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SCR Catalyst Mercury Oxidation Laboratory Testing Guideline – Industry Version

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ABSTRACT

Mercury oxidation has recently become an important characteristic of selective catalytic reduction (SCR) catalysts for many end-users. The Electric Power Research Institute (EPRI) has developed a mercury oxidation testing guideline, which 1) aids in the design and specification of appropriate laboratory testing equipment, 2) suggests best-practice operating procedures, 3) aids in the selection of test parameters, and 4) defines quality assurance/quality control measures. EPRI's ultimate goal is to develop a testing protocol for mercury oxidation similar to what is currently in place for NOx reduction and SO_2 conversion. The current guideline represents a first step toward this goal.

Keywords

Selective catalytic reduction (SCR)
Catalyst
Mercury oxidation
Testing protocol
Testing guidelines
Laboratory

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INTRODUCTION

Background

SCR catalysts are known to oxidize elemental mercury in coal-fired flue gases under certain conditions. The rate of oxidation is dependent on the catalyst formulation, as well as many operational parameters including flow rate, catalyst volume, temperature, and the presence of ammonia and other flue gas constituents. Halogens, in particular, may strongly influence the rate of mercury oxidation. Catalyst testing in the laboratory plays a key role in helping researchers understand these sensitivities.

SCR catalysts are routinely tested in the laboratory to determine NOx reduction and SO_2 conversion activities. EPRI and others have developed standard testing protocols which govern this testing.[1] However, these documents do not address SCR mercury oxidation, which is now considered an important catalyst characteristic. SCR owners and operators are interested in using laboratory mercury oxidation tests to compare catalyst performance, track deactivation, and provide a foundation for the prediction of full-scale performance. Appropriate testing methodologies are needed for these purposes, and a common "language" must be developed which allows mercury oxidation to be discussed on the same basis.

To help address the above needs, EPRI has developed a guideline which 1) aids in the design and specification of appropriate laboratory testing equipment, 2) suggests best-practice operating procedures, 3) aids in the selection of test parameters, and 4) defines quality assurance/quality control measures. Ultimately, EPRI's goal is to develop a standard testing protocol for mercury oxidation similar to what is currently in place for NOx reduction and SO_2 conversion. The current guideline represents a first step toward this goal.

Methodology

The methodology for developing this laboratory mercury oxidation testing guideline included: 1) a survey of interested parties and compilation of the results, 2) construction of a semi-bench scale laboratory test facility and parametric testing of representative catalyst samples,[2] 3) development of a draft guideline based on all available information, and 4) a draft guideline review and comment period involving a committee of SCR catalyst vendors, regenerators, test labs and end users. Committee feedback during the draft review process included two key concerns:

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[1] See "Protocol for Laboratory Testing of SCR Catalyst: 2nd Edition," EPRI Technical Update, 1014256, and "VGB Guideline VGB-R302He, 2nd Edition," VGB Technical Association of Large Power Plant Operators.

[2] The details of this effort are reported elsewhere (see: EPRI Technical Update 3002005088, to be issued April, 2015). Information acquired as part of this effort helped to provide guidance with respect to facility construction and operation, equilibrium considerations, parameter sensitivities, accuracy and reporting requirements, sample preparation, and facility maintenance.

- The influences of many operational and gas species parameters (temperature, flow, moisture, O_2, ammonia, halogens and others) are not presently well understood. As a result, the industry's ability to predict full-scale SCR mercury oxidation performance based on laboratory simulations is not yet fully developed.
- Due to slower equilibrium times and the need to include additional key gas components, the costs for performing SCR mercury oxidation tests in the laboratory are significantly higher than standard NOx reduction and SO_2 conversion activity measurements.

It is certain that additional technical clarity and cost efficiency will be gained as more work is performed in this area. In the interim, however, a reference laboratory test approach is presented in this guideline for the comparison of SCR mercury oxidation rates (e.g., between different catalyst types, or new and aged versions of the same catalyst). Based on industry input, the approach is simplified to provide the intrinsic catalyst mercury oxidation rate across a catalyst sample while minimizing testing costs, and does not attempt to directly simulate the complexity of full-scale systems (e.g., ammonia and NOx variations for different layers, SO_3, CO, HBr). It is anticipated that the approach will be modified in future iterations of the guideline as the industry develops additional knowledge.

If a laboratory chooses to perform tests which are not in alignment with this guideline, including apparatus, operating procedures, testing parameters, quality assurance/quality control and reporting, the laboratory must discuss the reasons for any deviations with the end user.

Mechanism of Mercury Oxidation

The detailed mechanism of mercury oxidation, as well as the relationship of various operating parameters to mercury oxidation, is still not fully understood, although the industry has made significant progress over the past several years. A number of species found in coal-fired flue gas may participate in the oxidation of mercury; however, for most coals the predominant <u>native</u> oxidative species is chlorine. Native bromine will also play a role, as well as other native halogens, but the levels of halogens other than chlorine are typically quite low. Bromine is frequently added to coal or flue gas to enhance mercury oxidation, in which case the oxidation of mercury with bromine becomes a primary reaction pathway under these circumstances. The following equation offers a conceptual schematic of the mercury oxidation reaction, showing the reaction of elemental mercury (Hg^0) with an oxidizing species (typically halogens in the case of coal-fired flue gas) to form oxidized mercury (Hg^{2+}).

$$Hg^0 + oxidizing\ species \xrightarrow{catalyst} Hg^2 + reduced\ species$$

In coal-fired flue gases, "oxidized" mercury is not normally present as mercury oxide, as the term would imply, but as mercury halides, such as $HgCl_2$ and $HgBr_2$. Although the catalytic formation of oxidized mercury is probably responsible for the majority of oxidized mercury that is formed downstream of the boiler, gas-phase non-catalytic reactions may also contribute to the overall level of oxidized mercury that is present. The actual *mechanism* of mercury oxidation likely involves complex pathways and intermediate species, with multiple pathways which both form and decompose oxidized mercury species probably taking place in most flue gas environments. A detailed discussion of the reaction mechanism is somewhat academic, however, and outside of the scope of this document.

Factors Affecting Mercury Oxidation

A complicating issue in developing a mercury oxidation testing guideline is that mercury oxidation is potentially sensitive to a large number of parameters, as listed below. All of these parameters must be considered when setting test conditions for laboratory testing. Furthermore, the selection of testing parameters will be influenced by the purpose of testing, such as performance tracking, field predictions, or comparative behavior.

- Catalyst Design
- Catalyst Volume
- Catalyst Age
- Flow Rate
- Temperature
- Ammonia Profile
- Halogen Level (HCl, HBr)
- Flue Gas Constituents (Hg^0, Hg^{2+}, SO_2, CO, NOx, O_2, H_2O, etc.)

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DEFINITIONS AND CONVENTIONS

Introduction

There are a number of parameters and conventions associated with SCR technology and catalyst testing that warrant consideration. It is important that these parameters be defined carefully and that a common reporting convention is utilized. The following sub-sections specify various definitions and reporting conventions to be utilized in relation to mercury oxidation testing. Note that these conventions generally follow those set forth in the EPRI SCR catalyst testing protocol.[3]

Standard Conditions

A number of SCR parameters are expressed in terms of flow rates measured at standard conditions. There is no single convention as to standard temperature and pressure utilized in the United States. As a result, it is important that the standard temperature and pressure basis for any data references be reported. Table 2-1 shows the standard temperature and pressure as defined by this guideline in both English and S.I. units.

Table 2-1
Standard Temperature and Pressure Conditions as Defined for the Guideline

Convention	Temperature	Pressure
English	32°F	1 atm.
S.I.	273.15 K (0°C)	1.01325×10^5 Pa

Specific Geometric Surface Area

The specific geometric surface area (A_{gs}) is the total catalyst surface area[4] per unit volume of catalyst. It is a "specific" parameter since it is expressed as area per unit volume of catalyst (m^2/m^3). The parameter is a fundamental property of the catalyst and is determined by the pitch and general geometry of the catalyst, including wall thickness and length.

Space Velocity

The space velocity is defined as the flue gas volumetric flow rate (expressed at standard conditions) divided by the overall catalyst volume. Typically this is expressed in units of inverse hours (i.e., 1/hr or hr^{-1}).

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[3] See the previously referenced EPRI SCR catalyst testing protocol.

[4] This is the geometric surface area presented to the gas, not the internal surface area, as typically measured by BET.

$$SV = \frac{Q_{fg}}{V_{cat}}$$

Where; SV = space velocity *(hr^{-1})*
Q_{fq} = flue gas volumetric flow rate *(m^3/h, STP)*
V_{cat} = catalyst volume *(m^3)*

Area Velocity

The area velocity (AV) is the flue gas flow rate (expressed at standard conditions) divided by the total catalyst geometric surface area, or more simply, the space velocity divided by the specific geometric surface area of the catalyst. This quantity is usually expressed in units of m/hr.

$$AV = \frac{Q_{fg}}{A_{cat}} = \frac{SV}{A_{gs}}$$

Where; AV = area velocity *(m/hr)*
Q_{fq} = flue gas volumetric flow rate *(m^3/h, STP)*
A_{cat} = total surface area of catalyst (m^2) $= V_{CAT} * A_{gs}$
SV = space velocity *(hr^{-1})*
A_{gs} = specific geometric surface area (m^2/m^3)

Linear Velocity

The linear velocity (LV) is defined as the flue gas velocity within the catalyst channels, and is expressed as the flue gas volumetric flow rate (at standard conditions) divided by the open face area of the catalyst. The parameter is defined as follows and is typically converted to units of m/s.

