

Laboratory Testing Guidelines for Combustion Turbine Selective Catalytic Reduction (SCR) and Carbon Monoxide (CO) Oxidation Catalysts, 2nd Edition

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EPRI Project Manager A. Jimenez

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W.S. Hinton and Associates 115 Calle de Santiago CT Pensacola, FL 32502

Principal Investigator W.S. Hinton

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ABSTRACT

Selective catalytic reduction (SCR) and carbon monoxide (CO) oxidation catalysts are widely employed on both simple cycle and combined cycle combustion turbine systems. Laboratorybased approaches for measuring catalyst performance give end users a means of tracking and forecasting the capabilities and working life of the installed catalyst. In 2015, EPRI published laboratory testing guidelines for gas turbine SCR and CO catalysts. The current effort seeks to update those guidelines with updated best practices, additional background information, and more definitive guidance where possible. As with the prior report, current practices for conducting catalyst performance measurements vary among commercial laboratories. Notwithstanding this finding, catalyst system owners and operators desire standardization of procedures and related terminology to promote consistency in catalyst performance testing, estimation of catalyst life, and performance tracking. These updated guidelines, developed in collaboration with end users, catalyst suppliers, and third-party laboratories, represent continued efforts by EPRI to develop standard guidelines and protocols that are accepted and utilized by the industry. The guidelines presented here provide separate recommendations for SCR and CO catalyst testing and encompass the following principal areas: 1) general background, 2) test apparatus, 3) testing approaches, 4) testing methods and procedures, 5) preparation of catalyst samples, 6) chemical and physical property measurements, 7) various supporting technical discussions, and 8) recommendations for future work.

Keywords

Combustion turbine NO_x SCR catalyst CO oxidation catalyst Laboratory testing Guideline

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1 INTRODUCTION

Background

Selective Catalytic Reduction (SCR) and CO oxidation catalysts are widely used on both simple cycle and combined cycle combustion turbine systems. These catalysts may also be applied to various stationary and mobile sources, including conventional boilers, process heaters and internal combustion engines. Laboratory testing provides a means of assessing catalyst performance, both initially and over time. In particular, many system owners and operators perform periodic laboratory testing to monitor the catalyst and predict when fresh catalyst will be needed. Laboratory tests usually gauge performance in the form of a deNOx activity measurement (for SCR catalysts) or conversion measurement (for CO catalysts). When the data is utilized to forecast when a catalyst charge will reach its "end of life", this gives the system owner time to procure and plan the installation of new catalyst as part of a pre-scheduled, plantwide maintenance outage, and avoids expensive forced outages due to non-compliance. Performance information may also be used to gauge the deNOx or CO oxidation capacity of the catalyst to determine the impact of operational changes or upgrades on the current catalyst charge, and its ability to meet emissions requirements.

A number of commercial laboratories, including catalyst suppliers and third-party entities, provide combustion turbine catalyst testing services. However, these organizations may not use similar testing procedures or terminology. This raises questions about data consistency, applicability to the full-scale installation, data interpretation, and usefulness if a utility or system owner utilizes multiple laboratories for catalyst testing. A similar situation occurred with testing SCR catalyst on coal-fired systems in the early 2000's. To address these issues, guidelines for coal SCR catalyst were developed both in Europe (VGB Guidelines^(1,2)) and in the United States (EPRI Protocol for Laboratory Testing of SCR Catalysts ⁽³⁾). These guidelines now ensure that test procedures among the various laboratories are consistent. Although there are many similarities between laboratory tests for coal and combustion turbine SCR catalysts, there are enough differences to justify a separate guidelines document. Perhaps the most significant difference is the higher activity (K, m/hr) of combustion turbine catalysts, which can be twice that of coal catalysts. This presents unique challenges for laboratory testing which must be addressed.

Applicability

This guideline applies primarily to combustion turbine SCR and CO catalysts testing using bench scale or semi-bench scale reactors. It will also generally apply to gas-fired boilers, process heaters, stationary engines etc. that use catalysts similar to those commonly employed for combustion turbines, and with similar NOx levels and linear/area velocities. Note that for coal-fired boilers that have been converted to natural gas firing, the EPRI guideline associated with coal firing may be more appropriate, depending on specifics of the installation.

This guideline is applicable primarily to bench and semi-bench test reactors. However, much of the information provided will also apply to micro test reactors and in some cases specific discussions associated with micro reactors are included. A definitive guideline specific to micro reactors is not included due to the large variation in micro reactor sizes, catalyst sample sizes, test reactor capabilities, and testing approaches. It is important to note, however, that many CO catalyst test reactors deemed "micro" reactors due to their small size actually qualify as semi-bench reactors under EPRI's definitions (described subsequently) and are therefore applicable to this guideline. The catalysts assumed for this guideline are single function catalysts, in other words catalysts applied specifically for deNOx or CO conversion. Much of the guidance will apply to multi-function catalysts as well (e.g. catalysts which combine both deNOx and CO conversion in a single layer), but nuances of this type of catalyst performance may not be considered, especially if performance for deNOx and CO conversion is measured simultaneously. Future identified work includes guidance for the testing of multi-function catalyst.

Objectives

Ultimately, the objective of this laboratory testing guideline is to provide definitive testing approaches and procedures, including the setting of test conditions and required accuracies, which will provide <u>standard tests</u> for SCR and CO catalysts according to this guideline. In some cases, provisions for <u>alternate</u> testing approaches and test conditions are provided that have certain advantages, especially in terms of data accuracy, which will also meet these guidelines, providing that certain criteria are met.

Fundamentally this second iteration guideline is similar to the previously published guideline, especially in terms of test approaches, test conditions, and required data accuracies. However, the current guideline provides for a more definitive "standard" test for both SCR catalyst and CO catalysts. Perhaps more importantly, the current guideline seeks to more fully inform the end-user as to catalyst testing purposes, selection of testing conditions, applicability of laboratory data to the field, and potential deviations of measured laboratory performance as compared to expected field performance.

These laboratory testing guidelines were developed in collaboration with end users, catalyst suppliers and third-party laboratories. Specifically, the following areas are addressed:

- General background
- Test apparatus
- Testing approaches
- Testing methods and procedures (including test conditions)
- Preparation of catalyst samples
- Chemical and physical properties
- Supporting data and technical discussions

Chapter 6 of this report contains a detailed technical data and discussion section. The intent of this discussion is to explain the impacts of key factors such as NH_3/NO_x set point, inlet NOx concentration, NO_2 fraction, area velocity, data accuracy and others. Future work recommendations are also included.

Significant Additions and Changes from the Previous Guideline Version

To help the reader in understanding the changes that have been incorporated in this version of the guideline as compared to the historical version, some highlights are shown below.

- This guideline provides for clear "standard" testing in terms of both testing approach and test conditions. Thus, if an end-user references a "standard" test, then the testing approach and the selection of test conditions will be well defined and there will be little or no ambiguity in the testing methodology and the selection of testing conditions. The standard test approach is to determine activity by measuring deNOx, at field conditions for most parameters, at an NH₃/NOx ratio of 1.2.
- Provisions have been made for "alternate" testing approaches and test conditions, in keeping with common industry practices. This allows flexibility in developing a tailored approach to testing such that the generated data is most valuable to the end user. In particular, for the standard testing approach, provisions are made for increasing the inlet NOx level or the AV (as compared to the field values) to improve testing accuracy. Provisions are also made for using an alternate NH₃/NOx ratio or developing a response curve based on multiple NH₃/NOx ratios. If alternate test conditions are utilized, specific criteria must be met, which include full disclosure of the use of alternate conditions to the data. An alternate testing approach is provided, where deNOx is measured and reported using field conditions, at some specified level of ammonia slip. Thus, with this approach, activity is not reported. Instead, deNOx will be the primary reported response variable.
- Additional technical discussions have been provided to help better educate end-users on the selection of test conditions, and the applicability of the test data to field performance. This includes, in particular, the effect of the NH₃/NOx ratio.
- The current version of the guideline provides better definition of test reactor sizes. The guideline is generally applicable only to bench and semi-bench facilities, and does not strictly encompass testing with micro reactors, although discussions related to micro reactors have been provided in terms of sample length and cross section. However, EPRI's standard definitions as to reactor sizes applies to this guideline, and according to these definitions, many reactors routinely labeled as "micro" reactors, actually qualify as semi-bench reactors. In most cases, the identifying characteristic of a micro reactor, according to the EPRI definitions, is a significantly shortened catalyst sample as compared to the field catalyst. However, even in these cases, certain topics such as sampling, test condition accuracy and drift, and data accuracy, apply to micro reactors as well.
- A number of needs have been identified as possible areas for future research and are discussed under the "Future Work" chapter. These reflect perceived gaps in the knowledge base, or where a better understanding of catalyst behavior would be of significant value to end-users.

2 DEFINITIONS AND CONVENTIONS

There are a number of parameters and conventions associated with combustion turbine SCR and CO catalyst testing that warrant consideration.

Catalyst Parameters

1. Standard Conditions

A number of catalyst parameters are expressed in terms of flow rates measured at standard conditions. There is no single convention for standard temperature and pressure in the U.S. As such, the standard conditions used by the laboratory must be specified when results are reported. Table 2-1 lists common standard temperatures and pressures used in the U.S. <u>For</u> the current guidelines, 0°C and 1atm are suggested for calculating normal, or standard, conditions. Other conditions are acceptable as long as they are clearly specified in the test report.

Table 2-1Examples of Standard Conditions

Temperature	Pressure
0°C	1 atm
15°C	1 atm
20°C	1 atm
25°C	1 atm
20°C	100 kpa (0.9869 atm)
60°F	1 atm (14.696 psia)
68°F	1 atm

Note: misinterpreting a standard temperature of 0°C instead of 15°C can lead to a 5% error in flow rate.

