrected for bias with a multiplicative constant dependent upon sample size. To correct for this bias, each *s* is multiplied by a bias factor to give corrected estimates, s^*, of true precision,

$$s^* = (bf) \cdot s \qquad \text{(eq. 3-40)}$$

where

$$bf = \frac{1}{c_4} \qquad \text{(eq. 3-41)}$$

and

$$C_4 = \frac{\sqrt{\frac{2}{\nu}}\left(\frac{\nu-1}{2}\right)!}{\left(\frac{\nu-1}{2}\right)!} \qquad \text{(eq. 3-42)}$$

where ν is the number of degrees of freedom with which the sample standard deviation is computed.

Standard Error Factor (sef). In addition, the standard error of the sample standard deviation, *s*, at a given concentration level, T, is also a function of the constant c_4.

$$\sigma_{s|T} = \sqrt{1-c_4^2} \cdot \sigma_{X|T} \qquad \text{(eq. 3-43)}$$

so that the standard error of *s** is

$$\sigma_{s^*|T} = \sigma_{(bf)\cdot s|T} \qquad \text{(eq. 3-44)}$$

$$= (bf) \cdot \sqrt{1-c_4^2} \cdot \sigma_{X|T} \qquad \text{(eq. 3-45)}$$

$$= (bf) \cdot (sef) \cdot \sigma_{X|T} \qquad \text{(eq. 3-46)}$$

which is a function of sample size and the true value of $\sigma_{x|T.}$

Need for Weights. Since regression analysis requires homogeneity of variances for the dependent variable, weighted least squares estimates must be used to fit the regression model to the values of *s*. The weights used must be proportional to the variances of the *s* values for the different concentration levels. The appropriate weights are

$$w = \frac{1}{(bf)^2 \cdot (sef)^2 \cdot \sigma^2_{X|T}} \qquad \text{(eq. 3-47)}$$

The true value of $\sigma^2_{X|T}$ is not known, however, so it must be estimated.

Preliminary Weights. There is a practical difficulty here since the values of b and a are not determined until the regression line has been fit, yet their values are needed to determine weights used to fit the regression line. To overcome this difficulty, an initial rough estimate of the precision regression line is obtained by assuming that the true values of $\sigma^2_{X|T}$ are constant, for which appropriate weights would be

$$w = \frac{1}{(bf)^2 \cdot (sef)^2} \qquad \text{(eq. 3-48)}$$

and weighted linear regression with these weights gives initial estimates, b_{init} and $a_{init,}$ of the slope and intercept, respectively, of the regression of s* on T.

$$s^* = b_{init} T + a_{init} \qquad \text{(eq. 3-49)}$$

Preliminary estimates of the method variance at each concentration level are now calculated using the values of b_{init} and $a_{init,}$ above. This calculation gives approximate weights

$$w = \frac{1}{(bf)^2 \cdot (sef)^2 \cdot (b_{init} T + a_{init})^2} \qquad \text{(eq. 3-50)}$$

which are then normalized, i.e. they are divided by their sum to give normalized weights, w_c

$$w_c \rightarrow \frac{w_c}{\sum_{c=1}^{C} w_c} \qquad \text{(eq. 3-51)}$$

As before, the bias and standard error factors are determined for the number of degrees of freedom with which the standard deviation is computed.

Refined estimates of the precision regression equation are now obtained using the preliminary weights above to perform a second weighted linear regression for the precision model using the weighted regression formulas in the next section with Y = s*, X = T to give

$$\hat{s}^* = bT + a \qquad \text{(eq. 3-52)}$$

Weighted Linear Regression Formulas. The formulas for computing weighted least squares regression estimates are similar to those for unweighted least squares. Suppose one has *C* observations on variables *U* (dependent) and *V* (independent), i.e.,

$$(V_c, U_c); \quad c = 1,2,\ldots,C \qquad \text{(eq. 3-53)}$$

Then the weighted least squares estimates, given normalized weights

$$w_c; c = 1,2,\ldots,C\left(\sum w_c = 1.0\right) \qquad \text{(eq. 3-54)}$$

and letting

$$WSS_{VU} = \sum w\left(V - \bar{V}\right)\left(U - \bar{U}\right) = \sum wUV - \left(\sum wV\right)\left(\sum wU\right) \qquad \text{(eq. 3-55)}$$

$$WSS_{VV} = \sum w\left(V - \bar{V}\right)^2 = \sum wV^2 - \left(\sum wV\right)^2 \qquad \text{(eq. 3-56)}$$

are

$$b = \frac{WSS_{VU}}{WSS_{VV}} \qquad \text{(eq. 3-57)}$$

and

$$a = \bar{U} - b\bar{V} \qquad \text{(eq. 3-58)}$$

where

$$\overline{V} = \sum wV \qquad \text{(eq. 3-59)}$$

and

$$\overline{U} = \sum wU \qquad \text{(eq. 3-60)}$$

and all sums are taken from 1 to C.

Precision Estimates by Weighted Curvilinear Regression. The STATCALC program uses essentially the same techniques described for the linear models described above to fit a curvilinear regression mode. To accomplish the curvilinear fit, the logarithms of the precisions are used, rather than the precisions themselves. The logarithmic form of the curvilinear precision equation is

$$\ln \hat{s}^* = bT + a \qquad \text{(eq. 3-61)}$$

After the fitting is done, the logarithmic model is converted back so that it is expressed in the original (nonlogarithmic) units. Use of the curvilinear model assumes that operator precision changes at a constant relative rate.

The nonlogarithmic form of the model is

$$\hat{s}^* = a'(b')^T \qquad \text{(eq. 3-62)}$$

where

$$b' = e^b \qquad \text{(eq. 3-63)}$$

and

$$a' = e^a \qquad \text{(eq. 3-64)}$$

General. For the curvilinear single operator regressions, the dependent variable is the logarithm of the bias corrected precision, i.e.,

$$\ln \hat{s}^*_{oc}; \quad c = 1,2,\ldots,C \qquad \text{(eq. 3-65)}$$

and the true concentration is the independent variables, i.e.,

$$T_c; \quad c = 1,2,\ldots,C \qquad \text{(eq. 3-66)}$$

Weighted linear regression is used, but, since the standard deviation of s* is proportional to the standard deviation of $\sigma_{X|T}$, the logarithmic transformation stabilizes the variance. The regression weights depend only on the number of retained data. In general, they are

$$w = \frac{1}{(bf)^2 \cdot (sef)^2} \qquad \text{(eq. 3-67)}$$

Recovery Estimates by Weighted Linear Regression. Recoveries are the measured amounts, X, reported by the laboratories. In fitting the regression models, it is assumed that, at a given concentration, the expected recovery is the same for all laboratories. STATCALC estimates the parameters of a linear model which relates recovery to true concentration.

$$\hat{X} = bT + a \qquad \text{(eq. 3-68)}$$

For estimating the recovery regression equation, STATCALC uses weighted regression techniques for the same reasons already discussed for precision regression, that is, to compensate for decreasing precision and rejected data to give approximately constant standard errors for recovery at each concentration.

The recovery regressions are performed with recovery as the dependent variable and true concentration as the independent variable. The actual calculations are performed on the C mean recovery-true concentration pairs, i.e.,

$$(T_c, \overline{X}_c); \quad c = 1,2,\ldots,C \qquad \text{(eq. 3-69)}$$

Weighted linear regression is used with (normalized) weights

$$w_c; \quad c = 1,2,\ldots,C \qquad \text{(eq. 3-70)}$$

Calculation of these weights differ from those for precision. To achieve the required homogeneity of variances the weights used must be proportional to the variances of the X values for the different concentration levels. The appropriate weights are

$$w = \frac{1}{N_T \cdot \sigma^2_{X|T}} \qquad \text{(eq. 3-71)}$$

where N_T is the total number of retained observations from which the mean recovery is computed and $\sigma^2_{x|T}$ is the true overall precision. The true value of $\sigma^2_{x|T}$ is not known, however, so it must be estimated using $\hat{s}^*_T$ from the previous precision regression analysis.

The weights are

$$w_c = \frac{N_c}{\hat{s}^*_c} \qquad \text{(eq. 3-72)}$$

where $\hat{s}^*_c$ is the appropriate regression equation evaluated at T_c.

Weighted least squares with $U = \overline{X}$, $V = T$ is used to obtain the recovery regression equation.

Precision Versus Recovery. A regression relationship between precision and recovery may be obtained by substituting the recovery regression results into the precision regression formulas.

Linear Models. The formulas output by the program for linear precision and linear recovery versus true concentration are:

- Single Operator Precision

$$\hat{s}^*_o = b_o T + a_o \qquad \text{(eq. 3-73)}$$

- Overall Precision

$$\hat{s}^*_t = b_t T + a_t \qquad \text{(eq. 3-74)}$$

- Recovery

$$\hat{X} = b_X T + a_X \qquad \text{(eq. 3-75)}$$

Solving for T in the recovery equation gives

$$T = \frac{\left(\hat{X} - a_X\right)}{b_X} \qquad \text{(eq. 3-76)}$$

which is substituted into the precision equations to obtain

$$\hat{s}^* = a_o + b_o \frac{\left(\hat{X} - a_X\right)}{b_X} \quad \text{(eq. 3-77)}$$

$$= \left(a_o - \frac{a_X}{b_X}\right) + \frac{b_o}{b_X}\hat{X} \quad \text{(eq. 3-78)}$$

$$= e_o + f_o \hat{X} \quad \text{(eq. 3-79)}$$

where

$$e_o = a_o - \frac{a_X}{b_X} \quad \text{(eq. 3-80)}$$

$$f_o = \frac{b_o}{b_X} \quad \text{(eq. 3-81)}$$

In a like manner, for the overall precision,

$$\hat{s}_t^* = e_t + f_t \hat{X} \quad \text{(eq. 3-82)}$$

where

$$e_t = a_t - \frac{a_X}{b_X} \quad \text{(eq. 3-83)}$$

$$f_t = \frac{b_t}{b_X} \quad \text{(eq. 3-84)}$$

Curvilinear Models. The formulas output by the program for curvilinear precision and linear recovery versus true concentration are:

- Single Operator Precision

$$\hat{s}_o^* = a_o \left(b_o\right)^T \quad \text{(eq. 3-85)}$$

- Overall Precision

$$\hat{s}_t^* = a_t \left(b_t\right)^T \quad \text{(eq. 3-86)}$$

- Recovery

$$\hat{X} = b_X T + a_X \quad \text{(eq. 3-87)}$$

Solving for T in the recovery equation gives

$$T = \frac{\left(\hat{X} - a_X\right)}{b_X} \quad \text{(eq. 3-88)}$$

which is substituted into the single operator precision equation to obtain

$$\hat{s}_o^* = a_o \left(b_o\right)^{\left(\hat{X} - a_X\right)/b_X} \quad \text{(eq. 3-89)}$$

$$\hat{s}_o^* = a_o \left(b_o\right)^{\left(-\frac{a_X}{b_X}\right)} \left(b_o\right)^{\left(\frac{\hat{X}}{b_X}\right)} \quad \text{(eq. 3-90)}$$

$$\hat{s}_o^* = e_o \left(f_o\right)^{\hat{X}} \quad \text{(eq. 3-91)}$$

where

$$e_o = a_o \left(b_o\right)^{\left(-\frac{a_X}{b_X}\right)} \left(b_o\right)^{\left(\frac{\hat{X}}{b_X}\right)} \quad \text{(eq. 3-92)}$$

$$f_o = \left(b_o\right)^{\left(1/b_X\right)} \quad \text{(eq. 3-93)}$$

In a like manner for the overall precision,

$$\hat{s}_t^* = e_t \left(f_t\right)^{\hat{X}} \quad \text{(eq. 3-94)}$$

3.8 Testing for Significant Bias

The program next performs the necessary calculations and tests to determine whether the observed bias values for each level are statistically significant.

3.8.1 General

The statistical procedure tests the null hypothesis that the true method bias at each concentration is zero, versus the alternative that it is nonzero, assuming the underlying distribution is normal. While a decision not to reject the null hypothesis is not proof that the method bias is zero, it lends credence to such a conclusion.

3.8.2 Rationale for the Procedure

If there is no method bias for a given level of concentration, then one would expect that the true and measured amounts of analyte should be the same, on average. A Student's t test procedure is used to judge, as prescribed by ASTM D 2777, whether the bias (difference between true and average measured concentrations) for the sample data at each concentration level is within the realm of acceptable variability, taking overall method precision into account. First, the program adjusts the overall precision (standard deviation) to reflect the number of observations over which recovery was averaged at the given concentration level. The adjusted value is called the standard deviation of the mean. Then, the program calculates the number of standard deviations (of the mean) by which the true and average recovered values differ. Finally, this latter figure is tested for acceptability under Student's t distribution.

3.8.3 Outline of the Calculations

The basic steps in the calculations at each concentration level are given below.

Student's t Test for Bias -

Step 1 - Calculate the bias:

$$B_c = \overline{X}_c - T_c \qquad \text{(eq. 3-95)}$$

Step 2 - Calculate the standard deviation of the mean (recovery):

$$s_{\overline{X}} = \frac{s_{tc}}{\sqrt{N_{tc}}} \qquad \text{(eq. 3-96)}$$

When the average of the Level 1 concentration recoveries is used as the true background concentration, an adjustment must be made in this calculation. The bias is then identically 0.0 for level 1 and for c greater than 1 it is

$$\overline{X}_c - T_c = \overline{X}_c - \overline{X}_1 - \sum_{i=2}^{c} \Delta T_i \qquad \text{(eq. 3-97)}$$

for which the standard deviation is estimated as

$$s_{\overline{X}_c - \overline{X}_1} = \sqrt{\frac{s_{tc}^2}{N_{tc}} + \frac{s_{t1}^2}{N_{t1}}} \qquad \text{(eq. 3-98)}$$

Step 3 - Calculate the observed t-value:

$$t_c = \frac{\left|\overline{X}_c - T_c\right|}{s_{tc} \Big/ \sqrt{N_{tc}}} \qquad \text{(eq. 3-99)}$$

Step 4 - Determine the critical value, $t_{.005}$, for a 1% (two-tailed) significance level for Student's t test with N_{tc} - 1 degrees of freedom, and compare t from Step 3 to the critical value. The null hypothesis is rejected (concluding there is significant bias) if

$$t_c > t_{.005} \qquad \text{(eq. 3-100)}$$

Step 5 - The results of the bias testing ("Yes" = "Yes, the bias is significant." or "No" = "No, the bias is not significant.") are reported to *.STT.

3.9 Use of STATCALC Output

The STATCALC output files, particularly the data preparation files (*.PRP) and statistical processing files (*.STT) provide all of the information necessary for the user to compile the tables and graphs required in ASTM D 2777. Copies of the data, data preparation and statistical processing files for all of the validation study data are contained in Appendix C. The precision and recovery regression data are used to calculate the IDE and IQE discussed in Chapter 5. Chapter 6 contains the performance data calculated from the Method 1638 validation study data.

3.10 References

1. American Society for Testing and Materials, "Standard Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water", D 2777-86.

2. American Society for Testing and Materials, "Standard Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water", D 2777-96.

3. F.E. Grubbs, "Procedures for Detecting Outlying Observations in Samples", *Technometrics*, 2, No. 1, February, 1969.

4. S.S. Shapiro and M. B. Wilk, "An Analysis of Variance Test for Normality", *Biometrika*, Vol 52, 1965, pp. 519-611.

5. S.S. Shapiro, M.B. Wilk and H.J. Chen, " A Comparative Study of Various Tests of Normality", *Journal of the American Statistical Association*, 63, 324, December 1968, pp. 1343-1372.

6. R.B. D'Agostino, "An Omnibus Test of Normality for Moderate and Large Samples", *Biometrika*, 58, 1971, pp. 341-348.

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4
DATA ANALYSIS

In the EPA/EPRI interlaboratory evaluation of EPA Method 1638, the EPA and EPRI performed analysis of the data independently. EPRI's approach to data reduction and analysis is presented in Sections 4.1 and 4.2.

4.1 Data Reduction

The laboratories submitted all of their results to the EPA's Sample Control Center (SCC) in electronic format. The SCC contacted each laboratory to resolve any discrepancies in the reported data. The corrected data files were forwarded from the SCC to EPRI in an Access® database. EPRI generated the appropriate ASCII files for use in the STATCALC statistical program.

The EPA flagged a number of values in the data set with the comment "not used in the final report." EPRI reviewed EPA's reason for removing each data point. If the reason was valid (e.g., the data point was beyond the calibration range), EPRI removed the data point prior to processing the data. However, EPA did not provide an explanation for all of the flagged data points, and to date, has declined to provide an explanation for these notations. EPRI retained these values in the data evaluation.

The following sections discuss the make-up of the data before and after processing.

4.1.1 Raw Data

A complete printout of the raw data by element is contained in Appendix B. The data are sorted by laboratory, matrix and concentration level.

4.1.2 Outlier Removal Results

EPRI's statistical program, STATCALC, performs the two outlier removal activities specified in ASTM D 2777 (1): lab ranking and individual outlier removal. These are described in detail in Section 3. When a lab is removed by the lab ranking procedure, all of its data are removed for a particular element and matrix. ASTM D 2777 permits up to 20% of the laboratories to be removed by lab ranking.

The individual outlier test is performed on the remaining data by element, matrix and concentration level. ASTM D 2777 permits up to a maximum of 10% of the data points that fail the outlier test to be removed at each element, matrix and concentration level. For small data sets

with fewer than 10 laboratories surviving the lab ranking test, ASTM D 2777 permits a maximum of one outlier data point that fails the outlier test to be removed at each element, matrix and concentration level.

Per the EPA study design, only the sample set for the freshwater matrix followed all requirements of the ASTM D 2777 design. The reagent grade water sample set contained 80% duplicates and ASTM D 2777 permits only up to 50% of the sample pairs for a given element and matrix to be duplicates. Nevertheless, it was still possible to use ASTM D 2777 to analyze the reagent grade water data. The sample sets for the other two matrices (filtered effluent, unfiltered effluent) consisted of a single duplicate pair for each matrix. The lab ranking test could not be performed on the effluent data since only a single pair of values were reported. However, the individual outlier test was performed on the data.

Table 4-1 summarizes the total number of data points (summed over all concentration levels) remaining in the reagent grade water and freshwater data sets after outlier removal. The largest percentage of outliers was removed by the laboratory ranking test. Only a small percentage was removed by the individual outlier procedure.

4.1.3 Normality Testing

The statistical program, STATCALC, tested the reagent grade water and freshwater data sets for normality using either the Shapiro/Wilk (sample size <50) or D'Agostino's (sample size >50) normality test. These tests are discussed in detail in Section 3. The test for individual outliers and much of the regression analysis assumes at least approximate normality. The results of the normality tests are either A (accept normality, the data passes the normality test) or R (reject normality, the data does not pass the normality test). The data sets for the other matrices were not tested since the normality tests in STATCALC were not designed to deal with data sets containing only one concentration level. Table 4-2 summarizes the results of the normality tests. A total of 149 of the 162 data sets tested normal (92%).

4.2 Data Analysis

4.2.1 Reagent Grade Water and Freshwater

The results of the statistical analysis of the Method 1638 data in reagent water and freshwater are summarized by element in Tables 4-3 through 4-20. The STATCALC input and output files are contained in Appendix C. The true concentration of the unspiked reagent grade water or the freshwater cannot be determined, especially for trace levels, by a single laboratory using the method under study. Since the unspiked reagent grade water and freshwater were sent blind to the participants along with the spiked samples, the true value of the lowest concentration can be determined as the consensus value obtained by using the method of standard additions on all of the data reported by the participants for each spiked and unspiked sample. For each matrix/analyte combination, the true concentration of the lowest concentration sample (or pair) was determined by using the method of standard additions, and the known spikes were added to this calculated background concentration to calculate the true concentrations of the remaining concentration levels.

Table 4-1
Data Points Remaining after Outlier Removal

			After Lab Ranking		After Individual Outlier Removal	
Element	**Matrix**	**Points as Received**	**Points**	**% (of Points Received)**	**Points**	**% (of Points Received)**
Sb	RGW	78	68	87.2	68	87.2
	FW	64	56	87.5	54	84.4
Cd	RGW	80	70	87.5	68	85
	FW	64	56	87.5	56	87.5
Cu	RGW	80	70	87.5	65	81.3
	FW	64	56	87.5	52	81.3
Pb	RGW	80	70	87.5	64	80.0
	FW	64	56	87.5	56	87.5
Ni	RGW	76	66	86.8	61	80.3
	FW	64	56	87.5	55	85.9
Se	RGW	74	66	89.2	64	86.5
	FW	64	56	87.5	55	85.9
Ag	RGW	80	70	87.5	68	85.0
	FW	64	56	87.5	52	81.3
Tl	RGW	80	70	87.5	70	87.5
	FW	64	56	87.5	55	85.9
Zn	RGW	78	68	87.2	64	82.1
	FW	64	56	87.5	54	84.4

RGW = Reagent Grade Water
FW = Freshwater
Percentages based on total data points received.

Table 4-2
Test of Data Normality after Outlier Removal

	Reagent Grade Water										Freshwater							
Element	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Sb	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A
Cd	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A
Cu	A	R	A	A	R	A	A	R	A	A	A	A	A	A	A	A	A	A
Pb	A	A	A	A	A	A	A	A	A	A	A	R	A	A	A	A	A	A
Ni	R	R	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A
Se	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A
Ag	A	A	A	R	A	R	A	A	A	A	A	R	R	R	A	A	A	A
Tl	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A
Zn	A	A	A	A	A	A	A	A	A	A	A	A	A	A	R	R	A	A

A = accept normality, R = reject normality

At each concentration level, ASTM D 2777 requires a minimum of six surviving pairs of data to calculate single operator precision and six individual data points to calculate overall or interlaboratory precision. The following data sets had only five pairs remaining:

- The lowest (unspiked) concentration of copper in freshwater.
- The lowest (unspiked) and fourth highest concentrations of lead in reagent grade water.
- The lowest (unspiked), lowest spiked, and highest spiked concentrations of nickel in reagent grade water.
- The highest spiked concentration of selenium in reagent grade water.

These data are presented in Tables 4-8, 4-9, 4-11 and 4-13, but are omitted from any further calculations.

Table 4-3
Statistical Summary for Antimony in Reagent Grade Water

	Blank	Blank	+0.1 µg/L	+0.12 µg/L	+1.0 µg/L	+1.0 µg/L	+5.0 µg/L	+5.0 µg/L	+20.0 µg/L	+20.0 µg/L
Number of retained values	7	7	7	7	7	7	7	7	6	6
True concentration, µg/L	0.0000	0.0000	0.1000	0.1200	1.0000	1.0000	5.0000	5.0000	20.0000	20.0000
Mean recovery, µg/L	0.0072	0.0013	0.052	0.0804	0.6423	0.6544	3.9763	3.9986	17.524	17.5356
Percent recovery	---	---	52.0	67.0	64.2	65.4	79.5	80.0	87.6	87.7
Overall standard deviation, S_t, µg/L	0.0199	0.0138	0.0133	0.0286	0.034	0.0511	0.2079	0.1873	1.1611	1.0801
Overall relative standard deviation, %	275.1	1027.7	25.5	35.5	5.3	7.8	5.2	4.7	6.6	6.2
Number of retained pairs	7		7		7		7		6	
Mean recovery (pairs), µg/L	0.0043		0.0662		0.6484		3.9875		17.5298	
Single operator standard deviation, S_o, µg/L	0.0085		0.0247		0.0246		0.0476		0.2318	
Single operator relative standard deviation, %	199.4		37.4		3.8		1.2		1.3	

Table 4-4
Statistical Summary for Antimony in Freshwater

	Blank	Blank	+0.08 µg/L	+0.12 µg/L	+0.28 µg/L	+0.38 µg/L	+0.68 µg/L	+0.88 µg/L
Number of retained values	7	7	6	7	7	7	7	6
True concentration, µg/L	0.1328	0.1328	0.2128	0.2528	0.4128	0.5128	0.8128	1.0128
Mean recovery, µg/L	0.1484	0.1265	0.2094	0.2473	0.3887	0.4805	0.7744	0.9789
Percent recovery	111.7	95.3	98.4	97.8	94.2	93.7	95.3	96.7
Overall standard deviation, S_t, µg/L	0.0309	0.014	0.0123	0.015	0.0163	0.0096	0.0171	0.0126
Overall relative standard deviation, %	20.8	11.1	5.9	6.1	4.2	2.0	2.2	1.3
Number of retained pairs	7		6		7		6	
Mean recovery (pairs), µg/L	0.1375		0.2298		0.4346		0.8688	
Single operator standard deviation, S_o, µg/L	0.0184		0.0041		0.0091		0.0089	
Single operator relative standard deviation, %	13.4		1.8		2.1		1.0	

Table 4-5
Statistical Summary for Cadmium in Reagent Grade Water

	Blank	Blank	+0.05 µg/L	+0.06 µg/L	+0.10 µg/L	+0.10 µg/L	+1.0 µg/L	+1.0 µg/L	+10.0 µg/L	+10.0 µg/L
Number of retained values	6	7	7	6	7	7	7	7	7	7
True concentration, µg/L	0.0041	0.0041	0.0541	0.0641	0.1041	0.1041	1.0041	1.0041	10.0041	10.0041
Mean recovery, µg/L	-0.005	0.0049	0.0501	0.0633	0.1022	0.1012	0.9825	0.9824	9.6056	9.6906
Percent recovery	-122.0	119.5	92.6	98.8	98.2	97.2	97.8	97.8	96.0	96.9
Overall standard deviation, S_t, µg/L	0.0201	0.0346	0.029	0.0366	0.0336	0.0373	0.0426	0.0789	0.5738	0.5262
Overall relative standard deviation, %	-404.2	707.6	57.8	57.8	32.9	36.9	4.3	8.0	6.0	5.4
Number of retained pairs	6		6		7		7		7	
Mean recovery (pairs), µg/L	0.0003		0.0562		0.1017		0.9825		9.6481	
Single operator standard deviation, S_o, µg/L	0.0027		0.0044		0.0075		0.037		0.0809	
Single operator relative standard deviation, %	783.4		7.8		7.4		3.8		0.8	

Table 4-6
Statistical Summary for Cadmium in Freshwater

	Blank	Blank	+0.04 µg/L	+0.05 µg/L	+0.12 µg/L	+0.14 µg/L	+0.25 µg/L	+0.25 µg/L
Number of retained values	7	7	7	7	7	7	7	7
True concentration, µg/L	0.0000	0.0000	0.0400	0.0500	0.1200	0.1400	0.2500	0.2500
Mean recovery, µg/L	-0.0138	-0.0056	0.0288	0.0483	0.1142	0.1352	0.2506	0.2401
Percent recovery	---	---	72.0	96.6	95.2	96.6	100.2	96.0
Overall standard deviation, S_t, µg/L	0.0296	0.0185	0.0203	0.0244	0.0211	0.0206	0.0307	0.0222
Overall relative standard deviation, %	---	---	70.4	50.6	18.5	15.2	12.2	9.3
Number of retained pairs	7		7		7		7	
Mean recovery (pairs), µg/L	-0.0097		0.0386		0.1247		0.2454	
Single operator standard deviation, S_o, µg/L	0.0199		0.0076		0.0046		0.0135	
Single operator relative standard deviation, %	-205.2		19.6		3.7		5.5	

Table 4-7
Statistical Summary for Copper in Reagent Grade Water

	Blank	Blank	+0.20 µg/L	+0.24 µg/L	+0.50 µg/L	+0.50 µg/L	+2.0 µg/L	+2.0 µg/L	+20 µg/L	+20 µg/L
Number of retained values	6	6	6	6	7	7	7	7	7	6
True concentration, µg/L	0.1052	0.1052	0.3052	0.3452	0.6052	0.6052	2.1052	2.1052	20.1052	20.1052
Mean recovery, µg/L	0.0662	0.0911	0.3461	0.3356	0.6993	0.6078	2.0463	2.3844	19.1832	19.6034
Percent recovery	62.9	86.6	113.4	97.2	115.5	100.4	97.2	113.3	95.4	97.5
Overall standard deviation, S_t, µg/L	0.0648	0.1198	0.1816	0.1378	0.3017	0.1441	0.1898	0.739	1.0918	1.119
Overall relative standard deviation, %	97.9	131.5	52.5	41.1	43.1	23.7	9.3	31.0	5.7	5.7
Number of retained pairs	6		6		7		7		6	
Mean recovery (pairs), µg/L	0.0787		0.3409		0.6536		2.2154		19.3771	
Single operator standard deviation, S_o, µg/L	0.0566		0.1122		0.18		0.4459		0.0681	
Single operator relative standard deviation, %	72.0		32.9		27.5		20.1		0.4	

Table 4-8
Statistical Summary for Copper in Freshwater

	Blank	Blank	+0.50 µg/L	+0.70 µg/L	+1.50 µg/L	+2.0 µg/L	+3.5 µg/L	+4.0 µg/L
Number of retained values	6	6	7	6	7	7	6	7
True concentration, µg/L	1.2599	1.2599	1.7599	1.9599	2.7599	3.2599	4.7599	5.2599
Mean recovery, µg/L	1.1199	1.1496	1.6311	1.7581	2.4874	3.1352	4.2865	4.7869
Percent recovery	88.9	91.2	92.7	89.7	90.1	96.2	90.1	91.0
Overall standard deviation, S_t, µg/L	0.1553	0.175	0.1472	0.1258	0.1616	0.3365	0.2118	0.3347
Overall relative standard deviation, %	13.9	15.2	9.0	7.2	6.5	10.7	4.9	7.0
Number of retained pairs	5		6		7		6	
Mean recovery (pairs), µg/L	1.1348		1.6897		2.8113		4.5559	
Single operator standard deviation, S_o, µg/L	0.078		0.073		0.1563		0.1022	
Single operator relative standard deviation, %	6.9		4.3		5.6		2.2	

Shaded values do not meet ASTM D 2777 minimum requirements for surviving data.

Table 4-9
Statistical Summary for Lead in Reagent Grade Water

	Blank	Blank	+0.03 µg/L	+0.036 µg/L	+0.10 µg/L	+0.10 µg/L	+0.50 µg/L	+0.50 µg/L	+5.0 µg/L	+5.0 µg/L
Number of retained values	6	6	7	7	6	7	6	6	7	6
True concentration, µg/L	0.0192	0.0192	0.0492	0.0552	0.1192	0.1192	0.5192	0.5192	5.0192	5.0192
Mean recovery, µg/L	0.0168	0.0179	0.0829	0.0635	0.1154	0.1289	0.4962	0.516	4.9776	4.9959
Percent recovery	87.5	93.2	168.5	115.0	96.8	108.1	95.6	99.4	99.2	99.5
Overall standard deviation, S_t, µg/L	0.0232	0.0216	0.0654	0.036	0.0246	0.0437	0.0215	0.0369	0.1751	0.1722
Overall relative standard deviation, %	138.0	120.7	78.9	56.6	21.3	33.9	4.3	7.1	3.5	3.4
Number of retained pairs	5		7		6		5		6	
Mean recovery (pairs), µg/L	0.0174		0.0732		0.1227		0.5061		4.9860	
Single operator standard deviation, S_o, µg/L	0.0032		0.0323		0.0114		0.0104		0.0728	
Single operator relative standard deviation, %	18.5		44.1		9.3		2.1		1.5	

Shaded values do not meet ASTM D 2777 minimum requirements for surviving data.

Table 4-10
Statistical Summary for Lead in Freshwater

	Blank	Blank	+0.04 µg/L	+0.05 µg/L	+0.19 µg/L	+0.19 µg/L	+0.30 µg/L	+0.35 µg/L
Number of retained values	7	7	7	7	7	7	7	7
True concentration, µg/L	0.0429	0.0429	0.0829	0.0929	0.2329	0.2329	0.3429	0.3929
Mean recovery, µg/L	0.0367	0.0612	0.0727	0.0918	0.237	0.2281	0.3348	0.371
Percent recovery	85.5	142.7	87.7	98.8	101.8	97.9	97.6	94.4
Overall standard deviation, S_t, µg/L	0.0201	0.0608	0.0183	0.0189	0.0624	0.0127	0.0512	0.0273
Overall relative standard deviation, %	54.7	99.5	25.1	20.6	26.3	5.6	15.3	7.4
Number of retained pairs	7		7		7		7	
Mean recovery (pairs), µg/L	0.0490		0.0823		0.2326		0.3529	
Single operator standard deviation, S_o, µg/L	0.03		0.0162		0.0409		0.0269	
Single operator relative standard deviation, %	61.3		19.7		17.6		7.6	

Table 4-11
Statistical Summary for Nickel in Reagent Grade Water

	Blank	Blank	+0.50 µg/L	+0.60 µg/L	+1.00 µg/L	+1.00 µg/L	+10.0 µg/L	+10.0 µg/L	+100.0 µg/L	+100.0 µg/L
Number of retained values	6	6	6	6	7	7	6	7	5	5
True concentration, µg/L	0.0000	0.0000	0.5000	0.6000	1.0000	1.0000	10.0000	10.0000	100.0000	100.0000
Mean recovery, µg/L	0.0092	-0.0075	0.4531	0.5651	0.9195	0.9286	9.1243	9.3206	91.7055	93.5939
Percent recovery	---	---	90.6	94.2	92.0	92.9	91.2	93.2	91.7	93.6
Overall standard deviation, S_t, µg/L	0.0119	0.0333	0.0234	0.0426	0.0502	0.0596	0.1579	0.3463	3.8264	3.2346
Overall relative standard deviation, %	129.9	---	5.2	7.5	5.5	6.4	1.7	3.7	4.2	3.5
Number of retained pairs	5		5		7		6		5	
Mean recovery (pairs), µg/L	0.0009		0.5091		0.9241		9.2300		92.6497	
Single operator standard deviation, S_o, µg/L	0.0039		0.0114		0.0568		0.1704		0.9933	
Single operator relative standard deviation, %	471.0		2.2		6.2		1.8		1.1	

Shaded values do not meet ASTM D 2777 minimum requirements for surviving data.

Table 4-12
Statistical Summary for Nickel in Freshwater

	Blank	Blank	+1.30 µg/L	+1.50 µg/L	+3.0 µg/L	+3.7 µg/L	+5.7 µg/L	+5.7 µg/L
Number of retained values	7	6	7	7	7	7	7	7
True concentration, µg/L	0.6716	0.6716	1.9716	2.1716	3.6716	4.3716	6.3716	6.3716
Mean recovery, µg/L	0.6647	0.6083	1.8396	2.041	3.3914	4.0243	6.1792	6.0398
Percent recovery	99.0	90.6	93.3	94.0	92.4	92.1	97.0	94.8
Overall standard deviation, S_t, µg/L	0.172	0.1054	0.1668	0.2009	0.259	0.3062	0.6599	0.3759
Overall relative standard deviation, %	25.9	17.3	9.1	9.8	7.6	7.6	10.7	6.2
Number of retained pairs	6		7		7		7	
Mean recovery (pairs), µg/L	0.6387		1.9403		3.7079		6.1095	
Single operator standard deviation, S_o, µg/L	0.0452		0.0389		0.0904		0.2802	
Single operator relative standard deviation, %	7.1		2.0		2.4		4.6	

Table 4-13
Statistical Summary for Selenium in Reagent Grade Water

	Blank	Blank	+1.00 µg/L	+1.20 µg/L	+5.00 µg/L	+5.00 µg/L	+20.0 µg/L	+20.0 µg/L	+100.0 µg/L	+100.0 µg/L
Number of retained values	6	7	6	7	7	7	7	7	5	5
True concentration, µg/L	0.0285	0.0285	1.0285	1.2285	5.0285	5.0285	20.0285	20.0285	100.0285	100.0285
Mean recovery, µg/L	0.0261	0.0645	0.9613	1.1716	4.819	4.966	19.7535	19.8695	99.5455	101.021
Percent recovery	91.6	226.3	93.5	95.4	95.8	98.8	98.6	99.2	99.5	101.0
Overall standard deviation, S_t, µg/L	0.0696	0.1244	0.1223	0.364	0.4707	0.6131	1.804	1.7017	10.6641	9.4256
Overall relative standard deviation, %	266.7	192.8	12.7	31.1	9.8	12.3	9.1	8.6	10.7	9.3
Number of retained pairs	6		6		7		7		5	
Mean recovery (pairs), µg/L	0.0468		1.0745		4.8925		19.8115		100.2831	
Single operator standard deviation, S_o, µg/L	0.0962		0.1589		0.4366		0.2501		1.4615	
Single operator relative standard deviation, %	205.6		14.8		8.9		1.3		1.5	

Shaded values do not meet ASTM D 2777 minimum requirements for surviving data.

Table 4-14
Statistical Summary for Selenium in Freshwater

	Blank	Blank	+0.60 µg/L	+0.80 µg/L	+1.60 µg/L	+2.00 µg/L	+3.60 µg/L	+3.60 µg/L
Number of retained values	7	6	7	7	7	7	7	7
True concentration, µg/L	0.2055	0.2055	0.8055	1.0055	1.8055	2.2055	3.8055	3.8055
Mean recovery, µg/L	0.2256	0.1567	0.7738	1.1332	1.7506	2.1067	3.7212	3.7927
Percent recovery	109.8	76.3	96.1	112.7	97.0	95.5	97.8	99.7
Overall standard deviation, S_t, µg/L	0.1174	0.0881	0.4486	0.2398	0.3362	0.4363	0.2105	0.2435
Overall relative standard deviation, %	52.0	56.2	58.0	21.2	19.2	20.7	5.7	6.4
Number of retained pairs	6		7		7		7	
Mean recovery (pairs), µg/L	0.1938		0.9535		1.9287		3.7570	
Single operator standard deviation, S_o, µg/L	0.1338		0.3717		0.2206		0.1807	
Single operator relative standard deviation, %	69.0		39.0		11.4		4.8	

Table 4-15
Statistical Summary for Silver in Reagent Grade Water

	Blank	Blank	+0.1 µg/L	+0.1 µg/L	+0.20 µg/L	+0.24 µg/L	+1.0 µg/L	+1.0 µg/L	+10.0 µg/L	+10.0 µg/L
Number of retained values	6	6	7	7	7	7	7	7	7	7
True concentration, µg/L	0.0062	0.0062	0.1062	0.1062	0.2062	0.2462	1.0062	1.0062	10.0062	10.0062
Mean recovery, µg/L	0.0075	0.0049	0.0373	0.0348	0.0701	0.083	0.3674	0.3702	9.2949	9.2115
Percent recovery	121.0	79.0	35.1	32.8	34.0	33.7	36.5	36.8	92.9	92.1
Overall standard deviation, S_t, µg/L	0.0067	0.0051	0.0433	0.0417	0.0705	0.1037	0.4129	0.3944	1.0013	1.0956
Overall relative standard deviation, %	89.7	104.4	116.0	119.7	100.6	124.9	112.4	106.5	10.8	11.9
Number of retained pairs	6		7		7		7		7	
Mean recovery (pairs), µg/L	0.0062		0.0361		0.0766		0.3688		9.2532	
Single operator standard deviation, S_o, µg/L	0.0019		0.0041		0.0272		0.0405		0.212	
Single operator relative standard deviation, %	31.4		11.3		35.5		11.0		2.3	

Table 4-16
Statistical Summary for Silver in Freshwater

	Blank	Blank	+0.07 µg/L	+0.08 µg/L	+0.20 µg/L	+0.20 µg/L	+0.30 µg/L	+0.36 µg/L
Number of retained values	6	6	7	7	7	7	6	6
True concentration, µg/L	0.0060	0.0060	0.0760	0.0860	0.2060	0.2060	0.3060	0.3660
Mean recovery, µg/L	0.0063	0.0058	0.0294	0.0344	0.1221	0.1128	0.0546	0.0658
Percent recovery	105.0	96.7	38.7	40.0	59.3	54.8	17.8	18.0
Overall standard deviation, S_t, µg/L	0.0041	0.0087	0.0247	0.0225	0.0464	0.0421	0.0254	0.029
Overall relative standard deviation, %	66.2	148.6	84.3	65.3	38.0	37.3	46.5	44.1
Number of retained pairs	6		7		7		6	
Mean recovery (pairs), µg/L	0.0061		0.0319		0.1175		0.0602	
Single operator standard deviation, S_o, µg/L	0.0041		0.0039		0.0096		0.0203	
Single operator relative standard deviation, %	67.0		12.3		8.2		33.7	

Table 4-17
Statistical Summary for Thallium in Reagent Grade Water

	Blank	Blank	+0.04 µg/L	+0.05 µg/L	+0.10 µg/L	+0.10 µg/L	+0.50 µg/L	+0.50 µg/L	+2.0 µg/L	+2.0 µg/L
Number of retained values	7	7	7	7	7	7	7	7	7	7
True concentration, µg/L	0.0000	0.0000	0.0400	0.0500	0.1000	0.1000	0.5000	0.5000	2.0000	2.0000
Mean recovery, µg/L	0.0002	0	0.0402	0.0473	0.0971	0.0954	0.4945	0.4873	1.9628	1.9908
Percent recovery	---	---	100.5	94.6	97.1	95.4	98.9	97.5	98.1	99.5
Overall standard deviation, S_t, µg/L	0.0028	0.0029	0.0056	0.0079	0.0058	0.0077	0.0423	0.0257	0.1069	0.1024
Overall relative standard deviation, %	---	---	13.9	16.8	5.9	8.1	8.6	5.3	5.4	5.1
Number of retained pairs	7		7		7		7		7	
Mean recovery (pairs), µg/L	0.0001		0.0438		0.0963		0.4909		1.9768	
Single operator standard deviation, S_o, µg/L	0.0003		0.0044		0.0027		0.0223		0.0297	
Single operator relative standard deviation, %	294.4		10.1		2.8		4.5		1.5	

Table 4-18
Statistical Summary for Thallium in Freshwater

	Blank	Blank	+0.02 µg/L	+0.023 µg/L	+0.06 µg/L	+0.06 µg/L	+0.10 µg/L	+0.12 µg/L
Number of retained values	6	7	7	7	7	7	7	7
True concentration, µg/L	0.0010	0.0010	0.0210	0.0240	0.0610	0.0610	0.1010	0.1210
Mean recovery, µg/L	0.0005	0.0006	0.0188	0.0218	0.0595	0.0581	0.0965	0.1157
Percent recovery	50.0	60.0	89.5	90.8	97.5	95.2	95.5	95.6
Overall standard deviation, S_t, µg/L	0.0006	0.0006	0.0035	0.0052	0.0078	0.0052	0.0082	0.0097
Overall relative standard deviation, %	125.5	100.1	18.7	24.0	13.2	9.0	8.5	8.4
Number of retained pairs	6		7		7		7	
Mean recovery (pairs), µg/L	0.0006		0.0203		0.0588		0.1061	
Single operator standard deviation, S_o, µg/L	0.0004		0.0016		0.0028		0.0025	
Single operator relative standard deviation, %	81.8		7.9		4.8		2.3	

Table 4-19
Statistical Summary for Zinc in Reagent Grade Water

	Blank	Blank	+0.5 µg/L	+0.5 µg/L	+2.0 µg/L	+2.4 µg/L	+5.0 µg/L	+5.0 µg/L	+50.0 µg/L	+50.0 µg/L
Number of retained values	6	6	6	6	7	6	7	7	6	6
True concentration, µg/L	0.1304	0.1304	0.6304	0.6304	2.1304	2.5304	5.1304	5.1304	50.1304	50.1304
Mean recovery, µg/L	0.4275	0.2537	0.5381	0.6174	2.3653	2.3323	4.8709	5.3294	47.2408	50.514
Percent recovery	327.8	194.6	85.4	97.9	111.0	92.2	94.9	103.9	94.2	100.8
Overall standard deviation, S_t, µg/L	0.7378	0.429	0.2024	0.3233	0.8147	0.3371	0.4551	0.9371	3.7256	5.8547
Overall relative standard deviation, %	172.6	169.1	37.6	52.4	34.4	14.5	9.3	17.6	7.9	11.6
Number of retained pairs	6		6		6		7		6	
Mean recovery (pairs), µg/L	0.3473		0.5778		2.3501		5.1002		48.8774	
Single operator standard deviation, S_o, µg/L	0.1031		0.1308		0.4954		0.4857		4.2774	
Single operator relative standard deviation, %	29.7		22.6		21.1		9.5		8.8	

Table 4-20
Statistical Summary for of Zinc in Freshwater

	Blank	Blank	+0.13 µg/L	+0.33 µg/L	+0.53 µg/L	+0.53 µg/L	+0.82 µg/L	+1.22 µg/L
Number of retained values	6	7	7	7	7	6	7	7
True concentration, µg/L	0.9413	0.9413	1.0713	1.2713	1.4713	1.4713	1.7613	2.1613
Mean recovery, µg/L	0.8165	0.9784	0.9967	1.2711	2.3827	1.5244	1.7416	2.0511
Percent recovery	86.7	103.9	93.0	100.0	161.9	103.6	98.9	94.9
Overall standard deviation, S_t, µg/L	0.2277	0.4371	0.2544	0.3173	1.5246	0.4571	0.3092	0.2977
Overall relative standard deviation, %	27.9	44.7	25.5	25.0	64.0	30.0	17.8	14.5
Number of retained pairs	6		7		6		7	
Mean recovery (pairs), µg/L	0.9037		1.1339		1.9866		1.8964	
Single operator standard deviation, S_o, µg/L	0.1934		0.0977		0.5636		0.1055	
Single operator relative standard deviation, %	21.4		8.6		28.4		5.6	

The following plots for reagent grade water and freshwater are presented in this section:

- Single operator precision: single operator standard deviation versus true concentration, where the latter is the average of the true values for each member of the pair.
- Overall precision: overall standard deviation versus true concentration
- Single operator relative standard deviation (RSD) versus mean concentration
- Overall relative standard deviation (RSD) versus mean concentration
- Mean result versus true concentration for both matrices. The 100% recovery line is plotted along with the recovery line for each matrix.

Each of the precision plots includes a regression line fit to the standard deviation versus true concentration data across the sample concentration range. The selection of the model that best fits the regression is incorporated into the calculation of the Interlaboratory Detection Estimate (IDE) and Interlaboratory Quantitation Estimate (IQE), described in Chapter 5.

The RSDs in the unspiked reagent grade water and freshwater are often quite high since these concentrations are so low. For most analytes, an RSD plot omitting low concentrations is included to show the plot on an expanded scale.

4.2.1.1 Antimony

Figure 4-1 shows the single operator (within laboratory) standard deviation data versus true concentration for antimony in reagent grade water and freshwater. A weighted least squares Rocke-Lorenzato ("hockey stick") curve fit is plotted across the concentration range for reagent grade water. The reagent grade water data exhibit increasing single operator standard deviation with increasing concentration. In the concentration range studied for freshwater, the single operator standard deviation data are clustered near the flat end of the reagent grade water "hockey stick" and are best fit by a constant model horizontal straight line.

The overall (among laboratories) standard deviation versus true concentration data for antimony in reagent grade water and freshwater are plotted in Figure 4-2. The reagent grade water data exhibit a trend of increasing standard deviation with increasing concentration. A Rocke-Lorenzato curve fit to the reagent grade water data is shown on the plot. As with the single operator data, the overall standard deviation data for freshwater are best fit with the constant model.

Figure 4-3 plots the single operator relative standard deviation (RSD) against mean concentration for antimony in both reagent grade water and freshwater. The RSD for the unspiked reagent grade water pair is quite high (199%) and has been omitted to make it easier to interpret the remaining data. The high RSD at the unspiked concentration level is not unexpected since the samples did not contain measurable antimony, yet the study required the reporting of measured values for each analyte even if such values were below detection limits. The data exhibit the expected trend of decreasing single operator RSD with increasing concentration, leveling off to less than 5% single operator RSDs in both matrices at concentrations higher than approximately 0.8 μg/L antimony.

The overall RSD data for antimony in reagent grade water and freshwater are plotted in Figure 4-4. The data from the unspiked pair of reagent grade water samples (RSDs 276% and 1028%) have been omitted from the plot. In general, the overall RSDs decrease with increasing concentration, leveling off to approximately 1% to 2% overall RSD in freshwater and approximately 5% overall RSD in reagent grade water.

Plots of recovery (mean result versus true concentration) for antimony in reagent grade water and freshwater are shown in Figures 4-5 (all concentration levels) and 4-6 (highest concentration level omitted for readability). The 100% recovery line is shown on the plot along with the mean recovery regression plots for reagent grade water and freshwater. At low concentrations (<5.0 μg/L), the recoveries in reagent grade water are less than 70%. At higher concentrations, the recoveries are within 80% to 90%. Recoveries in freshwater were within 90% to 100% across the entire concentration range studied.

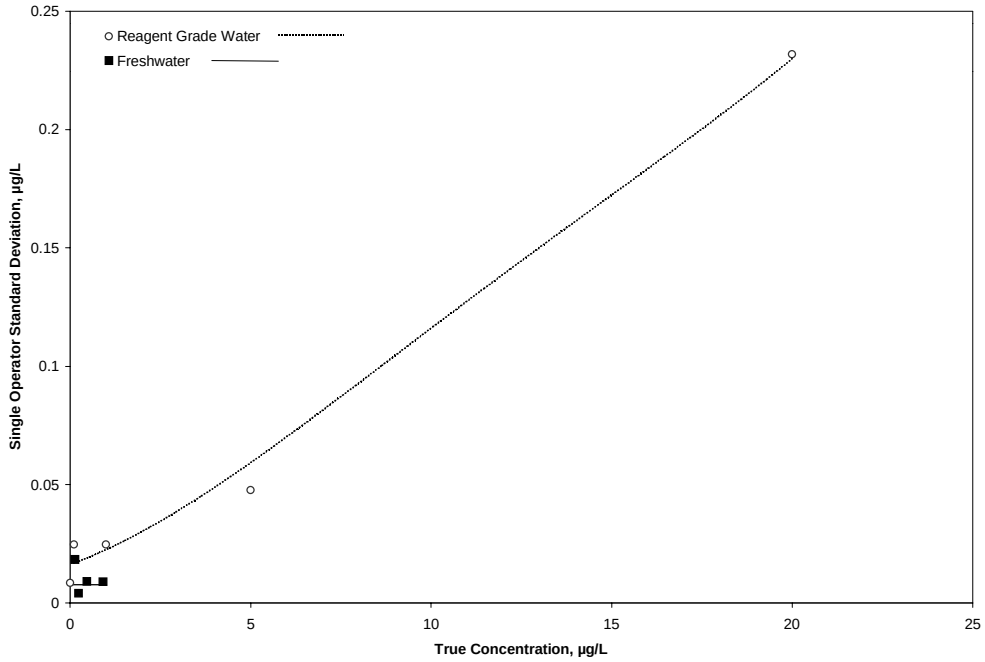

Figure 4-1
Plot of single operator standard deviation versus true concentration for antimony in reagent grade water and freshwater

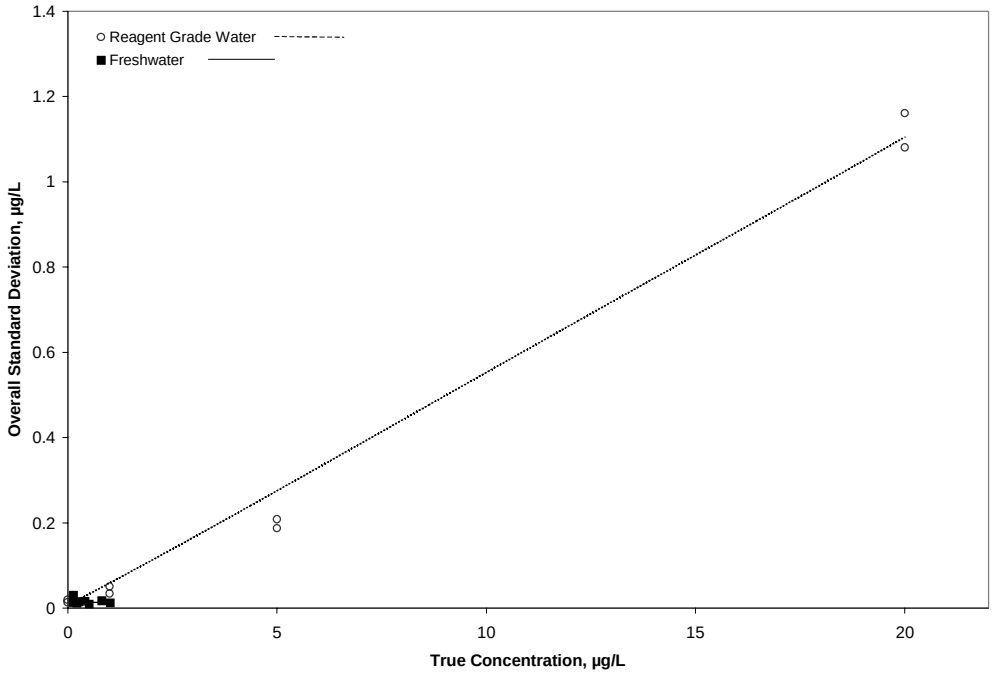

Figure 4-2
Plot of overall standard deviation versus true concentration for antimony in reagent grade water and freshwater

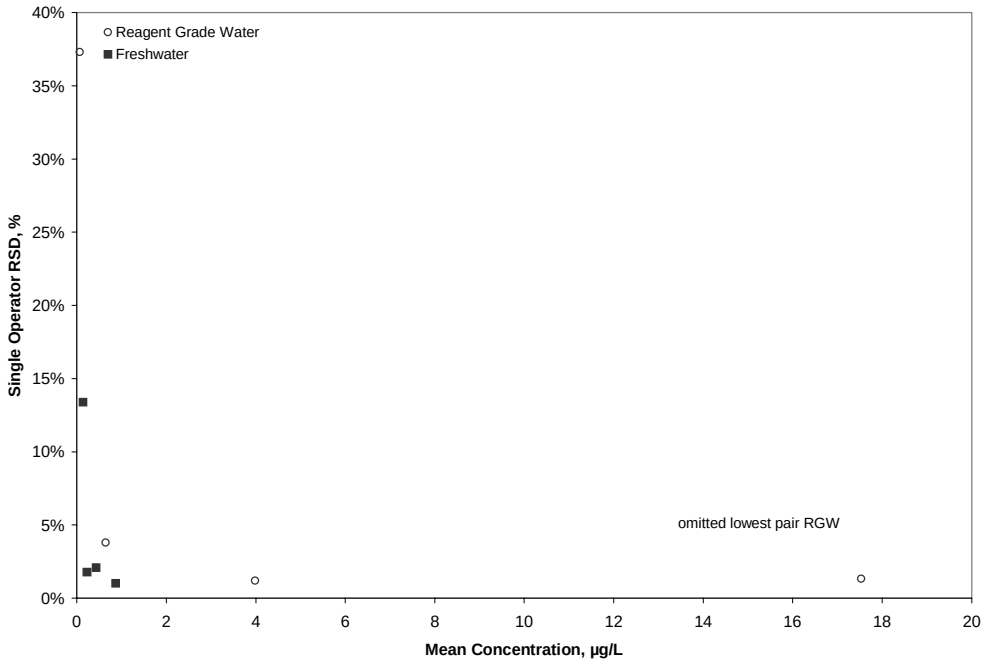

Figure 4-3
Plot of single operator relative standard deviation versus mean recovery for antimony in reagent grade water and freshwater (unspiked reagent grade water data omitted)

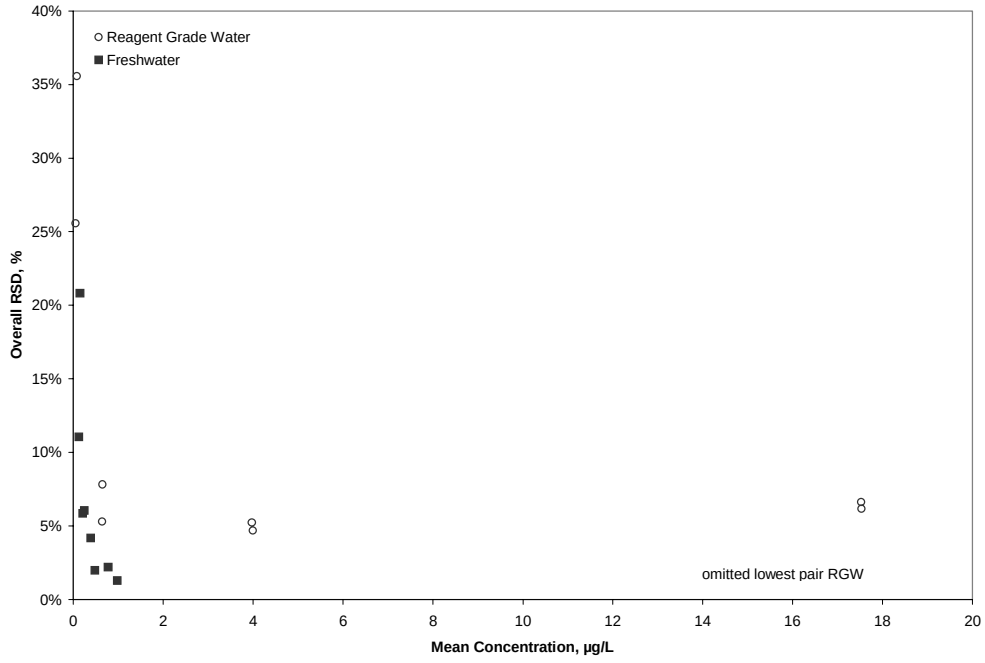

Figure 4-4
Plot of overall relative standard deviation versus mean recovery for antimony in reagent grade water and freshwater (unspiked reagent grade water data omitted)

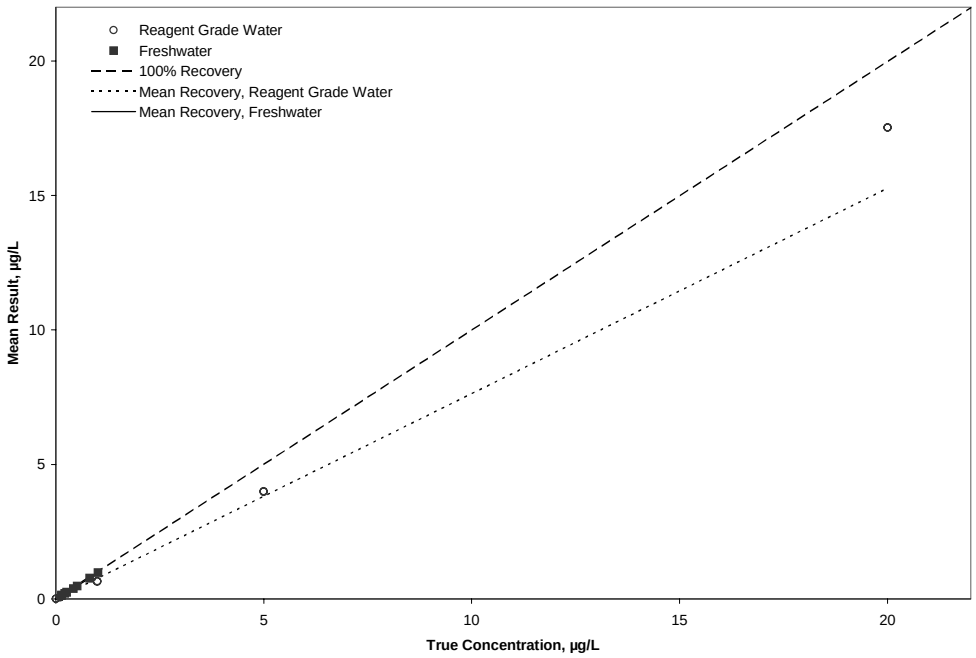

Figure 4-5
Plot of mean result versus true concentration for antimony in reagent grade water and freshwater (all concentration levels)

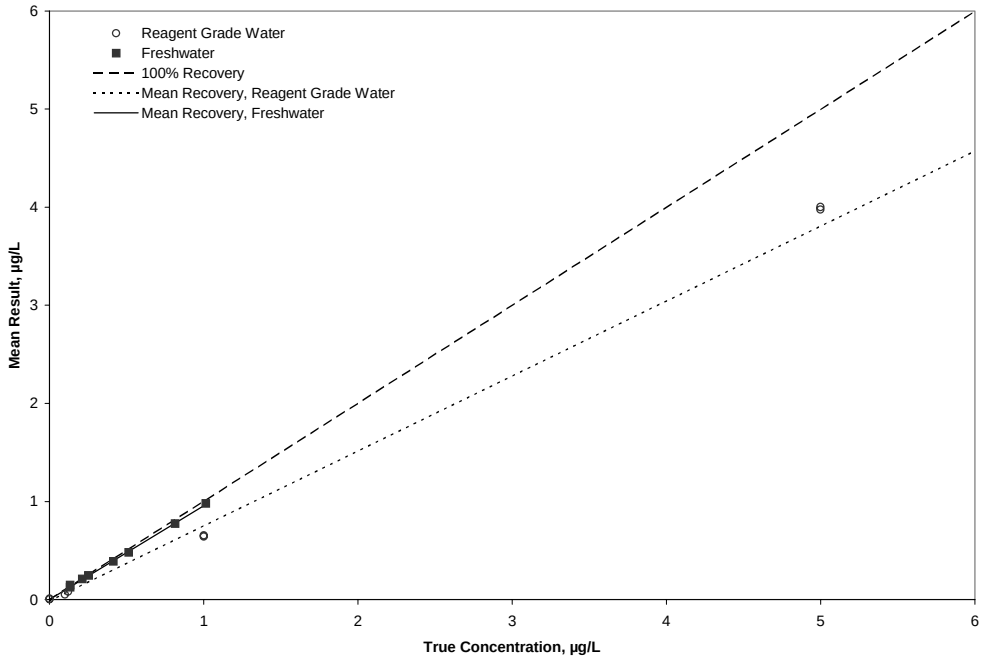

Figure 4-6
Plot of mean result versus true concentration for antimony in reagent grade water and freshwater (highest concentration level omitted)

4.2.1.2 Cadmium

Figure 4-7 shows the single operator precision (standard deviation versus true concentration) data for cadmium in reagent grade water and freshwater. The Rocke-Lorenzato fits to the data for each matrix are shown on the plot. Compared to reagent grade water, the data for freshwater show a trend of more sharply increasing single operator standard deviation with increasing concentration in the concentration range studied in freshwater.

The overall precision data for cadmium in reagent grade water and freshwater are shown in Figure 4-8. The Rocke-Lorenzato fit to the reagent grade water shows increasing standard deviation with increasing concentration. In the concentration range studied in freshwater, the overall standard deviation versus true concentration data are best fit by a constant model.

The single operator percent RSD versus mean concentration data for cadmium in both the reagent grade water and freshwater matrices are plotted in Figure 4-9. The data for the unspiked reagent grade water (RSD = 783%) and unspiked freshwater (RSD = -205%) have been omitted to better show the data for the higher concentrations. In reagent grade water, the single operator RSD data show the expected trend of generally decreasing single operator percent RSD with increasing concentration, decreasing to approximately 1% RSD near 10 μg/L cadmium. The freshwater single operator RSD data are more scattered, with RSDs of 4% to 6% between 0.1 μg/L and 0.25 μg/L.

The overall RSD data for cadmium in reagent grade water and freshwater are shown in Figure 4-10 (unspiked pair reagent grade water and freshwater omitted). The data exhibit decreasing overall percent RSD with increasing cadmium concentration, with approximately 10% overall RSD near 0.25 μg/L in freshwater and 6% overall RSD near 10 μg/L in reagent grade water.

The recovery (mean result versus true concentration) plot for all study concentrations of cadmium in reagent grade water and freshwater is shown in Figures 4-11. An expanded scale version of the plot is shown in Figure 4-12 with the highest concentration level in reagent grade water omitted from the plot. Recoveries of the cadmium spikes into reagent grade water were all in the 90% - 100% range. In freshwater, except for the lowest spiked concentration which had a recovery of 72%, recoveries were also in the 90% - 100% range.

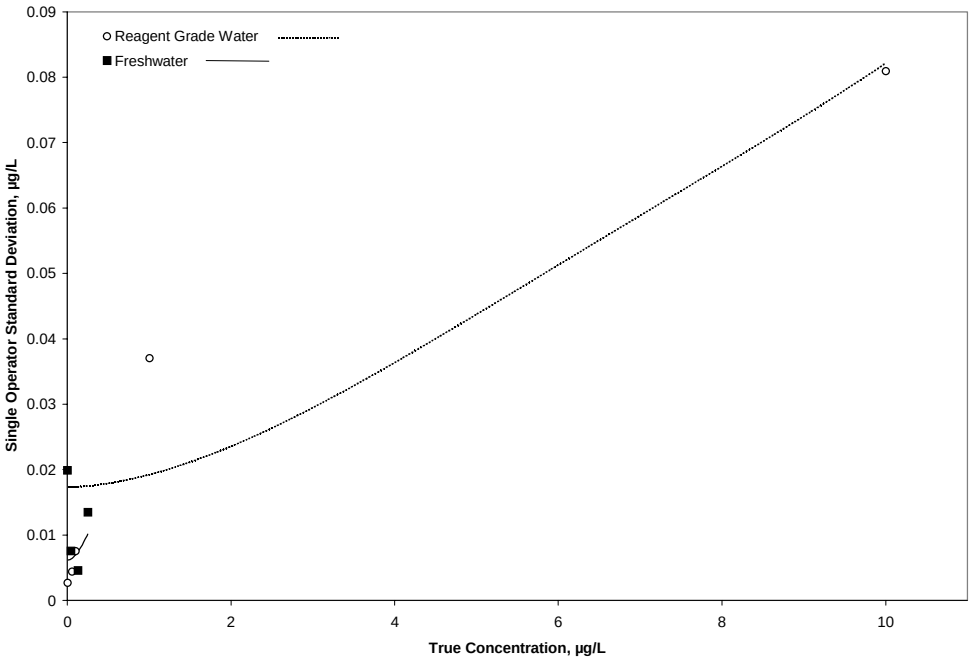

Figure 4-7
Plot of single operator standard deviation versus true concentration for cadmium in reagent grade water and freshwater

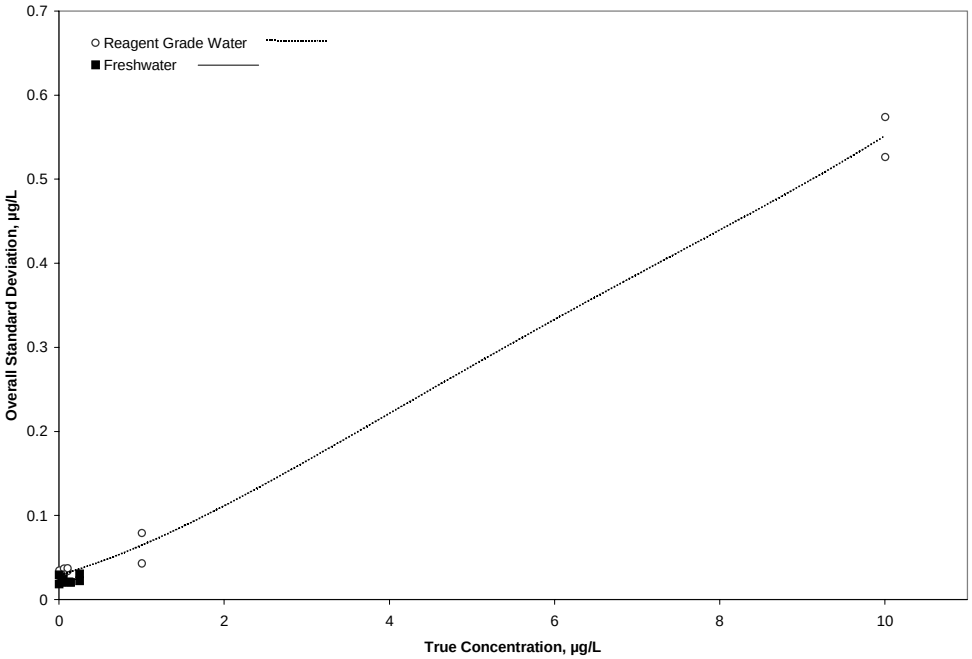

Figure 4-8
Plot of overall standard deviation versus true concentration for cadmium in reagent grade water and freshwater

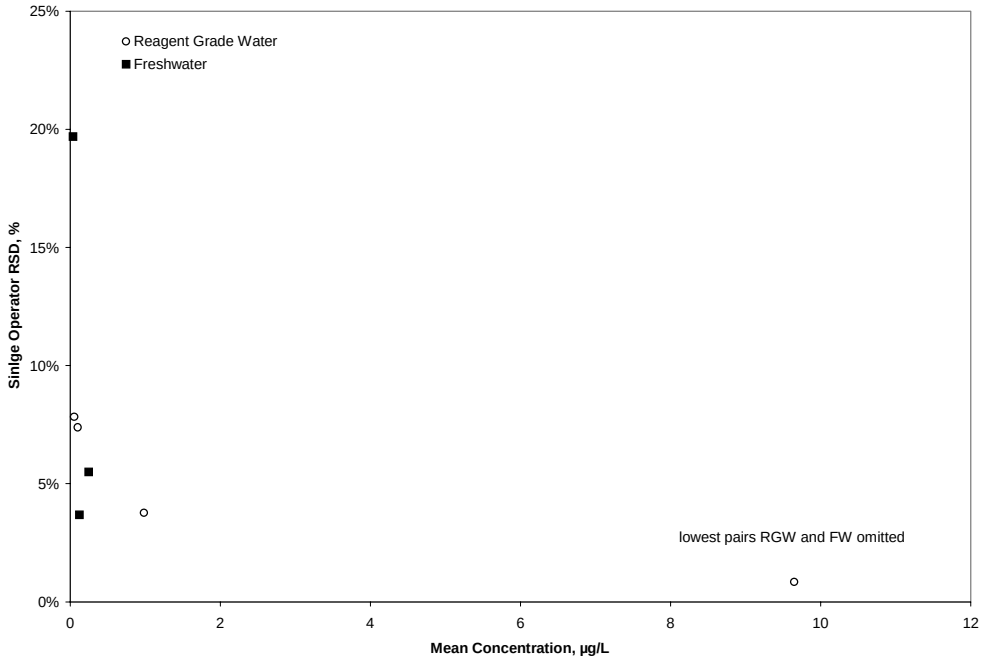

Figure 4-9
Plot of single operator relative standard deviation versus mean recovery for cadmium in reagent grade water and freshwater (unspiked reagent grade water and freshwater data omitted)

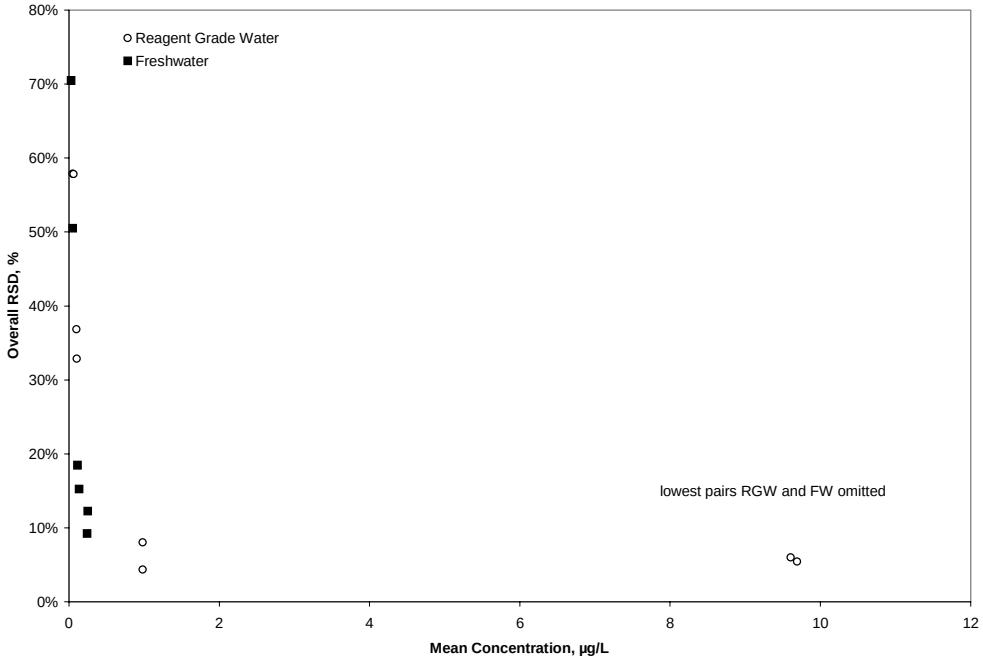

Figure 4-10
Plot of overall relative standard deviation versus mean recovery for cadmium in reagent grade water and freshwater (unspiked reagent grade water and freshwater data omitted)

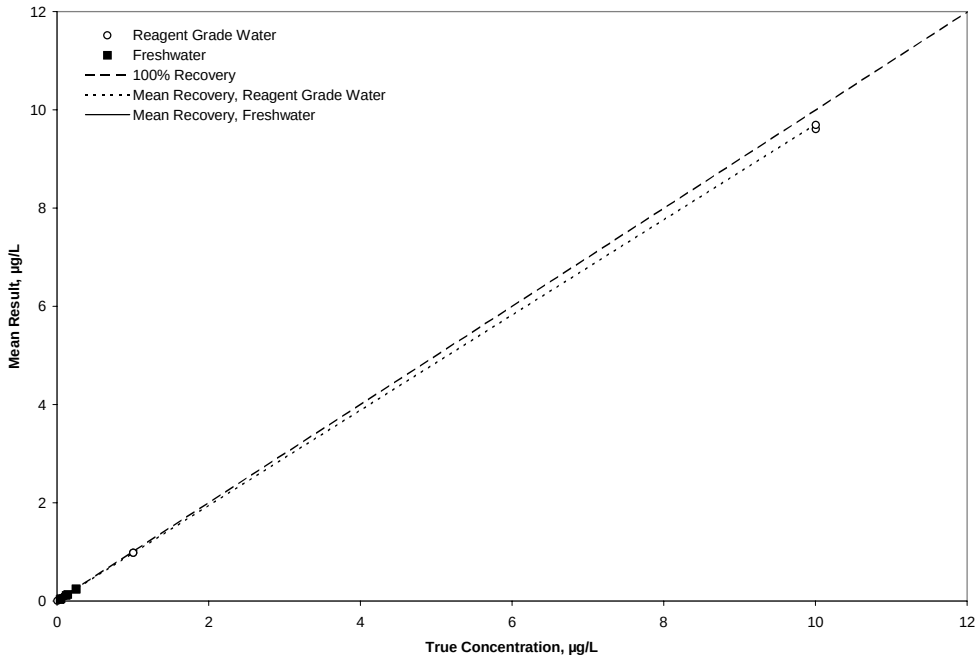

Figure 4-11
Plot of mean result versus true concentration for cadmium in reagent grade water and freshwater (all concentration levels)

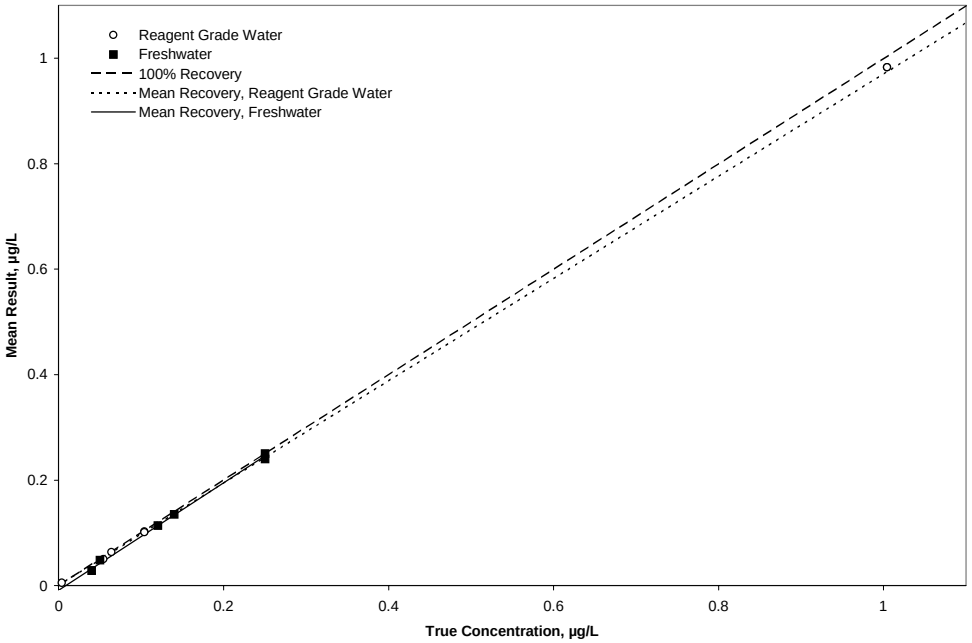

Figure 4-12
Plot of mean result versus true concentration for cadmium in reagent grade water and freshwater (highest concentration level omitted)

4.2.1.3 Copper

Figure 4-13 shows the single operator standard deviation versus true concentration (precision) data for copper in reagent grade water and freshwater. The single operator precision data for both matrices are best fit by a constant model (horizontal line). Based on the results of Cochran's test for the homogeneity of variances, the standard deviation data for reagent grade water with 2.1 μg/L copper were omitted from the curve fit. In the concentration ranges studied, the standard deviation data for both reagent grade water and freshwater were similar.