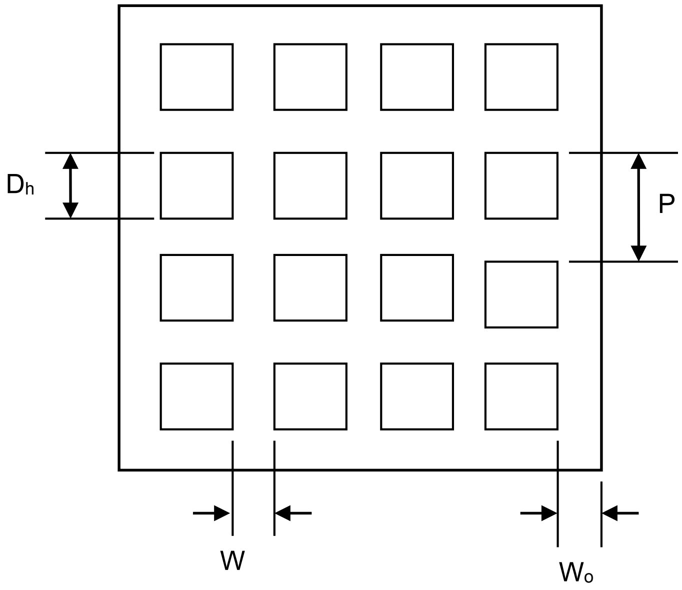
$$LV = \frac{Q_{fg}}{A_{open}}$$

Where; LV = linear velocity *(m/hr, typically converted to m/s)*
Q_{fq} = flue gas volumetric flow rate *(m^3/h, STP)*
A_{open} = total open area of catalyst face *(m^2)*

Catalyst Pitch

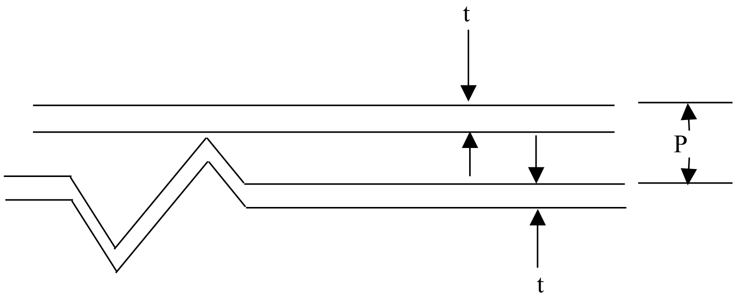
The determination of catalyst pitch depends on the catalyst type, as follows:

Honeycomb



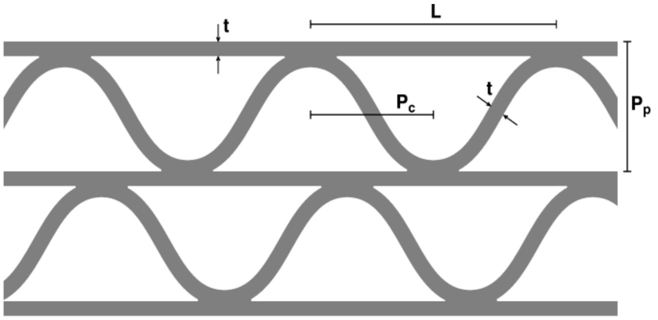
P = pitch, mm
W = inside wall thickness, mm
W_o = outside wall thickness, mm
D_h = hydraulic diameter, mm

Plate



t = plate thickness, mm
P = pitch, mm

Corrugated



Pp = is the plate pitch, mm
Pc = is the corrugated pitch, mm
t = is the wall thickness, mm
L = is the wave length, mm

Catalyst Condition

Catalyst samples that are subjected to laboratory analysis will have varied histories. These histories, in turn, will have some impact on how they are handled in the laboratory. Specifically, their history will impact conditioning times of the sample prior to testing. For the purpose of this guideline the following definitions are used:

a.	New Catalyst:	Catalyst that has been received from the manufacturer and has not been previously exposed to flue gas.
b.	Used Catalyst:	Catalyst that has been used in a SCR system – samples of which have been removed from the reactor and sent to the laboratory for testing without any processing.
c.	Dry Cleaned Catalyst:	Catalyst that has been cleaned of ash and deposits with a dry process (e.g., brushing, compressed air, etc.). This catalyst can be treated as Used Catalyst defined above.
d.	Washed or Wet Chemical Treated Catalyst:	Catalyst which was subjected to a water-based wet washing solution for the removal of physical restrictions, such as fly ash pluggage, large particle ash (LPA), or surface blinding layers.
e.	Rejuvenated Catalyst:	Catalyst which was washed and wet cleaned, as well as subjected to a water-based wet rejuvenation solution for the removal of chemical catalyst poisons, such as arsenic (As), phosphorus (P), potassium (K), sodium (Na), etc.
f.	Regenerated Catalyst:	Catalyst which has been treated by means of impregnating active ingredients (such as Mo, V, and W) in order to restore the catalyst's activity.

Note: Any catalyst sample that has undergone a wet process (d, e, and f) should be treated as a new catalyst in the context of laboratory testing when considering initial conditioning and stabilization times.

NOx Reduction

NOx reduction will be designated either as an absolute level of NOx reduction (i.e., NOx_{in} – NOx_{out}) in ppm, or as a percentage NOx reduction. The absolute NOx reduction is denoted by ΔNOx, and the percentage NOx reduction by ηNOx, according to the following equation.

$$\eta NOx\ (\%) = 100\% * \frac{(NOx_{in} - NOx_{out})}{NOx_{in}} = 100\% * \left(\frac{\Delta NOx}{NOx_{in}}\right)$$

Note that both NOx_{in} *and* NOx_{out} *must be expressed on a common gas-phase concentration basis.*

DeNOx Activity

The deNOx activity is designated as K (sometimes K_{NOx}) and is expressed in units of m/hr. The calculation includes AV, which is based on flow rates expressed at standard conditions (0°C, 1 atm.). The equation for computing the deNOx activity from the laboratory test follows.

$$K = -AV * \ln\left(1 - \frac{\eta NOx(\%)}{100}\right)$$

DeNOx Potential

The deNOx potential characterizes the performance of an SCR catalyst in terms of its ability to reduce NOx. The parameter is often used in catalyst management and process modeling as a convenient representation of the available “potential” to remove NOx. The quantity has no units and is calculated by the following equation.

$$DeNOx\ Potential = P_{NOx} = \frac{K}{AV}$$

The deNOx potential for each layer of catalyst in an SCR reactor can be summed to determine the deNOx potential for the reactor as a whole. If a portion of the catalyst is blocked due to the accumulation of fly ash, then the following equation can be used to determine the effective potential based on P_{clean}, which is the potential determined using K-values and area velocities for clean catalyst.

$$Effective\ P_{NOx} = (1 - B)P_{clean}$$

Where: B = fraction of the catalyst that is experiencing blockage.

Mercury Speciation

Mercury speciation, as defined in this guideline, is shown below.

- Hg^0 (elemental mercury)
- Hg^{2+} (oxidized mercury)
- Hg^T (total mercury)

Actual concentrations of the above mercury species are expressed in units of µg/m^3, at standard conditions. The standard flue gas basis for reporting mercury concentrations is dry at 3% O_2. In cases where it is meaningful to present mercury data at actual conditions, the basis of the data must be clearly stated.

Mercury Oxidation Rate

The mercury oxidation rate for test measurements is reported as the proportion of available elemental mercury entering the catalyst that is oxidized by the catalyst. This parameter may be calculated in multiple ways, as follows. The guideline does not currently specify which calculation methodology must be utilized, since this depends to some degree on how mercury is measured. Various calculation methods may provide better or worse repeatability, depending on the test apparatus and measurement methods, and various laboratories will typically develop an internal preference for the oxidation rate calculation. Under most circumstances, if the facility meets the performance requirements specified within this guideline, the mercury oxidation rate will be comparable regardless of which calculation method is used.

Elemental Mercury Basis

The equation below is based on the measurement of elemental mercury only, and implies that theoretically, for any particular test, only elemental mercury measurements are required to determine the mercury oxidation rate. However, speciated mercury measurements, as well as total mercury measurements would be required to meet the requirements for facility performance. The approach implies that any differences in the elemental mercury between the inlet and outlet measurement points are attributable to mercury oxidation by the catalyst. As a result, any mercury that is “lost” through the system will be attributed to mercury oxidation. System design, validation, and QA/QC procedures ensure that oxidation by the system itself, or loss in mercury, is minimal and therefore the change in elemental mercury may indeed be attributed to mercury oxidation by the catalyst. Note that the mercury oxidation rate must not be confused with the % oxidized mercury (% Hg^{2+}) at the catalyst outlet, which is simply the proportion of oxidized mercury compared to total mercury as measured at the catalyst outlet.

$$Hg^0 \text{ Oxidation Rate } = \frac{Hg^0_{in} - Hg^0_{out}}{Hg^0_{in}}$$

Note that the above mercury oxidation rate may be expressed as either a percentage or as a fraction, and that Hg^0_{in} and Hg^0_{out} are expressed on a common gas-phase concentration basis (i.e., wet/dry, O_2 basis, etc.).

Oxidized Mercury Basis

The following equation calculates mercury oxidation rate more directly than the previous equation, and requires that the mercury speciation be measured directly at both the inlet and outlet of the catalyst. Here the difference in the oxidized mercury is determined by subtraction, and divided by the inlet elemental mercury to determine the mercury oxidation rate. In this case, a loss in elemental mercury across the system would not be attributed to mercury oxidation.

$$Hg^0 \text{ Oxidation Rate } = \frac{Hg^{2+}_{out} - Hg^{2+}_{in}}{Hg^0_{in}}$$

Normalized Basis

The following equation essentially normalizes the inlet and outlet total mercury values by utilizing relative speciation at the inlet and outlet to calculate the mercury oxidation rate. The absolute levels of mercury are not required, only the relative amounts of oxidized and elemental mercury. This approach tends to correct data for which absolute mercury values are skewed, but the relative proportion of oxidized to elemental mercury is considered accurate. It requires that the relative speciation be known at both the catalyst inlet and outlet. The equation can also be written in a number of alternate mathematically equivalent ways since $\%Hg^0 = 100\% - \%Hg^{2+}$.

$$Hg^0 \text{ Oxidation Rate } = \frac{\%Hg_{out}^{2+} - \%Hg_{in}^{2+}}{\%Hg_{in}^{0}}$$

Where $\%Hg_{in}^{2+}$ and $\%Hg_{out}^{2+}$ refer to the proportion of oxidized mercury compared to total at the inlet and outlet, respectively, expressed as a percentage, while $\%Hg_{in}^{0}$ refers to the proportion of elemental mercury at the inlet.

SO_2 to SO_3 Conversion or Oxidation

Similar to the conversion for NOx, the absolute change in SO_2 or SO_3 across the catalyst is designated as ΔSO_2 or ΔSO_3, and is expressed on a gas-phase concentration basis.

$$\Delta SO_2 = SO_2 in - SO_2 out$$

$$\Delta SO_3 = SO_3 in - SO_3 out$$

The percentage oxidation (conversion) of SO_2 to SO_3 is expressed in the first equation below. Although the percentage oxidation can be calculated using the difference in SO_2 concentration (as in the second equation below), this method is difficult due to the accuracy required in the SO_2 measurement. As a result, in most instances, the SO_2 conversion is calculated using the first equation below, which relies on the direct measurement of the SO_3 concentration.

$$\eta SO_3 = 100\% * \left(\frac{\Delta SO_3}{SO_{2\,in}}\right)$$

or

$$\eta SO_3 = 100\% * \left(\frac{\Delta SO_2}{SO_{2\,in}}\right)$$

NH_3/NOx Molar Ratio

The molar ratio (α) of NH_3 to NOx at the SCR inlet is designated as a dimensionless quantity as shown below, where both NH_{3in} and NOx_{in} are expressed on a common gas-phase concentration basis. The molar ratio at any other location is defined similarly, where both the NH_3 and NOx concentrations correspond to the same location.

$$\alpha = \frac{NH_{3\,in}}{NOx_{in}}$$

Apparatus Scale

For the purposes of this document, apparatus scales are defined below. Note that “full-length” honeycomb samples refer to honeycomb element lengths as utilized in the field, which are usually on the order of 1,000 mm in length. For plate samples, however, “full-length” usually refers to a single plate length. A typical plate catalyst layer will have two sub-layers, or twice this length total. For mercury oxidation testing, if simulation of a complete reactor layer is desired, the sample length must be twice that of a single plate. This will reproduce the configuration of the full layer as installed in the field. This is required to simulate the ammonia profile for the layer as it would operate in the field (if ammonia is included in the test).