2. Space Velocity (SV)

The space velocity (SV) is the flue gas flow rate (Q_{fg} , expressed at standard conditions) divided by the overall catalyst volume (V_{CAT}). Typically, this is expressed in units of 1/hr. The space velocity is used for both SCR and CO catalysts.

$$SV(hr^{-1}) = \frac{Q_{fq}(Nm^3/hr)}{V_{CAT}(m^3)}$$
(2-1)

3. Area Velocity (AV)

The area velocity (AV) is the flue gas flow rate (expressed at standard conditions) divided by the total catalyst surface area exposed to the flow (A_{CAT} , which is the geometric surface area, not the catalyst pore surface area, and includes the entire geometric surface area exposed to the flue gas. This is distinct from the open area at the face of the catalyst, or cross-sectional area of the catalyst). This quantity is usually expressed in units of m/hr.

$$AV(m/hr) = \frac{Q_{fq}(Nm^3/hr)}{A_{CAT}(m^2)}$$
(2-2)

Often AV is calculated as follows, where A_{CAT} in the above equation is expressed as the product of the catalyst volume (V_{CAT}) and catalyst specific surface area (A_{SP} , as defined below).

$$AV(m/hr) = \frac{Q_{fq}(Nm^3/hr)}{V_{CAT}(m^3) * A_{SP}(m^2/m^3)}$$
(2-3)

4. Specific Surface Area (A_{sp})

The specific surface area of the catalyst is the geometric catalyst surface area per unit volume of catalyst, typically expressed as m^2/m^3 . When multiplied by the installed catalyst volume (V_{CAT}), the product becomes the total installed geometric surface area of catalyst (A_{CAT}). Specific surface area should be obtained from the catalyst supplier when possible (or alternately it can be calculated via geometry measurements.) Note that the specific surface area should not be confused with the catalyst pore surface area (often termed "BET" surface area).

$$A_{sp}(m^2/m^3) = \frac{A_{CAT}(m^2)}{V_{CAT}(m^3)}$$
(2-4)

5. ΔNOx or DeNOx

 ΔNOx (often expressed as deNOx, %deNOx, or % NOx conversion) is the NOx conversion across the sample or catalyst layer. It is calculated as follows;

$$\Delta \mathbf{NOx} = (\mathbf{NOx}_{in} - \mathbf{NOx}_{out}) / \mathbf{NOx}_{in}$$
(2-5)

Where NOx_{in} and NOx_{out} out are gas-phase concentrations of NOx (typically ppmv) at a consistent basis (e.g., moisture and O_2). When this parameter is used in the following activity calculation, it is expressed as a fraction. However, the value is often expressed as a percentage in other contexts.

6. DeNOx and CO Oxidation Activity (K)

Catalyst deNOx activity (K) is a metric utilized by the industry and represents the performance of the catalyst in reducing NO_x or CO concentrations. The parameter may also be referred to as simply "activity," or "K-value." The activity is not a fundamental property of a particular catalyst material, but depends on the catalyst composition, geometry, and test conditions.

Catalyst activity is typically only used in reference to SCR catalyst, but in some instances may also be used to quantify CO oxidation as well. The activity is measured in a test

apparatus and is expressed as m/hr for SCR activity and 1/hr for CO oxidation activity. The equation for deNOx activity for SCR catalyst is as follows:

$$\boldsymbol{K}(\mathrm{m/hr}) = -AV * \ln(1 - \Delta \mathrm{NO}_{x})$$
(2-6)

where ΔNO_x is the NO_x conversion (deNOx) measured in the laboratory (expressed as a fraction, see above).

For a CO catalyst, the CO oxidation activity is defined as a function of space velocity, rather than the area velocity.

$$\boldsymbol{K}(1/\mathrm{hr}) = -SV * \ln(1 - \Delta \mathrm{CO}) \tag{2-7}$$

where ΔCO is the CO oxidation (expressed as a fraction) measured in the laboratory at a particular SV.

Fresh catalyst activity is often defined as K_o, indicating the fresh/unexposed catalyst performance at time zero.

7. Reactor Potential (RP)

The reactor potential (RP) is a quantity that expresses the overall deNOx "capacity" of a system. As with activity, this term is primarily a SCR catalyst parameter. This quantity has no units and is described by the following equation:

$$\mathbf{RP} = \mathbf{K}/\mathbf{AV}$$

(2-8)

The applicability of the calculated reactor potential to the field conditions will be consistent with the applicability of the deNOx activity to the field conditions.

8. Cells Per Square Inch (CPSI)

This is a term used for both SCR and CO catalyst, but is primarily used for honeycomb catalysts. If the CPSI of a catalyst is known along with the wall thickness, the specific area of the catalyst can be calculated.

Catalyst Types

SCR catalysts are available primarily in three different configurations: honeycomb, corrugated, and plate. Descriptions of the various terms typically used to document catalyst dimensions are provided below:

<u>Honeycomb</u>



Corrugated







3 SCR CATALYST LABORATORY TESTING

Purposes of Testing

In general, laboratory-based approaches for measuring catalyst performance give end users a means of estimating current catalyst performance/capabilities, and for tracking and forecasting the future capabilities and working life of the installed catalyst. The typical approach for routine catalyst testing generates a "deactivation" curve showing the catalyst performance over time, allowing for performance to be extrapolated into the future, such that catalyst replacements can be predicted and managed. Deactivation curves for SCR catalyst are generally generated as K/Ko vs. operating hours, where "K" refers to the activity at the time of sampling, and Ko refers to the initial or fresh catalyst activity of the catalyst. An alternate approach for testing would be to report deNOx capability at a specific set of test conditions, which again can be tracked over time to generate a curve, and used to predict catalyst replacements needs. This approach also gives the maximum deNOx that can be achieved at the ammonia slip limit at the time of sampling. Catalyst testing also provides a near real-time assessment of the performance capabilities of the catalyst, such that impacts of changing operating conditions, for example, can be assessed. Laboratory testing may also be useful in troubleshooting SCR performance issues, helping to identify if the catalyst performance is an issue. For any one operating unit, the purpose of laboratory testing may differ from time to time. The preferred testing approach could change as well. However, it is important to note that if performance tracking is a primary purpose of testing, the testing approach, and specific test conditions, should not change for a particular catalyst layer.

Standard and Alternate Testing Approaches and Test Conditions

This guideline provides a <u>standard</u> test methodology and standard test conditions which are the default. Thus, when this guideline is referenced it is assumed that the standard methodology and standard test conditions will be used unless otherwise stated. However, alternate methodologies and test conditions are provided for under this guideline and can be used assuming that the following criteria are met.

Required Criteria for the Utilization of Alternate Test Approaches and Test Conditions

- 1. The use of alternate test approaches and/or test conditions is fully disclosed and agreed upon by all parties.
- 2. The resulting accuracies of generated test data are consistent with the accuracies required by the standard tests.
- 3. Any inconsistencies between the alternate approach/test conditions and the standard approach/test conditions are understood by all parties, especially with respect to the resulting data and its applicability to the field installation. If data "corrections" are applied, those should be clearly stated, including assumptions utilized and the actual correction factor that has been applied.

Standard Test Approach – Measuring SCR Catalyst DeNOx Activity

Under the standard approach for testing SCR catalyst, catalyst activity (K) is measured in the laboratory by placing a catalyst sample in a test apparatus and flowing actual or simulated combustion products through the sample under a given set of conditions. This is the most common testing approach in the industry and is the default approach used by the majority of testing laboratories. For this testing approach, laboratory conditions are set which include the gas composition (including NOx, O₂, NH₃, H₂O, etc.), temperature, and flow rate. Generally, these parameters are set to simulate a selected field condition. Choosing the appropriate test conditions is critical to the usefulness of the laboratory generated data. Most combustion turbines have many operating modes which vary in terms of volumetric flow rate, NOx, temperature, and potentially fuel (e.g., natural gas, fuel oil, etc.). If the laboratory conditions are designed to simulate a specific field condition, care must be taken in the choice of that field condition. Often the laboratory conditions are set to simulate the "design" condition for the catalyst, which is typically the "controlling" condition (i.e., the condition at which there is the highest catalyst demand). In cases where fuel oil is utilized as an optional fuel, there may be a controlling condition when firing oil, and a controlling condition when firing natural gas. In most cases, even though one of the fuel oil firing conditions may be the global controlling condition, the laboratory testing is designed to simulate the controlling condition when firing natural gas. In any event, consideration must be given to the test conditions as they compare to the field conditions, and any differences must be taken into account when assessing field performance using laboratory performance as a basis.

When an activity measurement is generated, this generally gives the most useful information for assessing performance at different operating conditions, along with an estimate of catalyst "life" for any specific operating condition. Most mathematical models for SCR performance utilize catalyst activity, along with NOx and flow rate, to simulate field SCR performance. These models allow for easy prediction of field performance under alternate operating conditions, including NOx, and uniformity in the NH₃/NOx ratio, etc. Thus, generating an activity measurement generally gives the most fundamental information possible related to catalyst NOx reduction capability. In addition, guarantees are often written in the form of guaranteed activity over time, under a specific set of test conditions. Guarantees may also be written in terms of minimum NOx reduction at a given set of operating/testing conditions, including outlet NH₃ (slip), as discussed subsequently. Generating activity in the laboratory allows for direct comparison to guarantee values. If the laboratory tests are not performed at the guaranteed conditions, catalyst suppliers will often provide "correction" or "performance" curves which help to establish the guaranteed activity under alternate conditions.

The required <u>standard</u> laboratory testing conditions for measuring catalyst activity are shown in Table 3-1 and may be applied to bench and semi-bench test apparatus (defined later in this section). Micro reactors may also be used providing certain criteria are met (see subsequent discussions related to micro reactors). The technical discussions below provide background for the development of these standard conditions, as well as other procedural instructions. As discussed above, the selected "field value" for target conditions is important and will dictate the direct applicability of the data. Alternate test conditions may be utilized provided they meet the criteria discussed previously.

Calculating DeNOx Activity

The deNOx activity is calculated as:

$$\mathbf{K} = -\mathbf{A}\mathbf{V}^*\ln\left(1 - \Delta \mathbf{N}\mathbf{O}\mathbf{x}/100\right) \tag{3-1}$$

Where ΔNO_x is the percent NO_x reduction measured across the sample and AV is the area velocity used for the test. When activity is measured on a fresh unexposed catalyst sample, it is typically designated as Ko.

