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Pressurized Water Reactor Steam Generator Lay-up: Corrosion Evaluation

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Pressurized Water Reactor Steam Generator Lay-up: Corrosion Evaluation

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Interim Report, December 2005

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REPORT SUMMARY

This interim report summarizes work completed to date for a project to develop improved lay-up guidance for PWR Steam Generators (SG). Phase 1 of this project included a detailed literature review and a gap analysis of additional work needed to quantify the corrosion behavior of SG materials under wet lay-up conditions. As a result of the gap analysis, EPRI designed a corrosion test program (Phase 2) to measure general corrosion rates of steam generator materials under lay-up conditions. This report summarizes Phases 1 and 2 of the project. Phase 3 of this project (to be completed) will develop additional localized corrosion measurements, including galvanic corrosion for important SG material galvanic couples.

Background

Within the past 10 years, outage lengths have decreased dramatically. Many nuclear units routinely perform refueling outages in • 25 days. Steam generator lay-up times have decreased accordingly. Some units have been able to take advantage of the EPRI guidance allowing lower concentrations of hydrazine and amine when the steam generators are in lay-up for less than 7 days. However, EPRI bases this guidance on industry practice and limited laboratory studies. Furthermore, there is no leeway in dealing with outages that are slightly longer than 7 days. There is little laboratory data to verify the effectiveness of lay-up solutions on critical steam generator metallurgies as a function of time. However, this guidance would be very beneficial to utilities both in refueling outages, as well as in forced outages where the outage length is not well known at the time when lay-up decisions need to be made. In order to maintain the integrity of the steam generator tubes during lay-up conditions, it would be prudent to develop the appropriate guidance for lay-up solutions based on time, steam generator critical metallurgy, deposit conditions, and existing steam generator corrosion conditions.

Objectives

- To develop corrosion test data for steam generator materials under lay-up conditions.
- To provide the industry with improved guidance for laying up steam generators.

Approach

The project team divided this work into four phases. Phase 1 included a detailed literature review and a gap analysis of additional work needed to quantify the corrosion behavior of SG materials under wet lay-up conditions. They designed Phase 2 to measure general corrosion rates of SG materials under lay-up conditions. This report summarizes Phases 1 and 2 of the project to date. Phase 3 of this project, to be completed, will analyze additional localized corrosion

measurements, including galvanic corrosion for important SG material galvanic couples. Phase 4 will assess the results in full and provide improved guidance for SG wet lay-up.

Results

The experimental work performed in this study demonstrated that the corrosion resistant alloys I600, I690 and 405 SS experience low and acceptable general corrosion rates under all of the deaerated conditions tested. Although there were a few I600 measurements with higher than acceptable corrosion rates (probably as a result of experimental error), the majority of the data was at the limit of detection in this program. However, the project team determined that evaluation of galvanic and local corrosion is necessary before drawing any overall general conclusions with respect to these alloys. Under aerated conditions without pH control, both I600 and I690 experienced relatively high general corrosion rates for some specimens, but below LLD for other specimens.

The remainder of the work focused on the carbon steel and weld materials, including an exploration of trends with respect to oxygen scavenger concentration and pH. In addition, the project team made comparisons to the literature data and discussed recommendations for future testing.

EPRI Perspective

Wet lay-up of steam generators during outages with chemically treated water (amine and oxygen scavenger) is desirable to minimize corrosion and oxidation during the lay-up period itself, and also minimize corrosion during subsequent startups and power operation. EPRI based the current lay-up guidance on industry experience with long outages and limited laboratory data. However, improved guidance is necessary to address current concerns with respect to shorter outage durations and discharge limits. EPRI will base the new guidance on a detailed review of existing field and laboratory data supplemented with an experimental program to fill critical voids in the corrosion database. This interim report addresses the literature review and gap analysis performed in the first phases of this project. Additional work is required before the development of improved guidance.

Keywords

Steam generator
Corrosion
Wet-layup

ABSTRACT

This interim report summarizes work completed to date on a project to evaluate the current PWR steam generator lay-up guidance as described in sections 2.4.12 and 5.5.1 of the PWR Secondary Water chemistry Guidelines – Revision 6 (EPRI TR-1008224, December 2004). Phase 1 of this project included an extensive literature review of the corrosion test data and basis for the current lay-up chemistry guidelines. A gap analysis was completed to determine what additional data would be needed to update the current guideline recommendations.

Phase 2 is a corrosion test measurement program to evaluate the general corrosion rates of several SG materials under wet lay-up conditions. In the test program, weight loss measurements were made in varying concentrations of oxygen scavengers with solutions containing <20 ppb dissolved oxygen and the pH controlled to >9.0. Additional tests were performed in initially air saturated and nitrogen purged solutions with and without oxygen scavengers. The oxygen scavengers tested in this work include; hydrazine, carbohydrazide and di-ethyl hydroxylamine (DEHA).

Phase 3, yet to be completed, will evaluate and measure localized and galvanic corrosion under steam generator lay-up chemistries.

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INTRODUCTION

This interim report summarizes work completed to date for a project to develop improved lay-up guidance for PWR Steam Generators (SG). Phase 1 of this project included a detailed literature review and a gap analysis of additional work needed to quantify the corrosion behavior of SG materials under wet lay-up conditions. As a result of the gap analysis, a corrosion test program (Phase 2) was designed to measure general corrosion rates of steam generator materials under lay-up conditions. This report summarizes Phases 1 and 2 of the project. Phase 3 of this project (to be completed) will include additional localized corrosion measurements including galvanic corrosion for important SG material galvanic couples.

The current lay-up guidance is based on industry experience with long outages and limited cost and or regulatory pressure on environmental discharge. Improved guidance is needed to address current concerns. The new guidance will be based on a detailed review of existing field and laboratory data supplemented with an experimental program to fill critical voids in the corrosion database. This interim report addresses the literature review and gap analysis that was performed in the first phase of the project. Additionally, this report summarizes general corrosion test data that was performed as a result of the gap analysis.

Wet lay-up of steam generators during outages with chemically treated water is desirable to minimize corrosion and oxidation during the lay-up period itself and also corrosion during subsequent startups and power operation. Protection is provided by an amine for pH control and hydrazine (or other qualified oxygen scavenger) to maintain a protective oxide film and a reducing environment. Fossil fuel boiler experience and laboratory studies show that proper lay-up chemistry can provide corrosion protection for six months or longer. A positive nitrogen overpressure should be maintained during filling, draining, and cold shutdown to minimize oxygen ingress.

Hydrazine in concentrations of >75 ppm is an effective oxygen scavenger at ambient temperatures. Nuclear units typically add hydrazine to the steam generators following a unit shutdown. Once the unit is cooled off, nitrogen sparging is used to mix the hydrazine and remove oxygen. Maintaining a reducing environment in the steam generators minimizes the corrosion potential on the tube surface. Removing oxygen from the lay-up solution also allows metal deposits, such as copper, to remain in the reduced state. Oxidation of deposited metals such as copper can contribute to an increase in the electrochemical potential in a localized area of the tube during start up. It is very important that during lay-up conditions, the steam generator tubes and deposits remain in a reducing environment.

In more recent years, alternates to hydrazine have been introduced both in lay-up and in operating applications. Hydrazine is carcinogenic and its production of ammonia has contributed to hazardous working conditions in steam generators. Also, the discharge limitations on hydrazine have caused further complications in its application. Alternate chemical treatments such as carbohydrazide and diethyl hydroxylamine (DEHA) have been used with varying success. Application guidance primarily has been supplied by chemical vendors with an emphasis on lower lay-up concentrations and higher reaction rate kinetics in scavenging oxygen. However, breakdown products such as carbonates, have caused many to return to dealing with the high concentrations and discharge limitation issues of hydrazine.

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BACKGROUND

Within the past 10 years, outage lengths have decreased dramatically. Many nuclear units routinely perform refueling outages in ≤ 25 days. Steam generator lay-up times have decreased accordingly. Some units have been able to take advantage of the EPRI guidance allowing lower concentrations of hydrazine and amine when the steam generators are in lay-up for less than 7 days. However, this guidance is based on industry practice and not on specific laboratory studies. Furthermore, there is no leeway in dealing with outages that are slightly longer than 7 days. Very few laboratory data have been taken to verify the effectiveness of lay-up solutions on critical steam generator metallurgies as a function of time. However, this guidance would be very beneficial to utilities both in refueling outages, as well as in forced outages where the outage length is not well known at the time when lay-up decisions need to be made. In order to maintain the integrity of the steam generator tubes during lay-up conditions, it would be prudent to develop the appropriate guidance for lay-up solutions based on time, steam generator critical metallurgy, deposit conditions, and existing steam generator corrosion conditions.

The objectives of this project are as follows:

- To develop corrosion test data for steam generators materials under lay-up conditions.
- Provide the industry with improved guidance for laying up steam generators.

Current Guidelines

The EPRI PWR Secondary Chemistry Guidelines, Revision 6 currently have the following specifications for lay-up chemistry:

CONTROL PARAMETERS FOR STEAM GENERATOR SAMPLE

Monitor values until stable at a frequency of three times per week, then weekly once values are stable.

Parameter	Initiate Action	Value Necessary Prior to Heatup (>200°F)
pH @ 25°C	<9.5	—
Hydrazine, ppm[a]	<75	—
Sodium, ppb	>1000	≤100
Chloride, ppb	>1000	≤100
Sulfate, ppb	>1000	≤100

DIAGNOSTIC PARAMETERS FOR STEAM GENERATOR SAMPLE

Parameter	Consideration
Boron	Applies only to plants using secondary side boric acid treatment.
Sludge analysis	When sludge is available as a result of sludge lancing or other maintenance.

CONTROL PARAMETERS FOR STEAM GENERATOR FILL SOURCE

Parameter	Frequency	Initiate Action
Dissolved O_2, ppb	(b)	>100[c]

a. Alternatives to hydrazine may be used if qualified by the utility. Appropriate limits for any hydrazine alternative should be substituted.

b. Required prior to and/or during fill for plants with oxygen control of their fill water source.

c. For plants without control of oxygen in their fill source, appropriate compensatory actions shall be taken to minimize steam generator exposure to oxygen, e.g., nitrogen sparging or addition of a reducing agent to the fill source or directly to the steam generators.

Figure 2-1
RSG Full Wet Layup (RCS ≤200°F) Steam Generator Sample

The Guidelines provides the following instructions; “During outages, when the time period between cold shutdown and draining or partial draining of the SGs for maintenance or startup is expected to be less than seven days or the time period between completion of the maintenance and startup is expected to be less than seven days, it is not necessary to place the steam generators in full wet layup. However, to the extent practicable considering personnel safety and environmental issues, the critical elements of full wet layup, i.e., elevated pH, hydrazine (or alternative oxygen scavenger) at greater than 75 ppm, nitrogen overpressure, low dissolved oxygen, and low impurities should be maintained. If one or more of these conditions cannot be maintained, the remaining conditions should still be applied.

When filling the steam generators from a drained or partially drained condition, the hydrazine concentration in the steam generator should be >5 ppm and a nitrogen overpressure should be established. If condenser vacuum is broken, steps should be taken to ensure that oxygenated feedwater is not introduced into the steam generators via feedwater. Water used to fill the steam generators should contain less than 100 ppb dissolved oxygen".

CONTROL PARAMETERS

Monitor all values every other day until stable, then weekly
or after significant water additions [a]

Parameter	Initiate Action	Value Necessary Prior to Heatup Above >200°F
pH @ 25°C	≤9.5	–
Hydrazine, ppm[b]	<75 or >500	–
Sodium, ppb	>1000	≤100
Chloride, ppb	>1000	≤100
Sulfate, ppb	>1000	≤100
Oxygen, ppb[c]	–	≤100

DIAGNOSTIC PARAMETERS

Parameter	Justification
Nitrogen Overpressure,[d] psig	Minimization of oxygen ingress to the steam generators during wet layup.
Hideout Return Database[e] (Na, Cl, SO_4, SiO_2, K, Mg, Ca, Al)	Assessment of OTSG crevice and superheat region solution chemistry during operation; impurity source term assessment.

a. Chemical addition and OTSG recirculation should be initiated as soon as possible after entering Mode 5.

b. Alternatives to hydrazine may be used if qualified by the utility. Appropriate limits for any hydrazine alternative should be substituted.

c. Routine monitoring not required prior to initial heatup if hydrazine concentration is within normal range.

d. A nitrogen overpressure should be maintained on the steam generators when personnel safety will not be compromised.

e. Hideout return assessments generally should be based on data collected during fill/drain operations immediately subsequent to shutdown.

Figure 2-2
OTSG Full Wet Layup (RCS ≤200°F) (Technical Specification Modes 5 and 6)
Steam Generator Sample

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LITERATURE REVIEW

The purpose of the literature review was to gather all relevant corrosion data that has served as the basis for the current lay-up chemistry guidelines. A listing and brief summary of the pertinent data for the literature review is given at the end of this Chapter. The main requirement of wet lay-up is to reduce or eliminate oxygen from the gas space of the steam generators. An oxygen scavenger is added to the water to remove any oxygen that enters the SG during lay-up. Finally, an elevated pH is maintained to reduce the solubility principally of iron in SG steels.

Rabeau et. al. **[19]** showed that a hydrazine concentration of 50 ppm to 100 ppm was necessary to minimize the corrosion rate for carbon steel in an aerated, pH 10.0 environment. This is in agreement with the current EPRI guidelines. However, deoxygenating the lay-up solution with nitrogen removed the dependency of the corrosion rate on hydrazine. Thus, the driving force for corrosion is available oxygen in the lay-up solution. The reaction of oxygen with metal surfaces is much preferred over the reaction of oxygen with hydrazine in solution, particularly at lay-up temperatures.

The following is an overview of the data from the literature reviewed that contain corrosion rate information for carbon steel. The data were taken from referenced sources and are sorted by the reported corrosion rate, CR.

Table 3-1
Literature Review of Corrosion Data

pH(25)	O2, ppm	N2H4, ppm	CH, ppm	HQ, ppm	DEHA	MEK ppm	Iso ascorbic Acid, ppm	Carbon Steel CR, mpy
9.6	1	200						< LLD
9.6	1		100					< LLD
9.9		145						<LLD
9.3	< LLD	0.003						0.0048
8.7	0.003							0.089
9.3		0.03						0.137
9.9		145						0.167
10.6			360					0.57
10.6			36					0.66
	0.025 – 0.040	45 - 100						1.755
	0.075 – 0.090					290 - 315		1.755
	0.020 – 0.030						400 - 800	2.457
10.1		200						2.5
	0.020 – 0.030		40 - 100					2.691
	0.004 – 0.016				40 - 200			2.964
	0.003 – 0.010			170 - 460				20.67
9.6	1	50						30

MEK- methyl ethyl ketone, CH-Carbohydrazide, HQ-hydroquinone, DEHA-Diethyl hydroxylamine

The majority of the literature focused testing on carbon steel, however the SG contains a number of potentially susceptible materials. A combined listing of materials from two different steam generators (replacement B&W and Westinghouse D5) is given below: **[24]**

Table 3-2
Steam Generator Metallurgies

Component	Material	Component	Material
6"/8" Handhole Diaphragms	SB-168	SD Nozzles	E7018-A1
AFW Nozzle	SA-508	Sec. Shell Drain	SA-508
AFW Permanent Cap	SA-234	Secondary Manway	SA-508
AFW Safe End	SB-166	Secondary MW Diaphragm	SB-168
AFW Thermal Sleeve	SB-167	Secondary Shell	SA-533
Anti Vibration Bars	Inconel 600	Shell Cone	SA-508
Aux Nozzle Cap Plug	SA-350	Shroud	SA-516
Back-up Rings	SA-285	Stayrod	SA-696 Gr C
Baffle Plate Retaining Blocks	SA-285	Stayrod Bolting	SA-193
Channel Head	SA-216	Steam Drum Head	SA-508
Diaphragm Screws	SA-193	Steam Drum Shell	SA-533
Drum Internals	Carbon Steel	Steam Drum Support ring	SA-516
FW Header Pipe	SA-335	Steam Outlet Nozzle	SA-508
FW Inlet Nozzle	SA-508	Steam Outlet Safe End	SA-350
FW Safe End	SA-508	Stub Barrel	SA-533
Handhole Covers	SA-516	Transition Cone	SA-533
Inspection Port cover	SA-105	TSP AntiRotation blocks	ASTM A-576
Inspection Port Diaphragms	SB-168	TSP back-up bars	ASTM A-576
J tube Pipe	SB-167	TSP Shims	SB-168
Lattice Support Rings	SA-516	TSP Spacer Pipes	SA-106
Lower Shell Barrels	SA-533	TSP Wedges	ASTM A-576
Main FW Nozzle	SA-508	Tube Support Plates	A-240
Main FW Nozzle Limiter/Insert	SB-166	Tube Supports	SA-240-410S
Main FW Thermal Sleeve	SA-106 Gr B	Tubes	SB-163
Main FW Thermal Slv Transition	SA-516	Tubesheet	SA-508
Manway/HH Gaskets	Flexitallic	Upper Head	SA-533
Misc Internal Non-Pressure Parts	ASTM A-285 Gr C	Upper Shell Barrels	SA-533
Misc Internal Non-Pressure Parts	Inconel	Weld Filler Material - Typical SMAW	SFA 5.1 Class E7018
Misc Internal Non-Pressure Parts	SA-106 Gr B	Wet Lay-up Tap	SA-508
Misc Internal Non-Pressure Parts	SA-240 Type 405	Wide Range Level Tap	SA-508
Misc Internal Non-Pressure Parts	SA-36	WR Upper/Lower Level Taps	SA-350
Misc Internal Non-Pressure Parts	SA-508	Wrapper Antirotation Block	SA-516
Misc Internal Non-Pressure Parts	SA-516 Gr 70	Wrapper AntiRotation Key	SA-516
Misc Internal Non-Pressure Parts	SA-696 Gr C	Wrapper Barrel	ASTM A285
NR Level Tap	SA-508	Wrapper Jacking Blocks	SA-516
NR Upper/Lower Level Taps	SA-350	Wrapper Jacking Studs	SA-36
Pre-heater Partition Plate	ASTM A-285 Gr C	Wrapper Key Restraint	SA-516
Recirc Nozzle	E7018-A1	Wrapper Position Blocks	SA-516
Sample Tap	SA-508		

The material analysis performed by B&W for the qualification of a polymer dispersant in the ANO steam generators helped to identify specific materials whose susceptibility bounded the corrosion susceptibility in the steam generators. These included steam generator tubing material, support plate material, high susceptible carbon steels, and high susceptible welding material. Similar considerations are applicable to this program's test matrix.

Analysis of Literature Data

Bounding Materials

The literature survey supplied a short list of steam generator materials that were used as bounding conditions in the approval for a steam generator polymer dispersant **[15]**.

Materials
LTMA Inconel 600
405 SS
SA-335 Gr P11
ER70S-6 GMAW
E7018-A1SMAW
SA-569
SA-36

Acceptable Corrosion Rates

An acceptable corrosion rate for carbon steel in a high pH aerated environment can be determined from the literature survey data **[1, 6, 7, 17, and 19]**.