Ultimately, the sample length selected for the laboratory testing will depend on the purposes of testing, test conditions, and the laboratory itself.

Micro – This is the smallest scale of apparatus utilized for laboratory testing. By definition in this guideline, this apparatus does not have the capability of operating with full-length catalyst samples. As a result, it does not simultaneously maintain both AV and LV values consistent with the field. However, it is often operated at either an AV consistent with the field or an LV consistent with the field, depending on the test mode that is desired. Also, if the sample is cut from a deactivated catalyst, it will only represent a portion of the full-length deactivation profile. The performance of the sample may depend on where it is cut (e.g., leading edge or trailing edge). The micro-reactor physical set-up deviates substantially from that of the field, and care must be taken in the evaluation of the data.

Semi-Bench – This is the intermediate scale of apparatus for laboratory testing. By definition in this guideline, this apparatus operates with full-length honeycomb or plate samples (two plate lengths), but with cross-sections smaller than standard catalyst elements (i.e., less than 150mm x 150mm cross-section). Semi-bench facilities have the ability to simultaneously operate at AV and LV values consistent with the field.

Bench – This is the largest scale of apparatus utilized for laboratory testing. By definition in this guideline, this apparatus operates with full-length, standard honeycomb elements or plate samples (two plate lengths) of equivalent cross-sectional area (150mm x 150mm). Both AV and LV values are typically maintained consistent with the field. This apparatus may be able to accommodate multiple catalyst layers in series, thereby directly simulating multi-layer full-scale reactors.

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TEST APPARATUS

The following discussions outline general test apparatus design parameters in terms of scale, materials of construction, flue gas generation, etc.

Apparatus Scale Summary

Apparatus scale definitions and characteristics were addressed in Section 2, and are summarized in Table 3-1. Bench, semi-bench and micro reactors may be used to perform reference tests, as described in Section 5.

Design of Apparatus Components

The following discussions highlight issues associated with the primary components of the test apparatus. With the exception of mercury introduction, the requirements for these components for mercury oxidation testing are nearly identical to those for deNOx and SO_2 conversion. Note that materials of construction and flue gas monitoring/analytical systems are addressed separately in subsequent report sections.

Synthetic Flue Gas Generation

The general approach to the generation of flue gas for the test reactor will be a function of test apparatus scale. Bench-scale apparatus will typically use some type of combustion system to generate the bulk of the flue gas (primarily N_2, CO_2, and O_2). The primary components may be trimmed (fine-tuned) using bottled gases. Additional flue gas components will be added using bottled gases, including: CO, NOx, and NH_3. Laboratory combustion systems typically utilize light hydrocarbons, such as natural gas or propane. This usually results in an overabundance of moisture in the generated flue gas (as compared to coal-fired systems) due to the high hydrogen content of the fuel. As a result, it will most likely be necessary to reduce the moisture content of the combustion gas by some means. This is often done with current bench-scale systems that are utilized for deNOx and SO_2 conversion testing, but not always. Given that under some conditions there may be a significant mercury oxidation dependency on moisture, it is envisioned that most applicable bench-scale systems will have the capability to independently adjust moisture levels.

Depending on the exact scale, semi-bench devices may also use small combustion systems, as previously mentioned, but may also use bottled gases, or liquefied gases (particularly nitrogen), which are subsequently vaporized. Micro-reactors typically will use bottled gases, although nitrogen may come from a variety of sources, including liquid nitrogen, especially if it is used for other purposes at the laboratory.

Table 3-1
Summary of Test Facility Characteristics According to Scale

Characteristic	Micro-Scale	Semi-Bench Scale	Bench-Scale
General Facility Size	Usually a small footprint, suitable for laboratory placement, possibly within a hood system or cabinet.	Moderately sized footprint, suitable for laboratory placement, often with various sub-systems distributed to different areas of the laboratory.	Relatively large footprint, equipment usually considered small industrial or “pilot” sized. Includes combustion systems which add to complexity and size.
Simulated Flue Gas Source	Bottled/liquefied gases	Typically bottled/liquefied gases. Some systems may utilize very small combustion systems.	Combustion system trimmed/augmented with bottled/liquefied gases.
Capital Cost	Lowest	Moderate, typically strikes a balance between testing capabilities and cost of facility.	Highest, requires substantial capital and long-term commitment
Operational Cost	Lowest	Moderate, typically strikes a balance between adequacy of simulating full-scale conditions and test cost.	Highest, requires significant man-power and utilities, resulting in relatively high per-hour operational costs.
Velocity Capability	Cannot achieve both field LV and AV simultaneously.	Can achieve both field LV and AV simultaneously.	Can achieve both field LV and AV simultaneously.
Sample Size	Small cross-section (less than 150mm x 150mm), shorter sample length than full length field layer.	Small cross-section (less than 150mm x 150mm), sample equivalent in length to field layer.	Cross-section equivalent to full honeycomb element (150mm x 150mm), length equivalent to field layer.
Reactor Construction Materials	Both glass and steel possible, glass often utilized. Glass-coated steel may be used.	Primarily steel, some glass components possible. Glass-coated steel may be used.	Steel only, glass-coating may be possible in some critical areas.
General Notes	Small size allows for very economical capital and operational cost, but inability to match both AV and LV in the field simultaneously causes conditions to deviate substantially from that of a field layer. Data must be evaluated with limitations in mind.	Strikes a balance between testing costs and adequacy of the simulation. Full-length samples allow direct simulation of a full field layer.	Full-length samples allow direct simulation of a full layer, but higher costs compared to semi-bench may significantly impact economics. Facilities already operating for deNOx testing may be able to add mercury testing economically, however.

Gas Metering and Composition

The exact gas metering devices utilized for the test apparatus will depend on the scale of the facility, the particular design of the facility, the gases that are being metered, and the preferences of the laboratory. For bench-scale systems, where the bulk of the flue gas is generated using a combustion system, the primary gas flow will probably be measured using a venturi meter or calibrated orifice plate. Supplemental gases will likely be metered using mass flow controllers. Smaller systems will most often use mass flow controllers for all of the gases, although some semi-bench devices may have primary gas flows that are measured in a manner similar to that for bench-scale devices. No specific methodology for gas metering is specified for the guideline as long as the gases are measured within the required accuracy for the tests, assuming that the flow rates have been verified through actual measurements using standard methodologies.

Clearly, the simulated flue gas composition will have a direct effect on mercury oxidation. The facility must have the ability to accurately set the reactor inlet flue gas composition, and to verify that composition. Further, the system must be able to hold those conditions over extended periods of time (i.e., avoid drift). Since SCR catalysts often require substantial time to fully establish mercury oxidation equilibrium under the specific test conditions, periodic fluctuations in flue gas speciation during a test or during conditioning periods may greatly hinder the ability to obtain accurate data. Systems will operate over-night, for example, to allow equilibrium to be attained, with or without supervision. As a result, a robust system for the control of flue gas composition is needed for an accurate and efficient test facility. In many cases, bottled gases may be used for flue gas make-up. In these cases, ganged bottles which allow for steady gas supply during periods of bottle change-out are particularly helpful. Flue gas composition is typically measured using a variety of analytical devices. FTIR[5] systems are popular due to their ability to measure a multitude of flue gas components simultaneously, and for their relative ease in operation. Various other measurement techniques are available for specific species, such as chemiluminescence for NOx, zirconia cell for oxygen, UV fluorescence for SO_2, and controlled condensation for SO_3. The selection of the overall monitoring system(s) and its specific components will be very much facility-specific depending on the experience and preferences of the laboratory. Accurate mercury measurements are of course critical to the test facility. Mercury doping and speciation measurements are of course a primary concern, and are discussed in a separate section, below.

Flue Gas Mixing

Ensuring that the flue gas is well-mixed prior to contact with the catalyst is crucial to obtaining accurate and repeatable data. The requirements for mixing will depend largely on the apparatus scale and the design of the particular facility. In-line static mixers generally offer the most convenient method of gas mixing, although facilities with long tubing runs with respect to the tubing diameter may not require dedicated mixing devices. Large bench-scale facilities are more likely to require separate mixing devices. Due to the sensitivity of mercury oxidation to virtually all flue gas components, mixing must be ensured after all components have been introduced into the flue gas. Flow modeling is recommended for all facilities to ensure adequate mixing; unless through other calculation means the design parameters clearly indicate that the flue gas will be

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[5] Fourier Transform Infrared Spectroscopy.

fully mixed prior to contacting the catalyst. Mixing verification is also recommended, if possible, as part of the commissioning of any test facility.

System Temperature Control and Gas Preheat

Due to the small size of many laboratory reactor systems, heat loss can be problematic. As a result, a variety of heat sources may be applied throughout the system. The test reactor chambers themselves will generally include heating capability of some sort. Care must be taken to ensure that the catalyst is evenly heated, avoiding hot or cold spots, and that the actual temperature at which the catalyst is operating is well-quantified. Typical thermocouple locations may in include the following; 1) immediately prior to the catalyst inlet, 2) sample intermediate locations (if the sample is divided), 3) reactor outer wall temperatures in various locations, and 4) immediately after the catalyst exit.

Reactor chamber heating capability alone is not typically sufficient for the system as a whole, and some type of gas preheat is therefore used. This helps to ensure that no catalyst temperature inconsistencies occur and better simulates the actual operation of full-scale SCRs. Preheating may be accomplished in a variety of ways, depending on the design of the facility and scale. Large bench-scale facilities may use heat exchangers and supplemental electric heaters to control the flue gas temperature entering the catalyst test chamber. Smaller systems may utilize simpler systems, such as heating tape, etc., applied to the piping/tubing. In any event, gas preheating must take into consideration accuracy, materials of construction (to avoid unwanted side reactions), and flexibility (to ensure that all required temperatures can be easily met). Further, as will be discussed in more detail in subsequent report sections, the facility must be validated to make certain inherent system mercury oxidation is minimized.