Table 3-1 SCR Catalyst Activity (K) - Standard Test Conditions

Parameter ¹	Target Level	Accuracy ²	Maximum Drift ³
Temperature	Field value	± 4.5°F (±2.5°C)	4.5°F (2.5°C)
Area Velocity	Field value or alternate of 35 m/hr ^{5,10}	\pm 5% of target	\pm 2% of value
Linear Velocity	Field, or as needed to achieve alternate AV	\pm 5 % of target	\pm 2% of value
O ₂	Field value (nominally 15% dry)	\pm 1% absolute of target	$\pm 0.5\%$ absolute of value
H ₂ O	As generated ⁴ or field value ⁸	\pm 1% absolute of target	$\pm 0.5\%$ absolute of value
CO ₂	As generated ⁴ or field value (nominally 3% dry)	\pm 1% absolute of target	$\pm 0.5\%$ absolute of value
NO _x	Field value or alternate ^{9,10}	\pm 1% of target	$\pm 0.5\%$ of value
NO ₂ /NO _x	Less than 5% or field ⁷		
SO ₂	None added		
NH ₃ /NO _x ratio	1.2 or alternate ⁶	$\pm 2\%$ of target	±2% of value
N ₂	Balance	NA	NA

1. As measured at the reactor inlet.

2. Accuracy refers to the absolute deviation of the test condition from the target condition.

3. Drift refers to the maximum amount of drift that a parameter may have during the duration of a single test.

4. "As generated" assumes a natural gas-fired combustion source and does not need to be controlled unless a specific target is set.

5. Use field value if measured NOx reduction < 95%; otherwise increase AV to 35 m/hr. If NOx reduction is not <95% at AV of 35 m/hr, then inlet NOx may be increased as necessary.

6. If the required accuracy cannot be met for the NH₃/NOx ratio, then see discussion under alternate multi-point testing.

7. If the field NO_2/NOx ratio is known to be high (especially for values >50%), then field values should be used. See discussion in Chapter 6.

8. Note that field moisture levels vary widely according to the reference field condition as a function of assumed ambient relative humidity and temperature, as well as if water injection (such as steam augmentation) is included.

9. Inlet NOx may be increased to improve test accuracy if measured deNOx is not <95% after having increased the AV to 35 m/hr.

10. The use of alternate values are provided for subject to the required criteria for their use. See individual discussion on selecting alternate values.

NOx and Area Velocity

The discussions below describe the impact of NOx and area velocity on activity measurements, along with the standard and alternate test conditions for these parameters. In all cases, it is important for both the laboratory and the customer to understand the impact of NOx and area velocity on resulting activity measurements, especially when these parameters deviate from the field. Large deviations from the field will result in deNOx activities that will not be directly applicable to the field. Chapter 6 contains additional discussion related to these parameters.

Impact of NO_x Level and Area Velocity on Activity Measurement Applicability

For new combustion turbine catalyst samples tested in the laboratory at the full-scale area velocity condition ("field value"), the high initial activity may result in a measured NO_x reduction near 100% during the activity test. In this case, the activity is difficult to determine accurately due to the very low catalyst outlet NO_x level. This is illustrated in Figure 3-1, which shows how the measured outlet NO_x varies with both area velocity and inlet NO_x level for a catalyst with an activity of 85 m/hr. For an area velocity less than 30 m/hr (not unusual for a full-scale design) and an inlet NO_x level of 20 ppm or less, the outlet NO_x values will be less than 1 ppm, which may be difficult to measure accurately.



Figure 3-1 Effect of Area Velocity and Inlet NO_x on Measured Outlet NO_x for Catalyst with K = 85 m/hr

The accuracy of the outlet NO_x measurement at very low levels is important due to the sensitivity of the activity calculation at high NO_x removal rates. Figure 3-2 shows how catalyst activity varies with small variations in outlet NO_x , particularly when the outlet NO_x approaches 1 ppm (in this case, fixed area velocity of 35 m/hr and K = 85 m/hr). At these conditions, an inlet NO_x level of 20 ppm results in an outlet NO_x of 1.8 ppm, and a variation of \pm 0.5 ppm in the measured NO_x will cause the catalyst activity to vary from 97 to 76 m/hr. This sensitivity decreases significantly at higher inlet NO_x values.



Figure 3-2 Effect of NO_x Levels on Catalyst Activity, K, for $A_V = 35$ m/hr

Test data provided in the technical discussion section of this document (Chapter 6) show that catalyst activity is relatively insensitive to changes in area velocity (i.e., changes in sample length or flow, see Figure 6-5). In fact, this sensitivity may be assumed to be within the activity measurement error for area velocity ranges analyzed in Chapter 6. Conversely, test data show that activity may be significantly sensitive to variations in the inlet NOx concentration (see Figure 6-6).

Standard Test Conditions for NOx and Area Velocity

The standard test conditions for NOx and area velocity are the field conditions and should be used if there is not a specific concern as to data accuracy. This gives the most directly applicable catalyst performance data since it most closely matches the actual field conditions. However, in cases where data accuracy is a concern, the following alternate conditions may be utilized, assuming that the criteria set forth at the beginning of this report section are met.

Alternate Test Conditions for NOx and Area Velocity

The following alternate conditions may be utilized when data accuracy is a concern and therefore deviation from the field conditions is justifiable. When making adjustments to either inlet NOx or area velocity, it is recommended not to deviate from the field conditions more than is necessary to provide an acceptable level of data accuracy. Fresh catalyst will exhibit a higher deNOx activity than exposed catalyst, and in most cases, therefore, the selected alternate test conditions, if required, will be consistent with the testing of fresh catalyst. It is recommended that these selected test conditions not be changed as the catalyst performance is tracked over time (i.e., as the catalyst becomes more deactivated with operating hours), even though the alternate test conditions may not be necessary for aged catalyst. This is done to ensure that the test conditions remain constant throughout the testing program for a particular catalyst layer, thus providing accurate relative performance data over time.

If the inlet NOx or area velocity are adjusted to improve accuracy, the criteria for using alternate test conditions set forth in at the beginning of this report section must be met. In addition, in the test report, the effects of the changes on the measured DeNOx activity should be estimated and disclosed when possible. If "corrections" for the effects of a change in AV or inlet NOx are included in the reported data, this should be disclosed.

Area Velocity

To improve accuracy of the activity measurement under high deNOx conditions (see discussions above, and discussion in Section 6 of this report), the area velocity (AV) may be increased by increasing flow rate, thus creating a higher area velocity and linear velocity. This approach for decreasing deNOx and therefore test accuracy theoretically provides more accurate results due to the lower sensitivity of calculated deNOx activity to differences in area velocity as compared to differences in inlet NOx. Consequently, an AV increase is recommended as the first tier adjustment to improve test accuracy. In this case the standard recommended AV is 35 m/hr. In rare cases, where field AV is higher than 35 m/hr, AV may be increased reasonably above the field value. However, the selected alternate area velocity should not deviate more from the field value than is necessary to provide for the required data accuracy. Note that shortening of the sample length to increase area velocity is generally not recommended, as this affects the NH₃ and NOx profile (inlet to outlet) through the tested sample, and for samples which have a deactivation profile (in other words the inlet and outlet of the catalyst have different levels of deactivation), shortening the sample result in a non-representative sample as compared to the field. Further, ammonia oxidation is significantly affected by catalyst length, thus if the catalyst length is adjusted, the impact of ammonia oxidation on overall performance may not be adequately assessed.¹³

Inlet NOx

Adjustment of the NOx level to improve the data accuracy is commonly utilized in laboratory testing, especially in cases where the field NOx value (i.e. the target value for the standard conditions) is below 20 ppmvd. However, data provided in this guideline demonstrates that activity is more sensitive to differences in inlet NOx than is the case for differences in AV. As a result, the first tier adjustment for improving test accuracy is to adjust AV, as described above. If, however, adjustment of AV to 35 m/hr does not provide the necessary lowering of exhibited deNOx, then an increase in inlet NOx is warranted. No strict guidance is provided as to the preferred higher NOx level, but the selected alternate NOx value should not deviate more from the field value than is necessary to provide for the required data accuracy.

NH₃/NOx Ratio

Standard Test Conditions for NH₃/NOx Ratio

Detailed discussion related to the impact of the NH₃/NOx ratio on test data, as well as the selection of the NH₃/NOx ratio for testing purposes, can be found in Chapter 6. Most laboratories test at a "standard" NH₃/NOx ratio of 1.0 or 1.2, with 1.2 being more common. In addition, many SCR models operate using an assumed NH₃/NOx ratio of 1.0 or 1.2 as the testing basis for input deNOx activities, again with 1.2 appearing to be the most common. For most facilities, an NH₃/NOx ratio of 1.2 is more consistent with the end-of-life actual operating ratio. Further, deNOx (and resulting calculated activity) is much more sensitive near an NH₃/NOx ratio of 1.0

than it is at 1.2 (see discussions in Chapter 6). As a result, and in keeping with the previous version of this guideline, the standard single-point NH_3/NOx ratio for testing as provided by this guideline is 1.2. This value should be used if alternate values, as described below, are not specified.

Chapter 6 contains detailed discussions related to the effects of the NH₃/NOx ratio on activity data, as well as discussions related to selecting an appropriate NH₃/NOx ratio if the standard value is not used. Note that regardless of the NH₃/NOx condition(s) selected for testing, laboratories may choose to include testing at a low NH₃/NOx ratio (such as 0.5) as a QA/QC measure. In this case, the ammonia conversion should be near 100%, allowing for ammonia slip to be excluded from the material balance (this is particularly useful in cases where ammonia slip is not typically measured). Thus, if the reduction in ammonia does not closely match the reduction in NOx, there would be an indication of an issue, such as a measurement error, or that ammonia oxidation is occurring. This QA/QC measure is highly recommended as part of routine system performance checks.

Alternate Test Conditions for NH₃/NOx Ratio

Single-Point Tests

Laboratories and their customers may select an alternate single-point NH₃/NOx ratio for testing compared to the standard NH₃/NOx ratio of 1.2, if agreed upon. Alternate NH₃/NOx conditions must be selected with care, and must consider the field conditions, as well as the test accuracies. Very high NH₃/NOx ratios are generally not recommended since they may create high deNOx, especially with fresh catalyst, and thus reduce the test accuracy. This will depend somewhat on the catalyst and field specifics. It should be noted, in particular, that once an NH₃/NOx ratio is selected for a particular catalyst layer, it should not be changed as the layer is tracked over its life (unless there is a specific reason to do so). This will provide data that is consistent for tracking purposes. As a result, NH₃/NOx ratios will generally be selected to ensure test accuracies etc., based on fresh catalyst performance, even though test accuracies may improve as the catalyst ages due to lower deNOx.