The overall precision data for copper in reagent grade water and freshwater are plotted in Figure 4-14. The data for reagent grade water exhibit a general trend of increasing standard deviation with increasing concentration. The Rocke-Lorenzato fit to the data is plotted. The overall standard deviation data for copper in freshwater are best fit by a constant model.

The single operator RSD data versus mean concentration for copper in both reagent grade water and freshwater are plotted in Figure 4-15. The lowest concentration pair in reagent grade water has been omitted because there were fewer than 6 pairs of data remaining. The data show decreasing RSD with increasing concentration. In freshwater, the single operator RSDs level off to <5% near 5 μg/L. The single operator RSDs in reagent grade water are <1% near 20 μg/L.

The overall percent RSD data are shown in Figure 4-16. In general, the data show a trend of decreasing percent RSD with increasing concentration. Both the reagent grade water and freshwater RSDs level off to <10% at the highest concentrations studied in the matrices.

Figure 4-17 shows the recovery plot for copper in reagent grade water and freshwater at all study concentrations. An expanded scale version of the recovery data plot is shown in Figure 4-18 with the highest concentration pair in reagent grade water omitted. In reagent grade water, mean recoveries at the lowest concentration averaged 75%. At the higher concentrations, mean recoveries in reagent grade water ranged from 95% to 115%. Recoveries in freshwater were between 85% and 100% across the study concentration range.

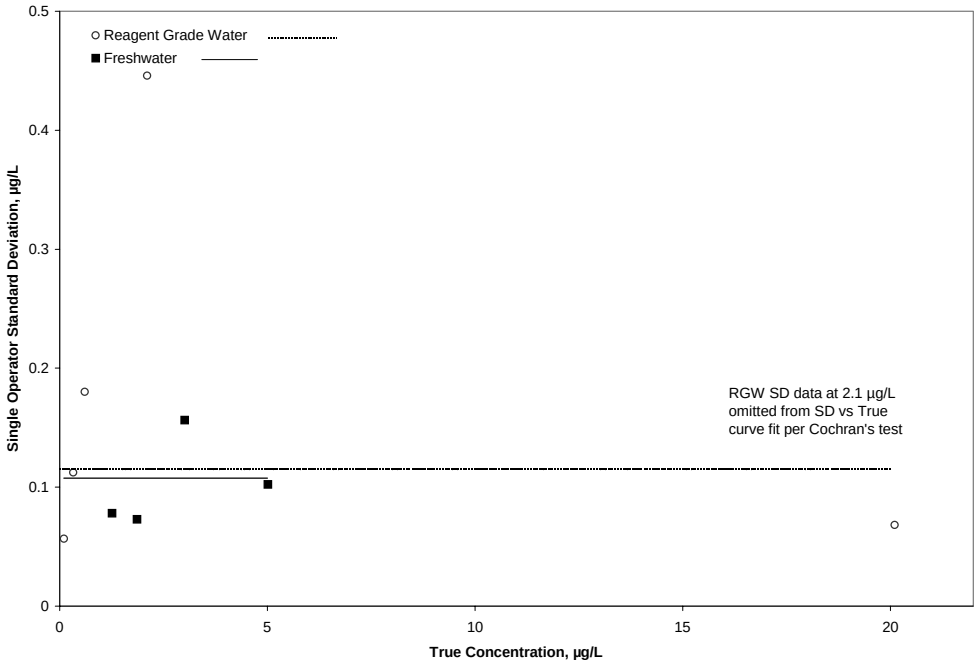

Figure 4-13
Plot of single operator standard deviation versus true concentration for copper in reagent grade water and freshwater

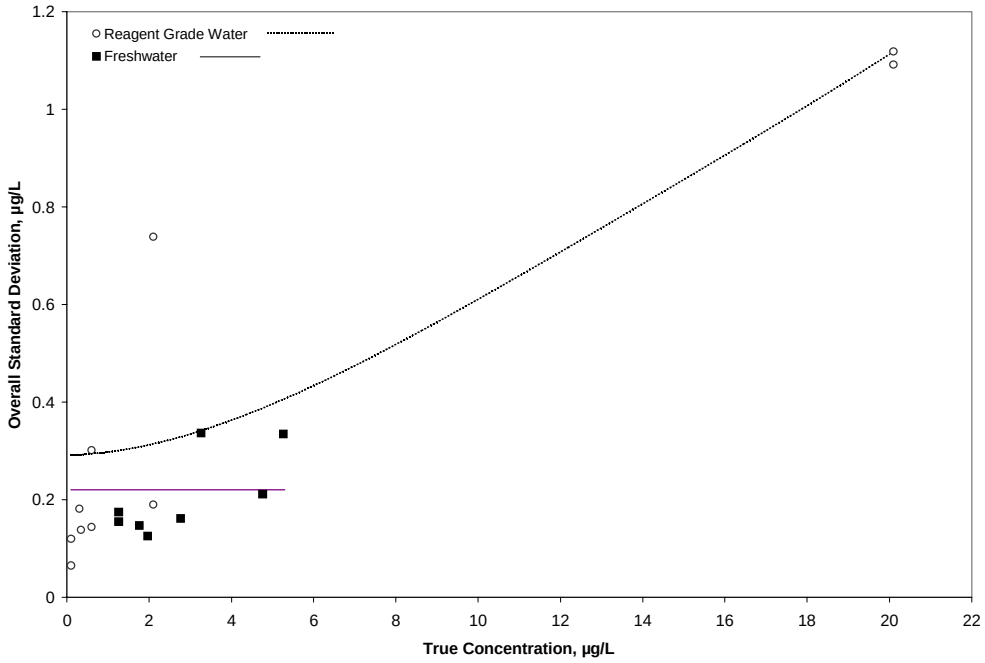

Figure 4-14
Plot of overall standard deviation versus true concentration for copper in reagent grade water and freshwater

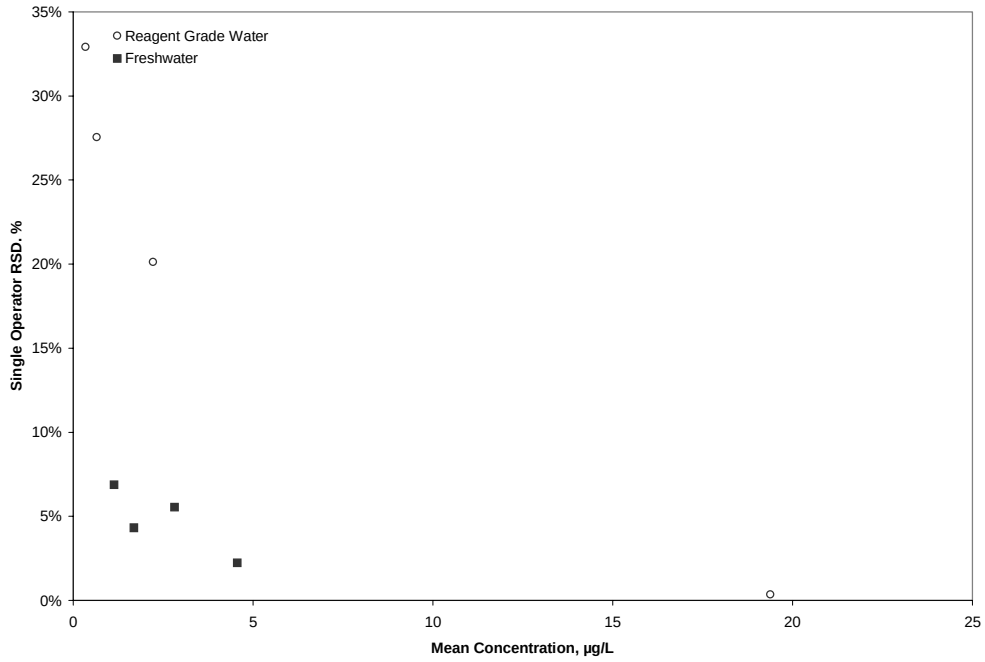

Figure 4-15
Plot of single operator relative standard deviation versus mean recovery for copper in reagent grade water and freshwater

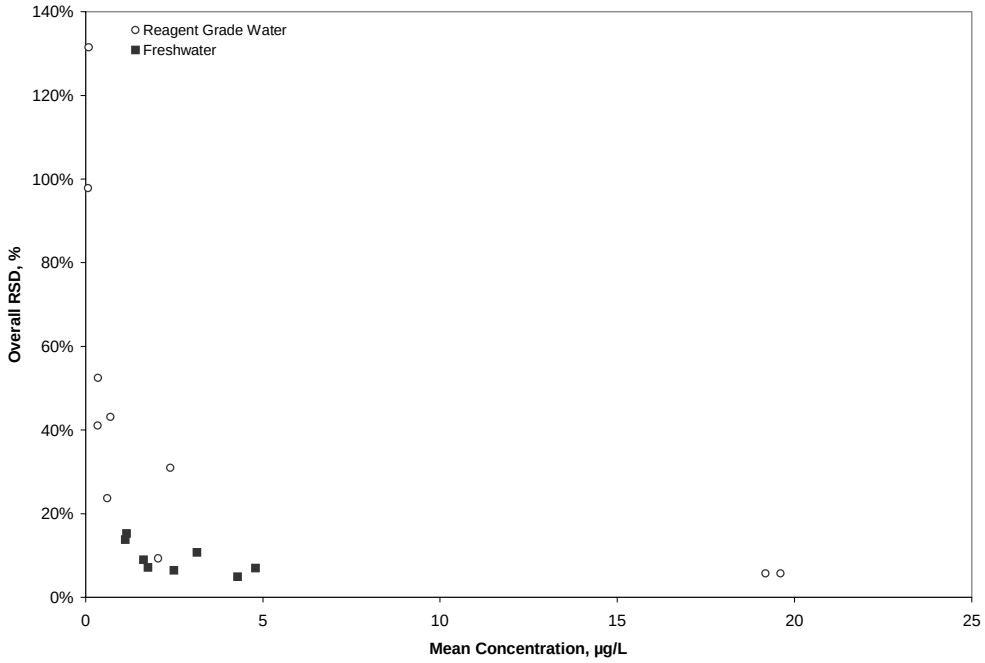

Figure 4-16
Plot of overall relative standard deviation versus mean recovery for copper in reagent grade water and freshwater

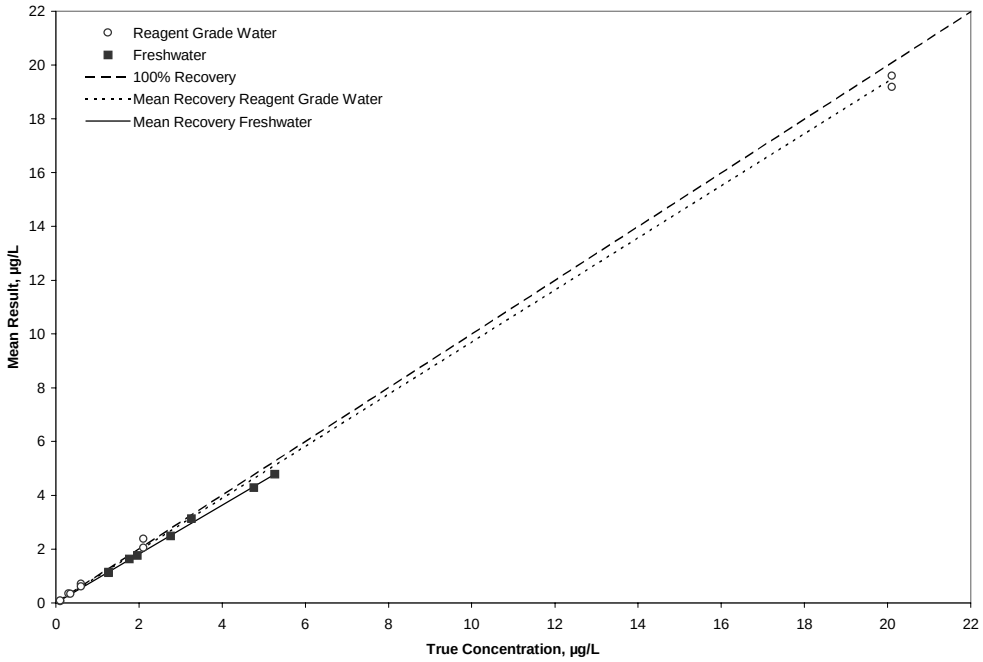

Figure 4-17
Plot of mean result versus true concentration for copper in reagent grade water and freshwater (all concentration levels)

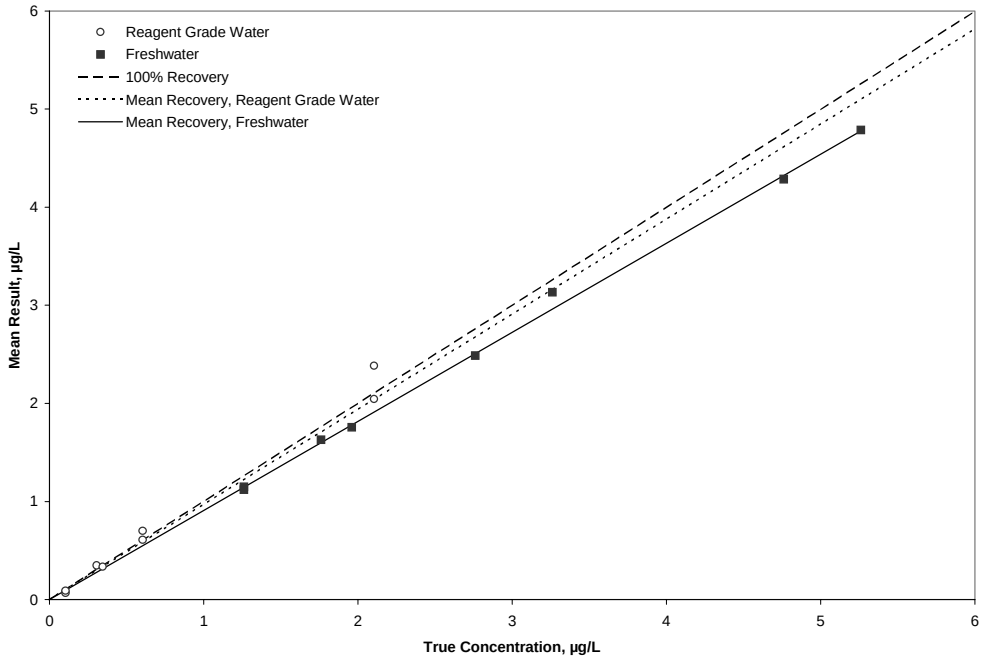

Figure 4-18
Plot of mean result versus true concentration for copper in reagent grade water and freshwater (highest concentration level omitted)

4.2.1.4 Lead

The single operator precision data for lead in reagent grade water and freshwater are shown in Figure 4-19. The reagent grade water display a trend of increasing standard deviation with increasing concentration. The Rocke-Lorenzato fit to the reagent grade water is shown on the plot. In the concentration range studied in freshwater, the data are best fit by a constant model, shown as a horizontal line on the plot.

The overall precision plot for lead in reagent grade water and freshwater is shown in Figure 4-20. The overall standard deviation data for reagent grade water exhibit a trend of increasing overall standard deviation with increasing concentration. The Rocke-Lorenzato fit to the reagent grade water data is included on the plot. The freshwater overall standard deviation data are best fit by a constant model over the concentration range studied, shown on the plot as a horizontal line. The freshwater overall standard deviation data correlate with the flat part of the Rocke-Lorenzato "hockey stick" for reagent grade water.

Figure 4-21 shows the plot of single operator percent RSD versus mean concentration for reagent grade water and freshwater. For reagent grade water, data at the lowest concentration and the next to highest concentration pairs are not included in the plot since only five pairs of data remained at these concentrations after outlier removal. The overall percent RSD is shown in Figure 4-22. The data exhibit a general trend of decreasing percent RSD with increasing lead concentration in both reagent grade water and freshwater.

The recovery data for lead in reagent grade water and freshwater are plotted in Figure 4-23 for all study concentrations. Figure 4-24 shows the same plot with the data for the highest concentration of reagent grade water omitted for readability. Above approximately 0.08 μg/L lead, mean recoveries were between 87% and 108% in both matrices. At approximately 0.05 μg/L, mean recoveries in reagent grade water were between 115% and 169%. Near the same concentration level in freshwater, mean recoveries were between 85% and 143%.

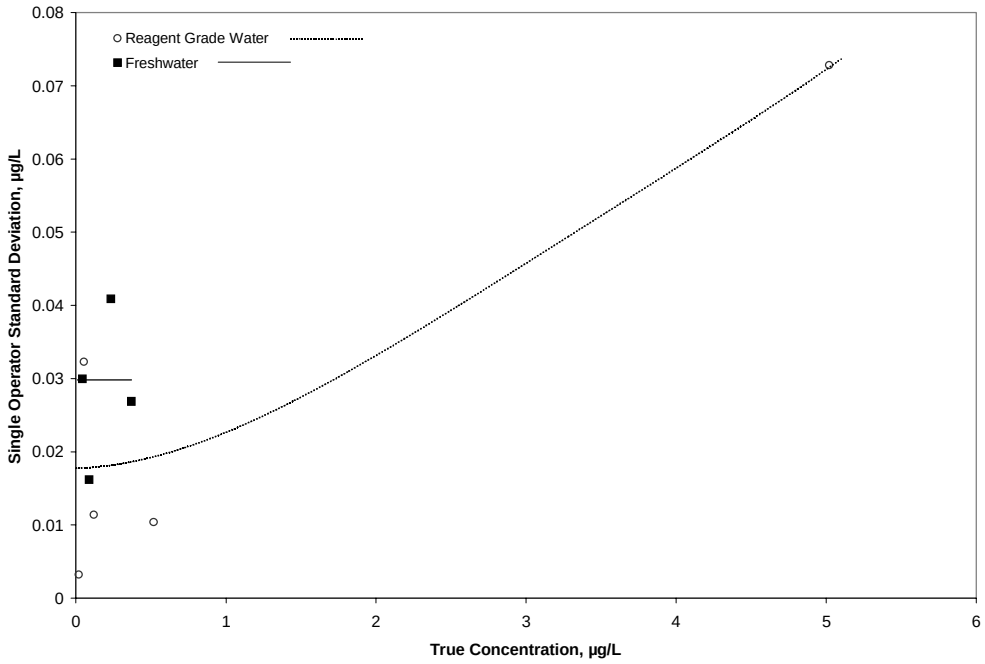

Figure 4-19
Plot of single operator standard deviation versus true concentration for lead in reagent grade water and freshwater

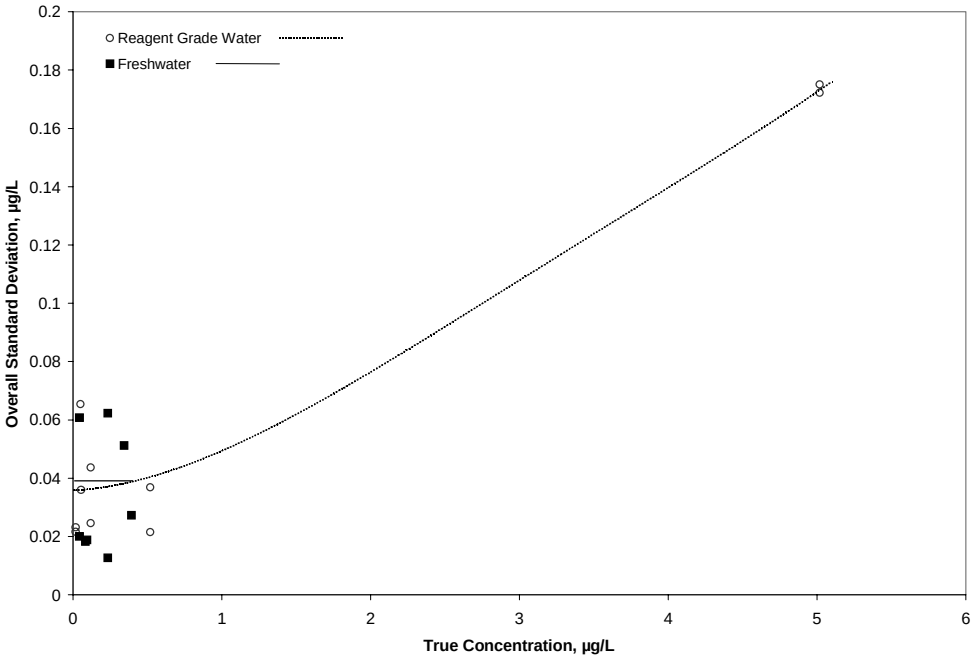

Figure 4-20
Plot of overall standard deviation versus true concentration for lead in reagent grade water and freshwater

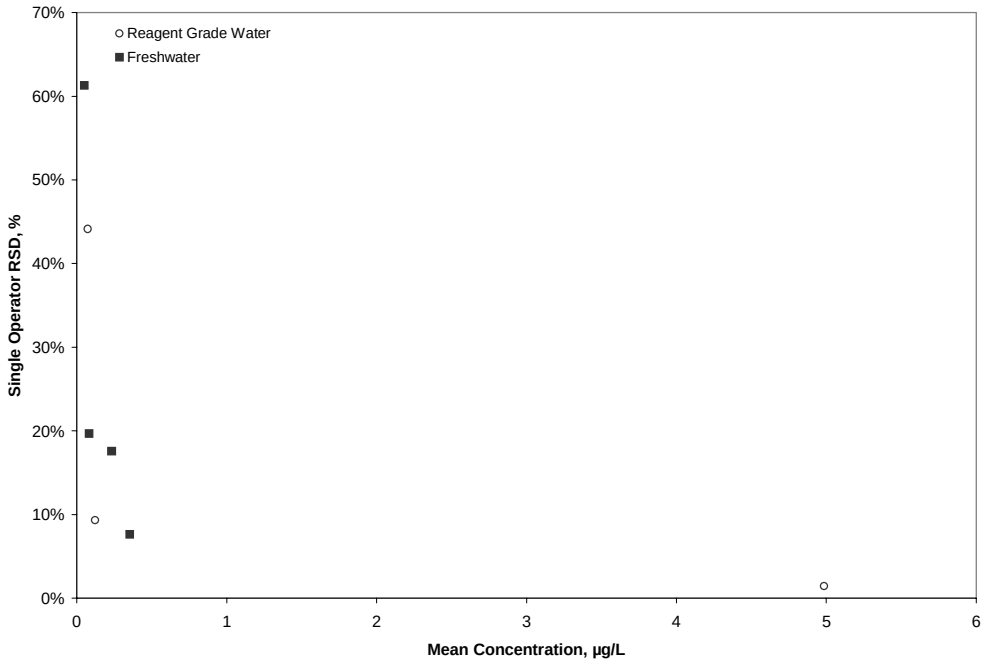

Figure 4-21
Plot of single operator relative standard deviation versus mean recovery for lead in reagent grade water and freshwater (all data)

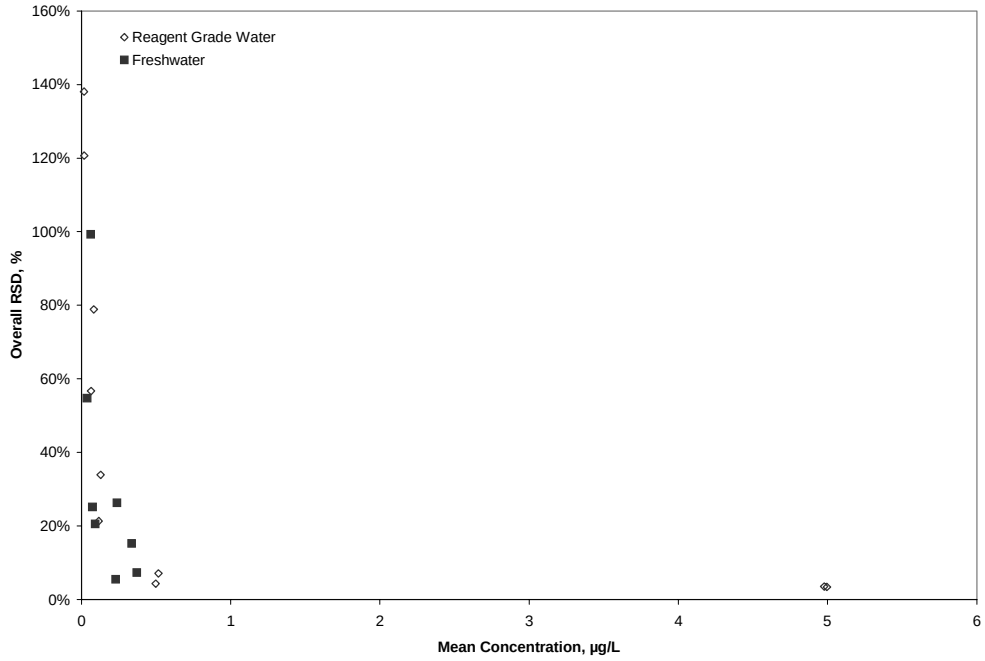

Figure 4-22
Plot of overall relative standard deviation versus mean recovery for lead in reagent grade water and freshwater (all data)

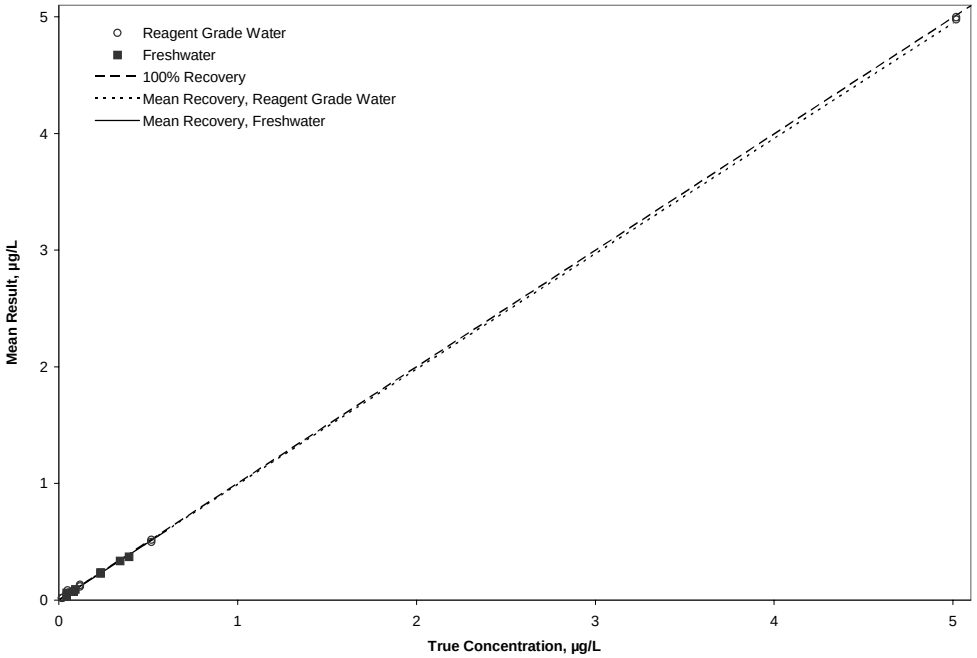

Figure 4-23
Plot of mean result versus true concentration for lead in reagent grade water and freshwater (all concentration levels)

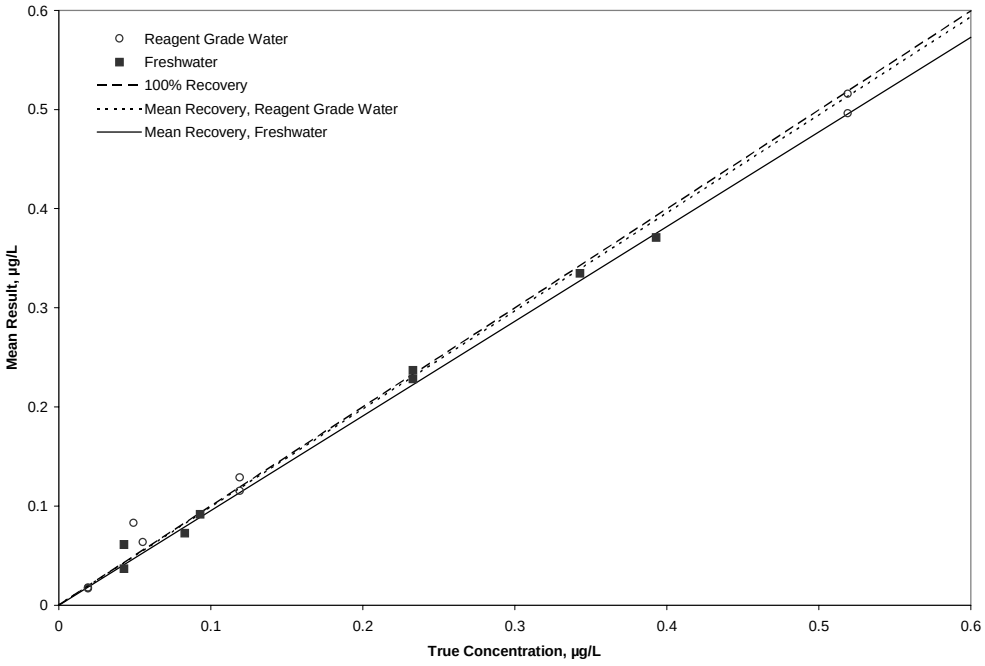

Figure 4-24
Plot of mean result versus true concentration for lead in reagent grade water and freshwater (highest concentration level omitted)

4.2.1.5 Nickel

Data from only five laboratories remained at the highest spiked concentration of nickel in reagent grade water, 100 µg/L, after outlier data removal. ASTM D2777 requires data from a minimum of six laboratories. The 100 µg/L data for nickel in reagent grade water was omitted from all further calculations.

The single operator standard deviation versus true concentration (precision) plot for nickel in reagent grade water and freshwater is shown in Figure 4-25. A Rocke-Lorenzato fit to the data for each matrix is included on the plot. The data show the expected trend of increasing standard deviation with increasing concentration. Over the concentration range studied, the single operator standard deviation increases more steeply in freshwater than in reagent grade water.

The overall standard deviation versus true concentration plot for nickel in reagent grade water and freshwater, shown in Figure 4-26, exhibits the same trends.

Figure 4-27 shows the plot of single operator RSD versus mean concentration for nickel in reagent grade water and freshwater. The data for the lowest two concentration pairs in reagent grade water have been omitted since fewer than five pairs remained at these concentrations after outlier removal. Single operator RSDs in both matrices were less than 6% across the range of concentrations examined.

The overall RSD data for nickel in reagent grade water and freshwater are plotted in Figure 4-28. The reagent grade water data show generally decreasing overall RSDs with increasing nickel concentration, leveling off to <5% overall RSD above approximately 10 µg/L nickel. With the exception of one of the samples at approximately 6 µg/L, the freshwater data exhibit a trend of decreasing overall RSD with increasing nickel concentration.

The recovery data for all study concentrations of nickel in reagent grade water and freshwater are plotted in Figure 4-29. An expanded plot, omitting the highest concentration nickel data in reagent grade water, is shown in Figure 4-30. Recoveries in both matrices were between 90% and 100% at all study concentrations.

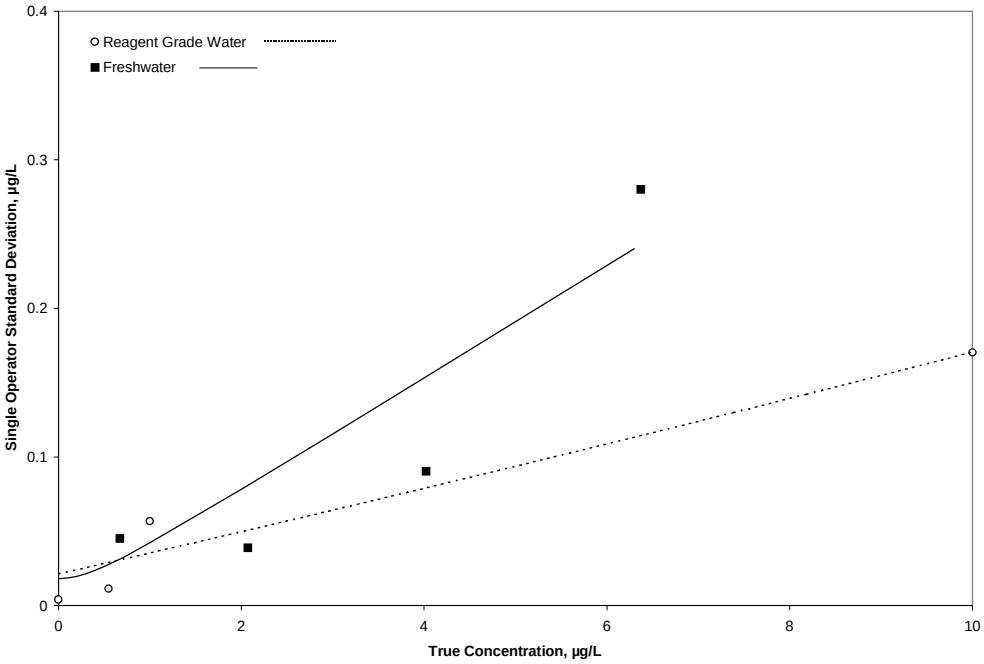

Figure 4-25
Plot of single operator standard deviation versus true concentration for nickel in reagent grade water and freshwater

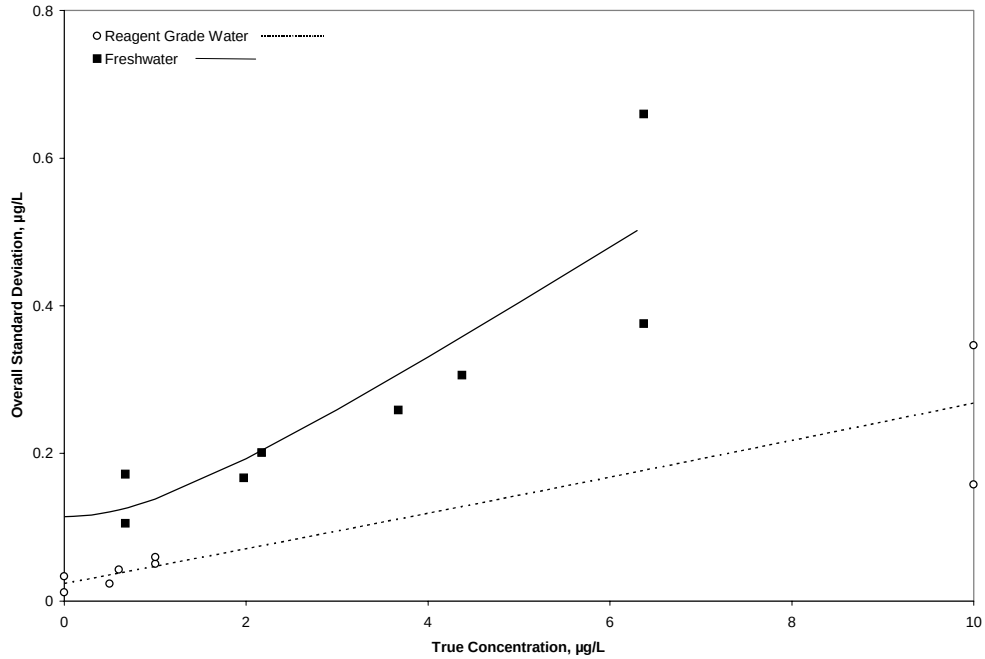

Figure 4-26
Plot of overall standard deviation versus true concentration for nickel in reagent grade water and freshwater

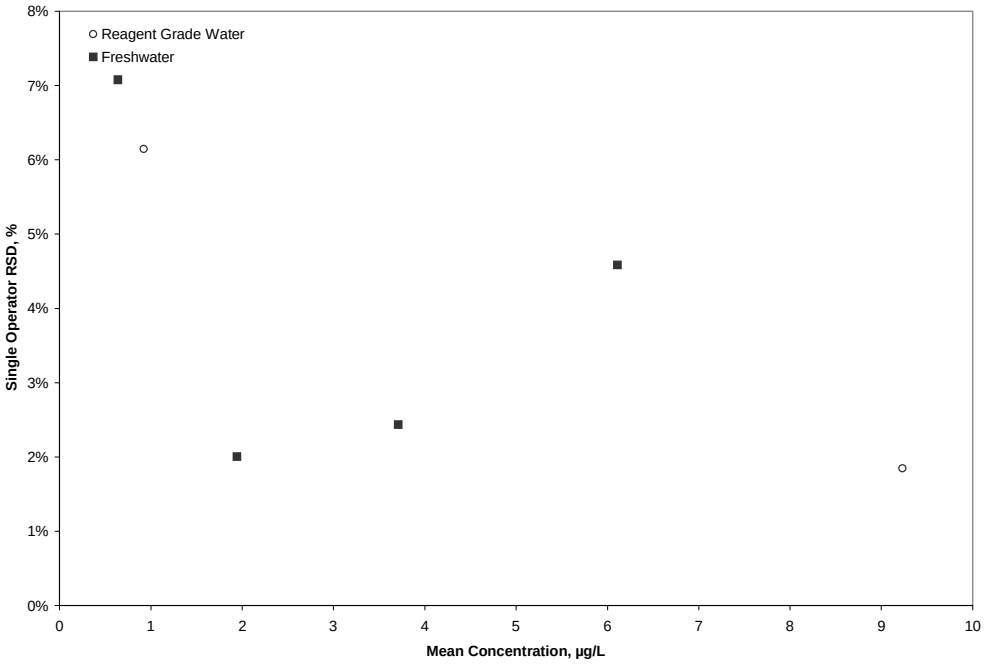

Figure 4-27
Plot of single operator relative standard deviation versus mean recovery for nickel in reagent grade water and freshwater (unspiked reagent grade water data omitted)

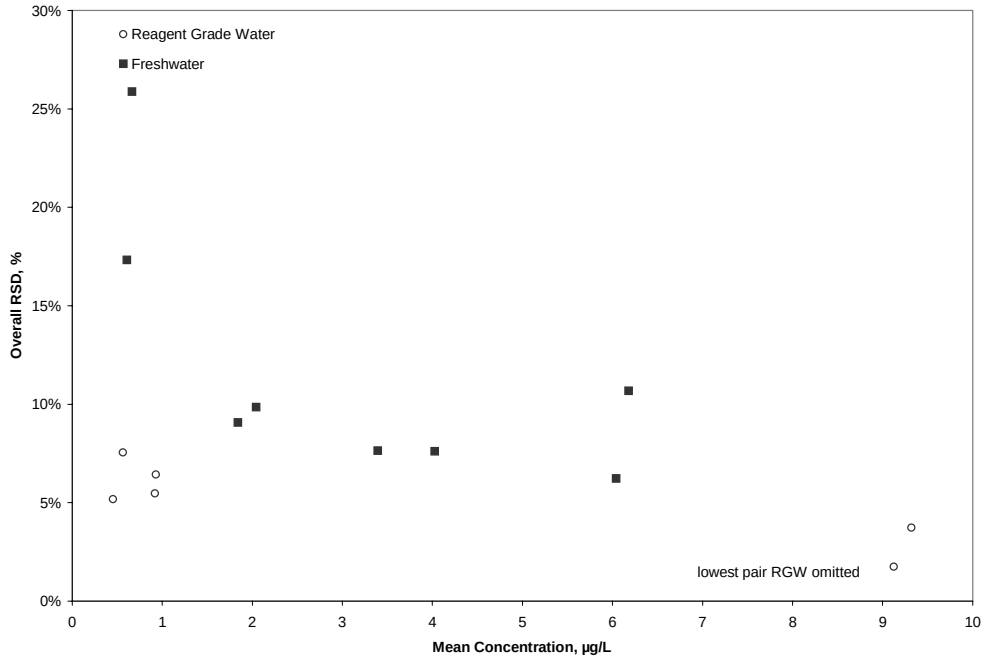

Figure 4-28
Plot of overall relative standard deviation versus mean recovery for nickel in reagent grade water and freshwater (unspiked reagent grade water data omitted)

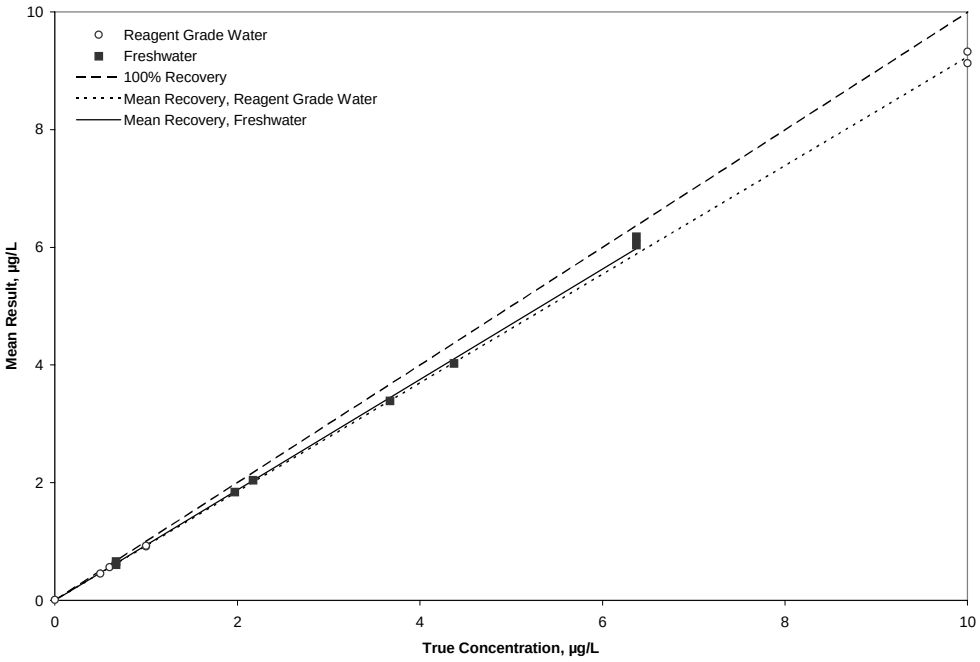

Figure 4-29
Plot of mean result versus true concentration for nickel in reagent grade water and freshwater (all concentration levels)

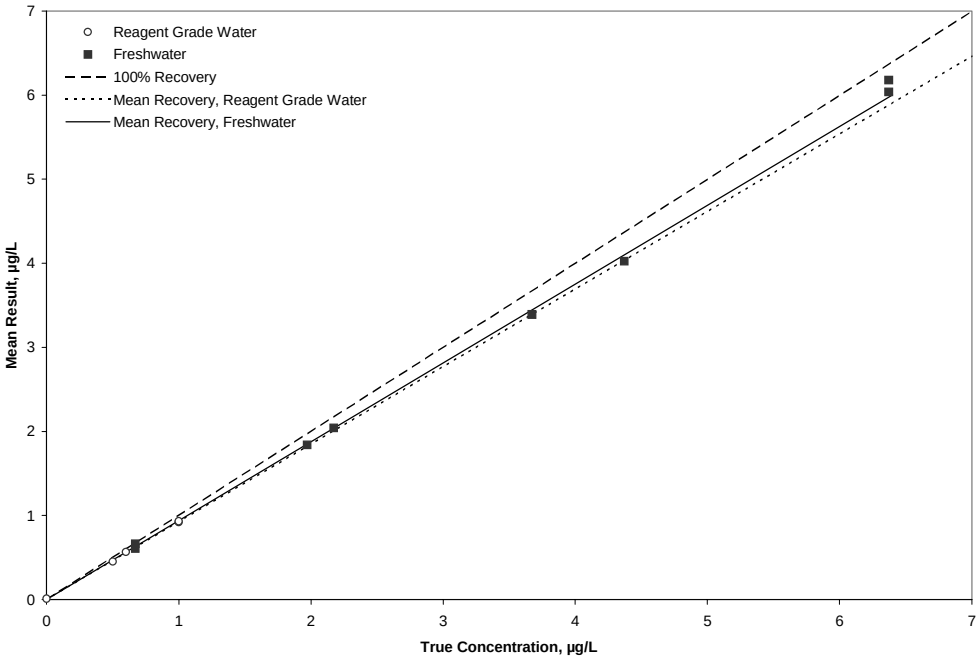

Figure 4-30
Plot of mean result versus true concentration for nickel in reagent grade water and freshwater (highest concentration level omitted)

4.2.1.6 Selenium

Data from only five laboratories remained at the highest spiked concentration of selenium in reagent grade water, 100 µg/L, after outlier data removal. ASTM D2777 requires data from a minimum of six laboratories. The 100 µg/L data for selenium in reagent grade water were omitted from all further calculations.

Figure 4-31 plots the single operator standard deviation versus true concentration data for selenium in reagent grade water and freshwater. Over the concentration ranges studied, both sets of data are best fit by a constant model (horizontal line).

The overall standard deviation versus true concentration data for selenium in reagent grade water and freshwater are plotted in Figure 4-32. The data for reagent grade water show a trend of increasing standard deviation with increasing concentration. A Rocke-Lorenzato fit to the reagent grade water data is plotted. The overall standard deviation data for selenium in freshwater are scattered and best fit with by a constant model, shown on the plot as a horizontal line.

The single operator percent RSD versus mean concentration data for selenium in reagent grade water and freshwater are plotted in Figure 4-33 with the data for the unspiked pair in reagent grade water (RSD = 206%) omitted. A trend of decreasing single operator RSD with increasing selenium concentration is evident in both matrices.

The overall percent RSDs versus mean concentrations for selenium in reagent grade water and freshwater are plotted in Figure 4-34 with the data for the unspiked pair reagent grade water omitted (RSDs = 267% and 193%). A general trend of decreasing overall RSD with increasing selenium concentration is noted in both matrices.

Figure 4-35 plots the recovery of selenium in reagent grade water and freshwater for all study concentrations. In Figure 4-36, the 100 µg/L spike in reagent grade water has been omitted to better show the recoveries at lower concentrations. Recoveries in reagent grade water were between 90% and 100% at all spiked concentration levels. In freshwater, mean recoveries ranged from 90% to 115% for all study concentration levels.

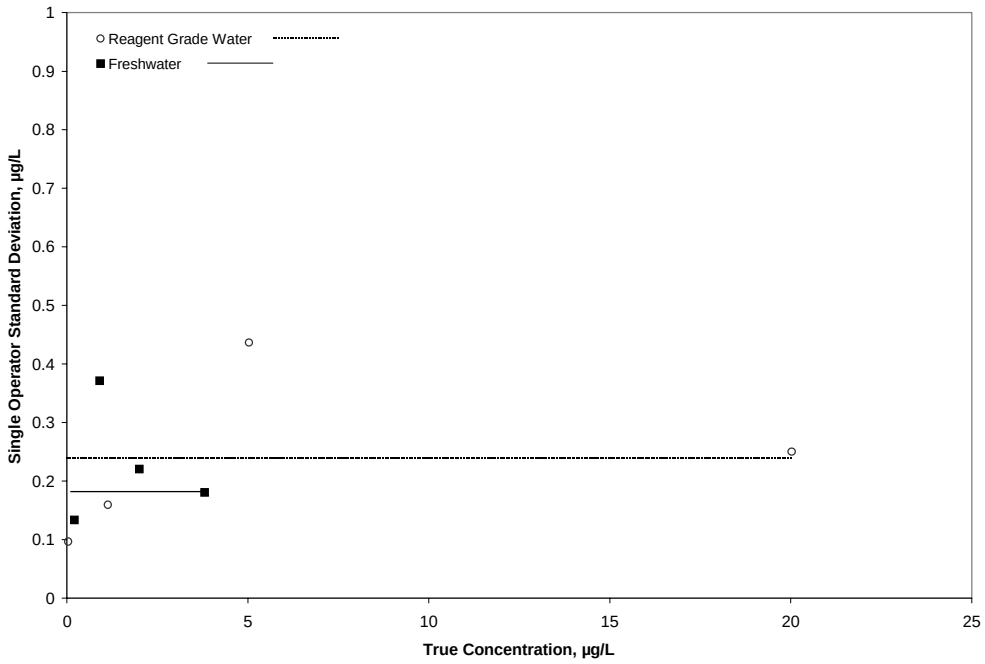

Figure 4-31
Plot of single operator standard deviation versus true concentration for selenium in reagent grade water and freshwater

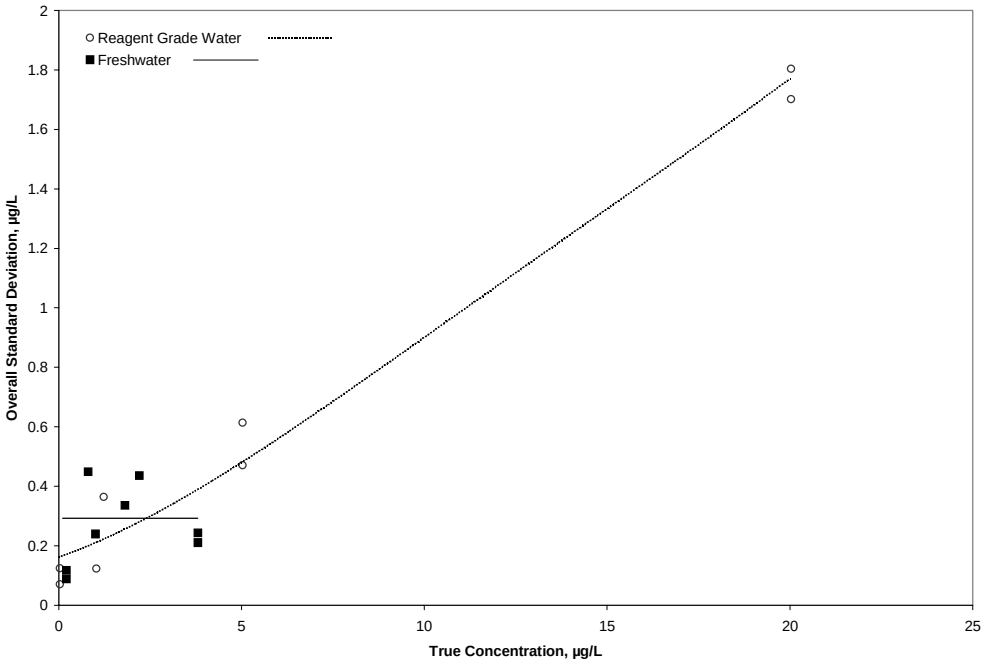

Figure 4-32
Plot of overall standard deviation versus true concentration for selenium in reagent grade water and freshwater

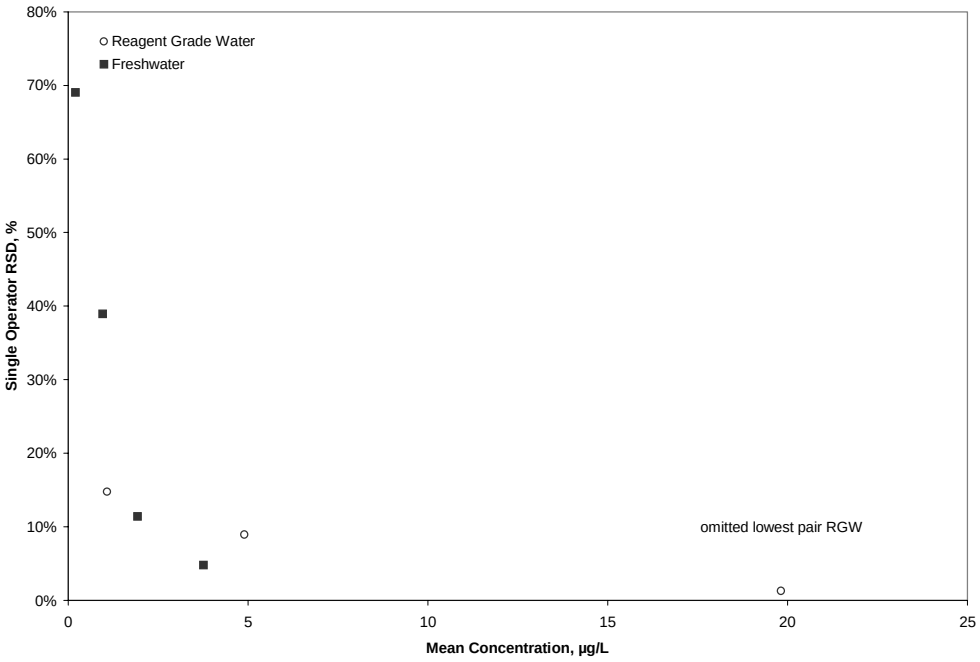

Figure 4-33
Plot of single operator relative standard deviation versus mean recovery for selenium in reagent grade water and freshwater (unspiked reagent grade water data omitted)

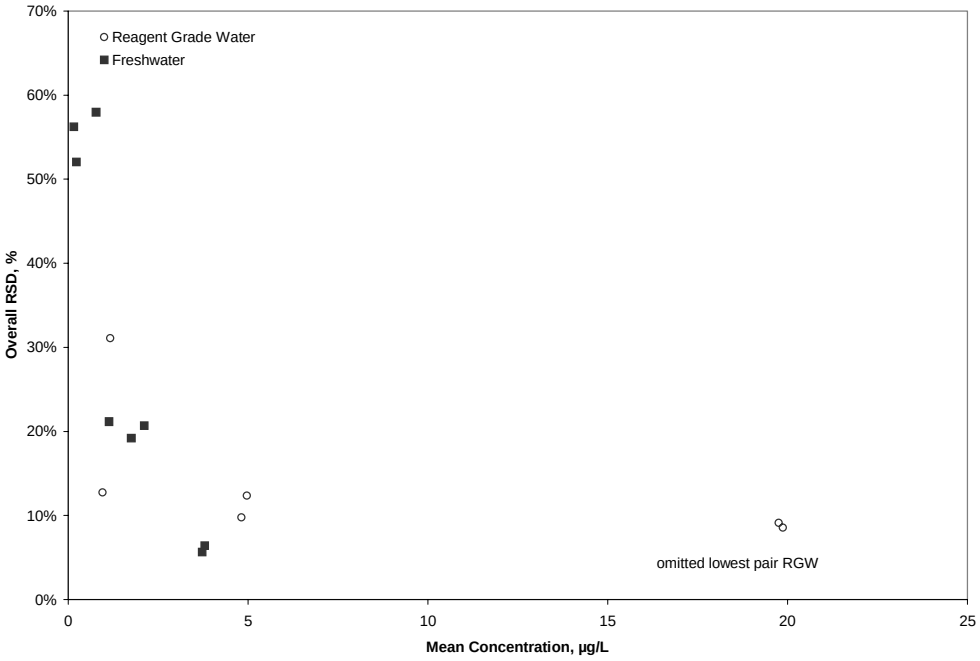

Figure 4-34
Plot of overall relative standard deviation versus mean recovery for selenium in reagent grade water and freshwater (unspiked reagent grade water data omitted)

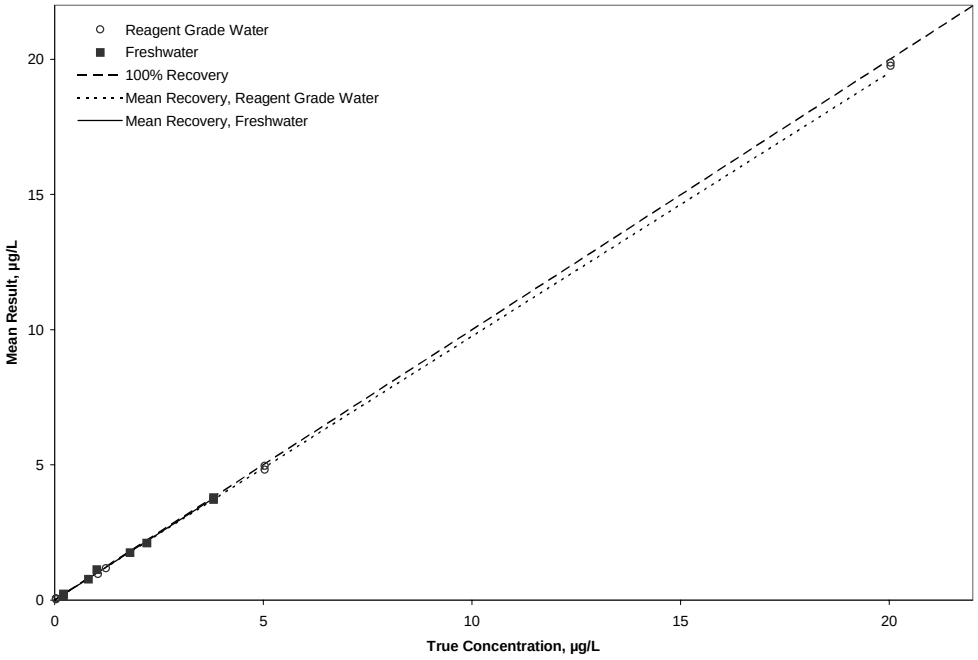

Figure 4-35
Plot of mean result versus true concentration for selenium in reagent grade water and freshwater (all concentration levels)

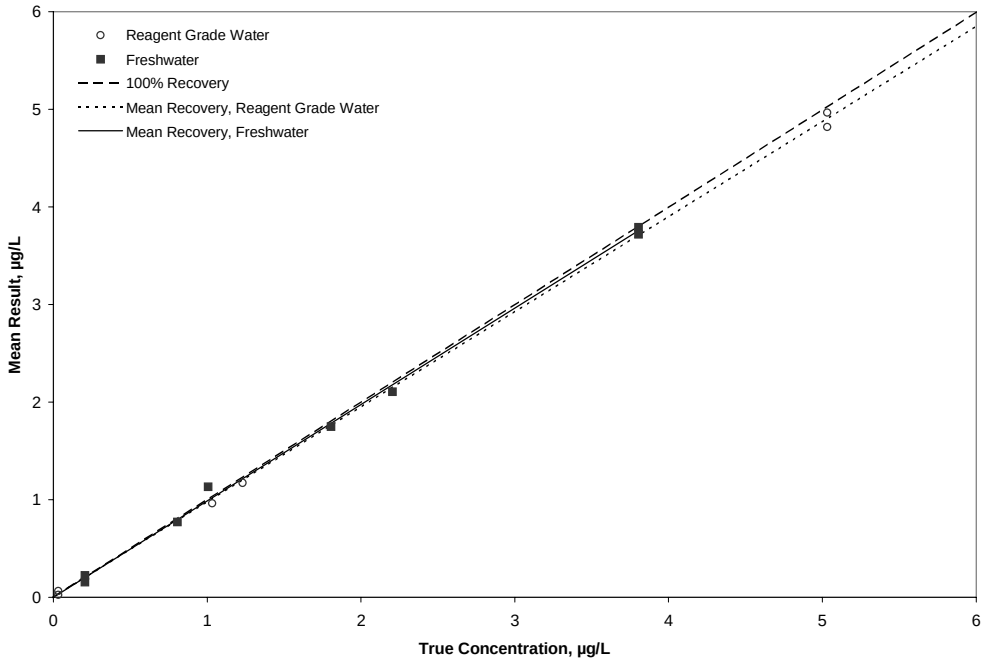

Figure 4-36
Plot of mean result versus true concentration for selenium in reagent grade water and freshwater (highest concentration level omitted)

4.2.1.7 Silver

The single operator precision (single operator standard deviation versus true concentration) plot for silver in reagent grade water and freshwater is shown in Figure 4-37. The data show a trend of increasing standard deviation with increasing concentration of silver. The Rocke-Lorenzato fits to the data are included on the plots.

The overall precision plot for silver in reagent grade water and freshwater is shown in Figure 4-38. The reagent grade water data show a general trend of increasing overall standard deviation with increasing silver concentration. The Rocke-Lorenzato fit of the data is included on the plot. Over the concentration range studied, the freshwater data are best fit with a constant model, shown on the plot as a horizontal line.

Figure 4-39 plots the single operator percent RSD versus mean concentration for silver in reagent grade water and freshwater. The data are scattered, exhibiting no obvious trend.

The overall percent RSD data for silver in reagent grade water and freshwater are plotted in Figure 4-40. The reagent grade water data are scattered with overall RSDs between approximately 90% and 125% up to a mean recovery of 0.4 μg/L. The overall percent RSDs in freshwater exhibit a trend of generally decreasing percent RSDs, leveling off at about 40% overall RSD near a mean recovery of 0.13 μg/L silver.

The silver spikes into the sample matrices were not stable. Recoveries were poor across most of the study concentration range. Recoveries in both reagent grade water and freshwater are plotted in Figures 4-41 (all concentrations) and 4-42 (highest concentration in reagent grade water omitted). Mean recoveries of the 10 μg/L spikes into reagent grade water were approximately 93%. At all other spiked concentrations, mean recoveries were between 33% and 37% in reagent grade water and 18% to 59% in freshwater.

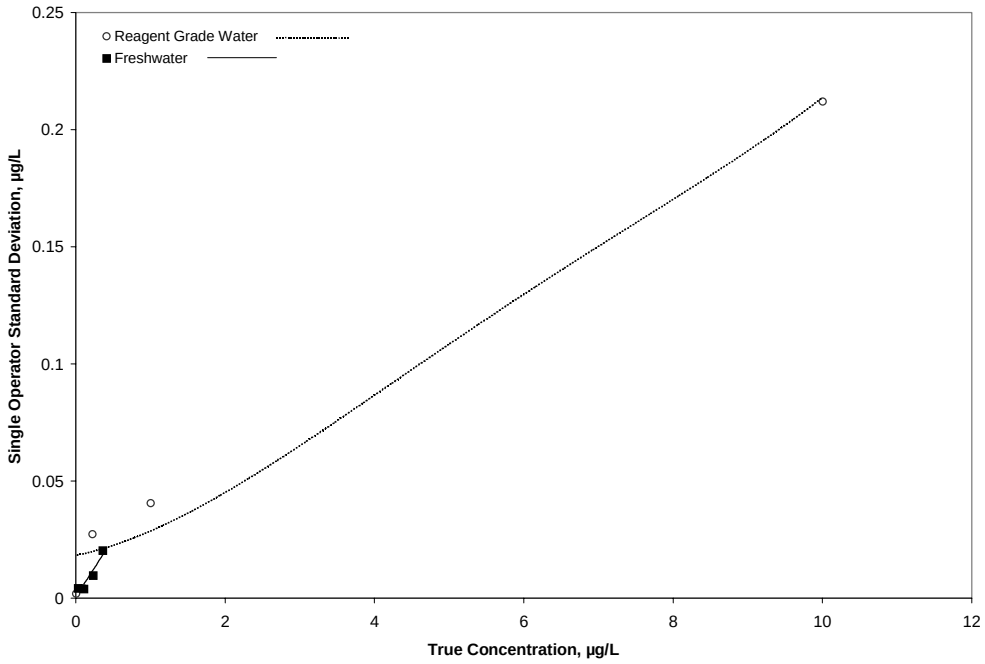

Figure 4-37
Plot of single operator standard deviation versus true concentration for silver in reagent grade water and freshwater

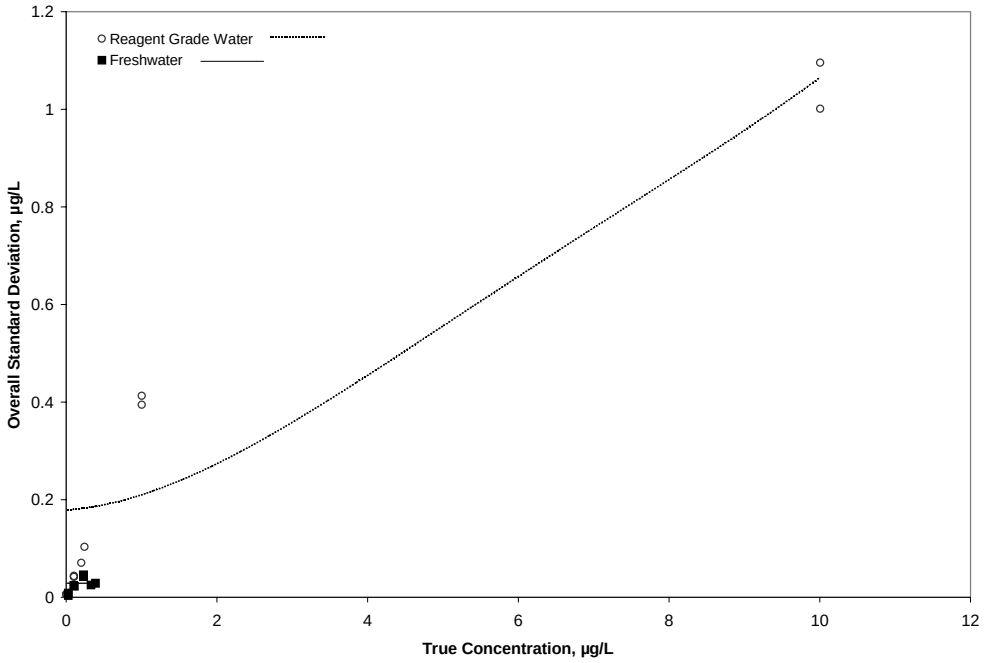

Figure 4-38
Plot of overall standard deviation versus true concentration for silver in reagent grade water and freshwater

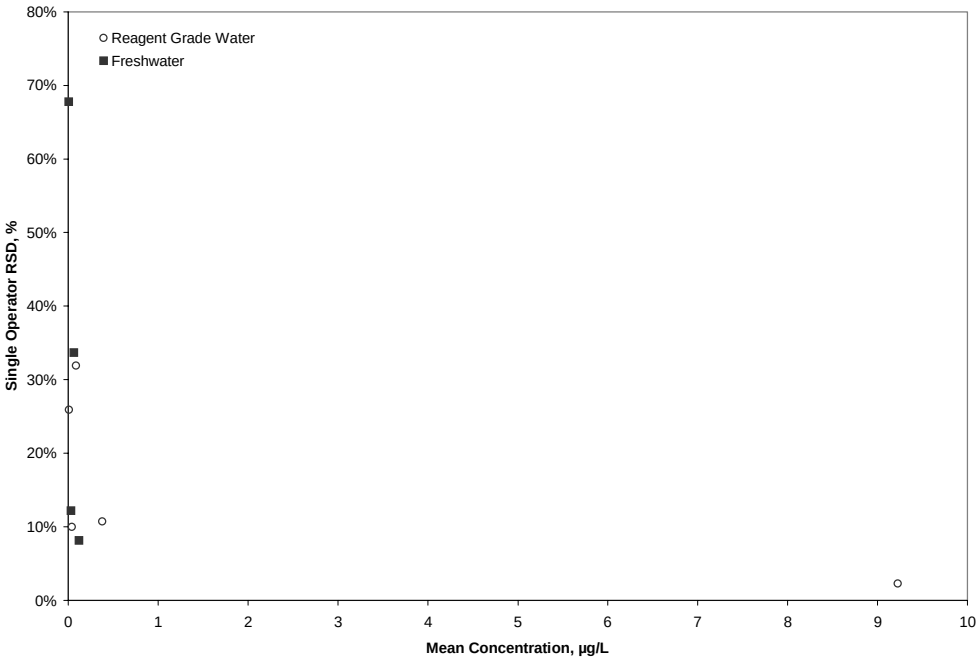

Figure 4-39
Plot of single operator relative standard deviation versus mean recovery for silver in reagent grade water and freshwater (all data)

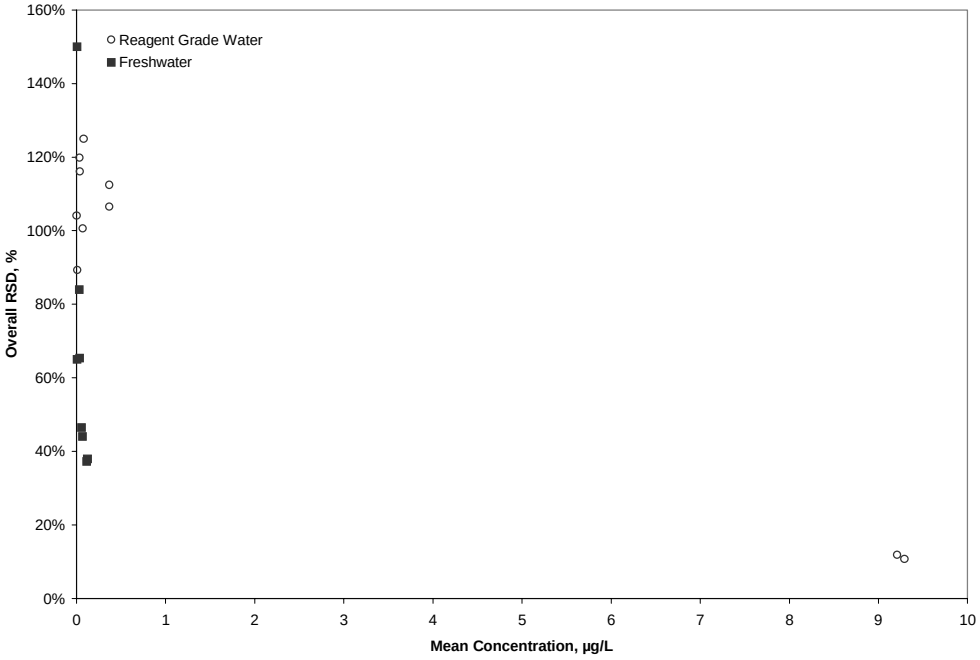

Figure 4-40
Plot of overall relative standard deviation versus mean recovery for silver in reagent grade water and freshwater (all data)

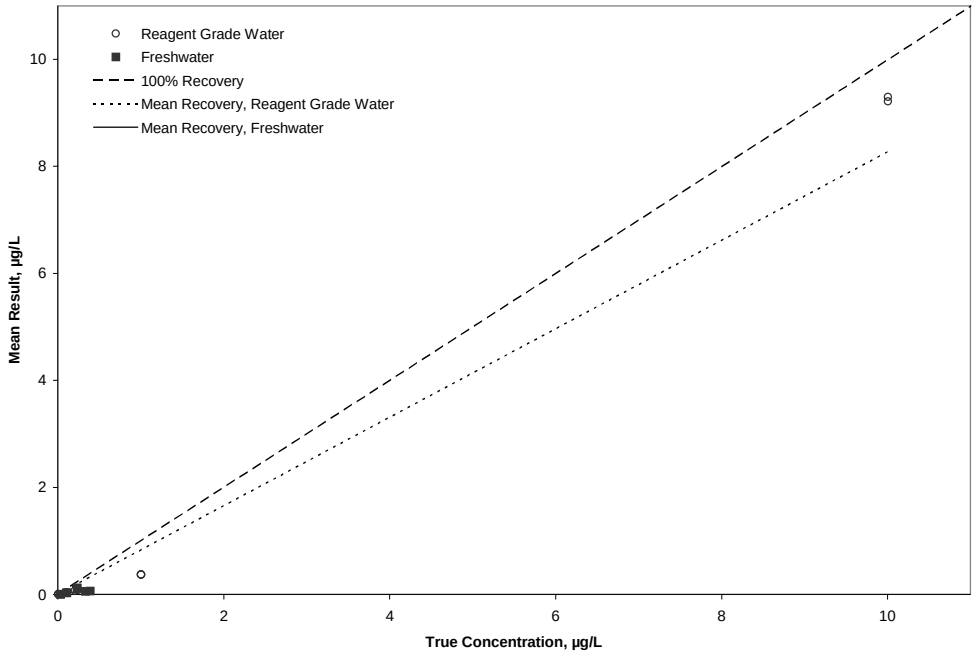

Figure 4-41
Plot of mean result versus true concentration for silver in reagent grade water and freshwater (all concentration levels)

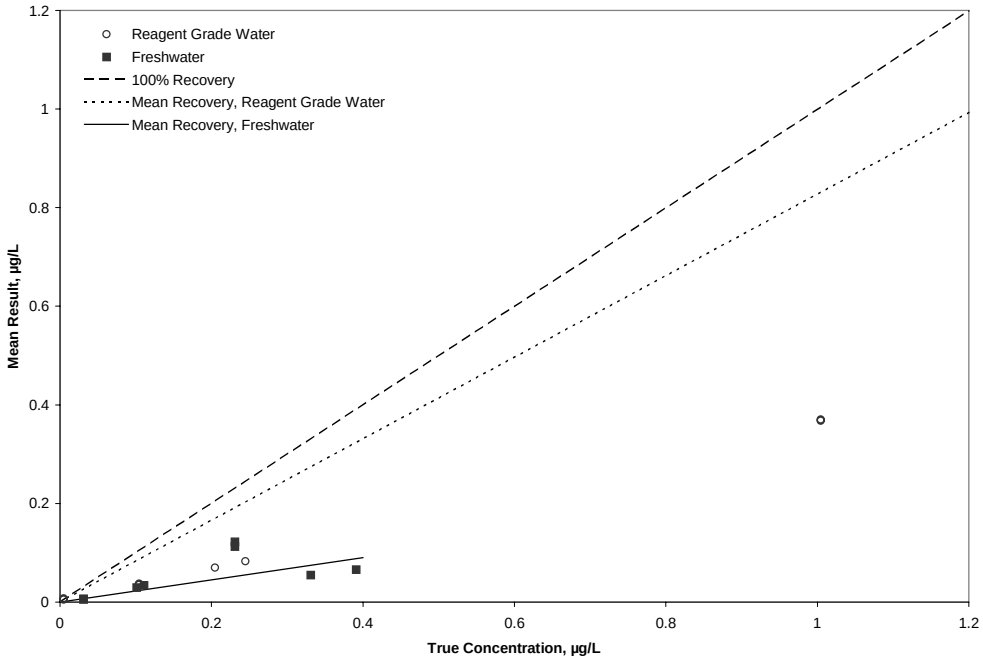

Figure 4-42
Plot of mean result versus true concentration for silver in reagent grade water and freshwater (highest concentration level omitted)

4.2.1.8 Thallium

Figure 4-43 shows the single operator precision plot (single operator standard deviation versus true concentration) for thallium in reagent grade water and freshwater. A Rocke-Lorenzato fit to each set of data is shown on the plot. The data show a general trend of increasing standard deviation with increasing concentration.