Measurement and Control of Mercury

Mercury-related factors critical to accurate testing include: 1) the ability to add mercury to the simulated flue gas consistently, accurately and in known concentration, and 2) the ability to confidently measure mercury concentrations at the reactor inlet and outlet, including total and speciated mercury. For mercury oxidation testing, mercury must be added to the flue gas upstream of the catalyst. Both elemental and oxidized mercury may be introduced by a variety of means. These include permeation tubes, dilution of saturated mercury-containing gases, and vaporization of mercury-containing solutions. Many of these systems were developed commercially as methods for spiking flue gas for various purposes, most often associated with monitor calibrations, etc. The selected methodology must be verifiable in terms of absolute concentrations and long-term repeatability using independent speciated mercury analyses.

Various approaches are available for measuring speciated mercury in a test facility. Several of these are mentioned in general below, each having its own pros and cons. However, detailed descriptions and comparisons of different types of mercury test equipment and instrumentation (e.g., dry continuous monitors (CEMS), wet semi-continuous monitor (SCEMS), speciated sorbent traps, etc.) are outside the scope of the current guideline. Specific laboratories must make their own determination as to the optimal approach given their testing needs, availability of equipment, and familiarity and confidence with measurement methodologies. It should be noted that automated systems which provide near continuous data at the reactor outlet are extremely valuable in identifying equilibrium trends, dynamic responses to process variables, and process upsets. Continuous outlet data lends itself well to the procedures described subsequently for establishing that equilibrium has been reached for a particular test, or that a catalyst sample has been sufficiency conditioned prior to testing. In all cases, robust QA/QC procedures must be in place to verify the accuracy of the mercury measurement systems for both total mercury and relative speciation. The procedures differ according to monitor type and specific design, but in general various calibration and spike/recovery systems are utilized as part of the long-term mercury measurement QA/QC plan.

Dedicated Continuous Inlet and Outlet Measurements

Under this approach, a dedicated continuous or semi-continuous mercury monitoring system is located at both the reactor inlet and outlet. This is a highly rigorous approach and assumes that the mercury measurement system provides mercury speciation data near real-time. This would be consistent with dry measurement systems that provide near-continuous speciated mercury data, or wet systems with automated switching that provides speciation data frequently. One of the concerns of this approach is that since two separate monitors are used, any bias between the two measurement systems would be seen as a catalyst response.

Single Continuous Monitor with Automated Inlet/Outlet Switching

An alternate approach to that above, is to use a single continuous monitor with automated switching to periodically sample gas from both the reactor inlet and the outlet. This approach would still give data in a relatively continuous fashion, but alleviate the need for two separate monitoring systems as well as alleviate the concern over monitor bias.

Dedicated Continuous Outlet with Intermittent Inlet Measurement

Under this scenario a continuous monitor would be dedicated primarily to the reactor outlet, but inlet speciation measurements would be made intermittently to verify the inlet speciation and mercury concentration. If it has been established that the inlet speciation is constant, and that the mercury injection system is capable of maintaining a constant supply of mercury, then this approach may work very well, since it still provides continuous or near-continuous outlet data. The inlet measurement may be made by a valving or piping change such that the outlet monitor is utilized for the inlet measurement, or a separate system of some type may be used for the inlet mercury verification. In any event, inlet mercury data are acquired only intermittently.

Manual Measurement System

Under this general scenario, a very basic mercury measurement system is employed, such as a wet impinger or speciated sorbent trap system where non-continuous speciated mercury measurements are made. One or more systems can be used with this approach, but in general the inlet and outlet mercury are measured intermittently to verify concentration and speciation, with the understanding that the test rig has been validated and shows little drift at the inlet in either speciation or total concentration.

Reactor Design

Although the reactor itself may represent only a small portion of the total physical equipment present, its design is critical to the overall operation the facility. The reactor design must 1) ensure the materials of construction are relatively inert, avoiding any inherent mercury oxidation or other reactions that may skew the test results, 2) be durable to withstand repeated assembly/disassembly for catalyst loading, and 3) be capable of holding catalyst samples in the appropriate geometrical configuration without flue-gas bypass. Typically, commercial test facilities will be designed to accept all types of catalyst samples (plate, honeycomb, and corrugated). This may be done using a single general reactor, or separate reactors designed specifically for each catalyst type. Semi-bench reactors are often quite long and narrow, and multiple sections may be used to facilitate catalyst sample loading. This type of multi-section reactor also facilitates intermediate temperature monitoring. Reactors often incorporate some type of transition from relatively small diameter piping/tubing to a larger round or square section which constitutes the reactor itself. Any transitions must be done in a manner that ensures even flue gas flow at the catalyst face and accommodates any change in materials that might be present between the reactor and the piping. For micro-reactors, where glass reactors are often used, the transition from metal tubing to the glass reactor section is an important design consideration.

Wetted components must minimize unwanted reactions that potentially confound the data. This is paramount to developing a suitable test apparatus. Glass or specialized metal alloys may be utilized to help minimize mercury oxidation through the system, or avoid other side reactions that might skew the data (such as deNOx, SO_2 conversion, etc.). Construction materials must also be consistent with required duties (i.e., high-temperature alloys in high-temperature applications, or non-corrosive alloys in areas where corrosive gases may be present, etc.). Large bench-scale systems will typically use metal alloys for nearly all of the large piping components, although some components associated with individual gas introduction, such as SO_3, halogens, or mercury, may be at least partly constructed of glass or polymeric materials. Smaller systems may more readily take advantage of the inert nature of glass or ceramic materials, especially in the case of micro-reactors where glass reactors and glass tubing are often utilized. In many cases, glass-coated metals may provide a balance between durability and inertness. No specific materials are required by this guideline; however, system validation testing (without catalyst present) must be performed to determine the inherent behavior of the system, as described in Section 5. Overall, systems which minimize surface area, maximize the use of non-reactive materials such as glass or glass coatings, and avoid cold-spots will generally fair best in terms of avoiding unwanted side reactions.

Exhaust Gas Measurement and Venting

Downstream of the reactor, flue gas compositional measurements are typically performed, which include mercury, as discussed above, and other constituents that may have changed across the reactor, such as SO_3, NOx, and NH_3. A total flow measurement may also be made. Subsequently, the flue gas may pass through a number of components, including various conditioning systems (to remove acid gases, mercury, NH_3, etc.), vacuum pumps, and coolers, before venting. Small laboratory systems may simply vent to an existing laboratory hood, while larger permanent systems may have dedicated vents. In many cases, the use of mercury may trigger regulatory requirements, so the final flue gas conditioning and venting process may be dictated to some degree by the permit requirements.

Apparatus Commissioning and Validation

The commissioning and validation of the laboratory apparatus includes several functions, as listed below. These validation efforts will be an integral part of the QA/QC plan and may be repeated periodically throughout the life of the facility. In addition to these validation steps, a complete understanding of the behavior of the mercury injection and measurement systems is critical. For most facilities, a high proportion of the general operational effort will be devoted to these mercury-related systems, and they will be a focus of the QA/QC plan. The documentation of the apparatus validation efforts must be available to laboratory customers.

- Verification of flue gas flow rates and flow measurement device accuracy
- Verification of ability to measure and control flue gas composition
- Verification of ability to measure and control mercury, including closing material balance
- Verification of ability to maintain desired temperatures throughout the system
- Blank reactor tests to establish inherent system behavior
- Facility conditioning and break-in period

Verification of Flue Gas Flow Rates

The actual flue gas flow rate through the test reactor is of course a fundamental parameter affecting mercury oxidation across the catalyst. In most cases, some specific flow target, usually expressed as an area velocity or linear velocity will be required. Sufficient accuracy must be present in the flow measurement devices so that the actual flow through the test reactor is known with confidence. Various flow devices may be used, such as mass flow controllers, rotameters, or orifice meters, often with some redundancy. In other words, individual flue gas components may be metered separately, at known flow rates, allowing for these flows to be summed to compute a total flow rate. The total flow rate may also be measured directly. Thus two independent flow measurements may be present. Further, reactor exit flow measurements may also be present, potentially providing additional flow data. In any event, the facility commissioning must include a verification of flow rate by some independent method, and be repeated periodically as part of a QA/QC plan.

Verification of Flue Gas Composition

Depending on the method of gas metering, some indication as to the expected flue gas composition will typically be available, with varying degrees of perceived accuracy. Mass flow meters, in particular, may be reasonably accurate in predicting the gas-phase concentration of various constituents. However, it is important that this be independently verified, usually through direct gas-phase measurements of the flue gas species. This may be done periodically as part of a QA/QC plan, or done continuously during testing using a gas monitoring system. The flue gas composition is typically verified at the reactor inlet since some species, such as ammonia, NOx, and SO_3, will change in concentration substantially across the SCR catalyst. For non-reactive species, however, verification of their concentrations can be done at the reactor exit if so desired. For tests that require both ammonia and NOx, it may be appropriate to determine the deNOx activity value under the test conditions, in which case NOx and/or ammonia must be measured at both the inlet and outlet of the reactor.

Verification of Speciated Mercury Concentrations

As part of facility commissioning, mercury concentrations in the simulated flue gas must be measured to ensure the appropriate amount of mercury is being injected and the speciation is not strongly partitioned to the oxidized phase (assuming only elemental mercury is being utilized). If both elemental and oxidized mercury are being introduced, inlet speciation measurements must be used to verify the appropriate speciation is being obtained. Assuming only elemental mercury is being introduced, high levels of oxidized mercury imply that some component of the system is oxidizing mercury, which can lead to skewed data. The system's ability to maintain a mercury concentration set point for the duration of a particular test must also be validated. Inlet concentrations and speciation will typically be confirmed periodically, or in some cases continuously, during routine testing. Total mercury concentrations at the reactor outlet help to close the mercury material balance and confirm that no adsorption/desorption of mercury is occurring across the catalyst, or that there is no bias between the reactor inlet and outlet mercury measurements. The actual speciation of mercury at the reactor outlet is of course the principal dependent parameter measured for the test, and by calculating the proportion of elemental mercury that is oxidized, the mercury oxidation rate can be calculated. Steady-state operation of the catalyst sample with respect to mercury oxidation is of course critical to the data quality – this will be discussed in more detail below.

Verification of Temperature

The verification of temperature is typically straightforward, assuming the facility has been designed with sufficient thermocouple locations. Since flow rate affects the thermal mass throughput of the system, substantial changes in operating flow rate may affect temperature behavior. Temperature must be verified for each test and include at a minimum the reactor inlet and exit flue gas temperatures. Other temperature validations may be required, such as gas preheat temperature, or flue gas sample temperatures. Temperature measurement around flow control and measurement devices may also be necessary to accurately compute flow rates on a standard basis.