Multi-Point Tests

Figure 6-1 in Chapter 6 demonstrates conceptually how variations in the NH₃/NOx ratio affect the activity measurement. The most rigorous and technically useful approach to setting the NH₃/NOx ratio would be to essentially generate the NH₃/NOx ratio response curve for deNOx activity. With this information, the deNOx activity corresponding to any NH₃/NOx ratio within the measured range could be accurately estimated, and thus highly accurate deNOx activities can be applied for various purposes such as to determine catalyst life, and to assess real-time performance capabilities. Further, generating a curve can help determine ammonia oxidation behavior.¹³ Typically, this approach would involve generating deNOx activities for a minimum of three (3) points across a range. For example, this range may include NH₃/NOx = 1.0, the highest NH₃/NOx ratio expected for the facility over its life, and a midpoint value. *Multi-point testing is also applicable if it is determined that the laboratory cannot measure the catalyst activity for a single NH₃/NO_x ratio setting with the required precision. In this case, a series of tests (4 minimum) is recommended, varying the NH₃/NO_x ratio over a range encompassing the desired NH₃/NO_x ratio and then interpolating the data to the desired NH₃/NO_x ratio. For example, if the target NH₃/NO_x ratio is 1.2, then 4 data points across the range of nominally 0.95*

to 1.50 can be measured, and the data then interpolated to $NH_3/NO_x = 1.20$. When testing using this approach, the data acquisition may be somewhat streamlined as compared to that below for generating a response curve where several NH_3/NOx ratio data points will ultimately be used, with the requirement being stable data averaged for at least 5 minutes at each NH_3/NOx ratio.

Catalyst Activity Measurement Error Analysis

A formal error analysis of the catalyst activity calculation is provided in Appendix A. For the assumed conditions and measurement uncertainties shown previously in Table 3-1, the overall activity uncertainty is nominally 2.7%, as shown in Table 3-2. It should be noted that this uncertainty applies to the actual measured deNOx activity at the test conditions. It does not indicate the expected deviation from the exhibited field performance, especially if alternate conditions are used rather than field conditions.

Table 3-2			
Assumed Uncertainties ((K = 85 m/hr,	NO _x -in=100 ppm	i, Av=20-45 m/hr)

Parameter		Uncertainty
Flow	Q	2%
Sample X-Section Dimension	d	1 mm
Sample Length	L	1 mm
Specific Surface Area	Asp	$0 \text{ m}^2/\text{m}^3$ (a)
Outlet NO_x	$NO_{x \text{ out}}$	0.5 ppm
Uncertainty	K	2.3 m/hr 2.7%

^(a)Assumed zero as this parameter is provided by the catalyst supplier

Conditioning

Prior to conducting an activity test, a fresh catalyst sample (i.e., a sample not previously exposed to flue gas) shall be conditioned at the activity test conditions for at least one (1) hour. However, conditioning times of much longer may be necessary depending on the specific catalyst characteristics and flue gas conditions. In particular, if tests are being conducted with SO₂ in the flue gas (although not required by this protocol), conditioning times may be significantly impacted due to the sulfation process. In all cases, laboratories should abide by best practices to ensure that catalyst samples are adequately conditioned prior to testing. No conditioning is needed for samples previously exposed to flue gas.

Number of Activity Tests

Discrete Testing

When discrete activity tests are performed, three (3) tests shall be conducted at each NH_3/NOx ratio setpoint tested, at stable conditions. If alternate multi-point testing is used to produce a curve, this shall be for each NH_3/NOx ratio tested. If the calculated activity of the three (3) tests differ by more than 2.0 m/hr as calculated between the highest and lowest measured value, then additional tests shall be conducted until this criteria is met. Laboratories should use best practices

to ensure that no trending is occurring and thus "steady-state" has been reached for the test, and in particular the drift requirements set forth in Table 3-1 should be met, which helps to confirm that steady-state has been reached. Note that if the catalyst is extremely slow in reaching steadystate, the drift requirement may be met even though true steady-state has not been reached.

Continuous Testing

In cases where continuous data are taken, data shall be collected and averaged over a period of 30 minutes of steady operation. Steady state shall be defined as drift of no larger than ± 2.0 m/hr in calculated activity at any time between the beginning and end of the test. This criteria must be met for all NH₃/NOx ratio values tested.

Measurement Methods

Gas Flow

A variety of methods may be used to measure gas flows. These guidelines do not require specific instrumentation to be used. However, recommended instrument accuracy is $\pm 2\%$.

Temperature

Temperatures may be measured using either thermocouples or RTDs. These devices shall have a calibrated accuracy of $\pm 2^{\circ}F$ ($\pm 1^{\circ}C$).

Gas Composition

The recommended gas analysis techniques are as follows:

 NO/NO_x : Chemiluminescent analyzer with a NO_2 to NO converter suitable for use in the presence of ammonia, or FTIR

O2: Paramagnetic, electrochemical, or zirconia oxide analyzer

CO₂: NDIR analyzer, or FTIR

H₂O: EPA Method 4 or FTIR

 NH_3 : Tunable Diode Laser, FTIR, Wet Chemical (specific ion electrode or IC), or chemiluminescent (by NO_x difference)

All continuous gas analyzers shall be calibrated before and after each test using EPA protocol calibration gases, where applicable.

Data Analysis and Reporting

At the completion of a test, the activity shall be calculated using equation 3-3.

$$\boldsymbol{K} = -\mathrm{AV} * \ln\left(1 - \frac{\Delta \mathrm{NO}_{\mathcal{X}}}{100}\right) \tag{3-3}$$

where;

$$AV = \frac{Q(0^{\circ}C, 1atm)}{A_{cat}}$$

Following the tests, a report will be prepared containing the following (as a minimum):

- Description of the apparatus
- Description of the test procedure and measurement methods
- Identity of the sample(s) tested
- Sample dimensions (a, b, L) and pitch
- Sample type, honeycomb/corrugated/plate
- Number of total cells/plates
- Number of blocked cells/channels if applicable, if any
- Amount of blockage (%) for each sample, if any
- Specific surface area (m²/m³)
- Standard temperature and pressure conditions used in the calculations
- Test conditions and results for each test
 - Number of tests conducted
 - Gas flow rates
 - Temperature (in/out)
 - Gas composition (O₂, H₂O, CO₂, NO_x)
 - Area velocity for the test (including calculation procedure)
 - Linear velocity at the face of the catalyst (not the cell velocity)
 - Differential pressure across the catalyst, with accuracy of the instrument noted
 - NO_x reduction for each test
 - Calculated activity (K and K/K₀)
 - Documentation of sample conditioning
 - NH₃/NO_x ratio
 - NH₃ flow rate
 - Start and stop time of each test

Alternate Test Approach - Measuring NO_x Reduction at NH₃ Slip Limit

For combustion turbine SCR systems, the SCR performance requirement is usually an outlet NO_x level (or NO_x reduction requirement) with an NH_3 slip limit. With this in mind, laboratory performance tests may focus on measuring the outlet NO_x level, or NO_x reduction, achievable at some desired NH_3 slip limit. This approach gives end-users an indication of the maximum achievable deNOx in the field system at the time of sampling (assuming that the test conditions are consistent with the field) while meeting the specified ammonia slip limit, and also allows for prediction of end-of-life at these conditions. In this sense, the data is more operationally-oriented than measuring deNOx activity. Guarantees may also be written in terms of NOx reduction. When this is the case, verification of the guaranteed performance will require testing using this alternate test approach to generate a NOx reduction value at the specified test/guarantee condition. In any case, when deNOx data is generated as the primary catalyst performance measurement, it is less flexible in its usage. For example, 1) most SCR models utilize deNOx activity, and this method will therefore not provide this primary input directly,

2) the data only apply to the specific test conditions (or corresponding field conditions), and thus are not directly useful to assess performance at alternate operating conditions, 3) if testing is being utilized to confirm guaranteed deNOx activities, then it will not provide a direct deNOx activity for comparison, and 4) this is not the most common testing approach, and therefore will not typically provide data which is directly comparable to other laboratories' measurements.

The required operating conditions for this alternate test measurement are shown in Table 3-4 and may be applied to bench-scale and semi-bench scale test apparatus. (See discussion below for applicability to micro facilities). The testing accuracy and other criteria applicable to measurement of deNOx activity apply to this alternate testing approach as well, with the exception of the accuracy and criteria related to the NH₃/NOx ratio. However, unlike the standard activity measurement approach, an accurate method for measuring ammonia slip is required for this alternate approach. For this, a continuous ammonia analyzer (i.e., TDL or FTIR) is preferred, but not required.
Table 3-3 NO_x Reduction at NH₃ Slip Limit (alternate test approach) – Standard Test Conditions

Parameter ¹	Target Level	Accuracy ²	Maximum Drift ³
Temperature	Field value	± 4.5°F (±2.5°C)	4.5°F (2.5°C)
Area Velocity	Field value	\pm 5% of target	$\pm 2\%$ of value
Linear Velocity	As needed to achieve desired AV	\pm 5 % of target	$\pm 2\%$ of value
O ₂	Field value (nominally 15% dry)	\pm 1% absolute of target	$\pm 0.5\%$ absolute of value
H ₂ O	As generated ⁴ or field value ⁷	\pm 1% absolute of target	$\pm 0.5\%$ absolute of value
CO ₂	As generated ⁴ or field value (nominally 3% dry)	\pm 1% absolute of target	$\pm 0.5\%$ absolute of value
NO _x	Field value	\pm 1% of target	$\pm 0.5\%$ of value
NO ₂ /NO _x	Less than 5% or field ⁶		
SO ₂	None added		
NH ₃ Slip	Permit level or customer specified value	± 0.2 ppm	± 0.2 ppm
NH ₃ /NO _x ratio	As measured/calculated at NH ₃ slip permit level		
N ₂	Balance	NA	NA

1. As measured at the reactor inlet.

2. Accuracy refers to the absolute deviation of the test condition from the target condition.

3. Drift refers to the maximum amount of drift that a parameter may have during the duration of a single test.

4. "As generated" assumes a natural gas-fired combustion source and does not need to be controlled unless a specific target is set..

5. The use of alternate values are provided for subject to the required criteria for their use. See individual discussion on selecting alternate values.

6. If the field NO_2/NOx ratio is known to be high (especially for values >50%), then field values should be used. See discussion in Chapter 6.

7. Note that field moisture levels vary widely according the reference field condition as a function of assumed ambient relative humidity and temperature, as well as if water injection (such as steam augmentation) is included.