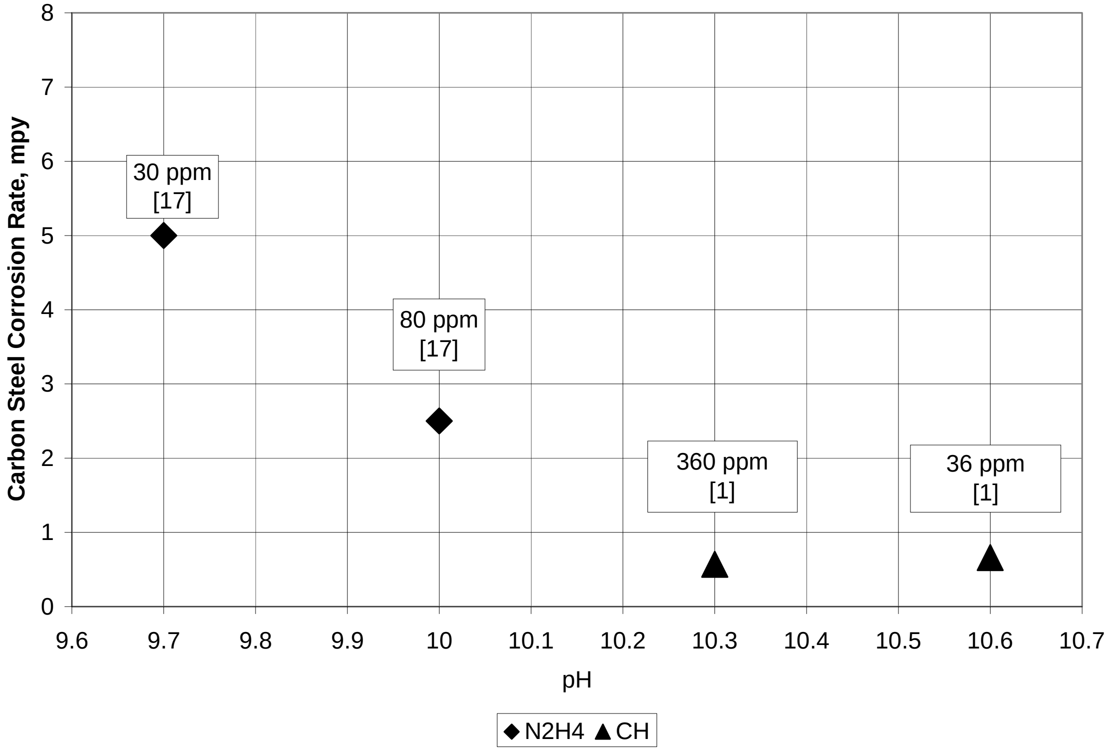


Figure 3-1
Carbon Steel Corrosion Rates with Oxygen Scavengers under Aerated Conditions

This is compared with the recommendations of Marks **[1]** where to prevent corrosion, the following correlation was presented:

$[N_2H_4] > -210pH + 2200$ ppm

and

[CH] > 125 ppm.

The hydrazine data in Figure 3-1 shows that a lay-up solution with hydrazine at 30 ppm and pH of 9.7 yields a corrosion rate for carbon steel of 5 mpy in an aerated solution. At an increased pH of 9.9 and hydrazine of 80 ppm, the carbon steel corrosion rate is 2.5 mpy.

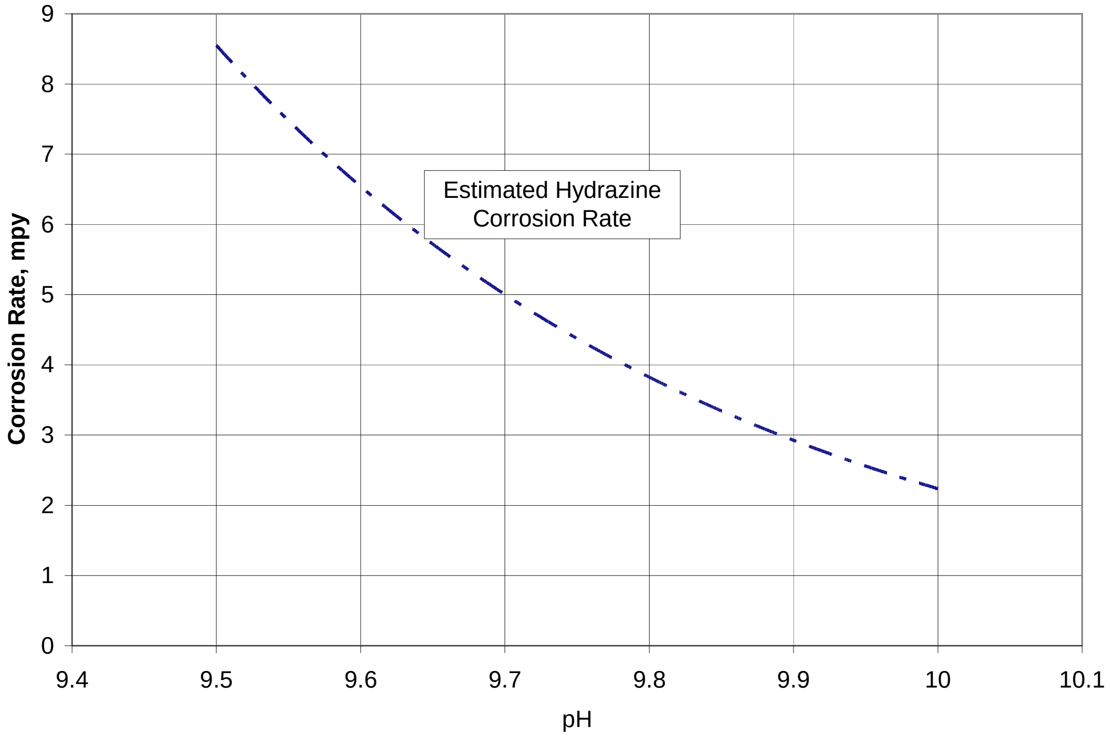


Figure 3-2
Estimated Corrosion Rate of Carbon Steel in Aerated Hydrazine Solution

Assuming that the corrosion rate is a function of pH within the hydrazine concentration range of 30 ppm to 145 ppm, the corrosion rate at the EPRI lay-up limit of pH 9.5 is estimated to be 9 mpy. It is inferred that the corrosion rate of carbon steel in the EPRI guideline values of pH 9.5 and hydrazine at 75 ppm would be less than or equal to 9 mpy in an aerated solution.

The testing performed in low oxygen environments are shown in Figure 3-3.

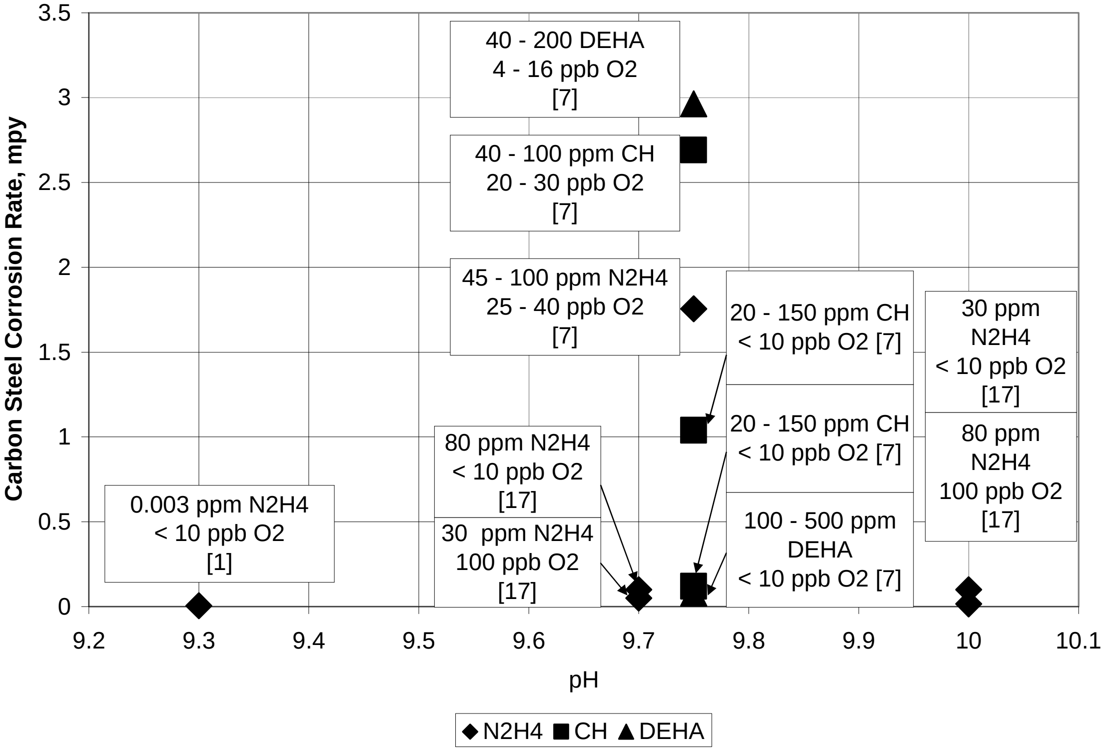


Figure 3-3
Corrosion Rate of Carbon Steel as a function of pH and Oxygen Scavenger

Corrosion rates were dramatically lower in this environment for hydrazine, and slightly elevated for the one set of data for DEHA and two sets of data for carbohydrazide. It is important to note that the concentration of DEHA and carbohydrazide were typically lower as compared with the tests run in fully aerated environments.

Further data were reviewed from two steam generator chemical cleaning qualifications **[22, 23]**. These provided the allowable amount of corrosion over a 40 year operating life.

These results are summarized from Table 5-8 of the report **[22]** in the first 3 columns of Table 3-3. A fourth column was added to give a calculated corrosion rate (Utility limit/40 years). It is assumed, for simplicity, that the average corrosion rate is the same in all modes of operations.

Table 3-3
Millstone 2 SG Tubesheet Chemical Cleaning Corrosion Coupon Results

Material	Category	Utility Limits (40 Year Allowance), mils	*Calculated Ave. Corrosion Rate, mpy* *(Utility Limits/40 yrs.)*
SB 163	Tubing	7.0	*0.175*
SA 570 Gr 40	Eggcrate	11.0	*0.275*
SA 516 Gr 70	Secondary Shell	91.2	*2.28*
	Welds	270	*6.75*
SA 508 Cl2	Tubesheet	550	*13.75*

The resulting corrosion rates for carbon steel in steam generators are summarized:

- In aerated lay-up solutions using the EPRI guideline pH and hydrazine amounts, 9 mpy
- In low oxygen environments using elevated concentrations of oxygen scavengers, < 3 mpy, and
- Performing a simple calculation using chemical cleaning criteria, 2.3 mpy.

As a point of conservatism, the allowable carbon steel corrosion rate of < 2.3 mpy will be used for this evaluation. Following this simple calculation of average corrosion rate, the corrosion rates for the other metallurgies listed as boundary conditions from the polymer dispersant application **[15]** will also be used as corrosion criteria for this evaluation. Table 3-4 lists the acceptable corrosion rates for all metallurgies to be tested based on the chemical cleaning allowable corrosion divided by a 40 year operating life.

Table 3-4
Allowable Corrosion Rates for Susceptible Steam Generator Metallurgies

Materials	Allowable Corrosion Rate, mpy
LTMA Inconel 600	0.175
405 SS	0.175
SA-335 Gr P11	***2.28***
ER70S-6 GMAW	6.75
E7018-A1SMAW	6.75
SA-569	***2.28***
SA-36	***2.28***

Proposed Lay-up Corrosion Testing

Data for aerated corrosion testing of carbon steel in high pH water has supported the current guidelines values of 75 ppm hydrazine and pH ≥ 9.5 **[2, 17, 19]**. Data for acceptable alternates to hydrazine (carbohydrazide, DEHA) along with unacceptable alternatives (hydroquinone, methyl ethyl ketoxime, and isoascorbic acid) were found in the literature search **[6, 7]**. Additional testing is needed to determine the concentrations of hydrazine, carbohydrazide, and DEHA that yield corrosion rates that are compatible with the allowable corrosion rates summarized in Table 3-4.

To support this, corrosion data are needed over a wider range of SG pH's and oxygen scavenger concentrations. A standard test program using ASTM G-31-72, Standard Practice for Laboratory Immersion Corrosion Testing of Metals was used for measuring weight loss of coupons in this project. In the test program, oxygen concentrations were maintained at < 20 ppb with continuous nitrogen sparging and initial oxygen scavenger concentrations in the test solutions were varied. Additional control test runs were performed without oxygen scavengers and using different nitrogen sparging procedures to simulate different plant operating procedures. The corrosion rates were calculated based on measured weight loss using the procedures provided in ASTM G-31-72.

There are concerns about the effects of steam generator sludge and deposits, particularly in regards to copper. Copper can catalyze the breakdown of oxygen scavengers and in some cases, affect the system pH. The instability of carbohydrazide in the presence of simulated steam generator sludge led Ontario Hydro to choose DEHA as the preferred oxygen scavenger over hydrazine **[7]**. However, the effects of the sludge pile will not be included in the scope of this work, but may be included in testing proposed for Phase 3 of this program.

The test program defined here is not designed to identify either local corrosion (e.g. pitting, crevice corrosion) or galvanic corrosion. Although visual observations were made on all corrosion test coupons for localized corrosion, a more detailed program will be carried out in Phase 3.

Initial Test Matrix

The following Test Matrix was proposed for this project. As will be discussed in Section 5, the experimental matrix was varied based on results obtained during the test program.

Fixed Parameters:

- pH >9.0 using ammonia
- Initial Oxygen < 20 ppb by nitrogen sparging
- Temperature = 25 C
- Time ≥ 500 hours

The metallurgies tested are listed in Table 3-4 above. Control Testing was also performed with no oxygen scavenger and the above conditions.

Varied Parameters:

Oxygen Scavenger	Concentrations, ppm			
Hydrazine	50	25	10	1
Carbohydrazide	100	50	25	1
DEHA	150	100	50	1

Measurements made in the test program were as follows:

- pH (3x/week)
- Dissolved oxygen (3x/week)
- Conductivity (3x/week)
- Calculated Corrosion Rate (end of experiment)
- Pit Depths (if any, at the end of experiment)

The purpose of this testing is to provide support for conditions where the steam generator oxygen concentration remains low (i.e. system integrity is not broken) or for lay-up where deoxygenation of the lay-up solution is possible. Although not all situations would be covered under this testing, issues 1, 2, and to some extent 3 listed below would be addressed.

Issues that cause utility personnel to not establish full wet lay-up typically include:

1. Short lay-up time (< 7 days)
2. Discharge limitations for chemicals such as hydrazine
3. Steam generator support system isolation – cannot add or properly mix chemicals
4. Steam generator drained or partially drained for maintenance activities

References

1. **Marks, C.R., "Evaluation of Hydrazine and Carbohydrazide Levels during Lay-up as a Function of pH," EPRI Secondary Water Chemistry Guidelines Review, February, 2000.**

   Data were reviewed based on pH and oxygen scavenger concentrations on carbon steel. Corrosion prevention was described by the author for hydrazine (ppm) as:

   $$[N_2H_4] > -210*pH + 2200$$

   and for carbohydrazide (ppm) as:

   $$[Cz] > 125$$

Oxygen concentrations and the amount of copper and iron deposits were not considered in this data set. This resource cited 23 pertinent references.
Additional data that was referenced in this report and is not cited elsewhere in this literature search are as follows:

Test Solution	Corrosion Rate, mpy
200 ppm N2H4, 10 ppm NH3	2.5
360 ppm CH, 200 ppm NH3	0.57
36 ppm CH, 200 ppm NH3	0.66
pH 8.7, 3 ppb O2	0.089
pH 9.3, 3 ppb N2H4, < LLD O2	0.0048
pH 9.3, 30 ppb N2H4	0.137
pH 9.9, N2H4 145 ppm	< LLD
pH 9.9, N2H4 145 ppm, lube oil	0.167

2. **Armentano, J.A., V.P. Murphy, "Stand-By Protection of High Pressure Boilers," 25th Annual International Water Conference, Pittsburgh, September, 1964.**

Test Solution	Corrosion Information
100 ppm N2H4, pH 9.2	Some Discoloration after 24 hours
200 ppm N2H4, pH 9.5	Some Discoloration after 24 hours
300 ppm N2H4, pH 9.9	No Discoloration after 24 hours
200 ppm N2H4, pH 10.0	No Discoloration after 5 days
200 ppm, pH 10.2 to 11.0	No corrosion detected after 6 months

3. **Bohnsack, G. "Chemistry of Corrosion Inhibition and Surface Passivation of Mild Steel by Hydrazine in Power Plant Circuits," Corrosion 89, April, 1989, New Orleans, LA.**
   Paper looks at the Shikorr reaction and the hydrazine influences on iron oxide formation. Oxide layers that prevent corrosion are formed at $T \leq 105$ C.

4. **Romaine, S., Cotton, I. J., "Effectiveness of a New Volatile Oxygen Scavenger," American Power Conference, Illinois Institute of Technology, Chicago, IL April, 1986.**
   A case study is provided to show the effectiveness of hydroquinone over hydrazine at the Virginia Power, Chesterfield Station. Laboratory corrosion data were reported. Metal coupons were sealed in bottles containing deionized, deaerated, pH 9.5 water for 2 years. The corrosion rate of the coupon in hydrazine was 0.675 mil/yr. and the corrosion rate for the coupon in hydroquinone was 0.085 mil/yr. The concentrations of hydrazine and hydroquinone in the lab data were not given in the paper.

5. **Ellis, D. M., Cuisia, D. G., Thompson, H. W., "The Oxidation and Degradation Products of Volatile Oxygen Scavengers and their Relevance in Plant Applications," Corrosion 87, Paper Number 432, Moscone Center San Francisco, CA March, 1987.**
   The paper reported principal breakdown products of DEHA to be acetaldehyde, dialkylamines, acetate, and acetaldoxime. No corrosion data was presented in the paper.

6. **Miller, A.D., "Survey of Alternate Reducing Agents for Secondary Chemistry Control," EPRI TR-107949, March 1997.**
   The report summarizes industry experience with hydrazine and alternate reducing agents. Sandra Pagan's work at Ontario Hydro listed the following corrosion rates:

Test Solution	Corrosion Rates
45 - 100 ppm N2H4, 25 - 40 ppb O2	45 um/yr
40 - 200 ppm DEHA, 4 - 16 ppb O2	76 um/yr
170 - 460 ppm HQ, 3 - 10 ppb O2	530 um/yr
290 - 315 ppm MEK, 75 - 90 ppb O2	45 um/yr
40 - 100 ppm CH, 20 - 30 ppb O2	69 um/yr
400 - 800 ppm IsoAsc, 20 - 30 ppb O2	63 um/yr

7. **Pagan, S., "Evaluation and Application of an Alternative Reducing Agent for Steam Generator Lay-Up at Pickering A NGS," EPRI Amine Workshop, New Orleans, LA, September, 1993.**
   Two alternates to hydrazine were tested based on initial screening for stability and corrosion rates; DEHA and Carbohydrazide. These were tested for 3 weeks in aqueous, static and stirred, 2% O2 and N2 cover, with and without sludge, 10, 25, and 50 C, and pH ranges of 5 - 5.3, 9.5 – 10, and 10.8 – 11. The materials tested were carbon steel, Monel 400, and 90/10 Cu Ni.