Blank Reactor Testing

Empty or "blank" reactor testing (as described in Section 5) is a fundamental part of facility commissioning and validation. For these tests, the system is operated at one or more conditions commensurate with planned testing conditions, but without any catalyst in place, using an empty reactor. These tests help to establish the inherent rate of mercury oxidation that occurs through the system. They also provide a convenient condition for verifying the inlet/outlet mercury balance, since with no catalyst present the potential for adsorption/loss of mercury across the reactor is minimized. Typically, the commissioning of a facility will begin with these blank reactor tests, where flue gas metering, compositional measurement, flow control, etc. can all be verified. These tests must be repeated when major equipment modifications are made, and each reactor utilized in the test facility must be characterized.

Facility Conditioning and Break-In Period

Initial results from the blank reactor tests described above may show unusual behavior, such as high levels of inherent mercury oxidation, or significant deNOx (if ammonia and NOx are included in the simulated flue gas). These phenomena may be ameliorated as system surfaces become passivated, so it is beneficial to operate a new facility for a week or more to help condition the facility. This often occurs simultaneously with various start-up and check-out activities. This conditioning period also helps in terms of facility troubleshooting and break-in, since it allows operational time to ensure all system components are operating as designed.

4

EQUILIBRIUM CONSIDERATIONS AND REPORTABLE DATA

Sample Equilibration Time

Ensuring that catalyst samples have reached steady state mercury oxidation conditions (i.e., equilibrium) is critical to the validity of the generated data. Data which are acquired prior to the establishment of equilibrium are transient and will lead to erroneous interpretation of the results. In many cases, the time required to reach equilibrium for mercury oxidation is much longer than historically required for deNOx and SO_2 conversion testing. As a result, a catalyst may be assumed to have reached equilibrium, when in fact it is simply moving towards equilibrium very slowly. This behavior may be difficult to recognize, so care must be taken to ensure definitively that a sample has indeed reached equilibrium.

The exact time for any particular catalyst sample to achieve equilibrium for mercury oxidation will be variable, depending on such factors as catalyst design, age of the sample (fresh samples typically require longer stabilization times than exposed samples), and to some degree equipment design, since the system flow rates and sample sizes may impact stabilization. It is difficult to provide definitive required equilibrium times at present, due to the many influential factors. Instead, appropriate equilibrium times must be determined on a case-by-case basis by monitoring with repetitive or continuous measurements. Each laboratory will develop internal guidelines for determining when stable operation has been reached.

The initial equilibrium time for a new catalyst sample (often referred to as “conditioning” time) may be particularly lengthy, and much longer than would be required for a used sample. Further, the time required for a sample to stabilize for a change in test conditions will differ depending on if it is a “shut down / start up” cycle (i.e., the system has shut down and restarted as a scheduled event or as an upset), or a simple intentional condition change (e.g., temperature shift) during operation. Equilibrium times will of course also be affected by the degree to which any one parameter is changed, as well as which parameter is changed (i.e., a change in temperature may require more equilibrium time than a change in O_2, for instance).

Procedure for Establishing that Equilibrium Has Been Reached

In general, establishing equilibrium requires mercury speciation measurements be made periodically until no trending is present in the data. Table 4-1 gives approximate equilibrium times and recommended frequency of measurements. The table makes a distinction as to the catalyst type (new, freshly regenerated, aged, etc.) and to the test type (i.e., initial conditioning/testing or a change in testing conditions). ***These are general guidelines. Each laboratory will need to establish definitively that equilibrium has been reached for each test performed.***

Table 4-1 assumes continuous, 24-hour operation of the test facility as equilibrium is reached and test conditions are generated. Test conditions must be confirmed periodically (typically when mercury speciation measurements are made).[6] The table also assumes either continuous (automated) mercury measurement capability or staffing available round-the-clock to perform mercury measurements. However, as noted in the table, an exception is made for laboratories not able to perform mercury measurements during overnight hours. In this case, the determination of equilibrium is a longer process due to the extended time between evening and morning tests.

Table 4-1 also specifies the allowable loss/gain in total mercury across the catalyst (<5% of the inlet value), as well as the actual deviation allowed from the average of the six (6) measurements to establish that no trending is occurring (± 5%, absolute). These values imply that the accuracy of the mercury oxidation rate is expected to be roughly ± 5%.

Reportable Data

Table 4-1 gives guidance for establishing equilibrium, essentially requiring that the deviation be limited over a certain number of tests depending on the sample/test type. The actual reportable data may include the results of these tests, as demonstrated in Table 4-2. The actual number of data sets reported will depend on the sample/test type, as defined in Table 4-1 (either 3 or 6 data measurement sets are required, depending on the sample/test scenario). Table 4-2 only reflects the mercury speciation data, along with the calculated individual and average mercury oxidation rates. Complimentary data, such as gas composition, temperature, flow rate, area velocity, linear velocity, etc. must also be provided, as discussed in Chapter 6.

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[6] Test conditions should be held constant, generally conforming to the specifications set forth in Table 5-1, although some drift in conditions is allowable during early stages of attainment of equilibrium.

Table 4-1
Approximate Equilibrium Times and Recommended Frequency of Mercury Measurement

Sample/Test Type	Approximate Time to Establish Equilibrium	Recommended Frequency of Mercury Measurements	Number of Measurements Recommended to Show Established Equilibrium[7]	Allowable Loss/Gain in Total Mercury across Catalyst	Allowable Deviation in Elemental Mercury Oxidation Rate across Catalyst
New or freshly regenerated catalyst, initial conditioning and lab testing	72-168 hours	One (1) set of inlet/outlet measurements every 4-8 hours	Minimum of 6 sets of inlet/outlet measurements spaced evenly across minimum 24 hour period	<5% of inlet value, no trending	AVG[8] ± 5% (absolute), no trending
Aged catalyst, [9] initial lab testing or after lab system SD/SU [10]	48-72 hours	One (1) set of inlet/outlet measurements every 4-8 hours	Minimum of 6 sets of inlet/outlet measurements spaced evenly across minimum 24 hour period	<5% of inlet value, no trending	AVG ± 5% (absolute), no trending
Aged catalyst, change in lab test conditions, not including lab system SD/SU	12-24 hours	One (1) set of inlet/outlet measurements every 2-4 hours	Minimum of 3 sets of inlet/outlet measurements spaced evenly across minimum 8 hour period	<5% of inlet value, no trending	AVG ± 5% (absolute), no trending

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[7] The table assumes either continuous (automated) mercury measurement capability or staffing available round-the-clock to perform mercury measurements. If this is not the case, then a longer time gap (up to 16 hours) is allowed between the evening and morning measurements. For example, the six (6) sets of inlet/outlet data may be obtained as follows: 08:00, 12:00, 16:00 on the first day, and then 08:00, 12:00, 16:00 on the next day.

[8] Refers to the average of the six (6) sets of inlet/outlet measurements.

[9] New or freshly regenerated catalyst that has undergone conditioning qualifies as “aged” catalyst.

[10] SD/SU = Lab system shut-down and start-up cycle, while the catalyst sample is installed, either as a planned event or due to a system upset.

Table 4-2
Reportable Data Example

Data Measurement Set[11]	Date	Time	Catalyst Inlet		Catalyst Outlet		Calculated Oxidation Rate[12] (%)
			Hg Total (μg/m^3)[13]	Hg 2+ (μg/m^3)	Hg Total (μg/m^3)	Hg 2+ (μg/m^3)	
#1							
#2							
#3							
#4							
#5							
#6							
Average Oxidation Rate (%)							
Maximum Deviation[14], +/-% of average							

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[11] See Table 4-1 for required number of data sets (3 or 6 depending on test scenario).

[12] The calculation methodology should be stated in the test report.

[13] The basis for the reported values should be stated, i.e., definition of standard temperature and pressure, wet vs. dry conditions, and O_2 basis, as discussed elsewhere in the guideline.

[14] Must be less than 5% (absolute).

5

REFERENCE TEST CONDITIONS AND FACILITY PERFORMANCE REQUIREMENTS

Reference Test Conditions, Allowable Deviation, and Drift

Reference test conditions have been developed for mercury oxidation testing, which include allowable deviation from the target conditions and maximum allowable drift. These reference conditions are shown in Table 5-1.

Following Table 5-1 is an explanatory section which describes additional details related to the sensitivities of the various testing conditions, as well as the approach in selecting the reference test conditions, deviation from target, and allowable drift. The reader is urged to refer to this section for a better understanding of the information presented in Table 5-1.

The reference test conditions are simplified in that they do not require NH_3, NOx, SO_3, CO, or HBr. In particular, the lack of NH_3 and NOx will cause the test to deviate from real-world conditions, where virtually all catalyst in an SCR is exposed to some level of ammonia and NOx. The reference conditions are valuable, however, since they give an intrinsic mercury oxidation rate, without the interferences of the omitted flue gas components.[15] As such, they are useful for comparative purposes (e.g., between different catalyst types, or new and aged versions of the same catalyst), or may ultimately provide a metric for field predictions. Additional long-term experience will be required to determine exactly how a laboratory-measured intrinsic mercury oxidation rate actually corresponds to field performance.

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[15] HBr of course enhances mercury oxidation. The omission of this component simplifies the testing, but does not drastically affect the qualitative behavior of the catalyst since halogens are still present, i.e., HCl.

Table 5-1
Reference Test Conditions (Typical Eastern Bituminous Coal), Allowable Deviations, and Drift

Parameter	Reference Test Condition Target Value	Allowable Deviation from Target[16]	Allowable Drift[17]
Temperature	700°F	± 10°F	± 5°F
AV[18]	18.0 m/hr (STP)	± 0.5 m/hr	± 0.3 m/hr
O_2	3.0% (dry)	± 0.5%, absolute	± 0.3%, absolute
H_2O	8.0% (actual, 3% O_2)	± 2%, absolute	± 1%, absolute
CO_2	0.0% or As-Generated	--	--
NOx	0.0 ppmv	± 10% of value	± 5% of value
α (NH_3/NOx ratio)	0.0	± 0.02 absolute	± 0.01 absolute
SO_2	1,000 ppmv (dry, 3% O_2)	± 10% of value	± 5% of value
SO_3	0.0 ppmv	--	--
HCl	75 ppmv (dry, 3% O_2)	± 10% of value	± 5% of value
HBr	0.0 ppmv	--	--
CO	0.0 ppmv	± 10% of value	± 5% of value
Hg^0	10-100 $\mu g/m^3$ (STP, dry, 3% O_2)	± 10% of value	± 5% of value
Native Hg^{2+} (reactor Inlet)	Less than 7% of total mercury	--	--
Added Hg^{2+} (reactor Inlet)	0.0 $\mu g/m^3$	± 10% (as proportion of Hg^T) absolute	± 5% (as proportion of Hg^T) absolute

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[16] Allowable deviation from target refers to the deviation from the target that is allowed in setting test conditions. Although the condition may deviate from the target, once the condition has been set, it must be maintained within the allowable drift limits. For instance, 700°F may be the target temperature for a test, however, the system settles at 705°F. Under the guideline, this is acceptable. However, the drift must not exceed the specified values using 705°F as the reference.