Number of NOx Reduction Tests

Discrete Testing

When discrete activity tests are performed, three (3) tests shall be conducted. If the measured deNOx values of the three (3) tests differ by more than 2% (relative) between the highest and lowest measured value, or if the measured ammonia slip varies by more \pm 0.2 ppm from the target value for any test, then additional tests shall be conducted until this criteria is met.

Continuous Testing

In cases where continuous data are taken, data shall be collected and averaged over a period of 30 minutes of steady operation. Steady state shall be defined as drift no larger than $\pm 2\%$ (relative) in measured deNOx at any time between the beginning and the end of the test, and drift in measured ammonia slip by more ± 0.2 ppm from the target value at any time between the beginning and the end of the test.

Data Analysis and Reporting

The data analysis and reporting for the alternative test approach of measuring deNOx shall follow that of the standard test approach of measuring activity, with the following exceptions.

- 1. Catalyst activity calculation and reporting is not required.
- 2. Ammonia slip will be reported.
- 3. The test resulting NH₃/NOx ratio shall be calculated and reported within the accuracy of available measurements needed to make this determination (e.g., ammonia and flue gas flow rates, NOx values, slip values).

Catalyst Test Apparatus

Various reactor sizes with differing capabilities are used by the industry for the laboratory testing of SCR and CO catalysts. However, the industry is inconsistent in their definition/designation of laboratory test reactors. Generally, three (3) types of reactors are used; 1) bench scale, 2) semibench scale, and 3) micro scale. For clarity and consistency with other EPRI reports, this report utilizes the following definitions of laboratory reactors. These apply to both SCR catalyst and CO catalyst test apparatus, and regardless of the test approach being utilized.

Bench Scale

This is the largest scale of apparatus utilized for laboratory testing. By definition in this guideline, a bench-scale apparatus can accommodate full-length, standard honeycomb elements (150 mm x 150 mm cross-section) or plate samples of equivalent cross-sectional area and full-length plates. Both AV and LV values are typically maintained consistent with the field.

Semi-Bench Scale

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, but with cross-sections smaller than standard catalyst elements (i.e., less than 150mm x 150mm cross-section). As with bench scale facilities, semi-bench scale facilities have the ability to simultaneously operate at AV and LV values consistent with the field.

Micro Scale

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. 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.

Bench and Semi-Bench Scale Reactors

In practice, bench scale and semi-bench scale test reactors operate in much the same way, with the primary differences between the two reactor types being the smaller cross-sectional sample area utilized in semi-bench reactors. Since full length samples are utilized in both reactor types, and since linear and area velocities are maintained consistent with the field, these two reactor types accurately simulate field flow and mass transfer characteristics of the field application. Prior EPRI programs have demonstrated the consistency of data between the two reactor sizes for coal-fired catalyst testing. In light of the capabilities of bench and semi-bench reactors, and the requirements of this guideline, these reactor sizes would be required for testing under this guideline. This is consistent with the utilization of full-length samples.

It is important to note that although the industry may identify test reactors that use small crosssectional area samples as "micro" reactors, this alone does not necessarily qualify them as micro reactors according to EPRI's definitions. If full length samples are utilized, and other parameters such as AV can be met according to the guidelines set forth above, then the test rig will qualify as a semi-bench reactor, regardless of the sample cross-sectional area.

Micro Scale Reactors

Micro reactors use small "core" catalyst samples, and according to EPRI's definitions, do not represent the full catalyst length. As discussed above, test reactors capable of utilizing full-length samples, regardless of cross-sectional area, likely qualify as semi-bench reactors under EPRI's definitions. For instance, a micro reactor might accommodate a core-drilled sample 25 mm in diameter and 150 mm long, cut from a catalyst layer with a depth of 300mm. Due to the shorter sample length, it is not possible in a micro reactor to match the field linear velocity and area velocity simultaneously, and therefore would not strictly meet the requirements of this guideline (i.e., in a shortened catalyst sample, if area velocity is maintained consistent to the field application, then linear velocity must be decreased. Conversely, if the linear velocity is maintained, then area velocity will be higher than the field). Note that a difference in area or linear velocity alone is not necessarily problematic, and in fact this guideline includes provisions for increasing area velocity to improve accuracy under the "alternate" test condition provisions. In practice, though, since micro reactors typically include multiple deviations from the field application, including differences in catalyst length, and potentially large differences in linear and/or area velocities, they may generate data that may be significantly inconsistent with the field application unless adjustments are made. Further, the degree of deviation from field application may be much larger with micro reactors than would typically be the case for bench

and semi-bench facilities using alternate test conditions as defined in this guideline. Considering these factors, micro facilities are not applicable to this guideline. <u>However, this should not be</u> <u>construed as indicating that micro reactors are not applicable to catalyst testing in general.</u> In fact micro reactors do provide very useful data if the limitations and direct applicability of the data to field installations is well understood. If a micro reactor is utilized for catalyst testing, the end-user would depend on the laboratory's understanding of the applicability of the test data to the field application.

Obtaining Catalyst Samples

The first task associated with laboratory SCR catalyst testing is obtaining an appropriate sample. The discussions below provide guidance related to sampling frequency and location, special considerations for built-in test elements and core samples, and documentation of the sample dimensions.

Sampling Frequency

As the catalyst ages, the NH₃ slip versus time will become steeper, as illustrated in Figure 3-3. As a result, it is common practice to sample less frequently (typically every two years) during the initial one-third to one-half of the catalyst's expected life, then more frequently (typically yearly) thereafter. For example, for a catalyst with an expected life of 60,000 hours, the catalyst may be sampled every two years for the first 4 years (roughly 30,000 hours), then yearly thereafter. Ultimately, the sampling frequency will be guided by the expected catalyst life, the availability of sampling outages, and end-user preferences. In cases where data show unexpected deactivation, sampling frequency will generally be increased to provide closer monitoring and troubleshooting information.



Figure 3-3 Example Relationship Between SCR Catalyst Activity and NH₃ Slip

Sampling Location

Most large combustion turbines operate with a "two on one" configuration where two combustion turbine generators (each with a heat-recovery steam generator and an SCR) are applied to a single steam turbine generator. This creates an "A" and a "B" side for the unit, but from an SCR perspective this represents two individual SCR systems. When catalyst sampling is undertaken, it generally occurs on both SCR systems, with a single sample being taken from each. In cases where there is only a single SCR, then a single sample is generally taken. However, in all cases, if there is concern that a single sample may not be representative of the catalyst layer as a whole, multiple samples may be taken. This is typically based on end-user preferences and catalyst supplier or test laboratory recommendations. There is no concrete industry data to suggest one region of a catalyst layer will deactivate at a different rate than another, under normal circumstances. In this case, samples are usually acquired from reasonably accessible locations, usually on the lower part of the reactor where sampling can be conducted without the need for platforms, etc. However, if there are large temperature non-uniformities or other abnormalities across the catalyst layer, sampling at multiple locations may be warranted to better approximate the performance of the catalyst layer as a whole. In this case, it is preferrable to take multiple samples from the catalyst layer; for example, one where the catalyst experiences average/normal temperatures, and one from areas with high temperatures. This helps to establish any temperature impacts that may be occurring. In cases where core samples are taken, there is a great deal of latitude in the sampling locations. Under normal circumstances, core samples are acquired from easily accessed areas. However, in troubleshooting cases, where it is desirable to take samples from multiple locations, the actual sampling location is limited only by access. When built-in test elements are utilized for sampling, there will be more limitations on sampling locations. Generally, the majority of test elements will be placed in easily accessible locations, and these will be used for routine testing. However, it may be advantageous to randomly place test elements cross the entire reactor for troubleshooting purposes. These may not be used for routine sampling, but could be accessed for troubleshooting purposes if needed. In these cases, the end-user and catalyst supplier will typically work to balance the technical benefits against additional costs associated with the larger number of test elements. In all cases, when a catalyst sample is removed, the location must be documented, and the inlet/outlet orientation of the sample noted.

Built-in Test Elements – Special Considerations

Built-in test elements are typically sized to be used in bench or semi-bench reactors. However, if the need arises, a core sample may be taken from one of these elements for use in a micro reactor, as described below. If the SCR has built-in test elements, these are located within specific catalyst modules in the layer (see above discussions related to the distribution of these test elements within the catalyst layer). Since the number of these test elements is finite, removal must be planned to ensure there are a sufficient number of samples to test throughout the life of the catalyst. For instance, if an SCR has 8 test elements and an expected life of 60,000 hours, an element could be taken every two years for the first four years (roughly 32,000 hours, requiring 2 samples), then annually thereafter (roughly 32,000 hours remaining, requiring a total of roughly 4 samples), requiring a total of 6 samples. This would leave 2 samples remaining as margin. When test elements are removed, fresh replacement elements should be used to replace the element just removed (these can be provided by the catalyst supplier as part of the original catalyst purchase, or may be provided, as needed, at the time of sampling). Care must be taken to

ensure a replacement element is not inadvertently sampled at a later date as it will not have the same operating time as the bulk of the catalyst.

Core Sampling – Special Considerations

In the case of core sampling, there is no practical limit to the number of samples that can be taken from a catalyst layer. This allows for more frequent sampling than would be the case with a finite number of built-in test elements. Sampling location selection is more flexible in the case of core samples than with pre-installed test elements, allowing for any specific area in the catalyst layer to be sampled, which may be particularly valuable if there are areas of concern, such as areas that appear to have been impacted by high temperatures. Full-length core samples typically have cross-sectional areas smaller than full test elements and therefore would be used in semi-bench scale test reactors. If the core sample is shorter than the full catalyst layer, then the sample qualifies as a micro reactor sample and is not strictly governed by this guideline. However, a discussion of micro reactor sampling is provided below.

Core samples may be taken in-situ, or the entire block, or cassette, may be removed and sent to the laboratory for coring/preparation. For ceramic honeycomb catalysts, a dry diamond core drill may be used to obtain the samples. For corrugated catalysts, a stainless steel tube with a sharpened end may be used. Examples of both of these tools are shown in Figure 3-4. Note that in some cases the manufacturer may provide pre-drilled cores at various locations within the catalyst layer. In this case, the number of samples and sample locations available will be limited, similar to the limitations associated with built-in test elements.