The overall precision plot for thallium in reagent grade water is shown in Figure 4-44. In both reagent grade water and freshwater, there is a trend of increasing standard deviation with increasing concentration. The data from both matrices correlate well, with the freshwater data overlaying the reagent grade water data.

The single operator RSD data for thallium in reagent grade water and freshwater are plotted in Figures 4-45. The unspiked reagent grade water data (RSD = 300%) have been omitted from the plot. A trend of generally decreasing single operator RSD is evident in both matrices, leveling off to less than 5% RSD in the study concentration range.

The thallium overall RSD data are plotted in Figures 4-46. An overall RSD could not be calculated for unspiked reagent grade water because the calculated true concentration of thallium in the unspiked sample was zero. The unspiked freshwater data (RSD = 100%, 125%) have been omitted from the plot to improve readability. The thallium data exhibit a general trend of decreasing percent overall RSD with increasing mean concentration of thallium, leveling off to between 5% and 10% overall RSD over the concentration range.

Figure 4-47 plots the recovery data for thallium at all study concentrations in reagent grade water and freshwater. In Figure 4-48, the data for the highest spike in reagent grade water have been omitted. Thallium recoveries were between 90% and 100% at all spike levels in both matrices.

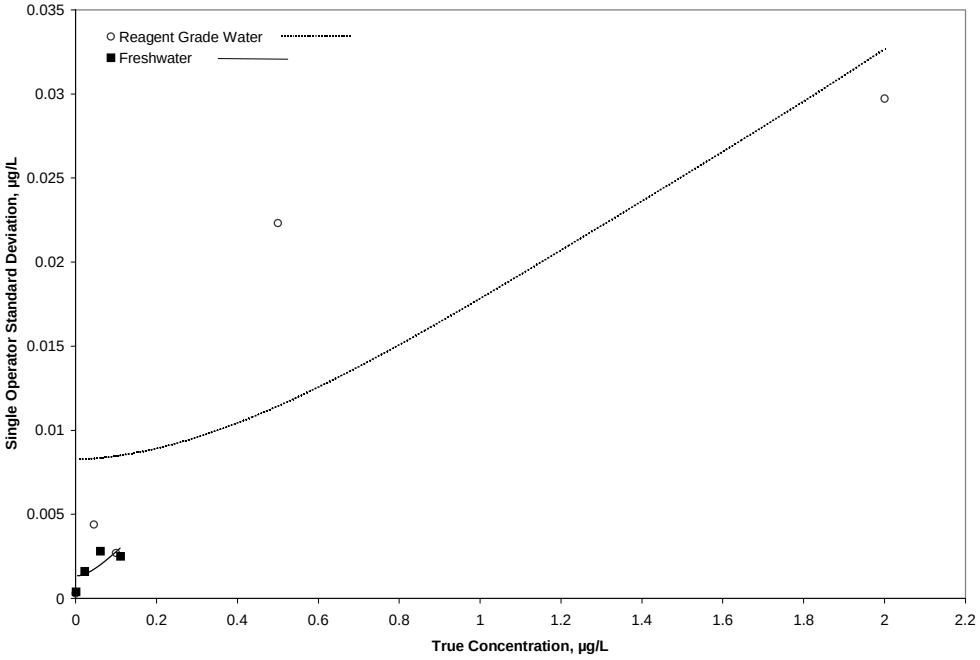

Figure 4-43
Plot of single operator standard deviation versus true concentration for thallium in reagent grade water and freshwater

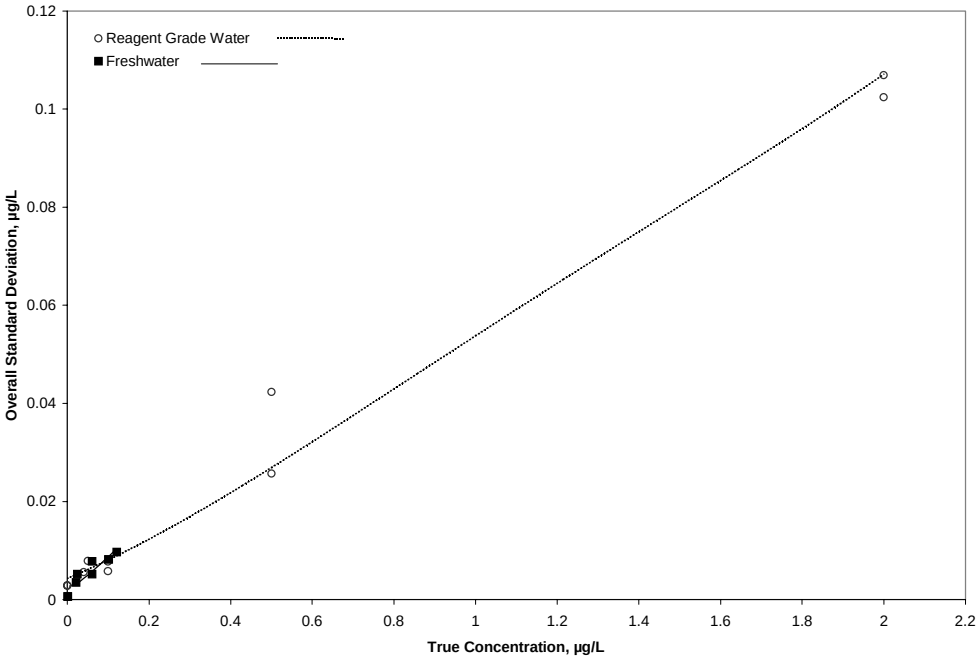

Figure 4-44
Plot of overall standard deviation versus true concentration for thallium in reagent grade water and freshwater

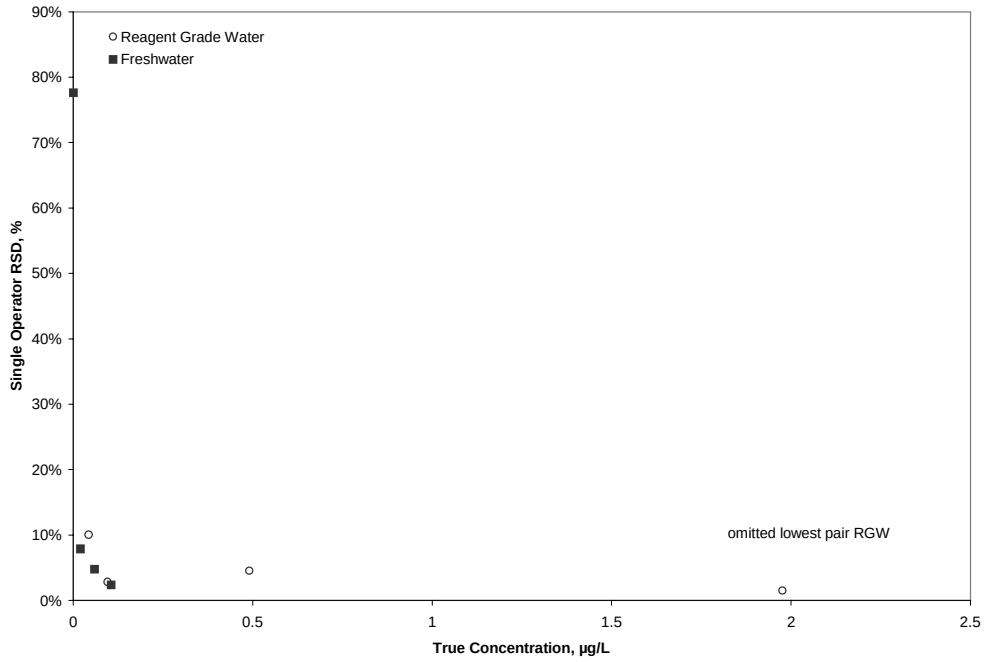

Figure 4-45
Plot of single operator relative standard deviation versus mean recovery for thallium in reagent grade water and freshwater (unspiked reagent grade water data omitted)

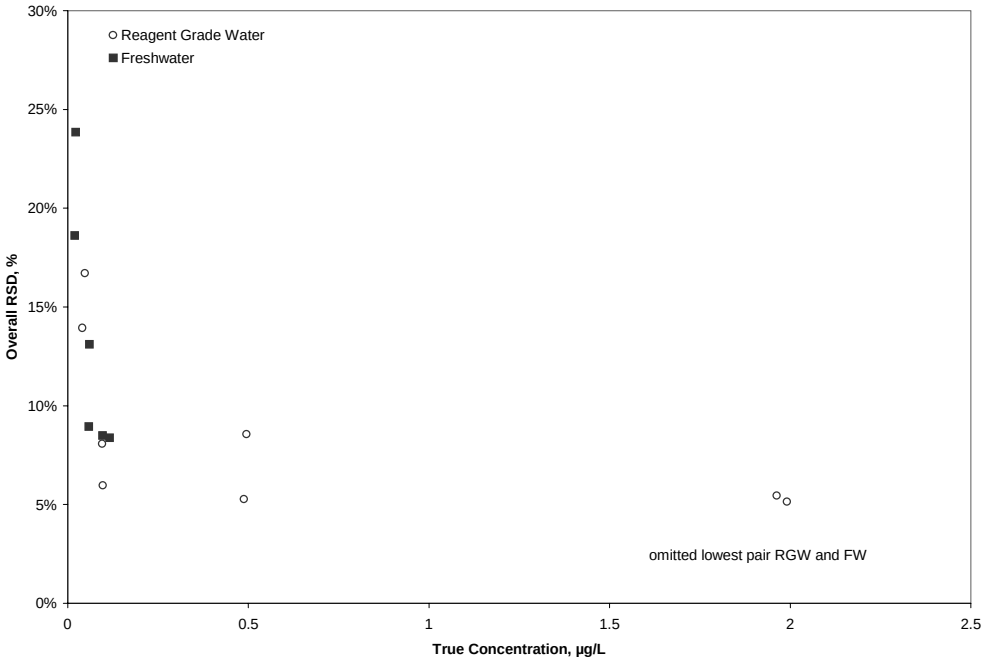

Figure 4-46
Plot of overall relative standard deviation versus mean recovery for thallium in reagent grade water and freshwater (unspiked reagent grade water and freshwater data omitted)

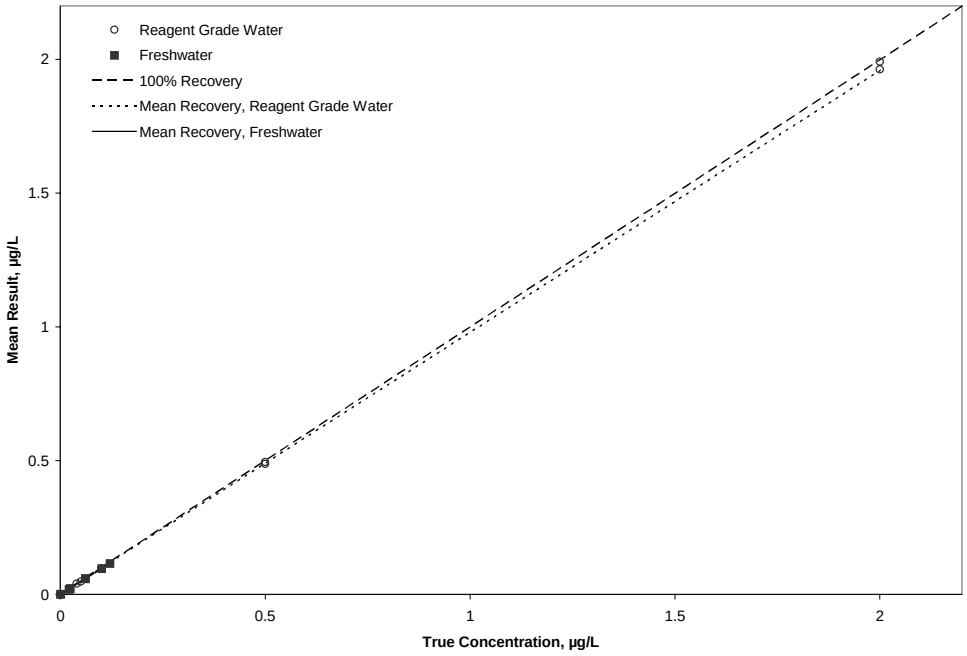

Figure 4-47
Plot of mean result versus true concentration for thallium in reagent grade water and freshwater (all concentration levels)

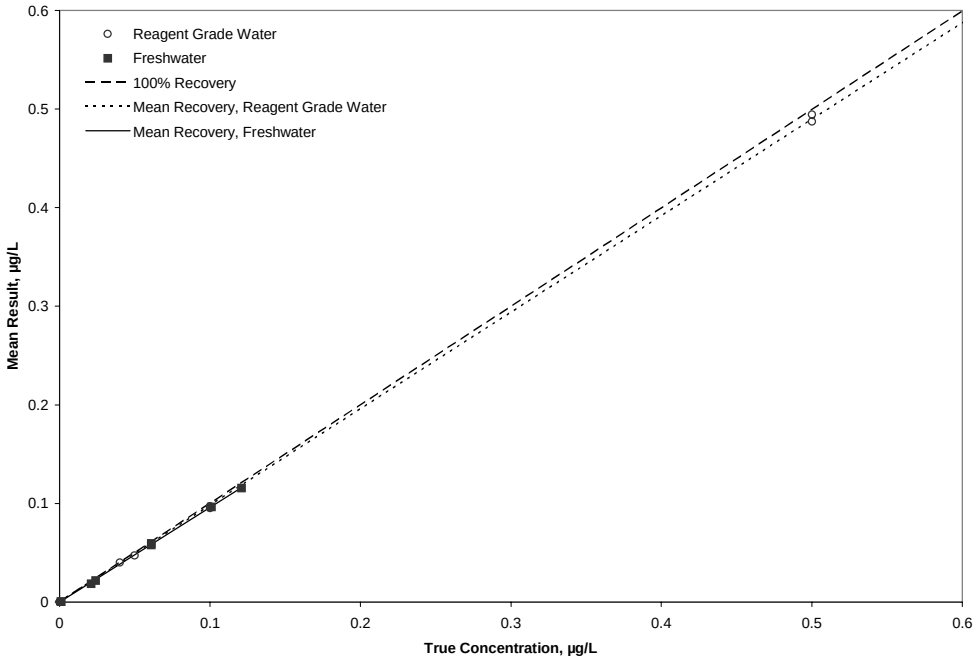

Figure 4-48
Plot of mean result versus true concentration for thallium in reagent grade water and freshwater (highest concentration level omitted)

4.2.1.9 Zinc

The plots of single operator standard deviation versus true concentration of zinc in reagent grade water and freshwater is shown in Figure 4-49. The Rocke-Lorenzato fit to the reagent grade water data is plotted, illustrating increasing single operator standard deviation with increasing concentration of zinc in reagent grade water. The freshwater data are clustered near the flat end of the reagent grade water Rocke-Lorenzato “hockey stick” and are best fit by a constant model, indicated on the plot as a horizontal line.

The overall precision data for zinc in reagent grade water and freshwater are plotted in Figure 4-50. The reagent grade water exhibit increasing overall standard with increasing concentration. Over the concentration range studied, the freshwater data exhibit essentially constant overall standard deviation.

Figure 4-51 plots the single operator percent RSD data versus mean concentration for zinc in reagent grade water and freshwater. The reagent grade water data exhibit a trend of decreasing single operator percent RSD with increasing concentration, leveling off to about 10% RSD. The freshwater single operator RSD data are scattered with no apparent trend.

The overall percent RSD data for zinc in reagent grade water and freshwater are plotted in Figure 4-52. Overall percent RSDs in reagent grade water generally decrease with increasing concentration, leveling off to between 15% and 20% at the higher study concentrations. The freshwater data are more scattered, generally tending toward decreasing overall percent RSD with increasing concentration with the exception of a particularly high RSD at a mean recovery of approximately 1.5 μg/L.

The recovery plot for all study concentrations in both reagent grade water and freshwater is shown in Figure 4-53. An expanded version which omits the reagent grade water data at 50 μg/L is shown in Figure 4-54. Mean recoveries in both matrices across most of the study range were in the range 85% to 115%. However, in reagent grade water, the mean recoveries for the unspiked pair of samples were 195% and 330%. In freshwater, the mean recovery for one of the sample pairs at a mean concentration of approximately 1.5 μg/L was 162%.

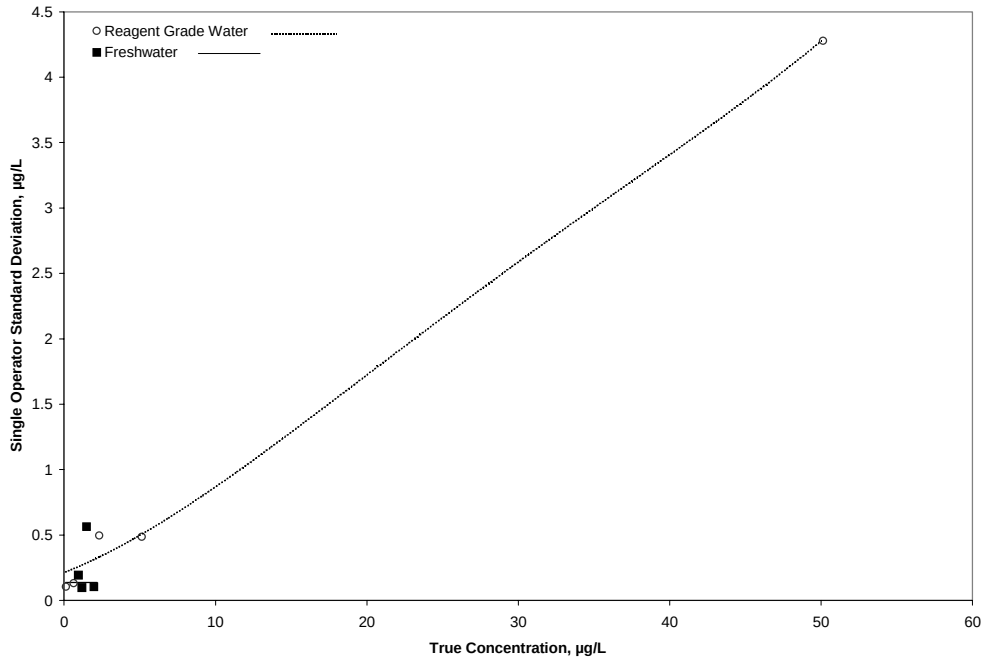

Figure 4-49
Plot of single operator standard deviation versus true concentration for zinc in reagent grade water and freshwater

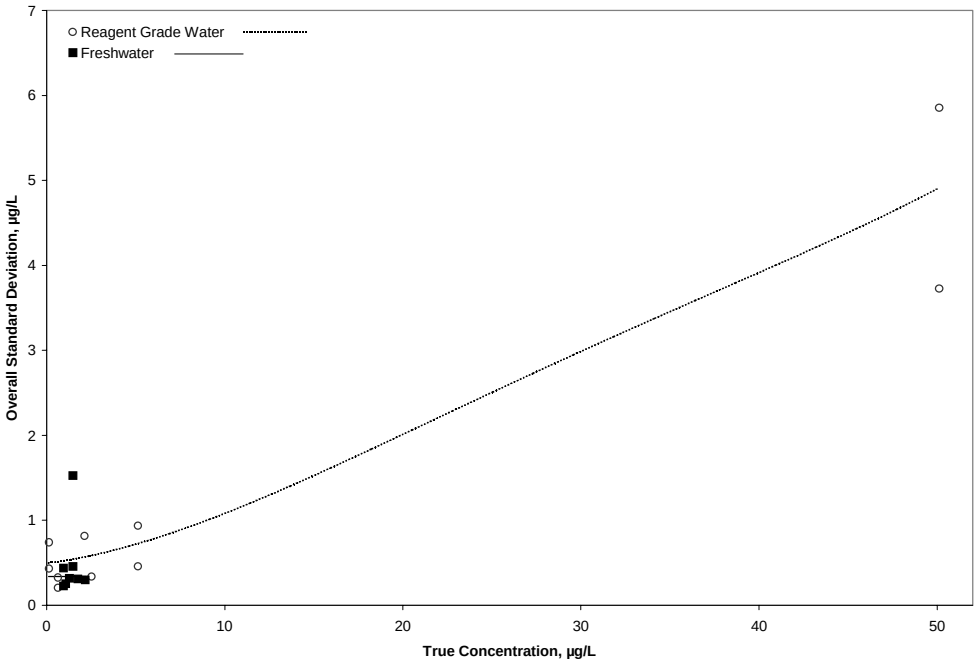

Figure 4-50
Plot of overall standard deviation versus true concentration for zinc in reagent grade water and freshwater

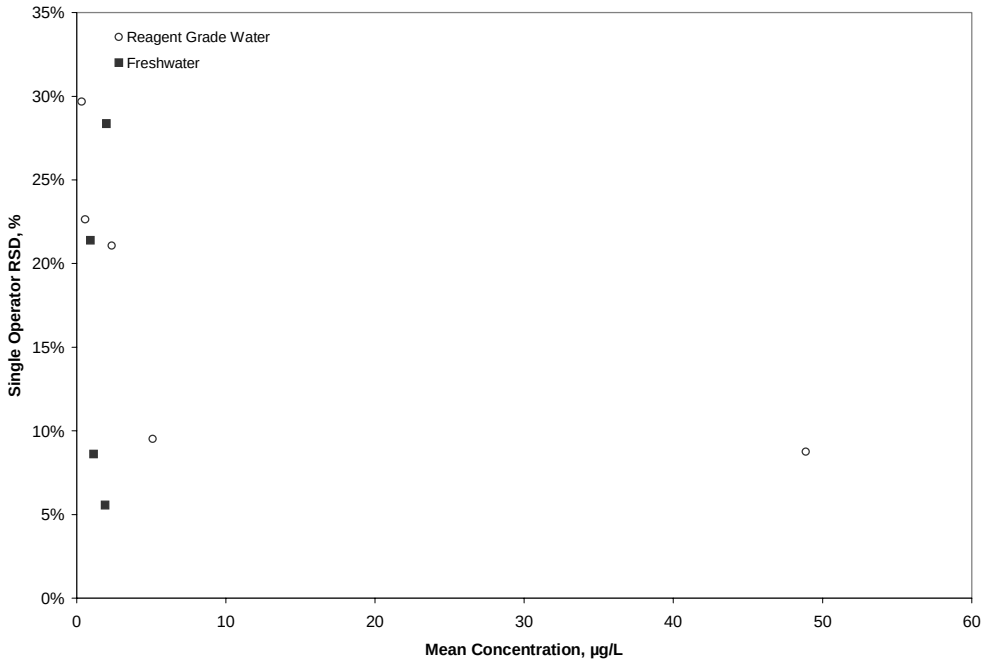

Figure 4-51
Plot of single operator relative standard deviation versus mean recovery for zinc in reagent grade water and freshwater (all data)

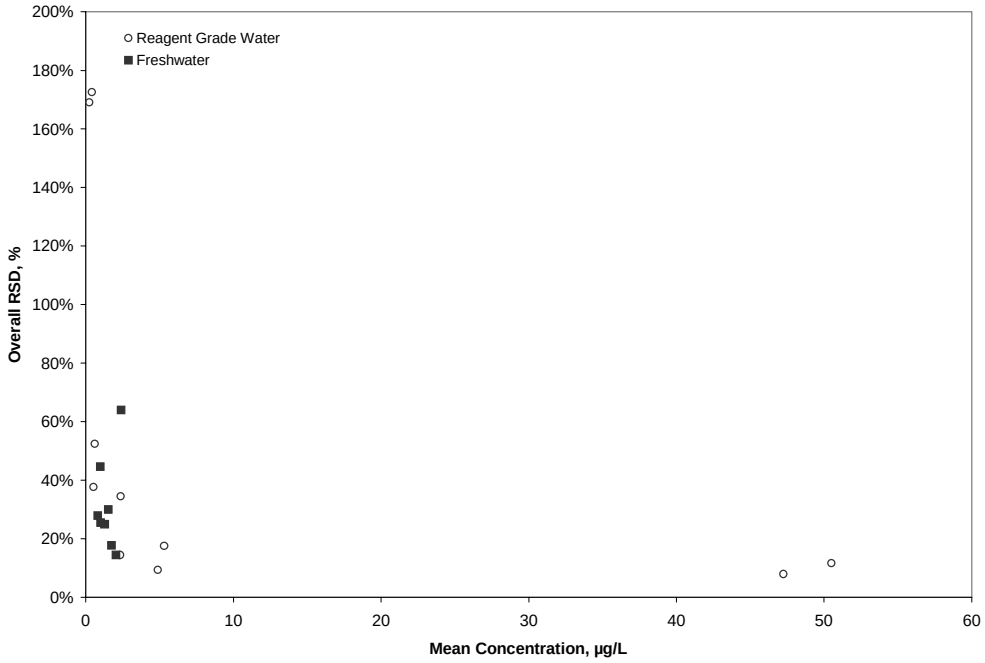

Figure 4-52
Plot of overall relative standard deviation versus mean recovery for zinc in reagent grade water and freshwater (all data)

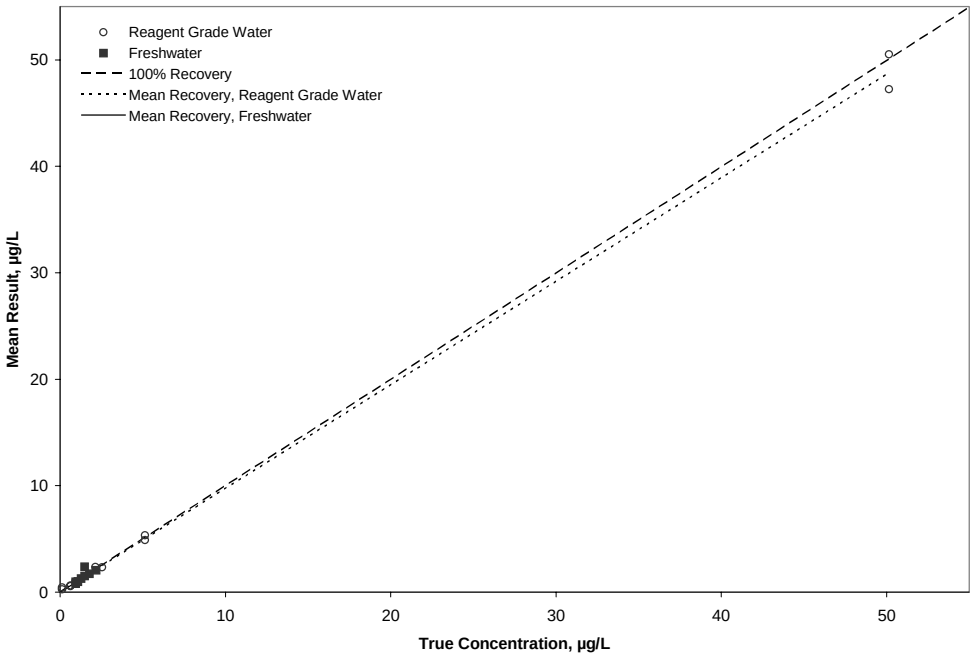

Figure 4-53
Plot of mean result versus true concentration for zinc in reagent grade water and freshwater (all concentration levels)

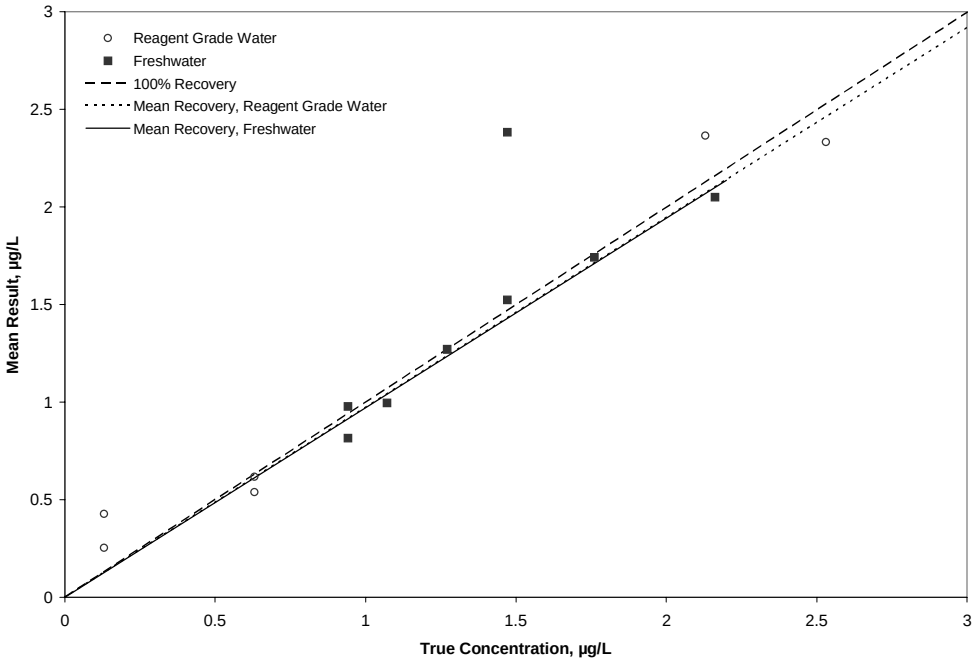

Figure 4-54
Plot of mean result versus true concentration for zinc in reagent grade water and freshwater (highest concentration level omitted)

4.2.2 Effluent Matrices

Recoveries in filtered effluent (FEF) and unfiltered effluent (UEF) are summarized in Tables 4-21 through 4-29. The EPA provided a single pair of duplicate samples in each matrix. The true concentration was calculated from the referee laboratory's background analysis plus any added spikes. It was not possible to perform laboratory ranking with only a single concentration level per matrix. The ASTM D 2777 outlier procedure was used to remove individual outliers.

With the exception of silver, mean recoveries reported by the study participants ranged from 94.1% for antimony in filtered effluent to 141.2% for selenium in filtered effluent. Mean recoveries for silver were 79.9% in filtered effluent and 46.7% in unfiltered effluent. Single operator RSDs ranged from 1.9% (lead in unfiltered effluent) to 16.2% (cadmium in filtered effluent). Overall RSDs ranged from 3.7% (nickel in filtered effluent) to 26.2% (cadmium in filtered effluent).

Table 4-21
Statistical Summary for Antimony in Effluent Matrices

	FEF + 0.03 µg/L Sb	**UEF + 9.6 µg/L Sb**
Number of retained values	8	7
True concentration, µg/L	0.986	10.011
Mean recovery, µg/L	1.0370	9.4326
Percent recovery	105.2%	94.1%
Overall standard deviation, S_t, µg/L	0.0772	0.5118
Overall relative standard deviation, %	7.4%	5.4%
Number of retained pairs	8	7
Mean recovery (pairs), µg/L	1.0370	9.4236
Single operator standard deviation, S_o, µg/L	0.0830	0.2151
Single operator relative standard deviation, %	8.0%	2.3%

FEF = Filtered Effluent
UEF = Unfiltered Effluent

Table 4-22
Statistical Summary for Cadmium in Effluent Matrices

	FEF + 0.2 µg/L Cd	**UEF + 0.38 µg/L Cd**
Number of retained values	8	8
True concentration, µg/L	0.308	0.505
Mean recovery, µg/L	0.2945	0.4819
Percent recovery	95.6%	95.4%
Overall standard deviation, S_t, µg/L	0.0772	0.0994
Overall relative standard deviation, %	26.2%	20.6%
Number of retained pairs	8	8
Mean recovery (pairs), µg/L	0.2945	0.4819
Single operator standard deviation, S_o, µg/L	0.0476	0.0167
Single operator relative standard deviation, %	16.2%	3.5%

FEF = Filtered Effluent
UEF = Unfiltered Effluent

Table 4-23
Statistical Summary for Copper in Effluent Matrices

	FEF + 0 µg/L Cu	**UEF + 0 µg/L Cu**
Number of retained values	8	8
True concentration, µg/L	13.8	15.1
Mean recovery, µg/L	14.2191	16.3690
Percent recovery	103.0%	108.4%
Overall standard deviation, S_t, µg/L	0.9401	1.7201
Overall relative standard deviation, %	6.6%	10.5%
Number of retained pairs	7	8
Mean recovery (pairs), µg/L	14.0836	16.3690
Single operator standard deviation, S_o, µg/L	0.5442	1.1316
Single operator relative standard deviation, %	3.9%	6.9%

FEF = Filtered Effluent
UEF = Unfiltered Effluent

Table 4-24
Statistical Summary for Lead in Effluent Matrices

	FEF + 0.05 µg/L Pb	**UEF + 0 µg/L Pb**
Number of retained values	8	7
True concentration, µg/L	0.531	1.11
Mean recovery	0.6043	1.0709
Percent recovery	113.8%	96.5%
Overall standard deviation, St	0.0709	0.1166
Overall relative standard deviation, %	11.7%	10.9%
Number of retained pairs	8	7
Mean recovery (pairs)	0.6043	1.0709
Single operator standard deviation, So	0.0572	0.0202
Single operator relative standard deviation, %	9.5%	1.9%

FEF = Filtered Effluent
UEF = Unfiltered Effluent

Table 4-25
Statistical Summary for Nickel in Effluent Matrices

	FEF + 4.4 µg/L Ni	**UEF + 5.3 µg/L Ni**
Number of retained values	7	8
True concentration, µg/L	7.0	8.01
Mean recovery, µg/L	6.8926	7.6556
Percent recovery	98.5%	95.6%
Overall standard deviation, S_t, µg/L	0.2559	0.9152
Overall relative standard deviation, %	3.7%	12.0%
Number of retained pairs	7	8
Mean recovery (pairs), µg/L	6.8926	7.6556
Single operator standard deviation, S_o, µg/L	0.2166	0.3487
Single operator relative standard deviation, %	3.1%	4.6%

FEF = Filtered Effluent
UEF = Unfiltered Effluent

Table 4-26
Statistical Summary for Selenium in Effluent Matrices

	FEF + 4.7 µg/L Se	**UEF + 5.7 µg/L Se**
Number of retained values	7	7
True concentration, µg/L	5	5.97
Mean recovery, µg/L	7.0619	8.1699
Percent recovery	141.2%	136.8%
Overall standard deviation, S_t, µg/L	1.2312	1.4987
Overall relative standard deviation, %	17.4%	18.3%
Number of retained pairs	7	7
Mean recovery (pairs), µg/L	7.0619	8.1699
Single operator standard deviation, S_o, µg/L	0.2270	0.3262
Single operator relative standard deviation, %	3.2%	4.0%

FEF = Filtered Effluent
UEF = Unfiltered Effluent

Table 4-27
Statistical Summary for Silver in Effluent Matrices

	FEF + 0 µg/L Ag	**UEF + 0 µg/L Ag**
Number of retained values	8	8
True concentration, µg/L	0.478	2.88
Mean recovery, µg/L	0.3819	1.3441
Percent recovery	79.9%	46.7%
Overall standard deviation, S_t, µg/L	0.0748	0.1505
Overall relative standard deviation, %	19.6%	11.2%
Number of retained pairs	8	8
Mean recovery (pairs), µg/L	0.3819	1.3441
Single operator standard deviation, S_o, µg/L	0.0428	0.0632
Single operator relative standard deviation, %	11.2%	4.7%

FEF = Filtered Effluent
UEF = Unfiltered Effluent

Table 4-28
Statistical Summary for Thallium in Effluent Matrices

	FEF + 0.2 µg/L Tl	**UEF + 0.9 µg/L Tl**
Number of retained values	8	8
True concentration, µg/L	0.2	0.9
Mean recovery, µg/L	0.1968	0.8895
Percent recovery	98.4%	98.8%
Overall standard deviation, S_t, µg/L	0.0147	0.0831
Overall relative standard deviation, %	7.4%	9.3%
Number of retained pairs	7	8
Mean recovery (pairs), µg/L	0.1985	0.8895
Single operator standard deviation, S_o, µg/L	0.0084	0.0326
Single operator relative standard deviation, %	4.2%	3.7%

FEF = Filtered Effluent
UEF = Unfiltered Effluent

Table 4-29
Statistical Summary for Zinc in Effluent Matrices

	FEF + 0 µg/L Zn	**UEF + 0 µg/L Zn**
Number of retained values	7	8
True concentration, µg/L	43.25	48.47
Mean recovery, µg/L	43.7457	50.8318
Percent recovery	101.1%	104.9%
Overall standard deviation, S_t, µg/L	1.9487	4.4021
Overall relative standard deviation, %	4.5%	8.7%
Number of retained pairs	7	8
Mean recovery (pairs), µg/L	44.0055	50.8318
Single operator standard deviation, S_o, µg/L	2.1179	3.6302
Single operator relative standard deviation, %	4.8%	7.1%

FEF = Filtered Effluent
UEF = Unfiltered Effluent

4.2.3 Standard Reference Material

Each study participant analyzed a trace elements in water standard produced by the National Institute of Standards & Technology (NIST). Standard Reference Material (SRM) 1643d is intended to simulate the elemental composition of fresh water. It consists of filtered and acidified water in a polyethylene bottle. The NIST certified values for 26 trace elements are listed in Table 4-30.

Table 4-30
Certified Concentrations for Trace Elements in NIST SRM 1643d

Element	Concentration, µg/L	Element	Concentration, µg/L
Aluminum	127.6 +/- 3.5	Lead	18.15 +/- 0.64
Antimony	54.1 +/- 1.1	Lithium	16.50 +/- 0.55
Arsenic	56.02 +/- 0.73	Manganese	37.66 +/- 0.83
Barium	506.5 +/- 8.9	Molybdenum	112.9 +/- 1.7
Beryllium	12.53 +/- 0.28	Nickel	58.1 +/- 2.7
Boron	144.8 +/- 5.2	Selenium	11.43 +/- 0.17
Cadmium	6.47 +/- 0.37	Silver	1.270 +/- 0.057
Chromium	18.53 +/- 0.20	Strontium	294.8 +/- 3.4
Cobalt	25.00 +/- 0.59	Thallium	7.28 +/- 0.25
Copper	20.5 +/- 3.8	Vanadium	35.1 +/- 1.4
Iron	91.2 +/- 3.9	Zinc	72.48 +/- 0.65
Calcium (mg/L)	31.04+/- 0.50	Potassium (mg/L)	2.356 +/- 0.035
Magnesium (mg/L)	7.989 +/- 0.035	Sodium (mg/L)	22.07 +/- 0.64

All of the SRM values reported by the study participants are contained in Appendix B. In their data analysis, EPA removed a large number of the reported values without explanation. Table 4-31 summarizes all of the reported SRM data. Table 4-32 summarizes only the data points retained by EPA in their data analysis. There are only slight differences in the statistics calculated from the two data sets. Using all of the reported data, recoveries ranged from 93.3% (thallium) to 102% (copper). RSDs ranged from 3% (cadmium) to 14.1% (selenium). Using only those data points retained by EPA, recoveries ranged from 91.8% (lead) to 101.2% (copper). RSDs calculated from only the EPA-retained data ranged from 3.1% (cadmium) to 15.2% (selenium). None of the SRM certified values lie within the concentration ranges of the freshwater study samples, ranging from 3 to 50 times the highest spiked concentration.

Table 4-31
Summary of All Reported SRM Data Compared to Study Samples

	SRM Results					Study Samples	
Analyte	**Certified Conc. µg/L**	**Reported Mean µg/L**	**Standard Deviation µg/L**	**RSD, %**	**Recovery, %**	**RGW Range µg/L**	**FW, Range µg/L**
Sb	54.1	54.1	2.2	4.1	99.9	0.0-20	0.133-1.01
Cd	6.47	6.14	0.2	3.0	94.9	0.004-10.0	0.0-0.25
Cu	20.5	20.9	1.6	7.6	102	0.105-20.1	1.26-5.26
Pb	18.15	17.3	2.3	13.2	95.3	0.019-5.0	0.043-0.39
Ni	58.1	55.6	2.7	4.9	95.6	0.0-10.0	0.67-6.37
Se	11.43	11.1	1.6	14.1	96.8	0.029-100	0.21-3.81
Ag	1.27	1.26	0.07	5.9	99.3	0.006-10.0	0.006-0.37
Tl	7.28	6.79	0.8	11.1	93.3	0.0-2.0	0.001-0.12
Zn	72.48	69.5	6.3	9.1	95.9	0.13-50.1	0.941-2.16

RGW = Reagent Grade Water; FW = Freshwater

Table 4-32
Summary of EPA-Retained SRM Data Compared to Study Samples

	SRM Results					Study Samples	
Analyte	**Certified Conc. µg/L**	**Reported Mean µg/L**	**Standard Deviation µg/L**	**RSD, %**	**Recovery, %**	**RGW Range µg/L**	**FW, Range µg/L**
Sb	54.1	53.1	1.9	3.6	98.1	0.0-20	0.133-1.01
Cd	6.47	6.09	0.2	3.1	94.1	0.004-10.0	0.0-0.25
Cu	20.5	20.8	1.7	8.2	101.2	0.105-20.1	1.26-5.26
Pb	18.15	16.7	2.1	12.7	91.8	0.019-5.0	0.043-0.39
Ni	58.1	55.4	1.9	3.5	95.3	0.0-10.0	0.67-6.37
Se	11.43	10.9	1.7	15.2	95.2	0.029-100	0.21-3.81
Ag	1.27	1.26	0.07	5.9	99.3	0.006-10.0	0.006-0.37
Tl	7.28	6.80	0.9	13.2	93.4	0.0-2.0	0.001-0.12
Zn	72.48	68.6	5.6	8.2	94.7	0.13-50.1	0.941-2.16

RGW = Reagent Grade Water; FW = Freshwater

4.3 References

1. American Society for Testing and Materials, "Standard Practice for the Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water," D 2777-98.

5
ESTIMATES OF DETECTION AND QUANTIFICATION

In order to evaluate and negotiate permit levels and for other NPDES or other regulatory applications, owners/operators of permitted facilities require detection and quantification level definitions and values that specifically incorporate the following:

- An interlaboratory standard deviation, to account for the variability associated with the analysis of split samples by the permittee and the regulator, and the use of different laboratories over the life of the permit.
- Estimates of the standard deviation over a range of concentrations including zero, if possible, to account for changes in standard deviation with concentration.
- Terms and definitions readily discernible by users as different from existing definitions based on single-operator or pooled single-operator standard deviation.
- Statistical treatment of data appropriate for future monitoring decisions.

In the 1994 draft EPA guidance (1) for setting and determining compliance with water quality-based effluent limitations (WQBEL) in the National Pollutant Discharge Elimination System (NPDES), the EPA proposed that when measured data are below the quantification level:

- Zero would be used in place of all analytical results below the quantification level.
- Quantification would be computed using the interim Minimum Level (ML).

During the summer of 1995, EPRI conducted an extensive review of available detection and quantification level definitions. This evaluation produced two major outputs: a list of properties and performance standards to evaluate detection and quantification level definitions, and a consensus agreement on an Alternative Minimum Level (AML) (2) to estimate a method's quantification level. The AML was based on the pioneering work of Currie (3, 4). EPRI's report on the AML (2) contains a complete discussion of the shortcomings with the EPA ML, the development of the AML, and AMLs calculated from EPRI interlaboratory data.

EPRI continued to refine definitions for detection and quantification, working within the American Society of Testing and Materials (ASTM) Committee D19 on Water Task Group D19.02.04.07 on Detection and Quantitation. The task group issued ASTM Standard Practices for an Interlaboratory Detection Estimate (IDE) (5) in 1997 and an Interlaboratory Quantitation Estimate (IQE) (6) in 2000.

The following sections review the various definitions of detection and quantification levels and discusses their applicability to environmental monitoring and permitting.

5.1 Currie Definitions of Detection and Quantification

Currie (3) defined two analytical measurement levels; the critical level (L_C) and the detection level (L_D). The L_C is the point at which "one may decide whether or not the result of an analysis indicates detection." The L_D is the point at which "a given analytical procedure may be relied upon to lead to a detection." There is an important distinction between these two levels. At the L_C one has confidence that a measurement is not a “false positive,” minimizing the chance of erroneously reporting that something is present when it is not. However, one must go up to the detection level before one can be confident that the analytical procedure is not “missing” a substance when it is actually present. At the L_D one is confident that a measurement is not a “false negative” or a “false positive.”

Statistically, the L_C is the concentration above which the response signal is significantly different from zero (Figure 5-1). When a measurement exceeds L_C, one can make the binary decision, "detected." A measured result at or above the critical level tells us the analyte is present. But a measured result below the critical level does not tell us with confidence that the analyte is absent. That is because replicate measurements of a sample with a true value equal to the critical level will fall both above and below the critical level at roughly the same frequency.

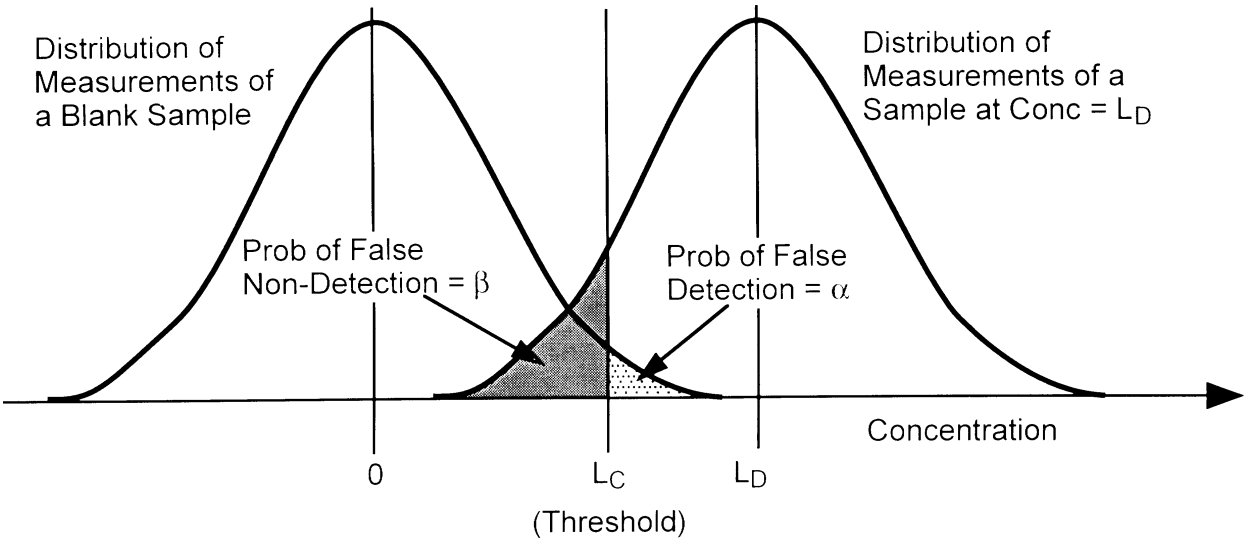

Figure 5-1
Relationship between Currie's L_c and L_D

When the true value equals the critical level, measured results falling below the critical level will be deemed non-detects. But those measured results really are false non-detects, given that the true value is equal to the critical level (and thus we know the analyte is present). Note that when the "true" concentration is zero, the probability of the correct decision "not detected" is $(1 - \alpha)$ where α is the Type I error rate or false positive rate of the statistical test.

Assuming a symmetric distribution of measurement errors, when the "true" concentration is equal to L_C, the probability of reporting it below L_C is 50%. This is termed the Type II error rate or false negative rate (β).

To accommodate both Type I and II errors Currie developed the concept of the "detection level," L_D. At the detection level, the non-detect problem is controlled. While replicate measurements of a sample with a true value equal to the L_D will fall both above and below the L_D, virtually all of the measurements falling below it will nonetheless fall above the L_C. Thus, for a true value equal to the L_D, virtually all of the measurements that fall below the L_D will be deemed detected. Only a very small, set percentage (e.g., 1 %) of those measurements will fall below the critical level and be deemed erroneously non-detects.

When the true concentration is L_D, then the Type II error rate (β) using L_C as the critical level is small. For example, assuming that $\alpha = \beta = 0.01$, another way of stating this is that 99% of the measured concentrations for samples not containing the analyte will be less than the L_C and 99% of the measured concentrations for samples with true concentration equal to L_D will be greater than L_C. That is, when the true concentration is equal to the L_D, the probability of a measurement below the L_C is 1% (3).

As can be seen from these definitions, the critical level and detection level are quite distinct. Above the critical level, we can have (1 - α) 100% confidence that the true concentration is greater than zero; whereas above the detection level we can have (1 - β) 100% confidence that the true concentration is greater than L_C. Table 5-1 summarizes the definitions and implications of the Currie scheme for defining detection and quantification levels.

Currie (3) defined the quantification level (L_Q) as the concentration "at which a given procedure will be sufficiently precise to yield a satisfactory quantitative estimate."

Table 5-1
Summary of Currie's Scheme for Detection and Quantification Levels

Level	Definition	Statistical Implications
Critical level, L_C	“The point at which one may decide whether or not the result of an analysis indicates detection.”	False positive (Type I) error rate controlled; signal is statistically different from zero.
Detection level, L_D	“The point at which a given analytical procedure may be relied upon to lead to a detection."	False positive and false negative (Type II) error rate controlled.
Quantification level, L_Q	“The concentration at which a given procedure will be sufficiently precise to yield a satisfactory quantitative estimate.”	Lowest concentration at which an RSD of 10% obtained.

5.2 EPA Definitions of Detection and Quantification

The EPA Method Detection Limit (7) currently applied by EPA in 40 CFR Part 136, computes a detection level based on the standard deviation of replicate measurements at a single concentration. For a particular sample analyte, *n* replicate analyses (minimum of 7) are performed at a single spiking concentration. All *n* replications typically are performed at the same time from a split sample. Based on the standard deviation(s) of these *n* measurements, a Currie L_C type parameter which EPA calls the "Method Detection Limit" (MDL) is calculated as:

$$MDL = t_{(0.01,n-1)} \cdot s \qquad \text{(eq. 5-1)}$$

where t is the appropriate value from the t-distribution for *n*-1 and s is sigma, the standard deviation of *n* measurements.

The procedure described in 40 CFR Part 136 does not address calculating an MDL using data from multiple laboratories. The MDLs published in EPA analytical methods sometimes are calculated from pooled single operator standard deviations from multiple laboratories. The single operator based MDL is typically a factor of 2 times lower than the MDL calculated from the interlaboratory standard deviation, which incorporates the random bias between laboratories.

The EPA's current estimate of a limit resembling a quantification level is based loosely on the "Limit of Quantitation (LOQ)" defined by the American Chemical Society (8) as the lowest concentration with an RSD of 10% (i.e., the ratio of measurement standard deviation to measurement mean concentration = 0.1 or, the measurement mean is ten times the standard deviation). While Currie also expressed the quantification level (L_Q) as a "10 sigma" limit, the sigma actually refers to the standard deviation of a net signal or concentration and not the true population standard deviation. If one assumes the "sigma" to be constant versus concentration, the 10 sigma quantification level can be shown to give an RSD of 10% because:

$$RSD = \left(\frac{s}{L_Q} \right) \times 100\% \qquad \text{(eq. 5-2)}$$

$$RSD = \left(\frac{s}{10s} \right) \times 100\% \qquad \text{(eq. 5-3)}$$

$$RSD = (0.10) \times 100\% \qquad \text{(eq. 5-4)}$$

or

$$RSD = 10\% \qquad \text{(eq. 5-5)}$$

The EPA interim Minimum Level (ML) was derived from the "10 sigma" definition by assuming that the standard deviation at the MDL and the interim ML are the same hence:

$$MDL = 3.14s \qquad \text{(eq. 5-6)}$$

and

$$ML \cong 10s \qquad \text{(eq. 5-7)}$$

so

$$\frac{ML}{MDL} = \frac{10s}{3.14s} = 3.18 \qquad \text{(eq. 5-8)}$$

thus:

$$ML \cong 3.18 \times MDL \qquad \text{(eq. 5-9)}$$

5.3 Modeling Standard Deviation Data

One significant shortcoming of EPA's estimates of detection and quantification is that they are based on an intralaboratory standard deviation at a single concentration. For permit level setting and monitoring, owners/operators of permitted facilities require definitions based on the interlaboratory standard deviation to account for the variability associated with the analysis of split samples and the use different laboratories over the life of a permit. The estimates must be developed over a range of concentrations to account for changes in standard deviation with concentration.

Figure 5-2 plots interlaboratory standard deviation versus true concentration data for all of the EPRI graphite furnace atomic absorption spectroscopy (GFAAS) studies for the freshwater matrices studied - reagent grade water (RGW), river water (RW) and ash pond overflow (APO) collected in the EPRI AMQ studies (note: the x-axis is logarithmic to accentuate the low concentrations). At or below the critical level the standard deviation is nearly constant, primarily influenced by instrument noise. At some concentration the standard deviation starts to increase and eventually becomes proportional to the true concentration. A Rocke and Lorenzato (9) fit in the form

$$s_t = \left(a_0 + a_1 T^2\right)^{1/2} \qquad \text{(eq. 5-10)}$$

is shown, where s_t is the interlaboratory standard deviation as defined by ASTM D 2777 (10).

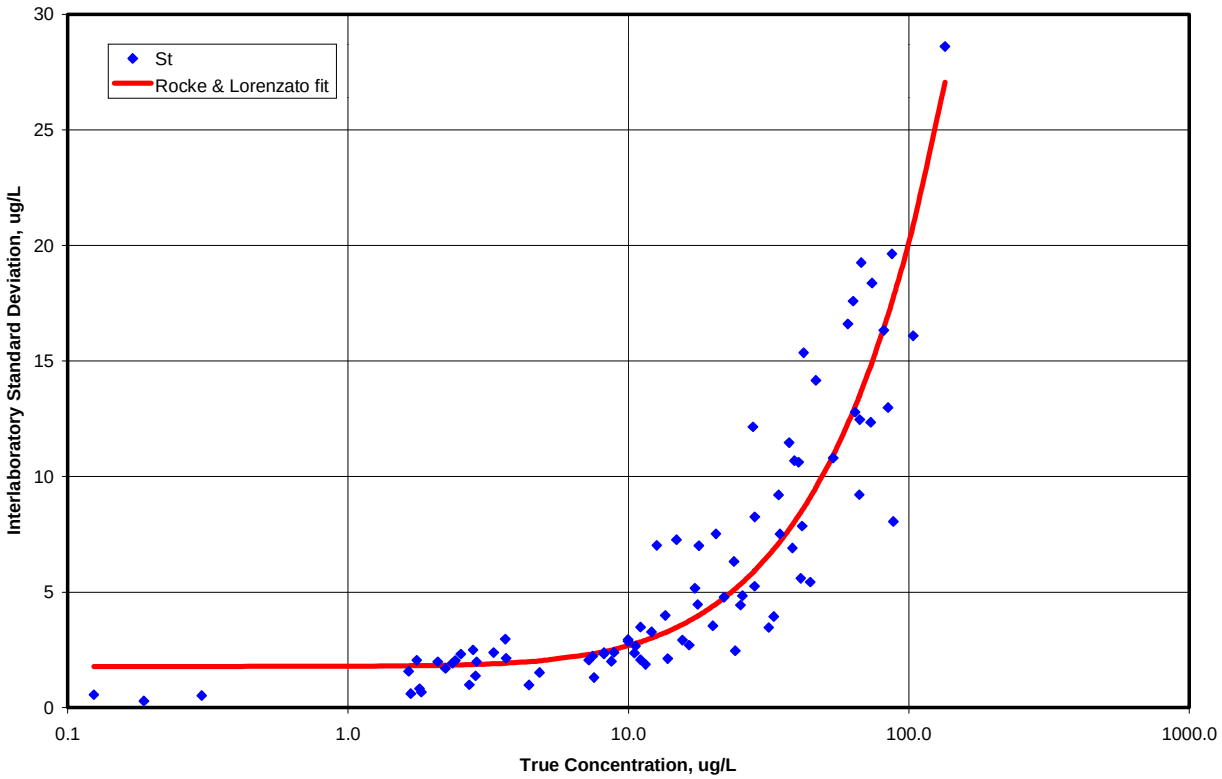

Figure 5-2
Curvilinear relationship of interlaboratory standard deviation versus true concentration; EPRI GFAAS data for RGW, RW and APO

The Rocke and Lorenzato (9) model reflects the "hockey stick" form of the relationship between standard deviation and the true concentration. This model is based on the assumption that the variance is composed principally of two error terms: 1) errors that are unrelated to analyte concentration (e.g. ambient contamination) and 2) errors that are proportional to analyte concentration (e.g. nebulizer flow variability).

5.4 Alternative Minimum Level

Shortly after the EPA issued their definition for the interim ML, EPRI began to develop an Alternative Minimum Level (AML). The AML is defined as 10 times the interlaboratory standard deviation at the lowest concentration that is differentiable from zero. The lowest concentration that is differentiable from zero can be statistically determined and is therefore anchored, unlike the MDL which is used to compute the interim ML.

EPRI's report on the AML (2) contains a complete discussion of the shortcomings with the EPA ML, the development of the AML, and AMLs calculated from EPRI interlaboratory data. A shortcoming of the AML is that it is not possible to select a relative standard deviation (RSD) associated with the calculated AML. To provide users with a quantification estimate with a selectable RSD, the ASTM developed the Interlaboratory Quantitation Estimate (IQE), discussed in the following section.

5.5 ASTM Interlaboratory Detection and Quantitation Estimates

In parallel with the development of the AML, the ASTM Committee D19 on Water Task Group D19.02.04.07 on Detection and Quantitation was also developing alternatives to EPA's MDL and interim ML. EPRI was directly involved in the task group's consensus development of the Interlaboratory Detection Estimate (5) and Interlaboratory Quantitation Estimate (6). Both the IDE and IQE incorporate interlaboratory standard deviation over a range of concentrations.

5.5.1 Interlaboratory Detection Estimate (IDE)

The IDE is defined as "the lowest concentration at which there is 90% confidence that a single measurement from a laboratory selected from the population of qualified laboratories represented in an interlaboratory study will have a true detection probability of a least 95% and a true non-detection probability of at least 99%" (5). It is an implementation of the basic principles for detection outlined by Currie (3) combined with the general approaches advocated by developers of calibration based detection levels. A measured critical level is computed from the standard deviation data and corrected to a true concentration using the recovery equation. A detection level is then computed. The key feature of the IDE is that it is based on interlaboratory standard deviation models, including the Rocke and Lorenzato model, and it uses tolerance limits to address the issue of estimating performance of multiple laboratories in the future.

The IDE addresses all of the problems with the MDL outlined in EPRI's AML report (2) and provides a true consensus standard for detection for use in permitting. The IDE calculation procedure is detailed in ASTM Standard Practice D 6091 (5).

5.5.2 Interlaboratory Quantitation Estimate (IQE)

Building on the success of the IDE, the same ASTM task group developed a standard practice for an Interlaboratory Quantitation Estimate (IQE) (6). The $IQE_{Z\%}$ is defined as "the lowest concentration for which a single measurement from a laboratory selected from the population of qualified laboratories represented in an interlaboratory study will have an estimated Z% relative standard deviation (Z% RSD, based on interlaboratory standard deviation), where Z is typically an integer multiple of 10, such as 10, 20 or 30." $IQE_{10\%}$ is consistent with Currie's (3) quantitation approach. The standard deviation data from an interlaboratory study are fit to one of three models, including constant, linear and the Rocke and Lorenzato fit, that describe the interlaboratory standard deviation (ILSD) of measurements and the true concentration. The principal difference between the AML and the IQE is that the IQE provides an estimate of the quantification level for a selected RSD. Details regarding the calculation of the IQE are found in the ASTM Standard Practice, D 6512 (6).

The IDE and IQE, as well as EPRI's AML, share a common heritage based on Currie (3). Together, they represent a serious effort to address the shortcomings in the EPA definitions for detection (MDL) and quantification (interim ML).

5.5.3 Computation of the IDE and IQE

As described in Section 3, all of the data in this report were first processed through EPRI's software program STATCALC according to ASTM D 2777 (9) to remove laboratories with consistent bias (lab ranking), remove individual outliers, and produce an outlier-free data set. EPRI developed a program called QCalc to calculate IDEs and IQEs in accordance with ASTM D 6091 and D 6512, respectively, from the outlier-free data sets produced by STATCALC.

5.6 References

1. USEPA, National Guidance for the Permitting, Monitoring, and Enforcement of Water Quality-based Effluent Limitations Set Below Analytical Detection/Quantitation Levels, March 22, 1994.

2. Scott, J.W., N.T. Whiddon and R.F. Maddalone, Alternative Minimum Levels for Utility Aqueous Discharges: Chemical Analytical Measurement Guide for National Pollutant Discharge Elimination Systems (NPDES) Permits, Electric Power Research Institute, Palo Alto, CA: November 1996. Report TR-106220.

3. Currie, L.A., "Limits For Qualitative Detection And Quantitative Determination: Application To Radiochemistry," Analytical Chemistry, Vol 40, p. 586 (1968).

4. Currie, L.A., "Nomenclature in Evaluation of Analytical Methods Including Detection and Quantification Capabilities," Pure & Appl. Chem., Vol. 67, No. 10, p1699 (1995).

5. American Society for Testing and Materials, "Standard Practice for a 99%/95% Interlaboratory Detection Estimate (IDE) for Analytical Methods with Negligible Calibration Error," D 6091.

6. American Society for Testing and Materials, "Standard Practice for an Interlaboratory Quantitation Estimate," D 6512.

7. USEPA, "Appendix B to Part 136 - Definition and Procedure for the Determination of the Method Detection Limit - Revision 1.11," Federal Register, 49, (209), 43430, Friday, October 26, 1984.

8. Kaiser, H., "Quantitation in Elemental Analysis (Part 2)," Anal. Chem. Vol. 42, No. 4, p. 26A (1970).

9. Rocke, D.M. and S. Lorenzato, "A Two-Component Model for Measurement Error in Analytical Chemistry," Technometrics, Vol. 37(2), p. 176 (1995).

10. American Society for Testing and Materials, "Standard Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water," D 2777.

6
RESULTS OF DATA ANALYSIS

This section presents the IDEs and IQEs calculated from the study data, and compares these parameters to EPA's detection and quantification measures. The precision and recovery regression coefficients required for calculation of the IDE and IQE are presented in Table 6-1. To obtain these coefficients, a regression model was selected for each matrix/element data set, based on the curvatures of the interlaboratory standard deviation versus true concentration (precision) plots shown in Section 4. When the precision data showed evidence of curvature, the data were fit with the Rocke-Lorenzato equation. If the data did not show curvature, they were fit with the constant model. In Table 6-1, if both a_0 and a_1 are shown, the precision equation was fit by the Rocke-Lorenzato function. If only a_0 is shown, the data are fit by the constant model.

The Interlaboratory Detection Estimates (IDEs) and Interlaboratory Quantitation Estimates (IQE) were computed as described in Section 5. Table 6-2 compares these values to the EPA Method Detection Limit (MDL) and Minimum Level (ML) listed in EPA draft Method 1638. This table also shows the lowest freshwater or human health Water Quality Criterion (WQC) for each analyte. The WQCs shown are calculated for 25 mg/L hardness, where applicable. IDEs and IQEs that are lower than the lowest spiked concentration or higher than the highest spiked concentration are highlighted to indicate that they do not meet the criteria specified in the ASTM standard practices.

6.1 Data Evaluation

Graphical illustrations of the freshwater and reagent grade water IDEs and IQEs compared to the EPA MDLs, MLs and lowest EPA WQCs are shown in Figures 6-1 through 6-8. Data for silver are omitted as silver was not stable in the study matrices.

As shown in Figure 6-1, the freshwater and reagent grade water IDEs for antimony were 11 and 7 times higher than the EPA MDL, respectively. The $IQE_{20\%}$ values were 4 to 6 times higher than the ML. The IDEs and IQEs were well below the lowest EPA WQC for antimony.

Figure 6-2 shows the cadmium IDEs and IQEs compared to the EPA values. The calculated IDE for reagent grade water was 6 times the EPA MDL and the IDE for freshwater was 4 times the EPA MDL. The $IQE_{20\%}$for cadmium in reagent grade water was about twice the EPA ML and the freshwater $IQE_{20\%}$ was approximately equal to the EPA ML. The IDEs and IQEs were less than the WQC, indicating that the method performs satisfactorily for measuring cadmium concentrations in reagent grade water and freshwater at the WQC.

The data for copper are shown in Figure 6-3. The reagent grade water IDE was 9 times the EPA MDL and the freshwater IDE was 13 times the EPA MDL. The $IQE_{20\%}$ were 5 and 6 times the EPA ML. The IDEs and $IQE_{20\%}$ were less than the WQC. Laboratories should be able to use this method for monitoring copper in reagent grade water and similar freshwater at the WQC.

Table 6-1
Regression Coefficients for Precision and Recovery, Method 1638 Validation Study

		Recovery $X = b_0 + b_1 T$		**Single Operator Precision** $s_o = \sqrt{a_0 + a_1 T^2}$		**Overall Precision** $s_t = \sqrt{a_0 + a_1 T^2}$	
Analyte	**Matrix**	b_0	b_1	a_o	a_1	a_o	a_1
Sb	RGW	-0.016	0.765	0.0003328	0.0001306	0.0002495	0.0030467
	FW	0.0	0.956	0.0000596		0.0001973	
Cd	RGW	0.0	0.971	0.000301	0.0000643	0.0010292	0.0030176
	FW	-0.009	1.02	0.0000382	0.0010392	0.000566	
Cu	RGW	0.0	0.97	0.0132075		0.0854685	0.0028767
	FW	0.0	0.908	0.011572		0.048559	
Pb	RGW	0.0	0.99	0.000319	0.0001961	0.0013176	0.0011392
	FW	0.0	0.955	0.00089		0.001527	
Ni	RGW	-0.001	0.924	0.0008421	0.0002867	0.0011572	0.0007170
	FW	0.0	0.938	0.0003302	0.0014455	0.0130709	0.0060176
Se	RGW	0.0	0.976	0.057535		0.038505	0.007721
	FW	0.0	0.988	0.0330731		0.08591	
Ag	RGW	0.0	0.828	0.000359	0.0004502	0.0325708	0.0109851
	FW	0.0	0.225	0.0000071	0.0025911	0.028767	
Tl	RGW	0	0.98	0.0000689	0.000249	0.0000295	0.002847
	FW	0.0	0.96	0.0000018	0.0005865	0.0000075	0.0068777
Zn	RGW	0.0	0.974	0.0559765	0.0072785	0.2609354	0.0094852
	FW	0.0	0.971	0.0193597		0.114496	

RGW = Reagent Grade Water, FW = Freshwater

Shaded values were obtained using data sets that included less than 5 pairs of data at one or more concentration level and therefore do no not meet ASTM D 2777criteria.