[17] Drift refers to the maximum amount of drift that a parameter may have during the duration of a single test. The reference point for the drift is the target value including any deviation (see above footnote).

[18] For plate catalysts, the specified AV assumes that samples equivalent to two plates in length (i.e., a full layer) are utilized.

Explanatory Notes Related to Reference Test Conditions

The following bullet items provide additional information related to Table 5-1. Important sensitivity information is summarized, and the general philosophy associated with the selection of reference test conditions, deviation, and drift, is given.

- **Eastern Bituminous Coal Reference Test Conditions**
  The reference conditions have been selected to conform roughly to typical flue gas conditions associated with the firing of eastern bituminous coal. If a laboratory chooses to perform reference tests which are not in alignment with Table 5-1, the laboratory must discuss the reasons for any deviations with the end user. For example, if the end user is burning a PRB coal, the laboratory may run the reference test at higher moisture, lower SO_2, and lower HCl levels than shown in Table 5-1 with permission from the end user.
- **Test Apparatus**
  Bench, semi-bench and micro reactors may be used to perform reference tests. However, there are two important cautions for micro-reactors that must be noted. First, the physical set-up (i.e., shortened sample length) will not allow for both the AV and LV to be matched to a field application simultaneously (according to EPRI's definition of micro-reactors). For the reference tests, the laboratory must match the AV reference target by adjusting the LV, accounting for the shortened catalyst length. Second, when working with deactivated samples, the mercury oxidation behavior of the shortened sample may deviate from the behavior of the sample as a whole due to a non-uniform deactivation profile. This is a key limitation for micro-reactors, and labs must clearly document and report where the sample was cut from the larger catalyst block. In order to define activity boundaries, testing at least two micro-scale samples (inlet face and outlet face) is recommended. This is also addressed in Section 6.
- **Temperature**
  Temperature is a fundamental condition of testing. Any data generated will necessarily correspond only to the tested temperature, unless known correlations can be applied to allow for temperature adjustments. For field applications, temperature will typically vary in tandem with reactor flow rate as well as to some degree with flue gas composition. Mercury oxidation is negatively impacted by increasing temperature under most conditions, but the effect is not particularly strong. As a result the deviation and drift values are not highly stringent. The reference temperature value of 700°F was selected as being roughly consistent with the average full-load operating temperature of the domestic SCR fleet as a whole.
- **AV**
  In most cases the tested sample length will be equivalent to a full-scale layer. In these cases if the AV is set to the field condition, then the LV will also match the field condition, and vice versa. This assumes that for plate catalysts, the sample length is equivalent to that of two plates, consistent with that of a field layer. If the testing conditions are set to match a field application, then then determination of AV and LV is made based upon the catalyst properties as well as the unit flow rate. The unit flow rate is typically full load as this is the controlling, or worst-case, condition for SCR mercury oxidation. However, laboratory testing may be utilized to predict performance at alternate load/flow conditions. In this case, other parameters may change as well, including temperature and flue gas composition. AV issues with micro-reactors were discussed above under the Test Apparatus bullet. The deviation and

drift values require that the AV be controlled tightly since AV can have a substantial effect on mercury oxidation (although the effect is probably not directly proportional under most conditions). The specified AV target for reference testing is 18 m/hr (STP), representing a typical value for a single layer.

- **O_2**
  Oxygen affects mercury oxidation, with the exact effect varying according to the specific conditions of testing. As a result, O_2 values must be selected with care, ensuring that when the tests are devised to be consistent with a field application that selected O_2 values match, within reason, those of the field. The reference O_2 condition selected (3%, dry) conforms roughly to a typical boiler outlet O_2 value operating to minimum excess O_2.
- **H_2O**
  Moisture affects mercury oxidation, with the exact sensitivity being dependent on test conditions. As a result, it is best for the tested H_2O values to be consistent with the associated field application, or in the case of the reference conditions, roughly average for eastern bituminous coal (8%). Moisture generally has an inhibiting effect on mercury oxidation. Therefore, conservative test conditions would include H_2O levels which are generally in the upper part of the H_2O range expected in the field.
- **CO_2**
  CO_2 is considered inert for mercury oxidation testing. As a result, CO_2 may be omitted altogether from the test conditions. For test apparatuses that have combustion systems, native CO_2 will be present. In any event, N_2 will typically be used to balance the inert content of the flue gas, and CO_2 and N_2 can be utilized interchangeably for this purpose.
- **NOx**
  NOx is not believed to have a direct impact on mercury oxidation. However, if NH_3 is present, then NOx will impact the ammonia level as the flue gas passes through the catalyst, due to the deNOx reaction. As a result, NOx will impact mercury oxidation in a secondary manner. If NOx is included in the test conditions, then NH_3 will typically also be included. In this case, it is likely that some NH_3/NOx ratio is being simulated, for which the NOx and NH_3 values will be consistent with that of the field. For instance, at the reactor inlet, the NH_3/NOx ratio will be relatively high, often around 0.9. In this location, the NOx will also be relatively high. As the flue gas passes through the catalyst, however, both NOx and NH_3 decrease. As a result, lower NH_3/NOx ratios correspond to a lower absolute level of both NOx and NH_3. In testing which simulates full-scale installations, therefore, the absolute values of both NOx and NH_3 must be considered, along with their ratio. Since tested NOx values potentially may vary widely, the deviation and drift values are on a relative basis. The simplified reference conditions do not require NOx or NH_3, and as a result deviate from a realistic operating scenario in the field unless the field reactor is being operated without ammonia (i.e., idling).
- **α (NH_3/NOx ratio)**
  From the above discussion, it is clear that setting an α value will result in setting an absolute level of NH_3, once the NOx value has been selected. The selected α will generally conform to some location within a reactor which is being simulated. If NH_3 is utilized without NOx being present, this produces constant ammonia throughout the tested sample. Although this situation may be valuable in some instances, especially for research purposes, it is not a

condition which conforms to any real-world operating scenario. The simplified reference test conditions set α to 0.0 which results in no ammonia being present. This is a condition which deviates from the field, where all catalyst is exposed to at least some level of ammonia. As a result, tests performed without NH_3 will inflate the mercury oxidation as compared to a real-world operating scenario, and care must be taken in evaluating the data and making any extrapolations to real-world SCR operation. Since NH_3 has a strong effect on mercury oxidation, the deviation and drift values are stringent.

- **SO_2**
  SO_2 has a negative impact on mercury oxidation. The effect is dependent on other testing conditions, and the sensitivity is not typically extreme, allowing for very reasonable deviation and drift values. In cases where field conditions are being duplicated, a conservative approach is to utilize SO_2 values that are on the upper end of the range expected in the field. The reference conditions specify 1,000 ppmvd SO_2, a value that is roughly consistent with a moderately low-sulfur eastern bituminous coal.
- **SO_3**
  The exact effect of SO_3 on mercury oxidation is not well understood, especially as a function of other operating conditions. Including SO_3 will allow for the testing conditions to more closely match those of the field, but the injection and operation of a test rig with SO_3 can lead to difficulties, and users may therefore opt to omit SO_3 from the testing, as is the case with the reference conditions.
- **HCl**
  HCl is of course one of the most critical factors affecting mercury oxidation. Mercury oxidation will be very sensitive to HCl levels in the range of 0-50 ppmv. Above this range, the sensitivity decreases. In any event, regardless of the other testing conditions, the selection of the HCl value will be critical to the data applicability. In cases where field conditions are simulated, it may be desirable to at least bound the range of HCl expected in the field by testing at several HCl conditions. The reference test conditions use 75 ppmvd HCl. This is consistent with relatively high chlorine eastern bituminous coals, but it should be cautioned that at this HCl value, the sensitivity to other parameters will be suppressed to some degree. Because of the increased sensitivity of mercury oxidation to HCl at low HCl values, the allowable deviation and drift is proportional to the value, i.e., the HCl conditions must be controlled more tightly in the lower, more sensitive, range.
- **HBr**
  HBr has the same general effect as HCl, but much less HBr is required to produce similar effects. Coal bromine is often poorly quantified, but all coals will contain some bromine. The inclusion of HBr tends to complicate testing, and many users will omit bromine. However, in cases where the testing is designed to mimic full-scale conditions, the selection of HBr value, if included at all, must be made with great care. Because HBr and HCl operate in tandem to promote mercury oxidation, they must be considered together. There will be a very high sensitivity to HBr at low HCl levels, as compared to higher HCl levels. In this case, sub-ppm control of HBr may be necessary. The reference test conditions omit HBr altogether from the testing.
- **CO**
  The effect of CO is not well quantified, especially as a function of other test conditions. From the available data, it appears that under some conditions (such as low HCl) mercury

oxidation may be particularly sensitive to CO, especially across low CO ranges. The issue is further complicated by the variability in CO in most field applications. Since CO generally has a suppressive effect on mercury oxidation, tests which mimic field conditions may utilize CO values in the upper range of those expected in the field as a conservative measure. The reference test conditions omit CO from the test requirements.

- **Hg^0**
  It appears that the absolute level of elemental mercury has little effect on the mercury oxidation rate that is measured. As a result, laboratories have a great deal of latitude in setting the value. In some cases, it may be desirable to set the elemental mercury value at a relatively high level as compared to a typical field application, to allow greater accuracy in the measurements. However, since this produces more mercury emissions from the test rig, there may be an incentive to lower the absolute mercury concentrations. As a result, there may be a very broad range in elemental mercury values utilized by the laboratory fleet as a whole. The very broad range specified (10-100 $\mu g/m^3$ STP, dry, 3% O_2) essentially leaves the setting of the elemental mercury level to the discretion of the laboratory.
- **Native Hg^{2+}** (Reactor Inlet)
  The level of native oxidized mercury entering the test reactor will be a function of the inherent oxidation that takes place upstream. The current guidelines specify that this oxidation not be more than 7% of the total mercury. Higher levels would be indicative of high rates of system mercury oxidation and lead to data validity concerns. Note that at low mercury oxidation rates for the catalyst/conditions being testing, deviations in the inlet speciation have more effect on the determined mercury oxidation rate than would be the case for samples/conditions which have a high rate of mercury oxidation.
- **Added Hg^{2+}** (Reactor Inlet)
  Users may opt to add oxidized mercury upstream of the catalyst to more closely mimic certain field conditions, especially in cases where lower catalyst layers are being simulated. However, adding oxidized mercury can be difficult and may add greatly to the complexity of the test rig. As a result, most laboratories do not add oxidized mercury as part of routine testing. It appears that this approach is satisfactory for most testing scenarios. However, again, if lower catalyst layers are being simulated, a more realistic simulation is obtained by matching the laboratory speciation to that of the field. The reference test conditions do not require the addition of oxidized mercury.
- **N_2**
  Similar to CO_2, N_2 is considered inert for mercury oxidation testing. N_2 will typically be used to balance the inert content of the flue gas, and CO_2 and N_2 may be utilized interchangeably for this purpose.