   Testing results:

Metallurgy	Temp	pH	Cover Gas	CR (DEHA 100 – 500 ppm), um/y	CR (Carbo 20 – 150 ppm), um/y
Monel 400				None	None
90/10 Cu/Ni				None	None
Carbon Steel	25	9.5 – 10.0	2% O2/N2	1 – 3	1 – 5
Carbon Steel	50	9.5 – 10.0	2% O2/N2	164, occasional pits	26, minor pits
Carbon Steel	25	5.0 – 5.3	2% O2/N2	180, occasional pits	220

   DEHA was chosen as the preferred alternative to hydrazine because carbohydrazide displayed stability problems, and there was a concern about degradation to ammonia in the steam generator. A DEHA specification was developed for lay-up at 100 ppm – 300 ppm, typical value of 250 ppm, and a pH of 9.5 – 11.0, typical value of 10.2 and dissolved oxygen typical value of 30 ppb

8. **Rohani-Rad, A., Mofidi, J., Modaress-Tehrani, Z., "Anticorrosive behaviour of octadecylamine for protection of boiler surfaces," Technical note, Corrosion Engineering, Science and Technology 2003, Vol 38. No. 1.**
   Weight loss and electrochemical tests were used to determine the anticorrosive behavior of octadecylamine (ODA) on carbon steel, brass, and austenitic stainless steel. The corrosive solution was soft water with 1 ppm Fe, 240 ppm Cl, 445 ppm Na, 4 ppm K, 0.5 ppm PO4, 5.75 ppm O2, 730 ppm SO4, 1270 ppm TOC, 280 ppm HCO3, 2 ppm Ca, and 2 ppm Mg. The pH was 8.27 and ODA concentration of about 10 ppm. Coupon testing

was performed on steel 20, brass and 18/10 steel over 1100 hours with average rates determined ever 100 hours.

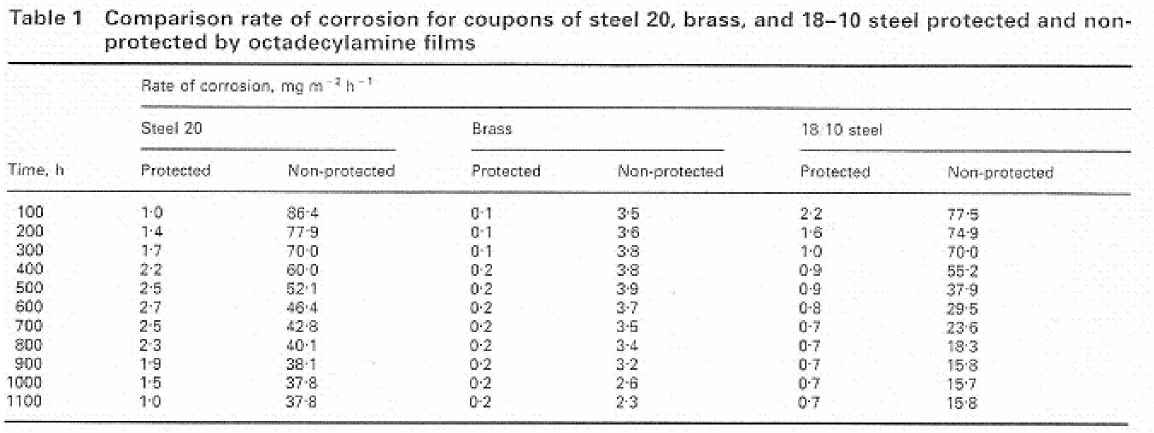


Table 1 Comparison rate of corrosion for coupons of steel 20, brass, and 18–10 steel protected and non-protected by octadecylamine films

	Rate of corrosion, mg m^{-2} h^{-1}					
	Steel 20		Brass		18/10 steel	
Time, h	Protected	Non-protected	Protected	Non-protected	Protected	Non-protected
100	1·0	86·4	0·1	3·5	2·2	77·5
200	1·4	77·9	0·1	3·6	1·6	74·9
300	1·7	70·0	0·1	3·8	1·0	70·0
400	2·2	60·0	0·2	3·8	0·9	55·2
500	2·5	52·1	0·2	3·9	0·9	37·9
600	2·7	46·4	0·2	3·7	0·8	29·5
700	2·5	42·8	0·2	3·5	0·7	23·6
800	2·3	40·1	0·2	3·4	0·7	18·3
900	1·9	38·1	0·2	3·2	0·7	15·8
1000	1·5	37·8	0·2	2·6	0·7	15·7
1100	1·0	37·8	0·2	2·3	0·7	15·8

Potentiostatic measurements were made for the steel 20. Corrosion rates were determined at constant potentials for exposure periods of 5 hours.

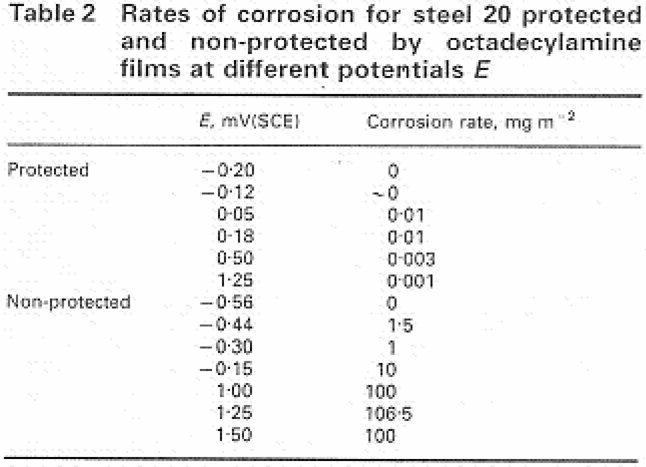


Table 2 Rates of corrosion for steel 20 protected and non-protected by octadecylamine films at different potentials E

	E, mV(SCE)	Corrosion rate, mg m^{-2}
Protected	−0·20	0
	−0·12	~0
	0·05	0·01
	0·18	0·01
	0·50	0·003
	1·25	0·001
Non-protected	−0·56	0
	−0·44	1·5
	−0·30	1
	−0·15	10
	1·00	100
	1·25	106·5
	1·50	100

9. **Marks, C., Varrin, R., D., "Oxidation and Reduction of PWR Steam Generator Secondary Side Deposits: Experimental Data and Predictive Models," EPRI 1003591, July 2002.**
   Report models the decomposition of hydrazine and carbohydrazide in steam generator scale, crevice, and sludge pile environments. Participation in the crevices is predicted to be minimal.
10. **Syrett, B. C., "Low Temperature Corrosion Problems in Fossil Power Plants – State of Knowledge Report," EPRI 1004924, December 2003.**
    This report is an extensive overview of corrosion problems in the fossil industry. Report contains no boiler lay-up data or oxygen scavenger corrosion rate information.

11. **Dooley, R.B., "Interim Cycle Chemistry Guidelines for Combined Cycle Heat Recovery Steam Generators (HRSGs)," EPRI TR-110051, Final Report, November 1998.**

Successful extended boiler wet lay-ups contain a pH of 10.0 and up to 200 ppm hydrazine. Nitrogen blanketing is recommended for short-term outages with no additional pH or hydrazine concentrations given.

12. **Allen, G. C., "Sourcebook for Plant Lay-up and Equipment Preservation (Revision 1)," EPRI NP-5106 Revision 1, May 1992.**
    Report overviews the PWR and BWR lay-up experiences and reflects the guidance given in the PWR and BWR Guidelines.

13. **Millett, P., "PWR Secondary Water Chemistry Guidelines – Revision 5," EPRI TR-102134-R5, Final Report May 2000.**
    Report specifies lay-up for RSGs with $\geq$ 9.5 pH and $\geq$ 75 ppm hydrazine for outages of 7 days or greater. For lay-up of less than 7 days hydrazine should be > 5 ppm and oxygen should be < 100 ppb. For OTSGs, the lay-up chemistry specifies pH > 9.8 and hydrazine to be between 75 ppm and 500 ppm.

14. **Marks, C., Varrin, R., D., "Oxidation and Reduction of Copper in Steam Generator Deposits: Experimental Data and Predictive Models," EPRI 1001204, September 2001.**
    The report applies the work of Bowers et. al. to the reduction of copper deposit in thin films using hydrazine. The reaction follows an Arrhenius model with a pre-exponential factor of 32 ± 4 s^{-1} and an activation energy of 32 ± 2 kJ/mol.

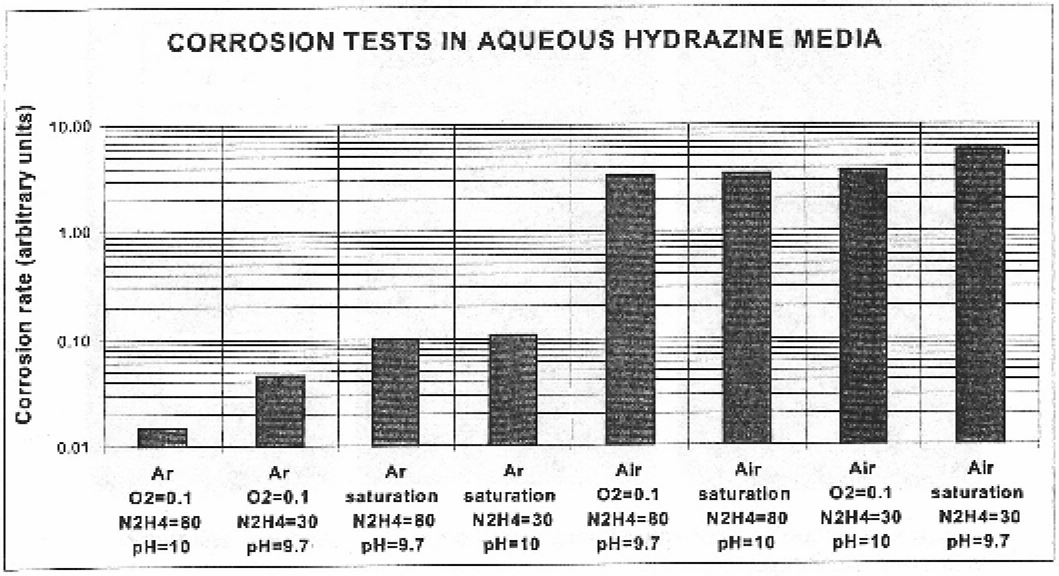
15. **Robbins, P., Frattini, P., "Dispersants for Tube Fouling Control Volume 1: Qualification for a Short-Term Trial at ANO-2," EPRI 1001422, Final Report March 2001.**
    The report identifies a short list of steam generator metallurgies that bound the testing concerns for application of a new polymer in nuclear steam generators. The final recommendations for material and testing were:

Material	Test(s)
TT Inconel 690	CERT, LPR, PDS
405SS	CERT (optional), LPR, PDS
SA-335 Gr P11	LPR, PDS
ER70S-6 GMAW	LPR, PDS
E7018-A1SMAW	LPR, PDS
SA-569	LPR, PDS, Weight Loss
SA-36	Weight Loss

16. **Whyte, D. D., "Laboratory Program to Examine Effects of Lay-up Conditions on Pitting of Inconel 600," EPRI NP-3012, Final Report, April 1983.**
    Pitting of Inconel 600 tubing for steam generators was studied at 40 C. Pitting solutions included copper chloride or sea water, plus sludge containing copper, copper oxide, and magnetite. In less than 3 weeks, pitting of A600 occurred in (700 ppm Cl) copper chloride solution. Similar results occurred in sea water (6000 ppm Cl). Pitting was reduced by decreasing oxygen, decreasing copper, or increasing the pH.

17. **Long, A., Organista, M., Brun, C., Combrade, P, "Optimization of Wet Lay-Up Conditions for Steam Generators Hydrazine Chemical Treatment," Presented at the**

International Conference: Water Chemistry of Nuclear Reactor Systems, Avignon, France, April 2002.
The paper outlines a test program to reduce the amount of hydrazine used during lay-up. The study showed the importance of nitrogen blanketing or performing dry lay-up. Testing was performed on two types of samples; free unalloyed A42, coupled with A600, fully immersed in water, and unalloyed A42, positioned at the water line. Corrosion was monitored by sample weight loss. The air interaction with the lay-up solution greatly increased corrosion rates as shown below:



18. **Foucault, M.. Long, A., Combrade, P., Rodet, I., Bouchacourt, M., "Secondary Chemistry of Steam Generator. Influence of Hydrazine on Corrosion Potential. Optimization of Shutdown Conditions and Return to Nominal Conditions," Presented at the International Conference: Water Chemistry of Nuclear Reactor Systems, Avignon, France, April 2002.**
    Autoclave testing was performed to determine the effects of increased oxygen in systems with high, and low hydrazine concentrations. Corrosion potential measurements were taken at 280 C. The results showed that increased oxygen with low hydrazine resulted in increased corrosion potential in carbon steel at 280 C. No data at lay-up temperatures were taken.
19. **Rabeau, A.M., et. al., "Steam Generators Lay-Up Optimization and Derived Wastes Reduction," Presented at the International Conference: Water Chemistry of Nuclear Reactor Systems, Avignon, France, April 2002**
    Corrosion testing of carbon steel (NF A 35-501) was performed in aerated and deaerated solutions with ammonia (pH(25) = 10.0), and hydrazine (0 ppm – 400 ppm). Under aerated conditions, the corrosion testing supported the current PWR guidelines for pH ≥ 9.5 and hydrazine ≥ 75 ppm.

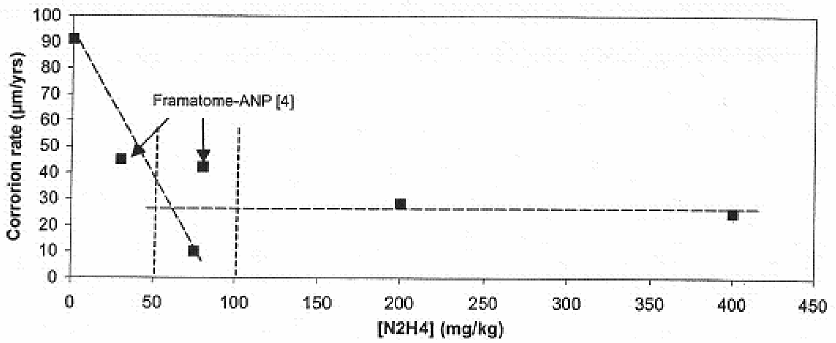


Figure 4 : Evolution of the corrosion rate with the hydrazine content (25 °C ; pH =10 ; aerated conditions)

With nitrogen blanketing, the corrosion rates were lower than 3 um/yr, which is 10 to 30 times less than the aerated conditions in the absence of hydrazine.

20. **Casar, D.J., "San Onofre Generating Station Control of Oxidants in the Secondary System during Shutdown, Lay-up and Startup," Revision 0, December 2001.** Document describes utilities compliance to the EPRI guidelines, but does not contain any new corrosion data for lay-up.

21. **McIlree, A., R., "Proceedings: 1999 EPRI Workshop on Startup Oxidant Control," TR-112815, Final Report June 1999.** Document describes utilities implementation of lay-up and start-up oxidant control, but does not contain any new corrosion data for lay-up.

22. **Pearl, W. L., "Chemical Cleaning of Millstone Unit 2," EPRI NP-4597, Final Report, May, 1986.** This report was used to determine allowable corrosion rates for various steam generator components. These results are summarized from Table 5-8 of the report listed below in the first 3 columns. A forth column was added to give a calculated corrosion rate:

Material	Category	Utility Limits (40 Year Allowance), mils	Calculated Ave. Corrosion Rate, mpy
SB 163	Tubing	7.0	0.175
SA 570 Gr 40	Eggcrate	11.0	0.275
SA 516 Gr 70	Secondary Shell	91.2	2.28
	Welds	270	6.75
SA 508 Cl2	Tubesheet	550	13.75

23. **Prestegiacomo, J. B., et al, "Qualification of PWR Steam Generator Chemical Cleaning For Indian Point-2," Volume 1 Summary Report, EPRI NP-6356-SL, Final Report, May 1989.** This report was used to determine corrosion limits for various steam generator components. A selection of the results is included to show allowable corrosion amounts and a calculated rate based on a 40 year life is added to the table. The corrosion results listed are from Table 4-8 in the report:

Material	Category	Limit that includes design corrosion allowance, mils	Calculated Ave. Corrosion Rate, mpy

SA508	Tubesheet	125	3.125
SA533	Stub Barrel	125	3.125
SA285	Wrapper	37	0.925
SA106	Blowdown Pipe	70	1.75
SA105	Boss	48 – 64	1.2 – 1.6
A234	Blowdown Elbow	70	1.75
SAE 1020	End Plate	70	1.75
A36	Channel	37	0.925

24. Personal communication with Jay Smith, Byron Station.

4

EXPERIMENTAL PROCEDURES

The effect of varying the concentrations of oxygen scavengers on different metals was investigated using the ASTM G-31-72, Standard Practice for Laboratory Immersion Corrosion Testing of Metals Procedure. The metals examined were Inconel 600, Inconel 690, 405SS, 1010, ER70S-GMAW, E7018-A1SMAW, SA-569, and SA-36.

Experimental Apparatus

Corrosion coupons from Metal Samples, Inc. (www.metalsamples.com) were used for the experiments. All of the coupons with the exception of 405SS had the following dimensions: 2" x 3/4" by 1/8". The 405SS coupons were 1/16" thick. Figure 4-1 shows a typical corrosion coupon. The hole in the coupon was used to attach the coupons to the coupon test rack (see Figure 4-2). All coupons were exposed in the as- received mill finished condition.

Figure 4-1
Metal Samples Corrosion Coupon

The calculated wetted area of the coupons and the allowable corrosion rate in mils per year based on the analysis in Section 3 is shown in Table 4-1.

Table 4-1
Materials Tested

Material	Density (g/cm^2)	A (cm^2)	Allowable Corrosion Rate mpy
Inconel 600	8.47	22.387	0.175
Inconel 690	8.19	22.387	0.175
405 SS	7.80	27.484	0.175
1010	7.87	27.484	2.28
ER70S-6 GMAW	7.87	22.387	6.75
E7018-A1SMAW	7.87	22.387	6.75
SA-569	7.87	27.484	2.28
SA-36	7.60	27.484	2.28

Each experiment consisted of exposing a number of test coupons to a test solution. The coupons were attached to a coupon test rack that was supplied by Metal Samples, Inc. A test rack similar to that shown in Figure 4-2 was used. In this test program, only flat (e.g. no U-bend) coupons were used. The flat bar rack, hex bolts and hex nuts were made of HASTELLOY C-276. The insulating washers and shoulder washers were Teflon.