Table 6-2
Summary of Results Computed from the Method 1638 Validation Study

	Sb		Cd		Cu		Pb		Ni	
	RGW	**FW**	**RGW**	**FW**	**RGW**	**FW**	**RGW**	**FW**	**RGW**	**FW**
Lower Test Concentration, µg/L	0	0.1328	0.0041	0	0.1052	1.2599	0.0192	0.0429	0	0.6716
Lowest Spiked Concentration, µg/L	0.1	0.2128	0.0541	0.04	0.3052	1.7599	0.0492	0.0829	0.5	1.9716
Upper Test Concentration, µg/L	20	1.0128	10.0041	0.25	20.1052	5.2599	5.0192	0.3929	10	6.3716
IDE, µg/L	0.11	0.07	0.15	0.11	0.77	1.12	0.16	0.19	0.13	0.64
$IQE_{10\%}$, µg/L	0.27	0.15	0.38	0.23	3.02	2.40	0.37	*0.41*	0.33	1.91
$IQE_{20\%}$, µg/L	0.12	0.07	0.17	0.12	0.90	1.20	0.18	0.20	0.14	0.71
$IQE_{30\%}$, µg/L	0.08	0.05	0.11	0.08	0.56	0.80	0.12	0.14	0.09	0.46
EPA MDL, µg/L	0.0097		0.025		0.087		0.015		0.33	
EPA ML, µg/L	0.02		0.1		0.2		0.05		1	
Lowest WQC, µg/L*		14*		0.8		2.7		0.54		16
RSDo at ML	91.2%	38.6%	17.4%	6.2%	57.5%	53.8%	36%	60%	5%	2%
RSDt at ML	79%	70%	33%	24%	146%	110%	73%	78%	4%	11%

RGW = Reagent Grade Water, FW = Freshwater

* Lowest of the freshwater or human health WQC, EPA 822-Z-99-001(April 1999) from 40CFR131.36. All values dissolved criteria. Hardness dependent freshwater dissolved WQCs calculated for 25 mg/L hardness as $CaCO_3$.
*** = Value could not be calculated
Shaded values are outside of the spiked concentration range and therefore do not meet criteria in the applicable ASTM standard.

Table 6-2, continued
Summary of Results Computed from the Method 1638 Validation Study

	Se		Ag		Tl		Zn	
	RGW	**FW**	**RGW**	**FW**	**RGW**	**FW**	**RGW**	**FW**
Lower Test Concentration, µg/L	0.0318	0.2055	0.0048	0.0311	0	0.001	0.1304	0.9413
Lowest Spiked Concentration, µg/L	1.0318	0.8055	0.1048	0.1011	0.04	0.021	0.6304	1.0713
Upper Test Concentration, µg/L	20.0318	3.8055	10.0048	0.3911	2	0.121	50.1304	2.1613
IDE, µg/L	0.6	1.39	***	0.384	0.02	0.003	2.1	1.52
$IQE_{10\%}$, µg/L	***	2.99	***	0.82	0.05	***	***	3.25
$IQE_{20\%}$, µg/L	0.72	1.49	***	0.41	0.02	***	2.56	1.62
$IQE_{30\%}$, µg/L	0.44	1.00	***	0.27	0.01	***	1.55	1.08
EPA MDL, µg/L	0.45		0.029		0.0079		0.14	
EPA ML, µg/L	1		0.1		0.02		0.5	
Lowest WQC, µg/L*		4.6		3.4		1.7		37
RSDo at ML	24%	18%	19%	3%	42%	7%	48%	28%
RSDt at ML	22%	29%	181%	170%	28%	14%	103%	68%

RGW = Reagent Grade Water, FW = Freshwater

* Lowest of the freshwater, marine or human health WQC, EPA 822-Z-99-001(April 1999) from 40CFR131.36. All values dissolved criteria. Hardness dependent freshwater dissolved WQCs calculated for 25 mg/L hardness as $CaCO_3$.
*** = Value could not be calculated
Shaded values are outside of the spiked concentration range and therefore do not meet criteria in the applicable ASTM standard.

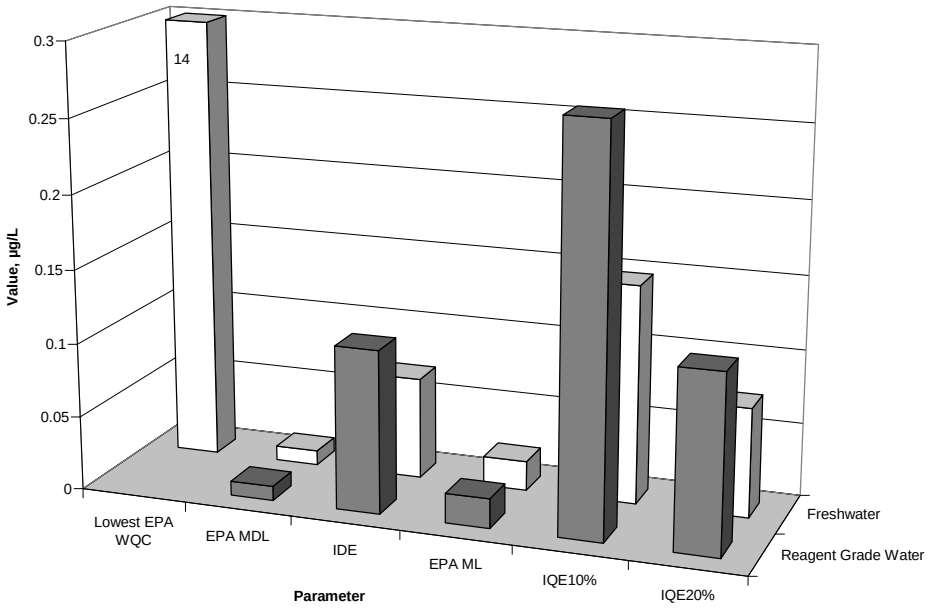

Figure 6-1
Comparison of Antimony IDE and IQE to EPA WQC, MDL and ML

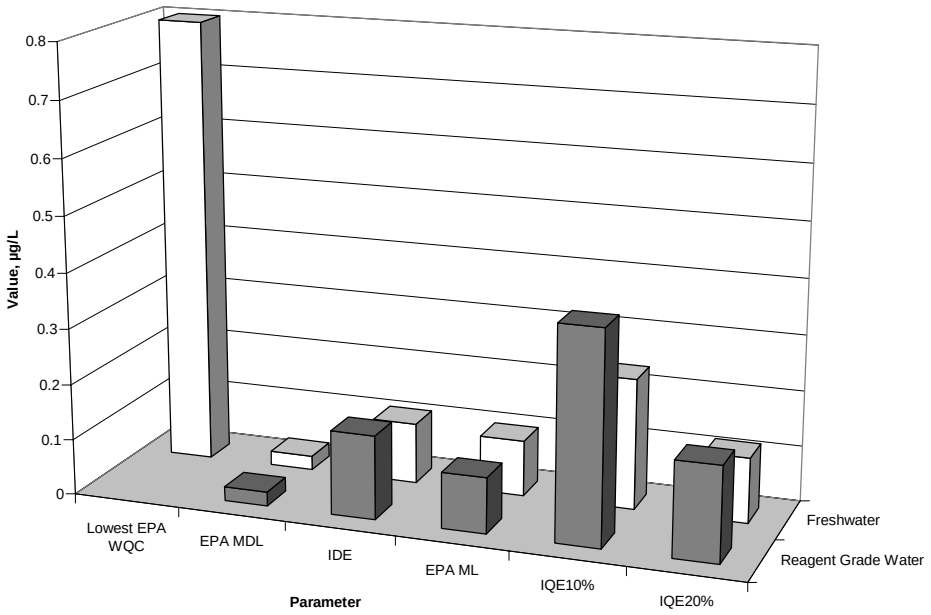

Figure 6-2
Comparison of Cadmium IDE and IQE to EPA WQC, MDL and ML

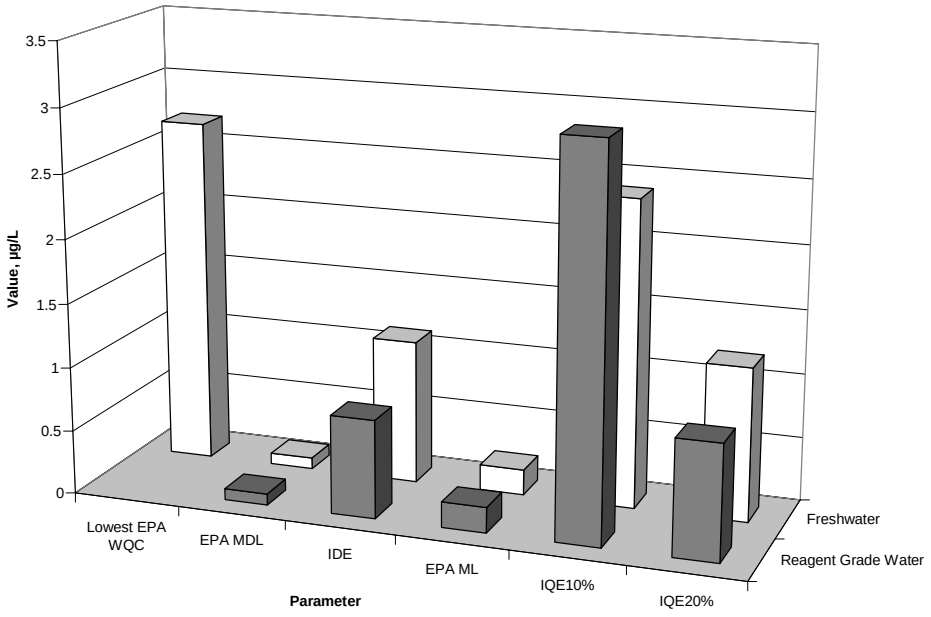

Figure 6-3
Comparison of Copper IDE and IQE to EPA WQC, MDL and ML

Figure 6-4 shows the detection and quantitation parameters for lead. The IDE in reagent grade water was 11 times higher than the EPA MDL, while the IDE in freshwater was approximately 13 times higher. The $IQE_{20\%}$ in both matrices were approximately 4 times the EPA ML. The IDE and $IQE_{20\%}$ were all below the WQC, indicating that the method is suitable for analysis of lead at the WQC level in these matrices.

The parameters for nickel are shown in Figure 6-5. The IDE in reagent grade water was less than the EPA MDL. The IDE in freshwater was approximately 2 times the EPA MDL. The $IQE_{20\%}$ in both matrices were lower than the EPA ML, which indicates that this method can be used to quantify nickel at the EPA ML. The IDEs and IQEs were all well below the WQC.

The detection and quantitation parameters for selenium are shown in Figure 6-6. The reagent grade water IDE was slightly higher than the EPA MDL while the $IQE_{20\%}$ was lower than the EPA ML. The method can provide quantitative data for selenium at the EPA ML in reagent grade water. In freshwater, the IDE was 3 times higher than the EPA MDL and the $IQE_{20\%}$ was 1.5 times the EPA ML. The IDEs and $IQE_{20\%}$ were significantly lower than the WQC, indicating that the method could be used to monitor selenium in reagent grade water and similar freshwater at the WQC.

The IDEs and IQEs for thallium compared to the EPA MDL and ML are shown in Figure 6-7. In freshwater, the IDE was less than the corresponding MDL. An IQE could not be reported for freshwater, because the calculated $IQE_{20\%}$ and $IQE_{30\%}$ values were below the lowest concentration reportable by the software program used to calculate these values (0.01 μg/L). The IDE in reagent grade water was 3 times higher than the EPA MDL and the $IQE_{20\%}$ was approximately

equal to the EPA ML. The data indicate that the method would be suitable for quantitation of thallium in a similar freshwater matrix at the EPA ML. The IDE and $IQE_{20\%}$ for thallium in freshwater are well below the lowest EPA WQC.

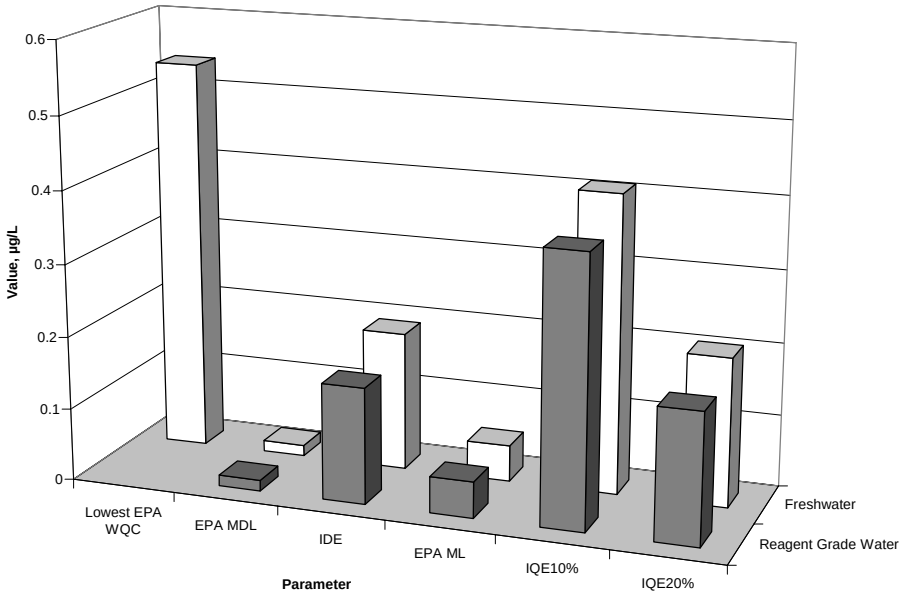

Figure 6-4
Comparison of Lead IDE and IQE to EPA WQC, MDL and ML

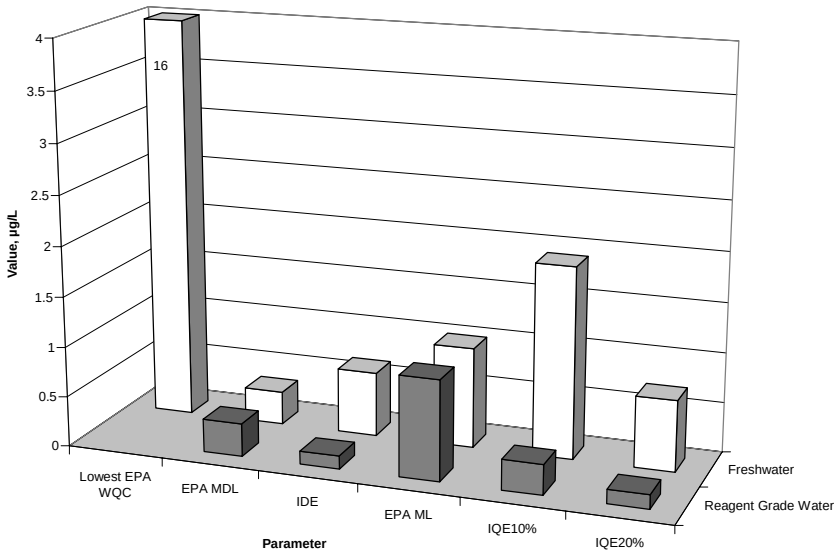

Figure 6-5
Comparison of Nickel IDE and IQE to EPA WQC, MDL and ML

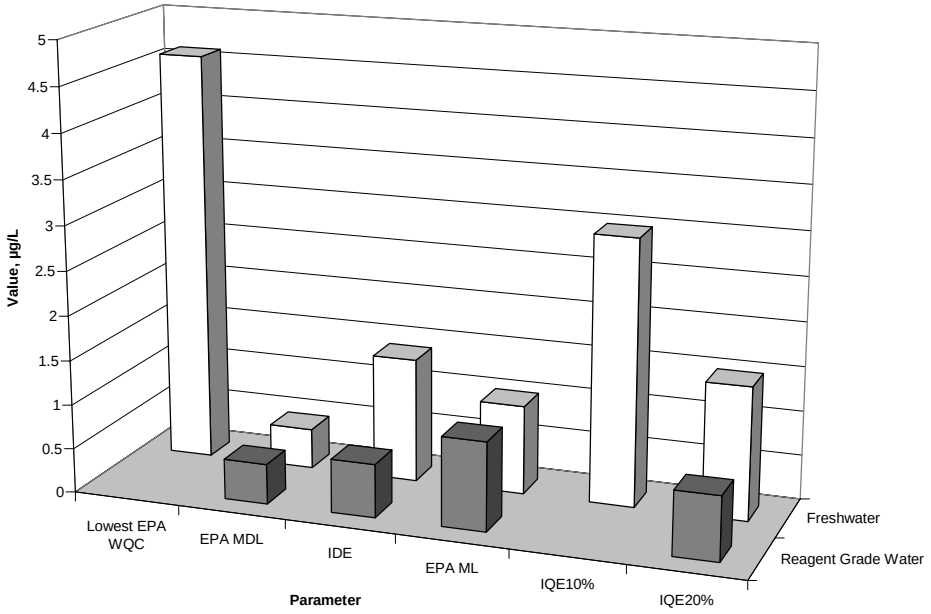

Figure 6-6
Comparison of Selenium IDE and IQE to EPA WQC, MDL and ML

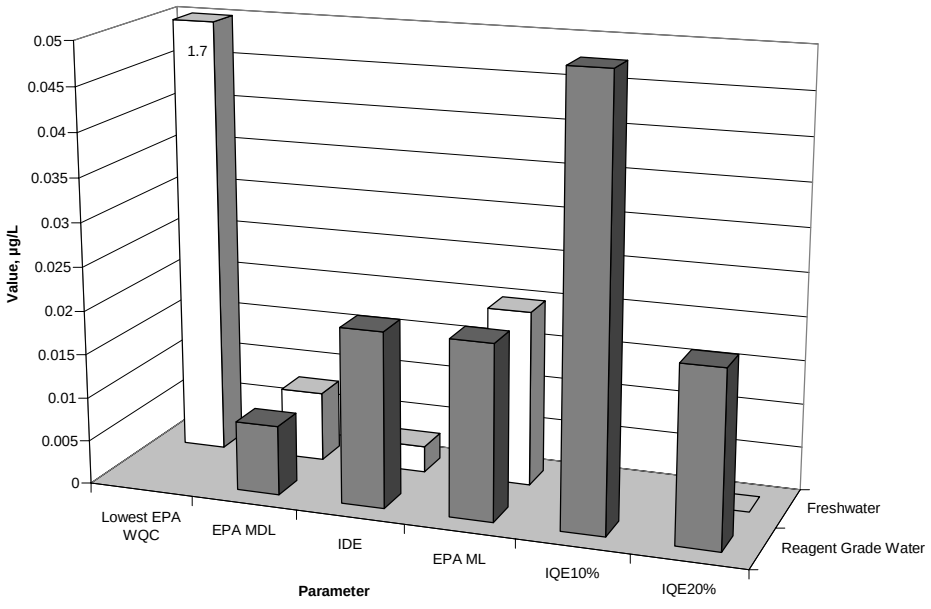

Figure 6-7
Comparison of Thallium IDE and IQE to EPA WQC, MDL and ML

The detection and quantitation parameters for zinc are plotted in Figure 6-8. The IDE in reagent grade water was 15 times the EPA MDL and in freshwater it was 11 times the EPA MDL. The $IQE_{20\%}$ in reagent grade water was 5 times the EPA ML and in freshwater it was 3 times the EPA ML. IDEs and $IQE_{20\%}$ were well below the freshwater WQC, indicating that the method can be used for analysis of zinc in reagent grade water and freshwater at the WQC.

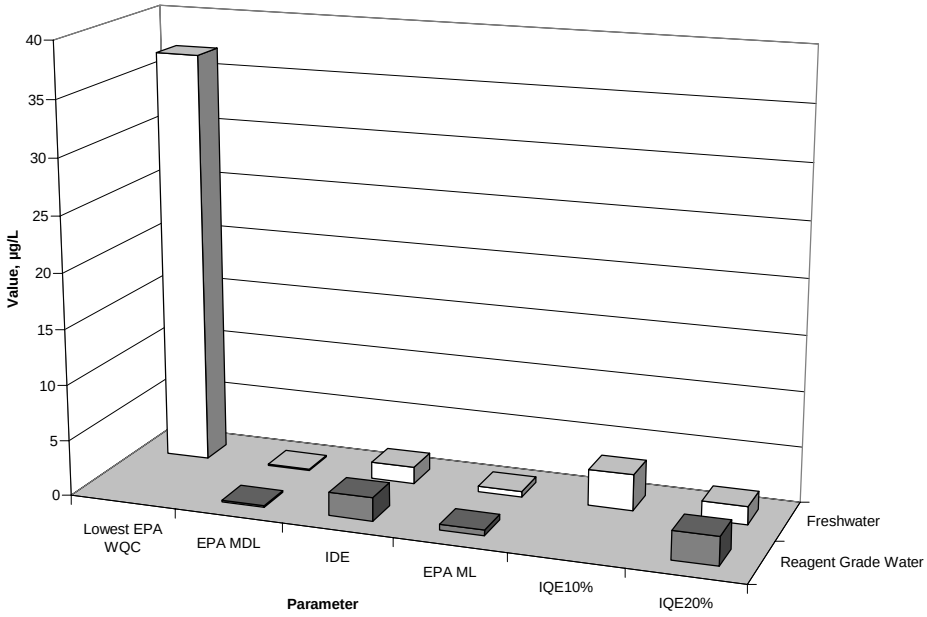

Figure 6-8
Comparison of Zinc IDE and IQE to EPA WQC, MDL and ML

7
SUMMARY AND CONCLUSIONS

EPA Draft Method 1638 was evaluated in reagent water and in filtered lake water (Lake Washington, WA). Filtered and unfiltered secondary treated municipal wastewater were also included in the study; however, since only a single pair of filtered effluent samples and a single pair of unfiltered effluent samples were analyzed, the method remains unvalidated in those matrices. The method's performance at concentrations other than those contained in the effluent samples can not be determined from the current study. The method has not been validated in other matrices.

The findings of EPRI's evaluation of the Method 1638 data include the following:

- For most elements, the samples were stable (>80% recovery) when tested by the referee laboratory one month after shipment to the participants, with the following exceptions:

  Recoveries of antimony spiked into the lowest concentration pair of reagent grade water samples were 8% and 57%.

  Recoveries of silver spiked into the reagent grade water matrix at 0.2 and 0.24 μg/L were negative.

  Recoveries of silver spiked into freshwater at 0.07 and 0.08 μg/L were negative.

  Recoveries of silver spiked into freshwater at 0.3 and 0.36 μg/L were 27% and 22%, respectively.

  Recovery of zinc spiked into reagent grade water at 0.5 μg/L was 70%.

- For all data sets except antimony, reported MDL data for two or more laboratories failed the Hald test for equality of variances and therefore a pooled MDL could not be calculated.

  Reported cadmium MDLs ranged from 0.002 to 0.077 μg/L. The EPA MDL is 0.0025 μg/L.

  Reported copper MDLs ranged from 0.008 to 0.148 μg/L. The EPA MDL is 0.087 μg/L.

  Reported lead MDLs ranged from 0.0017 to 0.0129 μg/L. The EPA MDL is 0.015 μg/L.

  Reported nickel MDLs ranged from 0.009 to 0.109 μg/L. The EPA MDL is 0.33 μg/L.

  Reported selenium MDLs ranged from 0.152 to 0.927 μg/L. The EPA MDL is 0.45 μg/L.

  Reported silver MDLs ranged from 0.006 to 0.028 μg/L. The EPA MDL is 0.029 μg/L.

  Reported thallium MDLs ranged from 0.001 to 0.015 μg/L. The EPA MDL is 0.0079 μg/L.

  Reported zinc MDLs ranged from 0.043 to 0.187 μg/L. The EPA MDL is 0.14 μg/L.

- The pooled MDL for antimony was 0.008 µg/L, compared to the EPA MDL of 0.0097 µg/L. Reported antimony MDLs ranged from 0.005 to 0.048 µg/L.
- Mean recoveries reported by the participants for each element in each matrix were generally good with the exception of silver. Silver was determined to be unstable under the conditions of this study, and therefore EPRI did not evaluate detection and quantification levels for this element. Recoveries of antimony in reagent grade water tended to be a little low. Lead in reagent grade water and zinc in freshwater had some unusually high recoveries, perhaps due to contamination of the samples in the laboratory with these ubiquitous metals.
- Recoveries of the study elements from the standard reference material, NIST SRM 1632d, ranged from 93.3% to 102% for all data reported by the participants. Although this would tend to indicate that the method is free from bias, the concentrations of the study elements in the NIST sample ranged from 3 to 50 times higher than the highest spiked concentrations in the freshwater study samples. Thus, recoveries from this SRM are not a good predictor of the method's performance with real-world samples.

EPRI calculated IDE and IQE values for each of the elements and compared these values (except silver) with the EPA's MDL and ML, as well as the lowest of the National water quality criteria (WQC) for protection of human health and aquatic organisms. The results of these comparisons were as follows:

- All IDEs were greater than the corresponding EPA MDL, with the exception of nickel in reagent grade water and thallium in freshwater. The other IDEs ranged from 1.3 to 15 times the EPA MDL.
- $IQE_{20\%}$ values exceeded the corresponding EPA ML in most cases. The IQE at a 20% RSD was used for comparison because most other quantitation levels are set at this RSD. Only nickel in reagent grade water and freshwater, thallium in reagent grade water, and selenium in reagent grade water had $IQE_{20\%}$ less than or equal to the corresponding EPA MLs. The other $IQE_{20\%}$ values ranged from 1.2 to 6 times the EPA ML.
- For each of the study elements having a WQC, the IDE and $IQE_{20\%}$ for the freshwater matrix were both below the lowest WQC.

A comparison of the IDEs and IQEs calculated from the study data with EPA's parameters for detection and quantification indicates that Draft Method 1638 generally is not appropriate for compliance monitoring at or near the EPA MDLs and MLs quoted in the method. Few of the tested elements could be detected or quantified with an acceptable level of precision at or near the EPA MDLs and MLs. On the other hand, Method 1638 is suitable for the analysis of the tested analytes at the lowest National WQC in freshwater matrices.

The analyst should use caution when utilizing the IDE and IQE values in this report when such values are below the lowest spiked concentration or above the highest spiked concentration in the study.

A
NOMENCLATURE

Table A-1
Nomenclature

Abbreviation	Description
AAS	Atomic Absorption Spectroscopy
Ag	Silver
AMQ	Analytical Methods Qualification
AML	Alternative Minimum Level
APO	Ash Pond Overflow
ASTM	American Society for Testing and Materials
Cd	Cadmium
CFR	Code of Federal Regulations
Cu	Copper
EPA	Environmental Protection Agency
FEF	Filtered Effluent
FR	Federal Register
FW	Freshwater
GFAAS	Graphite Furnace Atomic AAS
IDE	Interlaboratory Detection Estimate
IIAG	Inter-Industry Analytical Group
ILSD	Interlaboratory Standard Deviation
IQE	Interlaboratory Quantitation Estimate
L_C	Critical Level (Currie)
L_D	Detection Level (Currie)
L_Q	Determination Level

Abbreviation	Description
LOD	Limit of Detection
LOQ	Limit of Quantitation
MDL	Method Detection Limit
ML	Minimum Level
MSA	Method of Standard Additions
Ni	Nickel
NIST	National Institute of Standards & Technology
Pb	Lead
QC	Quality Control
R-L	Rocke-Lorenzato
RGW	Reagent Grade Water
RSD	Relative Standard Deviation
RW	River Water
SCC	Sample Control Center
Se	Selenium
SRM	Standard Reference Material
Tl	Thallium
UEF	Unfiltered Effluent
WQC	Water Quality Criteria
Zn	Zinc

B
RAW DATA

Table B-1
Raw Sample Data

Values in italics were above the reported calibration limit (per the SCC) and were not used in EPRI data processing. Values in parentheses were flagged by the SCC as not used in their final report but no explanation was provided. These data were used in EPRI's evaluation of the data.

Element	Matrix	Lab	1	2	3	4	5	6	7	8	9	10
Sb	5	1	0.0000	0.0000	0.0338	0.0957	0.6190	0.6800	3.7500	3.8800	17.1000	16.9000
Sb	5	2	-0.0041	-0.0158	0.0461	0.0518	0.6302	0.6413	4.2280	4.2610	18.9000	18.4100
Sb	5	3	0.0027	0.0017	0.0426	0.1099	0.6640	0.7064	3.9114	3.9958	16.1164	16.3849
Sb	5	4	0.0260	0.0240	0.0500	0.0620	0.6990	0.7120	4.1680	4.1230	16.5480	16.6010
Sb	5	5	-0.0132	-0.0108	0.0691	0.0424	0.6278	0.6425	4.1322	4.1184	*18.7491*	*18.6683*
Sb	5	6	0.0115	0.0131	0.0971	0.1484	0.7091	0.6579	4.4013	4.5073	19.0607	19.0956
Sb	5	7	-0.0017	-0.0006	0.0564	0.1016	0.6017	0.5732	3.7466	3.7478	18.0506	18.1348
Sb	5	8	0.0409	0.0109	0.0662	0.0992	0.6547	0.6252	3.8980	3.8641	18.4290	18.7830
Sb	16	1	0.1240	0.1190	0.2000	0.2260	0.3720	0.4660	0.7460	0.9420		
Sb	16	2	0.1588	0.1116	0.2013	0.2331	0.3645	0.4666	0.7920	0.9939		
Sb	16	3	0.1300	0.1246	0.2053	0.2454	0.3884	0.4765	0.7639	0.9755		
Sb	16	4	0.1410	0.1380	0.2070	0.2460	0.3880	0.4820	0.7440	0.9060		
Sb	16	5	0.1102	0.1050	0.1984	0.2290	0.3728	0.4789	0.7775	0.9871		
Sb	16	6	0.2055	0.1388	0.2307	0.2563	0.4046	0.4759	0.7725	0.9653		
Sb	16	7	0.1419	0.1326	0.2136	0.2490	0.3957	0.4948	0.7850	0.9860		
Sb	16	8	0.1514	0.1352	0.2939	0.2723	0.4069	0.4886	0.7861	0.9654		
Sb	17	1	1.0900	0.9940								
Sb	17	2	0.9923	1.0330								
Sb	17	3	1.0196	0.9876								
Sb	17	4	0.9800	1.1340								
Sb	17	5	(0.8571)	1.0284								

Raw Data

Element	Matrix	Lab	1	2	3	4	5	6	7	8	9	10
Sb	17	6	1.0240	1.1957								
Sb	17	7	1.1239	1.0088								
Sb	17	8	1.0597	1.0647								
Sb	18	1	9.7600	9.6000								
Sb	18	2	(8.4000)	9.0020								
Sb	18	3	9.0936	9.2436								
Sb	18	4	8.7510	8.9420								
Sb	18	5	*10.5392*	*9.7860*								
Sb	18	6	9.9574	9.6147								
Sb	18	7	9.8225	9.9053								
Sb	18	8	9.9690	9.8699								
Cd	5	1	0.0000	0.0000	0.0469	1.8400	0.0672	0.0786	0.9460	0.8640	8.6700	8.7400
Cd	5	2	0.0170	0.0123	0.0678	0.0785	0.1233	0.1166	1.0400	1.0600	9.8920	9.9640
Cd	5	3	0.0038	0.0031	0.0541	0.0633	0.0987	0.0981	0.9950	0.9564	10.0215	9.9182
Cd	5	4	0.0050	-0.0010	0.0470	0.0600	0.1100	0.0980	1.0180	1.0490	10.0840	10.1590
Cd	5	5	0.0895	0.0715	0.0977	0.1210	0.1595	0.1717	0.9707	1.0440	9.1160	9.3767
Cd	5	6	0.0088	0.0148	0.0558	0.0977	0.1240	0.1038	1.0202	1.1519	10.0737	10.1802
Cd	5	7	-0.0348	-0.0347	0.0162	0.0222	0.0726	0.0598	0.9869	0.9937	10.0357	10.1000
Cd	5	8	-0.0208	-0.0170	0.0207	0.0349	0.0840	0.0857	0.9206	0.9097	9.4199	9.5763
Cd	16	1	0.0000	0.0001	0.0224	0.0576	0.1190	0.1510	0.2080	0.2180		
Cd	16	2	-0.0634	0.0042	0.0582	0.0699	0.1212	0.1466	0.2926	0.2465		
Cd	16	3	0.0040	0.0048	0.0424	0.0635	0.1281	0.1426	0.2497	0.2538		
Cd	16	4	-0.0090	-0.0070	0.0320	0.0530	0.1140	0.1320	0.2750	0.2600		
Cd	16	5	0.0420	0.0360	0.1028	0.1133	0.1829	0.1926	0.3067	0.3099		
Cd	16	6	0.0132	0.0157	0.0349	0.0640	0.1414	0.1570	0.2666	0.2657		
Cd	16	7	-0.0373	-0.0370	-0.0008	0.0108	0.0816	0.1053	0.2298	0.2180		
Cd	16	8	-0.0218	-0.0203	0.0127	0.0194	0.0942	0.1121	0.2322	0.2185		
Cd	17	1	0.3040	0.2910								
Cd	17	2	0.1854	0.2856								
Cd	17	3	0.3243	0.3054								
Cd	17	4	0.1990	0.3680								

Element	Matrix	Lab	1	2	3	4	5	6	7	8	9	10
Cd	17	5	0.3996	0.4039								
Cd	17	6	0.3644	0.3490								
Cd	17	7	0.1585	0.1832								
Cd	17	8	0.2916	0.2988								
Cd	18	1	0.4620	0.4940								
Cd	18	2	0.4082	0.4050								
Cd	18	3	0.5163	0.5320								
Cd	18	4	0.3690	0.3580								
Cd	18	5	0.6642	0.6992								
Cd	18	6	0.5390	0.5277								
Cd	18	7	0.4040	0.3690								
Cd	18	8	0.4775	0.4846								
Cu	5	1	0.0000	0.0000	0.1750	0.2460	0.4490	0.4420	1.7700	1.8000	17.9000	18.1000
Cu	5	2	0.1589	0.1326	0.4544	0.2860	1.0490	0.8788	2.2070	2.1410	19.9900	20.2300
Cu	5	3	0.0323	0.0481	0.3242	0.2824	0.6168	0.6020	2.1453	3.2563	19.4746	19.7283
Cu	5	4	0.0440	0.0260	2.0470	0.9950	1.1720	0.5570	2.1880	2.2190	21.0530	21.3540
Cu	5	5	0.5282	0.6445	0.6310	0.4641	0.5643	0.6304	1.9111	1.8558	18.6020	18.8151
Cu	5	6	0.1286	0.3061	0.3025	0.5329	0.5794	0.6422	2.2046	3.5277	18.5092	43.9433
Cu	5	7	0.0161	0.0160	0.1925	0.2383	0.4596	0.4643	1.9044	1.8799	18.5410	18.9762
Cu	5	8	0.0173	0.0180	0.1719	0.2100	0.4540	0.4798	1.7638	1.8114	18.1127	18.5166
Cu	16	1	0.9110	0.9870	1.4400	1.6900	2.4000	2.8800	4.3400	4.7200		
Cu	16	2	6.2910	1.4400	1.8150	1.8170	2.5870	3.2650	4.5170	5.1440		
Cu	16	3	1.0159	1.0749	1.5634	1.6089	2.2561	2.8573	3.9508	4.3348		
Cu	16	4	1.2270	1.2380	1.7160	1.8940	2.7240	3.4490	5.8480	5.2620		
Cu	16	5	1.1891	8.0865	1.7400	5.4500	2.5788	3.6508	4.3962	4.7973		
Cu	16	6	1.3090	1.1262	1.6680	1.8756	2.4713	2.9969	4.3591	4.5801		
Cu	16	7	1.0672	1.0314	1.4750	1.6631	2.3944	2.8477	4.1560	4.6702		
Cu	16	8	1.0520	1.0250	1.4739	1.6273	2.3142	2.8229	4.0287	4.5219		
Cu	17	1	14.0000	13.0000								
Cu	17	2	13.8600	13.1800								
Cu	17	3	14.6823	15.2322								

Raw Data

Element	Matrix	Lab	1	2	3	4	5	6	7	8	9	10
Cu	17	4	16.1150	17.7950								
Cu	17	5	(15.4151)	14.4274								
Cu	17	6	12.7543	13.7450								
Cu	17	7	14.6566	14.7489								
Cu	17	8	13.8010	13.6680								
Cu	18	1	14.8000	14.4000								
Cu	18	2	15.5200	15.2000								
Cu	18	3	17.6339	16.2906								
Cu	18	4	17.5450	20.1940								
Cu	18	5	18.9784	17.9623								
Cu	18	6	17.4718	14.4560								
Cu	18	7	15.1818	15.3126								
Cu	18	8	15.4382	15.5194								
Pb	5	1	0.0004	0.0035	0.0366	0.0271	0.0790	0.0832	0.4540	0.4510	4.2700	4.2600
Pb	5	2	0.1292	0.0541	0.1964	0.0852	0.2634	0.1817	0.6876	0.5714	5.1920	5.0520
Pb	5	3	0.0103	0.0145	0.0605	0.0519	0.1167	0.1194	0.5013	0.5341	4.9543	5.0154
Pb	5	4	0.0070	0.0040	0.0670	0.0390	0.1060	0.1030	0.5160	0.5170	5.1970	5.2670
Pb	5	5	0.0594	0.1421	0.1423	0.1253	0.1596	0.1953	0.4931	0.5157	4.7988	4.8011
Pb	5	6	0.0208	0.0291	0.0528	0.0777	0.1160	0.1135	0.5201	2.4625	5.0285	6.3171
Pb	5	7	0.0051	0.0052	0.0375	0.0366	0.0996	0.0974	0.4792	0.4807	4.8454	4.8580
Pb	5	8	-0.0016	0.0004	0.0239	0.0290	0.0943	0.0921	0.4677	0.4772	4.8273	4.9817
Pb	16	1	0.0160	0.0131	0.0487	0.1090	0.1570	0.2200	0.2690	0.3220		
Pb	16	2	0.0852	0.0770	0.1464	0.2292	0.3806	0.4883	0.3636	0.5197		
Pb	16	3	0.0437	0.0486	0.0835	0.1106	0.2403	0.2358	0.3460	0.3924		
Pb	16	4	0.0280	0.0310	0.0690	0.0820	0.2200	0.2220	0.3540	0.3860		
Pb	16	5	0.0630	0.1640	0.0958	0.0835	0.3552	0.2326	0.4236	0.3797		
Pb	16	6	0.0603	0.1225	0.0890	0.1112	0.2493	0.2500	0.3379	0.3967		
Pb	16	7	0.0280	0.0278	0.0678	0.0791	0.2166	0.2143	0.3156	0.3653		
Pb	16	8	0.0180	0.0211	0.0550	0.0670	0.2203	0.2217	0.2972	0.3550		
Pb	17	1	0.5310	0.4870								
Pb	17	2	0.6331	0.7302								

Element	Matrix	Lab	1	2	3	4	5	6	7	8	9	10
Pb	17	3	0.6978	0.6215								
Pb	17	4	0.5650	0.7330								
Pb	17	5	0.5874	0.5899								
Pb	17	6	0.6123	0.6286								
Pb	17	7	0.5339	0.5326								
Pb	17	8	0.6138	0.5709								
Pb	18	1	0.9680	0.9730								
Pb	18	2	1.1380	1.1410								
Pb	18	3	1.2198	1.1774								
Pb	18	4	1.0530	1.0840								
Pb	18	5	1.7818	2.7456								
Pb	18	6	1.1253	1.1737								
Pb	18	7	0.8502	0.8549								
Pb	18	8	1.1202	1.1145								
Ni	5	1	0.0000	0.0000	0.4200	0.5230	0.9190	0.8190	8.9500	8.8200	*87.1000*	*86.9000*
Ni	5	2	0.0303	0.0256	0.5463	0.6395	1.4040	0.9972	10.1900	10.6900	98.7800	99.2600
Ni	5	3	0.0069	0.0152	0.4731	0.5952	0.9903	0.9265	9.2682	9.3891	91.9907	94.0182
Ni	5	4	0.0010	0.0030	1.5430	0.6310	0.9670	0.9890	10.2880	9.9170	97.5750	97.7140
Ni	5	5	3.3073	-0.0714	0.4734	0.5540	0.8445	0.9866	9.0384	9.3564	*91.7846*	*92.9108*
Ni	5	6	0.0312	0.2107	0.4697	0.8110	0.8887	0.9097	8.9976	9.4099	87.9207	89.4755
Ni	5	7	0.0077	0.0025	0.4378	0.5348	0.9141	0.9261	9.3127	9.1843	90.3164	92.2033
Ni	5	8	0.0083	0.0055	0.4448	0.5525	0.9131	0.9435	9.1786	9.1672	(90.7248)	(94.5584)
Ni	16	1	0.4940	0.5130	1.7200	1.8800	3.2800	3.8600	5.1800	5.7500		
Ni	16	2	0.8529	0.7177	1.9840	2.2680	3.7520	4.4710	6.9970	6.7370		
Ni	16	3	0.6528	0.6711	1.6941	1.9425	3.1383	3.5813	6.1252	5.8139		
Ni	16	4	0.6570	0.6600	1.9240	2.1060	3.5990	4.1060	6.2400	6.1380		
Ni	16	5	0.9110	3.1600	2.0831	2.3118	3.5877	4.2850	6.9102	6.2186		
Ni	16	6	0.0000	0.0000	1.0149	1.1366	2.4122	3.0205	5.6038	4.6596		
Ni	16	7	0.4790	0.4592	1.6675	1.8115	3.1570	3.8903	5.7563	5.7324		
Ni	16	8	0.6062	0.6288	1.8047	1.9671	3.2257	3.9768	6.0458	5.8885		
Ni	17	1	7.1100	6.5200								

Raw Data

Element	Matrix	Lab	1	2	3	4	5	6	7	8	9	10
Ni	17	2	7.0590	7.3030								
Ni	17	3	6.6059	6.3867								
Ni	17	4	6.8580	7.1600								
Ni	17	5	6.9976	7.0286								
Ni	17	6	4.5314	5.1111								
Ni	17	7	6.9029	6.8971								
Ni	17	8	6.9385	6.7290								
Ni	18	1	7.5900	7.6000								
Ni	18	2	7.6590	7.8510								
Ni	18	3	7.0975	7.1137								
Ni	18	4	8.9260	8.7740								
Ni	18	5	9.0821	8.5336								
Ni	18	6	6.6759	5.3956								
Ni	18	7	7.3304	7.4195								
Ni	18	8	7.6763	7.7658								
Se	5	1	0.0000	0.0000	0.9430	1.0700	5.6400	5.3500	22.4000	22.3000	*107.0000*	*111.0000*
Se	5	2	0.4753	0.2495	0.9426	0.6453	4.5400	5.9650	18.7500	19.3300	95.3600	96.5400
Se	5	3	0.0862	-0.1122	1.1618	1.3055	5.0323	4.9782	20.9289	21.1048	113.2210	113.7347
Se	5	4	-0.0010	0.0100	0.8750	1.2170	5.0620	4.9660	21.1320	21.1440	106.7670	106.0850
Se	5	5	-0.0605	0.1500	1.6423	1.7892	4.5147	4.4402	17.8544	18.3850	*89.2289*	*91.3304*
Se	5	6	20.6772	20.3840	21.7354	20.3721	24.9791	24.8939	37.9875	39.8635	*110.3332*	*110.4997*
Se	5	7	0.0101	0.0261	0.8322	0.9676	4.3423	4.1599	18.1392	18.1058	89.5153	91.3544
Se	5	8	0.1219	0.1280	1.0135	1.2068	4.6020	4.9030	19.0698	18.7166	92.8644	97.3893
Se	16	1	0.2890	0.1760	0.9890	1.0400	1.4000	2.2900	3.7900	4.1900		
Se	16	2	0.1495	-0.2973	0.0069	1.4730	1.3650	1.2640	3.9550	3.5530		
Se	16	3	0.1444	0.1777	0.7959	1.1142	2.0142	2.2558	3.7678	3.9146		
Se	16	4	0.1020	0.1380	0.6450	0.9580	1.8240	2.1020	3.7410	3.7900		
Se	16	5	0.4310	0.0000	1.4480	1.4273	2.2192	2.6318	3.8992	3.9011		
Se	16	6	19.7109	19.0204	20.3957	20.7004	21.1349	20.6159	27.4137	23.3279		
Se	16	7	0.1985	0.2125	0.7142	0.8711	1.5490	2.0332	3.4203	3.5249		
Se	16	8	0.2646	0.2360	0.8178	1.0490	1.8828	2.1701	3.4754	3.6753		

Element	Matrix	Lab	1	2	3	4	5	6	7	8	9	10
Se	17	1	8.8700	8.6700								
Se	17	2	7.2890	7.5030								
Se	17	3	8.0103	8.1828								
Se	17	4	6.2770	6.3980								
Se	17	5	8.0251	7.4642								
Se	17	6	24.8955	27.6746								
Se	17	7	5.5799	5.4285								
Se	17	8	5.3906	5.7788								
Se	18	1	9.9700	11.0000								
Se	18	2	8.4000	9.1760								
Se	18	3	9.4909	9.7532								
Se	18	4	7.3640	7.6150								
Se	18	5	7.9873	7.6959								
Se	18	6	35.2663	26.0733								
Se	18	7	6.5611	6.5048								
Se	18	8	6.3760	6.4850								
Ag	5	1	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0370	0.0357	6.7900	6.5800
Ag	5	2	0.0020	-0.0009	0.0091	0.0038	0.0347	0.0139	0.3276	0.3034	9.3500	9.4880
Ag	5	3	0.0003	0.0010	0.0870	0.0908	0.1611	0.2192	0.9276	0.8484	10.0446	9.9293
Ag	5	4	0.0050	0.0050	0.0970	0.0840	0.1560	0.2280	0.9000	0.9400	9.9770	10.1060
Ag	5	5	0.0576	0.0540	0.0542	0.0515	0.0510	0.0510	0.0935	0.0713	8.8829	8.4280
Ag	5	6	0.0105	0.0078	0.0107	0.0082	0.0879	0.0697	0.3156	0.3295	10.4827	10.2625
Ag	5	7	0.0096	0.0038	0.0019	0.0040	-0.0015	-0.0005	0.0061	0.0027	8.5905	8.9060
Ag	5	8	0.0177	0.0126	0.0015	0.0012	0.0014	0.0000	0.0012	0.0960	7.7363	7.3608
Ag	16	1	0.0052	0.0000	0.0109	0.0221	0.0974	0.0934	0.0546	0.1100		
Ag	16	2	0.0052	0.0021	0.0139	0.0269	0.1545	0.1189	0.0289	0.0618		
Ag	16	3	0.0028	0.0015	0.0636	0.0640	0.1929	0.1901	0.2361	0.3016		
Ag	16	4	0.0420	0.0090	0.0800	0.0940	0.1910	0.2040	0.2930	0.3580		
Ag	16	5	0.0460	0.0450	0.0640	0.0671	0.1265	0.1113	0.0972	0.0880		
Ag	16	6	0.0115	0.0217	0.0189	0.0178	0.1373	0.1275	0.0637	0.0508		
Ag	16	7	0.0107	0.0080	0.0200	0.0261	0.0637	0.0646	0.0453	0.0396		

Element	Matrix	Lab	1	2	3	4	5	6	7	8	9	10
Ag	16	8	0.0022	0.0017	0.0142	0.0171	0.0826	0.0839	0.0380	0.0448		
Ag	17	1	0.3550	0.3270								
Ag	17	2	0.3787	0.3816								
Ag	17	3	0.4471	0.4172								
Ag	17	4	0.4720	0.5090								
Ag	17	5	0.3230	0.3182								
Ag	17	6	0.4657	0.4473								
Ag	17	7	0.3379	0.3065								
Ag	17	8	0.3964	0.2275								
Ag	18	1	1.2000	1.1400								
Ag	18	2	1.2830	1.3120								
Ag	18	3	1.3798	1.3991								
Ag	18	4	1.5160	1.4980								
Ag	18	5	1.6205	1.4659								
Ag	18	6	1.4265	1.4569								
Ag	18	7	1.2007	1.3045								
Ag	18	8	1.0881	1.2143								
Tl	5	1	0.0000	0.0000	0.0345	0.0457	0.0838	0.0888	0.4290	0.4330	1.7600	1.7300
Tl	5	2	0.0012	0.0002	0.0502	0.0500	0.0962	0.0999	0.5734	0.4985	2.0030	2.0140
Tl	5	3	0.0001	0.0002	0.0394	0.0489	0.1005	0.0968	0.4960	0.5049	2.0253	2.0039
Tl	5	4	0.0010	0.0010	0.0360	0.0400	0.0890	0.0830	0.4510	0.4500	1.8130	1.8310
Tl	5	5	-0.0050	-0.0056	0.0358	0.0387	0.0917	0.0877	0.4848	0.4820	2.0091	2.0679
Tl	5	6	0.0041	0.0039	0.0425	0.0609	0.1005	0.0966	0.4782	0.4851	1.9235	1.9254
Tl	5	7	0.0002	0.0001	0.0423	0.0500	0.1051	0.1042	0.5151	0.5243	2.1066	2.1338
Tl	5	8	0.0000	0.0000	0.0352	0.0429	0.0970	0.0995	0.4630	0.4665	1.8590	1.9596
Tl	16	1	0.0000	0.0000	0.0168	0.0175	0.0496	0.0544	0.0867	0.1050		
Tl	16	2	0.0086	0.0012	0.0229	0.0305	0.0670	0.0590	0.1041	0.1190		
Tl	16	3	0.0009	0.0009	0.0208	0.0243	0.0620	0.0600	0.1028	0.1211		
Tl	16	4	0.0000	0.0010	0.0180	0.0200	0.0520	0.0530	0.0880	0.1050		
Tl	16	5	0.0000	0.0000	0.0131	0.0155	0.0551	0.0537	0.0977	0.1202		
Tl	16	6	0.0116	0.0080	0.0266	0.0269	0.0680	0.0640	0.1056	0.1229		

Element	Matrix	Lab	1	2	3	4	5	6	7	8	9	10
Tl	16	7	0.0013	0.0008	0.0218	0.0245	0.0695	0.0673	0.1050	0.1298		
Tl	16	8	0.0006	0.0000	0.0179	0.0206	0.0611	0.0594	0.0911	0.1097		
Tl	17	1	0.1830	0.1720								
Tl	17	2	0.2092	0.1841								
Tl	17	3	0.2019	0.2119								
Tl	17	4	0.1730	0.3200								
Tl	17	5	0.1961	0.1948								
Tl	17	6	0.2119	0.2188								
Tl	17	7	0.2089	0.2067								
Tl	17	8	0.1887	0.1904								
Tl	18	1	0.7830	0.7750								
Tl	18	2	0.8521	0.9424								
Tl	18	3	0.9219	0.9505								
Tl	18	4	0.7610	0.7680								
Tl	18	5	1.0243	0.9540								
Tl	18	6	0.9264	0.9030								
Tl	18	7	0.9770	0.9665								
Tl	18	8	0.8704	0.8559								
Zn	5	1	0.0713	0.2800	0.4650	0.4740	1.8900	2.3600	4.4600	4.5800	43.2000	43.4000
Zn	5	2	0.6914	0.4415	4.0300	1.1550	3.0660	2.8680	6.0460	5.6360	51.0000	51.3700
Zn	5	3	0.0697	0.1315	0.7130	0.6415	2.1609	2.6430	4.9954	5.8975	52.5187	51.9073
Zn	5	4	-0.2020	-0.2190	0.2510	0.2380	3.2120	2.0620	4.9730	4.9380	50.4520	50.7220
Zn	5	5	1.7024	4.5007	2.1958	3.4642	3.7071	4.2327	5.7298	6.3932	*47.7843*	*48.1603*
Zn	5	6	0.2636	0.4230	0.7293	0.8695	2.1349	2.7856	4.8052	6.4784	45.8536	59.8960
Zn	5	7	-0.0374	-0.0397	0.4118	0.4133	1.7942	2.1073	4.6699	4.5940	46.6399	46.6981
Zn	5	8	1.1251	0.9461	0.6587	1.0679	1.6577	2.0357	4.4630	4.4245	44.7807	50.4607
Zn	16	1	0.8690	0.7900	0.9050	1.3700	1.2800	1.4000	1.6300	2.0000		
Zn	16	2	1.9980	1.3810	1.4150	1.8460	4.3210	2.3680	2.1890	2.5790		
Zn	16	3	1.0036	0.9580	0.9369	1.1145	1.5506	1.3816	2.0570	2.0747		
Zn	16	4	0.5140	0.4470	0.6370	0.9220	1.4320	1.1660	1.6130	1.8390		
Zn	16	5	19.9924	2.6400	2.2861	2.8336	3.5617	14.3135	3.3408	3.6014		

Raw Data

Element	Matrix	Lab	1	2	3	4	5	6	7	8	9	10
Zn	16	6	1.0932	1.6887	1.1470	1.4080	2.1583	1.5672	1.8281	2.2378		
Zn	16	7	0.7638	0.7708	0.8760	1.0735	1.3056	1.2636	1.4933	1.8992		
Zn	16	8	0.6552	0.8136	1.0597	1.1635	4.6312	3.8415	1.3807	1.7278		
Zn	17	1	45.8000	41.0000								
Zn	17	2	44.3000	42.7000								
Zn	17	3	42.7116	44.2444								
Zn	17	4	44.9860	45.2430								
Zn	17	5	60.3392	*40.1084*								
Zn	17	6	43.3113	47.8295								
Zn	17	7	42.9109	42.8596								
Zn	17	8	42.8472	45.3330								
Zn	18	1	48.5000	48.2000								
Zn	18	2	49.4400	50.5500								
Zn	18	3	49.6040	49.7807								
Zn	18	4	50.6250	52.3870								
Zn	18	5	*59.7132*	*53.4459*								
Zn	18	6	62.0914	48.8716								
Zn	18	7	45.5374	46.2510								
Zn	18	8	49.5120	48.7990								

Matrix Code
5 = RGW
16 = Freshwater
17 = Filtered Effluent
18 = Unfiltered Effluent

Table B-2
Raw SRM Data

Raw Data submitted to Sample Control Center (SCC) along with SCC comments on reason data were removed.

Element	Lab	True, µg/L	Measured, µg/L	Reason (SCC Notation)
Sb	1	54.1	58.5	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Sb	2	54.1	53.56	
Sb	2	54.1	56.47	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Sb	2	54.1	58.74	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Sb	2	54.1	54.1	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Sb	3	54.1	55.972	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Sb	4	54.1	52.98	Result not used for the final study report
Sb	4	54.1	50.316	
Sb	4	54.1	51.975	
Sb	4	54.1	51.946	
Sb	4	54.1	52.089	
Sb	4	54.1	52.754	Result not used for the final study report
Sb	4	54.1	53.376	Result not used for the final study report
Sb	4	54.1	51.434	
Sb	5	54.1	54.4204	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Sb	5	54.1	55.0184	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Sb	5	54.1	52.7	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Sb	5	54.1	55.6	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Sb	6	54.1	52.7413	
Sb	6	54.1	54.8678	
Sb	7	54.1	53.4482	
Sb	7	54.1	53.966	
Sb	7	54.1	52.8257	
Sb	8	54.1	57.75	
Cd	1	6.47	6.11	
Cd	2	6.47	6.416	

Element	Lab	True, µg/L	Measured, µg/L	Reason (SCC Notation)
Cd	2	6.47	5.741	
Cd	2	6.47	6.288	Result not used for the final study report
Cd	2	6.47	5.791	
Cd	2	6.47	6.246	
Cd	2	6.47	6.363	Result not used for the final study report
Cd	3	6.47	6.335	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Cd	4	6.47	6.224	Result not used for the final study report
Cd	4	6.47	6.059	
Cd	4	6.47	6.195	
Cd	4	6.47	6.123	
Cd	4	6.47	6.236	
Cd	4	6.47	6.22	Result not used for the final study report
Cd	4	6.47	6.298	Result not used for the final study report
Cd	4	6.47	6.148	
Cd	5	6.47	6.1624	
Cd	5	6.47	6.1664	
Cd	5	6.47	5.89	
Cd	5	6.47	6.21	
Cd	6	6.47	6.3598	
Cd	6	6.47	6.413	
Cd	7	6.47	5.9226	
Cd	7	6.47	5.8877	
Cd	7	6.47	5.9414	
Cd	8	6.47	5.9093	
Cu	1	20.5	20.9	
Cu	2	20.5	20.35	
Cu	2	20.5	23.86	
Cu	2	20.5	21.5	
Cu	3	20.5	21.164	
Cu	4	20.5	20.96	Result not used for the final study report

Element	Lab	True, µg/L	Measured, µg/L	Reason (SCC Notation)
Cu	4	20.5	22.611	
Cu	4	20.5	22.986	
Cu	4	20.5	23.072	
Cu	4	20.5	23.219	
Cu	4	20.5	20.926	Result not used for the final study report
Cu	4	20.5	21.369	Result not used for the final study report
Cu	4	20.5	22.514	
Cu	5	20.5	20.3218	
Cu	5	20.5	20.3218	
Cu	5	20.5	18.5	
Cu	5	20.5	19.9	
Cu	6	20.5	20.2877	
Cu	6	20.5	19.4861	
Cu	7	20.5	19.5542	
Cu	7	20.5	18.9842	
Cu	7	20.5	18.3279	
Cu	8	20.5	18.9054	
Pb	1	18.15	18.4	
Pb	2	18.15	11.62	
Pb	2	18.15	14.07	
Pb	2	18.15	13.22	Result not used for the final study report
Pb	3	18.15	18.537	
Pb	4	18.15	16.45	Result not used for the final study report
Pb	4	18.15	18.329	
Pb	4	18.15	17.841	
Pb	4	18.15	17.47	
Pb	4	18.15	17.473	
Pb	4	18.15	16.714	Result not used for the final study report
Pb	4	18.15	16.858	Result not used for the final study report
Pb	4	18.15	17.923	

Element	Lab	True, µg/L	Measured, µg/L	Reason (SCC Notation)
Pb	5	18.15	16.446	
Pb	5	18.15	16.5942	
Pb	5	18.15	14.8	
Pb	5	18.15	14.8	
Pb	6	18.15	18.84661	Result not used for the final study report
Pb	6	18.15	19.9694	Result not used for the final study report
Pb	6	18.15	19.4343	Result not used for the final study report
Pb	6	18.15	19.9694	Result not used for the final study report
Pb	6	18.15	19.52037	Result not used for the final study report
Pb	6	18.15	20.61012	Result not used for the final study report
Pb	6	18.15	19.0657	
Pb	6	18.15	20.0256	
Pb	7	18.15	0.9248	
Pb	7	18.15	0.9217	
Pb	7	18.15	0.9199	
Pb	8	18.15	17.671	
Ni	1	58.1	57.9	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Ni	2	58.1	56.39	
Ni	2	58.1	59.76	
Ni	2	58.1	52.89	
Ni	2	58.1	60.07	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Ni	2	58.1	63.35	Result not used for the final study report
Ni	3	58.1	53.394	
Ni	4	58.1	53.992	Result not used for the final study report
Ni	4	58.1	55.674	
Ni	4	58.1	55.209	
Ni	4	58.1	55.925	
Ni	4	58.1	56.626	
Ni	4	58.1	54.007	Result not used for the final study report
Ni	4	58.1	54.235	Result not used for the final study report

Element	Lab	True, µg/L	Measured, µg/L	Reason (SCC Notation)
Ni	4	58.1	55.599	
Ni	5	58.1	54.5246	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Ni	5	58.1	55.8504	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Ni	5	58.1	51.4	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Ni	5	58.1	52.8	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Ni	6	58.1	57.2109	
Ni	6	58.1	54.2362	
Ni	7	58.1	55.7067	
Ni	7	58.1	54.2869	
Ni	7	58.1	52.3275	
Ni	8	58.1	55.2891	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Se	1	11.43	12.7	
Se	2	11.43	6.311	
Se	2	11.43	10.98	
Se	2	11.43	11.81	
Se	2	11.43	11.64	Result not used for the final study report
Se	3	11.43	11.562	
Se	4	11.43	12.079	Result not used for the final study report
Se	4	11.43	12.102	
Se	4	11.43	12.387	
Se	4	11.43	12.014	
Se	4	11.43	12.439	
Se	4	11.43	12.186	Result not used for the final study report
Se	4	11.43	11.983	Result not used for the final study report
Se	4	11.43	12.055	
Se	5	11.43	9.6472	
Se	5	11.43	11.2972	
Se	5	11.43	9.4	
Se	5	11.43	9.86	
Se	6	11.43	13.272	

Element	Lab	True, µg/L	Measured, µg/L	Reason (SCC Notation)
Se	6	11.43	11.353	
Se	7	11.43	9.7063	
Se	7	11.43	9.4371	
Se	7	11.43	9.1685	
Se	8	11.43	10.1865	
Ag	1	1.27	1.05	
Ag	2	1.27	1.17	
Ag	2	1.27	1.182	
Ag	2	1.27	1.295	
Ag	2	1.27	1.184	
Ag	2	1.27	1.222	
Ag	2	1.27	1.261	
Ag	3	1.27	1.193	
Ag	4	1.27	1.309	
Ag	4	1.27	1.311	
Ag	4	1.27	1.301	
Ag	4	1.27	1.329	
Ag	4	1.27	1.335	
Ag	4	1.27	1.292	
Ag	4	1.27	1.319	
Ag	4	1.27	1.302	
Ag	5	1.27	1.3044	
Ag	5	1.27	1.2902	
Ag	5	1.27	1.2	
Ag	5	1.27	1.23	
Ag	6	1.27	1.4157	
Ag	6	1.27	1.3449	
Ag	7	1.27	1.2611	
Ag	7	1.27	1.2607	
Ag	7	1.27	1.2463	

Element	Lab	True, µg/L	Measured, µg/L	Reason (SCC Notation)
Ag	8	1.27	1.273	
Tl	1	7.28	7.47	
Tl	2	7.28	6.449	
Tl	2	7.28	4.956	
Tl	2	7.28	5.863	
Tl	2	7.28	5.856	
Tl	2	7.28	5.791	
Tl	2	7.28	6.767	Result not used for the final study report
Tl	3	7.28	7.3	
Tl	4	7.28	6.766	Result not used for the final study report
Tl	4	7.28	6.77	
Tl	4	7.28	6.683	
Tl	4	7.28	6.442	
Tl	4	7.28	6.503	
Tl	4	7.28	6.937	Result not used for the final study report
Tl	4	7.28	6.994	Result not used for the final study report
Tl	4	7.28	6.575	
Tl	5	7.28	7.1402	
Tl	5	7.28	7.0934	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Tl	5	7.28	6.48	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Tl	5	7.28	6.46	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Tl	6	7.28	7.5666	
Tl	6	7.28	7.8181	
Tl	7	7.28	7.9586	
Tl	7	7.28	8.0726	
Tl	7	7.28	8.1705	
Tl	8	7.28	7.1355	
Zn	1	72.48	71.1	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Zn	2	72.48	67.01	
Zn	2	72.48	67.23	

Element	Lab	True, µg/L	Measured, µg/L	Reason (SCC Notation)
Zn	2	72.48	84.64	Result not used for the final study report
Zn	2	72.48	69.51	
Zn	2	72.48	70.96	
Zn	2	72.48	78.31	Result not used for the final study report
Zn	3	72.48	78.48	
Zn	4	72.48	71.23	Result not used for the final study report
Zn	4	72.48	71.747	
Zn	4	72.48	72.471	
Zn	4	72.48	72.091	
Zn	4	72.48	73.36	
Zn	4	72.48	71.591	Result not used for the final study report
Zn	4	72.48	72.445	Result not used for the final study report
Zn	4	72.48	70.789	
Zn	5	72.48	66.7382	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Zn	5	72.48	67.5834	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Zn	5	72.48	57.3	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Zn	5	72.48	66.1	ANALYTE DETECTED ABOVE UPPER CALIBRATION LIMIT
Zn	6	72.48	67.6902	
Zn	6	72.48	70.9326	
Zn	7	72.48	61.4301	
Zn	7	72.48	59.9528	
Zn	7	72.48	58.124	
Zn	8	72.48	68.2588	

C
STATCALC INPUT/OUTPUT

For each Method 1638 study analyte in reagent grade water and freshwater, the following tables are provided:

- Data File (.DAT) - Displays original raw data submitted by each laboratory.
- Data Validation File (.DA~) - Audit record documenting the fate of each original data point. Includes information on values removed by lab ranking and outlier testing.
- Final Data File (.DAF) - Final data set submitted for statistical processing after removal of outliers.
- Data Preparation File (.PRP) - Displays the results of the factor of 5 screening, laboratory ranking, individual outlier removal, and normality testing.
- Summary Statistics (.STT) - Contains recovery, single operator standard deviation, and overall standard deviation results at each concentration; results of bias testing; linear and curvilinear equations for single operator and overall standard deviation; linear regression equations for recovery; and linear regression equations for both single operator and overall standard deviation versus recovery (obtained by substitution).

The effluent samples consisted of a single pair of duplicate samples for each matrix (filtered effluent and unfiltered effluent). Only limited data processing was possible with these small data sets.

The Data Validation and Final Data files have a flag field to the left of each value which is used to provide information about the value. The following table lists the flags and their meanings.