Facility Performance Requirements

Firms offering commercial mercury oxidation testing for SCR catalysts must be able to demonstrate they can meet several criteria related to facility performance. These criteria include inherent mercury oxidation rates and repeatability, among others. Table 5-2 outlines the recommended facility performance requirements. It is understood that the basic conditions of the testing will be similar to the reference conditions described in Table 5-1. However, the presence of mercury is not required for the measurement of inherent SO_2 conversion. Note that these requirements do not have to be demonstrated with each sample tested, but only at system start-up and periodically over the life of the facility. Many facilities will maintain a reference sample which represents a well-characterized “standard” catalyst that can be utilized to validate system performance. This type of sample is especially helpful in troubleshooting problems, or confirming that the facility is operating properly when unexpected results are obtained.

Facility Maintenance and Cleaning

Facility performance may degrade over time and therefore require routine maintenance and cleaning. In particular, build-up of contaminants due to long-term operation, or operation at extreme conditions, may result in performance issues such as high levels of intrinsic mercury oxidation leading to high reactor inlet oxidized mercury levels, or high levels of native oxidation through the reactor (empty reactor oxidation). In addition, loss in total mercury between the inlet and outlet measurements locations may become problematic, leading to poor total mercury material balance closure.

The actual mode by which contamination adversely affects performance is not completely understood, but there are a number of possibilities. These include 1) the formation of salts (ammonium, sulfate, chloride, etc.) that lead to mercury deposition or oxidation, 2) low temperature areas that lead to condensation of compounds and subsequent mercury adsorption, 3) high moisture conditions leading to condensation and mercury adsorption, 4) high surface area/porosity in some materials/surfaces leading to mercury adsorption or surface catalyzed mercury oxidation, and 5) deposits of catalyst dust leading to mercury oxidation not directly attributable to the catalyst sample itself. Each individual test apparatus will have its own specific characteristics in terms of fouling/contamination, but one common aspect of all systems is that high levels of NH_3 and SO_3 tend to exacerbate fouling/contamination.

Table 5-2
Facility Performance Requirements

Criteria	Requirement
Inherent Reactor Inlet Mercury Oxidation	The system shall not oxidize more than 7% of the available Hg^0 prior to the flue gas entering the test reactor (as measured at the nearest available Hg measurement point to the reactor inlet).
Inherent Reactor and Downstream Equipment Mercury Oxidation	The SCR reactor and downstream equipment (prior to outlet Hg measurement point) shall not oxidize more than 5% of the available Hg^0 entering the test reactor (as measured between the typical inlet and outlet mercury test points). Empty reactor test is assumed.
Inherent SO_2 Conversion	The system SO_2 conversion shall not exceed 0.2% of the available SO_2 as measured at the reactor inlet, with an additional SO_2 conversion not to exceed 0.2% through the remaining equipment as measured at the typical reactor outlet measurement point. Empty reactor test is assumed.
Repeatability	The standard deviation of the mercury oxidation rate (expressed as a percentage) for repeat baseline measurements for a single reference catalyst sample operating at the prior described reference test conditions shall not exceed 4% (absolute), with the additional requirements that the mercury oxidation rate be greater than 50%, and less than 90%, as tested. The baseline measurements shall be repeated a minimum of 5 times with not less than ~4 hours intervening between each test. This repeatability check shall be performed annually or after any significant system modification which may impact inherent system mercury oxidation.

Cleaning Frequency

For the flue gas general piping and mercury sampling lines, a routine cleaning schedule should be in place that dictates frequency in cleaning as a function of calendar or operational time. The required frequency will be a function of the design of the specific facility, as well as the testing conditions that are typically employed. Long-term experience will help facility owners to determine a routine cleaning schedule that is appropriate for their particular circumstances. As-needed cleaning may also be conducted based on facility performance, such as inlet mercury speciation, intrinsic mercury oxidation between the inlet and outlet measurement points, or material balance closure. Note that system performance degradation, indicating a need for cleaning may occur gradually, or suddenly.

For the reactor chamber itself, cleaning is required between each different catalyst sample tested, since the removal of catalyst particles is necessary to limit sample cross-contamination. This may be conducted by physical wiping of the internal reactor surfaces followed by washing/flushing (see below) or by washing/flushing alone, depending on the reactor size and design.

Cleaning Procedure

Both in-situ cleaning and ex-situ cleaning options are available, depending on the nature and severity of the contamination. In-situ cleaning generally refers to the flushing of the system with various gases, and does not require significant alteration of the system configuration. Ex-situ cleaning is typically conducted using liquid solutions, and therefore requires at least some disassembly of system components. In all cases, the actual procedures involved will be a function of the facility design and preferences of the operators. Information provided here is therefore suitable only for general guidance. Individual facilities will of course develop their own internal cleaning procedures that are best-suited to their specific needs.

In-Situ Cleaning

In-situ cleaning has advantages in that it can be performed relatively quickly with a minimal amount of disruption. In particular, since it does not involve disassembly of system components, it provides a streamlined cleaning approach. However, in-situ cleaning is not generally considered as rigorous as ex-situ cleaning, and may not be suitable for all cleaning applications, especially in cases where physical fouling is present. In-situ cleaning does appear to be particularly effective in flushing adsorbed mercury compounds from internal surfaces, which over time may lead to erroneous results.

In-situ cleaning procedures involve flushing the facility with particular gas compositions that facilitate mercury removal. The exact composition will vary according to operator preference, but typically humid gas is utilized with significant levels of HCl. The duration of flushing may vary, but usually 1-2 hours minimum is required. The system can then be purged with humid inert gas for several hours or overnight. The temperatures utilized will typically be consistent with routine testing or slightly elevated in comparison.[19]

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[19] One specific recommendation offered by a laboratory that employs in-situ cleaning routinely is to use flushing gas that contains 6-8 vol% H_2O, with HCl on the order of 60-120 ppmv (balance N_2) for a minimum of 1-2 hours, followed by purging with similarly humid nitrogen gas, for a minimum of 2-3 hours (overnight preferred).

Ex-Situ Cleaning

Ex-situ cleaning is typically conducted by flushing the piping/tubing and reactor chamber with dilute acid, such as dilute hydrochloric or nitric acid[20] followed by rinsing with de-ionized water. This will usually include (as much as feasible) flushing of all inlet piping downstream of the mercury introduction point, the reactor chamber itself, and outlet piping between the reactor chamber and the outlet mercury measurement point. In addition, depending on the type of mercury measurement system employed, flushing of the sample lines may be appropriate.

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[20] Dilute nitric acid solution may be prepared according to EPA Method, 29 (see section 7.4, preparation of HNO_3 solution)

6

SAMPLE PREPARATION, REPORTING, AND QUALITY ASSURANCE/QUALITY CONTROL

Sample Preparation

Sample Cleaning

The test sample should not be subjected to any aggressive cleaning procedures. A light cleaning to remove loose ash is allowed. For honeycomb and corrugated catalyst samples, gentle cleaning with compressed air is the preferred method. After this loose cleaning, the number of plugged catalyst channels should be documented. For plate catalysts, the sample assembly process itself typically results in the removal of loose ash deposits. In cases where there is a heavy, strongly adhered ash deposit, the plate should be rejected, if possible.

Sample Cutting and Assembly

Sample preparation is of course a function of catalyst type (honeycomb, plate, or corrugated), and also a function of the sample size that is required (according to test apparatus type/size). The discussions, below, outline the sample cutting and assembly process for test samples as a function of these two parameters.

Honeycomb

Raw honeycomb catalyst samples are normally available in nominally 150 mm x 150 mm cross-section elements. For bench-scale testing, the full cross-sectional area of the element is typically utilized. Full length samples are also normally utilized, consistent with the field application, although they may be trimmed slightly to provide a smooth face. For semi-bench sample preparation, the sample will be reduced in cross-sectional area. This is typically done using a band-saw or similar device that avoids stressing the catalyst leading to breakage. In cases where multiple sections of catalyst will be installed end-to-end to simulate a full-length element, the catalyst element may be cut cross-wise prior to length-wise cutting to reduce the chances of breakage. When these multiple sections are used, the location from which the sections were cut from the original sample element should be documented. This is important for samples that have been exposed in the field, since portions of the catalyst nearer to the inlet face typically experience a higher rate of deactivation than those further downstream.[21]

Sample preparation for a micro-reactor facility generally follows that for a semi-bench facility, but in the case of the micro-reactor, the sample may be shortened considerably. When samples are shortened, it is important to be aware that the mercury oxidation behavior of the sample actually tested may deviate from the behavior of the sample as a whole, as

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[21] In terms of deNOx activity, this mainly occurs on samples associated with eastern bituminous coal firing where arsenic poisoning is the primary deactivation mechanism. Catalysts exposed to PRB flue gas may exhibit more uniform deactivation throughout the catalyst length. Freshly regenerated, or new catalyst samples will typically exhibit relatively uniform activity throughout the raw sample. It is assumed that the mercury oxidation rate will also trend with these deNOx deactivation trends.

discussed above. This is a key limitation for micro-reactors, and labs must clearly document and report where the sample was cut from the larger catalyst block. In order to define activity boundaries, testing at least two micro-scale samples (inlet face and outlet face) is recommended.

Plate Catalyst

For plate catalysts, test modules are made by cutting portions of full-size plates to create an assembly with roughly the same geometric configuration as that in the field application. For bench-scale testing, a test module for plate catalysts is made using a metal box with cross-sectional dimensions roughly equivalent to a honeycomb element. The plate-to-plate spacing is generally the same as full scale, and the undulations of the plates are used to create the spacing, as in the full-scale assemblies. Additionally, a special fixture may be needed to ensure that the spacing is maintained at the edges of the box. Care should be taken in cutting to avoid "frayed" plate edges. When a full-scale layer is being simulated, two of these assemblies will be placed end-to-end, similar to a typical field installation. For semi-bench testing, the width of the plates is typically considerably narrower than 150 mm, and as a result it may not be practical to include the undulations present with the raw sample. In this case, only flat portions of the plates are utilized and they are assembled in the test reactor using some type of holder which maintains the geometry throughout the length of the sample, but also minimizes the amount of catalyst that is blocked from exposure to the bulk flue gas. The spacing of these parallel plates is typically identical to that of the parallel portions in the full-scale assembly. For samples that have been exposed in the field, they will most likely originate from the upper half of the field layer. As a result, they may have elevated deactivation compared to the layer as a whole, as discussed with honeycomb samples, above.