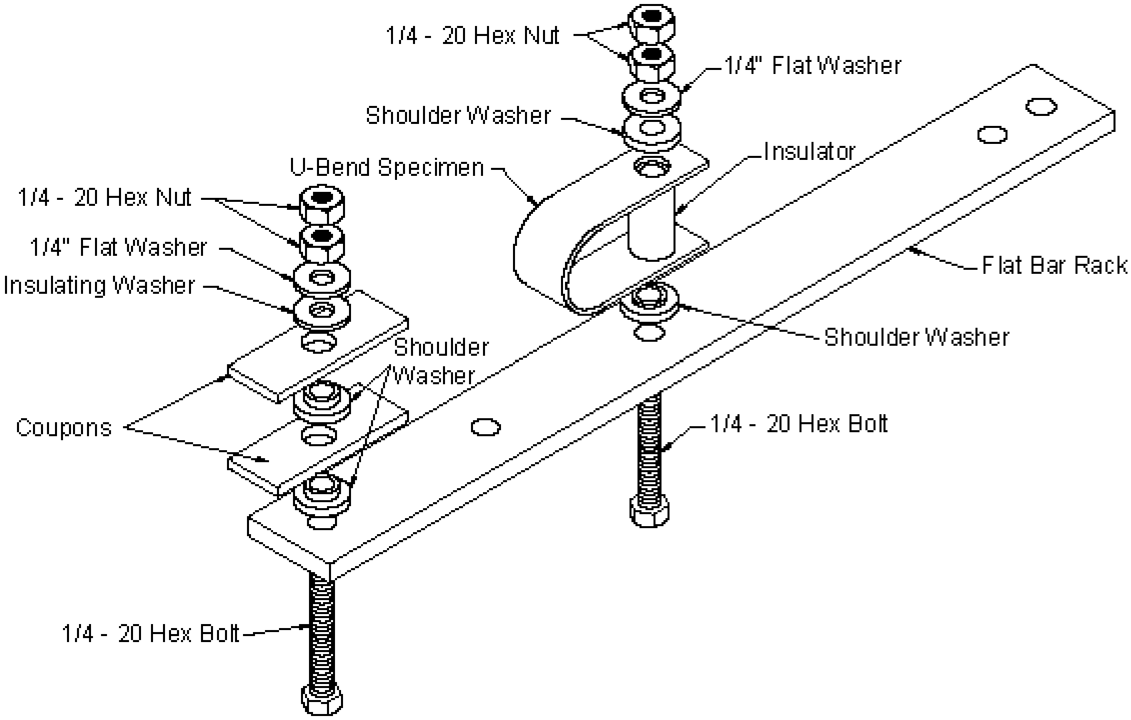


Figure 4-2
Metal Samples Coupon Rack

The coupon racks were constructed as shown in Figure 4-2. In most experiments, twelve or more coupons were attached to the coupon rack. Typically, three coupons were attached to each side of the flat bar rack at two elevations. In most experiments, the coupons were exposed to the test solution, however in some cases the upper coupons were exposed to the gas or vapor space above the test solution.

Test solutions were prepared using reagent grade chemicals and each coupon rack was inserted into a 1 liter high density polyethylene bottle. The bottle top was modified to pass through the flat bar rack. Tygon tubing was used to deliver nitrogen to the test solution and three probes were inserted through the vessel top to monitor the chemical environment in the test solution. Figure 4-3 is a photograph of the experimental apparatus completely assembled and inserted in the isothermal bath. Typically up to three tests were run simultaneously in an isothermal bath controlled to 25C. The apparatus is shown with a single probe in Figure 4-3. During most experiments, three probes; dissolved oxygen, conductivity and pH were used. Any unused openings in the bottle top were plugged with a glass or rubber stopper during the experiments. Otherwise there was no attempt to seal the vessels from oxygen ingress. The continuous nitrogen purge was used to maintain the solution oxygen free.

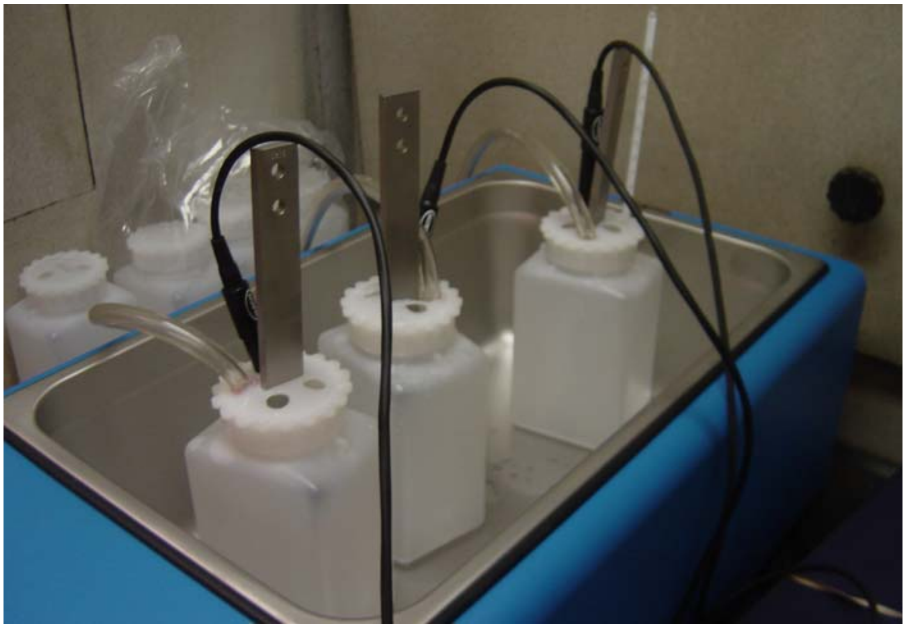


Figure 4-3
Assembled Test Apparatus

For some of the air saturated control experiments and for those without continuous nitrogen sparging, a standard 2-3 L pyrex glass beaker was used in place of the HDPE bottles.

Solutions were prepared with reagent grade chemicals. The initial oxygen scavenger concentrations were prepared and added directly to the HDPE (or glass) test vessel. The air space above the test solution was initially evacuated using high purity nitrogen. The nitrogen was introduced through tygon tubing which was inserted into each test vessel below the solution level. In most experiments, continuous nitrogen sparging was maintained by slowly bubbling nitrogen into the vessel. As the tops of the vessels were not air tight, atmospheric or slightly above atmospheric pressure was maintained with the nitrogen purge throughout the experiments.

Experimental Procedures

The oxygen scavengers studied were hydrazine, carbohydrazide, and DEHA. The metal coupons were submerged in solutions of various concentrations of oxygen scavengers. While in solution, pH, dissolved oxygen, conductivity, and temperature were monitored. The amount of corrosion is determined by comparing the weights of metal coupons before and after exposure to the solutions. For each experimental run, the coupons were left in solution for ≥ 20 days.

Pre Cleaning

ASTM G-31-72 was used for preparing the coupons for the test. The procedures for the pre cleaning are as follows:

- Brush coupon and rinse in deionized water
- Soak coupon in methanol for 5 minutes
- Air dry coupon for 10 minutes
- weigh coupon and record

Experimental Matrix

The coupons were submerged in different concentration of oxygen scavengers for ≥ 20 days and corrosion rates of the metals were calculated based on the weight difference of the test materials. Each experiment with an oxygen scavenger consisted of preparing approximately 750 mls of the desired concentration of scavenger and adding it to the test vessel. Nitrogen sparging was then initiated and the oxygen concentration in the vessel was continuously monitored until the concentration was reduced to < 20 ppb. This normally took less than 5 minutes, and the dissolved oxygen was less than detectable shortly thereafter. The initial pH and conductivity was then measured and recorded. If necessary, a pH adjustment was made to each test solution by adding reagent grade ammonia. During the subsequent 20+ days, readings of the dissolved oxygen, conductivity, pH and test temperature were recorded at a frequency of at least 3x/week. Adjustments to maintain the pH above 9.0 were made as needed using reagent grade ammonia.

The experimental matrix consisted of using different concentrations of oxygen scavengers as shown in Table 4-2. Nitrogen was sparged into these solutions to assure that the oxygen concentrations were less than 20 ppb. The temperature of the solution was kept constant at or near 25 C using an isothermal water bath and the pH of the solutions was maintained >9.0 using ammonia. The set up of the apparatus was described in experimental apparatus section.

Other sets of experiments were performed containing no oxygen scavenger. The experiments were performed to determine the effect of pH and/or the use of nitrogen sparging on the test materials. The experimental matrix for these experiments is shown in Table 4-3.

Table 4-2
Experimental Matrix of Metals Exposed to Oxygen Scavengers

Metal Type	N2H4 (ppm)	DEHA (ppm)	CHZ (ppm)
Inconel 600	1, 25, 50, 1000,10000, 25000	1, 150	1, 100
Inconel 690	1, 25, 50, 1000,10000,25000	1, 150	1, 100
405 SS	1, 25, 50, 1000,10000,25000	1, 150	1, 100
1010 CS	1, 25, 50, 1000,10000,25000	1, 150	1, 100
ER70S-GMAW	1, 25, 50, 1000,10000,25000	1, 150	1, 100
E7018-A1SMAW	1, 25, 50, 1000,10000,25000	1, 150	1, 100
SA-569	1, 25, 50, 1000,10000,25000	1, 150	1, 100
SA-36	1, 25, 50, 1000,10000,25000	1, 150	1, 100

Table 4-3
Experimental Matrix of Metals Exposed to No Oxygen Scavengers (Control)

Metal Type	O_2 Scavengers (ppm)	Control Conditions
Inconel 600	0	a,b,d
Inconel 690	0	a,b,d
405 SS	0	a,d
1010 CS	0	a,b,c,d
ER70S-GMAW	0	c,d
E7018-A1SMAW	0	a,c
SA-569	0	a,b,c,d
SA-36	0	a,b,c,d

a- indicates that the test coupons were placed above DI water. The dissolved oxygen is at air-saturated level and the pH is about 5.5
b- indicates that no nitrogen has been purged into the DI water in which the test coupons have been submerged, so the liquid is at air-saturated level. The pH is maintained >9.0.
c- indicate that nitrogen has been purged into the DI water only at the beginning of the experiment. Typcially dissolved oxygen reached air-saturated level within 10 minutes of securing the nitrogen purge. The pH is maintained >9.0.
d- indicates that nitrogen has been purged continuously into the DI water through out the experiment

Post Cleaning

After exposing the coupons for at least 20 days, each experiment was terminated by removing the test racks from the test vessels. Prior to re-weighing the coupons, ASTM G1-03, Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens was used for cleaning and preparing the coupons for the weight measurements.

For chemical cleaning the 1010 CS, SA 36, SA 569, ER 70S-GMAW, and E7018-A1SMAW coupons, the procedure was as follows:

1. Prepare a 1000 ml hydrochloric acid (HCl), 20 g antimony trioxide (Sb_2O_3) and 50g of stannous chloride ($SnCl_2$) solution

2. Maintain the temperature between 20 to 25°C

3. Stir the solution

4. Soak the test coupons in the solution for 4 minutes

5. Take the coupons out from solution and brush to remove loose corrosion products

6. Rinse with DI water

7. Air dry
8. Weigh and record

For chemical cleaning the Inconel 600 and Inconel 690 coupons, the following procedure was used:

1. Prepare a solution with 150 ml hydrochloric acid (HCl) and 1000 ml DI water
2. Maintain the temperature between 20 to 25°C
3. Soak the test metals for 2 minutes
4. Take the coupons out from solution and brush to remove loose corrosion products
5. Rinse with DI water
6. Air dry
7. Weigh and record

For chemical cleaning the 405 SS coupons, the following procedure was used:

1. Prepare a solution with 100 ml nitric acid (HNO3) and 1000 ml DI water
2. Maintain the temperature at 60°C
3. Soak the test metals for 20 minutes
4. Take the coupons out from solution and brush to remove loose corrosion products
5. Rinse with DI water
6. Air dry
7. Weigh and record

5

EXPERIMENTAL RESULTS

The results for the first seventeen experiments are reported here. A brief summary of the conditions for each experiment is provided in Table 5-1. As described in Section 4, Experimental Procedures, continuous nitrogen sparging was used in most of the experimental runs. The sparging rate was not directly measured, but was sufficient to maintain the dissolved oxygen levels to <20 ppb when employed. In some experiments, no sparging was used. The solutions for these experiments were air saturated. In experiment 16, the test vessel was initially purged with nitrogen for less than 5 minutes at the beginning of the experiment to reduce the dissolved oxygen to <20 ppb. During the course of experiment 16, three subsequent 5 minute nitrogen sparges were used to bring the dissolved oxygen levels to <20 ppb. As shown in the Appendix, the dissolved oxygen level in experiment 16 typically re-saturated within 1 day. It should also be noted that all coupons were exposed directly in the test solution except where noted in Table 5-1. In some cases the coupons were above the solution level in the air space of the test vessel. The coupons were exposed to the vapor in equilibrium with the test solution. Many of the tests employed pH control by NH3 addition. In some cases, the initial scavenger concentration was sufficient to maintain the pH above the target of 9.0 for the entire test period. The average pH for each test is presented in Table 5-1, however, it should be noted that in many cases the pH varied by 0.5 units or more during the test period. Appendix A includes trend plots of the pH for each test.

Table 5-1
Summary of Experimental Runs

Experiment No.	Materials (No. of coupons)	Oxygen Scavenger	Conc.ppm	pH Range in Experiment	Ave. pH	Nitrogen Sparging	Notes
1	1010 (2)* I600 (2) I690 (1) SA36 (2)* 405 (2) SA569 (2)	N2H4	50,000	10.3-10.8	10.5	Continuous	No NH3 added
2	1010 (2) I600 (1) I690 (2) SA36 (2) 405 (3) SA569 (2)	DEHA	150	9.0-10.4	9.4	Continuous	No NH3 added
3	1010 (1) I600 (1) 405 (3) SA569 (1)	CHZ	100	8.1-9.3	9.0	Continuous	NH3 used to adjust pH
4	1010 (2) I600 (2) I690 (1) SA36 (1) 405 (2) SA569 (2) E70S (2) E7018 (1)	N2H4	25,000	10.1-11.0	10.6	Continuous	No NH3 added
5	1010 (2) I600 (2) I690 (2) SA36 (1) 405 (2) SA569 (1) E70S (1) E7018 (2)	N2H4	10,000	9.4-10.7	10.0	Continuous	No NH3 added
6	1010 (3) I600 (2) I690 (2) SA36 (2) 405 (2) SA569 (2)	N2H4	1,000	8.8-10.2	9.2	Continuous	No NH3 added
7	1010 (3) I600 (3) I690 (3) SA36 (3) 405 (1) SA569 (2)	None	N/A	N/A	~5.5	None	Air Saturated Control, No Scavenger or NH3 added

Experiment No.	Materials (No. of coupons)	Oxygen Scavenger	Conc.ppm	pH Range in Experiment	Ave. pH	Nitrogen Sparging	Notes
8	1010 (2) I600 (1) I690 (1) SA36 (2) SA569 (2) E70S (1) E7018 (1)	DEHA	1	9.8-10.3	10.0	Continuous	NH3 used to adjust pH
9	1010 (2) I600 (1) I690 (1) SA36 (2) SA569 (3) E70S (1) E7018 (1)	CHZ	1	8.7-9.9	9.4	Continuous	NH3 used to adjust pH
10	1010 (2) I600 (1) I690 (1) SA36 (2) SA 569 (2) E70S (1) E7018 (1)	None	N/A	9.0-10.3	9.4	Continuous	NH3 used to adjust pH
11	1010 (2) I600 (2) I690 (2) SA36 (3) SA569 (3)	None	N/A	N/A	~5.5	None	Air Saturated, Coupons exposed above a pool of stagnant water. Coupons sprayed with DI water at beginning of run
12	1010 (2) I600 (2) I690 (1) SA36 (1) 405 (2) SA569 (2) E70S (2) E7018 (1)	N2H4	50	9.1-10.0	9.4	Continuous	NH3 used to adjust pH
13	1010 (1) I600 (2) I690 (2) SA36 (1) 405 (2) SA569 (1) E70S (1) E7018 (2)	N2H4	25	9.1-10.0	9.4	Continuous	NH3 used to adjust pH
14	1010 (3) I600 (2) I690 (2) SA36 (2) 405 (1) SA569 (2)	N2H4	1	9.2-10.1	9.4	Continuous	NH3 used to adjust pH
15	1010 (2) SA36 (2)	DEHA	1	9.0-9.9	9.3	Continuous	NH3 used to adjust pH

Experiment No.	**Materials (No. of coupons)**	**Oxygen Scavenger**	**Conc.ppm**	**pH Range in Experiment**	**Ave. pH**	**Nitrogen Sparging**	**Notes**
	405 (3) SA569 (2)						
16	Above Waterline 1010 (3) SA36 (2) SA569 (2) Below Waterline 1010 (3) SA36 (4) SA569 (4) E70S (3)	None	N/A	8.9-10.3	9.85	Initial to reduce DO <20 ppb, 3x for 5 minutes during remainder of the experiment	NH3 used to adjust pH. Coupons exposed above and below waterline
17	Above Waterline SA569 (3) I690 (2) I600 (1) Below Waterline 1010 (2) SA36 (3) E7018 (3) I690 (3) I600(3)	None	N/A	N/A	~5.5	None	Air Saturated Control, No Scavenger or NH3 added. Coupons exposed above and below waterline. Coupons above waterline sprayed with DI water at beginning of run.

Effects of Oxygen Scavengers

The weight loss measurements are presented in the following tables for each of the oxygen scavengers and for each material. The results for the control and air saturated tests without an oxygen scavenger are presented later in this section. In the tables below, W1 represents the weight of the coupon before exposure to the test solution. W2 represents the weight of the coupon after exposure and chemical cleaning of the corrosion products. The concentration column is for the oxygen scavenger, and represents the initial concentration of oxygen scavenger. No measurements were made of the oxygen scavenger concentration either during or at the end of each test. There were no additions of oxygen scavenger made after each test was initiated. However in two of the tests, some additional test solution (at the initial concentration) was added to the test vessels during the course of the experiment. T is the total mils of corrosion during the experiment and R is the calculated corrosion rate in mils per year. The lower limit reported for corrosion of the I600 and I690 corrosion coupons is 0.013 mpy, for all other materials the lower limit is 0.014 mpy. It should be noted that in some cases, W2 the weight after the test and cleaning was slightly greater than W1, the weight before exposure. In these cases, the corrosion rate was reported as less than the lower limit of 0.013 or 0.014 mpy, since the positive weight gain must represent experimental error. The pH was not held constant in any tests but was maintained above 9.0 as indicated in Table 5-1.

Inconel 600

Table 5-2
Corrosion Rate of Inconel 600 as a function of Hydrazine Concentration

Coupon Number	W1 (g)	W2 (g)	Hydrazine Concentration (ppm)	T (mils)	R (mils per year)
I 600-4	23.799	23.798	1	0.001	0.020
I 600-5	23.834	23.833	1	<0.001	<0.013
I 600-3	23.771	23.770	25	0.001	0.020
I 600-7	23.769	23.769	25	<0.001	<0.013
I 600-1	23.835	23.835	50	<0.001	<0.013
I 600-2	23.633	23.632	50	<0.001	<0.013
I 600-4	23.799	23.804	1000	<0.001	<0.013
I 600-6	23.682	23.681	1000	0.002	0.023
I 600-3	23.770	23.768	10000	0.003	0.048
I 600-7	23.771	23.769	10000	0.005	0.065
I 600-1	23.834	23.842	25000	<0.001	<0.013
I 600-2	23.633	23.623	25000	0.020	0.289
I 600-1	23.834	23.834	50000	<0.001	<0.013
I 600-2	23.633	23.633	50000	<0.001	<0.013

Table 5-3
Corrosion Rate of Inconel 600 as a function of DEHA Concentration

Coupon Number	W1 (g)	W2 (g)	DEHA Concentration (ppm)	T (mils)	R (mils per year)
I 600-1	23.842	23.835	1	0.016	0.182
I 600-3	23.770	23.770	150	<0.001	<0.013

Table 5-4
Corrosion Rate of Inconel 600 as a function of Carbohydrazide Concentration

Coupon Number	W1 (g)	W2 (g)	Carbohydrazide Concentration (ppm)	T (mils)	R (mils per year)
I 600-3	23.768	23.771	1	<0.001	<0.013
I 600-4	23.799	23.799	100	<0.001	<0.013

The Inconel 600 results for the three oxygen scavengers are summarized in Figure 5-1. Note, with the exception of two points all of results were at or near the lower limit of detection (0.013 mpy) and well below the allowable corrosion rate of 0.175 mpy. The two extreme points may well have been due to experimental error. The number in parentheses indicates the number of test results at the specified point.