Flag	Description	File
r	Value removed by lab ranking test	.DA~, .DAF
o	Value removed by individual outlier removal test	.DA~, .DAF
!	Value flagged for removal by lab ranking test but retained. Removal would have exceeded 20% cap on data removal by lab ranking.	.DA~
?	Value flagged for removal by individual outlier test but retained. Removal would have exceeded 10% cap on data removed by outlier testing.	.DA~
e	Temporary fill value for missing value. Calculated by linear regression through remaining data submitted by laboratory. Used for lab ranking test and then deleted.	.DA~
*	No value submitted	.DAF

sb-5.dat

```
Sb     5   1     0.0000    0.0000    0.0338    0.0957    0.6190    0.6800    3.7500    3.8800   17.1000   16.9000
Sb     5   2    -0.0041   -0.0158    0.0461    0.0518    0.6302    0.6413    4.2280    4.2610   18.9000   18.4100
Sb     5   3     0.0027    0.0017    0.0426    0.1099    0.6640    0.7064    3.9114    3.9958   16.1164   16.3849
Sb     5   4     0.0260    0.0240    0.0500    0.0620    0.6990    0.7120    4.1680    4.1230   16.5480   16.6010
Sb     5   5    -0.0132   -0.0108    0.0691    0.0424    0.6278    0.6425    4.1322    4.1184
Sb     5   6     0.0115    0.0131    0.0971    0.1484    0.7091    0.6579    4.4013    4.5073   19.0607   19.0956
Sb     5   7    -0.0017   -0.0006    0.0564    0.1016    0.6017    0.5732    3.7466    3.7478   18.0506   18.1348
Sb     5   8     0.0409    0.0109    0.0662    0.0992    0.6547    0.6252    3.8980    3.8641   18.4290   18.7830
```

```
File Name:  sb-5
Data Validation File (.DA~)

*****************************************************************************
***                  Parameter and Data Validation File                   ***
*****************************************************************************
   Analyte:  Sb              Matrix:  DI Water (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:   5
      Date:  06/28/2000       Method:  1638
     Pairs:  5
     Units:  ug/L
```

Level	1	2	3	4	5	6	7	8	9	10
Spike	.0001	.0000	.1000	.0200	.8800	.0000	4.0000	.0000	15.0000	.0000
Spike Increment	.0000	.0000	.1000	.1200	1.0000	1.0000	5.0000	5.0000	20.0000	20.0000

	Final Concentration									
Lab ID	.0001	.0001	.1001	.1201	1.0001	1.0001	5.0001	5.0001	20.0001	20.0001
1	.0000	.0000	.0338	.0957	.6190	.6800	3.7500	3.8800	17.1000	16.9000
2	-.0041	-.0158	.0461	.0518	.6302	.6413	4.2280	4.2610	18.9000	18.4100
3	.0027	.0017	.0426	.1099	.6640	.7064	3.9114	3.9958	16.1164	16.3849
4	.0260	.0240	.0500	.0620	.6990	.7120	4.1680	4.1230	16.5480	16.6010
5	-.0132	-.0108	.0691	.0424	.6278	.6425	4.1322	4.1184 e	16.6279e	16.6279
6 r	.0115r	.0131 r	.0971r	.1484 r	.7091r	.6579 r	4.4013r	4.5073 r	19.0607r	19.0956
7	-.0017	-.0006	.0564	.1016	.6017	.5732	3.7466	3.7478	18.0506	18.1348
8	.0409	.0109	.0662	.0992	.6547	.6252	3.8980	3.8641	18.4290	18.7830

Sb-5.daf

```
Sb     5   1      .0000     .0000     .0338     .0957     .6190     .6800    3.7500    3.8800   17.1000   16.9000
Sb     5   2     -.0041    -.0158     .0461     .0518     .6302     .6413    4.2280    4.2610   18.9000   18.4100
Sb     5   3      .0027     .0017     .0426     .1099     .6640     .7064    3.9114    3.9958   16.1164   16.3849
Sb     5   4      .0260     .0240     .0500     .0620     .6990     .7120    4.1680    4.1230   16.5480   16.6010
Sb     5   5     -.0132    -.0108     .0691     .0424     .6278     .6425    4.1322    4.1184*         *
Sb     5   7     -.0017    -.0006     .0564     .1016     .6017     .5732    3.7466    3.7478   18.0506   18.1348
Sb     5   8      .0409     .0109     .0662     .0992     .6547     .6252    3.8980    3.8641   18.4290   18.7830
```

```
File Name:  sb-5
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  sb-5
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***-------------------------------------***
 ***     Upper Critical Value:     65.0   ***
 ***     Lower Critical Value:     25.0   ***
-----------------------------------------------

      Ranks
     ------------------------------------------------------------------------------
      Rank  Level  Level  Level  Level  Level  Level  Level  Level  Level  Level
Lab    Sums    1      2      3      4      5      6      7      8      9     10
---- ------------------------------------------------------------------------------
  1    34.0   4.00   4.00   1.00   4.00   2.00   6.00   2.00   3.00   4.00   4.00
  2    42.0   2.00   1.00   3.00   2.00   4.00   3.00   7.00   7.00   7.00   6.00
  3    42.0   5.00   5.00   2.00   7.00   6.00   7.00   4.00   4.00   1.00   1.00
  4    53.0   7.00   8.00   4.00   3.00   7.00   8.00   6.00   6.00   2.00   2.00
  5    34.0   1.00   2.00   7.00   1.00   3.00   4.00   5.00   5.00   3.00   3.00
  6    74.0   6.00   7.00   8.00   8.00   8.00   5.00   8.00   8.00   8.00   8.00
  7    31.0   3.00   3.00   5.00   6.00   1.00   1.00   1.00   1.00   5.00   5.00
  8    50.0   8.00   6.00   6.00   5.00   5.00   2.00   3.00   2.00   6.00   7.00
-------------------------------------------------
 *** Laboratory   6 Rejected; Rank Sum   74.0 ***
-------------------------------------------------

File Name:  sb-5
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
-----------------------------------------


-----------------------------------------
 - No Outlier Rejections in Iteration 1 -
 - No Further Outlier Testing Necessary -
-----------------------------------------
```

File Name: sb-5
Data Preparation File (.PRP)

Analyte: Sb Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Results of Normality Testing ***

- Normality Tests -

Level	n	Test Type	Normality Accept/Reject	Test Statistic	Critical Value(s)	
1	7	W	A	.8647	.803	---
2	7	W	A	.9559	.803	---
3	7	W	A	.9634	.803	---
4	7	W	A	.8632	.803	---
5	7	W	A	.9538	.803	---
6	7	W	A	.9409	.803	---
7	7	W	A	.8847	.803	---
8	7	W	A	.9621	.803	---
9	6	W	A	.9446	.788	---
10	6	W	A	.8817	.788	---

- 0 Normality Rejection(s) -

File Name: sb-5
Data Preparation File (.PRP)

Analyte: Sb Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Data Removal Tracking ***
*** Simple Count of Remaining Data Points ***
*** After Removal Tests ***

Lev	Mtrx	Points As Received	After Lab Ranking Points	After Lab Ranking %	After Outlier Testing Points	After Outlier Testing %
1	5	8	7	87.5	7	87.5
2	5	8	7	87.5	7	87.5
3	5	8	7	87.5	7	87.5
4	5	8	7	87.5	7	87.5
5	5	8	7	87.5	7	87.5
6	5	8	7	87.5	7	87.5
7	5	8	7	87.5	7	87.5
8	5	8	7	87.5	7	87.5
9	5	7	6	85.7	6	85.7
10	5	7	6	85.7	6	85.7
Totals:		78	68	87.2	68	87.2

File Name: sb-5
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Sb Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8	9	10
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)	Pair 5 (Low)	Pair 5 (High)
CONCENTRATION:	.0001	.0001	.1001	.1201	1.0001	1.0001	5.0001	5.0001	20.0001	20.0001
RECOVERY:										
Observations	7	7	7	7	7	7	7	7	6	6
Mean Result	.0072	.0013	.0520	.0804	.6423	.6544	3.9763	3.9986	17.5240	17.5356
Bias	.0071	.0012	-.0481	-.0397	-.3578	-.3457	-1.0238	-1.0015	-2.4761	-2.4645
Relative Bias %	7128.5710	1242.8570	-48.0234	-33.0796	-35.7721	-34.5694	-20.4753	-20.0299	-12.3804	-12.3224
Maximum Result	.0409	.0240	.0691	.1099	.6990	.7120	4.2280	4.2610	18.9000	18.7830
Minimum Result	-.0132	-.0158	.0338	.0424	.6017	.5732	3.7466	3.7478	16.1164	16.3849

SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4	Pair 5
Observations	7	7	7	7	6
Standard Deviation	.0082	.0237	.0236	.0456	.2206
Correction Factor	1.0424	1.0424	1.0424	1.0424	1.0509
Corrected Std Dev	.0085	.0247	.0246	.0476	.2318
Relative Std Dev (%)	199.3734	37.3689	3.7979	1.1933	1.3225

OVERALL PRECISION:										
Observations	7	7	7	7	7	7	7	7	6	6
Standard Deviation	.0191	.0132	.0127	.0274	.0326	.0491	.1995	.1797	1.1049	1.0277
Correction Factor	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0509	1.0509
Corrected Std Dev	.0199	.0138	.0133	.0286	.0340	.0511	.2079	.1873	1.1611	1.0801
Relative Std Dev %	275.1312	1027.6810	25.4970	35.5481	5.2944	7.8136	5.2289	4.6843	6.6260	6.1594

File Name: sb-5
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Sb Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.0001	.0072	.0071	******	.990	3.707	NO
2	.0001	.0013	.0012	******	.248	3.707	NO
3	.1001	.0520	-.0481	-48.02	9.994	3.707	YES
4	.1201	.0804	-.0397	-33.08	3.835	3.707	YES
5	1.0001	.6423	-.3578	-35.77	29.011	3.707	YES
6	1.0001	.6544	-.3457	-34.57	18.648	3.707	YES
7	5.0001	3.9763	-1.0238	-20.48	13.580	3.707	YES
8	5.0001	3.9986	-1.0015	-20.03	14.746	3.707	YES
9	20.0001	17.5240	-2.4761	-12.38	5.490	4.032	YES
10	20.0001	17.5356	-2.4645	-12.32	5.874	4.032	YES

File Name: sb-5
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Single Operator Precision ***

Analyte: Sb Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Single Operator Precision ***

- Linear Model - s = b*T + a

Intercept (a): .0152
Slope (b): .0093

Pair	Sample Size	Weights (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	7	47.71	.0001	.0085	.0152
2	7	38.80	.1101	.0247	.0162
3	7	12.05	1.0001	.0246	.0245
4	7	1.35	5.0001	.0476	.0616
5	6	.09	20.0001	.2318	.2009

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a: .0178 Intercept (a'): -4.0298
b: 1.1432 Slope (b'): .1338

Pair	Sample Size	Weight (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	7	20.71	.0001	.0085	.0178
2	7	20.71	.0001	.0247	.0180
3	7	20.71	.1001	.0246	.0203
4	7	20.71	.1201	.0476	.0347
5	6	17.15	1.0001	.2318	.2585

File Name: sb-5
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Overall Precision ***

Analyte: Sb Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Overall Precision ***

- Linear Model - s = b*T + a

Intercept (a): .0145
Slope (b): .0358

Conc Level	Sample Size	Weights (%)	Conc	Overall Std Dev	Estimated Std Dev
1	7	18.09	.0001	.0199	.0146
2	7	18.09	.0001	.0138	.0146
3	7	17.38	.1001	.0133	.0181
4	7	17.24	.1201	.0286	.0189
5	7	12.14	1.0001	.0340	.0504
6	7	12.14	1.0001	.0511	.0504
7	7	2.46	5.0001	.2079	.1936
8	7	2.46	5.0001	.1873	.1936
9	6	.01	20.0001	1.1611	.7309
10	6	.01	20.0001	1.0801	.7309

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a: .0280 Intercept (a'): -3.5762
b: 1.2207 Slope (b'): .1994

Conc Level	Sample Size	Weight (%)	Conc	Overall Std Dev	Estimated Std Dev
1	7	10.36	.0001	.0199	.0280
2	7	10.36	.0001	.0138	.0280
3	7	10.36	.1001	.0133	.0285
4	7	10.36	.1201	.0286	.0287
5	7	10.36	1.0001	.0340	.0342
6	7	10.36	1.0001	.0511	.0342
7	7	10.36	5.0001	.2079	.0758
8	7	10.36	5.0001	.1873	.0758
9	6	8.57	20.0001	1.1611	1.5094
10	6	8.57	20.0001	1.0801	1.5094

```
File Name:  sb-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
---------------------------------------
Analyte:  Sb          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):     -.0122
          Slope (b):      .7950
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       7     29.25       .0001       .0072      -.0121
      2       7     29.25       .0001       .0013      -.0121
      3       7     18.84       .1001       .0520       .0674
      4       7     17.43       .1201       .0804       .0833
      5       7      2.44      1.0001       .6423       .7830
      6       7      2.44      1.0001       .6544       .7830
      7       7       .17      5.0001      3.9763      3.9632
      8       7       .17      5.0001      3.9986      3.9632
      9       6       .01     20.0001     17.5240     15.8889
     10       6       .01     20.0001     17.5356     15.8889
    ------------------------------------------------------------


File Name:  sb-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
---------------------------------------
Analyte:  Sb          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .0305
            Slope (f):      .0117
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .0178
                  (f):     1.1833
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .0299
            Slope (f):      .0451
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .0281
                  (f):     1.2851
     ==================================
```

```
Sb-16.dat
Sb    16   1     0.1240    0.1190    0.2000    0.2260    0.3720    0.4660    0.7460    0.9420
Sb    16   2     0.1588    0.1116    0.2013    0.2331    0.3645    0.4666    0.7920    0.9939
Sb    16   3     0.1300    0.1246    0.2053    0.2454    0.3884    0.4765    0.7639    0.9755
Sb    16   4     0.1410    0.1380    0.2070    0.2460    0.3880    0.4820    0.7440    0.9060
Sb    16   5     0.1102    0.1050    0.1984    0.2290    0.3728    0.4789    0.7775    0.9871
Sb    16   6     0.2055    0.1388    0.2307    0.2563    0.4046    0.4759    0.7725    0.9653
Sb    16   7     0.1419    0.1326    0.2136    0.2490    0.3957    0.4948    0.7850    0.9860
Sb    16   8     0.1514    0.1352    0.2939    0.2723    0.4069    0.4886    0.7861    0.9654


File Name:  sb-16
Data Validation File (.DA~)

*****************************************************************************
***                  Parameter and Data Validation File                   ***
*****************************************************************************
   Analyte:  Sb              Matrix:  Freshwater (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:  16
      Date:  06/28/2000      Method:  1638
     Pairs:  4
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------
Level          1          2          3          4          5          6          7          8
-------------------------------------------------------------------------------------------------------------
Spike          .1328      .0000      .0800      .0400      .1600      .1000      .3000      .2000
Spike
Increment      .0000      .0000      .0800      .1200      .2800      .3800      .6800      .8800
-------------------------------------------------------------------------------------------------------------

                Final Concentration
          ----------------------------------------------------------------------------------------------
Lab ID         .1328      .1328      .2128      .2528      .4128      .5128      .8128     1.0128
-------------------------------------------------------------------------------------------------------------
    1    r     .1240r     .1190  r   .2000r     .2260  r   .3720r     .4660  r   .7460r     .9420
    2          .1588      .1116      .2013      .2331      .3645      .4666      .7920      .9939
    3          .1300      .1246      .2053      .2454      .3884      .4765      .7639      .9755
    4          .1410      .1380      .2070      .2460      .3880      .4820      .7440o     .9060
    5          .1102      .1050      .1984      .2290      .3728      .4789      .7775      .9871
    6          .2055      .1388      .2307      .2563      .4046      .4759      .7725      .9653
    7          .1419      .1326      .2136      .2490      .3957      .4948      .7850      .9860
    8          .1514      .1352  o   .2939      .2723      .4069      .4886      .7861      .9654
-------------------------------------------------------------------------------------------------------------


Sb-16.daf
Sb    16   2      .1588     .1116     .2013     .2331     .3645     .4666     .7920     .9939
Sb    16   3      .1300     .1246     .2053     .2454     .3884     .4765     .7639     .9755
Sb    16   4      .1410     .1380     .2070     .2460     .3880     .4820     .7440o
Sb    16   5      .1102     .1050     .1984     .2290     .3728     .4789     .7775     .9871
Sb    16   6      .2055     .1388     .2307     .2563     .4046     .4759     .7725     .9653
Sb    16   7      .1419     .1326     .2136     .2490     .3957     .4948     .7850     .9860
Sb    16   8      .1514     .1352o              .2723     .4069     .4886     .7861     .9654
```

```
File Name:  sb-16
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  sb-16
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***-------------------------------------***
 ***     Upper Critical Value:     54.0   ***
 ***     Lower Critical Value:     18.0   ***
-----------------------------------------------

      Ranks
     ------------------------------------------------------------------
       Rank  Level  Level  Level  Level  Level  Level  Level  Level
Lab    Sums    1      2      3      4      5      6      7      8
---- ------------------------------------------------------------------
  1    15.0   2.00   3.00   2.00   1.00   2.00   1.00   2.00   2.00
  2    34.0   7.00   2.00   3.00   3.00   1.00   2.00   8.00   8.00
  3    32.0   3.00   4.00   4.00   4.00   5.00   4.00   3.00   5.00
  4    33.0   4.00   7.00   5.00   5.00   4.00   6.00   1.00   1.00
  5    25.0   1.00   1.00   1.00   2.00   3.00   5.00   5.00   7.00
  6    47.0   8.00   8.00   7.00   7.00   7.00   3.00   4.00   3.00
  7    48.0   5.00   5.00   6.00   6.00   6.00   8.00   6.00   6.00
  8    54.0   6.00   6.00   8.00   8.00   8.00   7.00   7.00   4.00
-------------------------------------------------
 *** Laboratory   1 Rejected; Rank Sum   15.0 ***
-------------------------------------------------

File Name:  sb-16
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
-------------------------------------------------------------------------------
  3     1     8   1       .2939      .2215      .0337   2.151   2.020    7
  8     1     4   1       .9060      .9685      .0296   2.107   2.020    7
-------------------------------------------------------------------------------
```

```
File Name:  sb-16
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
              Test  Normality      Test          Critical
   Level  n   Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     7     W     A               .9184      .803   ---
     2     7     W     A               .8610      .803   ---
     3     6     W     A               .8729      .788   ---
     4     7     W     A               .9530      .803   ---
     5     7     W     A               .9373      .803   ---
     6     7     W     A               .9751      .803   ---
     7     7     W     A               .9177      .803   ---
     8     6     W     A               .8943      .788   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  sb-16
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    16        8            7    87.5           7       87.5
  2    16        8            7    87.5           7       87.5
  3    16        8            7    87.5           6       75.0
  4    16        8            7    87.5           7       87.5
  5    16        8            7    87.5           7       87.5
  6    16        8            7    87.5           7       87.5
  7    16        8            7    87.5           7       87.5
  8    16        8            7    87.5           6       75.0
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         64           56    87.5          54       84.4
-----------------------------------------------------------------
```

File Name: sb-16
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Sb Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)
CONCENTRATION:	.1328	.1328	.2128	.2528	.4128	.5128	.8128	1.0128
RECOVERY:								
Observations	7	7	6	7	7	7	7	6
Mean Result	.1484	.1265	.2094	.2473	.3887	.4805	.7744	.9789
Bias	.0156	-.0063	-.0034	-.0055	-.0241	-.0323	-.0384	-.0339
Relative Bias %	11.7470	-4.7117	-1.6056	-2.1756	-5.8382	-6.3043	-4.7209	-3.3504
Maximum Result	.2055	.1388	.2307	.2723	.4069	.4948	.7920	.9939
Minimum Result	.1102	.1050	.1984	.2290	.3645	.4666	.7440	.9653
SINGLE OPERATOR PRECISION:	Pair 1		Pair 2		Pair 3		Pair 4	
Observations	7		6		7		6	
Standard Deviation	.0177		.0039		.0087		.0084	
Correction Factor	1.0424		1.0509		1.0424		1.0509	
Corrected Std Dev	.0184		.0041		.0091		.0089	
Relative Std Dev (%)	13.3979		1.7757		2.0825		1.0187	
OVERALL PRECISION:								
Observations	7	7	6	7	7	7	7	6
Standard Deviation	.0296	.0134	.0117	.0144	.0157	.0092	.0164	.0120
Correction Factor	1.0424	1.0424	1.0509	1.0424	1.0424	1.0424	1.0424	1.0509
Corrected Std Dev	.0309	.0140	.0123	.0150	.0163	.0096	.0171	.0126
Relative Std Dev %	20.8213	11.0732	5.8554	6.0855	4.2004	1.9905	2.0224	1.2889

File Name: sb-16
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Sb Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.1328	.1484	.0156	11.75	1.392	3.707	NO
2	.1328	.1265	-.0063	-4.71	1.231	3.707	NO
3	.2128	.2094	-.0034	-1.61	.717	4.032	NO
4	.2528	.2473	-.0055	-2.18	1.008	3.707	NO
5	.4128	.3887	-.0241	-5.84	4.071	3.707	YES
6	.5128	.4805	-.0323	-6.30	9.322	3.707	YES
7	.8128	.7744	-.0384	-4.72	6.204	3.707	YES
8	1.0128	.9789	-.0339	-3.35	6.924	4.032	YES

```
File Name:  sb-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***   Single Operator Precision    ***
----------------------------------------
Analyte:  Sb          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .0131
          Slope (b):     -.0064
    ============================
                                          Single
           Sample Weights                Operator   Estimated
    Pair   Size       (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       7     27.35       .1328       .0184       .0123
      2       6     22.65       .2328       .0041       .0117
      3       7     27.35       .4628       .0091       .0102
      4       6     22.65       .9128       .0089       .0073
    ------------------------------------------------------------



    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ============================================================
      a:      .0103      Intercept (a'):    -4.5755
      b:      .7516           Slope (b'):     -.2856
    ============================================================
                                                 Single
           Sample Weight                       Operator   Estimated
    Pair   Size      (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       7    27.35       .1328       .0184       .0099
      2       6    22.65       .1328       .0041       .0096
      3       7    27.35       .2128       .0091       .0090
      4       6    22.65       .2528       .0089       .0079
    ------------------------------------------------------------
```

File Name: sb-16
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Overall Precision ***

Analyte: Sb Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Overall Precision ***

- Linear Model - s = b*T + a

Intercept (a): .0191
Slope (b): -.0070

Conc Level	Sample Size	Weights (%)	Conc	Overall Std Dev	Estimated Std Dev
1	7	13.06	.1328	.0309	.0182
2	7	13.06	.1328	.0140	.0182
3	6	10.82	.2128	.0123	.0176
4	7	13.06	.2528	.0150	.0174
5	7	13.06	.4128	.0163	.0162
6	7	13.06	.5128	.0096	.0155
7	7	13.06	.8128	.0171	.0134
8	6	10.82	1.0128	.0126	.0120

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a: .0176 Intercept (a'): -4.0401
b: .7121 Slope (b'): -.3396

Conc Level	Sample Size	Weight (%)	Conc	Overall Std Dev	Estimated Std Dev
1	7	13.06	.1328	.0309	.0168
2	7	13.06	.1328	.0140	.0168
3	6	10.82	.2128	.0123	.0164
4	7	13.06	.2528	.0150	.0161
5	7	13.06	.4128	.0163	.0153
6	7	13.06	.5128	.0096	.0148
7	7	13.06	.8128	.0171	.0134
8	6	10.82	1.0128	.0126	.0125

File Name: sb-16
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
********** Recovery **********

Analyte: Sb Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Recovery ***

- Linear Model - X = b*T + a

Intercept (a): .0058
Slope (b): .9492

Conc Level	Sample Size	Weights (%)	Conc	Mean Result	Estimated Result
1	7	12.96	.1328	.1484	.1319
2	7	12.96	.1328	.1265	.1319
3	6	11.11	.2128	.2094	.2078
4	7	12.96	.2528	.2473	.2458
5	7	12.96	.4128	.3887	.3977
6	7	12.96	.5128	.4805	.4926
7	7	12.96	.8128	.7744	.7773
8	6	11.11	1.0128	.9789	.9672

```
File Name:  sb-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
----------------------------------------
Analyte:  Sb          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

    - Linear Model - s = f*X + e
   ==============================
       Intercept (e):      .0070
           Slope (f):     -.0067
   ==============================


    - Curvilinear Model - s = e*(f**X)
   ====================================
       Intercept (e):      .0103
                 (f):      .7402
   ====================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

    - Linear Model - s = f*X + e
    ============================
       Intercept (e):      .0130
           Slope (f):     -.0074
    ============================


    - Curvilinear Model - s = e*(f**X)
    ==================================
       Intercept (e):      .0176
                 (f):      .6992
    ==================================
```

```
Cd-5.dat
Cd     5   1     0.0000    0.0000    0.0469    1.8400    0.0672    0.0786    0.9460    0.8640    8.6700    8.7400
Cd     5   2     0.0170    0.0123    0.0678    0.0785    0.1233    0.1166    1.0400    1.0600    9.8920    9.9640
Cd     5   3     0.0038    0.0031    0.0541    0.0633    0.0987    0.0981    0.9950    0.9564   10.0215    9.9182
Cd     5   4     0.0050   -0.0010    0.0470    0.0600    0.1100    0.0980    1.0180    1.0490   10.0840   10.1590
Cd     5   5     0.0895    0.0715    0.0977    0.1210    0.1595    0.1717    0.9707    1.0440    9.1160    9.3767
Cd     5   6     0.0088    0.0148    0.0558    0.0977    0.1240    0.1038    1.0202    1.1519   10.0737   10.1802
Cd     5   7    -0.0348   -0.0347    0.0162    0.0222    0.0726    0.0598    0.9869    0.9937   10.0357   10.1000
Cd     5   8    -0.0208   -0.0170    0.0207    0.0349    0.0840    0.0857    0.9206    0.9097    9.4199    9.5763


File Name:  cd-5
Data Validation File (.DA~)

*****************************************************************************
***                  Parameter and Data Validation File                   ***
*****************************************************************************
   Analyte:  Cd              Matrix:  DI Water (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:   5
      Date:  06/28/2000      Method:  1638
     Pairs:  5
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------------------------
Level          1          2          3          4          5          6          7          8          9         10
-------------------------------------------------------------------------------------------------------------------------------
Spike          .0041      .0000      .0500      .0100      .0400      .0000      .9000      .0000     9.0000      .0000
Spike
Increment      .0000      .0000      .0500      .0600      .1000      .1000     1.0000     1.0000    10.0000    10.0000
-------------------------------------------------------------------------------------------------------------------------------

                Final Concentration
          ---------------------------------------------------------------------------------------------------------------------
Lab ID         .0041      .0041      .0541      .0641      .1041      .1041     1.0041     1.0041    10.0041    10.0041
-------------------------------------------------------------------------------------------------------------------------------
    1          .0000      .0000      .0469o    1.8400      .0672      .0786      .9460      .8640     8.6700     8.7400
    2          .0170      .0123      .0678      .0785      .1233      .1166     1.0400     1.0600     9.8920     9.9640
    3          .0038      .0031      .0541      .0633      .0987      .0981      .9950      .9564    10.0215     9.9182
    4          .0050     -.0010      .0470      .0600      .1100      .0980     1.0180     1.0490    10.0840    10.1590
    5    o     .0895      .0715      .0977      .1210      .1595      .1717      .9707     1.0440     9.1160     9.3767
    6    r     .0088r     .0148  r   .0558r     .0977  r   .1240r     .1038  r  1.0202r    1.1519  r  10.0737r   10.1802
    7         -.0348     -.0347      .0162      .0222      .0726      .0598      .9869      .9937    10.0357    10.1000
    8    !    -.0208!    -.0170  !   .0207!     .0349  !   .0840!     .0857  !   .9206!     .9097  !   9.4199!    9.5763
-------------------------------------------------------------------------------------------------------------------------------

Cd-5.daf
Cd     5   1      .0000     .0000     .0469o             .0672     .0786     .9460     .8640    8.6700    8.7400
Cd     5   2      .0170     .0123     .0678     .0785     .1233     .1166    1.0400    1.0600    9.8920    9.9640
Cd     5   3      .0038     .0031     .0541     .0633     .0987     .0981     .9950     .9564   10.0215    9.9182
Cd     5   4      .0050    -.0010     .0470     .0600     .1100     .0980    1.0180    1.0490   10.0840   10.1590
Cd     5   5 o              .0715     .0977     .1210     .1595     .1717     .9707    1.0440    9.1160    9.3767
Cd     5   7     -.0348    -.0347     .0162     .0222     .0726     .0598     .9869     .9937   10.0357   10.1000
Cd     5   8     -.0208    -.0170     .0207     .0349     .0840     .0857     .9206     .9097    9.4199    9.5763
```

```
File Name:  cd-5
Data Preparation File (.PRP)


Analyte:  Cd     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
------------------------------------------------------------
 Conc  Lab  Rep     Mean
 Lev   No   No     Result     Result                   Ratio
------------------------------------------------------------
  1      5  1       .0155      .0895                   5.77
  2      5  1       .0127      .0715                   5.62
  4      1  1       .2897     1.8400                   6.35
  4      7  1       .2897      .0222                    .08
  4      8  1       .2897      .0349                    .12
------------------------------------------------------------
Total Number of Questionable Observations:     5


------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  cd-5
Data Preparation File (.PRP)


Analyte:  Cd     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***---------------------------------------***
 ***     Upper Critical Value:     65.0   ***
 ***     Lower Critical Value:     25.0   ***
-----------------------------------------------

      Ranks
     ------------------------------------------------------------------------------
       Rank  Level  Level  Level  Level  Level  Level  Level  Level  Level  Level
Lab    Sums    1      2      3      4      5      6      7      8      9     10
---- ------------------------------------------------------------------------------
  1    26.0   3.00   4.00   3.00   8.00   1.00   2.00   2.00   1.00   1.00   1.00
  2    62.0   7.00   6.00   7.00   5.00   6.00   7.00   8.00   7.00   4.00   5.00
  3    44.0   4.00   5.00   5.00   4.00   4.00   5.00   5.00   3.00   5.00   4.00
  4    51.0   5.00   3.00   4.00   3.00   5.00   4.00   6.00   6.00   8.00   7.00
  5    59.0   8.00   8.00   8.00   7.00   8.00   8.00   3.00   5.00   2.00   2.00
  6    68.0   6.00   7.00   6.00   6.00   7.00   6.00   7.00   8.00   7.00   8.00
  7    27.0   1.00   1.00   1.00   1.00   2.00   1.00   4.00   4.00   6.00   6.00
  8    23.0   2.00   2.00   2.00   2.00   3.00   3.00   1.00   2.00   3.00   3.00
-----------------------------------------------
 *** Laboratory   6 Rejected; Rank Sum   68.0 ***
-----------------------------------------------

File Name:  cd-5
Data Preparation File (.PRP)


Analyte:  Cd     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

-------------------
   - Outlier(s) -
-------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
------------------------------------------------------------------------------
  1     1     5   1       .0895      .0085      .0397   2.038   2.020    7
  4     1     1   1      1.8400      .3171      .6723   2.265   2.020    7
------------------------------------------------------------------------------
```

File Name: cd-5
Data Preparation File (.PRP)

Analyte: Cd Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Results of Normality Testing ***

- Normality Tests -

Level	n	Test Type	Normality Accept/Reject	Test Statistic	Critical Value(s)	
1	6	W	A	.9164	.788	---
2	7	W	A	.8671	.803	---
3	7	W	A	.9435	.803	---
4	6	W	A	.9536	.788	---
5	7	W	A	.9398	.803	---
6	7	W	A	.8956	.803	---
7	7	W	A	.9885	.803	---
8	7	W	A	.9083	.803	---
9	7	W	A	.8516	.803	---
10	7	W	A	.8772	.803	---

- 0 Normality Rejection(s) -

File Name: cd-5
Data Preparation File (.PRP)

Analyte: Cd Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Data Removal Tracking ***
*** Simple Count of Remaining Data Points ***
*** After Removal Tests ***

Lev	Mtrx	Points As Received	After Lab Ranking Points	%	After Outlier Testing Points	%
1	5	8	7	87.5	6	75.0
2	5	8	7	87.5	7	87.5
3	5	8	7	87.5	7	87.5
4	5	8	7	87.5	6	75.0
5	5	8	7	87.5	7	87.5
6	5	8	7	87.5	7	87.5
7	5	8	7	87.5	7	87.5
8	5	8	7	87.5	7	87.5
9	5	8	7	87.5	7	87.5
10	5	8	7	87.5	7	87.5
Totals:		80	70	87.5	68	85.0

File Name: cd-5
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Cd Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8	9	10
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)	Pair 5 (Low)	Pair 5 (High)
CONCENTRATION:	.0041	.0041	.0541	.0641	.1041	.1041	1.0041	1.0041	10.0041	10.0041
RECOVERY:										
Observations	6	7	7	6	7	7	7	7	7	7
Mean Result	-.0050	.0049	.0501	.0633	.1022	.1012	.9825	.9824	9.6056	9.6906
Bias	-.0091	.0008	-.0040	-.0008	-.0019	-.0029	-.0216	-.0217	-.3985	-.3135
Relative Bias %	-221.1382	19.1638	-7.4729	-1.2220	-1.8389	-2.7721	-2.1555	-2.1611	-3.9835	-3.1337
Maximum Result	.0170	.0715	.0977	.1210	.1595	.1717	1.0400	1.0600	10.0840	10.1590
Minimum Result	-.0348	-.0347	.0162	.0222	.0672	.0598	.9206	.8640	8.6700	8.7400

SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4	Pair 5
Observations	6	6	7	7	7
Standard Deviation	.0025	.0042	.0072	.0355	.0776
Correction Factor	1.0509	1.0509	1.0424	1.0424	1.0424
Corrected Std Dev	.0027	.0044	.0075	.0370	.0809
Relative Std Dev (%)	783.3989	7.8446	7.4155	3.7653	.8389

OVERALL PRECISION:										
Observations	6	7	7	6	7	7	7	7	7	7
Standard Deviation	.0191	.0332	.0278	.0348	.0322	.0358	.0409	.0757	.5505	.5048
Correction Factor	1.0509	1.0424	1.0424	1.0509	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424
Corrected Std Dev	.0201	.0346	.0290	.0366	.0336	.0373	.0426	.0789	.5738	.5262
Relative Std Dev %	-404.1588	707.5995	57.8350	57.8175	32.8800	36.8743	4.3411	8.0292	5.9740	5.4296

File Name: cd-5
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Cd Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.0041	-.0050	-.0091	******	1.159	4.032	NO
2	.0041	.0049	.0008	19.16	.063	3.707	NO
3	.0541	.0501	-.0040	-7.47	.385	3.707	NO
4	.0641	.0633	-.0008	-1.22	.055	4.032	NO
5	.1041	.1022	-.0019	-1.84	.157	3.707	NO
6	.1041	.1012	-.0029	-2.77	.213	3.707	NO
7	1.0041	.9825	-.0216	-2.16	1.399	3.707	NO
8	1.0041	.9824	-.0217	-2.16	.759	3.707	NO
9	10.0041	9.6056	-.3985	-3.98	1.915	3.707	NO
10	10.0041	9.6906	-.3135	-3.13	1.643	3.707	NO

File Name: cd-5
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Single Operator Precision ***

Analyte: Cd Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Single Operator Precision ***

- Linear Model - s = b*T + a

Intercept (a):	.0068
Slope (b):	.0112

Pair	Sample Size	Weights (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	6	28.88	.0041	.0027	.0069
2	6	26.92	.0591	.0044	.0075
3	7	30.76	.1041	.0075	.0080
4	7	12.82	1.0041	.0370	.0181
5	7	.62	10.0041	.0809	.1190

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a:	.0072	Intercept (a'):	-4.9287
b:	1.2903	Slope (b'):	.2549

Pair	Sample Size	Weight (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	6	17.78	.0041	.0027	.0072
2	6	17.78	.0041	.0044	.0073
3	7	21.48	.0541	.0075	.0074
4	7	21.48	.0641	.0370	.0093
5	7	21.48	.1041	.0809	.0927

```
File Name:  cd-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ***       Overall Precision        ***
---------------------------------------
Analyte:  Cd          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Overall Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .0287
          Slope (b):      .0459
    ============================
    Conc   Sample   Weights                Overall   Estimated
    Level  Size         (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6       17.09       .0041       .0201       .0289
      2       7       20.64       .0041       .0346       .0289
      3       7       16.80       .0541       .0290       .0312
      4       6       13.38       .0641       .0366       .0316
      5       7       13.94       .1041       .0336       .0335
      6       7       13.94       .1041       .0373       .0335
      7       7        2.06      1.0041       .0426       .0748
      8       7        2.06      1.0041       .0789       .0748
      9       7         .04     10.0041       .5738       .4879
     10       7         .04     10.0041       .5262       .4879
    ------------------------------------------------------------


    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ============================================================
      a:      .0337      Intercept (a'):    -3.3917
      b:     1.3255           Slope (b'):      .2818
    ============================================================
    Conc   Sample   Weight                 Overall   Estimated
    Level  Size        (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6       8.57       .0041       .0201       .0337
      2       7      10.36       .0041       .0346       .0337
      3       7      10.36       .0541       .0290       .0342
      4       6       8.57       .0641       .0366       .0343
      5       7      10.36       .1041       .0336       .0347
      6       7      10.36       .1041       .0373       .0347
      7       7      10.36      1.0041       .0426       .0447
      8       7      10.36      1.0041       .0789       .0447
      9       7      10.36     10.0041       .5738       .5640
     10       7      10.36     10.0041       .5262       .5640
    ------------------------------------------------------------
```

```
File Name:  cd-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
---------------------------------------
Analyte:  Cd          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):     -.0008
          Slope (b):      .9692
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       6     16.41       .0041      -.0050       .0032
      2       7     19.14       .0041       .0049       .0032
      3       7     16.43       .0541       .0501       .0516
      4       6     13.68       .0641       .0633       .0613
      5       7     14.25       .1041       .1022       .1001
      6       7     14.25       .1041       .1012       .1001
      7       7      2.85      1.0041       .9825       .9723
      8       7      2.85      1.0041       .9824       .9723
      9       7       .07     10.0041      9.6056      9.6949
     10       7       .07     10.0041      9.6906      9.6949
    ------------------------------------------------------------


File Name:  cd-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
---------------------------------------
Analyte:  Cd          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .0077
            Slope (f):      .0116
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .0072
                  (f):     1.3008
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .0295
            Slope (f):      .0474
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .0337
                  (f):     1.3374
     ==================================
```

```
Cd-16.dat
Cd    16   1     0.0000    0.0001    0.0224    0.0576    0.1190    0.1510    0.2080    0.2180
Cd    16   2    -0.0634    0.0042    0.0582    0.0699    0.1212    0.1466    0.2926    0.2465
Cd    16   3     0.0040    0.0048    0.0424    0.0635    0.1281    0.1426    0.2497    0.2538
Cd    16   4     0.0090   -0.0070    0.0320    0.0530    0.1140    0.1320    0.2750    0.2600
Cd    16   5     0.0420    0.0360    0.1028    0.1133    0.1829    0.1926    0.3067    0.3099
Cd    16   6     0.0132    0.0157    0.0349    0.0640    0.1414    0.1570    0.2666    0.2657
Cd    16   7    -0.0373   -0.0370   -0.0008    0.0108    0.0816    0.1053    0.2298    0.2180
Cd    16   8    -0.0218   -0.0203    0.0127    0.0194    0.0942    0.1121    0.2322    0.2185

File Name:  cd-16
Data Validation File (.DA~)

*****************************************************************************
***                    Parameter and Data Validation File                 ***
*****************************************************************************
   Analyte:  Cd              Matrix:  Freshwater (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:  16
      Date:  06/28/2000      Method:  1638
     Pairs:  4
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------
Level          1          2           3          4           5          6           7          8
-------------------------------------------------------------------------------------------------------------
Spike          .0001      .0000        .0400      .0100        .0700      .0200        .1100      .0000
Spike
Increment      .0000      .0000        .0400      .0500        .1200      .1400        .2500      .2500
-------------------------------------------------------------------------------------------------------------

               Final Concentration
          ---------------------------------------------------------------------------------------------------
Lab ID         .0001      .0001        .0401      .0501        .1201      .1401        .2501      .2501
-------------------------------------------------------------------------------------------------------------
    1          .0000      .0001        .0224      .0576        .1190      .1510        .2080      .2180
    2         -.0634      .0042        .0582      .0699        .1212      .1466        .2926      .2465
    3          .0040      .0048        .0424      .0635        .1281      .1426        .2497      .2538
    4          .0090     -.0070        .0320      .0530        .1140      .1320        .2750      .2600
    5    r     .0420r     .0360  r     .1028r     .1133  r     .1829r     .1926  r     .3067r     .3099
    6          .0132      .0157        .0349      .0640        .1414      .1570        .2666      .2657
    7    !    -.0373!    -.0370  !    -.0008!     .0108  !     .0816!     .1053  !     .2298!     .2180
    8         -.0218     -.0203        .0127      .0194        .0942      .1121        .2322      .2185
-------------------------------------------------------------------------------------------------------------

Cd-16.daf
Cd    16   1      .0000     .0001     .0224     .0576     .1190     .1510     .2080     .2180
Cd    16   2     -.0634     .0042     .0582     .0699     .1212     .1466     .2926     .2465
Cd    16   3      .0040     .0048     .0424     .0635     .1281     .1426     .2497     .2538
Cd    16   4      .0090    -.0070     .0320     .0530     .1140     .1320     .2750     .2600
Cd    16   6      .0132     .0157     .0349     .0640     .1414     .1570     .2666     .2657
Cd    16   7     -.0373    -.0370    -.0008     .0108     .0816     .1053     .2298     .2180
Cd    16   8     -.0218    -.0203     .0127     .0194     .0942     .1121     .2322     .2185
```

File Name: cd-16
Data Preparation File (.PRP)

Analyte: Cd Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Results of 5x - 1/5x Mean Error Check ***
*** Questionable Data (Positive Values) ***

Conc Lev	Lab No	Rep No	Mean Result	Result	Ratio
2	1	1	.0076	.0001	.01
4	7	1	.0564	.0108	.19

Total Number of Questionable Observations: 2

*** Results of Factor of 5 Error Check ***
*** Questionable Data (All Values) ***

Total Number of Questionable Observations: 0

File Name: cd-16
Data Preparation File (.PRP)

Analyte: Cd Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Laboratory Ranking Results ***
*** Two-Tailed 5% Significance Level ***
*** Upper Critical Value: 54.0 ***
*** Lower Critical Value: 18.0 ***

	Ranks							
Lab	Rank Sums	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1	27.5	4.00	4.00	3.00	4.00	4.00	6.00	1.00	1.50
2	41.0	1.00	5.00	7.00	7.00	5.00	5.00	7.00	4.00
3	41.0	5.00	6.00	6.00	5.00	6.00	4.00	4.00	5.00
4	34.0	6.00	3.00	4.00	3.00	3.00	3.00	6.00	6.00
5	64.0	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00
6	51.0	7.00	7.00	5.00	6.00	7.00	7.00	5.00	7.00
7	10.5	2.00	1.00	1.00	1.00	1.00	1.00	2.00	1.50
8	19.0	3.00	2.00	2.00	2.00	2.00	2.00	3.00	3.00

*** Laboratory 5 Rejected; Rank Sum 64.0 ***

File Name: cd-16
Data Preparation File (.PRP)

Analyte: Cd Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Outlier Testing Results ***
*** Two-Sided 5% Significance Level ***

- No Outlier Rejections in Iteration 1 -
- No Further Outlier Testing Necessary -

```
File Name:  cd-16
Data Preparation File (.PRP)


Analyte:  Cd     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
              Test  Normality      Test          Critical
   Level  n   Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     7     W     A               .8849      .803   ---
     2     7     W     A               .9323      .803   ---
     3     7     W     A               .9938      .803   ---
     4     7     W     A               .8154      .803   ---
     5     7     W     A               .9554      .803   ---
     6     7     W     A               .9038      .803   ---
     7     7     W     A               .9759      .803   ---
     8     7     W     A               .8252      .803   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------



File Name:  cd-16
Data Preparation File (.PRP)


Analyte:  Cd     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    16        8            7    87.5           7       87.5
  2    16        8            7    87.5           7       87.5
  3    16        8            7    87.5           7       87.5
  4    16        8            7    87.5           7       87.5
  5    16        8            7    87.5           7       87.5
  6    16        8            7    87.5           7       87.5
  7    16        8            7    87.5           7       87.5
  8    16        8            7    87.5           7       87.5
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         64           56    87.5          56       87.5
-----------------------------------------------------------------
```

File Name: cd-16
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Cd Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)
CONCENTRATION:	.0001	.0001	.0401	.0501	.1201	.1401	.2501	.2501
RECOVERY:								
Observations	7	7	7	7	7	7	7	7
Mean Result	-.0138	-.0056	.0288	.0483	.1142	.1352	.2506	.2401
Bias	-.0139	-.0057	-.0113	-.0018	-.0059	-.0049	.0005	-.0100
Relative Bias %	*********	*********	-28.1083	-3.5643	-4.9007	-3.4771	.1828	-4.0098
Maximum Result	.0132	.0157	.0582	.0699	.1414	.1570	.2926	.2657
Minimum Result	-.0634	-.0370	-.0008	.0108	.0816	.1053	.2080	.2180

SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4
Observations	7	7	7	7
Standard Deviation	.0191	.0072	.0045	.0130
Correction Factor	1.0424	1.0424	1.0424	1.0424
Corrected Std Dev	.0199	.0076	.0046	.0135
Relative Std Dev (%)	-205.1619	19.5892	3.7194	5.5198

OVERALL PRECISION:								
Observations	7	7	7	7	7	7	7	7
Standard Deviation	.0284	.0178	.0195	.0234	.0203	.0198	.0294	.0213
Correction Factor	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424
Corrected Std Dev	.0296	.0185	.0203	.0244	.0211	.0206	.0307	.0222
Relative Std Dev %	-215.0291	-328.2260	70.3760	50.5514	18.5089	15.2465	12.2496	9.2503

File Name: cd-16
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Cd Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.0001	-.0138	-.0139	******	1.292	3.707	NO
2	.0001	-.0056	-.0057	******	.855	3.707	NO
3	.0401	.0288	-.0113	-28.11	1.532	3.707	NO
4	.0501	.0483	-.0018	-3.56	.202	3.707	NO
5	.1201	.1142	-.0059	-4.90	.768	3.707	NO
6	.1401	.1352	-.0049	-3.48	.652	3.707	NO
7	.2501	.2506	.0005	.18	.041	3.707	NO
8	.2501	.2401	-.0100	-4.01	1.245	3.707	NO

```
File Name:  cd-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***   Single Operator Precision    ***
----------------------------------------
Analyte:  Cd          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .0129
          Slope (b):     -.0144
    ============================
                                        Single
           Sample Weights              Operator   Estimated
    Pair   Size       (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       7     25.00       .0001       .0199       .0129
      2       7     25.00       .0451       .0076       .0123
      3       7     25.00       .1301       .0046       .0111
      4       7     25.00       .2501       .0135       .0093
    ------------------------------------------------------------



    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ======================================================
      a:      .0108      Intercept (a'):    -4.5297
      b:      .4299          Slope (b'):     -.8442
    ======================================================
                                              Single
           Sample Weight              Operator   Estimated
    Pair   Size      (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       7    25.00       .0001       .0199       .0108
      2       7    25.00       .0001       .0076       .0104
      3       7    25.00       .0401       .0046       .0097
      4       7    25.00       .0501       .0135       .0087
    ------------------------------------------------------------
```

File Name: cd-16
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Overall Precision ***

Analyte: Cd Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Overall Precision ***

- Linear Model - s = b*T + a

Intercept (a):	.0224
Slope (b):	.0093

Conc Level	Sample Size	Weights (%)	Conc	Overall Std Dev	Estimated Std Dev
1	7	13.70	.0001	.0296	.0224
2	7	13.70	.0001	.0185	.0224
3	7	13.20	.0401	.0203	.0228
4	7	13.08	.0501	.0244	.0229
5	7	12.28	.1201	.0211	.0236
6	7	12.07	.1401	.0206	.0237
7	7	10.98	.2501	.0307	.0248
8	7	10.98	.2501	.0222	.0248

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a:	.0220	Intercept (a'):	-3.8165
b:	1.5690	Slope (b'):	.4504

Conc Level	Sample Size	Weight (%)	Conc	Overall Std Dev	Estimated Std Dev
1	7	12.50	.0001	.0296	.0220
2	7	12.50	.0001	.0185	.0220
3	7	12.50	.0401	.0203	.0224
4	7	12.50	.0501	.0244	.0225
5	7	12.50	.1201	.0211	.0232
6	7	12.50	.1401	.0206	.0234
7	7	12.50	.2501	.0307	.0246
8	7	12.50	.2501	.0222	.0246

```
File Name:  cd-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
----------------------------------------
Analyte:  Cd          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):     -.0086
          Slope (b):     1.0186
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       7     13.57       .0001      -.0138      -.0085
      2       7     13.57       .0001      -.0056      -.0085
      3       7     13.13       .0401       .0288       .0322
      4       7     13.02       .0501       .0483       .0424
      5       7     12.31       .1201       .1142       .1137
      6       7     12.12       .1401       .1352       .1341
      7       7     11.13       .2501       .2506       .2461
      8       7     11.13       .2501       .2401       .2461
    ------------------------------------------------------------


File Name:  cd-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
----------------------------------------
Analyte:  Cd          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .0214
            Slope (f):     -.0141
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .0107
                  (f):      .4366
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .0309
            Slope (f):      .0092
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .0221
                  (f):     1.5562
     ==================================
```

```
Cu-5.dat
Cu     5   1     0.0000    0.0000    0.1750    0.2460    0.4490    0.4420    1.7700    1.8000   17.9000   18.1000
Cu     5   2     0.1589    0.1326    0.4544    0.2860    1.0490    0.8788    2.2070    2.1410   19.9900   20.2300
Cu     5   3     0.0323    0.0481    0.3242    0.2824    0.6168    0.6020    2.1453    3.2563   19.4746   19.7283
Cu     5   4     0.0440    0.0260    2.0470    0.9950    1.1720    0.5570    2.1880    2.2190   21.0530   21.3540
Cu     5   5     0.5282    0.6445    0.6310    0.4641    0.5643    0.6304    1.9111    1.8558   18.6020   18.8151
Cu     5   6     0.1286    0.3061    0.3025    0.5329    0.5794    0.6422    2.2046    3.5277   18.5092   43.9433
Cu     5   7     0.0161    0.0160    0.1925    0.2383    0.4596    0.4643    1.9044    1.8799   18.5410   18.9762
Cu     5   8     0.0173    0.0180    0.1719    0.2100    0.4540    0.4798    1.7638    1.8114   18.1127   18.5166

File Name:  cu-5
Data Validation File (.DA~)

*****************************************************************************
***                    Parameter and Data Validation File                 ***
*****************************************************************************
   Analyte:  Cu              Matrix:  DI Water (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:   5
      Date:  06/28/2000      Method:  1638
     Pairs:  5
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------------------------
Level          1          2          3          4          5          6          7          8          9          10
-------------------------------------------------------------------------------------------------------------------------------
Spike          .1052      .0000       .2000      .0400       .2600      .0000      1.5000      .0000     18.0000      .0000
Spike
Increment      .0000      .0000       .2000      .2400       .5000      .5000      2.0000     2.0000     20.0000    20.0000
-------------------------------------------------------------------------------------------------------------------------------

                Final Concentration
            -------------------------------------------------------------------------------------------------------------------
Lab ID          .1052      .1052       .3052      .3452       .6052      .6052      2.1052     2.1052     20.1052    20.1052
-------------------------------------------------------------------------------------------------------------------------------
    1    r     .0000r     .0000  r     .1750r     .2460  r     .4490r     .4420  r    1.7700r    1.8000  r   17.9000r   18.1000
    2          .1589      .1326        .4544      .2860       1.0490      .8788       2.2070     2.1410      19.9900    20.2300
    3          .0323      .0481        .3242      .2824        .6168      .6020       2.1453     3.2563      19.4746    19.7283
    4          .0440      .0260  o    2.0470o     .9950       1.1720      .5570       2.1880     2.2190      21.0530    21.3540
    5    o     .5282o     .6445        .6310      .4641        .5643      .6304       1.9111     1.8558      18.6020    18.8151
    6          .1286      .3061        .3025      .5329        .5794      .6422       2.2046     3.5277      18.5092o   43.9433
    7          .0161      .0160        .1925      .2383        .4596      .4643       1.9044     1.8799      18.5410    18.9762
    8    !     .0173!     .0180  !     .1719!     .2100  !     .4540!     .4798  !    1.7638!    1.8114  !   18.1127!   18.5166
-------------------------------------------------------------------------------------------------------------------------------

Cu-5.daf
Cu     5   2      .1589     .1326     .4544     .2860    1.0490     .8788    2.2070    2.1410   19.9900   20.2300
Cu     5   3      .0323     .0481     .3242     .2824     .6168     .6020    2.1453    3.2563   19.4746   19.7283
Cu     5   4      .0440     .0260o         o            1.1720     .5570    2.1880    2.2190   21.0530   21.3540
Cu     5   5 o         o             .6310     .4641     .5643     .6304    1.9111    1.8558   18.6020   18.8151
Cu     5   6      .1286     .3061     .3025     .5329     .5794     .6422    2.2046    3.5277   18.5092o
Cu     5   7      .0161     .0160     .1925     .2383     .4596     .4643    1.9044    1.8799   18.5410   18.9762
Cu     5   8      .0173     .0180     .1719     .2100     .4540     .4798    1.7638    1.8114   18.1127   18.5166
```

```
File Name:  cu-5
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
-------------------------------------------------------------
 Conc  Lab  Rep     Mean
 Lev   No   No     Result     Result                   Ratio
-------------------------------------------------------------
  1      7  1       .1157      .0161                    .14
  1      8  1       .1157      .0173                    .15
  2      4  1       .1489      .0260                    .17
  2      7  1       .1489      .0160                    .11
  2      8  1       .1489      .0180                    .12
-------------------------------------------------------------
Total Number of Questionable Observations:     5


-------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
-------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  cu-5
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***-------------------------------------***
 ***     Upper Critical Value:     65.0   ***
 ***     Lower Critical Value:     25.0   ***
-----------------------------------------------

      Ranks
     ------------------------------------------------------------------------------
       Rank  Level  Level  Level  Level  Level  Level  Level  Level  Level  Level
Lab    Sums    1      2      3      4      5      6      7      8      9     10
---- ------------------------------------------------------------------------------
  1    14.0   1.00   1.00   2.00   3.00   1.00   1.00   2.00   1.00   1.00   1.00
  2    65.0   7.00   6.00   6.00   5.00   7.00   8.00   8.00   5.00   7.00   6.00
  3    52.0   4.00   5.00   5.00   4.00   6.00   5.00   5.00   7.00   6.00   5.00
  4    64.0   5.00   4.00   8.00   8.00   8.00   4.00   6.00   6.00   8.00   7.00
  5    54.0   8.00   8.00   7.00   6.00   4.00   6.00   4.00   3.00   5.00   3.00
  6    62.0   6.00   7.00   4.00   7.00   5.00   7.00   7.00   8.00   3.00   8.00
  7    29.0   2.00   2.00   3.00   2.00   3.00   2.00   3.00   4.00   4.00   4.00
  8    20.0   3.00   3.00   1.00   1.00   2.00   3.00   1.00   2.00   2.00   2.00
-----------------------------------------------
 *** Laboratory   1 Rejected; Rank Sum   14.0 ***
-----------------------------------------------

File Name:  cu-5
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

-------------------
   - Outlier(s) -
-------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
-------------------------------------------------------------------------------
  1     1     5   1       .5282      .1322      .1835   2.158   2.020    7
  2     1     5   1       .6445      .1702      .2336   2.030   2.020    7
  3     1     4   1      2.0470      .5891      .6619   2.202   2.020    7
  4     1     4   1       .9950      .4298      .2765   2.044   2.020    7
  *     1     6   1     43.9433    23.0805     9.2508   2.255   2.020    7
-------------------------------------------------------------------------------
```

File Name: cu-5
Data Preparation File (.PRP)

Analyte: Cu Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Results of Normality Testing ***

- Normality Tests -

Level	n	Test Type	Normality Accept/Reject	Test Statistic	Critical Value(s)	
1	6	W	A	.8091	.788	---
2	6	W	R	.7488	.788	---
3	6	W	A	.9206	.788	---
4	6	W	A	.8563	.788	---
5	7	W	R	.7983	.803	---
6	7	W	A	.8807	.803	---
7	7	W	A	.8285	.803	---
8	7	W	R	.7881	.803	---
9	7	W	A	.8841	.803	---
10	6	W	A	.9231	.788	---

- 3 Normality Rejection(s) -

File Name: cu-5
Data Preparation File (.PRP)

Analyte: Cu Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Data Removal Tracking ***
*** Simple Count of Remaining Data Points ***
*** After Removal Tests ***

Lev	Mtrx	Points As Received	After Lab Ranking Points	After Lab Ranking %	After Outlier Testing Points	After Outlier Testing %
1	5	8	7	87.5	6	75.0
2	5	8	7	87.5	6	75.0
3	5	8	7	87.5	6	75.0
4	5	8	7	87.5	6	75.0
5	5	8	7	87.5	7	87.5
6	5	8	7	87.5	7	87.5
7	5	8	7	87.5	7	87.5
8	5	8	7	87.5	7	87.5
9	5	8	7	87.5	7	87.5
10	5	8	7	87.5	6	75.0
Totals:		80	70	87.5	65	81.3

File Name: cu-5
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Cu Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8	9	10
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)	Pair 5 (Low)	Pair 5 (High)
CONCENTRATION:	.1052	.1052	.3052	.3452	.6052	.6052	2.1052	2.1052	20.1052	20.1052
RECOVERY:										
Observations	6	6	6	6	7	7	7	7	7	6
Mean Result	.0662	.0911	.3461	.3356	.6993	.6078	2.0463	2.3844	19.1832	19.6034
Bias	-.0390	-.0141	.0409	-.0096	.0941	.0026	-.0589	.2792	-.9220	-.5018
Relative Bias %	-37.0722	-13.3714	13.3956	-2.7762	15.5486	.4272	-2.7972	13.2644	-4.5858	-2.4960
Maximum Result	.1589	.3061	.6310	.5329	1.1720	.8788	2.2070	3.5277	21.0530	21.3540
Minimum Result	.0161	.0160	.1719	.2100	.4540	.4643	1.7638	1.8114	18.1127	18.5166

SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4	Pair 5
Observations	6	6	7	7	6
Standard Deviation	.0539	.1067	.1727	.4278	.0648
Correction Factor	1.0509	1.0509	1.0424	1.0424	1.0509
Corrected Std Dev	.0566	.1122	.1800	.4459	.0681
Relative Std Dev (%)	71.9945	32.9114	27.5399	20.1269	.3515

OVERALL PRECISION:										
Observations	6	6	6	6	7	7	7	7	7	6
Standard Deviation	.0617	.1140	.1728	.1311	.2895	.1383	.1821	.7090	1.0474	1.0648
Correction Factor	1.0509	1.0509	1.0509	1.0509	1.0424	1.0424	1.0424	1.0424	1.0424	1.0509
Corrected Std Dev	.0648	.1198	.1816	.1378	.3017	.1441	.1898	.7390	1.0918	1.1190
Relative Std Dev %	97.9391	131.5014	52.4588	41.0608	43.1480	23.7148	9.2764	30.9944	5.6912	5.7084

File Name: cu-5
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Cu Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.1052	.0662	-.0390	-37.07	1.549	4.032	NO
2	.1052	.0911	-.0141	-13.37	.302	4.032	NO
3	.3052	.3461	.0409	13.40	.580	4.032	NO
4	.3452	.3356	-.0096	-2.78	.179	4.032	NO
5	.6052	.6993	.0941	15.55	.860	3.707	NO
6	.6052	.6078	.0026	.43	.049	3.707	NO
7	2.1052	2.0463	-.0589	-2.80	.856	3.707	NO
8	2.1052	2.3844	.2792	13.26	1.042	3.707	NO
9	20.1052	19.1832	-.9220	-4.59	2.329	3.707	NO
10	20.1052	19.6034	-.5018	-2.50	1.154	4.032	NO

File Name: cu-5
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Single Operator Precision ***

Analyte: Cu Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Single Operator Precision ***

- Linear Model - s = b*T + a

Intercept (a): .2076
Slope (b): -.0055

Pair	Sample Size	Weights (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	6	18.47	.1052	.0566	.2070
2	6	18.47	.3252	.1122	.2058
3	7	22.30	.6052	.1800	.2043
4	7	22.30	2.1052	.4459	.1960
5	6	18.47	20.1052	.0681	.0965

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a: .1594 Intercept (a'): -1.8361
b: .9650 Slope (b'): -.0356

Pair	Sample Size	Weight (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	6	18.47	.1052	.0566	.1588
2	6	18.47	.1052	.1122	.1576
3	7	22.30	.3052	.1800	.1560
4	7	22.30	.3452	.4459	.1479
5	6	18.47	.6052	.0681	.0779

File Name: cu-5
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Overall Precision ***

Analyte: Cu Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Overall Precision ***

- Linear Model - s = b*T + a

Intercept (a): .1681
Slope (b): .0598

Conc Level	Sample Size	Weights (%)	Conc	Overall Std Dev	Estimated Std Dev
1	6	14.25	.1052	.0648	.1744
2	6	14.25	.1052	.1198	.1744
3	6	13.07	.3052	.1816	.1864
4	6	12.85	.3452	.1378	.1888
5	7	13.96	.6052	.3017	.2043
6	7	13.96	.6052	.1441	.2043
7	7	8.29	2.1052	.1898	.2941
8	7	8.29	2.1052	.7390	.2941
9	7	.59	20.1052	1.0918	1.3712
10	6	.49	20.1052	1.1190	1.3712

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a: .1717 Intercept (a'): -1.7619
b: 1.1006 Slope (b'): .0958

Conc Level	Sample Size	Weight (%)	Conc	Overall Std Dev	Estimated Std Dev
1	6	9.06	.1052	.0648	.1735
2	6	9.06	.1052	.1198	.1735
3	6	9.06	.3052	.1816	.1768
4	6	9.06	.3452	.1378	.1775
5	7	10.94	.6052	.3017	.1820
6	7	10.94	.6052	.1441	.1820
7	7	10.94	2.1052	.1898	.2101
8	7	10.94	2.1052	.7390	.2101
9	7	10.94	20.1052	1.0918	1.1790
10	6	9.06	20.1052	1.1190	1.1790

```
File Name:  cu-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
---------------------------------------
Analyte:  Cu          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):      .0344
          Slope (b):      .9777
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       6     15.91       .1052       .0662       .1372
      2       6     15.91       .1052       .0911       .1372
      3       6     13.93       .3052       .3461       .3328
      4       6     13.58       .3452       .3356       .3719
      5       7     13.52       .6052       .6993       .6261
      6       7     13.52       .6052       .6078       .6261
      7       7      6.53      2.1052      2.0463      2.0926
      8       7      6.53      2.1052      2.3844      2.0926
      9       7       .30     20.1052     19.1832     19.6907
     10       6       .26     20.1052     19.6034     19.6907
    ------------------------------------------------------------


File Name:  cu-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
---------------------------------------
Analyte:  Cu          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .1724
            Slope (f):     -.0057
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .1596
                  (f):      .9642
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .1329
            Slope (f):      .0612
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .1711
                  (f):     1.1030
     ==================================
```

```
Cu-16.dat
Cu    16   1     0.9110    0.9870    1.4400    1.6900    2.4000    2.8800    4.3400    4.7200
Cu    16   2     6.2910    1.4400    1.8150    1.8170    2.5870    3.2650    4.5170    5.1440
Cu    16   3     1.0159    1.0749    1.5634    1.6089    2.2561    2.8573    3.9508    4.3348
Cu    16   4     1.2270    1.2380    1.7160    1.8940    2.7240    3.4490    5.8480    5.2620
Cu    16   5     1.1891    8.0865    1.7400    5.4500    2.5788    3.6508    4.3962    4.7973
Cu    16   6     1.3090    1.1262    1.6680    1.8756    2.4713    2.9969    4.3591    4.5801
Cu    16   7     1.0672    1.0314    1.4750    1.6631    2.3944    2.8477    4.1560    4.6702
Cu    16   8     1.0520    1.0250    1.4739    1.6273    2.3142    2.8229    4.0287    4.5219


File Name:  cu-16
Data Validation File (.DA~)

*****************************************************************************
***                  Parameter and Data Validation File                   ***
*****************************************************************************
   Analyte:  Cu              Matrix:  Freshwater (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:  16
      Date:  06/28/2000      Method:  1638
     Pairs:  4
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------
Level          1          2           3          4           5          6           7          8
-------------------------------------------------------------------------------------------------------------
Spike         1.2599      .0000       .5000      .2000       .8000      .5000      1.5000      .5000
Spike
Increment      .0000      .0000       .5000      .7000      1.5000     2.0000      3.5000     4.0000
-------------------------------------------------------------------------------------------------------------

                Final Concentration
          ----------------------------------------------------------------------------------------------
Lab ID        1.2599     1.2599       1.7599     1.9599       2.7599     3.2599       4.7599     5.2599
-------------------------------------------------------------------------------------------------------------
    1          .9110      .9870       1.4400     1.6900       2.4000     2.8800       4.3400     4.7200
    2    o    6.2910!    1.4400  !    1.8150!    1.8170  !    2.5870!    3.2650  !    4.5170!    5.1440
    3    !    1.0159!    1.0749  !    1.5634!    1.6089  !    2.2561!    2.8573  !    3.9508!    4.3348
    4    !    1.2270!    1.2380  !    1.7160!    1.8940  !    2.7240!    3.4490  o    5.8480!    5.2620
    5         1.1891o    8.0865       1.7400o    5.4500       2.5788     3.6508       4.3962     4.7973
    6         1.3090     1.1262       1.6680     1.8756       2.4713     2.9969       4.3591     4.5801
    7         1.0672     1.0314       1.4750     1.6631       2.3944     2.8477       4.1560     4.6702
    8    r    1.0520r    1.0250  r    1.4739r    1.6273  r    2.3142r    2.8229  r    4.0287r    4.5219
-------------------------------------------------------------------------------------------------------------

Cu-16.daf
Cu    16   1      .9110     .9870    1.4400    1.6900    2.4000    2.8800    4.3400    4.7200
Cu    16   2 o             1.4400    1.8150    1.8170    2.5870    3.2650    4.5170    5.1440
Cu    16   3     1.0159    1.0749    1.5634    1.6089    2.2561    2.8573    3.9508    4.3348
Cu    16   4     1.2270    1.2380    1.7160    1.8940    2.7240    3.4490o             5.2620
Cu    16   5     1.1891o             1.7400o             2.5788    3.6508    4.3962    4.7973
Cu    16   6     1.3090    1.1262    1.6680    1.8756    2.4713    2.9969    4.3591    4.5801
Cu    16   7     1.0672    1.0314    1.4750    1.6631    2.3944    2.8477    4.1560    4.6702
```

```
File Name:  cu-16
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  cu-16
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***---------------------------------------***
 ***     Upper Critical Value:     54.0   ***
 ***     Lower Critical Value:     18.0   ***
-----------------------------------------------

      Ranks
     ------------------------------------------------------------------
       Rank  Level  Level  Level  Level  Level  Level  Level  Level
Lab    Sums    1      2      3      4      5      6      7      8
---- ------------------------------------------------------------------
  1    24.0   1.00   1.00   1.00   4.00   4.00   4.00   4.00   5.00
  2    55.0   8.00   7.00   8.00   5.00   7.00   6.00   7.00   7.00
  3    17.0   2.00   4.00   4.00   1.00   1.00   3.00   1.00   1.00
  4    56.0   6.00   6.00   6.00   7.00   8.00   7.00   8.00   8.00
  5    54.0   5.00   8.00   7.00   8.00   6.00   8.00   6.00   6.00
  6    41.0   7.00   5.00   5.00   6.00   5.00   5.00   5.00   3.00
  7    25.0   4.00   3.00   3.00   3.00   3.00   2.00   3.00   4.00
  8    16.0   3.00   2.00   2.00   2.00   2.00   1.00   2.00   2.00
-------------------------------------------------
 *** Laboratory   8 Rejected; Rank Sum   16.0 ***
-------------------------------------------------

File Name:  cu-16
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
-------------------------------------------------------------------------------
  1     1     2   1      6.2910     1.8586     1.9592   2.262   2.020    7
  2     1     5   1      8.0865     2.1406     2.6263   2.264   2.020    7
  4     1     5   1      5.4500     2.2855     1.3997   2.261   2.020    7
  7     1     4   1      5.8480     4.5096      .6182   2.165   2.020    7
-------------------------------------------------------------------------------
```

```
File Name:  cu-16
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
              Test  Normality      Test          Critical
   Level  n   Type  Accept/Reject  Statistic     Value(s)
-------------------------------------------------------------------
     1     6     W     A               .9719      .788   ---
     2     6     W     A               .9025      .788   ---
     3     7     W     A               .9399      .803   ---
     4     6     W     A               .9008      .788   ---
     5     7     W     A               .9749      .803   ---
     6     7     W     A               .8613      .803   ---
     7     6     W     A               .9243      .788   ---
     8     7     W     A               .9501      .803   ---
------------------------------
 - 0 Normality Rejection(s) -
------------------------------


File Name:  cu-16
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     ----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    16        8            7    87.5           6       75.0
  2    16        8            7    87.5           6       75.0
  3    16        8            7    87.5           7       87.5
  4    16        8            7    87.5           6       75.0
  5    16        8            7    87.5           7       87.5
  6    16        8            7    87.5           7       87.5
  7    16        8            7    87.5           6       75.0
  8    16        8            7    87.5           7       87.5
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         64           56    87.5          52       81.3
-------------------------------------------------------------
```

File Name: cu-16
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Cu Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)
CONCENTRATION:	1.2599	1.2599	1.7599	1.9599	2.7599	3.2599	4.7599	5.2599
RECOVERY:								
Observations	6	6	7	6	7	7	6	7
Mean Result	1.1199	1.1496	1.6311	1.7581	2.4874	3.1352	4.2865	4.7869
Bias	-.1400	-.1103	-.1288	-.2018	-.2725	-.1247	-.4734	-.4730
Relative Bias %	-11.1146	-8.7560	-7.3210	-10.2964	-9.8746	-3.8240	-9.9452	-8.9923
Maximum Result	1.3090	1.4400	1.8150	1.8940	2.7240	3.6508	4.5170	5.2620
Minimum Result	.9110	.9870	1.4400	1.6089	2.2561	2.8477	3.9508	4.3348

SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4
Observations	5	6	7	6
Standard Deviation	.0733	.0695	.1500	.0972
Correction Factor	1.0638	1.0509	1.0424	1.0509
Corrected Std Dev	.0780	.0730	.1563	.1022
Relative Std Dev (%)	6.8752	4.3199	5.5606	2.2432

OVERALL PRECISION:								
Observations	6	6	7	6	7	7	6	7
Standard Deviation	.1478	.1665	.1413	.1197	.1551	.3228	.2015	.3211
Correction Factor	1.0509	1.0509	1.0424	1.0509	1.0424	1.0424	1.0509	1.0424
Corrected Std Dev	.1553	.1750	.1472	.1258	.1616	.3365	.2118	.3347
Relative Std Dev %	13.8719	15.2253	9.0269	7.1567	6.4987	10.7328	4.9403	6.9917

File Name: cu-16
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Cu Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	1.2599	1.1199	-.1400	-11.11	2.321	4.032	NO
2	1.2599	1.1496	-.1103	-8.76	1.623	4.032	NO
3	1.7599	1.6311	-.1288	-7.32	2.413	3.707	NO
4	1.9599	1.7581	-.2018	-10.30	4.129	4.032	YES
5	2.7599	2.4874	-.2725	-9.87	4.650	3.707	YES
6	3.2599	3.1352	-.1247	-3.82	1.022	3.707	NO
7	4.7599	4.2865	-.4734	-9.95	5.754	4.032	YES
8	5.2599	4.7869	-.4730	-8.99	3.897	3.707	YES

```
File Name:  cu-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***   Single Operator Precision    ***
----------------------------------------
Analyte:  Cu          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .0707
          Slope (b):      .0125
    ============================
                                          Single
           Sample Weights               Operator   Estimated
    Pair   Size       (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       5     25.60      1.2599       .0780       .0865
      2       6     28.79      1.8599       .0730       .0940
      3       7     28.38      3.0099       .1563       .1084
      4       6     17.23      5.0099       .1022       .1334
    ------------------------------------------------------------



    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ======================================================
      a:      .0763      Intercept (a'):    -2.5735
      b:     1.1036          Slope (b'):      .0985
    ======================================================
                                                Single
           Sample Weight                Operator   Estimated
    Pair   Size      (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------
      1       5    19.82      1.2599       .0780       .0863
      2       6    25.00      1.2599       .0730       .0916
      3       7    30.19      1.7599       .1563       .1026
      4       6    25.00      1.9599       .1022       .1250
    ------------------------------------------------------
```

File Name: cu-16
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Overall Precision ***

Analyte: Cu Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Overall Precision ***

- Linear Model - s = b*T + a

Intercept (a): .0969
Slope (b): .0398

Conc Level	Sample Size	Weights (%)	Conc	Overall Std Dev	Estimated Std Dev
1	6	18.98	1.2599	.1553	.1470
2	6	18.98	1.2599	.1750	.1470
3	7	17.69	1.7599	.1472	.1669
4	6	13.32	1.9599	.1258	.1749
5	7	11.45	2.7599	.1616	.2067
6	7	9.50	3.2599	.3365	.2265
7	6	4.90	4.7599	.2118	.2862
8	7	5.17	5.2599	.3347	.3061

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a: .1170 Intercept (a'): -2.1452
b: 1.2003 Slope (b'): .1826

Conc Level	Sample Size	Weight (%)	Conc	Overall Std Dev	Estimated Std Dev
1	6	11.32	1.2599	.1553	.1473
2	6	11.32	1.2599	.1750	.1473
3	7	13.68	1.7599	.1472	.1614
4	6	11.32	1.9599	.1258	.1674
5	7	13.68	2.7599	.1616	.1937
6	7	13.68	3.2599	.3365	.2122
7	6	11.32	4.7599	.2118	.2791
8	7	13.68	5.2599	.3347	.3058

```
File Name:  cu-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
----------------------------------------
Analyte:  Cu          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):     -.0124
          Slope (b):      .9194
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       6     19.13      1.2599      1.1199      1.1459
      2       6     19.13      1.2599      1.1496      1.1459
      3       7     17.32      1.7599      1.6311      1.6056
      4       6     13.53      1.9599      1.7581      1.7895
      5       7     11.30      2.7599      2.4874      2.5250
      6       7      9.40      3.2599      3.1352      2.9847
      7       6      5.05      4.7599      4.2865      4.3637
      8       7      5.15      5.2599      4.7869      4.8234
    ------------------------------------------------------------


File Name:  cu-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
----------------------------------------
Analyte:  Cu          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .0842
            Slope (f):      .0136
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .0764
                  (f):     1.1131
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .1104
            Slope (f):      .0432
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .1173
                  (f):     1.2197
     ==================================
```

```
Pb-5.dat
Pb     5   1     0.0004    0.0035    0.0366    0.0271    0.0790    0.0832    0.4540    0.4510    4.2700    4.2600
Pb     5   2     0.1292    0.0541    0.1964    0.0852    0.2634    0.1817    0.6876    0.5714    5.1920    5.0520
Pb     5   3     0.0103    0.0145    0.0605    0.0519    0.1167    0.1194    0.5013    0.5341    4.9543    5.0154
Pb     5   4     0.0070    0.0040    0.0670    0.0390    0.1060    0.1030    0.5160    0.5170    5.1970    5.2670
Pb     5   5     0.0594    0.1421    0.1423    0.1253    0.1596    0.1953    0.4931    0.5157    4.7988    4.8011
Pb     5   6     0.0208    0.0291    0.0528    0.0777    0.1160    0.1135    0.5201    2.4625    5.0285    6.3171
Pb     5   7     0.0051    0.0052    0.0375    0.0366    0.0996    0.0974    0.4792    0.4807    4.8454    4.8580
Pb     5   8    -0.0016    0.0004    0.0239    0.0290    0.0943    0.0921    0.4677    0.4772    4.8273    4.9817

File Name:  pb-5
Data Validation File (.DA~)

*****************************************************************************
***                    Parameter and Data Validation File                 ***
*****************************************************************************
   Analyte:  Pb              Matrix:  DI Water (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:   5
      Date:  06/28/2000      Method:  1638
     Pairs:  5
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------------------------
Level          1          2          3          4          5          6          7          8          9          10
-------------------------------------------------------------------------------------------------------------------------------
Spike          .0192      .0000      .0300      .0060      .0640      .0000      .4000      .0000      4.5000      .0000
Spike
Increment      .0000      .0000      .0300      .0360      .1000      .1000      .5000      .5000      5.0000     5.0000
-------------------------------------------------------------------------------------------------------------------------------

                Final Concentration
          ------------------------------------------------------------------------------------------------------------------
Lab ID          .0192      .0192      .0492      .0552      .1192      .1192      .5192      .5192      5.0192     5.0192
-------------------------------------------------------------------------------------------------------------------------------
    1    r     .0004r     .0035  r   .0366r     .0271  r   .0790r     .0832  r   .4540r     .4510  r   4.2700r    4.2600
    2    o     .1292!     .0541  !   .1964!     .0852  o   .2634!     .1817  o   .6876!     .5714  !   5.1920!    5.0520
    3          .0103      .0145      .0605      .0519      .1167      .1194      .5013      .5341      4.9543     5.0154
    4          .0070      .0040      .0670      .0390      .1060      .1030      .5160      .5170      5.1970     5.2670
    5    ?     .0594o     .1421      .1423      .1253  ?   .1596      .1953      .4931      .5157      4.7988     4.8011
    6          .0208      .0291      .0528      .0777      .1160      .1135      .5201o    2.4625      5.0285o    6.3171
    7          .0051      .0052      .0375      .0366      .0996      .0974      .4792      .4807      4.8454     4.8580
    8    !    -.0016!     .0004  !   .0239!     .0290  !   .0943!     .0921  !   .4677!     .4772  !   4.8273!    4.9817
-------------------------------------------------------------------------------------------------------------------------------

Pb-5.daf
Pb     5   2 o             .0541     .1964     .0852o             .1817o             .5714    5.1920    5.0520
Pb     5   3      .0103     .0145     .0605     .0519     .1167     .1194     .5013     .5341    4.9543    5.0154
Pb     5   4      .0070     .0040     .0670     .0390     .1060     .1030     .5160     .5170    5.1970    5.2670
Pb     5   5      .0594o              .1423     .1253     .1596     .1953     .4931     .5157    4.7988    4.8011
Pb     5   6      .0208     .0291     .0528     .0777     .1160     .1135     .5201o              5.0285o
Pb     5   7      .0051     .0052     .0375     .0366     .0996     .0974     .4792     .4807    4.8454    4.8580
Pb     5   8     -.0016     .0004     .0239     .0290     .0943     .0921     .4677     .4772    4.8273    4.9817
```

```
File Name:  pb-5
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
------------------------------------------------------------
 Conc  Lab  Rep     Mean
 Lev   No   No     Result     Result                   Ratio
------------------------------------------------------------
  1      1  1       .0290      .0004                    .01
  1      7  1       .0290      .0051                    .18
  2      1  1       .0316      .0035                    .11
  2      4  1       .0316      .0040                    .13
  2      7  1       .0316      .0052                    .16
  2      8  1       .0316      .0004                    .01
------------------------------------------------------------
Total Number of Questionable Observations:     6


------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  pb-5
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***-------------------------------------***
 ***     Upper Critical Value:     65.0   ***
 ***     Lower Critical Value:     25.0   ***
-----------------------------------------------

      Ranks
     ------------------------------------------------------------------------------
       Rank  Level  Level  Level  Level  Level  Level  Level  Level  Level  Level
Lab    Sums    1      2      3      4      5      6      7      8      9     10
---- ------------------------------------------------------------------------------
  1    13.0   2.00   2.00   2.00   1.00   1.00   1.00   1.00   1.00   1.00   1.00
  2    73.0   8.00   7.00   8.00   7.00   8.00   7.00   8.00   7.00   7.00   6.00
  3    53.0   5.00   5.00   5.00   5.00   6.00   6.00   5.00   6.00   5.00   5.00
  4    51.0   4.00   3.00   6.00   4.00   4.00   4.00   6.00   5.00   8.00   7.00
  5    57.0   7.00   8.00   7.00   8.00   7.00   8.00   4.00   4.00   2.00   2.00
  6    61.0   6.00   6.00   4.00   6.00   5.00   5.00   7.00   8.00   6.00   8.00
  7    32.0   3.00   4.00   3.00   3.00   3.00   3.00   3.00   3.00   4.00   3.00
  8    20.0   1.00   1.00   1.00   2.00   2.00   2.00   2.00   2.00   3.00   4.00
-----------------------------------------------
 *** Laboratory   1 Rejected; Rank Sum   13.0 ***
-----------------------------------------------

File Name:  pb-5
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
------------------------------------------------------------------------------
  1     1     2   1       .1292      .0329      .0470   2.048   2.020    7
  2     1     5   1       .1421      .0356      .0506   2.106   2.020    7
  5     1     2   1       .2634      .1365      .0599   2.119   2.020    7
  7     1     2   1       .6876      .5236      .0747   2.196   2.020    7
  8     1     6   1      2.4625      .7941      .7364   2.266   2.020    7
  *     1     6   1      6.3171     5.1846      .5213   2.172   2.020    7
------------------------------------------------------------------------------
```