Figure 3-4 Examples of Catalyst Coring Tools

Documenting Sample Dimensions

To calculate the activity from the test data, it is necessary to accurately determine the catalyst surface area. This calculation depends on the type of catalyst being tested, as described below.

Honeycomb

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 may be used to determine the geometric surface area of the sample. The first, and recommended approach, is to use the specific surface area provided by the catalyst supplier (m^2/m^3) . The total surface area is then calculated by multiplying the specific surface area by the volume of the test sample and adjusting for any plugged catalyst cells (eq. 3-4).

$$\boldsymbol{A_{cat}} = [\boldsymbol{A_{SP}} * \boldsymbol{a} * \boldsymbol{b} * \boldsymbol{L}] \left[\frac{\boldsymbol{C_{Tot}} - \boldsymbol{C_{Pl}}}{\boldsymbol{C_{Tot}}} \right]$$
(3-4)

 A_{SP} = specific surface area, m^2/m^3 a, b = cross sectional dimension L = sample length C_{Tot} = total number of catalyst cells C_{Pl} = number of plugged cells

The second method requires measurement of the cell opening. 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 directions of the cell and averaged. These cell measurements are best done using a microscope with a measuring vernier (Figure 3-5). The surface area of the sample may then be calculated as follows (eq. 3-5):

(3-5)

$$A_{Cat} = (C_{Tot} - C_{Pl})(4d_{Ave})L$$

 $d_{Ave} = average cell opening$



Figure 3-5 Example of Microscope Used to Document Catalyst Cell Dimensions

Corrugated and Plate Catalyst

For relatively large cross-sectional area samples where the geometry of the sample is consistent with the bulk of the catalyst on a catalyst volume basis, the specific surface area (m^2/m^3) should be obtained from the supplier. The catalyst surface area is then determined by multiplying the volume of the corrugated or plate test sample by the specific surface area. In some cases (such as when small plate samples are generated by cutting flat plates which are installed in a holder to maintain plate spacing), the geometry of the test sample will not be consistent with the full-scale

catalyst layer. In these cases, the surface area of the sample must be computed based on the actual sample assembly geometry by taking geometric measurements of the individual components of the sample. In this case, measurements should be taken with an accuracy of ± 1 mm in length/width of each element of the sample. In the case of plate samples, the width of each plate in the assembly should be measured at a minimum of three locations and averaged. Note that when test assemblies for plate catalysts are generated using flat plates only, the geometry will differ from the field catalyst, where the undulations in the field catalyst produce areas of catalyst that "touch" that are not included in the test assembly. Various means may be utilized by the laboratory to account for this discrepancy, which should be clearly disclosed as part of the test report.

For plate catalyst samples, generating test sample geometries which fully mimic the field geometry can be difficult. As a result, flat sections of catalyst are often used as described above. As noted, however, this will influence the direct applicability of the test data to the field application, since the geometry will differ markedly between the test sample and the field application. Where cross-undulations are present in the catalyst (i.e. undulations/ridges which may not be parallel to the flow path) fully replicating the field installation required cutting "across" these undulations. This cannot be accomplished with typical cutting methods used for flat areas of a plate, such as a shear press, since this approach will create damage to the catalyst cut edges, and result in flattening of the ridges. Alternate cutting methods are necessary, such as laser cutters, which provide clean cuts with no damage to the catalyst ridges. This is a specialized technique and detailed guidance is not within the scope of this guideline at present.

4 CO CATALYST LABORATORY TESTING

CO Catalyst Performance Measurement

CO oxidation catalyst performance is measured in the laboratory by placing a catalyst sample in a test apparatus and flowing combustion products (actual or simulated) through the sample under a given set of operating conditions. The CO oxidation rate across the sample is defined as follows:

$$\Delta CO\% = 100\% * (CO_{inlet} - CO_{outlet})/CO_{inlet}$$
(4-1)

Where the units of CO are ppmv, dry corrected to 15% O₂.

The required operating conditions for a CO catalyst performance measurement are shown in Table 4-1 and may be applied to bench and semi-bench reactors, as discussed later in this section. Note that most test apparatus designated as micro reactors for CO catalyst testing actually qualify as semi-bench systems under EPRI's definitions. The technical discussions below provide background for the development of these conditions, as well as other procedural instructions. If a laboratory chooses to deviate from these conditions or procedures, the changes must be identified and discussed with the end user.

 Table 4-1

 CO Oxidation Catalyst Performance Test Conditions

Parameter ¹	Target Level	Accuracy ²	Maximum Drift ³
Temperature	Field value or light-off curve from 200°F at low end up to 100°F above full-load field value	± 10° F (6°C)	5° F (3°C)
Space Velocity ⁴	Field value	± 5 % of target	± 2% of value
Test Sample Length	Field value		
O ₂	Field value (nominally 15% vol. dry)	± 1% absolute of target	± 0.5% absolute of value
H ₂ O	Field value ⁵	± 1% absolute of target	± 0.5% absolute of value
СО	100 ppmvd @ 15% O2	± 1 % of target	± 0.5% of value
SO ₂	None added		
CO ₂	Field value (nominally 3% vol. dry)	± 1% absolute of target	± 0.5% absolute of value
NOx	None added		
N ₂	Balance	NA	NA

1. As measured at the reactor inlet.

2. Accuracy refers to the absolute deviation of the test condition from the target condition.

3. Drift refers to the maximum amount of drift that a parameter may have during the duration of a single test.

4. The flow rate is set to match the field space velocity taking into account cell blockage, unless the laboratory and end user agree to test a higher space velocity (i.e., higher flow or worst-case condition)

5. Note that field moisture levels vary widely according to the reference field condition as a function of assumed ambient relative humidity and temperature, as well as if water injection (such as steam augmentation) is included.

Test Conditions

Space Velocity

Figure 4-1⁽⁴⁾ demonstrates the relationship between CO oxidation and space velocity for various temperatures. For a given catalyst volume, there is higher CO conversion at lower flue gas flow rates (i.e., lower space velocities). To ensure laboratory CO oxidation rates represent field values, tests shall be performed at the field space velocity if possible.

In some instances, the laboratory test apparatus may not be able to achieve the field space velocity. In these cases, the space velocity shall be set as close as possible to the field value, and the measured CO oxidation shall be scaled by assuming a first order reaction similar to SCR catalyst activity:

$$\mathbf{K}_{\mathbf{CO-Lab}} = -\mathrm{SV}_{\mathrm{lab}} * \ln(1 - \Delta \mathrm{CO}) \tag{4-2}$$

CO reductions at field conditions shall then be calculated from the laboratory measurements using $K_{\text{CO-lab}}$:

$$\Delta CO_{\text{fullscale}} = 1 - e^{(-K_{\text{CO-lab}}/\text{SV}_{\text{fullscale}})}$$
(4-3)

This correction shall only be used for small deviations in space velocity, nominally +/-10%, between the field value and laboratory. If the laboratory space velocity deviates significantly (e.g., a much higher flow rate producing "worst case" results), the laboratory is required to have a database to interpret the results for the end user.



Figure 4-1 CO Oxidation as a Function of Space Velocity

Temperature

CO conversion is also a function of temperature and is typically characterized by a "light-off" curve, as shown in Figure 4-2. As temperature increases, CO oxidation increases rapidly and plateaus at a certain temperature unique to each catalyst and space velocity. A light-off curve may then be developed during testing by measuring the CO reduction at temperatures across an appropriate range. As CO catalyst ages or is poisoned, the light-off curve may change. This change may provide information regarding the nature of the deactivation.

For light-off temperature curve tests, full-load field space velocity (i.e., mass flow rate) shall be maintained for each temperature setting. The ability to perform a full light-off curve may depend on the apparatus and its ability to maintain a constant mass flow rate and composition while varying the temperature.

As indicated in Table 4-1 above, the laboratory temperature setting shall either be the field operating temperature of the catalyst, or a light-off curve that covers the temperature range from nominally 200°F (93°C) at the low end to 100°F (56°C) above the full-load field operating temperature of the catalyst.



Figure 4-2 CO Oxidation Light-Off Curve

Background Gas Composition

Catalyst activity tests shall be performed under gas composition conditions as close to field conditions as practical. The exception is the inlet CO level, which shall be increased to 100 ppmvd to improve the accuracy of the CO oxidation measurement.

Conditioning

Prior to conducting a CO oxidation test, a fresh catalyst sample (i.e., a sample not previously exposed to flue gas) shall be conditioned at the test conditions for at least two (2) hours. For single-temperature tests, the conditioning temperature shall be the same as the test temperature. When a light-off test is to be performed, the conditioning temperature shall be the normal full load field operating temperature. No conditioning is needed for samples previously exposed to flue gas.

Number of Oxidation Tests

Single Temperature Test

After the conditioning period, CO oxidation activity shall be determined by conducting and averaging three (3) separate measurements (CO reduction). Each test shall be conducted for a minimum of 20 minutes, with a minimum of 20 minutes between tests.

Light-Off Test

If a light-off study is preferred, three (3) separate tests shall be performed. The temperatures should range from nominally 200°F (93°C) up 100°F (56°C) higher than the full-load field condition). (Note that lower minimum temperatures may be appropriate depending on supplier guarantees, etc.) Temperature increments shall be no larger than 25°F (14°C) increments. When the average measured CO oxidation rates are similar (\pm 5%) at four (4) consecutive temperatures, the temperature increment shall be increased to 50°F (28°C). The test shall be considered

complete after the averaged measured CO oxidation rates are similar (\pm 5%) at the next four (4) temperature increments or the temperature is 100°F (56°C) above the full-scale field operating temperature. During each light-off test, the unit shall be held at each temperature until the CO concentration stabilizes, and then 5 minutes of data shall be collected.

Measurement Methods

Gas Flow

A variety of methods may be used to measure gas flows. These guidelines do not require specific instrumentation to be used. However, recommended instrument accuracy is $\pm 2\%$.

Temperature

Temperatures shall be measured using either thermocouples or RTDs. These devices shall have a calibrated accuracy of $\pm 2^{\circ}F$ ($\pm 1^{\circ}C$). Temperature measurement devices should be placed such that they are representative of the actual gas temperature, without bias associated with wall effects, etc.