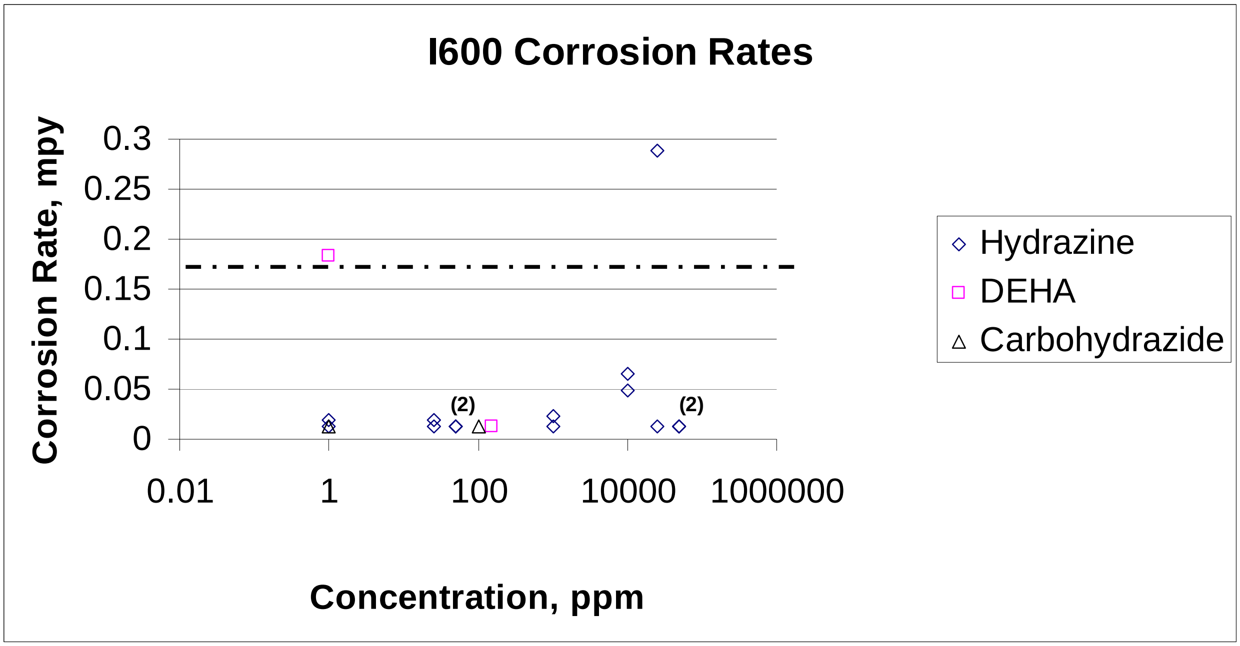


Figure 5-1
Corrosion Rate of Inconel 600 as a function of Oxygen Scavenger Concentration

Inconel 690

Table 5-5
Corrosion Rate of Inconel 690 as a function of Hydrazine Concentration

Coupon Number	W1 (g)	W2 (g)	Hydrazine Concentration (ppm)	T (mils)	R (mils per year)
I 690-4	26.665	26.665	1	<0.001	0.014
I 690-5	27.912	27.911	1	0.002	0.026
I 690-2	26.468	26.468	25	<0.001	<0.013
I 690-3	28.175	28.175	25	<0.001	<0.013
I 690-1	28.611	28.611	50	<0.001	<0.013
I 690-4	26.664	26.665	1000	<0.001	<0.013
I 690-5	27.912	27.910	1000	0.004	0.056
I 690-2	26.468	26.469	10000	<0.001	<0.013
I 690-3	28.175	28.173	10000	0.003	0.052
I 690-1	28.612	28.611	25000	0.001	0.020
I 690-2	26.469	26.468	50000	<0.001	<0.013

Table 5-6
Corrosion Rate of Inconel 690 as a function of DEHA Concentration

Coupon Number	W1 (g)	W2 (g)	DEHA Concentration (ppm)	T (mils)	R (mils per year)
I 690-1	28.611	28.6114	1	<0.001	<0.013
I 690-3	28.175	28.17501	150	<0.001	<0.013
I 690-4	26.664	26.66383	150	<0.001	<0.013

Table 5-7
Corrosion Rate of Inconel 690 as a function of Carbohydrazide Concentration

Coupon Number	W1 (g)	W2 (g)	Carbohydrazide Concentration (ppm)	T (mils)	R (mils per year)
I 690-10	28.287	28.290	1	<0.001	<0.013

The results for Inconel 690 are summarized in Figure 5-2. All of the measured corrosion rates are well below the corrosion allowance of 0.175 mpy. Most of the results are below the lower limit of detection of 0.013 mpy.

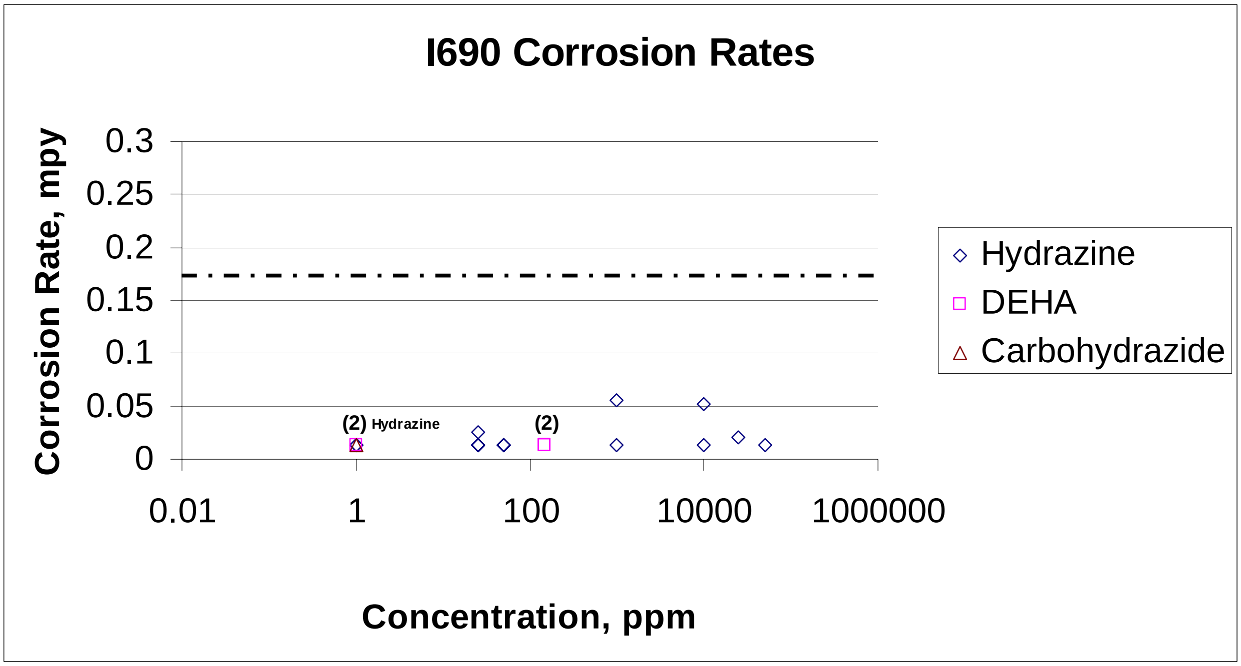


Figure 5-2
Corrosion Rate of Inconel 690 as a function of Oxygen Scavenger Concentration

405 Stainless Steel

Table 5-8
Corrosion Rate of 405 SS as a function of Hydrazine Concentration

Coupon Number	W1 (g)	W2 (g)	Hydrazine Concentration (ppm)	T (mils)	R (mils per year)
405-6	6.819	6.819	1	<0.001	<0.014
405-2	6.840	6.840	25	0.001	0.020
405-3	6.709	6.709	25	0.001	0.021
405-1	6.709	6.709	50	<0.001	<0.014
405-4	6.618	6.617	50	0.002	0.025
405-6	6.825	6.825	1000	<0.001	<0.014
405-7	6.776	6.779	1000	<0.001	<0.014
405-3	6.716	6.715	10000	<0.001	<0.014
405-2	6.8465	6.8648	10000	<0.001	<0.014
405-4	6.625	6.624	25000	<0.001	<0.014
405-1	6.7146	6.7748	25000	<0.001	<0.014
405-1	6.715	6.715	50000	<0.001	<0.014
405-2	6.847	6.847	50000	<0.001	0.021

Table 5-9
Corrosion Rate of 405 SS as a function of DEHA Concentration

Coupon Number	W1 (g)	W2 (g)	DEHA Concentration (ppm)	T (mils)	R (mils per year)
405-5	6.339	6.339	1	<0.001	<0.014
405-8	6.733	6.733	1	0.001	0.018
405-9	6.985	6.984	1	0.002	0.025
405-4	6.624	6.618	1	0.015	0.190
405-3	6.716	6.716	150	<0.001	<0.014
405-4	6.625	6.625	150	<0.001	<0.014
405-5	6.347	6.347	150	<0.001	<0.014

Table 5-10
Corrosion Rate of 405 SS as a function of Carbohydrazide Concentration

Coupon Number	W1 (g)	W2 (g)	Carbohydrazide Concentration (ppm)	T (mils)	R (mils per year)
405-6	6.825	6.825	100	<0.001	<0.014
405-7	6.776	6.776	100	<0.001	<0.014
405-8	6.743	6.743	100	0.002	0.019

The results for 405 SS are shown in Figure 5-3. All of the corrosion rates are well below the corrosion allowance and most are at or below the lower limit of detection of 0.014 mpy.

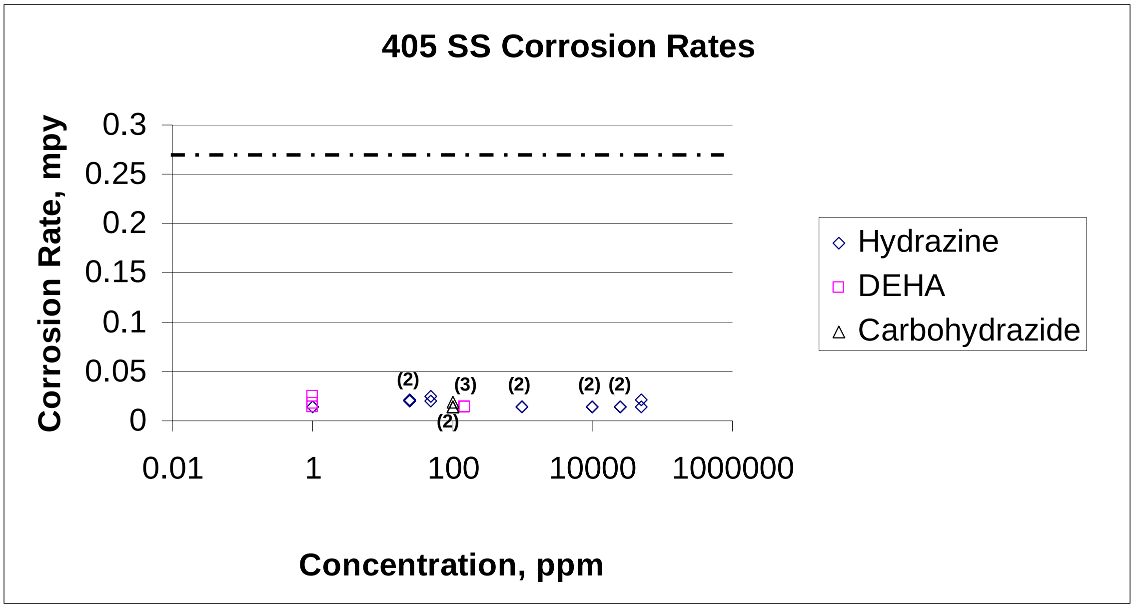


Figure 5-3
Corrosion Rate of 405 SS as a function of Oxygen Scavenger Concentration

1010 Carbon Steel

Table 5-11
Corrosion Rate of 1010 CS as a function of Hydrazine Concentration

Coupon Number	W1 (g)	W2 (g)	Hydrazine Concentration (ppm)	T (mils)	R (mils per year)
1010-4	21.355	21.313	1	0.094	1.149
1010-5	21.168	21.111	1	0.129	2.039
1010-7	21.003	20.969	1	0.076	1.206
1010-6	21.200	21.192	25	0.019	0.298
1010-1	21.082	21.033	50	0.109	1.734
1010-2	23.314	23.278	50	0.080	1.273
1010-4	21.398	21.380	1000	0.041	0.594
1010-5	21.208	21.195	1000	0.029	0.424
1010-7	21.043	21.036	1000	0.017	0.248
1010-3	21.216	21.216	10000	<0.001	<0.014
1010-6	21.215	21.215	10000	<0.001	<0.014
1010-1	21.168	21.150	25000	0.039	0.571
1010-2	23.360	23.339	25000	0.048	0.694
1010-1	21.174	21.168	50000	0.011	0.140

Table 5-12
Corrosion Rate of 1010 CS as a function of DEHA Concentration

Coupon Number	W1 (g)	W2 (g)	DEHA Concentration (ppm)	T (mils)	R (mils per year)
1010-8	20.092	20.036	1	0.125	1.986
1010-9	20.987	20.918	1	0.156	2.470
1010-1	21.150	21.082	1	0.153	1.741
1010-2	23.339	23.314	1	0.056	0.642
1010-3	21.216	21.216	150	<0.001	<0.014
1010-4	21.398	21.398	150	<0.001	<0.014

Table 5-13
Corrosion Rate of 1010 CS as a function of Carbohydrazide Concentration

Coupon Number	W1 (g)	W2 (g)	Carbohydrazide Concentration (ppm)	T (mils)	R (mils per year)
1010-6	21.215	21.200	1	0.034	0.385
1010-10	21.278	21.268	1	0.022	0.355
1010-5	21.210	21.208	100	0.003	0.037

The results for 1010 carbon steel are summarized in Figure 5-4. There is a correlation of corrosion rate with concentration for all three oxygen scavengers. The use of carbohydrazide appeared to produce lower corrosion rates than hydrazine and DEHA. At the 1ppm oxygen scavenger level, some of the corrosion rates were at or near the corrosion limit for hydrazine and DEHA, but well below the corrosion limit for carbohydrazide.

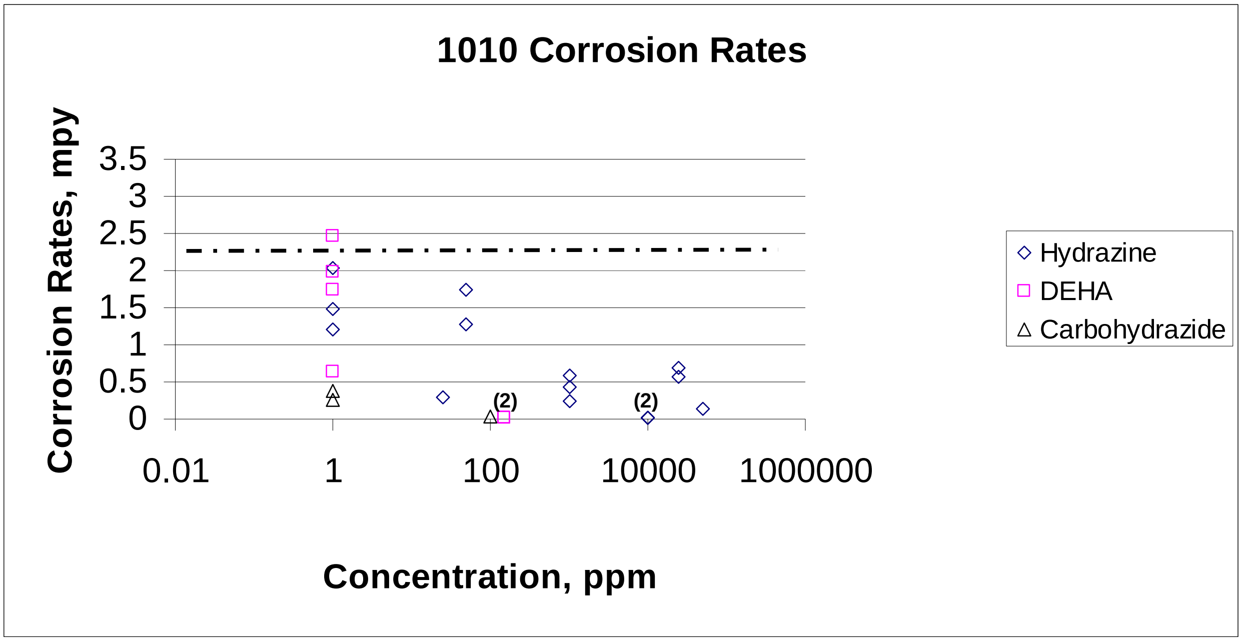


Figure 5-4
Corrosion Rate of 1010 CS as a function of Oxygen Scavenger Concentration

A569 Carbon Steel

Table 5-14
Corrosion Rate of A569 CS as a function of Hydrazine Concentration

Coupon Number	W1 (g)	W2 (g)	Hydrazine Concentration (ppm)	T (mils)	R (mils per year)
A 569-4	20.272	20.222	1	0.112	1.777
A 569-5	20.280	20.230	1	0.112	1.773
A 569-3	20.192	20.178	25	0.031	0.496
A 569-1	20.278	20.238	50	0.089	1.408
A 569-2	20.450	20.412	50	0.083	1.323
A 569-4	20.294	20.285	1000	0.020	0.293
A 569-5	20.347	20.331	1000	0.034	0.501
A 569-3	20.287	20.286	10000	0.004	0.051
A 569-1	20.367	20.352	25000	0.034	0.489
A 569-2	20.546	20.529	25000	0.039	0.563
A 569-1	20.375	20.367	50000	0.014	0.175
A 569-2	20.551	20.546	50000	0.008	0.102

Table 5-15
Corrosion Rate of A569 CS as a function of DEHA Concentration

Coupon Number	W1 (g)	W2 (g)	DEHA Concentration (ppm)	T (mils)	R (mils per year)
A 569-6	20.108	20.060	1	0.108	1.720
A 569-7	20.481	20.405	1	0.171	2.706
A 569-1	20.352	20.278	1	0.166	1.891
A 569-2	20.529	20.450	1	0.178	2.029
A 569-3	20.288	20.287	150	0.001	0.010
A 569-4	20.295	20.294	150	0.002	0.019

Table 5-16
Corrosion Rate of A569 CS as a function of Carbohydrazide Concentration

Coupon Number	W1 (g)	W2 (g)	Carbohydrazide Concentration (ppm)	T (mils)	R (mils per year)
A 569-3	20.286	20.192	1	0.211	2.406
A 569-4	20.285	20.272	1	0.030	0.336
A 569-5	20.331	20.280	1	0.114	1.300
A 569-5	20.349	20.347	100	0.003	0.043

The results for A569 are summarized in Figure 5-5   The results are similar to 1010. There is a correlation of corrosion rate with oxygen scavenger concentration.   On average, it also appears that carbohydrazide performed better with respect to corrosion rate, however, there is more scatter than with 1010 carbon steel.