File Name: pb-5
Data Preparation File (.PRP)

Analyte: Pb Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Results of Normality Testing ***

- Normality Tests -

Level	n	Test Type	Normality Accept/Reject	Test Statistic	Critical Value(s)	
1	6	W	A	.7892	.788	---
2	6	W	A	.8524	.788	---
3	7	W	A	.8452	.803	---
4	7	W	A	.9003	.803	---
5	6	W	A	.8296	.788	---
6	7	W	A	.8060	.803	---
7	6	W	A	.9532	.788	---
8	6	W	A	.9313	.788	---
9	7	W	A	.8729	.803	---
10	6	W	A	.9498	.788	---

- 0 Normality Rejection(s) -

File Name: pb-5
Data Preparation File (.PRP)

Analyte: Pb Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Data Removal Tracking ***
*** Simple Count of Remaining Data Points ***
*** After Removal Tests ***

Lev	Mtrx	Points As Received	After Lab Ranking Points	After Lab Ranking %	After Outlier Testing Points	After Outlier Testing %
1	5	8	7	87.5	6	75.0
2	5	8	7	87.5	6	75.0
3	5	8	7	87.5	7	87.5
4	5	8	7	87.5	7	87.5
5	5	8	7	87.5	6	75.0
6	5	8	7	87.5	7	87.5
7	5	8	7	87.5	6	75.0
8	5	8	7	87.5	6	75.0
9	5	8	7	87.5	7	87.5
10	5	8	7	87.5	6	75.0
Totals:		80	70	87.5	64	80.0

File Name: pb-5
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Pb Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8	9	10
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)	Pair 5 (Low)	Pair 5 (High)
CONCENTRATION:	.0192	.0192	.0492	.0552	.1192	.1192	.5192	.5192	5.0192	5.0192
RECOVERY:										
Observations	6	6	7	7	6	7	6	6	7	6
Mean Result	.0168	.0179	.0829	.0635	.1154	.1289	.4962	.5160	4.9776	4.9959
Bias	-.0024	-.0013	.0337	.0083	-.0038	.0097	-.0230	-.0032	-.0416	-.0233
Relative Bias %	-12.3264	-6.8576	68.5250	15.0880	-3.2159	8.1496	-4.4235	-.6131	-.8285	-.4649
Maximum Result	.0594	.0541	.1964	.1253	.1596	.1953	.5201	.5714	5.1970	5.2670
Minimum Result	-.0016	.0004	.0239	.0290	.0943	.0921	.4677	.4772	4.7988	4.8011
SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4	Pair 5					
Observations	5	7	6	5	6					
Standard Deviation	.0030	.0310	.0108	.0098	.0692					
Correction Factor	1.0638	1.0424	1.0509	1.0638	1.0509					
Corrected Std Dev	.0032	.0323	.0114	.0104	.0728					
Relative Std Dev (%)	18.4662	44.1239	9.2727	2.0642	1.4595					
OVERALL PRECISION:										
Observations	6	6	7	7	6	7	6	6	7	6
Standard Deviation	.0221	.0205	.0627	.0345	.0234	.0419	.0205	.0351	.1680	.1638
Correction Factor	1.0509	1.0509	1.0424	1.0424	1.0509	1.0424	1.0509	1.0509	1.0424	1.0509
Corrected Std Dev	.0232	.0216	.0654	.0360	.0246	.0437	.0215	.0369	.1751	.1722
Relative Std Dev %	138.0434	120.6695	78.8657	56.6020	21.3205	33.8914	4.3405	7.1424	3.5173	3.4461

File Name: pb-5
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Pb Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.0192	.0168	-.0024	-12.33	.262	4.032	NO
2	.0192	.0179	-.0013	-6.86	.157	4.032	NO
3	.0492	.0829	.0337	68.52	1.422	3.707	NO
4	.0552	.0635	.0083	15.09	.639	3.707	NO
5	.1192	.1154	-.0038	-3.22	.401	4.032	NO
6	.1192	.1289	.0097	8.15	.613	3.707	NO
7	.5192	.4962	-.0230	-4.42	2.745	4.032	NO
8	.5192	.5160	-.0032	-.61	.222	4.032	NO
9	5.0192	4.9776	-.0416	-.83	.655	3.707	NO
10	5.0192	4.9959	-.0233	-.46	.349	4.032	NO

File Name: pb-5
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Single Operator Precision ***

Analyte: Pb Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Single Operator Precision ***

- Linear Model - s = b*T + a

Intercept (a): .0157
Slope (b): .0095

Pair	Sample Size	Weights (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	5	24.37	.0192	.0032	.0159
2	7	35.28	.0522	.0323	.0162
3	6	26.45	.1192	.0114	.0168
4	5	12.62	.5192	.0104	.0206
5	6	1.28	5.0192	.0728	.0634

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a: .0113 Intercept (a'): -4.4819
b: 1.4450 Slope (b'): .3681

Pair	Sample Size	Weight (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	5	16.54	.0192	.0032	.0114
2	7	25.20	.0192	.0323	.0115
3	6	20.86	.0492	.0114	.0118
4	5	16.54	.0552	.0104	.0137
5	6	20.86	.1192	.0728	.0718

```
File Name:  pb-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ***       Overall Precision        ***
---------------------------------------
Analyte:  Pb          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Overall Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .0329
          Slope (b):      .0243
    ============================
    Conc   Sample   Weights                Overall   Estimated
    Level  Size         (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6       13.98       .0192       .0232       .0334
      2       6       13.98       .0192       .0216       .0334
      3       7       16.00       .0492       .0654       .0341
      4       7       15.83       .0552       .0360       .0343
      5       6       11.75       .1192       .0246       .0358
      6       7       14.20       .1192       .0437       .0358
      7       6        6.63       .5192       .0215       .0456
      8       6        6.63       .5192       .0369       .0456
      9       7         .55      5.0192       .1751       .1550
     10       6         .46      5.0192       .1722       .1550
    ------------------------------------------------------------


    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ============================================================
      a:      .0309      Intercept (a'):    -3.4774
      b:     1.4050          Slope (b'):      .3400
    ============================================================
    Conc   Sample   Weight                Overall   Estimated
    Level  Size        (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6       9.23       .0192       .0232       .0311
      2       6       9.23       .0192       .0216       .0311
      3       7      11.15       .0492       .0654       .0314
      4       7      11.15       .0552       .0360       .0315
      5       6       9.23       .1192       .0246       .0322
      6       7      11.15       .1192       .0437       .0322
      7       6       9.23       .5192       .0215       .0369
      8       6       9.23       .5192       .0369       .0369
      9       7      11.15      5.0192       .1751       .1702
     10       6       9.23      5.0192       .1722       .1702
    ------------------------------------------------------------
```

```
File Name:  pb-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
---------------------------------------
Analyte:  Pb          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):      .0065
          Slope (b):      .9894
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       6     13.73       .0192       .0168       .0255
      2       6     13.73       .0192       .0179       .0255
      3       7     15.34       .0492       .0829       .0552
      4       7     15.21       .0552       .0635       .0612
      5       6     11.93       .1192       .1154       .1245
      6       7     13.92       .1192       .1289       .1245
      7       6      7.38       .5192       .4962       .5202
      8       6      7.38       .5192       .5160       .5202
      9       7       .74      5.0192      4.9776      4.9723
     10       6       .64      5.0192      4.9959      4.9723
    ------------------------------------------------------------


File Name:  pb-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
---------------------------------------
Analyte:  Pb          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .0091
            Slope (f):      .0096
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .0113
                  (f):     1.4507
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .0263
            Slope (f):      .0246
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .0308
                  (f):     1.4102
     ==================================
```

```
Pb-16.dat
Pb    16   1     0.0160    0.0131    0.0487    0.1090    0.1570    0.2200    0.2690    0.3220
Pb    16   2     0.0852    0.0770    0.1464    0.2292    0.3806    0.4883    0.3636    0.5197
Pb    16   3     0.0437    0.0486    0.0835    0.1106    0.2403    0.2358    0.3460    0.3924
Pb    16   4     0.0280    0.0310    0.0690    0.0820    0.2200    0.2220    0.3540    0.3860
Pb    16   5     0.0630    0.1640    0.0958    0.0835    0.3552    0.2326    0.4236    0.3797
Pb    16   6     0.0603    0.1225    0.0890    0.1112    0.2493    0.2500    0.3379    0.3967
Pb    16   7     0.0280    0.0278    0.0678    0.0791    0.2166    0.2143    0.3156    0.3653
Pb    16   8     0.0180    0.0211    0.0550    0.0670    0.2203    0.2217    0.2972    0.3550

File Name:  pb-16
Data Validation File (.DA~)

*****************************************************************************
***                    Parameter and Data Validation File                 ***
*****************************************************************************
   Analyte:  Pb              Matrix:  Freshwater (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:  16
      Date:  06/28/2000      Method:  1638
     Pairs:  4
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------
Level          1          2          3          4          5          6          7          8
-------------------------------------------------------------------------------------------------------------
Spike          .0429      .0000      .0400      .0100      .1400      .0000      .1100      .0500
Spike
Increment      .0000      .0000      .0400      .0500      .1900      .1900      .3000      .3500
-------------------------------------------------------------------------------------------------------------

               Final Concentration
          -------------------------------------------------------------------------------------------------
Lab ID         .0429      .0429      .0829      .0929      .2329      .2329      .3429      .3929
-------------------------------------------------------------------------------------------------------------
    1    !     .0160!     .0131  !   .0487!     .1090  !   .1570!     .2200  !   .2690!     .3220
    2    r     .0852r     .0770  r   .1464r     .2292  r   .3806r     .4883  r   .3636r     .5197
    3          .0437      .0486      .0835      .1106      .2403      .2358      .3460      .3924
    4          .0280      .0310      .0690      .0820      .2200      .2220      .3540      .3860
    5          .0630      .1640      .0958      .0835      .3552      .2326      .4236      .3797
    6          .0603      .1225      .0890      .1112      .2493      .2500      .3379      .3967
    7          .0280      .0278      .0678      .0791      .2166      .2143      .3156      .3653
    8          .0180      .0211      .0550      .0670      .2203      .2217      .2972      .3550
-------------------------------------------------------------------------------------------------------------

Pb-16.daf
Pb    16   1      .0160     .0131     .0487     .1090     .1570     .2200     .2690     .3220
Pb    16   3      .0437     .0486     .0835     .1106     .2403     .2358     .3460     .3924
Pb    16   4      .0280     .0310     .0690     .0820     .2200     .2220     .3540     .3860
Pb    16   5      .0630     .1640     .0958     .0835     .3552     .2326     .4236     .3797
Pb    16   6      .0603     .1225     .0890     .1112     .2493     .2500     .3379     .3967
Pb    16   7      .0280     .0278     .0678     .0791     .2166     .2143     .3156     .3653
Pb    16   8      .0180     .0211     .0550     .0670     .2203     .2217     .2972     .3550
```

```
File Name:  pb-16
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  pb-16
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***---------------------------------------***
 ***     Upper Critical Value:     54.0   ***
 ***     Lower Critical Value:     18.0   ***
-----------------------------------------------

      Ranks
     ---------------------------------------------------------------
      Rank  Level  Level  Level  Level  Level  Level  Level  Level
Lab    Sums    1      2      3      4      5      6      7      8
---- ----------------------------------------------------------------
  1    13.0   1.00   1.00   1.00   5.00   1.00   2.00   1.00   1.00
  2    61.0   8.00   6.00   8.00   8.00   8.00   8.00   7.00   8.00
  3    43.0   5.00   5.00   5.00   6.00   5.00   6.00   5.00   6.00
  4    32.5   3.50   4.00   4.00   3.00   3.00   4.00   6.00   5.00
  5    50.0   7.00   8.00   7.00   4.00   7.00   5.00   8.00   4.00
  6    50.0   6.00   7.00   6.00   7.00   6.00   7.00   4.00   7.00
  7    20.5   3.50   3.00   3.00   2.00   2.00   1.00   3.00   3.00
  8    18.0   2.00   2.00   2.00   1.00   4.00   3.00   2.00   2.00
-----------------------------------------------
 *** Laboratory   2 Rejected; Rank Sum   61.0 ***
-----------------------------------------------

File Name:  pb-16
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
-----------------------------------------


-----------------------------------------
 - No Outlier Rejections in Iteration 1 -
 - No Further Outlier Testing Necessary -
-----------------------------------------
```

```
File Name:  pb-16
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
              Test  Normality      Test          Critical
   Level  n   Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     7     W     A               .8818      .803   ---
     2     7     W           R         .7981      .803   ---
     3     7     W     A               .9509      .803   ---
     4     7     W     A               .8497      .803   ---
     5     7     W     A               .8633      .803   ---
     6     7     W     A               .9135      .803   ---
     7     7     W     A               .9576      .803   ---
     8     7     W     A               .8996      .803   ---
------------------------------
 - 1 Normality Rejection(s) -
------------------------------



File Name:  pb-16
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    16        8            7    87.5           7       87.5
  2    16        8            7    87.5           7       87.5
  3    16        8            7    87.5           7       87.5
  4    16        8            7    87.5           7       87.5
  5    16        8            7    87.5           7       87.5
  6    16        8            7    87.5           7       87.5
  7    16        8            7    87.5           7       87.5
  8    16        8            7    87.5           7       87.5
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         64           56    87.5          56       87.5
-----------------------------------------------------------------
```

File Name: pb-16
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Pb Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)
CONCENTRATION:	.0429	.0429	.0829	.0929	.2329	.2329	.3429	.3929
RECOVERY:								
Observations	7	7	7	7	7	7	7	7
Mean Result	.0367	.0612	.0727	.0918	.2370	.2281	.3348	.3710
Bias	-.0062	.0183	-.0102	-.0011	.0041	-.0048	-.0081	-.0219
Relative Bias %	-14.4189	42.5574	-12.3212	-1.2148	1.7420	-2.0794	-2.3747	-5.5703
Maximum Result	.0630	.1640	.0958	.1112	.3552	.2500	.4236	.3967
Minimum Result	.0160	.0131	.0487	.0670	.1570	.2143	.2690	.3220

SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4
Observations	7	7	7	7
Standard Deviation	.0288	.0156	.0392	.0258
Correction Factor	1.0424	1.0424	1.0424	1.0424
Corrected Std Dev	.0300	.0162	.0409	.0269
Relative Std Dev (%)	61.3004	19.7246	17.5916	7.6169

OVERALL PRECISION:								
Observations	7	7	7	7	7	7	7	7
Standard Deviation	.0193	.0584	.0175	.0181	.0599	.0122	.0492	.0262
Correction Factor	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424
Corrected Std Dev	.0201	.0608	.0183	.0189	.0624	.0127	.0512	.0273
Relative Std Dev %	54.6996	99.4633	25.1330	20.5646	26.3429	5.5770	15.3078	7.3518

File Name: pb-16
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Pb Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.0429	.0367	-.0062	-14.42	.849	3.707	NO
2	.0429	.0612	.0183	42.56	.828	3.707	NO
3	.0829	.0727	-.0102	-12.32	1.542	3.707	NO
4	.0929	.0918	-.0011	-1.21	.165	3.707	NO
5	.2329	.2370	.0041	1.74	.179	3.707	NO
6	.2329	.2281	-.0048	-2.08	1.050	3.707	NO
7	.3429	.3348	-.0081	-2.37	.438	3.707	NO
8	.3929	.3710	-.0219	-5.57	2.213	3.707	NO

```
File Name:  pb-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***   Single Operator Precision    ***
----------------------------------------
Analyte:  Pb          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .0244
          Slope (b):      .0224
    ============================
                                        Single
           Sample Weights              Operator   Estimated
    Pair   Size       (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       7     29.92       .0429       .0300       .0254
      2       7     27.97       .0879       .0162       .0264
      3       7     22.85       .2329       .0409       .0296
      4       7     19.25       .3679       .0269       .0327
    ------------------------------------------------------------



    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ======================================================
      a:      .0233      Intercept (a'):    -3.7603
      b:     2.2713          Slope (b'):      .8204
    ======================================================
                                                Single
           Sample Weight               Operator   Estimated
    Pair   Size      (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       7    25.00       .0429       .0300       .0241
      2       7    25.00       .0429       .0162       .0250
      3       7    25.00       .0829       .0409       .0282
      4       7    25.00       .0929       .0269       .0315
    ------------------------------------------------------------
```

File Name: pb-16
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Overall Precision ***

Analyte: Pb Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Overall Precision ***

- Linear Model - s = b*T + a

Intercept (a): .0302
Slope (b): .0207

Conc Level	Sample Size	Weights (%)	Conc	Overall Std Dev	Estimated Std Dev
1	7	14.76	.0429	.0201	.0311
2	7	14.76	.0429	.0608	.0311
3	7	13.97	.0829	.0183	.0319
4	7	13.79	.0929	.0189	.0321
5	7	11.52	.2329	.0624	.0350
6	7	11.52	.2329	.0127	.0350
7	7	10.12	.3429	.0512	.0373
8	7	9.56	.3929	.0273	.0383

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a: .0248 Intercept (a'): -3.6985
b: 2.2655 Slope (b'): .8178

Conc Level	Sample Size	Weight (%)	Conc	Overall Std Dev	Estimated Std Dev
1	7	12.50	.0429	.0201	.0256
2	7	12.50	.0429	.0608	.0256
3	7	12.50	.0829	.0183	.0265
4	7	12.50	.0929	.0189	.0267
5	7	12.50	.2329	.0624	.0300
6	7	12.50	.2329	.0127	.0300
7	7	12.50	.3429	.0512	.0328
8	7	12.50	.3929	.0273	.0341

```
File Name:  pb-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
----------------------------------------
Analyte:  Pb          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):      .0049
          Slope (b):      .9526
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       7     14.68       .0429       .0367       .0458
      2       7     14.68       .0429       .0612       .0458
      3       7     13.93       .0829       .0727       .0839
      4       7     13.75       .0929       .0918       .0934
      5       7     11.56       .2329       .2370       .2268
      6       7     11.56       .2329       .2281       .2268
      7       7     10.19       .3429       .3348       .3316
      8       7      9.65       .3929       .3710       .3792
    ------------------------------------------------------------


File Name:  pb-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
----------------------------------------
Analyte:  Pb          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .0193
            Slope (f):      .0235
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .0232
                  (f):     2.3660
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .0250
            Slope (f):      .0218
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .0247
                  (f):     2.3597
     ==================================
```

```
Ni-5.dat
Ni     5   1     0.0000    0.0000    0.4200    0.5230    0.9190    0.8190    8.9500    8.8200
Ni     5   2     0.0303    0.0256    0.5463    0.6395    1.4040    0.9972   10.1900   10.6900   98.7800   99.2600
Ni     5   3     0.0069    0.0152    0.4731    0.5952    0.9903    0.9265    9.2682    9.3891   91.9907   94.0182
Ni     5   4     0.0010    0.0030    1.5430    0.6310    0.9670    0.9890   10.2880    9.9170   97.5750   97.7140
Ni     5   5     3.3073   -0.0714    0.4734    0.5540    0.8445    0.9866    9.0384    9.3564
Ni     5   6     0.0312    0.2107    0.4697    0.8110    0.8887    0.9097    8.9976    9.4099   87.9207   89.4755
Ni     5   7     0.0077    0.0025    0.4378    0.5348    0.9141    0.9261    9.3127    9.1843   90.3164   92.2033
Ni     5   8     0.0083    0.0055    0.4448    0.5525    0.9131    0.9435    9.1786    9.1672   90.7248   94.5584

File Name:  ni-5
Data Validation File (.DA~)

*****************************************************************************
***                    Parameter and Data Validation File                 ***
*****************************************************************************
   Analyte:  Ni              Matrix:  DI Water (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:   5
      Date:  06/28/2000      Method:  1638
     Pairs:  5
     Units:  ug/L

-----------------------------------------------------------------------------------------------------------------------------
Level          1          2          3          4          5          6          7          8          9          10
-----------------------------------------------------------------------------------------------------------------------------
Spike          .0001      .0000       .5000      .1000       .4000      .0000      9.0000     .0000     90.0000     .0000
Spike
Increment      .0000      .0000       .5000      .6000      1.0000     1.0000     10.0000    10.0000    100.0000   100.0000
-----------------------------------------------------------------------------------------------------------------------------

                Final Concentration
            ---------------------------------------------------------------------------------------------------------------
Lab ID          .0001      .0001       .5001      .6001      1.0001     1.0001     10.0001    10.0001    100.0001   100.0001
-----------------------------------------------------------------------------------------------------------------------------
    1    !     .0000!     .0000  !     .4200!     .5230  !     .9190!     .8190  !    8.9500!    8.8200  e   88.9513e   88.9513
    2    r     .0303r     .0256  r     .5463r     .6395  r    1.4040r     .9972  r   10.1900r   10.6900  r   98.7800r   99.2600
    3          .0069      .0152        .4731      .5952       .9903      .9265       9.2682     9.3891      91.9907    94.0182
    4          .0010      .0030  o    1.5430     .6310       .9670      .9890  o   10.2880     9.9170      97.5750    97.7140
    5    o    3.3073?    -.0714        .4734      .5540       .8445      .9866       9.0384     9.3564  e   85.6877e   85.6877
    6    ?     .0312o     .2107        .4697o     .8110       .8887      .9097       8.9976     9.4099      87.9207    89.4755
    7          .0077      .0025        .4378      .5348       .9141      .9261       9.3127     9.1843      90.3164    92.2033
    8          .0083      .0055        .4448      .5525       .9131      .9435       9.1786     9.1672      90.7248    94.5584
-----------------------------------------------------------------------------------------------------------------------------

Ni-5.daf
Ni     5   1      .0000     .0000     .4200     .5230     .9190     .8190    8.9500    8.8200*         *
Ni     5   3      .0069     .0152     .4731     .5952     .9903     .9265    9.2682    9.3891   91.9907   94.0182
Ni     5   4      .0010     .0030o              .6310     .9670     .9890o             9.9170   97.5750   97.7140
Ni     5   5 o             -.0714     .4734     .5540     .8445     .9866    9.0384    9.3564*         *
Ni     5   6      .0312o              .4697o              .8887     .9097    8.9976    9.4099   87.9207   89.4755
Ni     5   7      .0077     .0025     .4378     .5348     .9141     .9261    9.3127    9.1843   90.3164   92.2033
Ni     5   8      .0083     .0055     .4448     .5525     .9131     .9435    9.1786    9.1672   90.7248   94.5584
```

```
File Name:  ni-5
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
------------------------------------------------------------
 Conc  Lab  Rep     Mean
 Lev   No   No     Result     Result                   Ratio
------------------------------------------------------------
  1      2  1       .4241      .0303                    .07
  1      3  1       .4241      .0069                    .02
  1      4  1       .4241      .0010                    .00
  1      5  1       .4241     3.3073                   7.80
  1      6  1       .4241      .0312                    .07
  1      7  1       .4241      .0077                    .02
  1      8  1       .4241      .0083                    .02
  2      4  1       .0328      .0030                    .09
  2      6  1       .0328      .2107                   6.42
  2      7  1       .0328      .0025                    .08
  2      8  1       .0328      .0055                    .17
------------------------------------------------------------
Total Number of Questionable Observations:    11


------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  ni-5
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***---------------------------------------***
 ***     Upper Critical Value:     65.0   ***
 ***     Lower Critical Value:     25.0   ***
-----------------------------------------------

      Ranks
     ------------------------------------------------------------------------------
       Rank  Level  Level  Level  Level  Level  Level  Level  Level  Level  Level
Lab    Sums    1      2      3      4      5      6      7      8      9     10
---- ------------------------------------------------------------------------------
  1    18.0   1.00   2.00   1.00   1.00   5.00   1.00   1.00   1.00   3.00   2.00
  2    74.0   6.00   7.00   7.00   7.00   8.00   8.00   7.00   8.00   8.00   8.00
  3    51.0   3.00   6.00   5.00   5.00   7.00   4.00   5.00   5.00   6.00   5.00
  4    62.0   2.00   4.00   8.00   6.00   6.00   7.00   8.00   7.00   7.00   7.00
  5    35.0   8.00   1.00   6.00   4.00   1.00   6.00   3.00   4.00   1.00   1.00
  6    44.0   7.00   8.00   4.00   8.00   2.00   2.00   2.00   6.00   2.00   3.00
  7    35.0   4.00   3.00   2.00   2.00   4.00   3.00   6.00   3.00   4.00   4.00
  8    41.0   5.00   5.00   3.00   3.00   3.00   5.00   4.00   2.00   5.00   6.00
-----------------------------------------------
 *** Laboratory   2 Rejected; Rank Sum   74.0 ***
-----------------------------------------------

File Name:  ni-5
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
------------------------------------------------------------------------------
  1     1     5   1      3.3073      .4803     1.2466   2.268   2.020    7
  2     1     6   1       .2107      .0236      .0874   2.140   2.020    7
  3     1     4   1      1.5430      .6088      .4124   2.265   2.020    7
  4     1     6   1       .8110      .6002      .1000   2.107   2.020    7
  7     1     4   1     10.2880     9.2905      .4607   2.165   2.020    7
------------------------------------------------------------------------------
```

File Name: ni-5
Data Preparation File (.PRP)

Analyte: Ni Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Results of Normality Testing ***

- Normality Tests -

Level	n	Test Type	Normality Accept/Reject	Test Statistic	Critical Value(s)	
1	6	W	R	.7638	.788	---
2	6	W	R	.6518	.788	---
3	6	W	A	.8669	.788	---
4	6	W	A	.9108	.788	---
5	7	W	A	.9578	.803	---
6	7	W	A	.8844	.803	---
7	6	W	A	.9147	.788	---
8	7	W	A	.9328	.803	---
9	5	W	A	.8989	.762	---
10	5	W	A	.9877	.762	---

- 2 Normality Rejection(s) -

File Name: ni-5
Data Preparation File (.PRP)

Analyte: Ni Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Data Removal Tracking ***
*** Simple Count of Remaining Data Points ***
*** After Removal Tests ***

Lev	Mtrx	Points As Received	After Lab Ranking Points	After Lab Ranking %	After Outlier Testing Points	After Outlier Testing %
1	5	8	7	87.5	6	75.0
2	5	8	7	87.5	6	75.0
3	5	8	7	87.5	6	75.0
4	5	8	7	87.5	6	75.0
5	5	8	7	87.5	7	87.5
6	5	8	7	87.5	7	87.5
7	5	8	7	87.5	6	75.0
8	5	8	7	87.5	7	87.5
9	5	6	5	83.3	5	83.3
10	5	6	5	83.3	5	83.3
Totals:		76	66	86.8	61	80.3

File Name: ni-5
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Ni Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8	9	10
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)	Pair 5 (Low)	Pair 5 (High)
CONCENTRATION:	.0001	.0001	.5001	.6001	1.0001	1.0001	10.0001	10.0001	100.0001	100.0001
RECOVERY:										
Observations	6	6	6	6	7	7	6	7	5	5
Mean Result	.0092	-.0075	.4531	.5651	.9195	.9286	9.1243	9.3206	91.7055	93.5939
Bias	.0091	-.0076	-.0470	-.0350	-.0806	-.0715	-.8758	-.6795	-8.2946	-6.4062
Relative Bias %	9083.3330	*********	-9.3915	-5.8351	-8.0563	-7.1464	-8.7584	-6.7954	-8.2946	-6.4062
Maximum Result	.0312	.0152	.4734	.6310	.9903	.9890	9.3127	9.9170	97.5750	97.7140
Minimum Result	.0000	-.0714	.4200	.5230	.8445	.8190	8.9500	8.8200	87.9207	89.4755
SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4	Pair 5					
Observations	5	5	7	6	5					
Standard Deviation	.0037	.0107	.0545	.1621	.9337					
Correction Factor	1.0638	1.0638	1.0424	1.0509	1.0638					
Corrected Std Dev	.0039	.0114	.0568	.1704	.9933					
Relative Std Dev (%)	470.9625	2.2414	6.1504	1.8462	1.0722					
OVERALL PRECISION:										
Observations	6	6	6	6	7	7	6	7	5	5
Standard Deviation	.0114	.0317	.0223	.0405	.0482	.0572	.1503	.3323	3.5968	3.0405
Correction Factor	1.0509	1.0509	1.0509	1.0509	1.0424	1.0424	1.0509	1.0424	1.0638	1.0638
Corrected Std Dev	.0119	.0333	.0234	.0426	.0502	.0596	.1579	.3463	3.8264	3.2346
Relative Std Dev %	129.9005	-442.6499	5.1719	7.5412	5.4588	6.4177	1.7308	3.7157	4.1725	3.4560

File Name: ni-5
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Ni Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.0001	.0092	.0091	******	1.956	4.032	NO
2	.0001	-.0075	-.0076	******	.589	4.032	NO
3	.5001	.4531	-.0470	-9.39	5.159	4.032	YES
4	.6001	.5651	-.0350	-5.84	2.115	4.032	NO
5	1.0001	.9195	-.0806	-8.06	4.427	3.707	YES
6	1.0001	.9286	-.0715	-7.15	3.307	3.707	NO
7	10.0001	9.1243	-.8758	-8.76	14.277	4.032	YES
8	10.0001	9.3206	-.6795	-6.80	5.411	3.707	YES
9	100.0001	91.7055	-8.2946	-8.29	5.157	4.604	YES
10	100.0001	93.5939	-6.4062	-6.41	4.711	4.604	YES

File Name: ni-5
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Single Operator Precision ***

Analyte: Ni Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Single Operator Precision ***

- Linear Model - s = b*T + a

Intercept (a): .0187
Slope (b): .0128

Pair	Sample Size	Weights (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	5	35.77	.0001	.0039	.0187
2	5	27.11	.5501	.0114	.0257
3	7	33.78	1.0001	.0568	.0315
4	6	3.30	10.0001	.1704	.1471
5	5	.05	100.0001	.9933	1.3025

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a: .0265 Intercept (a'): -3.6320
b: 1.0389 Slope (b'): .0382

Pair	Sample Size	Weight (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	5	17.29	.0001	.0039	.0265
2	5	17.29	.0001	.0114	.0270
3	7	26.33	.5001	.0568	.0275
4	6	21.80	.6001	.1704	.0388
5	5	17.29	1.0001	.9933	1.2049

File Name: ni-5
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Overall Precision ***

Analyte: Ni Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Overall Precision ***

- Linear Model - s = b*T + a

Intercept (a): .0242
Slope (b): .0241

Conc Level	Sample Size	Weights (%)	Conc	Overall Std Dev	Estimated Std Dev
1	6	14.25	.0001	.0119	.0242
2	6	14.25	.0001	.0333	.0242
3	6	13.63	.5001	.0234	.0362
4	6	13.51	.6001	.0426	.0386
5	7	15.74	1.0001	.0502	.0482
6	7	15.74	1.0001	.0596	.0482
7	6	5.83	10.0001	.1579	.2647
8	7	7.04	10.0001	.3463	.2647
9	5	.00	100.0001	3.8264	2.4295
10	5	.00	100.0001	3.2346	2.4295

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a: .0474 Intercept (a'): -3.0485
b: 1.0457 Slope (b'): .0447

Conc Level	Sample Size	Weight (%)	Conc	Overall Std Dev	Estimated Std Dev
1	6	9.80	.0001	.0119	.0474
2	6	9.80	.0001	.0333	.0474
3	6	9.80	.5001	.0234	.0485
4	6	9.80	.6001	.0426	.0487
5	7	11.83	1.0001	.0502	.0496
6	7	11.83	1.0001	.0596	.0496
7	6	9.80	10.0001	.1579	.0742
8	7	11.83	10.0001	.3463	.0742
9	5	7.77	100.0001	3.8264	4.1440
10	5	7.77	100.0001	3.2346	4.1440

```
File Name:  ni-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
---------------------------------------
Analyte:  Ni          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):      .0002
          Slope (b):      .9246
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       6     29.04       .0001       .0092       .0003
      2       6     29.04       .0001      -.0075       .0003
      3       6     12.96       .5001       .4531       .4626
      4       6     11.39       .6001       .5651       .5551
      5       7      8.52      1.0001       .9195       .9249
      6       7      8.52      1.0001       .9286       .9249
      7       6       .24     10.0001      9.1243      9.2461
      8       7       .28     10.0001      9.3206      9.2461
      9       5       .00    100.0001     91.7055     92.4586
     10       5       .00    100.0001     93.5939     92.4586
    ------------------------------------------------------------


File Name:  ni-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
---------------------------------------
Analyte:  Ni          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .0184
            Slope (f):      .0139
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .0265
                  (f):     1.0422
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .0240
            Slope (f):      .0260
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .0474
                  (f):     1.0495
     ==================================
```

```
Ni-16.dat
Ni    16   1     0.4940    0.5130    1.7200    1.8800    3.2800    3.8600    5.1800    5.7500
Ni    16   2     0.8529    0.7177    1.9840    2.2680    3.7520    4.4710    6.9970    6.7370
Ni    16   3     0.6528    0.6711    1.6941    1.9425    3.1383    3.5813    6.1252    5.8139
Ni    16   4     0.6570    0.6600    1.9240    2.1060    3.5990    4.1060    6.2400    6.1380
Ni    16   5     0.9110    3.1600    2.0831    2.3118    3.5877    4.2850    6.9102    6.2186
Ni    16   6     0.0000    0.0000    1.0149    1.1366    2.4122    3.0205    5.6038    4.6596
Ni    16   7     0.4790    0.4592    1.6675    1.8115    3.1570    3.8903    5.7563    5.7324
Ni    16   8     0.6062    0.6288    1.8047    1.9671    3.2257    3.9768    6.0458    5.8885

File Name:  ni-16
Data Validation File (.DA~)

*****************************************************************************
***                    Parameter and Data Validation File                 ***
*****************************************************************************
   Analyte:  Ni              Matrix:  Freshwater (using mean as true)
   Project:  EPA/EPRI     Matrix ID:  16
      Date:  06/28/2000      Method:  1638
     Pairs:  4
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------
Level          1          2           3          4           5          6           7          8
-------------------------------------------------------------------------------------------------------------
Spike          .6716      .0000       1.3000      .2000       1.5000      .7000       2.0000      .0000
Spike
Increment      .0000      .0000       1.3000     1.5000       3.0000     3.7000       5.7000     5.7000
-------------------------------------------------------------------------------------------------------------

               Final Concentration
          ------------------------------------------------------------------------------------------------
Lab ID         .6716      .6716       1.9716     2.1716       3.6716     4.3716       6.3716     6.3716
-------------------------------------------------------------------------------------------------------------
    1          .4940      .5130       1.7200     1.8800       3.2800     3.8600       5.1800     5.7500
    2    !     .8529!     .7177  !    1.9840!    2.2680  !    3.7520!    4.4710  !    6.9970!    6.7370
    3          .6528      .6711       1.6941     1.9425       3.1383     3.5813       6.1252     5.8139
    4          .6570      .6600       1.9240     2.1060       3.5990     4.1060       6.2400     6.1380
    5    !     .9110o    3.1600  !    2.0831!    2.3118  !    3.5877!    4.2850  !    6.9102!    6.2186
    6    r     .0000r     .0000  r    1.0149r    1.1366  r    2.4122r    3.0205  r    5.6038r    4.6596
    7          .4790      .4592       1.6675     1.8115       3.1570     3.8903       5.7563     5.7324
    8          .6062      .6288       1.8047     1.9671       3.2257     3.9768       6.0458     5.8885
-------------------------------------------------------------------------------------------------------------

Ni-16.daf
Ni    16   1      .4940     .5130    1.7200    1.8800    3.2800    3.8600    5.1800    5.7500
Ni    16   2      .8529     .7177    1.9840    2.2680    3.7520    4.4710    6.9970    6.7370
Ni    16   3      .6528     .6711    1.6941    1.9425    3.1383    3.5813    6.1252    5.8139
Ni    16   4      .6570     .6600    1.9240    2.1060    3.5990    4.1060    6.2400    6.1380
Ni    16   5      .9110o             2.0831    2.3118    3.5877    4.2850    6.9102    6.2186
Ni    16   7      .4790     .4592    1.6675    1.8115    3.1570    3.8903    5.7563    5.7324
Ni    16   8      .6062     .6288    1.8047    1.9671    3.2257    3.9768    6.0458    5.8885
```

```
File Name:  ni-16
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  Freshwater (using mean as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  ni-16
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  Freshwater (using mean as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***---------------------------------------***
 ***     Upper Critical Value:     54.0   ***
 ***     Lower Critical Value:     18.0   ***
-----------------------------------------------

      Ranks
     ------------------------------------------------------------------
      Rank  Level  Level  Level  Level  Level  Level  Level  Level
Lab    Sums    1      2      3      4      5      6      7      8
---- ------------------------------------------------------------------
  1    25.0   3.00   3.00   4.00   3.00   5.00   3.00   1.00   3.00
  2    60.0   7.00   7.00   7.00   7.00   8.00   8.00   8.00   8.00
  3    31.0   5.00   6.00   3.00   4.00   2.00   2.00   5.00   4.00
  4    48.0   6.00   5.00   6.00   6.00   7.00   6.00   6.00   6.00
  5    59.0   8.00   8.00   8.00   8.00   6.00   7.00   7.00   7.00
  6     9.0   1.00   1.00   1.00   1.00   1.00   1.00   2.00   1.00
  7    20.0   2.00   2.00   2.00   2.00   3.00   4.00   3.00   2.00
  8    36.0   4.00   4.00   5.00   5.00   4.00   5.00   4.00   5.00
-------------------------------------------------
 *** Laboratory   6 Rejected; Rank Sum    9.0 ***
-------------------------------------------------

File Name:  ni-16
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  Freshwater (using mean as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
-------------------------------------------------------------------------------
  2     1     5   1      3.1600      .9728      .9688   2.258   2.020    7
-------------------------------------------------------------------------------
```

```
File Name:  ni-16
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  Freshwater (using mean as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
              Test  Normality      Test          Critical
   Level  n   Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     7     W     A               .9083      .803   ---
     2     6     W     A               .9045      .788   ---
     3     7     W     A               .9181      .803   ---
     4     7     W     A               .9182      .803   ---
     5     7     W     A               .8683      .803   ---
     6     7     W     A               .9837      .803   ---
     7     7     W     A               .9475      .803   ---
     8     7     W     A               .8485      .803   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  ni-16
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  Freshwater (using mean as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     ----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    16        8            7    87.5           7       87.5
  2    16        8            7    87.5           6       75.0
  3    16        8            7    87.5           7       87.5
  4    16        8            7    87.5           7       87.5
  5    16        8            7    87.5           7       87.5
  6    16        8            7    87.5           7       87.5
  7    16        8            7    87.5           7       87.5
  8    16        8            7    87.5           7       87.5
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         64           56    87.5          55       85.9
-----------------------------------------------------------------
```

File Name: ni-16
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Ni Matrix: Freshwater (using mean as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)
CONCENTRATION:	.6716	.6716	1.9716	2.1716	3.6716	4.3716	6.3716	6.3716
RECOVERY:								
Observations	7	6	7	7	7	7	7	7
Mean Result	.6647	.6083	1.8396	2.0410	3.3914	4.0243	6.1792	6.0398
Bias	-.0069	-.0633	-.1320	-.1306	-.2802	-.3473	-.1924	-.3318
Relative Bias %	-1.0274	-9.4253	-6.6936	-6.0147	-7.6319	-7.9435	-3.0194	-5.2079
Maximum Result	.9110	.7177	2.0831	2.3118	3.7520	4.4710	6.9970	6.7370
Minimum Result	.4790	.4592	1.6675	1.8115	3.1383	3.5813	5.1800	5.7324
SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4				
Observations	6	7	7	7				
Standard Deviation	.0430	.0373	.0868	.2688				
Correction Factor	1.0509	1.0424	1.0424	1.0424				
Corrected Std Dev	.0452	.0389	.0904	.2802				
Relative Std Dev (%)	7.0724	2.0031	2.4389	4.5868				
OVERALL PRECISION:								
Observations	7	6	7	7	7	7	7	7
Standard Deviation	.1650	.1003	.1600	.1927	.2485	.2937	.6331	.3606
Correction Factor	1.0424	1.0509	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424
Corrected Std Dev	.1720	.1054	.1668	.2009	.2590	.3062	.6599	.3759
Relative Std Dev %	25.8729	17.3315	9.0651	9.8423	7.6379	7.6078	10.6794	6.2230

File Name: ni-16
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Ni Matrix: Freshwater (using mean as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.6716	.6647	-.0069	-1.03	.111	3.707	NO
2	.6716	.6083	-.0633	-9.43	1.546	4.032	NO
3	1.9716	1.8396	-.1320	-6.69	2.182	3.707	NO
4	2.1716	2.0410	-.1306	-6.01	1.793	3.707	NO
5	3.6716	3.3914	-.2802	-7.63	2.983	3.707	NO
6	4.3716	4.0243	-.3473	-7.94	3.128	3.707	NO
7	6.3716	6.1792	-.1924	-3.02	.804	3.707	NO
8	6.3716	6.0398	-.3318	-5.21	2.435	3.707	NO

```
File Name:  ni-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***   Single Operator Precision    ***
----------------------------------------
Analyte:  Ni          Matrix:  Freshwater (using mean as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .0248
          Slope (b):      .0180
    ============================
                                          Single
           Sample Weights                Operator   Estimated
    Pair   Size       (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6     63.11       .6716       .0452       .0369
      2       7     28.37      2.0716       .0389       .0622
      3       7      7.16      4.0216       .0904       .0973
      4       7      1.36      6.3716       .2802       .1397
    ------------------------------------------------------------



    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ======================================================
      a:      .0252      Intercept (a'):    -3.6790
      b:     1.4233          Slope (b'):      .3530
    ======================================================
                                                 Single
           Sample Weight                       Operator   Estimated
    Pair   Size      (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6    21.63       .6716       .0452       .0320
      2       7    26.12       .6716       .0389       .0525
      3       7    26.12      1.9716       .0904       .1044
      4       7    26.12      2.1716       .2802       .2393
    ------------------------------------------------------------
```

File Name: ni-16
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Overall Precision ***

Analyte: Ni Matrix: Freshwater (using mean as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Overall Precision ***

- Linear Model - s = b*T + a

Intercept (a): .0978
Slope (b): .0538

Conc Level	Sample Size	Weights (%)	Conc	Overall Std Dev	Estimated Std Dev
1	7	36.90	.6716	.1720	.1340
2	6	30.55	.6716	.1054	.1340
3	7	11.27	1.9716	.1668	.2039
4	7	9.86	2.1716	.2009	.2146
5	7	4.48	3.6716	.2590	.2953
6	7	3.38	4.3716	.3062	.3330
7	7	1.78	6.3716	.6599	.4406
8	7	1.78	6.3716	.3759	.4406

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a: .1158 Intercept (a'): -2.1558
b: 1.2544 Slope (b'): .2266

Conc Level	Sample Size	Weight (%)	Conc	Overall Std Dev	Estimated Std Dev
1	7	12.77	.6716	.1720	.1348
2	6	10.58	.6716	.1054	.1348
3	7	12.77	1.9716	.1668	.1810
4	7	12.77	2.1716	.2009	.1894
5	7	12.77	3.6716	.2590	.2661
6	7	12.77	4.3716	.3062	.3119
7	7	12.77	6.3716	.6599	.4907
8	7	12.77	6.3716	.3759	.4907

```
File Name:  ni-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
----------------------------------------
Analyte:  Ni          Matrix:  Freshwater (using mean as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):     -.0046
          Slope (b):      .9433
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       7     30.95       .6716       .6647       .6290
      2       6     26.53       .6716       .6083       .6290
      3       7     13.36      1.9716      1.8396      1.8553
      4       7     12.06      2.1716      2.0410      2.0440
      5       7      6.37      3.6716      3.3914      3.4590
      6       7      5.01      4.3716      4.0243      4.1193
      7       7      2.86      6.3716      6.1792      6.0060
      8       7      2.86      6.3716      6.0398      6.0060
    ------------------------------------------------------------


File Name:  ni-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
----------------------------------------
Analyte:  Ni          Matrix:  Freshwater (using mean as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .0297
            Slope (f):      .0191
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .0253
                  (f):     1.4538
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .1027
            Slope (f):      .0570
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .1159
                  (f):     1.2716
     ==================================
```

```
Se-5.dat
Se     5   1     0.0000    0.0000    0.9430    1.0700    5.6400    5.3500   22.4000   22.3000
Se     5   2     0.4753    0.2495    0.9426    0.6453    4.5400    5.9650   18.7500   19.3300   95.3600   96.5400
Se     5   3     0.0862   -0.1122    1.1618    1.3055    5.0323    4.9782   20.9289   21.1048  113.2210  113.7347
Se     5   4    -0.0010    0.0100    0.8750    1.2170    5.0620    4.9660   21.1320   21.1440  106.7670  106.0850
Se     5   5    -0.0605    0.1500    1.6423    1.7892    4.5147    4.4402   17.8544   18.3850
Se     5   6    20.6772   20.3840   21.7354   20.3721   24.9791   24.8939   37.9875   39.8635
Se     5   7     0.0101    0.0261    0.8322    0.9676    4.3423    4.1599   18.1392   18.1058   89.5153   91.3544
Se     5   8     0.1219    0.1280    1.0135    1.2068    4.6020    4.9030   19.0698   18.7166   92.8644   97.3893
Note: Highest pair deleted due to insufficient data


File Name:  se-5
Data Validation File (.DA~)

*************************************************************************
***                  Parameter and Data Validation File                 ***
*************************************************************************
   Analyte:  Se              Matrix:  DI Water (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:   5
      Date:  07/31/2000       Method:  1638
     Pairs:  4
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------
Level          1          2           3          4           5          6           7          8
-------------------------------------------------------------------------------------------------------------
Spike          .0318      .0000       1.0000      .2000       3.8000      .0000      15.0000      .0000
Spike
Increment      .0000      .0000       1.0000     1.2000       5.0000     5.0000      20.0000    20.0000
-------------------------------------------------------------------------------------------------------------

                Final Concentration
        -----------------------------------------------------------------------------------------------------
Lab ID          .0318      .0318       1.0318     1.2318       5.0318     5.0318      20.0318    20.0318
-------------------------------------------------------------------------------------------------------------
    1           .0000      .0000        .9430     1.0700       5.6400     5.3500      22.4000    22.3000
    2    o      .4753      .2495        .9426      .6453       4.5400     5.9650      18.7500    19.3300
    3           .0862     -.1122       1.1618     1.3055       5.0323     4.9782      20.9289    21.1048
    4          -.0010      .0100        .8750     1.2170       5.0620     4.9660      21.1320    21.1440
    5          -.0605      .1500  o    1.6423     1.7892       4.5147     4.4402      17.8544    18.3850
    6    r   20.6772r   20.3840  r   21.7354r   20.3721  r   24.9791r   24.8939  r   37.9875r   39.8635
    7    !      .0101!     .0261  !     .8322!     .9676  !    4.3423!    4.1599  !   18.1392!   18.1058
    8           .1219      .1280       1.0135     1.2068       4.6020     4.9030      19.0698    18.7166
-------------------------------------------------------------------------------------------------------------


Se-5.daf
Se     5   1      .0000     .0000     .9430    1.0700    5.6400    5.3500   22.4000   22.3000
Se     5   2 o              .2495     .9426     .6453    4.5400    5.9650   18.7500   19.3300
Se     5   3      .0862    -.1122    1.1618    1.3055    5.0323    4.9782   20.9289   21.1048
Se     5   4     -.0010     .0100     .8750    1.2170    5.0620    4.9660   21.1320   21.1440
Se     5   5     -.0605     .1500o             1.7892    4.5147    4.4402   17.8544   18.3850
Se     5   7      .0101     .0261     .8322     .9676    4.3423    4.1599   18.1392   18.1058
Se     5   8      .1219     .1280    1.0135    1.2068    4.6020    4.9030   19.0698   18.7166
```

```
File Name:  se-5
Data Preparation File (.PRP)


Analyte:  Se     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  07/31/2000
  Units:  ug/L
------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
------------------------------------------------------------
 Conc  Lab  Rep     Mean
 Lev   No   No     Result     Result                   Ratio
------------------------------------------------------------
  1      2  1      2.6713      .4753                    .18
  1      3  1      2.6713      .0862                    .03
  1      6  1      2.6713    20.6772                   7.74
  1      7  1      2.6713      .0101                    .00
  1      8  1      2.6713      .1219                    .05
  2      2  1      2.6185      .2495                    .10
  2      4  1      2.6185      .0100                    .00
  2      5  1      2.6185      .1500                    .06
  2      6  1      2.6185    20.3840                   7.78
  2      7  1      2.6185      .0261                    .01
  2      8  1      2.6185      .1280                    .05
  3      6  1      3.6432    21.7354                   5.97
  4      2  1      3.5717      .6453                    .18
  4      6  1      3.5717    20.3721                   5.70
------------------------------------------------------------
Total Number of Questionable Observations:    14


------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  se-5
Data Preparation File (.PRP)


Analyte:  Se     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  07/31/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***-------------------------------------***
 ***     Upper Critical Value:     54.0   ***
 ***     Lower Critical Value:     18.0   ***
-----------------------------------------------

      Ranks
     ------------------------------------------------------------------
       Rank  Level  Level  Level  Level  Level  Level  Level  Level
Lab    Sums    1      2      3      4      5      6      7      8
---- ------------------------------------------------------------------
  1    39.0   3.00   2.00   4.00   3.00   7.00   6.00   7.00   7.00
  2    35.0   7.00   7.00   3.00   1.00   3.00   7.00   3.00   4.00
  3    38.0   5.00   1.00   6.00   6.00   5.00   5.00   5.00   5.00
  4    34.0   2.00   3.00   2.00   5.00   6.00   4.00   6.00   6.00
  5    28.0   1.00   6.00   7.00   7.00   2.00   2.00   1.00   2.00
  6    64.0   8.00   8.00   8.00   8.00   8.00   8.00   8.00   8.00
  7    16.0   4.00   4.00   1.00   2.00   1.00   1.00   2.00   1.00
  8    34.0   6.00   5.00   5.00   4.00   4.00   3.00   4.00   3.00
-----------------------------------------------
 *** Laboratory   6 Rejected; Rank Sum   64.0 ***
-----------------------------------------------

File Name:  se-5
Data Preparation File (.PRP)


Analyte:  Se     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  07/31/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
------------------------------------------------------------------------------
  1     1     2   1       .4753      .0903      .1802   2.136   2.020    7
  3     1     5   1      1.6423     1.0586      .2784   2.096   2.020    7
------------------------------------------------------------------------------
```

File Name: se-5
Data Preparation File (.PRP)

Analyte: Se Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 07/31/2000
Units: ug/L

*** Results of Normality Testing ***

- Normality Tests -

Level	n	Test Type	Normality Accept/Reject	Test Statistic	Critical Value(s)	
1	6	W	A	.9267	.788	---
2	7	W	A	.9675	.803	---
3	6	W	A	.9293	.788	---
4	7	W	A	.9553	.803	---
5	7	W	A	.8905	.803	---
6	7	W	A	.9597	.803	---
7	7	W	A	.9107	.803	---
8	7	W	A	.8959	.803	---

- 0 Normality Rejection(s) -

File Name: se-5
Data Preparation File (.PRP)

Analyte: Se Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 07/31/2000
Units: ug/L

*** Data Removal Tracking ***
*** Simple Count of Remaining Data Points ***
*** After Removal Tests ***

Lev	Mtrx	Points As Received	After Lab Ranking Points	%	After Outlier Testing Points	%
1	5	8	7	87.5	6	75.0
2	5	8	7	87.5	7	87.5
3	5	8	7	87.5	6	75.0
4	5	8	7	87.5	7	87.5
5	5	8	7	87.5	7	87.5
6	5	8	7	87.5	7	87.5
7	5	8	7	87.5	7	87.5
8	5	8	7	87.5	7	87.5
Totals:		64	56	87.5	54	84.4

File Name: se-5
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Se Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 07/31/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)
CONCENTRATION:	.0318	.0318	1.0318	1.2318	5.0318	5.0318	20.0318	20.0318
RECOVERY:								
Observations	6	7	6	7	7	7	7	7
Mean Result	.0261	.0645	.9613	1.1716	4.8190	4.9660	19.7535	19.8695
Bias	-.0057	.0327	-.0705	-.0602	-.2128	-.0658	-.2783	-.1623
Relative Bias %	-17.8721	102.7853	-6.8279	-4.8848	-4.2283	-1.3068	-1.3894	-.8104
Maximum Result	.1219	.2495	1.1618	1.7892	5.6400	5.9650	22.4000	22.3000
Minimum Result	-.0605	-.1122	.8322	.6453	4.3423	4.1599	17.8544	18.1058

SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4
Observations	6	6	7	7
Standard Deviation	.0915	.1512	.4188	.2399
Correction Factor	1.0509	1.0509	1.0424	1.0424
Corrected Std Dev	.0962	.1589	.4366	.2501
Relative Std Dev (%)	205.6023	14.7896	8.9231	1.2623

OVERALL PRECISION:								
Observations	6	7	6	7	7	7	7	7
Standard Deviation	.0663	.1193	.1164	.3492	.4515	.5882	1.7307	1.6326
Correction Factor	1.0509	1.0424	1.0509	1.0424	1.0424	1.0424	1.0424	1.0424
Corrected Std Dev	.0696	.1244	.1223	.3640	.4707	.6131	1.8040	1.7017
Relative Std Dev %	266.6845	192.8380	12.7228	31.0658	9.7669	12.3459	9.1327	8.5646

File Name: se-5
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Se Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 07/31/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.0318	.0261	-.0057	-17.87	.210	4.032	NO
2	.0318	.0645	.0327	102.79	.725	3.707	NO
3	1.0318	.9613	-.0705	-6.83	1.483	4.032	NO
4	1.2318	1.1716	-.0602	-4.88	.456	3.707	NO
5	5.0318	4.8190	-.2128	-4.23	1.247	3.707	NO
6	5.0318	4.9660	-.0658	-1.31	.296	3.707	NO
7	20.0318	19.7535	-.2783	-1.39	.425	3.707	NO
8	20.0318	19.8695	-.1623	-.81	.263	3.707	NO

File Name: se-5
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Single Operator Precision ***

Analyte: Se Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 07/31/2000
Units: ug/L

*** Single Operator Precision ***

- Linear Model - s = b*T + a

Intercept (a): .2034
Slope (b): .0061

Pair	Sample Size	Weights (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	6	27.62	.0318	.0962	.2036
2	6	26.54	1.1318	.1589	.2102
3	7	27.99	5.0318	.4366	.2340
4	7	17.86	20.0318	.2501	.3253

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a: .1739 Intercept (a'): -1.7492
b: 1.0280 Slope (b'): .0276

Pair	Sample Size	Weight (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	6	22.65	.0318	.0962	.1741
2	6	22.65	.0318	.1589	.1794
3	7	27.35	1.0318	.4366	.1998
4	7	27.35	1.2318	.2501	.3024

```
File Name:  se-5
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***       Overall Precision        ***
----------------------------------------
Analyte:  Se          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  07/31/2000
  Units:  ug/L

 *** Overall Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .1085
          Slope (b):      .0882
    ============================
    Conc   Sample   Weights                Overall   Estimated
    Level  Size         (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6       31.72       .0318       .0696       .1114
      2       7       38.31       .0318       .1244       .1114
      3       6       12.19      1.0318       .1223       .1995
      4       7       12.71      1.2318       .3640       .2172
      5       7        2.32      5.0318       .4707       .5522
      6       7        2.32      5.0318       .6131       .5522
      7       7         .22     20.0318      1.8040      1.8747
      8       7         .22     20.0318      1.7017      1.8747
    ------------------------------------------------------------


    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ============================================================
      a:      .1680      Intercept (a'):    -1.7835
      b:     1.1322          Slope (b'):      .1242
    ============================================================
    Conc   Sample   Weight                Overall   Estimated
    Level  Size        (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6      10.82       .0318       .0696       .1687
      2       7      13.06       .0318       .1244       .1687
      3       6      10.82      1.0318       .1223       .1910
      4       7      13.06      1.2318       .3640       .1958
      5       7      13.06      5.0318       .4707       .3139
      6       7      13.06      5.0318       .6131       .3139
      7       7      13.06     20.0318      1.8040      2.0228
      8       7      13.06     20.0318      1.7017      2.0228
    ------------------------------------------------------------
```

```
File Name:  se-5
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
----------------------------------------
Analyte:  Se          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  07/31/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):      .0035
          Slope (b):      .9766
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       6     34.63       .0318       .0261       .0345
      2       7     40.40       .0318       .0645       .0345
      3       6     10.79      1.0318       .9613      1.0111
      4       7     10.62      1.2318      1.1716      1.2065
      5       7      1.64      5.0318      4.8190      4.9176
      6       7      1.64      5.0318      4.9660      4.9176
      7       7       .14     20.0318     19.7535     19.5670
      8       7       .14     20.0318     19.8695     19.5670
    ------------------------------------------------------------


File Name:  se-5
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
----------------------------------------
Analyte:  Se          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  07/31/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .1998
            Slope (f):      .0062
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .1739
                  (f):     1.0287
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .1050
            Slope (f):      .0903
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .1680
                  (f):     1.1356
     ==================================
```

Se-16.dat

```
Se    16   1     0.2890    0.1760    0.9890    1.0400    1.4000    2.2900    3.7900    4.1900
Se    16   2     0.1495   -0.2973    0.0069    1.4730    1.3650    1.2640    3.9550    3.5530
Se    16   3     0.1444    0.1777    0.7959    1.1142    2.0142    2.2558    3.7678    3.9146
Se    16   4     0.1020    0.1380    0.6450    0.9580    1.8240    2.1020    3.7410    3.7900
Se    16   5     0.4310    0.0000    1.4480    1.4273    2.2192    2.6318    3.8992    3.9011
Se    16   6    19.7109   19.0204   20.3957   20.7004   21.1349   20.6159   27.4137   23.3279
Se    16   7     0.1985    0.2125    0.7142    0.8711    1.5490    2.0332    3.4203    3.5249
Se    16   8     0.2646    0.2360    0.8178    1.0490    1.8828    2.1701    3.4754    3.6753
```

```
File Name:  se-16
Data Validation File (.DA~)

*****************************************************************************
***                  Parameter and Data Validation File                   ***
*****************************************************************************
   Analyte:  Se              Matrix:  Freshwater (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:  16
      Date:  06/28/2000      Method:  1638
     Pairs:  4
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------
Level          1          2           3          4           5          6           7          8
-------------------------------------------------------------------------------------------------------------
Spike          .2055      .0000       .6000      .2000       .8000      .4000       1.6000     .0000
Spike
Increment      .0000      .0000       .6000      .8000       1.6000     2.0000      3.6000     3.6000
-------------------------------------------------------------------------------------------------------------

                Final Concentration
          ----------------------------------------------------------------------------------------------
Lab ID         .2055      .2055       .8055     1.0055       1.8055     2.2055      3.8055     3.8055
-------------------------------------------------------------------------------------------------------------
    1          .2890      .1760       .9890     1.0400       1.4000     2.2900      3.7900     4.1900
    2          .1495o    -.2973       .0069     1.4730       1.3650     1.2640      3.9550     3.5530
    3          .1444      .1777       .7959     1.1142       2.0142     2.2558      3.7678     3.9146
    4          .1020      .1380       .6450      .9580       1.8240     2.1020      3.7410     3.7900
    5          .4310      .0000      1.4480     1.4273       2.2192     2.6318      3.8992     3.9011
    6    r   19.7109r   19.0204  r   20.3957r   20.7004  r   21.1349r   20.6159  r   27.4137r   23.3279
    7          .1985      .2125       .7142      .8711       1.5490     2.0332      3.4203     3.5249
    8          .2646      .2360       .8178     1.0490       1.8828     2.1701      3.4754     3.6753
-------------------------------------------------------------------------------------------------------------
```

Se-16.daf

```
Se    16   1      .2890     .1760     .9890    1.0400    1.4000    2.2900    3.7900    4.1900
Se    16   2      .1495o              .0069    1.4730    1.3650    1.2640    3.9550    3.5530
Se    16   3      .1444     .1777     .7959    1.1142    2.0142    2.2558    3.7678    3.9146
Se    16   4      .1020     .1380     .6450     .9580    1.8240    2.1020    3.7410    3.7900
Se    16   5      .4310     .0000    1.4480    1.4273    2.2192    2.6318    3.8992    3.9011
Se    16   7      .1985     .2125     .7142     .8711    1.5490    2.0332    3.4203    3.5249
Se    16   8      .2646     .2360     .8178    1.0490    1.8828    2.1701    3.4754    3.6753
```

File Name: se-16
Data Preparation File (.PRP)

Analyte: Se Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Results of 5x - 1/5x Mean Error Check ***
*** Questionable Data (Positive Values) ***

Conc Lev	Lab No	Rep No	Mean Result	Result	Ratio
1	1	1	2.6612	.2890	.11
1	2	1	2.6612	.1495	.06
1	3	1	2.6612	.1444	.05
1	4	1	2.6612	.1020	.04
1	5	1	2.6612	.4310	.16
1	6	1	2.6612	19.7109	7.41
1	7	1	2.6612	.1985	.07
1	8	1	2.6612	.2646	.10
2	1	1	2.4951	.1760	.07
2	3	1	2.4951	.1777	.07
2	4	1	2.4951	.1380	.06
2	6	1	2.4951	19.0204	7.62
2	7	1	2.4951	.2125	.09
2	8	1	2.4951	.2360	.09
3	2	1	3.2266	.0069	.00
3	4	1	3.2266	.6450	.20
3	6	1	3.2266	20.3957	6.32
4	6	1	3.5791	20.7004	5.78
5	6	1	4.1736	21.1349	5.06

Total Number of Questionable Observations: 19

*** Results of Factor of 5 Error Check ***
*** Questionable Data (All Values) ***

Total Number of Questionable Observations: 0

File Name: se-16
Data Preparation File (.PRP)
Analyte: Se Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Laboratory Ranking Results ***
*** Two-Tailed 5% Significance Level ***
*** Upper Critical Value: 54.0 ***
*** Lower Critical Value: 18.0 ***

	Ranks							
Lab	Rank Sums	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1	39.0	6.00	4.00	6.00	3.00	2.00	6.00	5.00	7.00
2	23.0	3.00	1.00	1.00	7.00	1.00	1.00	7.00	2.00
3	37.0	2.00	5.00	4.00	5.00	6.00	5.00	4.00	6.00
4	22.0	1.00	3.00	2.00	2.00	4.00	3.00	3.00	4.00
5	47.0	7.00	2.00	7.00	6.00	7.00	7.00	6.00	5.00
6	64.0	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00
7	21.0	4.00	6.00	3.00	1.00	3.00	2.00	1.00	1.00
8	35.0	5.00	7.00	5.00	4.00	5.00	4.00	2.00	3.00

*** Laboratory 6 Rejected; Rank Sum 64.0 ***

File Name: se-16
Data Preparation File (.PRP)

Analyte: Se Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Outlier Testing Results ***
*** Two-Sided 5% Significance Level ***

- Outlier(s) -

Lev	Iter	Lab	Rep	Result	Mean	Std Dev	t	Crit t	n
2	1	2	1	-.2973	.0918	.1879	2.071	2.020	7

File Name: se-16
Data Preparation File (.PRP)

Analyte: Se Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Results of Normality Testing ***

- Normality Tests -

Level	n	Test Type	Normality Accept/Reject	Test Statistic	Critical Value(s)	
1	7	W	A	.9226	.803	---
2	6	W	A	.8505	.788	---
3	7	W	A	.9305	.803	---
4	7	W	A	.8774	.803	---
5	7	W	A	.9384	.803	---
6	7	W	A	.8621	.803	---
7	7	W	A	.8977	.803	---
8	7	W	A	.9435	.803	---

- 0 Normality Rejection(s) -

File Name: se-16
Data Preparation File (.PRP)

Analyte: Se Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Data Removal Tracking ***
*** Simple Count of Remaining Data Points ***
*** After Removal Tests ***

Lev	Mtrx	Points As Received	After Lab Ranking Points	%	After Outlier Testing Points	%
1	16	8	7	87.5	7	87.5
2	16	8	7	87.5	6	75.0
3	16	8	7	87.5	7	87.5
4	16	8	7	87.5	7	87.5
5	16	8	7	87.5	7	87.5
6	16	8	7	87.5	7	87.5
7	16	8	7	87.5	7	87.5
8	16	8	7	87.5	7	87.5
Totals:		64	56	87.5	55	85.9

File Name: se-16
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Se Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)
CONCENTRATION:	.2055	.2055	.8055	1.0055	1.8055	2.2055	3.8055	3.8055
RECOVERY:								
Observations	7	6	7	7	7	7	7	7
Mean Result	.2256	.1567	.7738	1.1332	1.7506	2.1067	3.7212	3.7927
Bias	.0201	-.0488	-.0317	.1277	-.0549	-.0988	-.0843	-.0128
Relative Bias %	9.7671	-23.7470	-3.9319	12.7030	-3.0407	-4.7497	-2.2141	-.3364
Maximum Result	.4310	.2360	1.4480	1.4730	2.2192	2.6318	3.9550	4.1900
Minimum Result	.1020	.0000	.0069	.8711	1.3650	1.2640	3.4203	3.5249

SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4
Observations	6	7	7	7
Standard Deviation	.1273	.3566	.2116	.1734
Correction Factor	1.0509	1.0424	1.0424	1.0424
Corrected Std Dev	.1338	.3717	.2206	.1807
Relative Std Dev (%)	69.0362	38.9779	11.4386	4.8103

OVERALL PRECISION:								
Observations	7	6	7	7	7	7	7	7
Standard Deviation	.1126	.0838	.4304	.2300	.3225	.4186	.2019	.2336
Correction Factor	1.0424	1.0509	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424
Corrected Std Dev	.1174	.0881	.4486	.2398	.3362	.4363	.2105	.2435
Relative Std Dev %	52.0499	56.2083	57.9776	21.1585	19.2048	20.7094	5.6558	6.4201

File Name: se-16
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Se Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.2055	.2256	.0201	9.77	.471	3.707	NO
2	.2055	.1567	-.0488	-23.75	1.426	4.032	NO
3	.8055	.7738	-.0317	-3.93	.195	3.707	NO
4	1.0055	1.1332	.1277	12.70	1.469	3.707	NO
5	1.8055	1.7506	-.0549	-3.04	.450	3.707	NO
6	2.2055	2.1067	-.0988	-4.48	.625	3.707	NO
7	3.8055	3.7212	-.0843	-2.21	1.104	3.707	NO
8	3.8055	3.7927	-.0128	-.34	.145	3.707	NO

```
File Name:  se-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***   Single Operator Precision    ***
----------------------------------------
Analyte:  Se          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .2568
          Slope (b):     -.0144
    ============================
                                          Single
           Sample Weights                Operator   Estimated
    Pair   Size       (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6     21.63       .2055       .1338       .2538
      2       7     26.12       .9055       .3717       .2437
      3       7     26.12      2.0055       .2206       .2279
      4       7     26.12      3.8055       .1807       .2020
    ------------------------------------------------------------



    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ======================================================
      a:      .2271      Intercept (a'):    -1.4821
      b:      .9708           Slope (b'):     -.0296
    ======================================================
                                                Single
           Sample Weight                      Operator   Estimated
    Pair   Size      (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6    21.63       .2055       .1338       .2258
      2       7    26.12       .2055       .3717       .2211
      3       7    26.12       .8055       .2206       .2141
      4       7    26.12      1.0055       .1807       .2030
    ------------------------------------------------------------
```

```
File Name:  se-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***       Overall Precision        ***
----------------------------------------
Analyte:  Se          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Overall Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .2268
          Slope (b):      .0241
    ============================
    Conc   Sample   Weights                Overall   Estimated
    Level  Size         (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       7       15.16       .2055       .1174       .2318
      2       6       12.55       .2055       .0881       .2318
      3       7       14.06       .8055       .4486       .2463
      4       7       13.73      1.0055       .2398       .2511
      5       7       12.49      1.8055       .3362       .2704
      6       7       11.93      2.2055       .4363       .2800
      7       7       10.04      3.8055       .2105       .3187
      8       7       10.04      3.8055       .2435       .3187
    ------------------------------------------------------------


    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ============================================================
      a:      .1891      Intercept (a'):    -1.6656
      b:     1.1363          Slope (b'):      .1278
    ============================================================
    Conc   Sample   Weight                Overall   Estimated
    Level  Size        (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       7      12.77       .2055       .1174       .1941
      2       6      10.58       .2055       .0881       .1941
      3       7      12.77       .8055       .4486       .2096
      4       7      12.77      1.0055       .2398       .2150
      5       7      12.77      1.8055       .3362       .2381
      6       7      12.77      2.2055       .4363       .2506
      7       7      12.77      3.8055       .2105       .3075
      8       7      12.77      3.8055       .2435       .3075
    ------------------------------------------------------------
```

```
File Name:  se-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
----------------------------------------
Analyte:  Se          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):      .0116
          Slope (b):      .9806
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       7     16.47       .2055       .2256       .2132
      2       6     14.11       .2055       .1567       .2132
      3       7     14.59       .8055       .7738       .8015
      4       7     14.03      1.0055      1.1332       .9976
      5       7     12.10      1.8055      1.7506      1.7821
      6       7     11.28      2.2055      2.1067      2.1744
      7       7      8.71      3.8055      3.7212      3.7433
      8       7      8.71      3.8055      3.7927      3.7433
    ------------------------------------------------------------


File Name:  se-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
----------------------------------------
Analyte:  Se          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .2449
            Slope (f):     -.0147
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .2272
                  (f):      .9703
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .2149
            Slope (f):      .0246
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .1888
                  (f):     1.1392
     ==================================
```

```
Ag-5.dat
Ag     5   1     0.0000    0.0000    0.0000    0.0000    0.0000   0.0000    0.0370    0.0357    6.7900    6.5800
Ag     5   2     0.0020   -0.0009    0.0091    0.0038    0.0347   0.0139    0.3276    0.3034    9.3500    9.4880
Ag     5   3     0.0003    0.0010    0.0870    0.0908    0.1611   0.2192    0.9276    0.8484   10.0446    9.9293
Ag     5   4     0.0050    0.0050    0.0970    0.0840    0.1560   0.2280    0.9000    0.9400    9.9770   10.1060
Ag     5   5     0.0576    0.0540    0.0542    0.0515    0.0510   0.0510    0.0935    0.0713    8.8829    8.4280
Ag     5   6     0.0105    0.0078    0.0107    0.0082    0.0879   0.0697    0.3156    0.3295   10.4827   10.2625
Ag     5   7     0.0096    0.0038    0.0019    0.0040   -0.0015  -0.0005    0.0061    0.0027    8.5905    8.9060
Ag     5   8     0.0177    0.0126    0.0015    0.0012    0.0014   0.0000    0.0012    0.0960    7.7363    7.3608

File Name:  ag-5
Data Validation File (.DA~)

*****************************************************************************
***                    Parameter and Data Validation File                 ***
*****************************************************************************
   Analyte:  Ag              Matrix:  DI Water (using mean as true)
   Project:  EPA/EPRI     Matrix ID:   5
      Date:  07/13/2000      Method:  1638
     Pairs:  5
     Units:  ug/L

-----------------------------------------------------------------------------------------------------------------------------
Level          1          2          3          4          5          6          7          8          9          10
-----------------------------------------------------------------------------------------------------------------------------
Spike          .0048      .0000      .1000      .0000      .1000      .0400      .7600      .0000     9.0000      .0000
Spike
Increment      .0000      .0000      .1000      .1000      .2000      .2400     1.0000     1.0000    10.0000    10.0000
-----------------------------------------------------------------------------------------------------------------------------

                Final Concentration
           -----------------------------------------------------------------------------------------------------------------
Lab ID          .0048      .0048      .1048      .1048      .2048      .2448     1.0048     1.0048    10.0048    10.0048
-----------------------------------------------------------------------------------------------------------------------------
    1    r     .0000r     .0000  r    .0000r     .0000  r    .0000r     .0000  r    .0370r     .0357  r   6.7900r    6.5800
    2          .0020     -.0009       .0091      .0038       .0347      .0139       .3276      .3034      9.3500     9.4880
    3          .0003      .0010       .0870      .0908       .1611      .2192       .9276      .8484     10.0446     9.9293
    4    !     .0050!     .0050  !    .0970!     .0840  !    .1560!     .2280  !    .9000!     .9400  !   9.9770!   10.1060
    5    o     .0576o     .0540       .0542      .0515       .0510      .0510       .0935      .0713      8.8829     8.4280
    6          .0105      .0078       .0107      .0082       .0879      .0697       .3156      .3295     10.4827    10.2625
    7          .0096      .0038       .0019      .0040      -.0015     -.0005       .0061      .0027      8.5905     8.9060
    8          .0177      .0126       .0015      .0012       .0014      .0000       .0012      .0960      7.7363     7.3608
-----------------------------------------------------------------------------------------------------------------------------

Ag-5.daf
g      5   2      .0020    -.0009     .0091     .0038     .0347     .0139     .3276     .3034    9.3500    9.4880
Ag     5   3      .0003     .0010     .0870     .0908     .1611     .2192     .9276     .8484   10.0446    9.9293
Ag     5   4      .0050     .0050     .0970     .0840     .1560     .2280     .9000     .9400    9.9770   10.1060
Ag     5   5 o         o             .0542     .0515     .0510     .0510     .0935     .0713    8.8829    8.4280
Ag     5   6      .0105     .0078     .0107     .0082     .0879     .0697     .3156     .3295   10.4827   10.2625
Ag     5   7      .0096     .0038     .0019     .0040    -.0015    -.0005     .0061     .0027    8.5905    8.9060
Ag     5   8      .0177     .0126     .0015     .0012     .0014     .0000     .0012     .0960    7.7363    7.3608
```

```
File Name:  ag-5
Data Preparation File (.PRP)


Analyte:  Ag     Matrix:  DI Water (using mean as true)
Project:  EPA/EPRI
   Date:  07/13/2000
  Units:  ug/L
------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
------------------------------------------------------------
 Conc  Lab  Rep     Mean
 Lev   No   No     Result     Result                   Ratio
------------------------------------------------------------
  1      2  1       .0128      .0020                    .16
  1      3  1       .0128      .0003                    .02
  2      3  1       .0105      .0010                    .10
  2      5  1       .0105      .0540                   5.13
  3      7  1       .0327      .0019                    .06
  3      8  1       .0327      .0015                    .05
  4      2  1       .0304      .0038                    .12
  4      7  1       .0304      .0040                    .13
  4      8  1       .0304      .0012                    .04
  5      8  1       .0615      .0014                    .02
  6      2  1       .0727      .0139                    .19
  7      1  1       .3261      .0370                    .11
  7      7  1       .3261      .0061                    .02
  7      8  1       .3261      .0012                    .00
  8      1  1       .3284      .0357                    .11
  8      7  1       .3284      .0027                    .01
------------------------------------------------------------
Total Number of Questionable Observations:    16


------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  ag-5
Data Preparation File (.PRP)


Analyte:  Ag     Matrix:  DI Water (using mean as true)
Project:  EPA/EPRI
   Date:  07/13/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***---------------------------------------***
 ***     Upper Critical Value:     65.0   ***
 ***     Lower Critical Value:     25.0   ***
-----------------------------------------------

      Ranks
     ---------------------------------------------------------------------------
       Rank  Level  Level  Level  Level  Level  Level  Level  Level  Level  Level
Lab    Sums    1      2      3      4      5      6      7      8      9     10
---- ----------------------------------------------------------------------------
  1    16.5   1.00   2.00   1.00   1.00   2.00   2.50   3.00   2.00   1.00   1.00
  2    40.0   3.00   1.00   4.00   3.00   4.00   4.00   6.00   5.00   5.00   5.00
  3    63.0   2.00   3.00   7.00   8.00   8.00   7.00   8.00   7.00   7.00   6.00
  4    67.0   4.00   5.00   8.00   7.00   7.00   8.00   7.00   8.00   6.00   7.00
  5    52.0   8.00   8.00   6.00   6.00   5.00   5.00   4.00   3.00   4.00   3.00
  6    61.0   6.00   6.00   5.00   5.00   6.00   6.00   5.00   6.00   8.00   8.00
  7    28.0   5.00   4.00   3.00   4.00   1.00   1.00   2.00   1.00   3.00   4.00
  8    32.5   7.00   7.00   2.00   2.00   3.00   2.50   1.00   4.00   2.00   2.00
-----------------------------------------------
 *** Laboratory   1 Rejected; Rank Sum   16.5 ***
-----------------------------------------------

File Name:  ag-5
Data Preparation File (.PRP)


Analyte:  Ag     Matrix:  DI Water (using mean as true)
Project:  EPA/EPRI
   Date:  07/13/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------
--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
-------------------------------------------------------------------------
  1     1     5   1       .0576      .0147      .0198   2.166   2.020    7
  2     1     5   1       .0540      .0119      .0191   2.206   2.020    7
-------------------------------------------------------------------------
```