Gas Composition

The recommended gas analysis techniques are as follows:

- O2: paramagnetic, electrochemical, or zirconia oxide analyzer
- CO₂: NDIR analyzer, or FTIR
- H₂O: EPA Method 4, or FTIR
- CO: gas filter correlation, or FTIR

CO analyzers range from gas filter correlation, to FTIR, to NDIR technologies. NDIR (nondispersive infrared) analyzers measure the absorption of infrared light in the sample over a broader wavelength region. These analyzers are not recommended for CO oxidation testing because there may be potential interference from CO_2 or H_2O at low CO levels.

FTIR (Fourier transform infrared) measures absorption over a wide spectral range and is designed to compensate for interfering species.

Gas filter correlation analyzers measure CO by comparing the infrared energy absorbed by a sample to that absorbed by a reference gas. As such, these analyzers are capable of more accurately measuring low range CO concentrations. In most cases the low range of these analyzers is 0 - 1 ppm.

All continuous gas analyzers shall be calibrated before and after each test using EPA protocol calibration gases.

NO to NO₂ Oxidation

NO to NO₂ oxidation measurements across a CO catalyst sample are not required by these guidelines. However, some end users may request these measurements, especially if the CO catalyst is upstream of an SCR catalyst. The ratio of NO to NO₂ may impact SCR catalyst activity.

To perform a NO to NO₂ oxidation test, the same conditions (flow rate, temperature, O_2) shall be maintained as the CO oxidation test. The NO shall be set at the field level exiting the turbine. A NO/NO_x analyzer, such as a chemiluminescent analyzer with a NO₂ to NO converter or a FTIR analyzer, shall be used to measure the NO and NO₂ at the inlet and outlet of the CO catalyst. Any change in the total NO_x across the catalyst shall be noted. The NO to NO₂ oxidation shall then be calculated using the following equation, and the results and parameters included in the test report:

$$\Delta NO\% = 100\% * (NO_{inlet} - NO_{outlet}) / NO_{inlet}$$
(4-4)

Reporting

Following the tests, a report shall be prepared containing the following (as a minimum):

- Description of the apparatus
- Description of the test procedure
- Identity of the sample(s) tested
- Sample dimensions (d, 1)
 - number of total cells
 - number of blocked cells
 - Amount of blockage for each sample
 - Sample geometry cross section of area, length, and volume
 - CPSI, if known
- Standard temperature and pressure conditions used in the calculations
- Test conditions and results for each test
 - Number of tests conducted
 - Gas flow rates
 - Differential pressure across the catalyst, with accuracy of the instrument noted
 - Temperature (in/out)
 - Gas composition (O₂, H₂O, CO)
 - Space velocity for the test (including calculation procedure)
 - CO oxidation rate for each test (including plotted light-off curve if developed)
 - Documentation of sample conditioning
 - Start and stop time of each test

CO Catalyst Testing Apparatus and Samples

CO oxidation tests may be performed using a micro, bench or semi-bench reactor (see definitions in Section 3). Some laboratories choose to test full-scale CO catalyst blocks in a larger reactor, which is also acceptable. The catalyst sample shall be either a core sample, test button or other suitable geometry for the laboratory reactor, and the sample shall be tested at the field value for space velocity whenever possible. Sample and test apparatus sizes are discussed in more detail below.

Types of CO Catalyst Samples and Associated Test Reactor Designation

In general, reactor designations for CO catalyst testing follow the definitions given above for SCR catalyst testing (i.e., bench, semi-bench, and micro). However, due to the typical short length of CO catalysts, a test reactor that cannot test full-length SCR catalyst samples can often test full length CO catalyst samples. In this case, the reactor may be designated as a micro-reactor for SCR catalyst testing, but as a semi-bench reactor for CO catalyst testing. As a result, under the EPRI definitions, the majority of test apparatus for CO catalyst testing will qualify as either semi-bench or bench reactors. CO catalyst samples are classified as follows.

Core Samples (Figure 4-3a)

Core samples are generally 1" diameter and full field length. These are generally the smallest samples utilized for CO catalyst testing and are often tested in what is designated a "micro" reactor, due its small size. However, if full length core samples are used, and the space velocity is set at or near the field value, then the test reactor would operate as a semi-bench reactor under EPRI's definitions. Conversely, if the space velocity differs substantially from the field value (due to the inability to achieve field flow rates, for instance), even though full length samples may be used, then the test apparatus would be more accurately described as a micro reactor. Reactors sized for testing core samples often use simulated flue gas constituted from bottled gases, rather than a combustion source, as is typically the case with reactors associated with larger samples, as described below. This may provide better control of flue gas conditions, especially flows.

Test "Buttons" (Figure 4-3b)

Catalyst test "buttons" are larger than core samples and are typically sized to fit within a maximum 3" diameter pipe (approximately 2.75" in diameter and full field length). If the test button contains catalyst of the same length as the actual catalyst layer, then test reactors capable of accommodating these samples would generally be considered semi-bench reactors, assuming that the field space velocity is achievable. Due to their larger size, test buttons are often tested in a reactor using flue gas from a combustor. In this case, it may be more difficult to maintain flows while varying temperatures to develop a light-off curve.

Large and Full-Scale Blocks (Figure 4-3c)

In certain instances, full-scale (600 mm x 600 mm) or half-scale (300 mm x 300mm) catalyst blocks may be sent to the laboratory for CO oxidation testing. For testing at this size, an apparatus larger than a typical bench reactor is required. If the sample is cut down to standard bench size (150 mm x 150 mm) then a typical bench scale facility may be used.



a) Core: 25mm dia.



b) Button: 76mm dia.



c) Full Scale Module: 600 x 600mm

Figure 4-3 Examples of CO Catalysts

Sampling from the Catalyst Layer

General guidance for obtaining CO catalyst samples is provided below. CO catalysts are relatively thin (i.e., short) in the direction of flow compared to SCR samples. Due to the short catalyst lengths, it is typically for the acquired catalyst sample to be the same length as the catalyst layer. Therefore, as mentioned above, in many cases reactors which qualify as micro reactors for SCR catalyst samples, actually qualify as semi-bench reactors for CO catalyst testing purposes.

Obtaining Samples

Some metal foil catalyst suppliers install test buttons within the catalyst layers. In these cases, obtaining samples for testing is relatively easy, as the button(s) merely needs to be removed from the reactor and replaced with a new test button. If test buttons are not available, often an entire element is removed for testing. In these instances, a core or button sample can be fabricated for testing. For ceramic honeycomb catalyst, either a core sample is drilled for testing in a semi-bench reactor, or a larger piece cut for a bench reactor. Care must be taken to maintain the physical integrity of the sample as some of these metal foil catalysts are not brazed together as a single unit.

Number of Samples

Generally, a single sample is sufficient if test buttons are provided. If core samples are drilled from larger elements, one to three samples shall be drilled. As with SCR catalyst samples, if an "A" and "B" side is present for the unit, both sides are typically sampled.

Location of Samples

If the CO catalyst has been performing as expected, the location of the catalyst samples is not important, and samples may be taken from where it is convenient within the reactor. However, if there is a known issue within the reactor, such as a large temperature gradient, then an attempt shall be made to obtain samples from the low temperature zone as well as the average temperature zone. In the case of core samples, there is great latitude in selecting sampling locations. However, similar to the discussions above related to built-in SCR catalyst samples, built-in CO catalyst samples will have more limitations. In most cases the majority of built-in samples are located with easy access, with these locations being utilized under normal circumstances. However, in troubleshooting cases, it is beneficial to have built-in test elements at locations better distributed across the entire catalyst layer, such that samples from specific "problem" areas may be acquired if needed. Again, these may not typically be utilized, but would be available for troubleshooting purposes if needed.

Sampling Frequency

CO catalysts are often sampled at the same time as the SCR catalyst, and thus the guidance above related to SCR catalyst sampling would generally apply to CO catalyst sampling as well. However, in cases where CO catalyst is sampled independently from the SCR catalyst, then sampling frequency will depend on end-user preference and catalyst supplier recommendations. Generally, if slow expected deactivation is occurring, then CO catalyst can be sampled relatively infrequently, every two years or more depending on the expected life of the catalyst. In cases where relatively rapid deactivation is occurring, or the catalyst is near its end of life, then more frequent sampling (yearly, for example) may be appropriate to more closely monitor the status of the catalyst and estimate end of life. In all cases, if the field reactor is designed with pre-installed test samples, there will be a limited number of available samples, and care must be taken not to exhaust these samples (in this case the above discussions related to built-in SCR catalyst samples apply).

Documenting Test Sample Dimensions

A core sample or test button shall be round in cross-section. The length of the sample (L) shall be determined using a ruler or tape measure to measure the length at 4 different points approximately 90° apart and averaging the measurements. The length shall be measured within ± 1 mm. The length shall be the full field catalyst length.

The diameter of the sample (d) shall be measured with a ruler across a minimum of four diameters approximately 45° to each other. The measurements shall be within ± 1 mm and shall be averaged.

The volume of the test sample shall be determined by multiplying the cross-sectional dimension by the length and adjusting for any plugged catalyst cells.

$$\mathbf{V_{cat}} = \left[L * \pi * \frac{d^2}{4} \right] \left[\frac{C_{\text{Tot}} - C_{\text{Pl}}}{C_{\text{Tot}}} \right]$$
(4-5)

 C_{Tot} = total number of catalyst cells C_{Pl} = total number of plugged cells

Alternatively, if the specification of a honeycomb CO catalyst is given in terms of cells per square inch (CPSI), the cross sectional area shall be calculated using this value and calculating the number of open cells.

$$\mathbf{V_{cat}} = \left((\mathbf{C_{Tot}} - \mathbf{C_{Pl}})^* \mathbf{L} \right) / (\mathbf{CPSI})$$
(4-6)

5 PHYSICAL AND CHEMICAL PROPERTIES

Background

A number of physical and chemical properties are commonly measured for both SCR and CO catalysts. While these tests are not required, they can be useful for troubleshooting cases in which the catalyst is deactivating faster than expected.

Excessive catalyst deactivation is generally caused by either a thermal event or by poisoning. Thermal events, such as exposure to high temperatures, can cause failure of the catalyst or substrate material, sintering of active sites or the supports, or reactions of active materials. Deactivation due to a thermal event is usually irreversible. Catalyst poisoning, however, is often reversible, and can either be chemical or physical. Chemical poisoning is the result of selective contamination of the active sites by elements such as sodium and phosphorous, and for CO catalysts, sulfur. Physical poisoning involves masking, fouling or plugging of catalyst cells or pores, or attrition of the active sites or wash coat. Alkaline earth metals such as calcium and potassium can affect the acidity of the catalyst, thereby masking the ability for ammonia absorption or CO oxidation. Silicas and siloxanes (silicon, oxygen and alkane compounds) can convert to silicon dioxide which also serves to mask catalyst the active sites. Poisoning can generally be reversed by catalyst cleaning or regeneration.