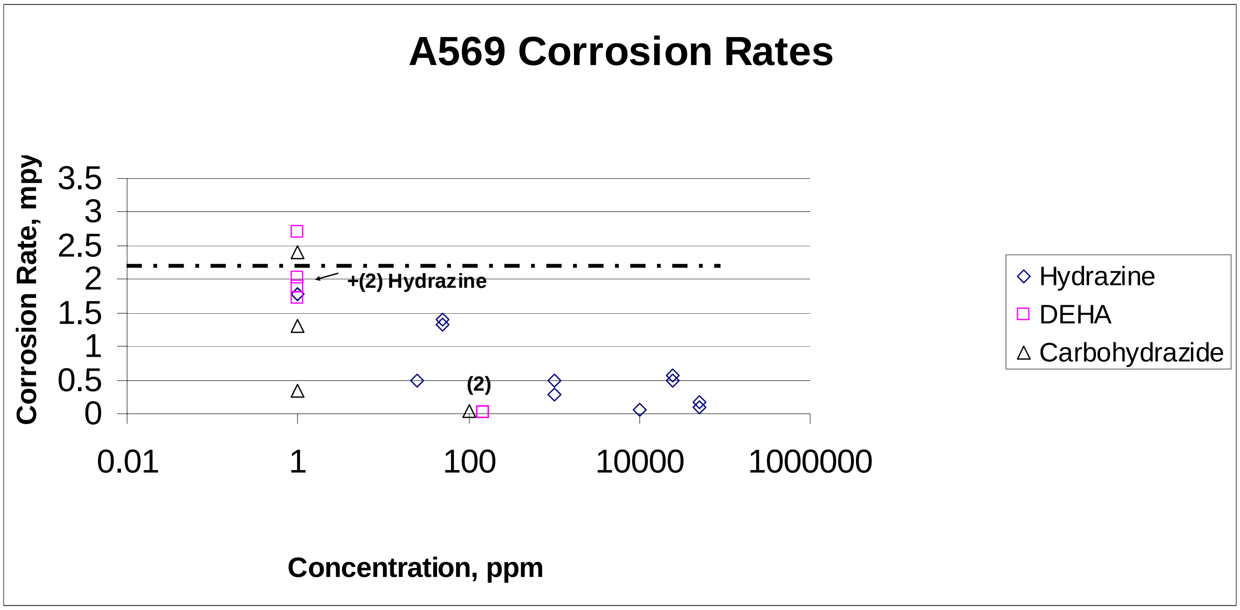


Figure 5-5
Corrosion Rate of A569 CS as a function of Oxygen Scavenger Concentration

A36 Carbon Steel

Table 5-17
Corrosion Rate of A36 CS as a function of Hydrazine Concentration

Coupon Number	W1 (g)	W2 (g)	Hydrazine Concentration (ppm)	T (mils)	R (mils per year)
A 36-3	23.091	23.054	1	0.085	1.351
A 36-4	23.251	23.208	1	0.100	1.594
A 36-2	23.055	23.042	25	0.029	0.459
A 36-1	21.122	21.083	50	0.090	1.432
A 36-3	23.125	23.109	1000	0.035	0.514
A 36-4	23.291	23.280	1000	0.026	0.375
A 36-2	23.093	23.089	10000	0.010	0.142
A 36-1	21.219	21.200	25000	0.042	0.611
A 36-2	23.096	23.093	50000	0.006	0.071

Table 5-18
Corrosion Rate of A36 CS as a function of DEHA Concentration

Coupon Number	W1 (g)	W2 (g)	DEHA Concentration (ppm)	T (mils)	R (mils per year)
A 36-5	23.260	23.188	1	0.167	2.648
A 36-6	23.304	23.237	1	0.155	2.460
A 36-1	21.200	21.122	1	0.182	2.077
A 36-2	23.089	23.055	1	0.079	0.897
A 36-3	23.126	23.125	150	0.003	0.038
A 36-4	23.292	23.291	150	0.002	0.021

Table 5-19
Corrosion Rate of A36 CS as a function of Carbohydrazine Concentration

Coupon Number	W1 (g)	W2 (g)	Carbohydrazide Concentration (ppm)	T (mils)	R (mils per year)
A 36-4	23.280	23.251	1	0.066	0.755
A 36-7	23.261	23.208	1	0.123	1.399

The results for A36 are summarized in Figure 5-6. The results are similar to both 1010 and A569.

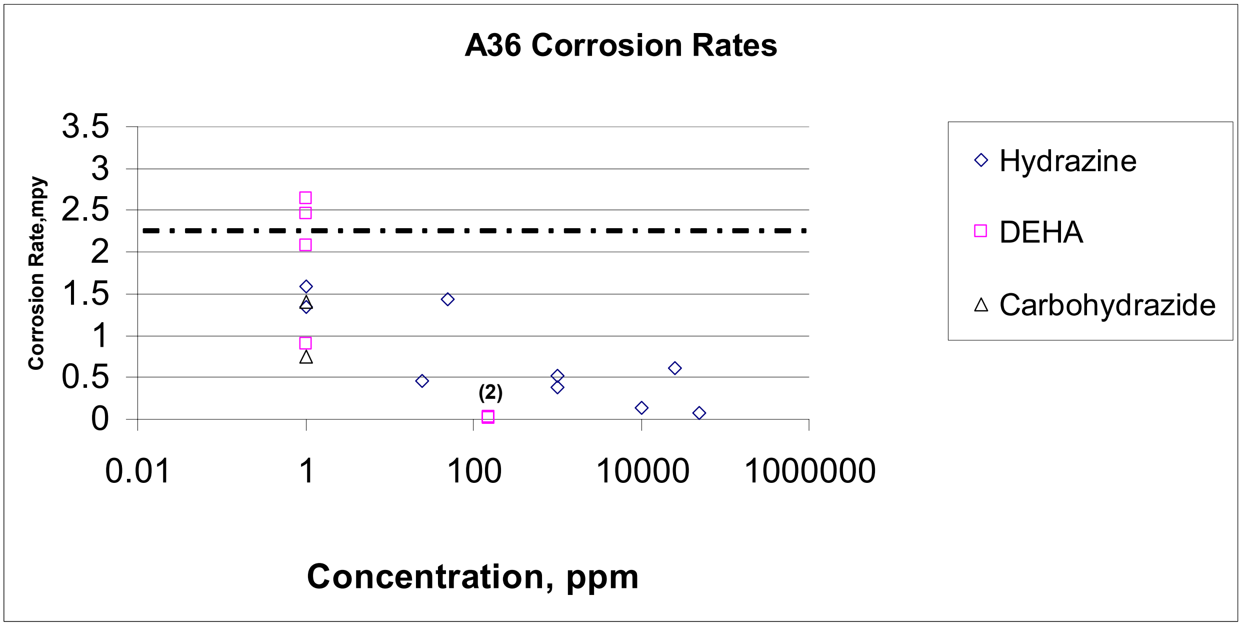


Figure 5-6
Corrosion Rate of A36 CS as a function of Oxygen Scavenger Concentration

E 70-S Weld Metal

Table 5-20
Corrosion Rate of E-70-S as a function o f Hydrazine Concentration

Coupon Number	W1 (g)	W2 (g)	Hydrazine Concentration (ppm)	T (mils)	R (mils per year)
E 70S-3	21.722	21.667	25	0.124	1.961
E 70S-1	21.439	21.360	50	0.176	2.798
E 70S-2	21.624	21.563	50	0.136	2.153
E 70S-3	21.744	21.743	10000	0.002	0.033
E 70S-1	21.532	21.526	25000	0.012	0.181
E 70S-2	21.710	21.698	25000	0.028	0.402

Table 5-21
Corrosion Rate of E-70-S as a function of DEHA Concentration

Coupon Number	W1 (g)	W2 (g)	DEHA Concentration (ppm)	T (mils)	R (mils per year)
E 70s-1	21.526	21.439	1	0.195	2.226

Table 5-22
Corrosion Rate of E-7018 as a function of Carbohydrazide Concentration

Coupon Number	W1 (g)	W2 (g)	Carbohydrazide Concentration (ppm)	T (mils)	R (mils per year)
E 70s-2	21.698	21.624	1	0.165	1.885

The data for E-70-S is summarized in Figure 5-7. The correlation of corrosion rate with oxygen scavenger concentration was less pronounced with E-70-S, although there was less experimental data for E-70-S. It is also noted that all corrosion rates were well below the corrosion allowance which is much higher than for the carbon steels previously shown.

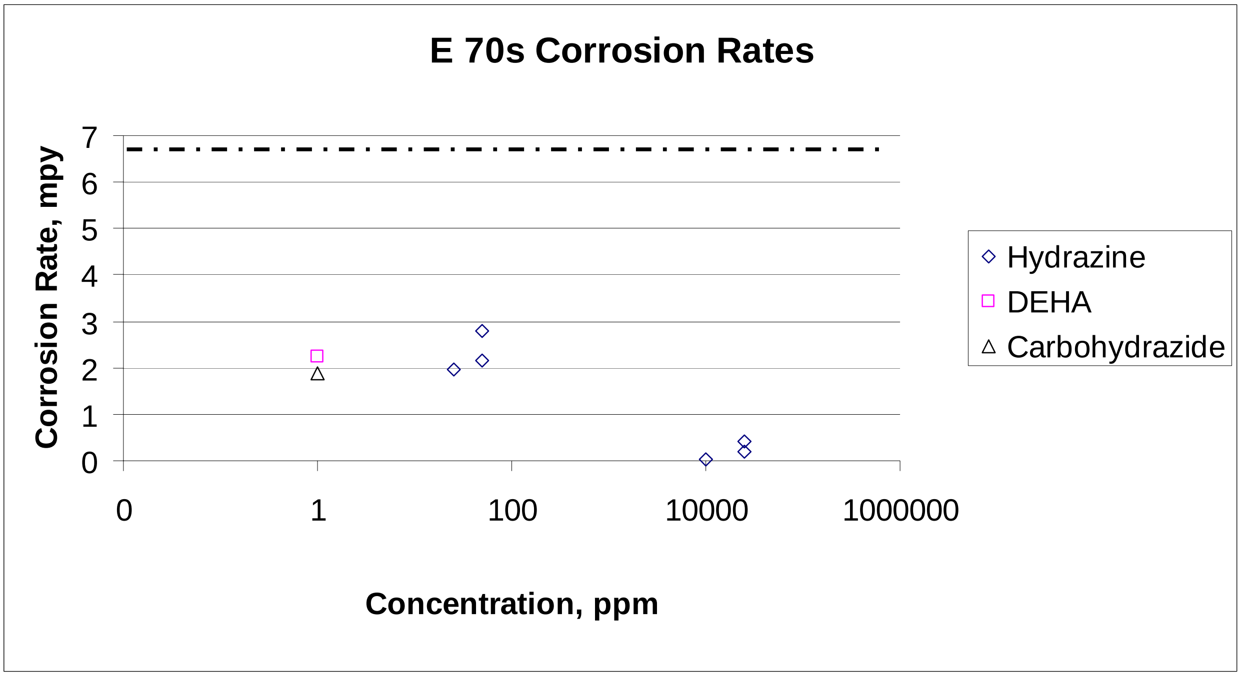


Figure 5-7
Corrosion Rate of E-70-S as a function of Oxygen Scavenger Concentration

E-7018 Weld Metal

Table 5-23
Corrosion Rate of E-7018 as a function of Hydrazine Concentration

Coupon Number	W1 (g)	W2 (g)	Hydrazine Concentration (ppm)	T (mils)	R (mils per year)
E 7018-2	21.807	21.780	25	0.060	0.954
E 7018-3	21.450	21.402	25	0.106	1.681
E 7018-1	21.749	21.698	50	0.113	1.794
E 7018-2	21.856	21.859	10000	<0.001	<0.014
E 7018-3	21.477	21.475	10000	0.004	0.054
E 7018-1	21.900	21.862	25000	0.083	1.210

Table 5-24
Corrosion Rate of E-7018 as a function of DEHA Concentration

Coupon Number	W1 (g)	W2 (g)	DEHA Concentration (ppm)	T (mils)	R (mils per year)
E 7018-1	21.862	21.749	1	0.255	2.903

Table 5-25
Corrosion Rate of E-7018 as a function of Carbohydrazide Concentration

Coupon Number	W1 (g)	W2 (g)	Carbohydrazide Concentration (ppm)	T (mils)	R (mils per year)
E 7018-2	21.859	21.807	1	0.116	1.322

The results for E-7018 are summarized in Figure 5-8. The results are similar to E-7018.

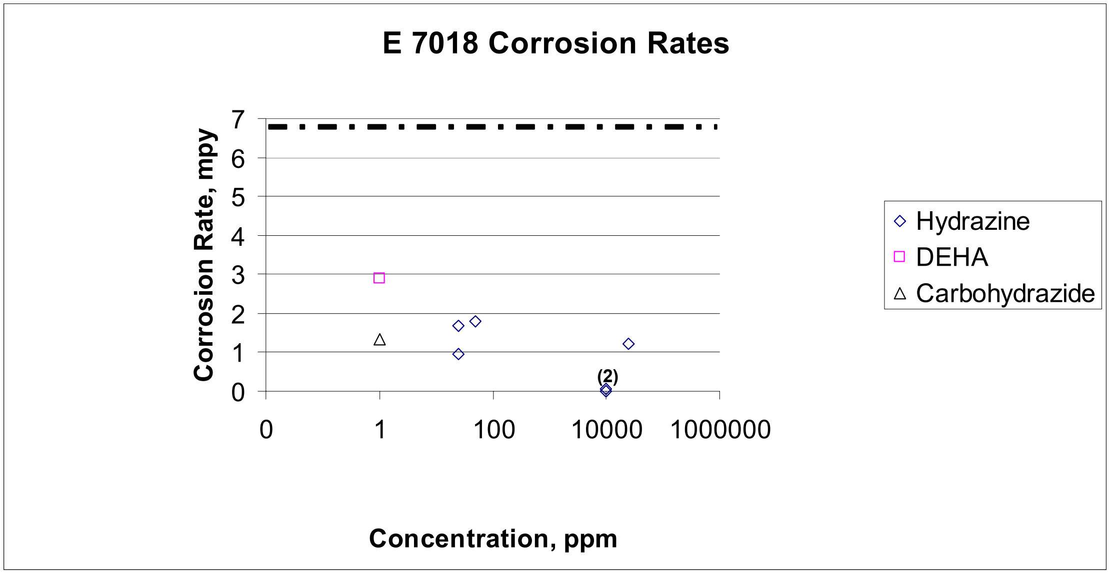


Figure 5-8
Corrosion Rate of E-7018 as a Function of Oxygen Scavenger Concentration

Control Environments

Air Saturated Conditions

Three sets of experiments were completed under air saturated conditions with no chemical additives as described in Table 5-1. The main difference in these sets of experiments was whether the coupons were exposed within the demineralized water test solution or above the pool of demineralized water in the vapor space. The latter case is meant to simulate conditions where the SG's have been partially drained and some portions of the SG would be exposed above the waterline. The experimental results are presented in the following sets of tables.

I600

Table 5-26
Corrosion Rate of I600 in Air Saturated Demineralized Water

Coupon	W1 (g)	W3 (g)	Experiment No.	T (mils)	R (mils per year)
I 600-5	23.835	23.824	7	0.023	0.336
I 600-9	23.672	23.661	7	0.022	0.327
I 600-10	23.762	23.765	7	<0.001	<0.013
I 600-2	23.632	23.631	17b	<0.001	<0.013
I 600-3	23.770	23.771	17b	<0.001	<0.013
I 600-4	23.798	23.801	17b	<0.001	<0.013

Table 5-27
Corrosion Rate of I600 in Vapor above Air Saturated Demineralized Water

Coupon	W1 (g)	W3 (g)	Experiment No.	T (mils)	R (mils per year)
I 600-4	23.804	23.799	11	0.011	0.128
I 600-9	23.661	23.667	11	<0.001	<0.013
I 600-8	23.734	23.740	17a	<0.001	<0.013

I 690

Table 5-28
Corrosion Rate of I690 in Air Saturated Demineralized Water

Coupon	W1 (g)	W3 (g)	Experiment No.	T (mils)	R (mils per year)
I 690-8	28.058	28.024	7	0.071	1.031
I 690-9	26.561	26.514	7	0.097	1.416
I 690-10	28.290	28.287	7	0.006	0.092
I 690-2	26.468	26.469	17b	<0.001	<0.013
I 690-3	28.175	28.177	17b	<0.001	<0.013
I 690-4	26.665	26.665	17b	<0.001	<0.013

Table 5-29
Corrosion Rate of I690 in Vapor above Air Saturated Demineralized Water

Coupon	W1 (g)	W3 (g)	Experiment No.	T (mils)	R (mils per year)
I 690-5	27.910	27.912	11	<0.001	<0.013
I 690-8	28.024	28.058	11	<0.001	<0.013
I 690-1	28.611	28.611	17a	<0.001	<0.013
I 690-5	27.911	27.911	17a	<0.001	<0.013

405 SS

Table 5-30
Corrosion Rate of 405 SS in Air Saturated Demineralized Water

Coupon	W1 (g)	W3 (g)	Experiment No.	T (mils)	R (mils per year)
405-5	6.347	6.347	7	<0.001	<0.014

1010 Carbon Steel

Table 5-31
Corrosion Rate of 1010 CS in Air Saturated Demineralized Water

Coupon	W1 (g)	W3 (g)	Experiment No.	T (mils)	R (mils per year)
1010-8	21.531	21.433	7	0.217	3.173
1010-9	21.100	21.013	7	0.192	2.809
1010-10	21.388	21.278	7	0.246	3.592
1010-1	21.033	20.947	17b	0.191	2.793
1010-2	23.278	23.215	17b	0.140	2.049

Table 5-32
Corrosion Rate of 1010 CS in Vapor Above Air Saturated Demineralized Water

Coupon	W1 (g)	W3 (g)	Experiment No.	T (mils)	R (mils per year)
1010-4	21.380	21.355	11	0.055	0.632
1010-5	21.195	21.168	11	0.061	0.691

A569 Carbon Steel

Table 5-33
Corrosion Rate of A569 CS in Air Saturated Dimeralized Water

Coupon	W1 (g)	W3 (g)	Experiment No.	T (mils)	R (mils per year)
A 569-6	20.234	20.132	7	0.228	3.334
A 569-9	20.193	20.114	7	0.177	2.579

Table 5-34
Corrosion Rate of A569 CS in Vapor above Air Saturated Demineralized Water

Coupon	W1 (g)	W3 (g)	Experiment No.	T (mils)	R (mils per year)
A 569-7	20.492	20.481	11	0.024	0.278
A 569-8	20.538	20.525	11	0.027	0.311
A 569-10	20.337	20.326	11	0.024	0.275
A 569-1	20.238	20.200	17a	0.086	1.263
A 569-2	20.412	20.397	17a	0.035	0.512
A 569-3	20.178	20.156	17a	0.049	0.714

A36 Carbon Steel

Table 5-35
Corrosion Rate of A36 CS in Air Saturated Demineralized Water

Coupon	W1 (g)	W3 (g)	Experiment	T (mils)	R (mils per

			No.		year)
A 36-5	23.374	23.281	7	0.214	3.130
A 36-7	23.275	23.261	7	0.032	0.461
A 36-8	23.334	23.180	7	0.358	5.228
A 36-1	21.083	21.004	17b	0.181	2.645
A 36-2	23.042	22.963	17b	0.182	2.662
A 36-3	23.054	22.970	17b	0.194	2.831

Table 5-36
Corrosion Rate of A36 CS in Vapor above Air Saturated Demineralized Water

Coupon	W1 (g)	W3 (g)	Experiment No.	T (mils)	R (mils per year)
A 36-3	23.109	23.091	11	0.0435	0.496
A 36-6	23.325	23.304	11	0.0486	0.554
A 36-9	23.379	23.358	11	0.050	0.565

E-7018 Weld Metal

Table 5-37
Corrosion Rate of E-7018 in Air Saturated Demineralized Water

Coupon	W1 (g)	W3 (g)	Experiment No.	T (mils)	R (mils per year)
E 7018-1	21.698	21.593	17b	0.234	3.419
E 7018-2	21.780	21.668	17b	0.250	3.657
E 7018-3	21.402	21.306	17b	0.216	3.158

Controlled Conditions without Oxygen Scavengers

Two experiments were run with nitrogen sparging used to remove dissolved oxygen and pH control using ammonia. No oxygen scavengers were used in these tests. The test conditions are summarized in Table 5-1. Experiment No. 10 used a continuous nitrogen sparge throughout the entire test period. Experiment No. 16 used an initial nitrogen sparge to reduce the dissolved oxygen to <20 ppb. The nitrogen was discontinued except for five minute nitrogen sparges four times during the subsequent 25 day test period. In Experiment No. 10, the dissolved oxygen was always <20 ppb, where in Experiment No. 16, it was <20 ppb for only a short period in between the sparge procedures.