File Name: ag-5
Data Preparation File (.PRP)

Analyte: Ag Matrix: DI Water (using mean as true)
Project: EPA/EPRI
Date: 07/13/2000
Units: ug/L

*** Results of Normality Testing ***

- Normality Tests -

Level	n	Test Type	Normality Accept/Reject	Test Statistic	Critical Value(s)	
1	6	W	A	.9494	.788	---
2	6	W	A	.9712	.788	---
3	7	W	A	.8108	.803	---
4	7	W	R	.7865	.803	---
5	7	W	A	.8830	.803	---
6	7	W	R	.7903	.803	---
7	7	W	A	.8248	.803	---
8	7	W	A	.8459	.803	---
9	7	W	A	.9604	.803	---
10	7	W	A	.9134	.803	---

- 2 Normality Rejection(s) -

File Name: ag-5
Data Preparation File (.PRP)

Analyte: Ag Matrix: DI Water (using mean as true)
Project: EPA/EPRI
Date: 07/13/2000
Units: ug/L

*** Data Removal Tracking ***
*** Simple Count of Remaining Data Points ***
*** After Removal Tests ***

Lev	Mtrx	Points As Received	After Lab Ranking Points	%	After Outlier Testing Points	%
1	5	8	7	87.5	6	75.0
2	5	8	7	87.5	6	75.0
3	5	8	7	87.5	7	87.5
4	5	8	7	87.5	7	87.5
5	5	8	7	87.5	7	87.5
6	5	8	7	87.5	7	87.5
7	5	8	7	87.5	7	87.5
8	5	8	7	87.5	7	87.5
9	5	8	7	87.5	7	87.5
10	5	8	7	87.5	7	87.5
Totals:		80	70	87.5	68	85.0

File Name: ag-5
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Ag Matrix: DI Water (using mean as true)
Project: EPA/EPRI Method: 1638
Date: 07/13/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8	9	10
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)	Pair 5 (Low)	Pair 5 (High)
CONCENTRATION:	.0048	.0048	.1048	.1048	.2048	.2448	1.0048	1.0048	10.0048	10.0048
RECOVERY:										
Observations	6	6	7	7	7	7	7	7	7	7
Mean Result	.0075	.0049	.0373	.0348	.0701	.0830	.3674	.3702	9.2949	9.2115
Bias	.0027	.0001	-.0675	-.0700	-.1347	-.1618	-.6374	-.6346	-.7099	-.7933
Relative Bias %	56.5972	1.7361	-64.3675	-66.8075	-65.7785	-66.0773	-63.4384	-63.1583	-7.0960	-7.9290
Maximum Result	.0177	.0126	.0970	.0908	.1611	.2280	.9276	.9400	10.4827	10.2625
Minimum Result	.0003	-.0009	.0015	.0012	-.0015	-.0005	.0012	.0027	7.7363	7.3608

SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4	Pair 5
Observations	6	7	7	7	7
Standard Deviation	.0019	.0039	.0261	.0388	.2034
Correction Factor	1.0509	1.0424	1.0424	1.0424	1.0424
Corrected Std Dev	.0019	.0041	.0272	.0405	.2120
Relative Std Dev (%)	31.3624	11.3114	35.4821	10.9736	2.2909

OVERALL PRECISION:										
Observations	6	6	7	7	7	7	7	7	7	7
Standard Deviation	.0064	.0049	.0415	.0400	.0676	.0995	.3961	.3784	.9606	1.0511
Correction Factor	1.0509	1.0509	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424
Corrected Std Dev	.0067	.0051	.0433	.0417	.0705	.1037	.4129	.3944	1.0013	1.0956
Relative Std Dev %	89.6967	104.4453	115.9728	119.7454	100.5792	124.9036	112.3891	106.5438	10.7722	11.8938

File Name: ag-5
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Ag Matrix: DI Water (using mean as true)
Project: EPA/EPRI Method: 1638
Date: 07/13/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.0048	.0075	.0027	56.60	1.034	4.032	NO
2	.0048	.0049	.0001	1.74	.042	4.032	NO
3	.1048	.0373	-.0675	-64.37	4.296	3.707	YES
4	.1048	.0348	-.0700	-66.81	4.635	3.707	YES
5	.2048	.0701	-.1347	-65.78	5.270	3.707	YES
6	.2448	.0830	-.1618	-66.08	4.301	3.707	YES
7	1.0048	.3674	-.6374	-63.44	4.258	3.707	YES
8	1.0048	.3702	-.6346	-63.16	4.437	3.707	YES
9	10.0048	9.2949	-.7099	-7.10	1.955	3.707	NO
10	10.0048	9.2115	-.7933	-7.93	1.997	3.707	NO

File Name: ag-5
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Single Operator Precision ***

Analyte: Ag Matrix: DI Water (using mean as true)
Project: EPA/EPRI Method: 1638
Date: 07/13/2000
Units: ug/L

*** Single Operator Precision ***

- Linear Model - s = b*T + a

Intercept (a): .0068
Slope (b): .0273

Pair	Sample Size	Weights (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	6	37.02	.0048	.0019	.0069
2	7	32.77	.1048	.0041	.0097
3	7	23.84	.2248	.0272	.0129
4	7	6.23	1.0048	.0405	.0342
5	7	.14	10.0048	.2120	.2795

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a: .0089 Intercept (a'): -4.7172
b: 1.3905 Slope (b'): .3297

Pair	Sample Size	Weight (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	6	17.15	.0048	.0019	.0090
2	7	20.71	.0048	.0041	.0093
3	7	20.71	.1048	.0272	.0096
4	7	20.71	.1048	.0405	.0125
5	7	20.71	.2048	.2120	.2420

```
File Name:  ag-5
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***       Overall Precision        ***
----------------------------------------
Analyte:  Ag          Matrix:  DI Water (using mean as true)
Project:  EPA/EPRI    Method:  1638
   Date:  07/13/2000
  Units:  ug/L

 *** Overall Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .0464
          Slope (b):      .1568
    ============================
    Conc   Sample   Weights                Overall   Estimated
    Level  Size         (%)        Conc     Std Dev     Std Dev
    ---------------------------------------------------------------
      1       6       15.78       .0048       .0067       .0471
      2       6       15.78       .0048       .0051       .0471
      3       7       15.92       .1048       .0433       .0628
      4       7       15.92       .1048       .0417       .0628
      5       7       13.49       .2048       .0705       .0785
      6       7       12.67       .2448       .1037       .0848
      7       7        5.05      1.0048       .4129       .2040
      8       7        5.05      1.0048       .3944       .2040
      9       7         .18     10.0048      1.0013      1.6156
     10       7         .18     10.0048      1.0956      1.6156
    ---------------------------------------------------------------


    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ============================================================
      a:      .0510      Intercept (a'):    -2.9759
      b:     1.3776          Slope (b'):      .3203
    ============================================================
    Conc   Sample   Weight                Overall   Estimated
    Level  Size        (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6       8.57       .0048       .0067       .0511
      2       6       8.57       .0048       .0051       .0511
      3       7      10.36       .1048       .0433       .0527
      4       7      10.36       .1048       .0417       .0527
      5       7      10.36       .2048       .0705       .0545
      6       7      10.36       .2448       .1037       .0552
      7       7      10.36      1.0048       .4129       .0704
      8       7      10.36      1.0048       .3944       .0704
      9       7      10.36     10.0048      1.0013      1.2573
     10       7      10.36     10.0048      1.0956      1.2573
    ------------------------------------------------------------
```

```
File Name:  ag-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
---------------------------------------
Analyte:  Ag          Matrix:  DI Water (using mean as true)
Project:  EPA/EPRI    Method:  1638
   Date:  07/13/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):     -.0337
          Slope (b):      .6983
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       6     23.69       .0048       .0075      -.0304
      2       6     23.69       .0048       .0049      -.0304
      3       7     15.56       .1048       .0373       .0394
      4       7     15.56       .1048       .0348       .0394
      5       7      9.96       .2048       .0701       .1093
      6       7      8.54       .2448       .0830       .1372
      7       7      1.48      1.0048       .3674       .6679
      8       7      1.48      1.0048       .3702       .6679
      9       7       .02     10.0048      9.2949      6.9526
     10       7       .02     10.0048      9.2115      6.9526
    ------------------------------------------------------------


File Name:  ag-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
---------------------------------------
Analyte:  Ag          Matrix:  DI Water (using mean as true)
Project:  EPA/EPRI    Method:  1638
   Date:  07/13/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .0551
            Slope (f):      .0390
    ============================


     - Curvilinear Model - s = e*(f**X)
    ===================================
        Intercept (e):      .0091
                  (f):     1.6034
    ===================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .0947
            Slope (f):      .2246
     ============================


     - Curvilinear Model - s = e*(f**X)
     ===================================
        Intercept (e):      .0518
                  (f):     1.5821
     ===================================
```

```
Ag-16.dat
Ag    16   1     0.0052    0.0000    0.0109    0.0221    0.0974    0.0934    0.0546    0.1100
Ag    16   2     0.0052    0.0021    0.0139    0.0269    0.1545    0.1189    0.0289    0.0618
Ag    16   3     0.0028    0.0015    0.0636    0.0640    0.1929    0.1901    0.2361    0.3016
Ag    16   4     0.0420    0.0090    0.0800    0.0940    0.1910    0.2040    0.2930    0.3580
Ag    16   5     0.0460    0.0450    0.0640    0.0671    0.1265    0.1113    0.0972    0.0880
Ag    16   6     0.0115    0.0217    0.0189    0.0178    0.1373    0.1275    0.0637    0.0508
Ag    16   7     0.0107    0.0080    0.0200    0.0261    0.0637    0.0646    0.0453    0.0396
Ag    16   8     0.0022   0.0017     0.0142    0.0171    0.0826    0.0839    0.0380    0.0448

File Name:  ag-16
Data Validation File (.DA~)

*****************************************************************************
***                    Parameter and Data Validation File                 ***
*****************************************************************************
   Analyte:  Ag              Matrix:  Freshwater (using mean as true)
   Project:  EPA/EPRI     Matrix ID:  16
      Date:  07/13/2000      Method:  1638
     Pairs:  4
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------
Level          1          2           3          4           5          6           7          8
-------------------------------------------------------------------------------------------------------------
Spike          .0311      .0000       .0700      .0100       .1200      .0000       .1000      .0600
Spike
Increment      .0000      .0000       .0700      .0800       .2000      .2000       .3000      .3600
-------------------------------------------------------------------------------------------------------------

               Final Concentration
          ---------------------------------------------------------------------------------------------------
Lab ID         .0311      .0311       .1011      .1111       .2311      .2311       .3311      .3911
-------------------------------------------------------------------------------------------------------------
    1          .0052      .0000       .0109      .0221       .0974      .0934       .0546      .1100
    2          .0052      .0021       .0139      .0269       .1545      .1189       .0289      .0618
    3          .0028      .0015       .0636      .0640       .1929      .1901  o    .2361o     .3016
    4    r     .0420r     .0090  r    .0800r     .0940  r    .1910r     .2040  r    .2930r     .3580
    5    o     .0460o     .0450       .0640      .0671       .1265      .1113       .0972      .0880
    6          .0115?     .0217       .0189      .0178       .1373      .1275       .0637      .0508
    7          .0107      .0080       .0200      .0261       .0637      .0646       .0453      .0396
    8    !     .0022!     .0017  !    .0142!     .0171  !    .0826!     .0839  !    .0380!     .0448
-------------------------------------------------------------------------------------------------------------

Ag-16.daf
Ag    16   1      .0052     .0000     .0109     .0221     .0974     .0934     .0546     .1100
Ag    16   2      .0052     .0021     .0139     .0269     .1545     .1189     .0289     .0618
Ag    16   3      .0028     .0015     .0636     .0640     .1929     .1901o         o
Ag    16   5 o         o              .0640     .0671     .1265     .1113     .0972     .0880
Ag    16   6      .0115     .0217     .0189     .0178     .1373     .1275     .0637     .0508
Ag    16   7      .0107     .0080     .0200     .0261     .0637     .0646     .0453     .0396
Ag    16   8      .0022     .0017     .0142     .0171     .0826     .0839     .0380     .0448
```

File Name: ag-16
Data Preparation File (.PRP)

Analyte: Ag Matrix: Freshwater (using mean as true)
Project: EPA/EPRI
Date: 07/13/2000
Units: ug/L

*** Results of 5x - 1/5x Mean Error Check ***
*** Questionable Data (Positive Values) ***

Conc Lev	Lab No	Rep No	Mean Result	Result	Ratio
1	3	1	.0157	.0028	.18
1	8	1	.0157	.0022	.14
2	2	1	.0111	.0021	.19
2	3	1	.0111	.0015	.13
2	8	1	.0111	.0017	.15

Total Number of Questionable Observations: 5

*** Results of Factor of 5 Error Check ***
*** Questionable Data (All Values) ***

Total Number of Questionable Observations: 0

File Name: ag-16
Data Preparation File (.PRP)

Analyte: Ag Matrix: Freshwater (using mean as true)
Project: EPA/EPRI
Date: 07/13/2000
Units: ug/L

*** Laboratory Ranking Results ***
*** Two-Tailed 5% Significance Level ***
*** Upper Critical Value: 54.0 ***
*** Lower Critical Value: 18.0 ***

Ranks

Lab	Rank Sums	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1	24.5	3.50	1.00	1.00	3.00	3.00	3.00	4.00	6.00
2	30.5	3.50	4.00	2.00	5.00	6.00	5.00	1.00	4.00
3	45.0	2.00	2.00	6.00	6.00	8.00	7.00	7.00	7.00
4	60.0	7.00	6.00	8.00	8.00	7.00	8.00	8.00	8.00
5	49.0	8.00	8.00	7.00	7.00	4.00	4.00	6.00	5.00
6	38.0	6.00	7.00	4.00	2.00	5.00	6.00	5.00	3.00
7	25.0	5.00	5.00	5.00	4.00	1.00	1.00	3.00	1.00
8	16.0	1.00	3.00	3.00	1.00	2.00	2.00	2.00	2.00

*** Laboratory 4 Rejected; Rank Sum 60.0 ***

File Name: ag-16
Data Preparation File (.PRP)

Analyte: Ag Matrix: Freshwater (using mean as true)
Project: EPA/EPRI
Date: 07/13/2000
Units: ug/L

*** Outlier Testing Results ***
*** Two-Sided 5% Significance Level ***

- Outlier(s) -

Lev	Iter	Lab	Rep	Result	Mean	Std Dev	t	Crit t	n
1	1	5	1	.0460	.0119	.0154	2.205	2.020	7
2	1	5	1	.0450	.0114	.0166	2.021	2.020	7
7	1	3	1	.2361	.0805	.0721	2.159	2.020	7
8	1	3	1	.3016	.0995	.0926	2.182	2.020	7

File Name: ag-16
Data Preparation File (.PRP)

Analyte: Ag Matrix: Freshwater (using mean as true)
Project: EPA/EPRI
Date: 07/13/2000
Units: ug/L

*** Results of Normality Testing ***

- Normality Tests -

Level	n	Test Type	Normality Accept/Reject	Test Statistic	Critical Value(s)	
1	6	W	A	.8629	.788	---
2	6	W	R	.7350	.788	---
3	7	W	R	.7098	.803	---
4	7	W	R	.7514	.803	---
5	7	W	A	.9791	.803	---
6	7	W	A	.9244	.803	---
7	6	W	A	.9242	.788	---
8	6	W	A	.8927	.788	---

- 3 Normality Rejection(s) -

File Name: ag-16
Data Preparation File (.PRP)

Analyte: Ag Matrix: Freshwater (using mean as true)
Project: EPA/EPRI
Date: 07/13/2000
Units: ug/L

*** Data Removal Tracking ***
*** Simple Count of Remaining Data Points ***
*** After Removal Tests ***

Lev	Mtrx	Points As Received	After Lab Ranking Points	After Lab Ranking %	After Outlier Testing Points	After Outlier Testing %
1	16	8	7	87.5	6	75.0
2	16	8	7	87.5	6	75.0
3	16	8	7	87.5	7	87.5
4	16	8	7	87.5	7	87.5
5	16	8	7	87.5	7	87.5
6	16	8	7	87.5	7	87.5
7	16	8	7	87.5	6	75.0
8	16	8	7	87.5	6	75.0
Totals:		64	56	87.5	52	81.3

File Name: ag-16
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Ag Matrix: Freshwater (using mean as true)
Project: EPA/EPRI Method: 1638
Date: 07/13/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)
CONCENTRATION:	.0311	.0311	.1011	.1111	.2311	.2311	.3311	.3911
RECOVERY:								
Observations	6	6	7	7	7	7	6	6
Mean Result	.0063	.0058	.0294	.0344	.1221	.1128	.0546	.0658
Bias	-.0248	-.0253	-.0717	-.0767	-.1090	-.1183	-.2765	-.3253
Relative Bias %	-79.8499	-81.2433	-70.9623	-68.9983	-47.1534	-51.1838	-83.5045	-83.1671
Maximum Result	.0115	.0217	.0640	.0671	.1929	.1901	.0972	.1100
Minimum Result	.0022	.0000	.0109	.0171	.0637	.0646	.0289	.0396
SINGLE OPERATOR PRECISION:	Pair 1		Pair 2		Pair 3		Pair 4	
Observations	6		7		7		6	
Standard Deviation	.0039		.0038		.0092		.0193	
Correction Factor	1.0509		1.0424		1.0424		1.0509	
Corrected Std Dev	.0041		.0039		.0096		.0203	
Relative Std Dev (%)	67.0058		12.2982		8.1546		33.6859	
OVERALL PRECISION:								
Observations	6	6	7	7	7	7	6	6
Standard Deviation	.0039	.0082	.0237	.0216	.0445	.0403	.0242	.0276
Correction Factor	1.0509	1.0509	1.0424	1.0424	1.0424	1.0424	1.0509	1.0509
Corrected Std Dev	.0041	.0087	.0247	.0225	.0464	.0421	.0254	.0290
Relative Std Dev %	66.1834	148.6238	84.2587	65.3377	37.9997	37.2757	46.5014	44.0948

File Name: ag-16
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Ag Matrix: Freshwater (using mean as true)
Project: EPA/EPRI Method: 1638
Date: 07/13/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.0311	.0063	-.0248	-79.85	15.414	4.032	YES
2	.0311	.0058	-.0253	-81.24	7.502	4.032	YES
3	.1011	.0294	-.0717	-70.96	7.999	3.707	YES
4	.1111	.0344	-.0767	-69.00	9.394	3.707	YES
5	.2311	.1221	-.1090	-47.15	6.476	3.707	YES
6	.2311	.1128	-.1183	-51.18	7.757	3.707	YES
7	.3311	.0546	-.2765	-83.50	28.024	4.032	YES
8	.3911	.0658	-.3253	-83.17	28.844	4.032	YES

```
File Name:  ag-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***   Single Operator Precision    ***
----------------------------------------
Analyte:  Ag          Matrix:  Freshwater (using mean as true)
Project:  EPA/EPRI    Method:  1638
   Date:  07/13/2000
  Units:  ug/L

 *** Single Operator Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .0030
          Slope (b):      .0290
    ============================
                                          Single
           Sample Weights                Operator   Estimated
    Pair   Size       (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6     87.66       .0311       .0041       .0039
      2       7      9.60       .1061       .0039       .0061
      3       7      2.05       .2311       .0096       .0097
      4       6       .70       .3611       .0203       .0135
    ------------------------------------------------------------


    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ======================================================
      a:      .0028      Intercept (a'):    -5.8820
      b:   206.2836          Slope (b'):     5.3293
    ======================================================
                                                Single
           Sample Weight                      Operator   Estimated
    Pair   Size      (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6    22.65       .0311       .0041       .0033
      2       7    27.35       .0311       .0039       .0049
      3       7    27.35       .1011       .0096       .0096
      4       6    22.65       .1111       .0203       .0191
    ------------------------------------------------------------
```

```
File Name:  ag-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***       Overall Precision        ***
----------------------------------------
Analyte:  Ag          Matrix:  Freshwater (using mean as true)
Project:  EPA/EPRI    Method:  1638
   Date:  07/13/2000
  Units:  ug/L

 *** Overall Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .0073
          Slope (b):      .1085
    ============================
    Conc   Sample   Weights                Overall   Estimated
    Level  Size         (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------------
      1       6       22.69       .0311       .0041       .0106
      2       6       22.69       .0311       .0087       .0106
      3       7       16.11       .1011       .0247       .0182
      4       7       15.09       .1111       .0225       .0193
      5       7        7.84       .2311       .0464       .0323
      6       7        7.84       .2311       .0421       .0323
      7       6        4.27       .3311       .0254       .0432
      8       6        3.45       .3911       .0290       .0497
    ------------------------------------------------------------------


    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ============================================================
      a:      .0101      Intercept (a'):    -4.5993
      b:    61.4519          Slope (b'):     4.1183
    ============================================================
    Conc   Sample   Weight                Overall   Estimated
    Level  Size        (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6      11.32       .0311       .0041       .0114
      2       6      11.32       .0311       .0087       .0114
      3       7      13.68       .1011       .0247       .0153
      4       7      13.68       .1111       .0225       .0159
      5       7      13.68       .2311       .0464       .0261
      6       7      13.68       .2311       .0421       .0261
      7       6      11.32       .3311       .0254       .0393
      8       6      11.32       .3911       .0290       .0504
    ------------------------------------------------------------
```

```
File Name:  ag-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
----------------------------------------
Analyte:  Ag          Matrix:  Freshwater (using mean as true)
Project:  EPA/EPRI    Method:  1638
   Date:  07/13/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):     -.0028
          Slope (b):      .3388
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       6     32.15       .0311       .0063       .0077
      2       6     32.15       .0311       .0058       .0077
      3       7     12.77       .1011       .0294       .0315
      4       7     11.38       .1111       .0344       .0348
      5       7      4.06       .2311       .1221       .0755
      6       7      4.06       .2311       .1128       .0755
      7       6      1.95       .3311       .0546       .1094
      8       6      1.47       .3911       .0658       .1297
    ------------------------------------------------------------


File Name:  ag-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
----------------------------------------
Analyte:  Ag          Matrix:  Freshwater (using mean as true)
Project:  EPA/EPRI    Method:  1638
   Date:  07/13/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .0112
            Slope (f):      .0857
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .0029
                  (f):  *********
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .0155
            Slope (f):      .3203
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .0104
                  (f):  *********
     ==================================
```

Tl-5.dat

```
Tl     5   1     0.0000    0.0000    0.0345    0.0457    0.0838    0.0888    0.4290    0.4330    1.7600    1.7300
Tl     5   2     0.0012    0.0002    0.0502    0.0500    0.0962    0.0999    0.5734    0.4985    2.0030    2.0140
Tl     5   3     0.0001    0.0002    0.0394    0.0489    0.1005    0.0968    0.4960    0.5049    2.0253    2.0039
Tl     5   4     0.0010    0.0010    0.0360    0.0400    0.0890    0.0830    0.4510    0.4500    1.8130    1.8310
Tl     5   5    -0.0050   -0.0056    0.0358    0.0387    0.0917    0.0877    0.4848    0.4820    2.0091    2.0679
Tl     5   6     0.0041    0.0039    0.0425    0.0609    0.1005    0.0966    0.4782    0.4851    1.9235    1.9254
Tl     5   7     0.0002    0.0001    0.0423    0.0500    0.1051    0.1042    0.5151    0.5243    2.1066    2.1338
Tl     5   8     0.0000    0.0000    0.0352    0.0429    0.0970    0.0995    0.4630    0.4665    1.8590    1.9596
```

```
File Name:  tl-5
Data Validation File (.DA~)

*****************************************************************************
***                  Parameter and Data Validation File                   ***
*****************************************************************************
   Analyte:  Tl              Matrix:  DI Water (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:   5
      Date:  06/28/2000      Method:  1638
     Pairs:  5
     Units:  ug/L
```

Level	1	2	3	4	5	6	7	8	9	10
Spike	.0001	.0000	.0400	.0100	.0500	.0000	.4000	.0000	1.5000	.0000
Spike Increment	.0000	.0000	.0400	.0500	.1000	.1000	.5000	.5000	2.0000	2.0000

	Final Concentration									
Lab ID	.0001	.0001	.0401	.0501	.1001	.1001	.5001	.5001	2.0001	2.0001
1 r	.0000r	.0000 r	.0345r	.0457 r	.0838r	.0888 r	.4290r	.4330 r	1.7600r	1.7300
2	.0012	.0002	.0502	.0500	.0962	.0999	.5734	.4985	2.0030	2.0140
3	.0001	.0002	.0394	.0489	.1005	.0968	.4960	.5049	2.0253	2.0039
4	.0010	.0010	.0360	.0400	.0890	.0830	.4510	.4500	1.8130	1.8310
5	-.0050	-.0056	.0358	.0387	.0917	.0877	.4848	.4820	2.0091	2.0679
6	.0041	.0039	.0425	.0609	.1005	.0966	.4782	.4851	1.9235	1.9254
7 !	.0002!	.0001 !	.0423!	.0500 !	.1051!	.1042 !	.5151!	.5243 !	2.1066!	2.1338
8	.0000	.0000	.0352	.0429	.0970	.0995	.4630	.4665	1.8590	1.9596

Tl-5.daf

```
Tl     5   2      .0012     .0002     .0502     .0500     .0962     .0999     .5734     .4985    2.0030    2.0140
Tl     5   3      .0001     .0002     .0394     .0489     .1005     .0968     .4960     .5049    2.0253    2.0039
Tl     5   4      .0010     .0010     .0360     .0400     .0890     .0830     .4510     .4500    1.8130    1.8310
Tl     5   5     -.0050    -.0056     .0358     .0387     .0917     .0877     .4848     .4820    2.0091    2.0679
Tl     5   6      .0041     .0039     .0425     .0609     .1005     .0966     .4782     .4851    1.9235    1.9254
Tl     5   7      .0002     .0001     .0423     .0500     .1051     .1042     .5151     .5243    2.1066    2.1338
Tl     5   8      .0000     .0000     .0352     .0429     .0970     .0995     .4630     .4665    1.8590    1.9596
```

```
File Name:  tl-5
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
-------------------------------------------------------------
 Conc  Lab  Rep     Mean
 Lev   No   No     Result     Result                   Ratio
-------------------------------------------------------------
  1      3  1       .0008      .0001                    .12
  2      6  1       .0007      .0039                   5.78
  2      7  1       .0007      .0001                    .15
-------------------------------------------------------------
Total Number of Questionable Observations:     3


-------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
-------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  tl-5
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***---------------------------------------***
 ***     Upper Critical Value:     65.0   ***
 ***     Lower Critical Value:     25.0   ***
-----------------------------------------------

      Ranks
     ---------------------------------------------------------------------------
      Rank  Level  Level  Level  Level  Level  Level  Level  Level  Level  Level
Lab   Sums    1      2      3      4      5      6      7      8      9     10
---- ---------------------------------------------------------------------------
  1   18.0   2.50   2.50   1.00   4.00   1.00   3.00   1.00   1.00   1.00   1.00
  2   63.0   7.00   5.50   8.00   6.50   4.00   7.00   8.00   6.00   5.00   6.00
  3   56.0   4.00   5.50   5.00   5.00   6.50   5.00   6.00   7.00   7.00   5.00
  4   30.0   6.00   7.00   4.00   2.00   2.00   1.00   2.00   2.00   2.00   2.00
  5   33.0   1.00   1.00   3.00   1.00   3.00   2.00   5.00   4.00   6.00   7.00
  6   57.5   8.00   8.00   7.00   8.00   6.50   4.00   4.00   5.00   4.00   3.00
  7   68.5   5.00   4.00   6.00   6.50   8.00   8.00   7.00   8.00   8.00   8.00
  8   34.0   2.50   2.50   2.00   3.00   5.00   6.00   3.00   3.00   3.00   4.00
-----------------------------------------------
 *** Laboratory   1 Rejected; Rank Sum   18.0 ***
-----------------------------------------------

File Name:  tl-5
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
-----------------------------------------


-----------------------------------------
 - No Outlier Rejections in Iteration 1 -
 - No Further Outlier Testing Necessary -
-----------------------------------------
```

File Name: tl-5
Data Preparation File (.PRP)

Analyte: Tl Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Results of Normality Testing ***

- Normality Tests -

Level	n	Test Type	Normality Accept/Reject	Test Statistic	Critical Value(s)	
1	7	W	A	.8642	.803	---
2	7	W	A	.8167	.803	---
3	7	W	A	.8726	.803	---
4	7	W	A	.9151	.803	---
5	7	W	A	.9689	.803	---
6	7	W	A	.9062	.803	---
7	7	W	A	.9039	.803	---
8	7	W	A	.9919	.803	---
9	7	W	A	.9543	.803	---
10	7	W	A	.9913	.803	---

- 0 Normality Rejection(s) -

File Name: tl-5
Data Preparation File (.PRP)

Analyte: Tl Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Data Removal Tracking ***
*** Simple Count of Remaining Data Points ***
*** After Removal Tests ***

Lev	Mtrx	Points As Received	After Lab Ranking Points	After Lab Ranking %	After Outlier Testing Points	After Outlier Testing %
1	5	8	7	87.5	7	87.5
2	5	8	7	87.5	7	87.5
3	5	8	7	87.5	7	87.5
4	5	8	7	87.5	7	87.5
5	5	8	7	87.5	7	87.5
6	5	8	7	87.5	7	87.5
7	5	8	7	87.5	7	87.5
8	5	8	7	87.5	7	87.5
9	5	8	7	87.5	7	87.5
10	5	8	7	87.5	7	87.5
Totals:		80	70	87.5	70	87.5

File Name: tl-5
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Tl Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8	9	10
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)	Pair 5 (Low)	Pair 5 (High)
CONCENTRATION:	.0001	.0001	.0401	.0501	.1001	.1001	.5001	.5001	2.0001	2.0001
RECOVERY:										
Observations	7	7	7	7	7	7	7	7	7	7
Mean Result	.0002	.0000	.0402	.0473	.0971	.0954	.4945	.4873	1.9628	1.9908
Bias	.0001	-.0001	.0001	-.0028	-.0030	-.0047	-.0056	-.0128	-.0373	-.0093
Relative Bias %	128.5715	-128.5714	.2494	-5.5033	-2.9542	-4.7096	-1.1198	-2.5538	-1.8656	-.4650
Maximum Result	.0041	.0039	.0502	.0609	.1051	.1042	.5734	.5243	2.1066	2.1338
Minimum Result	-.0050	-.0056	.0352	.0387	.0890	.0830	.4510	.4500	1.8130	1.8310

SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4	Pair 5
Observations	7	7	7	7	7
Standard Deviation	.0003	.0042	.0026	.0214	.0285
Correction Factor	1.0424	1.0424	1.0424	1.0424	1.0424
Corrected Std Dev	.0003	.0044	.0027	.0223	.0297
Relative Std Dev (%)	294.3825	10.0727	2.7964	4.5385	1.5015

OVERALL PRECISION:										
Observations	7	7	7	7	7	7	7	7	7	7
Standard Deviation	.0027	.0028	.0054	.0076	.0055	.0074	.0406	.0247	.1026	.0982
Correction Factor	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424
Corrected Std Dev	.0028	.0029	.0056	.0079	.0058	.0077	.0423	.0257	.1069	.1024
Relative Std Dev %	1234.0100	*********	13.8966	16.7845	5.9210	8.1159	8.5606	5.2835	5.4462	5.1429

File Name: tl-5
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Tl Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.0001	.0002	.0001	128.57	.125	3.707	NO
2	.0001	.0000	-.0001	******	.121	3.707	NO
3	.0401	.0402	.0001	.25	.049	3.707	NO
4	.0501	.0473	-.0028	-5.50	.957	3.707	NO
5	.1001	.0971	-.0030	-2.95	1.418	3.707	NO
6	.1001	.0954	-.0047	-4.71	1.679	3.707	NO
7	.5001	.4945	-.0056	-1.12	.365	3.707	NO
8	.5001	.4873	-.0128	-2.55	1.368	3.707	NO
9	2.0001	1.9628	-.0373	-1.87	.963	3.707	NO
10	2.0001	1.9908	-.0093	-.46	.251	3.707	NO

File Name: tl-5
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Single Operator Precision ***

Analyte: Tl Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Single Operator Precision ***

- Linear Model - s = b*T + a

Intercept (a):	.0018
Slope (b):	.0226

Pair	Sample Size	Weights (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	7	39.35	.0001	.0003	.0018
2	7	30.47	.0451	.0044	.0028
3	7	23.17	.1001	.0027	.0041
4	7	6.22	.5001	.0223	.0131
5	7	.79	2.0001	.0297	.0471

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a:	.0021	Intercept (a'):	-6.1722
b:	4.6528	Slope (b'):	1.5375

Pair	Sample Size	Weight (%)	Conc	Single Operator Std Dev	Estimated Std Dev
1	7	20.00	.0001	.0003	.0021
2	7	20.00	.0001	.0044	.0022
3	7	20.00	.0401	.0027	.0024
4	7	20.00	.0501	.0223	.0045
5	7	20.00	.1001	.0297	.0452

```
File Name:  tl-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ***       Overall Precision        ***
---------------------------------------
Analyte:  Tl          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Overall Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .0031
          Slope (b):      .0546
    ============================
    Conc   Sample   Weights                Overall   Estimated
    Level  Size         (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       7       30.39       .0001       .0028       .0031
      2       7       30.39       .0001       .0029       .0031
      3       7       13.76       .0401       .0056       .0053
      4       7       11.76       .0501       .0079       .0058
      5       7        6.19       .1001       .0058       .0085
      6       7        6.19       .1001       .0077       .0085
      7       7         .61       .5001       .0423       .0304
      8       7         .61       .5001       .0257       .0304
      9       7         .05      2.0001       .1069       .1123
     10       7         .05      2.0001       .1024       .1123
    ------------------------------------------------------------


    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ============================================================
      a:      .0059      Intercept (a'):    -5.1301
      b:     4.7299           Slope (b'):     1.5539
    ============================================================
    Conc   Sample   Weight                Overall   Estimated
    Level  Size        (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       7      10.00       .0001       .0028       .0059
      2       7      10.00       .0001       .0029       .0059
      3       7      10.00       .0401       .0056       .0063
      4       7      10.00       .0501       .0079       .0064
      5       7      10.00       .1001       .0058       .0069
      6       7      10.00       .1001       .0077       .0069
      7       7      10.00       .5001       .0423       .0129
      8       7      10.00       .5001       .0257       .0129
      9       7      10.00      2.0001       .1069       .1324
     10       7      10.00      2.0001       .1024       .1324
    ------------------------------------------------------------
```

File Name: tl-5
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
********** Recovery **********

Analyte: Tl Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Recovery ***

- Linear Model - X = b*T + a
Intercept (a): -.0002
Slope (b): .9809

Conc Level	Sample Size	Weights (%)	Conc	Mean Result	Estimated Result
1	7	34.47	.0001	.0002	-.0001
2	7	34.47	.0001	.0000	-.0001
3	7	11.76	.0401	.0402	.0391
4	7	9.64	.0501	.0473	.0489
5	7	4.46	.1001	.0971	.0980
6	7	4.46	.1001	.0954	.0980
7	7	.35	.5001	.4945	.4903
8	7	.35	.5001	.4873	.4903
9	7	.03	2.0001	1.9628	1.9617
10	7	.03	2.0001	1.9908	1.9617

File Name: tl-5
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Precision vs Recovery ***

Analyte: Tl Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Single Operator Precision vs Recovery ***
(Obtained by Substitution)

- Linear Model - s = f*X + e
Intercept (e): .0020
Slope (f): .0231

- Curvilinear Model - s = e*(f**X)
Intercept (e): .0021
(f): 4.7939

*** Overall Precision vs Recovery ***
(Obtained by Substitution)

- Linear Model - s = f*X + e
Intercept (e): .0033
Slope (f): .0557

- Curvilinear Model - s = e*(f**X)
Intercept (e): .0059
(f): 4.8749

```
Tl-16.dat
Tl    16   1     0.0000    0.0000    0.0168    0.0175    0.0496    0.0544    0.0867    0.1050
Tl    16   2     0.0086    0.0012    0.0229    0.0305    0.0670    0.0590    0.1041    0.1190
Tl    16   3     0.0009    0.0009    0.0208    0.0243    0.0620    0.0600    0.1028    0.1211
Tl    16   4     0.0000    0.0010    0.0180    0.0200    0.0520    0.0530    0.0880    0.1050
Tl    16   5     0.0000    0.0000    0.0131    0.0155    0.0551    0.0537    0.0977    0.1202
Tl    16   6     0.0116    0.0080    0.0266    0.0269    0.0680    0.0640    0.1056    0.1229
Tl    16   7     0.0013    0.0008    0.0218    0.0245    0.0695    0.0673    0.1050    0.1298
Tl    16   8     0.0006    0.0000    0.0179    0.0206    0.0611    0.0594    0.0911    0.1097


File Name:  tl-16
Data Validation File (.DA~)

*****************************************************************************
***                   Parameter and Data Validation File                  ***
*****************************************************************************
   Analyte:  Tl              Matrix:  Freshwater (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:  16
      Date:  06/28/2000      Method:  1638
     Pairs:  4
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------
Level          1          2            3          4            5          6            7          8
-------------------------------------------------------------------------------------------------------------
Spike          .0010      .0000        .0200      .0030        .0370      .0000        .0400      .0200
Spike
Increment      .0000      .0000        .0200      .0230        .0600      .0600        .1000      .1200
-------------------------------------------------------------------------------------------------------------

                 Final Concentration
          -------------------------------------------------------------------------------------------------
Lab ID         .0010      .0010        .0210      .0240        .0610      .0610        .1010      .1210
-------------------------------------------------------------------------------------------------------------
    1    !     .0000!     .0000  !     .0168!     .0175  !     .0496!     .0544  !     .0867!     .1050
    2    o     .0086      .0012        .0229      .0305        .0670      .0590        .1041      .1190
    3          .0009      .0009        .0208      .0243        .0620      .0600        .1028      .1211
    4          .0000      .0010        .0180      .0200        .0520      .0530        .0880      .1050
    5          .0000      .0000        .0131      .0155        .0551      .0537        .0977      .1202
    6    r     .0116r     .0080  r     .0266r     .0269  r     .0680r     .0640  r     .1056r     .1229
    7          .0013      .0008        .0218      .0245        .0695      .0673        .1050      .1298
    8          .0006      .0000        .0179      .0206        .0611      .0594        .0911      .1097
-------------------------------------------------------------------------------------------------------------

Tl-16.daf
Tl    16   1      .0000     .0000     .0168     .0175     .0496     .0544     .0867     .1050
Tl    16   2 o              .0012     .0229     .0305     .0670     .0590     .1041     .1190
Tl    16   3      .0009     .0009     .0208     .0243     .0620     .0600     .1028     .1211
Tl    16   4      .0000     .0010     .0180     .0200     .0520     .0530     .0880     .1050
Tl    16   5      .0000     .0000     .0131     .0155     .0551     .0537     .0977     .1202
Tl    16   7      .0013     .0008     .0218     .0245     .0695     .0673     .1050     .1298
Tl    16   8      .0006     .0000     .0179     .0206     .0611     .0594     .0911     .1097
```

```
File Name:  tl-16
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
------------------------------------------------------------
 Conc  Lab  Rep     Mean
 Lev   No   No     Result     Result                   Ratio
------------------------------------------------------------
  2      6  1       .0015      .0080                   5.38
------------------------------------------------------------
Total Number of Questionable Observations:     1


------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  tl-16
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***-------------------------------------***
 ***     Upper Critical Value:     54.0   ***
 ***     Lower Critical Value:     18.0   ***
-----------------------------------------------

      Ranks
     ------------------------------------------------------------------
       Rank  Level  Level  Level  Level  Level  Level  Level  Level
Lab    Sums    1      2      3      4      5      6      7      8
---- ------------------------------------------------------------------
  1    14.5   2.00   2.00   2.00   2.00   1.00   3.00   1.00   1.50
  2    49.0   7.00   7.00   7.00   8.00   6.00   4.00   6.00   4.00
  3    42.0   5.00   5.00   5.00   5.00   5.00   6.00   5.00   6.00
  4    21.5   2.00   6.00   4.00   3.00   2.00   1.00   2.00   1.50
  5    20.0   2.00   2.00   1.00   1.00   3.00   2.00   4.00   5.00
  6    60.0   8.00   8.00   8.00   7.00   7.00   7.00   8.00   7.00
  7    53.0   6.00   4.00   6.00   6.00   8.00   8.00   7.00   8.00
  8    28.0   4.00   2.00   3.00   4.00   4.00   5.00   3.00   3.00
-----------------------------------------------
 *** Laboratory   6 Rejected; Rank Sum   60.0 ***
-----------------------------------------------

File Name:  tl-16
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
------------------------------------------------------------------------------
  1     1     2   1       .0086      .0016      .0031   2.237   2.020    7
------------------------------------------------------------------------------
```

```
File Name:  tl-16
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
---------------------------------------------------------------------
     1     6     W     A               .8382      .788   ---
     2     7     W     A               .8070      .803   ---
     3     7     W     A               .9520      .803   ---
     4     7     W     A               .9585      .803   ---
     5     7     W     A               .9479      .803   ---
     6     7     W     A               .8864      .803   ---
     7     7     W     A               .8718      .803   ---
     8     7     W     A               .9075      .803   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  tl-16
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     ----------------  --------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    16        8            7    87.5           6       75.0
  2    16        8            7    87.5           7       87.5
  3    16        8            7    87.5           7       87.5
  4    16        8            7    87.5           7       87.5
  5    16        8            7    87.5           7       87.5
  6    16        8            7    87.5           7       87.5
  7    16        8            7    87.5           7       87.5
  8    16        8            7    87.5           7       87.5
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         64           56    87.5          55       85.9
---------------------------------------------------------------
```

File Name: tl-16
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Tl Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)
CONCENTRATION:	.0010	.0010	.0210	.0240	.0610	.0610	.1010	.1210
RECOVERY:								
Observations	6	7	7	7	7	7	7	7
Mean Result	.0005	.0006	.0188	.0218	.0595	.0581	.0965	.1157
Bias	-.0005	-.0004	-.0022	-.0022	-.0015	-.0029	-.0045	-.0053
Relative Bias %	-53.3333	-44.2857	-10.6803	-8.9881	-2.5058	-4.7307	-4.4696	-4.3920
Maximum Result	.0013	.0012	.0229	.0305	.0695	.0673	.1050	.1298
Minimum Result	.0000	.0000	.0131	.0155	.0496	.0530	.0867	.1050
SINGLE OPERATOR PRECISION:	Pair 1		Pair 2		Pair 3		Pair 4	
Observations	6		7		7		7	
Standard Deviation	.0004		.0015		.0027		.0024	
Correction Factor	1.0509		1.0424		1.0424		1.0424	
Corrected Std Dev	.0004		.0016		.0028		.0025	
Relative Std Dev (%)	81.7773		7.8698		4.8239		2.3314	
OVERALL PRECISION:								
Observations	6	7	7	7	7	7	7	7
Standard Deviation	.0006	.0005	.0034	.0050	.0075	.0050	.0078	.0093
Correction Factor	1.0509	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424	1.0424
Corrected Std Dev	.0006	.0006	.0035	.0052	.0078	.0052	.0082	.0097
Relative Std Dev %	125.5211	100.0865	18.6717	24.0248	13.1680	8.9625	8.4674	8.4209

File Name: tl-16
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Tl Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.0010	.0005	-.0005	-53.33	2.344	4.032	NO
2	.0010	.0006	-.0004	-44.29	2.190	3.707	NO
3	.0210	.0188	-.0022	-10.68	1.766	3.707	NO
4	.0240	.0218	-.0022	-8.99	1.134	3.707	NO
5	.0610	.0595	-.0015	-2.51	.538	3.707	NO
6	.0610	.0581	-.0029	-4.73	1.528	3.707	NO
7	.1010	.0965	-.0045	-4.47	1.524	3.707	NO
8	.1210	.1157	-.0053	-4.39	1.504	3.707	NO

```
File Name:  tl-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***   Single Operator Precision    ***
----------------------------------------
Analyte:  Tl          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .0007
          Slope (b):      .0252
    ============================
                                          Single
           Sample Weights                Operator   Estimated
    Pair   Size       (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6     47.52       .0010       .0004       .0007
      2       7     31.06       .0225       .0016       .0012
      3       7     14.30       .0610       .0028       .0022
      4       7      7.12       .1110       .0025       .0035
    ------------------------------------------------------------



    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ======================================================
      a:      .0008      Intercept (a'):    -7.1388
      b:  *********          Slope (b'):    13.2372
    ======================================================
                                                 Single
           Sample Weight                Operator   Estimated
    Pair   Size      (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------
      1       6    21.63       .0010       .0004       .0008
      2       7    26.12       .0010       .0016       .0011
      3       7    26.12       .0210       .0028       .0018
      4       7    26.12       .0240       .0025       .0034
    ------------------------------------------------------
```

File Name: tl-16
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Overall Precision ***

Analyte: Tl Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Overall Precision ***

- Linear Model - s = b*T + a

Intercept (a):	.0008
Slope (b):	.0938

Conc Level	Sample Size	Weights (%)	Conc	Overall Std Dev	Estimated Std Dev
1	6	31.07	.0010	.0006	.0009
2	7	37.52	.0010	.0006	.0009
3	7	11.75	.0210	.0035	.0028
4	7	10.34	.0240	.0052	.0031
5	7	3.32	.0610	.0078	.0066
6	7	3.32	.0610	.0052	.0066
7	7	1.54	.1010	.0082	.0103
8	7	1.15	.1210	.0097	.0122

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a:	.0013	Intercept (a'):	-6.6520
b:	*********	Slope (b'):	20.3871

Conc Level	Sample Size	Weight (%)	Conc	Overall Std Dev	Estimated Std Dev
1	6	10.58	.0010	.0006	.0013
2	7	12.77	.0010	.0006	.0013
3	7	12.77	.0210	.0035	.0020
4	7	12.77	.0240	.0052	.0021
5	7	12.77	.0610	.0078	.0045
6	7	12.77	.0610	.0052	.0045
7	7	12.77	.1010	.0082	.0101
8	7	12.77	.1210	.0097	.0152

```
File Name:  tl-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
----------------------------------------
Analyte:  Tl          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):     -.0005
          Slope (b):      .9559
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       6     40.63       .0010       .0005       .0005
      2       7     47.40       .0010       .0006       .0005
      3       7      5.16       .0210       .0188       .0196
      4       7      4.26       .0240       .0218       .0224
      5       7       .94       .0610       .0595       .0578
      6       7       .94       .0610       .0581       .0578
      7       7       .38       .1010       .0965       .0960
      8       7       .27       .1210       .1157       .1152
    ------------------------------------------------------------


File Name:  tl-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
----------------------------------------
Analyte:  Tl          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .0012
            Slope (f):      .0264
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .0008
                  (f):  *********
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .0013
            Slope (f):      .0982
     ============================
```

Zn-5.dat

```
Zn     5   1     0.0713    0.2800    0.4650    0.4740    1.8900    2.3600    4.4600    4.5800   43.2000   43.4000
Zn     5   2     0.6914    0.4415    4.0300    1.1550    3.0660    2.8680    6.0460    5.6360   51.0000   51.3700
Zn     5   3     0.0697    0.1315    0.7130    0.6415    2.1609    2.6430    4.9954    5.8975   52.5187   51.9073
Zn     5   4    -0.2020   -0.2190    0.2510    0.2380    3.2120    2.0620    4.9730    4.9380   50.4520   50.7220
Zn     5   5     1.7024    4.5007    2.1958    3.4642    3.7071    4.2327    5.7298    6.3932
Zn     5   6     0.2636    0.4230    0.7293    0.8695    2.1349    2.7856    4.8052    6.4784   45.8536   59.8960
Zn     5   7    -0.0374   -0.0397    0.4118    0.4133    1.7942    2.1073    4.6699    4.5940   46.6399   46.6981
Zn     5   8     1.1251    0.9461    0.6587    1.0679    1.6577    2.0357    4.4630    4.4245   44.7807   50.4607
```

```
File Name:  zn-5
Data Validation File (.DA~)

*************************************************************************
***                  Parameter and Data Validation File                 ***
*************************************************************************
   Analyte:  Zn              Matrix:  DI Water (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:   5
      Date:  06/28/2000      Method:  1638
     Pairs:  5
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------------------
Level          1          2          3          4          5          6          7          8          9         10
-------------------------------------------------------------------------------------------------------------------------
Spike         .1304      .0000      .5000      .0000     1.5000      .4000     2.6000      .0000    45.0000      .0000
Spike
Increment     .0000      .0000      .5000      .5000     2.0000     2.4000     5.0000     5.0000    50.0000    50.0000
-------------------------------------------------------------------------------------------------------------------------

                Final Concentration
          ------------------------------------------------------------------------------------------------------------
Lab ID        .1304      .1304      .6304      .6304     2.1304     2.5304     5.1304     5.1304    50.1304    50.1304
-------------------------------------------------------------------------------------------------------------------------
    1         .0713      .2800      .4650      .4740     1.8900     2.3600     4.4600     4.5800    43.2000    43.4000
    2    r   .6914r     .4415  r  4.0300r    1.1550  r  3.0660r    2.8680  r  6.0460r    5.6360  r 51.0000r   51.3700
    3         .0697      .1315      .7130      .6415     2.1609     2.6430     4.9954     5.8975    52.5187    51.9073
    4        -.2020     -.2190      .2510      .2380     3.2120     2.0620     4.9730     4.9380    50.4520    50.7220
    5        1.7024o    4.5007  o  2.1958o    3.4642     3.7071o    4.2327     5.7298     6.3932  e 34.7065e   34.7065
    6         .2636      .4230      .7293      .8695     2.1349     2.7856     4.8052     6.4784    45.8536    59.8960
    7        -.0374     -.0397      .4118      .4133     1.7942     2.1073     4.6699     4.5940    46.6399    46.6981
    8        1.1251      .9461      .6587     1.0679     1.6577     2.0357     4.4630     4.4245    44.7807    50.4607
-------------------------------------------------------------------------------------------------------------------------
```

Zn-5.daf

```
Zn     5   1      .0713     .2800     .4650     .4740    1.8900    2.3600    4.4600    4.5800   43.2000   43.4000
Zn     5   3      .0697     .1315     .7130     .6415    2.1609    2.6430    4.9954    5.8975   52.5187   51.9073
Zn     5   4     -.2020    -.2190     .2510     .2380    3.2120    2.0620    4.9730    4.9380   50.4520   50.7220
Zn     5   5    1.7024o         o         o             3.7071o             5.7298    6.3932*         *
Zn     5   6      .2636     .4230     .7293     .8695    2.1349    2.7856    4.8052    6.4784   45.8536   59.8960
Zn     5   7     -.0374    -.0397     .4118     .4133    1.7942    2.1073    4.6699    4.5940   46.6399   46.6981
Zn     5   8     1.1251     .9461     .6587    1.0679    1.6577    2.0357    4.4630    4.4245   44.7807   50.4607
```

```
File Name:  zn-5
Data Preparation File (.PRP)


Analyte:  Zn     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
-------------------------------------------------------------
 Conc  Lab  Rep     Mean
 Lev   No   No     Result     Result                   Ratio
-------------------------------------------------------------
  1      1  1       .4904      .0713                    .15
  1      3  1       .4904      .0697                    .14
  2      3  1       .8404      .1315                    .16
  2      5  1       .8404     4.5007                   5.36
-------------------------------------------------------------
Total Number of Questionable Observations:     4


-------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
-------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  zn-5
Data Preparation File (.PRP)


Analyte:  Zn     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***      Laboratory Ranking Results      ***
 ***   Two-Tailed 5% Significance Level   ***
 ***-------------------------------------***
 ***     Upper Critical Value:     65.0   ***
 ***     Lower Critical Value:     25.0   ***
-----------------------------------------------

      Ranks
     ---------------------------------------------------------------------------
       Rank  Level  Level  Level  Level  Level  Level  Level  Level  Level  Level
Lab    Sums    1      2      3      4      5      6      7      8      9     10
---- ---------------------------------------------------------------------------
  1    28.0   4.00   4.00   3.00   3.00   3.00   4.00   1.00   2.00   2.00   2.00
  2    66.0   6.00   6.00   8.00   7.00   6.00   7.00   8.00   5.00   7.00   6.00
  3    52.0   3.00   3.00   5.00   4.00   5.00   5.00   6.00   6.00   8.00   7.00
  4    33.0   1.00   1.00   1.00   1.00   7.00   2.00   5.00   4.00   6.00   5.00
  5    63.0   8.00   8.00   7.00   8.00   8.00   8.00   7.00   7.00   1.00   1.00
  6    55.0   5.00   5.00   6.00   5.00   4.00   6.00   4.00   8.00   4.00   8.00
  7    27.0   2.00   2.00   2.00   2.00   2.00   3.00   3.00   3.00   5.00   3.00
  8    36.0   7.00   7.00   4.00   6.00   1.00   1.00   2.00   1.00   3.00   4.00
-----------------------------------------------
 *** Laboratory   2 Rejected; Rank Sum   66.0 ***
-----------------------------------------------

File Name:  zn-5
Data Preparation File (.PRP)


Analyte:  Zn     Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
-----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
-------------------------------------------------------------------------------
  2     1     5   1      4.5007      .8604     1.6479   2.209   2.020    7
  3     1     5   1      2.1958      .7749      .6507   2.183   2.020    7
  4     1     5   1      3.4642     1.0241     1.1120   2.194   2.020    7
  6     1     5   1      4.2327     2.6038      .7757   2.100   2.020    7
-------------------------------------------------------------------------------
```

File Name: zn-5
Data Preparation File (.PRP)

Analyte: Zn Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Results of Normality Testing ***

- Normality Tests -

Level	n	Test Type	Normality Accept/Reject	Test Statistic	Critical Value(s)	
1	7	W	A	.8163	.803	---
2	6	W	A	.9551	.788	---
3	6	W	A	.9025	.788	---
4	6	W	A	.9667	.788	---
5	7	W	A	.8309	.803	---
6	6	W	A	.8658	.788	---
7	7	W	A	.8663	.803	---
8	7	W	A	.8352	.803	---
9	6	W	A	.9321	.788	---
10	6	W	A	.9424	.788	---

- 0 Normality Rejection(s) -

File Name: zn-5
Data Preparation File (.PRP)

Analyte: Zn Matrix: DI Water (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Data Removal Tracking ***
*** Simple Count of Remaining Data Points ***
*** After Removal Tests ***

Lev	Mtrx	Points As Received	After Lab Ranking Points	After Lab Ranking %	After Outlier Testing Points	After Outlier Testing %
1	5	8	7	87.5	7	87.5
2	5	8	7	87.5	6	75.0
3	5	8	7	87.5	6	75.0
4	5	8	7	87.5	6	75.0
5	5	8	7	87.5	7	87.5
6	5	8	7	87.5	6	75.0
7	5	8	7	87.5	7	87.5
8	5	8	7	87.5	7	87.5
9	5	7	6	85.7	6	85.7
10	5	7	6	85.7	6	85.7
Totals:		78	68	87.2	64	82.1

File Name: zn-5
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Zn Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8	9	10
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)	Pair 5 (Low)	Pair 5 (High)
CONCENTRATION:	.1304	.1304	.6304	.6304	2.1304	2.5304	5.1304	5.1304	50.1304	50.1304
RECOVERY:										
Observations	7	6	6	6	7	6	7	7	6	6
Mean Result	.4275	.2537	.5381	.6174	2.3653	2.3323	4.8709	5.3294	47.2408	50.5140
Bias	.2971	.1233	-.0923	-.0130	.2349	-.1981	-.2595	.1990	-2.8896	.3836
Relative Bias %	227.8593	94.5169	-14.6362	-2.0675	11.0241	-7.8301	-5.0581	3.8783	-5.7641	.7652
Maximum Result	1.7024	.9461	.7293	1.0679	3.7071	2.7856	5.7298	6.4784	52.5187	59.8960
Minimum Result	-.2020	-.2190	.2510	.2380	1.6577	2.0357	4.4600	4.4245	43.2000	43.4000

SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4	Pair 5
Observations	6	6	6	7	6
Standard Deviation	.0981	.1244	.4714	.4659	4.0701
Correction Factor	1.0509	1.0509	1.0509	1.0424	1.0509
Corrected Std Dev	.1031	.1308	.4954	.4857	4.2774
Relative Std Dev (%)	29.6784	22.6318	21.0805	9.5223	8.7513

OVERALL PRECISION:										
Observations	7	6	6	6	7	6	7	7	6	6
Standard Deviation	.7078	.4082	.1926	.3077	.7816	.3208	.4366	.8990	3.5450	5.5710
Correction Factor	1.0424	1.0509	1.0509	1.0509	1.0424	1.0509	1.0424	1.0424	1.0509	1.0509
Corrected Std Dev	.7378	.4290	.2024	.3233	.8147	.3371	.4551	.9371	3.7256	5.8547
Relative Std Dev %	172.5763	169.1374	37.6073	52.3713	34.4425	14.4538	9.3424	17.5829	7.8864	11.5903

File Name: zn-5
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Zn Matrix: DI Water (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.1304	.4275	.2971	227.86	1.112	3.707	NO
2	.1304	.2537	.1233	94.52	.740	4.032	NO
3	.6304	.5381	-.0923	-14.64	1.174	4.032	NO
4	.6304	.6174	-.0130	-2.07	.104	4.032	NO
5	2.1304	2.3653	.2349	11.02	.795	3.707	NO
6	2.5304	2.3323	-.1981	-7.83	1.513	4.032	NO
7	5.1304	4.8709	-.2595	-5.06	1.573	3.707	NO
8	5.1304	5.3294	.1990	3.88	.586	3.707	NO
9	50.1304	47.2408	-2.8896	-5.76	1.997	4.032	NO
10	50.1304	50.5140	.3836	.77	.169	4.032	NO

```
File Name:  zn-5
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***   Single Operator Precision    ***
----------------------------------------
Analyte:  Zn          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .0998
          Slope (b):      .0938
    ============================
                                        Single
           Sample Weights              Operator   Estimated
    Pair   Size       (%)        Conc    Std Dev     Std Dev
    ------------------------------------------------------------
      1       6     53.51       .1304       .1031       .1120
      2       6     32.02       .6304       .1308       .1589
      3       6     10.22      2.3304       .4954       .3184
      4       7      4.19      5.1304       .4857       .5810
      5       6       .06     50.1304      4.2774      4.8022
    ------------------------------------------------------------



    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ======================================================
      a:      .2112      Intercept (a'):    -1.5551
      b:     1.0637          Slope (b'):      .0618
    ======================================================
                                                Single
           Sample Weight               Operator   Estimated
    Pair   Size      (%)        Conc    Std Dev     Std Dev
    ------------------------------------------------------
      1       6    19.20       .1304       .1031       .2129
      2       6    19.20       .1304       .1308       .2195
      3       6    19.20       .6304       .4954       .2439
      4       7    23.19       .6304       .4857       .2899
      5       6    19.20      2.1304      4.2774      4.6724
    ------------------------------------------------------
```

```
File Name:  zn-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ***       Overall Precision        ***
---------------------------------------
Analyte:  Zn          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Overall Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .4257
          Slope (b):      .0729
    ============================
    Conc   Sample   Weights                Overall   Estimated
    Level  Size         (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       7       22.79       .1304       .7378       .4352
      2       6       18.87       .1304       .4290       .4352
      3       6       15.11       .6304       .2024       .4716
      4       6       15.11       .6304       .3233       .4716
      5       7       10.54      2.1304       .8147       .5809
      6       6        7.71      2.5304       .3371       .6101
      7       7        4.81      5.1304       .4551       .7995
      8       7        4.81      5.1304       .9371       .7995
      9       6         .12     50.1304      3.7256      4.0778
     10       6         .12     50.1304      5.8547      4.0778
    ------------------------------------------------------------


    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ============================================================
      a:      .4406      Intercept (a'):     -.8197
      b:     1.0485           Slope (b'):      .0474
    ============================================================
    Conc   Sample   Weight                 Overall   Estimated
    Level  Size        (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       7      11.15       .1304       .7378       .4433
      2       6       9.23       .1304       .4290       .4433
      3       6       9.23       .6304       .2024       .4539
      4       6       9.23       .6304       .3233       .4539
      5       7      11.15      2.1304       .8147       .4874
      6       6       9.23      2.5304       .3371       .4967
      7       7      11.15      5.1304       .4551       .5619
      8       7      11.15      5.1304       .9371       .5619
      9       6       9.23     50.1304      3.7256      4.7434
     10       6       9.23     50.1304      5.8547      4.7434
    ------------------------------------------------------------
```

```
File Name:  zn-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
---------------------------------------
Analyte:  Zn          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):      .1117
          Slope (b):      .9704
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       7     20.30       .1304       .4275       .2382
      2       6     17.40       .1304       .2537       .2382
      3       6     14.81       .6304       .5381       .7234
      4       6     14.81       .6304       .6174       .7234
      5       7     11.39      2.1304      2.3653      2.1791
      6       6      8.85      2.5304      2.3323      2.5673
      7       7      6.02      5.1304      4.8709      5.0904
      8       7      6.02      5.1304      5.3294      5.0904
      9       6       .20     50.1304     47.2408     48.7602
     10       6       .20     50.1304     50.5140     48.7602
    ------------------------------------------------------------


File Name:  zn-5
Statistical Analysis File (.STT)


---------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
---------------------------------------
Analyte:  Zn          Matrix:  DI Water (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):     -.0153
            Slope (f):      .0967
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .2097
                  (f):     1.0657
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .3107
            Slope (f):      .0751
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .4382
                  (f):     1.0501
     ==================================
```

Zn-16.dat

```
Zn    16   1     0.8690    0.7900    0.9050    1.3700    1.2800    1.4000    1.6300    2.0000
Zn    16   2     1.9980    1.3810    1.4150    1.8460    4.3210    2.3680    2.1890    2.5790
Zn    16   3     1.0036    0.9580    0.9369    1.1145    1.5506    1.3816    2.0570    2.0747
Zn    16   4     0.5140    0.4470    0.6370    0.9220    1.4320    1.1660    1.6130    1.8390
Zn    16   5    19.9924    2.6400    2.2861    2.8336    3.5617   14.3135    3.3408    3.6014
Zn    16   6     1.0932    1.6887    1.1470    1.4080    2.1583    1.5672    1.8281    2.2378
Zn    16   7     0.7638    0.7708    0.8760    1.0735    1.3056    1.2636    1.4933    1.8992
Zn    16   8     0.6552    0.8136    1.0597    1.1635    4.6312    3.8415    1.3807    1.7278
```

```
File Name:  zn-16
Data Validation File (.DA~)

*************************************************************************
***                  Parameter and Data Validation File                 ***
*************************************************************************
   Analyte:  Zn              Matrix:  Freshwater (using MSA as true)
   Project:  EPA/EPRI     Matrix ID:  16
      Date:  06/28/2000      Method:  1638
     Pairs:  4
     Units:  ug/L

-------------------------------------------------------------------------------------------------------------
Level          1          2           3          4           5          6           7          8
-------------------------------------------------------------------------------------------------------------
Spike         .9413      .0000       .1300      .2000       .2000      .0000       .2900      .4000
Spike
Increment     .0000      .0000       .1300      .3300       .5300      .5300       .8200     1.2200
-------------------------------------------------------------------------------------------------------------

                Final Concentration
          ------------------------------------------------------------------------------------------------
Lab ID         .9413      .9413      1.0713     1.2713      1.4713     1.4713      1.7613     2.1613
-------------------------------------------------------------------------------------------------------------
    1          .8690      .7900       .9050     1.3700      1.2800     1.4000      1.6300     2.0000
    2    o    1.9980     1.3810      1.4150     1.8460      4.3210?    2.3680      2.1890     2.5790
    3         1.0036      .9580       .9369     1.1145      1.5506     1.3816      2.0570     2.0747
    4    !     .5140!     .4470  !    .6370!     .9220  !    1.4320!    1.1660  !   1.6130!    1.8390
    5    r   19.9924r    2.6400  r   2.2861r    2.8336  r   3.5617r   14.3135  r   3.3408r    3.6014
    6         1.0932     1.6887      1.1470     1.4080      2.1583     1.5672      1.8281     2.2378
    7          .7638      .7708       .8760     1.0735      1.3056     1.2636      1.4933     1.8992
    8          .6552      .8136      1.0597     1.1635      4.6312o    3.8415      1.3807     1.7278
-------------------------------------------------------------------------------------------------------------
```

Zn-16.daf

```
Zn    16   1      .8690     .7900     .9050    1.3700    1.2800    1.4000    1.6300    2.0000
Zn    16   2 o             1.3810    1.4150    1.8460    4.3210    2.3680    2.1890    2.5790
Zn    16   3    1.0036     .9580     .9369    1.1145    1.5506    1.3816    2.0570    2.0747
Zn    16   4     .5140     .4470     .6370     .9220    1.4320    1.1660    1.6130    1.8390
Zn    16   6    1.0932    1.6887    1.1470    1.4080    2.1583    1.5672    1.8281    2.2378
Zn    16   7     .7638     .7708     .8760    1.0735    1.3056    1.2636    1.4933    1.8992
Zn    16   8     .6552     .8136    1.0597    1.1635    4.6312o             1.3807    1.7278
```

File Name: zn-16
Data Preparation File (.PRP)

Analyte: Zn Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Results of 5x - 1/5x Mean Error Check ***
*** Questionable Data (Positive Values) ***

Conc Lev	Lab No	Rep No	Mean Result	Result	Ratio
1	4	1	3.3611	.5140	.15
1	5	1	3.3611	19.9924	5.95
1	8	1	3.3611	.6552	.19

Total Number of Questionable Observations: 3

*** Results of Factor of 5 Error Check ***
*** Questionable Data (All Values) ***

Total Number of Questionable Observations: 0

File Name: zn-16
Data Preparation File (.PRP)

Analyte: Zn Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Laboratory Ranking Results ***
*** Two-Tailed 5% Significance Level ***
*** Upper Critical Value: 54.0 ***
*** Lower Critical Value: 18.0 ***

Ranks

Lab	Rank Sums	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1	28.0	4.00	3.00	3.00	5.00	1.00	4.00	4.00	4.00
2	54.0	7.00	6.00	7.00	7.00	7.00	6.00	7.00	7.00
3	35.0	5.00	5.00	4.00	3.00	4.00	3.00	6.00	5.00
4	13.0	1.00	1.00	1.00	1.00	3.00	1.00	3.00	2.00
5	62.0	8.00	8.00	8.00	8.00	6.00	8.00	8.00	8.00
6	46.0	6.00	7.00	6.00	6.00	5.00	5.00	5.00	6.00
7	18.0	3.00	2.00	2.00	2.00	2.00	2.00	2.00	3.00
8	32.0	2.00	4.00	5.00	4.00	8.00	7.00	1.00	1.00

*** Laboratory 5 Rejected; Rank Sum 62.0 ***

File Name: zn-16
Data Preparation File (.PRP)

Analyte: Zn Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI
Date: 06/28/2000
Units: ug/L