Sample preparation for micro-reactor testing will generally follow the same approach as for semi-bench testing, although the length may be considerably shorter. Methods of maintaining the plate geometry may differ considerably between the micro and semi-bench systems, however, especially if a glass reactor is utilized in the micro-reactor testing. As the sample cross-sectional area decreases, any non-idealities due to geometry inconsistencies or plate cutting (especially frayed edges) generally have a greater and greater impact on data quality. As a result, for extremely small cross-sectional area samples, extreme care must be taken to insure that the sample is edges are cut cleanly and that the plate geometry is maintained consistently throughout the sample. The micro-reactor cautions and recommendations mentioned for exposed honeycomb samples also apply to plate samples. In order to define activity boundaries, testing at least two micro-scale samples (inlet face and outlet face) is recommended.

Corrugated Catalyst

Preparation of corrugated catalyst samples generally follows that of honeycomb. However, since corrugated catalyst is not manufactured in elements that are similar in size to typical honeycomb elements, sample cutting will be required for all types of test apparatuses, including bench-scale systems.[22] The geometric surface area of corrugated catalyst is more

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[22] Corrugated catalyst is typically manufactured in "blocks" roughly similar in size to plate blocks that are utilized in field installations.

difficult to compute than for either plate or honeycomb samples, and the manufacturer can generally give guidance as to the proper methodology. Depending on the length of the raw sample material, multiple end-to-end sections of catalyst may be required to simulate a full-scale layer, even in a bench-scale system (similar to plate catalysts). Raw samples may be obtained as full-size blocks from the field installation, and then cut using a band-saw similar to the honeycomb samples, or samples may be retrieved from the field using a core-drill. In either case, field samples will typically require further cutting to prepare the test sample.

The micro-reactor cautions and recommendations mentioned for exposed honeycomb samples also apply to corrugated samples. In order to define activity boundaries, testing at least two micro-scale samples (inlet face and outlet face) is recommended.

Calculating Geometric Surface Area

To adequately determine the area velocity for laboratory testing, an accurate determination of the geometric surface area of the test sample is required. This determination depends on the type of catalyst being tested, as described below.

<u>Honeycomb</u>

Figure 6-1 shows the dimensions associated with a honeycomb sample. The length of the sample (l) should be determined by using a ruler or tape measure to measure the length of all four sides and averaging the measurements. The length should be measured within ±1 mm.

The cross sectional dimensions (a, b) should also be measured on all four sides and at a minimum of three positions along the length. This measurement should also be within ±1 mm. The six measurements for each dimension should be averaged.

Two methods can be used to determine the geometric surface area of the sample. The first is to use the geometric surface area provided by the catalyst vendor (m^2/m^3). The total surface area is calculated by multiplying the geometric surface area by the volume of the test sample and adjusting for any plugged catalyst cells (see below).

$$A = \left[A_{gs} \ x \ a \ x \ b \ x \ l\right]\left[\frac{C_{Tot} - C_{Pl}}{C_{Tot}}\right]$$

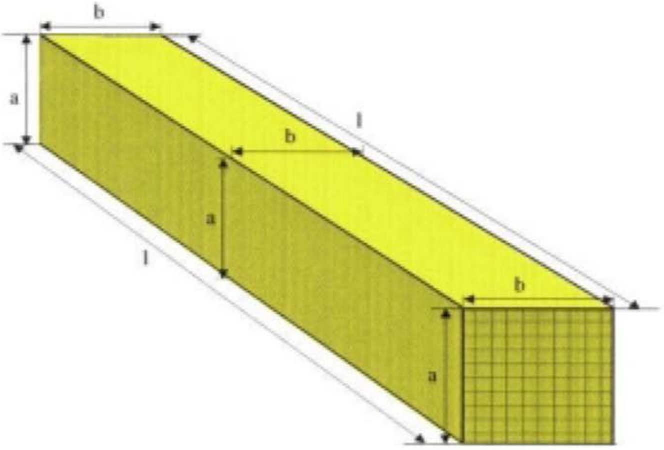
A = geometric surface area of sample, m^2
A_{gs} = specific geometric surface area, m^2/m^3
a, b, l = see Figure 6-1
C_{Tot} = total number of catalyst cells
C_{Pl} = number of plugged cells

The second method requires that the cell opening be measured. This measurement should be made within ±0.01 mm. It is recommended that the opening be measured for a minimum of 16 cells selected at random. The cell opening should be measured in both the "a" and "b" directions (see Figure 6-1). The "a" direction measurements and the "b" direction measurements should be averaged separately. The surface area of the sample can then be calculated as follows:

$$A = \left(C_{Tot} - C_{Pl}\right)\left(2d_a + 2d_b\right)l$$

d_a = average clear width channel opening in the a direction (a_{ch})
d_b = average clear width channel opening in the b direction (b_{ch})

Catalyst element



Catalyst cross-section

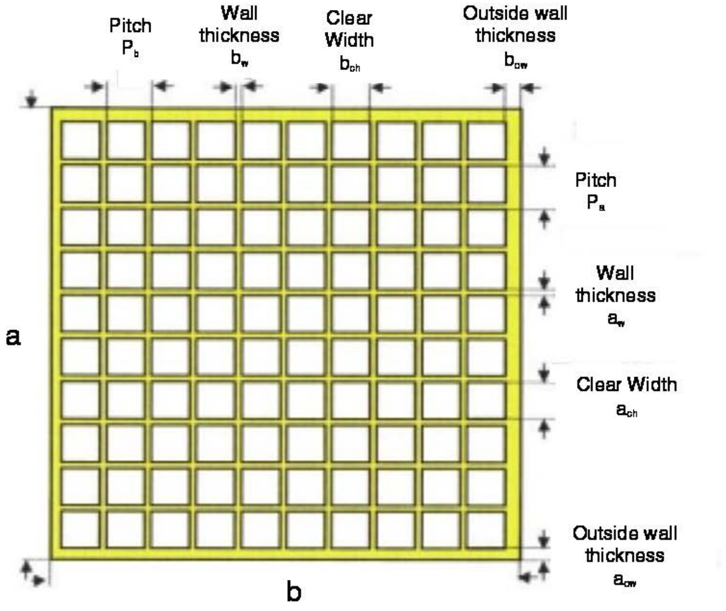


Figure 6-1
Dimensions of Honeycomb Catalyst[23]

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[23] See "Protocol for Laboratory Testing of SCR Catalyst: 2nd Edition," EPRI Technical Update, 1014256, and "VGB Guideline VGB-R302He, 2nd Edition," VGB Technical Association of Large Power Plant Operators.

Plate Catalyst

The width and length of each plate utilized in the sample assembly should be recorded. The length measurement should be made at the centerline of the plate, and the width measurement should be determined as the average of the plate width at the inlet, center, and outlet. The dimensions for each plate can then be averaged to create a width and length value for the assembly as a whole. The geometric surface area can then be calculated using the following equation.

$$A_{gs} = 2\, N_p\, W_p\, L$$

N_p = number of plates
W_p = averaged width of plates (see below)
L = average length of the plates

For bench sample assemblies, where the original plate undulations are utilized, it is often necessary to include the area of the undulations that provide the spacing. However, an adjustment needs to be made to account for the area that is not exposed to flue gas where the undulations make contact with the flat plate. One plate type catalyst manufacturer suggests using 95% of the plate area to account for this lost area. Alternately, the surface area calculation may be made by disregarding the undulations altogether and assuming that the plates are flat. In any event, it is recommended that the catalyst supplier be contacted to rectify any discrepancies between the measured specific surface area and the vendor's value of the specific surface area. For semi-bench and micro systems, the plates sections utilized will typically be flat and calculation of the surface area associated with the undulations is not applicable. In all cases, if the catalyst on some portion of the plate has eroded away, the calculated area should be reduced accordingly.

Corrugated Catalyst

Because of the complex geometry of the corrugated catalyst, it is recommended that the geometric surface area (m^2/m^3) be obtained from the vendor. The sample surface area is then determined similar to the first method for the honeycomb catalyst, by multiplying the volume of the corrugated test sample by the specific geometric surface area.

Reporting

The test report for catalyst sample mercury oxidation testing must contain the following items at a minimum:

- Calculated mercury oxidation rate and verification of equation used
- NOx conversion/deNOx activity, and SO_2 conversion (if tested)
- Absolute speciated mercury concentrations at the reactor inlet and outlet (see Chapter 5 for detailed guidance)
- Verification of standard conditions for which data are reported (including temperature, pressure, moisture, and O_2 basis)
- Description of the apparatus
- Description of the test procedure

- Description of the mercury addition procedure
- Description of the SO_3 addition procedure (if utilized)
- Description of the method of HCl and HBr addition (if utilized)
- Description of the measurement technique for all reported parameters
- Identification of the sample(s) tested
- Sample dimensions (a, b, L)
- Honeycomb/corrugated
  - Number of total cells
  - Number of blocked cells
  - If micro-scale, location of sample within larger block (e.g., inlet or outlet)
- Plate
  - Number of plates
  - Plate spacing
  - Plate thickness
  - If micro-scale, location of sample within larger plate (e.g., inlet or outlet)
- Specific geometric surface area
- Sample total surface area
- Gas flow rates
- Temperature (in/out)
- Gas composition
- Area velocity for the test
- Linear velocity for the test
- NOx reduction for each test
- NH_3/NOx ratio
- NH_3 concentration
- Documentation of all parameter measurement methods
- Documentation of conditioning/stabilization times and relevant data
- Documentation of reactor blank tests and relevant data
- Upon request, any relevant QA/QC data including standard test repeatability including data relating to the “Facility Performance Requirements” as described in Table 5-2.

Quality Assurance/Quality Control

Due to the difficulty in accurately measuring mercury concentrations under flue gas conditions, as well as the variability that is often present in mercury data, laboratories must have in place an aggressive QA/QC plan to insure data quality. Topics of particular importance include: 1) the mercury addition system, 2) the mercury measurement system(s), 3) sample conditioning and stabilization confirmation, and 4) test apparatus baseline characteristics.[24] All of these topics have been previously discussed in some detail. The exact QA/QC plan for any particular laboratory should be formally stated, and quality assurance testing should be done periodically to ensure the accuracy of the data that is generated. This plan, as well as test results, must be available to customers upon request.

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[24] This includes blank reactor tests, system validation tests, monitor calibration and validation, etc.

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