I600

Table 5-38
Corrosion Rate of I600 in pH Controlled Low Dissolved Oxygen Water

Coupon	W1 (g)	W3 (g)	Experiment	T (mils)	R (mils per year)

			No[1].		
I 600-5	23.824	23.834	10	<0.001	<0.013

1-continuous nitrogen sparge

Table 5-39
Corrosion Rate of I690 in pH Controlled Low Dissolved Oxygen Water

Coupon	W1 (g)	W3 (g)	Experiment No.[1]	T (mils)	R (mils per year)
I 690-2	26.469	26.468	10	0.001	<0.013

1-continuous nitrogen sparge

1010 Carbon Steel

Table 5-40
Corrosion Rate of 1010 CS in pH Controlled Low Dissolved Oxygen Water

Coupon	W1 (g)	W3 (g)	Experiment No.[1,2]	T (mils)	R (mils per year)
1010-7	21.036	21.003	10	0.074188	0.846
1010-9	21.013	20.988	10	0.058434	0.667
1010-3	21.131	21.042	16b	0.1991	2.907
1010-4	21.313	21.212	16b	0.226362	3.305
1010-5	21.111	21.020	16b	0.203793	2.975

1-Experiment No. 10: continuous nitrogen sparge
2-Experiment No. 16b: initial nitrogen sparge, followed by periodic sparging

Table 5-41
Corrosion Rate of 1010 CS in Vapor above pH Controlled Low Dissolved Oxygen Water

Coupon	W1 (g)	W3 (g)	Experiment No.[2]	T (mils)	R (mils per year)
1010-6	21.192	21.178	16a	0.030	0.440
1010-7	20.969	20.954	16a	0.033	0.480
1010-8	20.036	20.010	16a	0.059	0.858

2-initial nitrogen sparge, followed by periodic sparging

A569 Carbon Steel

Table 5-42
Corrosion Rate of A569 CS in pH Controlled Low Dissolved Oxygen Water

Coupon	W1 (g)	W3 (g)	Experiment	T (mils)	R (mils per year)

			No.[1,2]		
A 569-6	20.132	20.108	10	0.054	0.612
A 569-9	20.114	21.408	10	<0.001	<0.014
A 569-4	20.222	20.134	16b	0.196	2.868
A 569-5	20.230	20.141	16b	0.199	2.900
A 569-6	20.060	19.966	16b	0.208	3.041
A 569-7	20.405	20.330	16b	0.166	2.427

1-Experiment No. 10: continuous nitrogen sparge

2-Experiment No. 16b: initial nitrogen sparge, followed by periodic sparging

Table 5-43
Corrosion Rate of A569 CS in Vapor above pH Controlled Low Dissolved Oxygen Water

Coupon	W1 (g)	W3 (g)	Experiment No.[2]	T (mils)	R (mils per year)
A 569-8	20.525	20.508	16a	0.038	0.561
A 569-9	21.408	21.390	16a	0.040	0.584

2-initial nitrogen sparge, followed by periodic sparging

A36 Carbon Steel

Table 5-44
Corrosion Rate of A36 CS in pH Controlled Low Dissolved Oxygen Water

Coupon	W1 (g)	W3 (g)	Experiment No.[1,2]	T (mils)	R (mils per year)
A 36-5	23.281	23.260	10	0.049	0.562
A 36-8	23.179	23.153	10	0.062	0.705
A 36-4	23.208	23.108	16b	0.231	3.378
A 36-5	23.188	23.110	16b	0.181	2.645
A 36-6	23.237	23.127	16b	0.256	3.733
A 36-7	23.208	23.094	16b	0.263	3.838

1-Experiment No. 10: continuous nitrogen sparge

2-Experiment No. 16b: initial nitrogen sparge, followed by periodic sparging

Table 5-45
Corrosion Rate of A36 CS in Vapor above pH Controlled Low Dissolved Oxygen Water

Coupon	W1 (g)	W3 (g)	Experiment No.[2]	T (mils)	R (mils per year)
A 36-8	23.153	23.139	16a	0.032	0.470
A 36-9	23.358	23.340	16a	0.0407	0.595

2-initial nitrogen sparge, followed by periodic sparging

E-7018 Weld Metal

Table 5-46
Corrosion Rate of E-7018 in pH Controlled Low Dissolved Oxygen Water

Coupon	W1 (g)	W3 (g)	Experiment No.[1]	T (mils)	R (mils per year)
E 7018-3	21.475	21.450	10	0.056	0.641

1-continuous nitrogen sparge

E-70-S Weld Metal

Table 5-47
Corrosion Rate of E-70-S in pH Controlled Low Dissolved Oxygen Water

Coupon	W1 (g)	W3 (g)	Experiment No.[1,2]	T (mils)	R (mils per year)
E 70s-3	21.743	21.722	10	0.047149	0.538
E 70s-1	21.360	21.219	16b	0.313957	4.584
E 70s-2	21.563	21.465	16b	0.218988	3.197
E 70s-3	21.667	21.569	16b	0.219658	3.207

1-Experiment No. 10: continuous nitrogen sparge
2-Experiment No. 16b: initial nitrogen sparge, followed by periodic sparging

6

DISCUSSION OF RESULTS

The primary purpose of this section is to review and compare the results of the current experimental program with other relevant data obtained in the literature review reported in section 2 of this report. It is important to recognize that the current experimental program is not yet complete so that many important questions identified in the gap analysis are still unanswered. Furthermore, it is recognized that additional testing to investigate galvanic and other local corrosion processes is necessary before any formal recommendations can be made to change the existing secondary water lay-up guidance. Recommendations for future work related to general corrosion are discussed later in this section.

The first conclusion that can be reached from the experimental work performed in this study is that the corrosion resistant alloys I600, I690 and 405 SS experience low and acceptable general corrosion rates under all of the dearated conditions tested. Although there were a couple of I600 measurements with higher than acceptable corrosion rates (probably as a result of experimental error), the majority of the data was at the limit of detection in this program. A general caution, however, as noted above is that galvanic and local corrosion should be evaluated before any overall general conclusion with respect to these alloys is drawn. Under aerated conditions without pH control both I600 and I690 experienced relatively high general corrosion rates for some specimens, but below LLD for other specimens.

The remainder of this section discusses the corrosion measurements for the carbon steels; 1010, A569 and A36. Less testing of the weld metals E-70-S and E-7018 was performed but the results were all similar to the carbon steels.

The results for individual carbon steels were presented in Section 5. For the purpose of evaluating the reproducibility of the experimental results, it is useful to show the results for all of the carbon steels on one graph since their corrosion behavior is expected to be similar. Figure 6-1 includes all of the hydrazine data for the three carbon steels tested, 1010, A-569 and A-36. A trend line is also drawn through the dataset for discussion purposes.

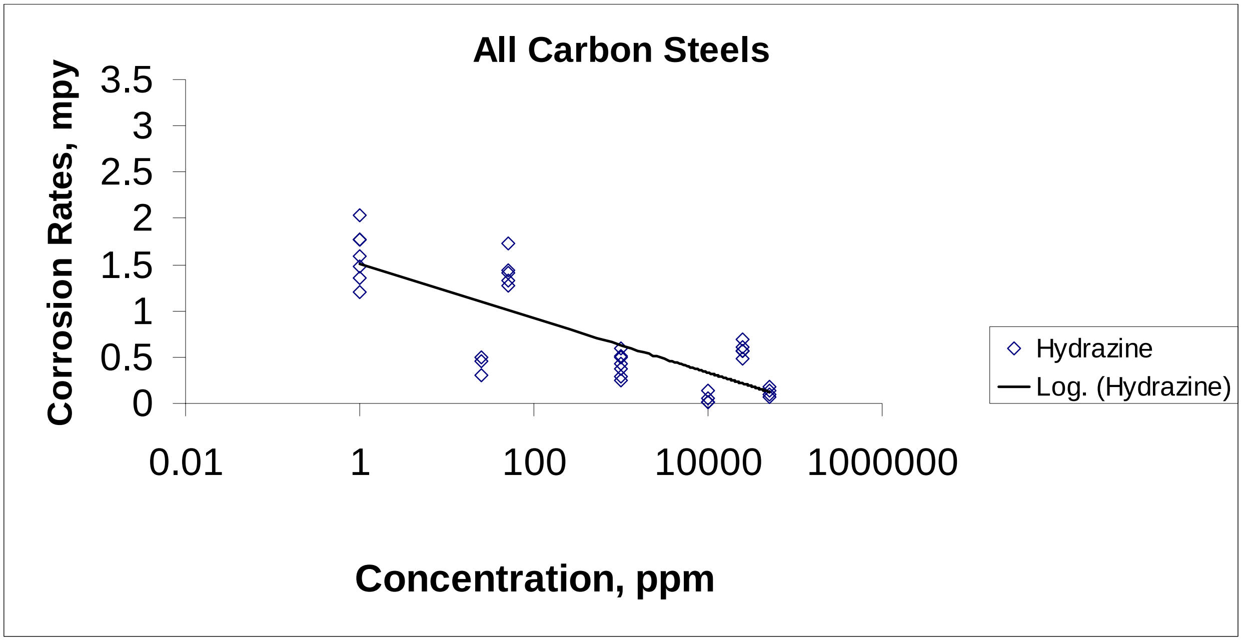


Figure 6-1
Summary of all Carbon Steel data with Hydrazine additions

Figure 6-1, in addition to showing the trend with hydrazine concentration gives a good representation of how repeatable individual test measurements were in this program. These results illustrate that individual measurements are quite reproducible when exposed to the same test solution in an individual test vessel. In other words, the scatter at each concentration level is quite low suggesting that experimental measurement error is quite low. At the 1 ppm concentration level, the scatter is larger and this may in fact be due to real differences in corrosion rates. On the other hand, the general trend of the data although linear on the corrosion rate vs. log concentration plot does exhibit a fair amount of scatter around the trend line. This suggests that the run to run variation in the corrosion rate is much greater. In other words, if multiple tests were repeated in different test vessels with the same solution composition and metals, the results may not be as repeatable. At any rate, the experimental results appear to be more than adequate to show the general trend as a function of solution composition and material type.

Effect of Solution pH on Corrosion Rates

Figures 6-2 through 6-4 show the effect of solution pH on the corrosion rates of the three carbon steels tested under deaerated conditions. The test results for I600, I690 and 405SS are not shown here since all the test results were at or near the limit of detection of corrosion. There was insufficient data to show correlations for either of the weld metals, E-70-S or E-7018.

The three carbon steels tested did not show any strong correlation with pH. The data for all three oxygen scavengers is shown in the figures. Since measurements were only made at two pHs for both DEHA and carbohydrazide, conclusions with respect to pH should not be drawn from this data alone. Hydrazine measurements were made at several pHs and thus this data is believed to be more representative. Two important points should be considered with respect to the hydrazine data. First, as shown in Table 5-1, the average solution pH did not correspond directly with the hydrazine concentration. In some cases, the pH was due to the scavenger alone and in some cases ammonia was added to control the pH >9.0. Secondly, the pH reported in Figures 6-2 through 6-4 is the average pH for the entire test period (typically 30 days). As described in Section 5 and as shown in the Appendix, the pH varied considerably during the test. Nonetheless, there is no clear correlation of corrosion data with pH. It is reasonable to conclude from this data that for oxygen free solutions over the range of pH from about 9.2-10.6 (e.g. approximately 1.5 pH units), the effect of oxygen scavenger concentration, over the practical range considered, is much more pronounced than the effect of solution pH on the observed carbon steel corrosion rate.

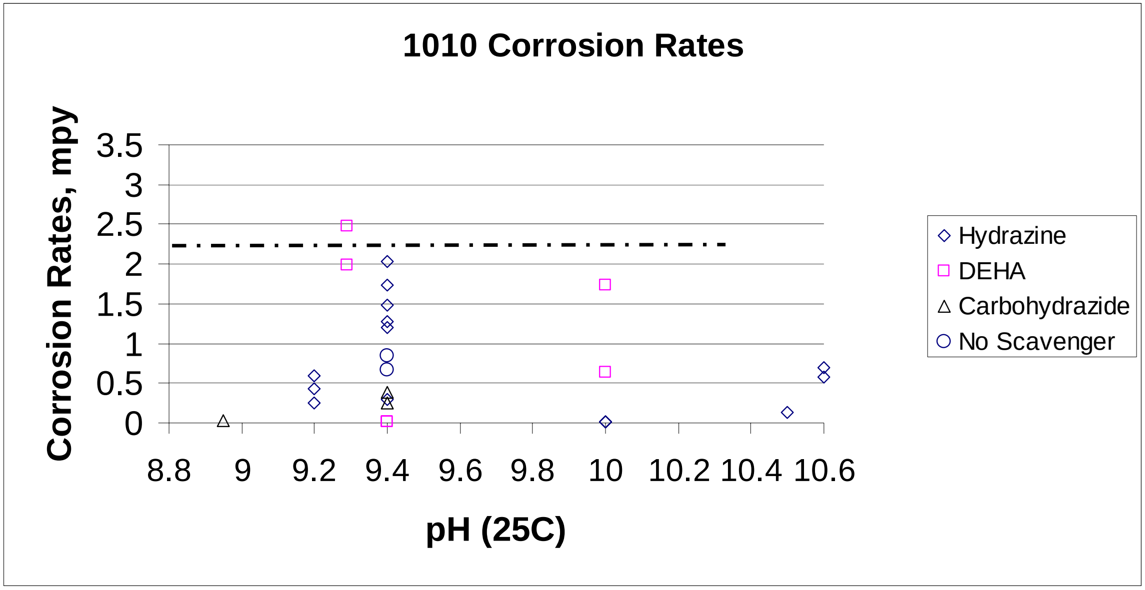


Figure 6-2
1010 Corrosion Rate as a Function of Average Solution pH

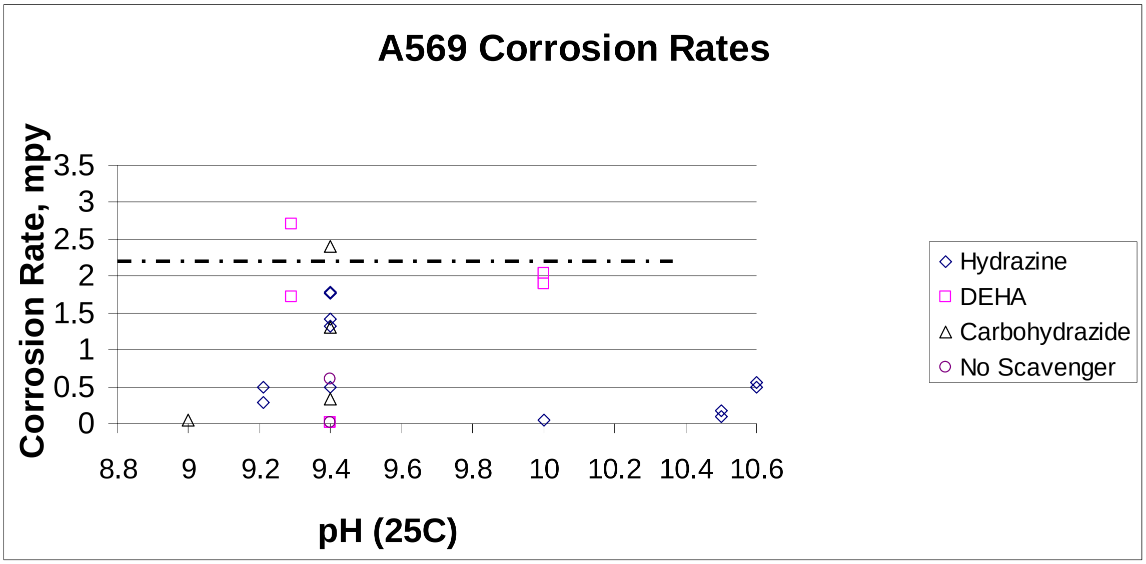


Figure 6-3
A569 Corrosion Rate as a Function of Average Solution pH

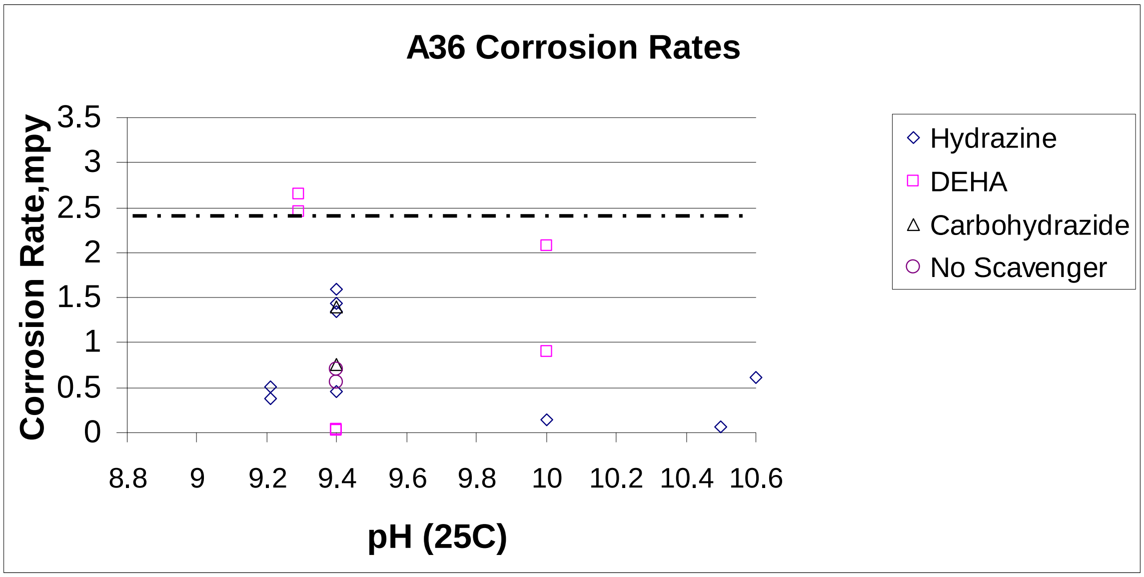


Figure 6-4
A36 Corrosion Rate as a Function of Average Solution pH

Comparison to Literature Data

In section 2, a detailed literature review of available corrosion data relevant to lay-up conditions was presented. Although the experimental program conducted here was designed to fill gaps in the available data rather than to repeat measurements, it is useful to compare the data from this study to the literature data where a comparison can be made.