*** Outlier Testing Results ***
*** Two-Sided 5% Significance Level ***

- Outlier(s) -

Lev	Iter	Lab	Rep	Result	Mean	Std Dev	t	Crit t	n
1	1	2	1	1.9980	.9853	.4884	2.074	2.020	7
6	1	8	1	3.8415	1.8554	.9616	2.065	2.020	7

```
File Name:  zn-16
Data Preparation File (.PRP)


Analyte:  Zn     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
              Test  Normality      Test          Critical
   Level  n   Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     6     W     A               .9797      .788   ---
     2     7     W     A               .9147      .803   ---
     3     7     W     A               .9696      .803   ---
     4     7     W     A               .9145      .803   ---
     5     7     W           R         .7509      .803   ---
     6     6     W           R         .7816      .788   ---
     7     7     W     A               .9408      .803   ---
     8     7     W     A               .9340      .803   ---
------------------------------
 - 2 Normality Rejection(s) -
------------------------------


File Name:  zn-16
Data Preparation File (.PRP)


Analyte:  Zn     Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI
   Date:  06/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     ----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    16        8            7    87.5           6       75.0
  2    16        8            7    87.5           7       87.5
  3    16        8            7    87.5           7       87.5
  4    16        8            7    87.5           7       87.5
  5    16        8            7    87.5           7       87.5
  6    16        8            7    87.5           6       75.0
  7    16        8            7    87.5           7       87.5
  8    16        8            7    87.5           7       87.5
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         64           56    87.5          54       84.4
-----------------------------------------------------------------
```

File Name: zn-16
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Zn Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

LEVEL:	1	2	3	4	5	6	7	8
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)	Pair 2 (Low)	Pair 2 (High)	Pair 3 (Low)	Pair 3 (High)	Pair 4 (Low)	Pair 4 (High)
CONCENTRATION:	.9413	.9413	1.0713	1.2713	1.4713	1.4713	1.7613	2.1613
RECOVERY:								
Observations	6	7	7	7	7	6	7	7
Mean Result	.8165	.9784	.9967	1.2711	2.3827	1.5244	1.7416	2.0511
Bias	-.1248	.0371	-.0746	-.0002	.9114	.0531	-.0197	-.1102
Relative Bias %	-13.2618	3.9459	-6.9675	-.0180	61.9433	3.6090	-1.1193	-5.1001
Maximum Result	1.0932	1.6887	1.4150	1.8460	4.6312	2.3680	2.1890	2.5790
Minimum Result	.5140	.4470	.6370	.9220	1.2800	1.1660	1.3807	1.7278

SINGLE OPERATOR PRECISION:	Pair 1	Pair 2	Pair 3	Pair 4
Observations	6	7	6	7
Standard Deviation	.1840	.0938	.5363	.1012
Correction Factor	1.0509	1.0424	1.0509	1.0424
Corrected Std Dev	.1934	.0977	.5636	.1055
Relative Std Dev (%)	21.3979	8.6209	28.3726	5.5651

OVERALL PRECISION:								
Observations	6	7	7	7	7	6	7	7
Standard Deviation	.2166	.4194	.2441	.3044	1.4626	.4349	.2967	.2856
Correction Factor	1.0509	1.0424	1.0424	1.0424	1.0424	1.0509	1.0424	1.0424
Corrected Std Dev	.2277	.4371	.2544	.3173	1.5246	.4571	.3092	.2977
Relative Std Dev %	27.8832	44.6749	25.5278	24.9647	63.9852	29.9846	17.7549	14.5124

File Name: zn-16
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Zn Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.9413	.8165	-.1248	-13.26	1.412	4.032	NO
2	.9413	.9784	.0371	3.95	.234	3.707	NO
3	1.0713	.9967	-.0746	-6.97	.809	3.707	NO
4	1.2713	1.2711	-.0002	-.02	.002	3.707	NO
5	1.4713	2.3827	.9114	61.94	1.649	3.707	NO
6	1.4713	1.5244	.0531	3.61	.299	4.032	NO
7	1.7613	1.7416	-.0197	-1.12	.176	3.707	NO
8	2.1613	2.0511	-.1102	-5.10	1.021	3.707	NO

```
File Name:  zn-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***   Single Operator Precision    ***
----------------------------------------
Analyte:  Zn          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision ***

    - Linear Model - s = b*T + a
    ============================
      Intercept (a):      .2424
          Slope (b):     -.0109
    ============================
                                          Single
           Sample Weights                Operator   Estimated
    Pair   Size       (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6     22.65       .9413       .1934       .2321
      2       7     27.35      1.1713       .0977       .2296
      3       6     22.65      1.4713       .5636       .2263
      4       7     27.35      1.9613       .1055       .2209
    ------------------------------------------------------------



    - Curvilinear Model - s = a*(b**T)  ( ln s = b'*T + a')
    ======================================================
      a:      .2355      Intercept (a'):    -1.4460
      b:      .8034          Slope (b'):     -.2189
    ======================================================
                                             Single
           Sample Weight                   Operator   Estimated
    Pair   Size      (%)        Conc     Std Dev     Std Dev
    ------------------------------------------------------------
      1       6    22.65       .9413       .1934       .1917
      2       7    27.35       .9413       .0977       .1823
      3       6    22.65      1.0713       .5636       .1707
      4       7    27.35      1.2713       .1055       .1533
    ------------------------------------------------------------
```

File Name: zn-16
Statistical Analysis File (.STT)

*** Performance Estimation Results ***
*** Overall Precision ***

Analyte: Zn Matrix: Freshwater (using MSA as true)
Project: EPA/EPRI Method: 1638
Date: 06/28/2000
Units: ug/L

*** Overall Precision ***

- Linear Model - s = b*T + a

Intercept (a):	.3193
Slope (b):	.1187

Conc Level	Sample Size	Weights (%)	Conc	Overall Std Dev	Estimated Std Dev
1	6	12.22	.9413	.2277	.4310
2	7	14.76	.9413	.4371	.4310
3	7	14.20	1.0713	.2544	.4464
4	7	13.40	1.2713	.3173	.4702
5	7	12.67	1.4713	1.5246	.4939
6	6	10.49	1.4713	.4571	.4939
7	7	11.71	1.7613	.3092	.5283
8	7	10.55	2.1613	.2977	.5758

- Curvilinear Model - s = a*(b**T) (ln s = b'*T + a')

a:	.3353	Intercept (a'):	-1.0927
b:	1.1173	Slope (b'):	.1109

Conc Level	Sample Size	Weight (%)	Conc	Overall Std Dev	Estimated Std Dev
1	6	10.82	.9413	.2277	.3722
2	7	13.06	.9413	.4371	.3722
3	7	13.06	1.0713	.2544	.3776
4	7	13.06	1.2713	.3173	.3861
5	7	13.06	1.4713	1.5246	.3948
6	6	10.82	1.4713	.4571	.3948
7	7	13.06	1.7613	.3092	.4077
8	7	13.06	2.1613	.2977	.4261

```
File Name:  zn-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ************  Recovery  **************
----------------------------------------
Analyte:  Zn          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Recovery ***

    - Linear Model - X = b*T + a
    ============================
      Intercept (a):     -.0614
          Slope (b):     1.1086
    ============================
    Conc   Sample   Weights                  Mean   Estimated
    Level  Size         (%)      Conc      Result      Result
    ------------------------------------------------------------
      1       6     13.67       .9413       .8165       .9821
      2       7     15.95       .9413       .9784       .9821
      3       7     14.87      1.0713       .9967      1.1263
      4       7     13.40      1.2713      1.2711      1.3480
      5       7     12.15      1.4713      2.3827      1.5697
      6       6     10.41      1.4713      1.5244      1.5697
      7       7     10.61      1.7613      1.7416      1.8912
      8       7      8.94      2.1613      2.0511      2.3346
    ------------------------------------------------------------


File Name:  zn-16
Statistical Analysis File (.STT)


----------------------------------------
 *** Performance Estimation Results ***
 ***     Precision vs Recovery      ***
----------------------------------------
Analyte:  Zn          Matrix:  Freshwater (using MSA as true)
Project:  EPA/EPRI    Method:  1638
   Date:  06/28/2000
  Units:  ug/L

 *** Single Operator Precision vs Recovery ***
          (Obtained by Substitution)

     - Linear Model - s = f*X + e
    ============================
        Intercept (e):      .2978
            Slope (f):     -.0099
    ============================


     - Curvilinear Model - s = e*(f**X)
    ==================================
        Intercept (e):      .2327
                  (f):      .8208
    ==================================



 *** Overall Precision vs Recovery ***
       (Obtained by Substitution)

     - Linear Model - s = f*X + e
     ============================
        Intercept (e):      .3746
            Slope (f):      .1071
     ============================


     - Curvilinear Model - s = e*(f**X)
     ==================================
        Intercept (e):      .3374
                  (f):     1.1052
     ==================================
```

```
Sb-17.dat
Sb    17   1     1.0900    0.9940
Sb    17   2     0.9923    1.0330
Sb    17   3     1.0196    0.9876
Sb    17   4     0.9800    1.1340
Sb    17   5     0.8571    1.0284
Sb    17   6     1.0240    1.1957
Sb    17   7     1.1239    1.0088
Sb    17   8     1.0597    1.0647

File Name:  sb-17
Data Validation File (.DA~)

*******************************************************************************
***                    Parameter and Data Validation File                   ***
*******************************************************************************
   Analyte:  Sb              Matrix:  Filtered Effluent (using Frontier as tru
   Project:  EPA/EPRI     Matrix ID:  17
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike          .9860      .0000
Spike
Increment      .0000      .0000
---------------------------------

                 Final Concentration
          ------------------------
Lab ID         .9860      .9860
---------------------------------
    1         1.0900      .9940
    2          .9923     1.0330
    3         1.0196      .9876
    4          .9800     1.1340
    5          .8571     1.0284
    6         1.0240     1.1957
    7         1.1239     1.0088
    8         1.0597     1.0647
---------------------------------


Sb-17.daf
Sb    17   1     1.0900     .9940
Sb    17   2      .9923    1.0330
Sb    17   3     1.0196     .9876
Sb    17   4      .9800    1.1340
Sb    17   5      .8571    1.0284
Sb    17   6     1.0240    1.1957
Sb    17   7     1.1239    1.0088
Sb    17   8     1.0597    1.0647
```

```
File Name:  sb-17
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  sb-17
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  sb-17
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------


-------------------------------------------
 - No Outlier Rejections in Iteration 1 -
 - No Further Outlier Testing Necessary -
-------------------------------------------
```

```
File Name:  sb-17
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     8     W     A              .9395      .818   ---
     2     8     W     A              .8613      .818   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  sb-17
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    17        8            8   100.0           8      100.0
  2    17        8            8   100.0           8      100.0
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          16      100.0
---------------------------------------------------------------
```

```
File Name:  sb-17
Statistical Analysis File (.STT)


----------------------------------------
 *** Summary Performance Statistics ***
----------------------------------------
Analyte:  Sb          Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI    Method:  1638
   Date:  08/28/2000
  Units:  ug/L

----------------------------------------
           LEVEL:         1         2
----------------------------------------
YOUDEN PAIRS:         Pair 1    Pair 1
                       (Low)    (High)
----------------------------------------
CONCENTRATION:         .9860     .9860
----------------------------------------
RECOVERY:
 Observations              8         8
 Mean Result          1.0183    1.0558
 Bias                  .0323     .0698
 Relative Bias %      3.2784    7.0766
 Maximum Result       1.1239    1.1957
 Minimum Result        .8571     .9876
----------------------------------------
SINGLE OPERATOR PRECISION:   Pair 1
   Observations                 8
   Standard Deviation         .0830
   Correction Factor         1.0362
   Corrected Std Dev          .0860
   Relative Std Dev (%)      8.2954
----------------------------------------
OVERALL PRECISION:
 Observations               8         8
 Standard Deviation     .0812     .0734
 Correction Factor     1.0362    1.0362
 Corrected Std Dev      .0842     .0761
 Relative Std Dev %    8.2640    7.2044
----------------------------------------



File Name:  sb-17
Statistical Analysis File (.STT)


----------------------------------
  *** Results of Bias Testing ***
----------------------------------
 Analyte:  Sb          Matrix:  Filtered Effluent (using Frontier as tru
 Project:  EPA/EPRI    Method:  1638
    Date:  08/28/2000
   Units:  ug/L

-------------------------------------------------------------------------
                                 Rel.    Obs     Crit   Statistically
Conc.            Mean            Bias      t        t     Significant
Level   Conc    Result      Bias     (%)   Value   Value   (1% Two-Tail)
-------------------------------------------------------------------------
 1     .9860    1.0183     .0323    3.28   1.126   3.499         NO
 2     .9860    1.0558     .0698    7.08   2.689   3.499         NO
-------------------------------------------------------------------------
```

```
Sb-18.dat
Sb    18   1     9.7600    9.6000
Sb    18   2     8.4000    9.0020
Sb    18   3     9.0936    9.2436
Sb    18   4     8.7510    8.9420
Sb    18   5
Sb    18   6     9.9574    9.6147
Sb    18   7     9.8225    9.9053
Sb    18   8     9.9690    9.8699

File Name:  sb-18
Data Validation File (.DA~)

*******************************************************************************
***                    Parameter and Data Validation File                   ***
*******************************************************************************
   Analyte:  Sb              Matrix:  UnFiltered Effluent (using Frontier as t
   Project:  EPA/EPRI     Matrix ID:  18
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike        10.0110      .0000
Spike
Increment      .0000      .0000
---------------------------------

                Final Concentration
          ------------------------
Lab ID       10.0110    10.0110
---------------------------------
    1         9.7600     9.6000
    2         8.4000     9.0020
    3         9.0936     9.2436
    4         8.7510     8.9420
    5    e     .0000e     .0000
    6         9.9574     9.6147
    7         9.8225     9.9053
    8         9.9690     9.8699
---------------------------------

Sb-18.daf
Sb    18   1     9.7600    9.6000
Sb    18   2     8.4000    9.0020
Sb    18   3     9.0936    9.2436
Sb    18   4     8.7510    8.9420
Sb    18   5
Sb    18   6     9.9574    9.6147
Sb    18   7     9.8225    9.9053
Sb    18   8     9.9690    9.8699
```

```
File Name:  sb-18
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  sb-18
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  sb-18
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------


-------------------------------------------
 - No Outlier Rejections in Iteration 1 -
 - No Further Outlier Testing Necessary -
-------------------------------------------
```

```
File Name:  sb-18
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
              Test  Normality      Test          Critical
   Level  n   Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     7     W     A              .8506      .803   ---
     2     7     W     A              .8968      .803   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  sb-18
Data Preparation File (.PRP)


Analyte:  Sb     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-------------------------------------------------
 ***         Data Removal Tracking          ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests           ***
-------------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    18        7            7   100.0           7      100.0
  2    18        7            7   100.0           7      100.0
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         14           14   100.0          14      100.0
------------------------
```

```
File Name:  sb-18
Statistical Analysis File (.STT)


----------------------------------------
 *** Summary Performance Statistics ***
----------------------------------------
Analyte:  Sb          Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI    Method:  1638
   Date:  08/28/2000
  Units:  ug/L

----------------------------------------
           LEVEL:         1         2
----------------------------------------
YOUDEN PAIRS:         Pair 1    Pair 1
                       (Low)    (High)
----------------------------------------
CONCENTRATION:       10.0110   10.0110
----------------------------------------
RECOVERY:
 Observations              7         7
 Mean Result          9.3934    9.4539
 Bias                 -.6176    -.5571
 Relative Bias %     -6.1696   -5.5646
 Maximum Result       9.9690    9.9053
 Minimum Result       8.4000    8.9420
----------------------------------------
SINGLE OPERATOR PRECISION:   Pair 1
   Observations                 7
   Standard Deviation         .2151
   Correction Factor         1.0424
   Corrected Std Dev          .2243
   Relative Std Dev (%)      2.3797
----------------------------------------
OVERALL PRECISION:
 Observations              7         7
 Standard Deviation     .6400     .3946
 Correction Factor     1.0424    1.0424
 Corrected Std Dev      .6671     .4114
 Relative Std Dev %    7.1016    4.3511
----------------------------------------



File Name:  sb-18
Statistical Analysis File (.STT)


----------------------------------
  *** Results of Bias Testing ***
----------------------------------
 Analyte:  Sb          Matrix:  UnFiltered Effluent (using Frontier as t
 Project:  EPA/EPRI    Method:  1638
    Date:  08/28/2000
   Units:  ug/L

---------------------------------------------------------------------------
                                 Rel.    Obs     Crit   Statistically
Conc.            Mean            Bias      t        t     Significant
Level   Conc    Result      Bias   (%)   Value   Value   (1% Two-Tail)
---------------------------------------------------------------------------
 1   10.0110    9.3934    -.6176   -6.17   2.553   3.707         NO
 2   10.0110    9.4539    -.5571   -5.56   3.735   3.707        YES
---------------------------------------------------------------------------
```

```
Cd-17.dat
Cd    17   1     0.3040   0.2910
Cd    17   2     0.1854   0.2856
Cd    17   3     0.3243   0.3054
Cd    17   4     0.1990   0.3680
Cd    17   5     0.3996   0.4039
Cd    17   6     0.3644   0.3490
Cd    17   7     0.1585   0.1832
Cd    17   8     0.2916   0.2988

File Name:  cd-17
Data Validation File (.DA~)

*****************************************************************************
***                    Parameter and Data Validation File                 ***
*****************************************************************************
   Analyte:  Cd              Matrix:  Filtered Effluent (using Frontier as tru
   Project:  EPA/EPRI     Matrix ID:  17
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike          .3080      .0000
Spike
Increment      .0000      .0000
---------------------------------

                 Final Concentration
          ------------------------
Lab ID         .3080      .3080
---------------------------------
    1          .3040      .2910
    2          .1854      .2856
    3          .3243      .3054
    4          .1990      .3680
    5          .3996      .4039
    6          .3644      .3490
    7          .1585      .1832
    8          .2916      .2988
---------------------------------

Cd-17.daf
Cd    17   1      .3040     .2910
Cd    17   2      .1854     .2856
Cd    17   3      .3243     .3054
Cd    17   4      .1990     .3680
Cd    17   5      .3996     .4039
Cd    17   6      .3644     .3490
Cd    17   7      .1585     .1832
Cd    17   8      .2916     .2988
```

```
File Name:  cd-17
Data Preparation File (.PRP)


Analyte:  Cd     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  cd-17
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  cd-17
Data Preparation File (.PRP)


Analyte:  Cd     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------


-------------------------------------------
 - No Outlier Rejections in Iteration 1 -
 - No Further Outlier Testing Necessary -
-------------------------------------------
```

```
File Name:  cd-17
Data Preparation File (.PRP)


Analyte:  Cd     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     8     W     A              .9321      .818   ---
     2     8     W     A              .9372      .818   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  cd-17
Data Preparation File (.PRP)


Analyte:  Cd     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    17        8            8   100.0           8      100.0
  2    17        8            8   100.0           8      100.0
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          16      100.0
---------------------
```

```
File Name:  cd-17
Statistical Analysis File (.STT)


----------------------------------------
 *** Summary Performance Statistics ***
----------------------------------------
Analyte:  Cd          Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI    Method:  1638
   Date:  08/28/2000
  Units:  ug/L

----------------------------------------
            LEVEL:         1         2
----------------------------------------
YOUDEN PAIRS:         Pair 1    Pair 1
                       (Low)    (High)
----------------------------------------
CONCENTRATION:         .3080     .3080
----------------------------------------
RECOVERY:
 Observations              8         8
 Mean Result           .2783     .3106
 Bias                 -.0297     .0026
 Relative Bias %     -9.6266     .8482
 Maximum Result        .3996     .4039
 Minimum Result        .1585     .1832
----------------------------------------
SINGLE OPERATOR PRECISION:   Pair 1
   Observations                 8
   Standard Deviation         .0476
   Correction Factor         1.0362
   Corrected Std Dev          .0493
   Relative Std Dev (%)     16.7490
----------------------------------------
OVERALL PRECISION:
 Observations              8         8
 Standard Deviation     .0881     .0664
 Correction Factor     1.0362    1.0362
 Corrected Std Dev      .0913     .0688
 Relative Std Dev %   32.8037   22.1581
----------------------------------------



File Name:  cd-17
Statistical Analysis File (.STT)


---------------------------------
  *** Results of Bias Testing ***
---------------------------------
 Analyte:  Cd          Matrix:  Filtered Effluent (using Frontier as tru
 Project:  EPA/EPRI    Method:  1638
    Date:  08/28/2000
   Units:  ug/L

------------------------------------------------------------------------
                                  Rel.    Obs     Crit   Statistically
Conc.             Mean            Bias      t        t     Significant
Level   Conc    Result      Bias     (%)   Value   Value   (1% Two-Tail)
------------------------------------------------------------------------
 1     .3080     .2783    -.0297   -9.63    .952   3.499         NO
 2     .3080     .3106     .0026     .85    .111   3.499         NO
------------------------------------------------------------------------
```

```
Cd-18.dat
Cd    18   1     0.4620   0.4940
Cd    18   2     0.4082   0.4050
Cd    18   3     0.5163   0.5320
Cd    18   4     0.3690   0.3580
Cd    18   5     0.6642   0.6992
Cd    18   6     0.5390   0.5277
Cd    18   7     0.4040   0.3690
Cd    18   8     0.4775   0.4846

File Name:  cd-18
Data Validation File (.DA~)

*******************************************************************************
***                    Parameter and Data Validation File                   ***
*******************************************************************************
   Analyte:  Cd              Matrix:  UnFiltered Effluent (using Frontier as t
   Project:  EPA/EPRI     Matrix ID:  18
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike          .5050      .0000
Spike
Increment      .0000      .0000
---------------------------------

                 Final Concentration
          ------------------------
Lab ID         .5050      .5050
---------------------------------
    1          .4620      .4940
    2          .4082      .4050
    3          .5163      .5320
    4          .3690      .3580
    5          .6642      .6992
    6          .5390      .5277
    7          .4040      .3690
    8          .4775      .4846
---------------------------------

Cd-18.daf
Cd    18   1      .4620     .4940
Cd    18   2      .4082     .4050
Cd    18   3      .5163     .5320
Cd    18   4      .3690     .3580
Cd    18   5      .6642     .6992
Cd    18   6      .5390     .5277
Cd    18   7      .4040     .3690
Cd    18   8      .4775     .4846
```

```
File Name:  cd-18
Data Preparation File (.PRP)


Analyte:  Cd     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  cd-18
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  cd-18
Data Preparation File (.PRP)


Analyte:  Cd     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------


-------------------------------------------
 - No Outlier Rejections in Iteration 1 -
 - No Further Outlier Testing Necessary -
-------------------------------------------
```

```
File Name:  cd-18
Data Preparation File (.PRP)


Analyte:  Cd     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     8     W     A              .9327      .818   ---
     2     8     W     A              .9119      .818   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  cd-18
Data Preparation File (.PRP)


Analyte:  Cd     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-------------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-------------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    18        8            8   100.0           8      100.0
  2    18        8            8   100.0           8      100.0
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          16      100.0
---------------------------------------------------------------
```

```
File Name:  cd-18
Statistical Analysis File (.STT)


----------------------------------------
 *** Summary Performance Statistics ***
----------------------------------------
Analyte:  Cd          Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI    Method:  1638
   Date:  08/28/2000
  Units:  ug/L

----------------------------------------
           LEVEL:         1         2
----------------------------------------
YOUDEN PAIRS:         Pair 1    Pair 1
                       (Low)    (High)
----------------------------------------
CONCENTRATION:          .5050     .5050
----------------------------------------
RECOVERY:
 Observations              8         8
 Mean Result           .4800     .4837
 Bias                 -.0250    -.0213
 Relative Bias %     -4.9455   -4.2203
 Maximum Result        .6642     .6992
 Minimum Result        .3690     .3580
----------------------------------------
SINGLE OPERATOR PRECISION:   Pair 1
   Observations                 8
   Standard Deviation         .0167
   Correction Factor         1.0362
   Corrected Std Dev          .0173
   Relative Std Dev (%)      3.5996
----------------------------------------
OVERALL PRECISION:
 Observations              8         8
 Standard Deviation     .0943     .1108
 Correction Factor     1.0362    1.0362
 Corrected Std Dev      .0978     .1148
 Relative Std Dev %   20.3670   23.7416
----------------------------------------



File Name:  cd-18
Statistical Analysis File (.STT)


----------------------------------
  *** Results of Bias Testing ***
----------------------------------
 Analyte:  Cd          Matrix:  UnFiltered Effluent (using Frontier as t
 Project:  EPA/EPRI    Method:  1638
    Date:  08/28/2000
   Units:  ug/L

---------------------------------------------------------------------------
                                 Rel.    Obs     Crit   Statistically
Conc.             Mean           Bias      t        t     Significant
Level   Conc    Result      Bias     (%)   Value   Value   (1% Two-Tail)
---------------------------------------------------------------------------
 1     .5050     .4800    -.0250   -4.95    .749   3.499         NO
 2     .5050     .4837    -.0213   -4.22    .544   3.499         NO
---------------------------------------------------------------------------
```

```
Cu-17.dat
Cu    17   1    14.0000   13.0000
Cu    17   2    13.8600   13.1800
Cu    17   3    14.6823   15.2322
Cu    17   4    16.1150   17.7950
Cu    17   5    15.4151   14.4274
Cu    17   6    12.7543   13.7450
Cu    17   7    14.6566   14.7489
Cu    17   8    13.8010   13.6680

File Name:  cu-17
Data Validation File (.DA~)

*******************************************************************************
***                    Parameter and Data Validation File                   ***
*******************************************************************************
   Analyte:  Cu              Matrix:  Filtered Effluent (using Frontier as tru
   Project:  EPA/EPRI     Matrix ID:  17
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike        13.8000      .0000
Spike
Increment      .0000      .0000
---------------------------------

                Final Concentration
          ------------------------
Lab ID       13.8000    13.8000
---------------------------------
    1        14.0000    13.0000
    2        13.8600    13.1800
    3        14.6823    15.2322
    4        16.1150o   17.7950
    5        15.4151    14.4274
    6        12.7543    13.7450
    7        14.6566    14.7489
    8        13.8010    13.6680
---------------------------------

Cu-17.daf
Cu    17   1    14.0000   13.0000
Cu    17   2    13.8600   13.1800
Cu    17   3    14.6823   15.2322
Cu    17   4    16.1150o
Cu    17   5    15.4151   14.4274
Cu    17   6    12.7543   13.7450
Cu    17   7    14.6566   14.7489
Cu    17   8    13.8010   13.6680
```

```
File Name:  cu-17
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  cu-17
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  cu-17
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
-------------------------------------------------------------------------------
  2     1     4   1     17.7950    14.4746     1.5450   2.149   2.126    8
-------------------------------------------------------------------------------
```

```
File Name:  cu-17
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     8     W     A              .9724      .818   ---
     2     7     W     A              .9475      .803   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  cu-17
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-------------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-------------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    17        8            8   100.0           8      100.0
  2    17        8            8   100.0           7       87.5
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          15       93.8
-----------
```

```
File Name:  cu-17
Statistical Analysis File (.STT)


----------------------------------------
 *** Summary Performance Statistics ***
----------------------------------------
Analyte:  Cu          Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI    Method:  1638
   Date:  08/28/2000
  Units:  ug/L

----------------------------------------
           LEVEL:         1         2
----------------------------------------
YOUDEN PAIRS:         Pair 1    Pair 1
                       (Low)    (High)
----------------------------------------
CONCENTRATION:       13.8000   13.8000
----------------------------------------
RECOVERY:
 Observations              8         7
 Mean Result         14.4105   14.0002
 Bias                  .6105     .2002
 Relative Bias %      4.4242    1.4508
 Maximum Result      16.1150   15.2322
 Minimum Result      12.7543   13.0000
----------------------------------------
SINGLE OPERATOR PRECISION:   Pair 1
   Observations                 7
   Standard Deviation         .5442
   Correction Factor         1.0424
   Corrected Std Dev          .5672
   Relative Std Dev (%)      3.9890
----------------------------------------
OVERALL PRECISION:
 Observations              8         7
 Standard Deviation    1.0444     .8276
 Correction Factor     1.0362    1.0424
 Corrected Std Dev     1.0822     .8626
 Relative Std Dev %    7.5098    6.1616
----------------------------------------



File Name:  cu-17
Statistical Analysis File (.STT)


----------------------------------
  *** Results of Bias Testing ***
----------------------------------
 Analyte:  Cu          Matrix:  Filtered Effluent (using Frontier as tru
 Project:  EPA/EPRI    Method:  1638
    Date:  08/28/2000
   Units:  ug/L

---------------------------------------------------------------------------
                                 Rel.    Obs     Crit   Statistically
Conc.            Mean            Bias      t        t     Significant
Level   Conc    Result      Bias   (%)   Value   Value   (1% Two-Tail)
---------------------------------------------------------------------------
 1   13.8000   14.4105     .6105    4.42   1.654   3.499         NO
 2   13.8000   14.0002     .2002    1.45    .640   3.707         NO
---------------------------------------------------------------------------
```

```
Cu-18.dat
Cu    18   1    14.8000   14.4000
Cu    18   2    15.5200   15.2000
Cu    18   3    17.6339   16.2906
Cu    18   4    17.5450   20.1940
Cu    18   5    18.9784   17.9623
Cu    18   6    17.4718   14.4560
Cu    18   7    15.1818   15.3126
Cu    18   8    15.4382   15.5194

File Name:  cu-18
Data Validation File (.DA~)

*******************************************************************************
***                    Parameter and Data Validation File                   ***
*******************************************************************************
   Analyte:  Cu              Matrix:  UnFiltered Effluent (using Frontier as t
   Project:  EPA/EPRI     Matrix ID:  18
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike        15.1000      .0000
Spike
Increment      .0000      .0000
---------------------------------

                Final Concentration
          ------------------------
Lab ID       15.1000    15.1000
---------------------------------
    1        14.8000    14.4000
    2        15.5200    15.2000
    3        17.6339    16.2906
    4        17.5450    20.1940
    5        18.9784    17.9623
    6        17.4718    14.4560
    7        15.1818    15.3126
    8        15.4382    15.5194
---------------------------------

Cu-18.daf
Cu    18   1    14.8000   14.4000
Cu    18   2    15.5200   15.2000
Cu    18   3    17.6339   16.2906
Cu    18   4    17.5450   20.1940
Cu    18   5    18.9784   17.9623
Cu    18   6    17.4718   14.4560
Cu    18   7    15.1818   15.3126
Cu    18   8    15.4382   15.5194
```

```
File Name:  cu-18
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  cu-18
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  cu-18
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------


-------------------------------------------
 - No Outlier Rejections in Iteration 1 -
 - No Further Outlier Testing Necessary -
-------------------------------------------
```

```
File Name:  cu-18
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     8     W     A              .8859      .818   ---
     2     8     W     A              .8412      .818   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  cu-18
Data Preparation File (.PRP)


Analyte:  Cu     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    18        8            8   100.0           8      100.0
  2    18        8            8   100.0           8      100.0
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          16      100.0
---------------------------------------------------------------
```

File Name: cu-18
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Cu Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

LEVEL:	1	2
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)
CONCENTRATION:	15.1000	15.1000
RECOVERY:		
Observations	8	8
Mean Result	16.5711	16.1669
Bias	1.4711	1.0669
Relative Bias %	9.7426	7.0653
Maximum Result	18.9784	20.1940
Minimum Result	14.8000	14.4000

SINGLE OPERATOR PRECISION:	Pair 1
Observations	8
Standard Deviation	1.1316
Correction Factor	1.0362
Corrected Std Dev	1.1726
Relative Std Dev (%)	7.1637

OVERALL PRECISION:		
Observations	8	8
Standard Deviation	1.5184	1.9853
Correction Factor	1.0362	1.0362
Corrected Std Dev	1.5735	2.0572
Relative Std Dev %	9.4951	12.7250

File Name: cu-18
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Cu Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	15.1000	16.5711	1.4711	9.74	2.740	3.499	NO
2	15.1000	16.1669	1.0669	7.07	1.520	3.499	NO

```
Pb-17.dat
Pb    17   1     0.5310    0.4870
Pb    17   2     0.6331    0.7302
Pb    17   3     0.6978    0.6215
Pb    17   4     0.5650    0.7330
Pb    17   5     0.5874    0.5899
Pb    17   6     0.6123    0.6286
Pb    17   7     0.5339    0.5326
Pb    17   8     0.6138    0.5709

File Name:  pb-17
Data Validation File (.DA~)

*******************************************************************************
***                    Parameter and Data Validation File                   ***
*******************************************************************************
   Analyte:  Pb              Matrix:  Filtered Effluent (using Frontier as tru
   Project:  EPA/EPRI     Matrix ID:  17
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike          .5310      .0000
Spike
Increment      .0000      .0000
---------------------------------

                 Final Concentration
          ------------------------
Lab ID         .5310      .5310
---------------------------------
    1          .5310      .4870
    2          .6331      .7302
    3          .6978      .6215
    4          .5650      .7330
    5          .5874      .5899
    6          .6123      .6286
    7          .5339      .5326
    8          .6138      .5709
---------------------------------

Pb-17.daf
Pb    17   1      .5310     .4870
Pb    17   2      .6331     .7302
Pb    17   3      .6978     .6215
Pb    17   4      .5650     .7330
Pb    17   5      .5874     .5899
Pb    17   6      .6123     .6286
Pb    17   7      .5339     .5326
Pb    17   8      .6138     .5709
```

```
File Name:  pb-17
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  pb-17
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  pb-17
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------


-------------------------------------------
 - No Outlier Rejections in Iteration 1 -
 - No Further Outlier Testing Necessary -
-------------------------------------------
```

```
File Name:  pb-17
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     8     W     A              .9439      .818   ---
     2     8     W     A              .9387      .818   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  pb-17
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-------------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-------------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    17        8            8   100.0           8      100.0
  2    17        8            8   100.0           8      100.0
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          16      100.0
--------------------
```

```
File Name:  pb-17
Statistical Analysis File (.STT)


----------------------------------------
 *** Summary Performance Statistics ***
----------------------------------------
Analyte:  Pb          Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI    Method:  1638
   Date:  08/28/2000
  Units:  ug/L

----------------------------------------
           LEVEL:         1         2
----------------------------------------
YOUDEN PAIRS:         Pair 1    Pair 1
                       (Low)    (High)
----------------------------------------
CONCENTRATION:          .5310     .5310
----------------------------------------
RECOVERY:
 Observations              8         8
 Mean Result           .5968     .6117
 Bias                  .0658     .0807
 Relative Bias %     12.3893   15.2001
 Maximum Result        .6978     .7330
 Minimum Result        .5310     .4870
----------------------------------------
SINGLE OPERATOR PRECISION:   Pair 1
   Observations                 8
   Standard Deviation         .0572
   Correction Factor         1.0362
   Corrected Std Dev          .0592
   Relative Std Dev (%)      9.8025
----------------------------------------
OVERALL PRECISION:
 Observations              8         8
 Standard Deviation     .0553     .0871
 Correction Factor     1.0362    1.0362
 Corrected Std Dev      .0573     .0902
 Relative Std Dev %    9.6082   14.7518
----------------------------------------



File Name:  pb-17
Statistical Analysis File (.STT)


----------------------------------
  *** Results of Bias Testing ***
----------------------------------
 Analyte:  Pb          Matrix:  Filtered Effluent (using Frontier as tru
 Project:  EPA/EPRI    Method:  1638
    Date:  08/28/2000
   Units:  ug/L

---------------------------------------------------------------------------
                                 Rel.    Obs     Crit   Statistically
Conc.             Mean           Bias      t        t     Significant
Level   Conc    Result      Bias   (%)   Value   Value   (1% Two-Tail)
---------------------------------------------------------------------------
 1     .5310     .5968     .0658   12.39   3.363   3.499         NO
 2     .5310     .6117     .0807   15.20   2.622   3.499         NO
---------------------------------------------------------------------------
```

```
Pb-18.dat
Pb    18   1     0.9680    0.9730
Pb    18   2     1.1380    1.1410
Pb    18   3     1.2198    1.1774
Pb    18   4     1.0530    1.0840
Pb    18   5     1.7818    2.7456
Pb    18   6     1.1253    1.1737
Pb    18   7     0.8502    0.8549
Pb    18   8     1.1202    1.1145

File Name:  pb-18
Data Validation File (.DA~)

*******************************************************************************
***                    Parameter and Data Validation File                   ***
*******************************************************************************
   Analyte:  Pb              Matrix:  UnFiltered Effluent (using Frontier as t
   Project:  EPA/EPRI     Matrix ID:  18
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike         1.1100      .0000
Spike
Increment      .0000      .0000
---------------------------------

                Final Concentration
          ------------------------
Lab ID        1.1100     1.1100
---------------------------------
    1          .9680      .9730
    2         1.1380     1.1410
    3         1.2198     1.1774
    4         1.0530     1.0840
    5    o    1.7818o    2.7456
    6         1.1253     1.1737
    7          .8502      .8549
    8         1.1202     1.1145
---------------------------------

Pb-18.daf
Pb    18   1      .9680     .9730
Pb    18   2     1.1380    1.1410
Pb    18   3     1.2198    1.1774
Pb    18   4     1.0530    1.0840
Pb    18   5 o         o
Pb    18   6     1.1253    1.1737
Pb    18   7      .8502     .8549
Pb    18   8     1.1202    1.1145
```

```
File Name:  pb-18
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  pb-18
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  pb-18
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
-------------------------------------------------------------------------------
  1     1     5   1      1.7818     1.1570      .2772   2.254   2.126    8
  2     1     5   1      2.7456     1.2830      .6012   2.433   2.126    8
-------------------------------------------------------------------------------
```

```
File Name:  pb-18
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     7     W     A              .9309      .803   ---
     2     7     W     A              .8559      .803   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  pb-18
Data Preparation File (.PRP)


Analyte:  Pb     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    18        8            8   100.0           7       87.5
  2    18        8            8   100.0           7       87.5
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          14       87.5
---------------------------------------------------------------
```

File Name: pb-18
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Pb Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

LEVEL:	1	2
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)
CONCENTRATION:	1.1100	1.1100
RECOVERY:		
Observations	7	7
Mean Result	1.0678	1.0741
Bias	-.0422	-.0359
Relative Bias %	-3.8031	-3.2368
Maximum Result	1.2198	1.1774
Minimum Result	.8502	.8549

SINGLE OPERATOR PRECISION:	Pair 1
Observations	7
Standard Deviation	.0202
Correction Factor	1.0424
Corrected Std Dev	.0211
Relative Std Dev (%)	1.9695

OVERALL PRECISION:		
Observations	7	7
Standard Deviation	.1236	.1191
Correction Factor	1.0424	1.0424
Corrected Std Dev	.1288	.1241
Relative Std Dev %	12.0623	11.5554

File Name: pb-18
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Pb Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	1.1100	1.0678	-.0422	-3.80	.904	3.707	NO
2	1.1100	1.0741	-.0359	-3.24	.798	3.707	NO

```
Ni-17.dat
Ni    17   1     7.1100    6.5200
Ni    17   2     7.0590    7.3030
Ni    17   3     6.6059    6.3867
Ni    17   4     6.8580    7.1600
Ni    17   5     6.9976    7.0286
Ni    17   6     4.5314    5.1111
Ni    17   7     6.9029    6.8971
Ni    17   8     6.9385    6.7290

File Name:  ni-17
Data Validation File (.DA~)

*******************************************************************************
***                    Parameter and Data Validation File                   ***
*******************************************************************************
   Analyte:  Ni              Matrix:  Filtered Effluent (using Frontier as tru
   Project:  EPA/EPRI     Matrix ID:  17
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike         6.8926      .0000
Spike
Increment      .0000      .0000
---------------------------------

                Final Concentration
          ------------------------
Lab ID        6.8926     6.8926
---------------------------------
    1         7.1100     6.5200
    2         7.0590     7.3030
    3         6.6059     6.3867
    4         6.8580     7.1600
    5         6.9976     7.0286
    6    o    4.5314o    5.1111
    7         6.9029     6.8971
    8         6.9385     6.7290
---------------------------------

Ni-17.daf
Ni    17   1     7.1100    6.5200
Ni    17   2     7.0590    7.3030
Ni    17   3     6.6059    6.3867
Ni    17   4     6.8580    7.1600
Ni    17   5     6.9976    7.0286
Ni    17   6 o         o
Ni    17   7     6.9029    6.8971
Ni    17   8     6.9385    6.7290
```

```
File Name:  ni-17
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  ni-17
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  ni-17
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
-------------------------------------------------------------------------------
  1     1     6   1      4.5314     6.6254      .8599   2.435   2.126    8
  2     1     6   1      5.1111     6.6419      .6919   2.213   2.126    8
-------------------------------------------------------------------------------
```

```
File Name:  ni-17
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     7     W     A              .9174      .803   ---
     2     7     W     A              .9681      .803   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  ni-17
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-------------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-------------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    17        8            8   100.0           7       87.5
  2    17        8            8   100.0           7       87.5
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          14       87.5
---------------------------------------------------------------
```

File Name: ni-17
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Ni Matrix: Filtered Effluent (using Frontier as tru
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

LEVEL:	1	2
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)
CONCENTRATION:	6.8926	6.8926
RECOVERY:		
Observations	7	7
Mean Result	6.9246	6.8606
Bias	.0320	-.0320
Relative Bias %	.4636	-.4639
Maximum Result	7.1100	7.3030
Minimum Result	6.6059	6.3867

SINGLE OPERATOR PRECISION:	Pair 1
Observations	7
Standard Deviation	.2166
Correction Factor	1.0424
Corrected Std Dev	.2258
Relative Std Dev (%)	3.2761

OVERALL PRECISION:		
Observations	7	7
Standard Deviation	.1655	.3349
Correction Factor	1.0424	1.0424
Corrected Std Dev	.1726	.3491
Relative Std Dev %	2.4919	5.0878

File Name: ni-17
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Ni Matrix: Filtered Effluent (using Frontier as tru
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	6.8926	6.9246	.0320	.46	.511	3.707	NO
2	6.8926	6.8606	-.0320	-.46	.253	3.707	NO

```
Ni-18.dat
Ni    18   1     7.5900    7.6000
Ni    18   2     7.6590    7.8510
Ni    18   3     7.0975    7.1137
Ni    18   4     8.9260    8.7740
Ni    18   5     9.0821    8.5336
Ni    18   6     6.6759    5.3956
Ni    18   7     7.3304    7.4195
Ni    18   8     7.6763    7.7658

File Name:  ni-18
Data Validation File (.DA~)

*****************************************************************************
***                    Parameter and Data Validation File                 ***
*****************************************************************************
   Analyte:  Ni              Matrix:  UnFiltered Effluent (using Frontier as t
   Project:  EPA/EPRI     Matrix ID:  18
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike         8.0100      .0000
Spike
Increment      .0000      .0000
---------------------------------

                Final Concentration
          ------------------------
Lab ID        8.0100     8.0100
---------------------------------
    1         7.5900     7.6000
    2         7.6590     7.8510
    3         7.0975     7.1137
    4         8.9260     8.7740
    5         9.0821     8.5336
    6         6.6759     5.3956
    7         7.3304     7.4195
    8         7.6763     7.7658
---------------------------------

Ni-18.daf
Ni    18   1     7.5900    7.6000
Ni    18   2     7.6590    7.8510
Ni    18   3     7.0975    7.1137
Ni    18   4     8.9260    8.7740
Ni    18   5     9.0821    8.5336
Ni    18   6     6.6759    5.3956
Ni    18   7     7.3304    7.4195
Ni    18   8     7.6763    7.7658
```

```
File Name:  ni-18
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  ni-18
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  ni-18
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------


-------------------------------------------
 - No Outlier Rejections in Iteration 1 -
 - No Further Outlier Testing Necessary -
-------------------------------------------
```

```
File Name:  ni-18
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     8     W     A              .8896      .818   ---
     2     8     W     A              .8919      .818   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  ni-18
Data Preparation File (.PRP)


Analyte:  Ni     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    18        8            8   100.0           8      100.0
  2    18        8            8   100.0           8      100.0
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          16      100.0
---------------------------------------------------------------
```

```
 Se-17.dat
Se    17   1     8.8700    8.6700
Se    17   2     7.2890    7.5030
Se    17   3     8.0103    8.1828
Se    17   4     6.2770    6.3980
Se    17   5     8.0251    7.4642
Se    17   6    24.8955   27.6746
Se    17   7     5.5799    5.4285
Se    17   8     5.3906    5.7788

File Name:  se-17
Data Validation File (.DA~)

********************************************************************************
***                    Parameter and Data Validation File                    ***
********************************************************************************
   Analyte:  Se              Matrix:  Filtered Effluent (using Frontier as tru
   Project:  EPA/EPRI     Matrix ID:  17
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike         7.0619      .0000
Spike
Increment      .0000      .0000
---------------------------------

                Final Concentration
          ------------------------
Lab ID        7.0619     7.0619
---------------------------------
    1         8.8700     8.6700
    2         7.2890     7.5030
    3         8.0103     8.1828
    4         6.2770     6.3980
    5         8.0251     7.4642
    6    o   24.8955o   27.6746
    7         5.5799     5.4285
    8         5.3906     5.7788
---------------------------------

se-17.daf
Se    17   1     8.8700    8.6700
Se    17   2     7.2890    7.5030
Se    17   3     8.0103    8.1828
Se    17   4     6.2770    6.3980
Se    17   5     8.0251    7.4642
Se    17   6 o         o
Se    17   7     5.5799    5.4285
Se    17   8     5.3906    5.7788
```

File Name: se-17
Data Preparation File (.PRP)

Analyte: Se Matrix: Filtered Effluent (using Frontier as tru
Project: EPA/EPRI
Date: 08/28/2000
Units: ug/L

*** Results of 5x - 1/5x Mean Error Check ***
*** Questionable Data (Positive Values) ***

Total Number of Questionable Observations: 0

*** Results of Factor of 5 Error Check ***
*** Questionable Data (All Values) ***

Total Number of Questionable Observations: 0

File Name: se-17
Data Preparation File (.PRP)

Insufficient Data to Complete Lab Ranking

File Name: se-17
Data Preparation File (.PRP)

Analyte: Se Matrix: Filtered Effluent (using Frontier as tru
Project: EPA/EPRI
Date: 08/28/2000
Units: ug/L

*** Outlier Testing Results ***
*** Two-Sided 5% Significance Level ***

- Outlier(s) -

Lev	Iter	Lab	Rep	Result	Mean	Std Dev	t	Crit t	n
1	1	6	1	24.8955	9.2922	6.4254	2.428	2.126	8
2	1	6	1	27.6746	9.6375	7.3753	2.446	2.126	8

```
File Name:  se-17
Data Preparation File (.PRP)


Analyte:  Se     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     7     W     A              .9243      .803   ---
     2     7     W     A              .9418      .803   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  se-17
Data Preparation File (.PRP)


Analyte:  Se     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    17        8            8   100.0           7       87.5
  2    17        8            8   100.0           7       87.5
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          14       87.5
---------------------------------------------------------------
```

File Name: se-17
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Se Matrix: Filtered Effluent (using Frontier as tru
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

LEVEL:	1	2
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)
CONCENTRATION:	7.0619	7.0619
RECOVERY:		
Observations	7	7
Mean Result	7.0631	7.0608
Bias	.0012	-.0011
Relative Bias %	.0174	-.0162
Maximum Result	8.8700	8.6700
Minimum Result	5.3906	5.4285

SINGLE OPERATOR PRECISION:	Pair 1
Observations	7
Standard Deviation	.2270
Correction Factor	1.0424
Corrected Std Dev	.2366
Relative Std Dev (%)	3.3506

OVERALL PRECISION:		
Observations	7	7
Standard Deviation	1.3388	1.2216
Correction Factor	1.0424	1.0424
Corrected Std Dev	1.3955	1.2733
Relative Std Dev %	19.7569	18.0341

File Name: se-17
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Se Matrix: Filtered Effluent (using Frontier as tru
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	7.0619	7.0631	.0012	.02	.002	3.707	NO
2	7.0619	7.0608	-.0011	-.02	.002	3.707	NO

```
Se-18.dat
Se    18   1     9.9700   11.0000
Se    18   2     8.4000    9.1760
Se    18   3     9.4909    9.7532
Se    18   4     7.3640    7.6150
Se    18   5     7.9873    7.6959
Se    18   6    35.2663   26.0733
Se    18   7     6.5611    6.5048
Se    18   8     6.3760    6.4850

File Name:  se-18
Data Validation File (.DA~)

*****************************************************************************
***                    Parameter and Data Validation File                 ***
*****************************************************************************
   Analyte:  Se              Matrix:  UnFiltered Effluent (using Frontier as t
   Project:  EPA/EPRI     Matrix ID:  18
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike         5.9700      .0000
Spike
Increment      .0000      .0000
---------------------------------

                Final Concentration
          ------------------------
Lab ID        5.9700     5.9700
---------------------------------
    1         9.9700    11.0000
    2         8.4000     9.1760
    3         9.4909     9.7532
    4         7.3640     7.6150
    5         7.9873     7.6959
    6    o   35.2663o   26.0733
    7         6.5611     6.5048
    8         6.3760     6.4850
---------------------------------

Se    18   1     9.9700   11.0000
Se    18   2     8.4000    9.1760
Se    18   3     9.4909    9.7532
Se    18   4     7.3640    7.6150
Se    18   5     7.9873    7.6959
Se    18   6 o         o
Se    18   7     6.5611    6.5048
Se    18   8     6.3760    6.4850
```

File Name: se-18
Data Preparation File (.PRP)

Analyte: Se   Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI
   Date: 08/28/2000
  Units: ug/L

*** Results of 5x - 1/5x Mean Error Check ***
*** Questionable Data (Positive Values) ***

Total Number of Questionable Observations: 0

*** Results of Factor of 5 Error Check ***
*** Questionable Data (All Values) ***

Total Number of Questionable Observations: 0

File Name: se-18
Data Preparation File (.PRP)

Insufficient Data to Complete Lab Ranking

File Name: se-18
Data Preparation File (.PRP)

Analyte: Se   Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI
   Date: 08/28/2000
  Units: ug/L

*** Outlier Testing Results ***
*** Two-Sided 5% Significance Level ***

- Outlier(s) -

Lev	Iter	Lab	Rep	Result	Mean	Std Dev	t	Crit t	n
1	1	6	1	35.2663	11.4269	9.7165	2.453	2.126	8
2	1	6	1	26.0733	10.5379	6.4735	2.400	2.126	8

```
File Name:  se-18
Data Preparation File (.PRP)


Analyte:  Se     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     7     W     A              .9421      .803   ---
     2     7     W     A              .9214      .803   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  se-18
Data Preparation File (.PRP)


Analyte:  Se     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    18        8            8   100.0           7       87.5
  2    18        8            8   100.0           7       87.5
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          14       87.5
---------------------------------------------------------------
```

File Name: se-18
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Se Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

LEVEL:	1	2
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)
CONCENTRATION:	5.9700	5.9700
RECOVERY:		
Observations	7	7
Mean Result	8.0213	8.3186
Bias	2.0513	2.3486
Relative Bias %	34.3606	39.3393
Maximum Result	9.9700	11.0000
Minimum Result	6.3760	6.4850

SINGLE OPERATOR PRECISION:	Pair 1
Observations	7
Standard Deviation	.3261
Correction Factor	1.0424
Corrected Std Dev	.3400
Relative Std Dev (%)	4.1611

OVERALL PRECISION:		
Observations	7	7
Standard Deviation	1.3768	1.7087
Correction Factor	1.0424	1.0424
Corrected Std Dev	1.4351	1.7811
Relative Std Dev %	17.8908	21.4108

File Name: se-18
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Se Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	5.9700	8.0213	2.0513	34.36	3.942	3.707	YES
2	5.9700	8.3186	2.3486	39.34	3.637	3.707	NO

```
ag-17.dat
Ag    17   1     0.3550    0.3270
Ag    17   2     0.3787    0.3816
Ag    17   3     0.4471    0.4172
Ag    17   4     0.4720    0.5090
Ag    17   5     0.3230    0.3182
Ag    17   6     0.4657    0.4473
Ag    17   7     0.3379    0.3065
Ag    17   8     0.3964    0.2275

File Name:  ag-17
Data Validation File (.DA~)

*******************************************************************************
***                    Parameter and Data Validation File                   ***
*******************************************************************************
   Analyte:  Ag              Matrix:  Filtered Effluent (using Frontier as tru
   Project:  EPA/EPRI     Matrix ID:  17
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike          .4780      .0000
Spike
Increment      .0000      .0000
---------------------------------

                 Final Concentration
          ------------------------
Lab ID         .4780      .4780
---------------------------------
    1          .3550      .3270
    2          .3787      .3816
    3          .4471      .4172
    4          .4720      .5090
    5          .3230      .3182
    6          .4657      .4473
    7          .3379      .3065
    8          .3964      .2275
---------------------------------

ag-17.daf
Ag    17   1      .3550     .3270
Ag    17   2      .3787     .3816
Ag    17   3      .4471     .4172
Ag    17   4      .4720     .5090
Ag    17   5      .3230     .3182
Ag    17   6      .4657     .4473
Ag    17   7      .3379     .3065
Ag    17   8      .3964     .2275
```

```
File Name:  ag-17
Data Preparation File (.PRP)


Analyte:  Ag     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  ag-17
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  ag-17
Data Preparation File (.PRP)


Analyte:  Ag     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------


-------------------------------------------
 - No Outlier Rejections in Iteration 1 -
 - No Further Outlier Testing Necessary -
-------------------------------------------
```

```
File Name:  ag-17
Data Preparation File (.PRP)


Analyte:  Ag     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     8     W     A              .9114      .818   ---
     2     8     W     A              .9803      .818   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  ag-17
Data Preparation File (.PRP)


Analyte:  Ag     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-------------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-------------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    17        8            8   100.0           8      100.0
  2    17        8            8   100.0           8      100.0
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          16      100.0
---------------------------------------------------------------
```

File Name: ag-17
Statistical Analysis File (.STT)

--- *** Summary Performance Statistics *** ---

Analyte: Ag Matrix: Filtered Effluent (using Frontier as tru
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

LEVEL:	1	2
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)
CONCENTRATION:	.4780	.4780
RECOVERY:		
Observations	8	8
Mean Result	.3970	.3668
Bias	-.0810	-.1112
Relative Bias %	-16.9508	-23.2662
Maximum Result	.4720	.5090
Minimum Result	.3230	.2275

SINGLE OPERATOR PRECISION:	Pair 1
Observations	8
Standard Deviation	.0428
Correction Factor	1.0362
Corrected Std Dev	.0443
Relative Std Dev (%)	11.6125

OVERALL PRECISION:		
Observations	8	8
Standard Deviation	.0585	.0898
Correction Factor	1.0362	1.0362
Corrected Std Dev	.0606	.0931
Relative Std Dev %	15.2599	25.3697

File Name: ag-17
Statistical Analysis File (.STT)

--- *** Results of Bias Testing *** ---

Analyte: Ag Matrix: Filtered Effluent (using Frontier as tru
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.4780	.3970	-.0810	-16.95	3.920	3.499	YES
2	.4780	.3668	-.1112	-23.27	3.503	3.499	YES

```
Ag-18.dat
Ag    18   1     1.2000    1.1400
Ag    18   2     1.2830    1.3120
Ag    18   3     1.3798    1.3991
Ag    18   4     1.5160    1.4980
Ag    18   5     1.6205    1.4659
Ag    18   6     1.4265    1.4569
Ag    18   7     1.2007    1.3045
Ag    18   8     1.0881    1.2143

File Name:  ag-18
Data Validation File (.DA~)

*******************************************************************************
***                    Parameter and Data Validation File                   ***
*******************************************************************************
   Analyte:  Ag              Matrix:  UnFiltered Effluent (using Frontier as t
   Project:  EPA/EPRI     Matrix ID:  18
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike         2.8800      .0000
Spike
Increment      .0000      .0000
---------------------------------

                Final Concentration
          ------------------------
Lab ID        2.8800     2.8800
---------------------------------
    1         1.2000     1.1400
    2         1.2830     1.3120
    3         1.3798     1.3991
    4         1.5160     1.4980
    5         1.6205     1.4659
    6         1.4265     1.4569
    7         1.2007     1.3045
    8         1.0881     1.2143
---------------------------------

ag-18.daf
Ag    18   1     1.2000    1.1400
Ag    18   2     1.2830    1.3120
Ag    18   3     1.3798    1.3991
Ag    18   4     1.5160    1.4980
Ag    18   5     1.6205    1.4659
Ag    18   6     1.4265    1.4569
Ag    18   7     1.2007    1.3045
Ag    18   8     1.0881    1.2143
```

```
File Name:  ag-18
Data Preparation File (.PRP)


Analyte:  Ag     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  ag-18
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  ag-18
Data Preparation File (.PRP)


Analyte:  Ag     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------


-------------------------------------------
 - No Outlier Rejections in Iteration 1 -
 - No Further Outlier Testing Necessary -
-------------------------------------------
```

```
File Name:  ag-18
Data Preparation File (.PRP)


Analyte:  Ag     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     8     W     A              .9717      .818   ---
     2     8     W     A              .9323      .818   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  ag-18
Data Preparation File (.PRP)


Analyte:  Ag     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-------------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-------------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    18        8            8   100.0           8      100.0
  2    18        8            8   100.0           8      100.0
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          16      100.0
-----------------------------------------------------------------
```

File Name: ag-18
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Ag Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

LEVEL:	1	2
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)
CONCENTRATION:	2.8800	2.8800
RECOVERY:		
Observations	8	8
Mean Result	1.3393	1.3488
Bias	-1.5407	-1.5312
Relative Bias %	-53.4957	-53.1654
Maximum Result	1.6205	1.4980
Minimum Result	1.0881	1.1400

SINGLE OPERATOR PRECISION:	Pair 1
Observations	8
Standard Deviation	.0632
Correction Factor	1.0362
Corrected Std Dev	.0655
Relative Std Dev (%)	4.8734

OVERALL PRECISION:		
Observations	8	8
Standard Deviation	.1791	.1283
Correction Factor	1.0362	1.0362
Corrected Std Dev	.1855	.1329
Relative Std Dev %	13.8536	9.8551

File Name: ag-18
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Ag Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	2.8800	1.3393	-1.5407	-53.50	24.337	3.499	YES
2	2.8800	1.3488	-1.5312	-53.17	33.760	3.499	YES

```
tl-17.dat
Tl    17   1     0.1830    0.1720
Tl    17   2     0.2092    0.1841
Tl    17   3     0.2019    0.2119
Tl    17   4     0.1730    0.3200
Tl    17   5     0.1961    0.1948
Tl    17   6     0.2119    0.2188
Tl    17   7     0.2089    0.2067
Tl    17   8     0.1887    0.1904

File Name:  tl-17
Data Validation File (.DA~)

*******************************************************************************
***                    Parameter and Data Validation File                   ***
*******************************************************************************
   Analyte:  Tl              Matrix:  Filtered Effluent (using Frontier as tru
   Project:  EPA/EPRI     Matrix ID:  17
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike          .2000      .0000
Spike
Increment      .0000      .0000
---------------------------------

                 Final Concentration
          ------------------------
Lab ID         .2000      .2000
---------------------------------
    1          .1830      .1720
    2          .2092      .1841
    3          .2019      .2119
    4          .1730o     .3200
    5          .1961      .1948
    6          .2119      .2188
    7          .2089      .2067
    8          .1887      .1904
---------------------------------

tl-17.daf
Tl    17   1      .1830     .1720
Tl    17   2      .2092     .1841
Tl    17   3      .2019     .2119
Tl    17   4      .1730o
Tl    17   5      .1961     .1948
Tl    17   6      .2119     .2188
Tl    17   7      .2089     .2067
Tl    17   8      .1887     .1904
```

```
File Name:  tl-17
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  tl-17
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  tl-17
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
-------------------------------------------------------------------------------
  2     1     4   1       .3200      .2123      .0461   2.335   2.126    8
-------------------------------------------------------------------------------
```

```
File Name:  tl-17
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     8     W     A              .9246      .818   ---
     2     7     W     A              .9750      .803   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  tl-17
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    17        8            8   100.0           8      100.0
  2    17        8            8   100.0           7       87.5
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          15       93.8
---------------------------------------------------------------
```

File Name: tl-17
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Tl Matrix: Filtered Effluent (using Frontier as tru
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

LEVEL:	1	2
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)
CONCENTRATION:	.2000	.2000
RECOVERY:		
Observations	8	7
Mean Result	.1966	.1970
Bias	-.0034	-.0030
Relative Bias %	-1.7063	-1.5214
Maximum Result	.2119	.2188
Minimum Result	.1730	.1720

SINGLE OPERATOR PRECISION:	Pair 1
Observations	7
Standard Deviation	.0084
Correction Factor	1.0424
Corrected Std Dev	.0087
Relative Std Dev (%)	4.4455

OVERALL PRECISION:		
Observations	8	7
Standard Deviation	.0140	.0165
Correction Factor	1.0362	1.0424
Corrected Std Dev	.0145	.0172
Relative Std Dev %	7.3926	8.7259

File Name: tl-17
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Tl Matrix: Filtered Effluent (using Frontier as tru
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.2000	.1966	-.0034	-1.71	.688	3.499	NO
2	.2000	.1970	-.0030	-1.52	.488	3.707	NO

```
tl-18.dat
Tl    18   1     0.7830    0.7750
Tl    18   2     0.8521    0.9424
Tl    18   3     0.9219    0.9505
Tl    18   4     0.7610    0.7680
Tl    18   5     1.0243    0.9540
Tl    18   6     0.9264    0.9030
Tl    18   7     0.9770    0.9665
Tl    18   8     0.8704    0.8559

File Name:  tl-18
Data Validation File (.DA~)

*******************************************************************************
***                    Parameter and Data Validation File                   ***
*******************************************************************************
   Analyte:  Tl              Matrix:  UnFiltered Effluent (using Frontier as t
   Project:  EPA/EPRI     Matrix ID:  18
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike          .9000      .0000
Spike
Increment      .0000      .0000
---------------------------------

                 Final Concentration
          ------------------------
Lab ID         .9000      .9000
---------------------------------
    1          .7830      .7750
    2          .8521      .9424
    3          .9219      .9505
    4          .7610      .7680
    5         1.0243      .9540
    6          .9264      .9030
    7          .9770      .9665
    8          .8704      .8559
---------------------------------

Tl    18   1      .7830     .7750
Tl    18   2      .8521     .9424
Tl    18   3      .9219     .9505
Tl    18   4      .7610     .7680
Tl    18   5     1.0243     .9540
Tl    18   6      .9264     .9030
Tl    18   7      .9770     .9665
Tl    18   8      .8704     .8559
```

```
File Name:  tl-18
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  tl-18
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  tl-18
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------


-------------------------------------------
 - No Outlier Rejections in Iteration 1 -
 - No Further Outlier Testing Necessary -
-------------------------------------------
```

```
File Name:  tl-18
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     8     W     A              .9673      .818   ---
     2     8     W     A              .8344      .818   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  tl-18
Data Preparation File (.PRP)


Analyte:  Tl     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    18        8            8   100.0           8      100.0
  2    18        8            8   100.0           8      100.0
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         16           16   100.0          16      100.0
---------------------------------------------------------------
```

File Name: tl-18
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Tl Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

LEVEL:	1	2
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)
CONCENTRATION:	.9000	.9000
RECOVERY:		
Observations	8	8
Mean Result	.8895	.8894
Bias	-.0105	-.0106
Relative Bias %	-1.1653	-1.1764
Maximum Result	1.0243	.9665
Minimum Result	.7610	.7680

SINGLE OPERATOR PRECISION:	Pair 1
Observations	8
Standard Deviation	.0326
Correction Factor	1.0362
Corrected Std Dev	.0338
Relative Std Dev (%)	3.8000

OVERALL PRECISION:		
Observations	8	8
Standard Deviation	.0909	.0809
Correction Factor	1.0362	1.0362
Corrected Std Dev	.0942	.0838
Relative Std Dev %	10.5909	9.4233

File Name: tl-18
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Tl Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	.9000	.8895	-.0105	-1.17	.326	3.499	NO
2	.9000	.8894	-.0106	-1.18	.370	3.499	NO

```
zn-17.dat
Zn    17   1    45.8000   41.0000
Zn    17   2    44.3000   42.7000
Zn    17   3    42.7116   44.2444
Zn    17   4    44.9860   45.2430
Zn    17   5    60.3392
Zn    17   6    43.3113   47.8295
Zn    17   7    42.9109   42.8596
Zn    17   8    42.8472   45.3330

File Name:  zn-17
Data Validation File (.DA~)

*******************************************************************************
***                    Parameter and Data Validation File                   ***
*******************************************************************************
   Analyte:  Zn              Matrix:  Filtered Effluent (using Frontier as tru
   Project:  EPA/EPRI     Matrix ID:  17
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike        43.2500      .0000
Spike
Increment      .0000      .0000
---------------------------------

                Final Concentration
          ------------------------
Lab ID       43.2500    43.2500
---------------------------------
    1        45.8000    41.0000
    2        44.3000    42.7000
    3        42.7116    44.2444
    4        44.9860    45.2430
    5    o   60.3392e     .0000
    6        43.3113    47.8295
    7        42.9109    42.8596
    8        42.8472    45.3330
---------------------------------

zn-17.daf
Zn    17   1    45.8000   41.0000
Zn    17   2    44.3000   42.7000
Zn    17   3    42.7116   44.2444
Zn    17   4    44.9860   45.2430
Zn    17   5 o         *
Zn    17   6    43.3113   47.8295
Zn    17   7    42.9109   42.8596
Zn    17   8    42.8472   45.3330
```

```
File Name:  zn-17
Data Preparation File (.PRP)


Analyte:  Zn     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  zn-17
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  zn-17
Data Preparation File (.PRP)


Analyte:  Zn     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
-------------------------------------------------------------------------------
  1     1     5   1     60.3392    45.9008     5.9404   2.431   2.126    8
-------------------------------------------------------------------------------
```

```
File Name:  zn-17
Data Preparation File (.PRP)


Analyte:  Zn     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     7     W     A              .8737      .803   ---
     2     7     W     A              .9735      .803   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  zn-17
Data Preparation File (.PRP)


Analyte:  Zn     Matrix:  Filtered Effluent (using Frontier as tru
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-------------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-------------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    17        8            8   100.0           7       87.5
  2    17        7            7   100.0           7      100.0
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         15           15   100.0          14       93.3
-----------------------------------------------------------------
```

File Name: zn-17
Statistical Analysis File (.STT)

--- *** Summary Performance Statistics *** ---

Analyte: Zn Matrix: Filtered Effluent (using Frontier as tru
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

LEVEL:	1	2
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)
CONCENTRATION:	43.2500	43.2500
RECOVERY:		
Observations	7	7
Mean Result	43.8381	44.1728
Bias	.5881	.9228
Relative Bias %	1.3599	2.1336
Maximum Result	45.8000	47.8295
Minimum Result	42.7116	41.0000

SINGLE OPERATOR PRECISION:	Pair 1
Observations	7
Standard Deviation	2.1179
Correction Factor	1.0424
Corrected Std Dev	2.2076
Relative Std Dev (%)	5.0167

OVERALL PRECISION:		
Observations	7	7
Standard Deviation	1.2089	2.2297
Correction Factor	1.0424	1.0424
Corrected Std Dev	1.2601	2.3241
Relative Std Dev %	2.8744	5.2614

File Name: zn-17
Statistical Analysis File (.STT)

--- *** Results of Bias Testing *** ---

Analyte: Zn Matrix: Filtered Effluent (using Frontier as tru
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	43.2500	43.8381	.5881	1.36	1.287	3.707	NO
2	43.2500	44.1728	.9228	2.13	1.095	3.707	NO

```
zn-18.dat
Zn    18   1    48.5000   48.2000
Zn    18   2    49.4400   50.5500
Zn    18   3    49.6040   49.7807
Zn    18   4    50.6250   52.3870
Zn    18   5
Zn    18   6    62.0914   48.8716
Zn    18   7    45.5374   46.2510
Zn    18   8    49.5120   48.7990

File Name:  zn-18
Data Validation File (.DA~)

*****************************************************************************
***                    Parameter and Data Validation File                 ***
*****************************************************************************
   Analyte:  Zn              Matrix:  UnFiltered Effluent (using Frontier as t
   Project:  EPA/EPRI     Matrix ID:  18
      Date:  08/28/2000      Method:  1638
     Pairs:  1
     Units:  ug/L

---------------------------------
Level          1          2
---------------------------------
Spike        48.4700      .0000
Spike
Increment      .0000      .0000
---------------------------------

                Final Concentration
          ------------------------
Lab ID       48.4700    48.4700
---------------------------------
    1        48.5000    48.2000
    2        49.4400    50.5500
    3        49.6040    49.7807
    4        50.6250    52.3870
    5    e     .0000e     .0000
    6    o   62.0914    48.8716
    7        45.5374    46.2510
    8        49.5120    48.7990
---------------------------------

zn-18.daf
Zn    18   1    48.5000   48.2000
Zn    18   2    49.4400   50.5500
Zn    18   3    49.6040   49.7807
Zn    18   4    50.6250   52.3870
Zn    18   5 *         *
Zn    18   6 o          48.8716
Zn    18   7    45.5374   46.2510
Zn    18   8    49.5120   48.7990
```

```
File Name:  zn-18
Data Preparation File (.PRP)


Analyte:  Zn     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
---------------------------------------------------------------
 ***        Results of 5x - 1/5x Mean Error Check        ***
 ***         Questionable Data (Positive Values)         ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


---------------------------------------------------------------
 ***        Results of Factor of 5 Error Check           ***
 ***          Questionable Data (All Values)             ***
---------------------------------------------------------------
Total Number of Questionable Observations:     0


File Name:  zn-18
Data Preparation File (.PRP)


-------------------------------------------
 Insufficient Data to Complete Lab Ranking
-------------------------------------------



File Name:  zn-18
Data Preparation File (.PRP)


Analyte:  Zn     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
----------------------------------------
 ***    Outlier Testing Results      ***
 *** Two-Sided 5% Significance Level ***
----------------------------------------

--------------------
   - Outlier(s) -
--------------------
 Lev  Iter  Lab  Rep     Result       Mean    Std Dev    t     Crit t   n
-------------------------------------------------------------------------------
  1     1     6   1     62.0914    50.7585     5.2510   2.158   2.020    7
-------------------------------------------------------------------------------
```

```
File Name:  zn-18
Data Preparation File (.PRP)


Analyte:  Zn     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------
 *** Results of Normality Testing ***
-----------------------------------------

-------------------------
   - Normality Tests -
-------------------------
               Test  Normality      Test          Critical
   Level  n    Type  Accept/Reject  Statistic     Value(s)
-----------------------------------------------------------------
     1     6     W     A              .8253      .788   ---
     2     7     W     A              .9818      .803   ---
-------------------------------
 - 0 Normality Rejection(s) -
-------------------------------


File Name:  zn-18
Data Preparation File (.PRP)


Analyte:  Zn     Matrix:  UnFiltered Effluent (using Frontier as t
Project:  EPA/EPRI
   Date:  08/28/2000
  Units:  ug/L
-----------------------------------------------
 ***         Data Removal Tracking         ***
 *** Simple Count of Remaining Data Points ***
 ***          After Removal Tests          ***
-----------------------------------------------

             Points   After Lab Ranking  After Outlier Testing
               As     -----------------  ---------------------
 Lev  Mtrx  Received     Points    %         Points       %

 ---  ----  --------  ---------  ------  ----------     ------
  1    18        7            7   100.0           6       85.7
  2    18        7            7   100.0           7      100.0
 ---  ----  --------  ---------  ------  ----------     ------
Totals:         14           14   100.0          13       92.9
---------------------------------------------------------------
```

File Name: zn-18
Statistical Analysis File (.STT)

*** Summary Performance Statistics ***

Analyte: Zn Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

LEVEL:	1	2
YOUDEN PAIRS:	Pair 1 (Low)	Pair 1 (High)
CONCENTRATION:	48.4700	48.4700
RECOVERY:		
Observations	6	7
Mean Result	48.8697	49.2628
Bias	.3997	.7928
Relative Bias %	.8247	1.6356
Maximum Result	50.6250	52.3870
Minimum Result	45.5374	46.2510

SINGLE OPERATOR PRECISION:	Pair 1
Observations	6
Standard Deviation	.6493
Correction Factor	1.0509
Corrected Std Dev	.6824
Relative Std Dev (%)	1.3903

OVERALL PRECISION:		
Observations	6	7
Standard Deviation	1.7662	1.9260
Correction Factor	1.0509	1.0424
Corrected Std Dev	1.8562	2.0075
Relative Std Dev %	3.7982	4.0752

File Name: zn-18
Statistical Analysis File (.STT)

*** Results of Bias Testing ***

Analyte: Zn Matrix: UnFiltered Effluent (using Frontier as t
Project: EPA/EPRI Method: 1638
Date: 08/28/2000
Units: ug/L

Conc. Level	Conc	Mean Result	Bias	Rel. Bias (%)	Obs t Value	Crit t Value	Statistically Significant (1% Two-Tail)
1	48.4700	48.8697	.3997	.82	.554	4.032	NO
2	48.4700	49.2628	.7928	1.64	1.089	3.707	NO

Target:

Plant MultimediaToxics Characterization (PISCES)

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