The majority of the data from the experimental program conducted here was under dearated conditions (dissolved oxygen <20 ppb). Figure 3-3 included corrosion data for carbon steel under low oxygen conditions. The data from this study (1010, A569, A36) is plotted with the data from Figure 3-3 below. No definitive conclusions or correlations can be drawn from the combination of this data other than the observation that the corrosion rates reported here are of the same order as previously reported. This increases our confidence in the use of the data from this experimental program for evaluating lay-up chemistries.

Figure 3-1 was a summary of available data using oxygen scavengers under aerated conditions. No comparable measurements were made in this program, however Experiment # 16 was conducted under partially aerated conditions with pH control using NH3. In Experiment #16, the test vessel was initially sparged with nitrogen and 3 times for 5 minutes during the remainder of the test. For the majority of the test, the coupons were exposed to aerated conditions since the test vessel re-saturated with air quickly after the nitrogen purge. The data for the three carbon steels (1010, A569, and A36) is included with the original data from Figure 3-1 on Figure 6-6 below. The results from this program (with no oxygen scavenger present) suggest that pH control alone may be as effective as nominally 100 ppm hydrazine at a similar pH level. This conclusion is very tentative however, since the exact experimental conditions from the literature are not well known. In addition, it must be noted that Experiment #10, under nominally the same conditions at Experiment #16 except continuous nitrogen purge was used to maintain oxygen less than 25 ppb, had carbon steel corrosion rates about 5-times lower than Experiment #16b as indicated in Table 5-40. These new results underscore the importance of minimizing or eliminating dissolved oxygen from the system.

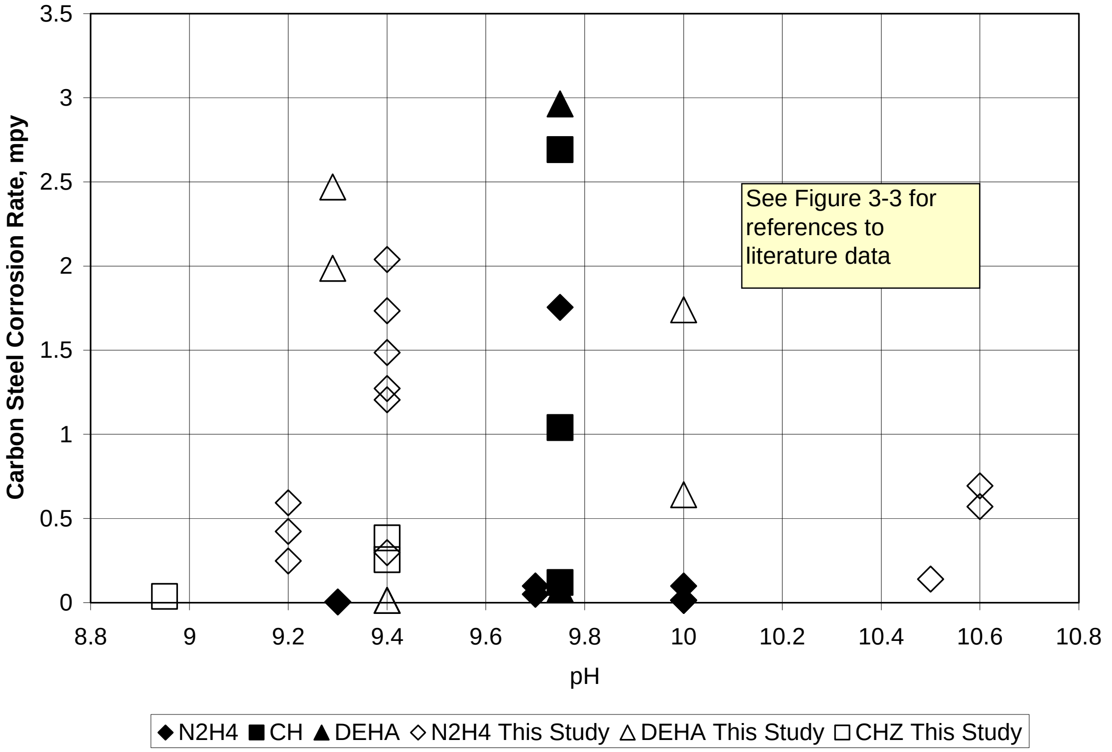


Figure 6-5
Comparison of Corrosion Data from This Study to Literature Data (Dearated Conditions)

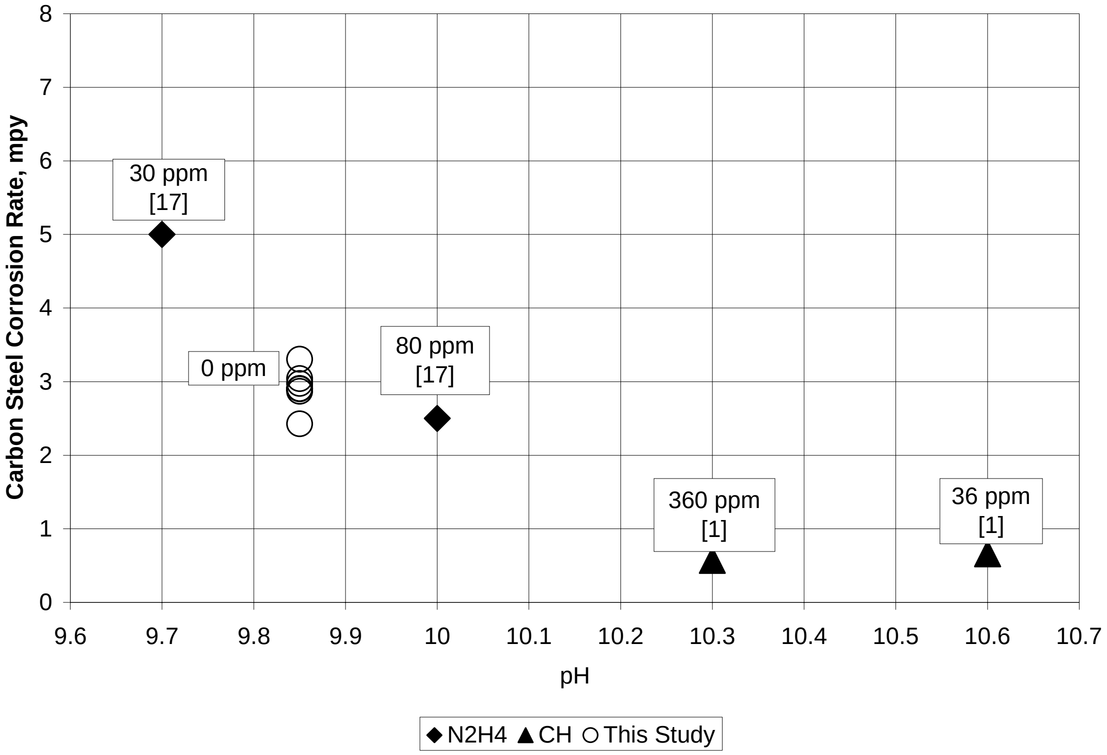


Figure 6-6
Comparison of Corrosion Data from This Study to Literature Data (Aerated Conditions)

Recommendations for Future Work

In addition to the general conclusions stated above, it is also clear from the test results that the introduction of oxygen markedly increases the corrosion rate and that the corrosion rate in the vapor space above air saturated demineralized or pH controlled water is much lower than in the water. Although this is not unexpected, the results from Experiments No. 10, 16 and 17 show this effect for all three carbon steels. This observation coupled with the earlier ones leads to some recommendations for future general corrosion testing. Although localized corrosion testing is also planned in the future, the scope of that testing is not discussed here. The following table contains recommendations for future general corrosion experiments. The list is not prioritized, however consideration would need to be given to which experiments should be completed first so that the design of future experiments could be refined.

Figure 6-7
Future General Corrosion Test Recommendations

Experiment Series	Materials	Test Conditions	Comments
A	Carbon Steels & Weld Metals-in liquid	Low Oxygen, vary pH with NH3. No oxygen scavenger	The experiments completed to date suggest that acceptable corrosion rates (e.g. less than allowance) can be achieved in dearated solutions without oxygen scavengers. The influence of pH is important and should be explored in a controlled manner.
B	Carbon Steels & Weld Metals-in liquid	Fixed pH (~9.5), vary oxygen from 0-200 ppb. No oxygen scavenger	The importance of oxygen control is clear from the current set of results. Tests to determine the dependence on concentration and/or the presence of a threshold concentration above which corrosion increases rapidly is needed.
C	Carbon Steels & Weld Metals-in vapor and liquid	Initial nitrogen purge, fixed pH, with and without oxygen scavengers	Experiment #16 was initially designed to simulate the plant practice of initial dearation at the beginning of the layup period. The test apparatus will need to be re-designed to better simulate plant conditions.
D	Carbon Steels & Weld Metals- vapor and liquid	Initial Aerated conditions, fixed pH with oxygen scavenger	Similar to Series C except use oxygen scavenger to reduce oxygen conentration

A

CHEMISTRY MEASUREMENT DATA

The chemistry measurements where available are reported in this Appendix. Average pH values are reported for each experiment. It should be noted that because the vessels were relatively stagnant and in many cases excessive corrosion product existed in the test vessel, the values reported here should be considered approximate. For the cases where the dissolved oxygen is reported <0.020 ppm, it is felt that the dissolved oxygen was less than this value. In most cases the oxygen probe reported 0.0 ppm.

Table A-1
Experiment No. 1 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	10.60	<0.020	325
3	10.87	0.31	206
4	10.73	<0.020	215
7	10.68	0.03	261
8	10.59	<0.020	291
9	10.50	<0.020	319
10	10.55	<0.020	326
11	10.50	<0.020	280.9
14	10.33	<0.020	427
15	10.40	<0.020	335
16	10.38	<0.020	449
17	10.38	<0.020	458
18	10.38	<0.020	468.5
19	10.40	<0.020	477
21	10.60	<0.020	542.5
22	10.58	<0.020	577
23	10.58	<0.020	605
24	10.52	<0.020	652
28	10.47	<0.020	769
29	10.44	<0.020	800
Avg.	10.02		

Table A-2
Experiment No. 2 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	10.40	<0.020	90.1
3	9.78	<0.020	91.7
4	9.53	<0.020	100.2
7	9.17	<0.020	119.35
8	9.23	<0.020	121.65
9	9.21	<0.020	126.4
10	9.27	<0.020	130.7
11	9.35	<0.020	73.2
14	9.04	<0.020	167.5
15	9.13	<0.020	167.4
16	9.20	<0.020	168.1
17	9.27	<0.020	169.5
18	9.27	<0.020	170.5
19	9.53	<0.020	171.7
21	9.44	<0.020	186.7
22	9.36	<0.020	193
23	9.34	<0.020	198.8
24	9.29	<0.020	206
28	9.24	<0.020	229.5
29	9.22	<0.020	235
Avg.	8.92		

Table A-3
Experiment No. 3 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	8.10	0.05	5.83
3	8.54	0.06	7.74
4	9.55	<0.020	54.6
7	9.27	<0.020	66.7
8	9.16	<0.020	73.6
9	9.04	<0.020	82.9
10	9.00	<0.020	81
11	9.06	<0.020	90.9
14	9.04	<0.020	148.6
15	8.83	<0.020	90.4
16	8.88	<0.020	191
17	8.85	<0.020	206.5
18	8.66	<0.020	223
19	9.04	<0.020	236
21	8.97	<0.020	223
22	8.98	<0.020	219
23	8.94	<0.020	219
24	8.98	<0.020	215
28	9.01	<0.020	197.3
29	9.04	<0.020	189.8
Avg.	8.52		

Table A-4
Experiment No. 4 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	11.42	<0.020	167.7
3	10.97	<0.020	268
4	10.88	<0.020	305
5	10.80	<0.020	336
6	10.74	<0.020	366.5
7	10.68	<0.020	397
10	10.53	<0.020	504
11	10.49	<0.020	538
12	10.42	<0.020	586
13	10.40	<0.020	602
14	10.38	<0.020	619
17	10.41	<0.020	665
18	10.51	<0.020	693
24	10.10	<0.020	943
25	10.16	<0.020	981
Avg	9.93		

Table A-5
Experiment No. 5 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	10.77	<0.020	117.3
3	10.26	<0.020	175.7
4	10.29	<0.020	178
5	10.21	<0.020	193
6	10.17	<0.020	173
7	10.16	<0.020	172
10	10.11	<0.020	152
11	10.08	<0.020	139.7
12	10.08	<0.020	136.1
13	9.90	<0.020	272
14	9.87	<0.020	299
17	9.90	<0.020	371
18	9.75	<0.020	383
24	9.41	<0.020	504
25	9.46	<0.020	568
Avg.	9.4		

Table A-6
Experiment No. 6 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	10.23	<0.020	40.3
3	9.33	<0.020	125.2
4	9.19	<0.020	193.5
5	9.14	<0.020	224
6	8.97	<0.020	253
7	8.99	<0.020	273
10	8.84	<0.020	311
11	8.85	<0.020	285
12	9.37	<0.020	292
13	9.34	<0.020	314
14	9.27	<0.020	328
17	9.15	<0.020	354
18	9.09	<0.020	362
24	9.13	<0.020	437
25	9.20	<0.020	494
Avg.	8.63		

Table A-7
Experiment No. 8 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	10.34	<0.020	97.9
2	10.13	<0.020	143.3
3	10.14	<0.020	173.8
4	10.32	<0.020	203
7	10.25	<0.020	284
9	10.14	<0.020	353
10	10.08	<0.020	353
11	10.15	<0.020	367
13	9.78	<0.020	249
14	10.24	<0.020	373
16	10.2	<0.020	394
17	10.08	<0.020	362
18	10.04	<0.020	237
21	9.56	<0.020	124.1
23	9.48	<0.020	55.9
24	9.8	<0.020	166.8
25	9.92	<0.020	79.8
Avg.	9.48		

Table A-8
Experiment No. 9 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	9.86	<0.020	114.4
2	9.45	<0.020	170.1
3	9.2	<0.020	190
4	9.22	<0.020	199.8
7	9.01	<0.020	224
9	8.85	<0.020	249
10	8.85	<0.020	260
11	8.73	<0.020	249
13	9.48	<0.020	334
14	9.95	<0.020	394
16	9.77	<0.020	451
17	9.69	<0.020	490
18	9.58	<0.020	478
21	9.31	<0.020	532
23	9.4	<0.020	570
24	9.43	<0.020	563
25	9.31	<0.020	574
Avg.	8.84		

Table A-9
Experiment No. 10 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	10.32	<0.020	36.9
2	9.6	<0.020	43.5
3	9.74	<0.020	45.1
4	9.02	<0.020	40.2
7	9.335	<0.020	12.19
9	8.52	<0.020	1.74
10	8.1	<0.020	0.18
11	8.11	<0.020	0.54
13	10.29	<0.020	81.6
14	10.47	<0.020	79.3
16	10	<0.020	52.4
17	9.65	<0.020	59.4
18	9.59	<0.020	49.59
21	9.4	<0.020	79
23	9.17	<0.020	69.3
24	9.04	<0.020	57.1
25	9.24	<0.020	44.4
Avg.	8.87		

Table A-10
Experiment No. 12 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	9.13	<0.020	10.14
2	9.97	<0.020	63.8
3	9.87	<0.020	0.21
5	9.52	<0.020	0.09
6	9.41	<0.020	0.17
7	9.22	<0.020	64.7
10	9.1	<0.020	75.2
12	9.41	<0.020	93.1
13	9.2	<0.020	115.2
14	9.01	<0.020	129.7
17	9.5	<0.020	171.1
18	9.26	<0.020	238
19	9.38	<0.020	181.4
20	9.17	<0.020	197.8
Avg.	8.74		

Table A-11
Experiment No. 13 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	9.97	<0.020	84.2
2	9.29	<0.020	56.2
3	9.33	<0.020	57.3
5	9.07	<0.020	58.7
6	9.14	<0.020	61.3
7	9.8	<0.020	93.9
10	9.27	<0.020	75.7
12	9.63	<0.020	111.9
13	9.22	<0.020	100.9
14	9.02	<0.020	99.6
17	9.7	<0.020	103.9
18	9.17	<0.020	91.8
19	9.8	<0.020	121.9
20	9.37	<0.020	36
Avg.	8.79		

Table A-12
Experiment No. 14 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	10.11	<0.020	71.4
2	9.45	<0.020	61.3
3	9.7	<0.020	58.1
5	9.27	<0.020	87.3
6	9.24	<0.020	85.9
7	9.18	<0.020	108.2
10	10.02	<0.020	137.5
12	9.49	<0.020	191.5
13	9.36	<0.020	160.3
14	9.21	<0.020	228
17	9.09	<0.020	219
18	9.06	<0.020	204
19	9.4	<0.020	213
20	9.28	<0.020	214
Avg.	8.79		

Table A-13
Experiment No. 15 Chemistry Data

time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	9.68	<0.020	Not Measured
2	9.90	<0.020	Not Measured
3	9.35	<0.020	Not Measured
5	9.11	<0.020	Not Measured
6	9.26	<0.020	Not Measured
7	9.11	<0.020	Not Measured
10	9.21	<0.020	Not Measured
12	9.26	<0.020	Not Measured
13	9.15	<0.020	Not Measured
14	9.00	<0.020	Not Measured
17	9.31	<0.020	Not Measured
18	9.29	<0.020	Not Measured
19	9.05	<0.020	Not Measured
20	9.36	<0.020	Not Measured
Avg.	8.67		

Table A-14
Experiment No. 16 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	11.1	<0.020	78.0
1	10.26	4.05	100.4
3	10.06	2.65	172.2
5	9.89	1.67	205
7	9.78	1.55	216
11	9.85	1.66	212
12	9.56	1.68	217
13	9.65	<0.020	230
14	9.84	1.40	320
18	9.66	1.23	366
19	9.62	1.37	393
21	9.87	1.55	449
25	8.91	1.58	411
Avg.	9.15		

Table A-15
Experiment No. 17 Chemistry Data

Time (days)	pH	D0 (mg/l)	Conductivity (•s/cm)
0	6.14	5.99	4.32
1	5.56	3.41	7.21
3	5.57	3.87	9.55
5	5.55	3.64	11.28
7	5.58	3.15	13.17
11	5.43	3.06	17.07
12	5.38	3.18	19.66
13	5.84	3.30	20.20
14	5.55	2.98	21.60
18	5.62	3.27	27.40
19	5.76	2.99	28.80
21	5.71	3.91	31.00
25	6.05	4.15	48.40
Avg.	5.67		

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