Testing for physical and chemical properties can help identify if any of these issues are present in the catalyst samples. Physical properties include surface area, and pore size distribution and volume. These properties can be useful in determining if catalyst deactivation is due to pluggage, catalyst structural defects or surface fouling, and are generally performed prior to catalyst cleaning or regeneration. Chemical property tests, which include both bulk and surface analyses, are useful for assessing amounts of catalytic active material and poisoning effects.

This guideline does not require any particular physical or chemical property be measured on a routine basis, but does provide guidance as to the preferred testing methods and reporting requirements, as discussed in the following sections.

Catalyst Sampling

Guidelines regarding obtaining samples for testing pertain mostly to chemical analysis. However, if samples are to be tested for physical properties, these guidelines may be used as well. Due to the differences in composition between SCR and CO catalysts, the procedure for obtaining catalyst samples for testing of physical and chemical properties will differ. SCR catalyst is homogenous in composition, and as such testing is performed on the catalyst as a whole structure. CO catalyst is instead comprised of an inert ceramic or metal foil substrate covered with a wash coat containing active catalyst materials. Therefore, for CO catalyst, only the active surface coating and wash coat need to be analyzed for chemical and physical properties. This is a difficult measurement, and consultation with the catalyst supplier is recommended. Two methods are acceptable for obtaining CO samples for chemical analysis:

- 1. The wash coat and embedded active ingredients shall be scraped from the substrate and analyzed for poisons; or
- 2. The sample shall be acid washed, and the wash analyzed for poisons.

Whichever method is used shall be agreed upon by the end user and laboratory and noted as such in the final analysis report.

Since the actual amount of catalyst or catalyst wash coat required for chemical analysis is miniscule compared to the typical amount of catalyst that is retrieved from a full-scale installation, the chemical analysis data may strongly depend on the actual location of the analyzed portion of catalyst within the catalyst as a whole. Leading edge samples will typically exhibit higher levels of poisons than trailing edge samples. Thus, aged catalyst will require multiple catalyst samples per element while new catalyst generally only needs one sample. Table 5-1 summarizes the number of samples and sample locations for new and used SCR catalyst for testing of chemical and physical properties, while Table 5-2 summarizes the catalyst sample requirements for CO catalyst. These guidelines assume catalyst samples are primarily obtained from the reactors for SCR activity or CO oxidation tests, and therefore are given per catalyst element. If two catalyst elements or test buttons are retrieved from the reactor, then each test element shall have the listed numbers of samples analyzed for chemical and physical properties.

Table 5-1 SCR Catalyst Sampling Guideline – Minimum Requirements

Parameter	New Catalyst	Aged/Regenerated
Sample Location – Corrugated and Honeycomb ¹	One sample per element, located 2" downstream of inlet face	Two samples per element, one sample located roughly 2" downstream of inlet face, and one sample located roughly 2" upstream of outlet face

¹ It is assumed that with a honeycomb element, or portion of element, that the entire catalyst layer depth (in the direction of flow) is represented.

 Table 5-2

 CO Catalyst Sampling Guideline – Minimum Requirements

Parameter	New Catalyst	Aged/Regenerated	
Sample Location – Honeycomb	One sample per piece, with the sample retrieved from roughly the center of the piece	Two samples per element, one sample located roughly 1/4 th downstream of inlet face, and one sample located 1/4 th upstream of outlet face	
Sample Location – Metal Foil	One sample per piece, with the sample retrieved from roughly the center of the piece	Two samples per piece, one sample located roughly 1/4 th downstream of inlet face, and one sample located 1/4 th upstream of outlet face	

Physical Properties

Various physical properties can be measured for both SCR and CO catalysts. These commonly include such parameters as surface area, pore size distribution and volume. The need to perform these analyses varies greatly, and will be a function of variables such as catalyst age, general performance, specific field application, and owner or manufacturer preference, etc. Physical property evaluations may be particularly important for troubleshooting activities to determine blockage or masking of active sites or structural breakdowns.

Surface Area Analysis

Catalyst surface area is typically measured by the Brunauer Emmett Teller (BET) method. The BET method relies on gaseous adsorbents to determine the total surface area of a solid sample. The method detects the total microscopic surface area of porous material and should not be confused with geometric surface area. BET surface area is usually reported in m²/g and is several orders of magnitude greater than geometric surface area. BET surface area be parameter does not translate directly to field performance. Along with catalyst surface area, the BET method can also provide some information regarding pore size distribution. There are a few different BET tests that can be performed, including single point, multiple point, and high resolution. The tests performed should be consistent year to year.

Most commonly a single-point BET method is used, with nitrogen as the adsorbent. By determining the amount of nitrogen gas adsorbed onto a catalyst sample's internal and external surfaces at a specific temperature, the total surface area of the sample can be determined. Careful out-gassing of the sample is required prior to testing, and testing occurs under highly controlled pressure and temperature conditions. The method is time and cost efficient. Loss in surface area can be attributed to a number of factors such as sintering or pore blockage, and as a result, BET analysis is often employed when abnormal deactivation is encountered, and the source of deactivation is not readily apparent. Some loss in BET surface area is common as catalyst ages, but a marked decline in surface area can be a sign of acute catalyst degradation. Multi-point BET methods are also available, and if used can provide insight on the pore size distributions, similar to the gas adsorption methods described below.

BET surface area is one of the most common physical analyses methods utilized for SCR and CO catalysts, and may be performed routinely, or on an as-needed basis. The location of the sample with respect to the catalyst element as a whole, as well as with respect to its location in the reactor, may influence the results of the BET analysis.

Pore Volume and Size Distribution

The pore volume and pore size distribution of a particular catalyst are major design parameters. In many ways they dictate the diffusion and reactivity properties of the catalyst, and as such are carefully controlled during catalyst manufacture. The two primary methods for determining pore volume and pore size distribution are mercury porosimetry and gas adsorption (various specific methods). Both are discussed below.

Mercury Porosimetry

Mercury porosimetry is a technique that uses liquid mercury to determine pore size, size distribution, and total pore volume in porous solids, such as catalysts. The method is very common in the catalyst industry, and the underlying fundamentals are well understood. Elemental mercury is a "non-wetting" liquid at ambient conditions. Due to its high surface tension, external pressure is required to force its intrusion into small pores, etc. The pressure required for intrusion is proportional to pore size, and the volume intruded at a particular pressure is proportional to the pore volume at that pore size. The pressure versus intruded volume data can be manipulated in various ways to determine pore diameter distributions, volume distributions, etc. The method is useful when applied to SCR catalysts because it can quickly determine the pore structure of the sample, including total porosity, and can be used as a troubleshooting method to help determine if changes have occurred in the microscopic physical structure of the catalysts. Samples can be tested in various forms, including powders and small catalyst pieces.

Gas Adsorption

Somewhat similar to BET analyses, various gases can be used to examine the adsorption isotherms of catalyst samples. These data can then be manipulated to determine such parameters as pore volume and size distribution. The method works especially well for delicate materials or materials with extremely fine pores, and can be applied well to SCR catalysts. In general, the method gives the same basic information as mercury porosimetry, but there will be differences between the two measurement methods and data should not be directly interchanged. In general, these gas absorption methods can measure smaller pore sizes than mercury porosimetry. Various instrument designs and operating conditions are utilized to determine pore characteristics with this method, and no single procedure or instrument type is specified. It is cautioned that data may not be directly comparable between different gas adsorption methods, although they may appear to be quite similar. Comparative data should always be generated with identical sample preparation, analysis, and data reduction procedures, in addition to utilizing equipment that is identical or near identical.

Test Methods and Reporting Conventions

Table 5-3 provides a summary of the various physical tests that are common for SCR and CO catalyst, along with the preferred testing method, when possible.

Table 5-3Common Physical Property Parameters and Test Methods

Parameter	Preferred Test Method	
Surface Area	Single-Point BET using Nitrogen	
Pore Volume and Distribution	Hg Porosimetry or Gas Adsorption	

Due to the non-standard nature of the testing for many of the physical parameters, it is very important for the test report to carefully delineate the testing methodology and data analysis techniques employed. As previously discussed, it is unlikely that physical property data would be translatable between different test methods, but a complete record of the test methodology may aid in future testing of the catalyst, as it ages for instance, or for troubleshooting scenarios, etc. Reporting of physical property testing should include the following parameters at a minimum.

- Measured parameter and units
- Detailed description of the test procedure
- Detailed description of the test apparatus²
- Specific test conditions employed
- Data interpretation method
- Sample preparation procedures
- Applicable QA/QC data and analyses

Chemical Properties

The measurement of chemical properties can aid significantly in the understanding of the deactivation mechanisms that are predominant for a particular field application and may serve as a predictor of catalyst deactivation problems prior to detection with actual activity measurements. In general, they are important in tracking the general "health" of a particular catalyst charge over time. Analysis of chemical properties is also important in troubleshooting scenarios, when the deactivation rate differs from that expected for a full-scale installation, for instance. However, due to the highly specific nature of testing for troubleshooting purposes, this guideline will be limited to chemical analyses that are performed as a manner of routine, typically conducted whenever catalyst is sampled.

Chemical analyses can be generally divided into two groups; bulk and surface. Each group of analyses has its own benefits. Bulk analyses provide highly quantitative measurements of the catalyst make-up, especially with respect to primary catalyst support materials and catalytically active species. Surface analyses generally provide data that are more qualitative in nature, but more sensitive to species which may be adsorbed from the flue gas, such as catalyst poisons, which by nature tend to be concentrated on the catalyst surface.

² Description of commercial equipment utilized should include manufacturer and specific model information.

Specific Analytes and Reporting Convention

The following two tables summarize the specific analytes for bulk and surface chemical analyses, which are identical. Table 5-4 gives the primary list of analytes that should be reported on a routine basis, while Table 5-5 gives a discretionary list of analytes. This discretionary list contains analytes that may be important for certain fuels or field applications but are not as commonly reported as those in the primary list. The reporting basis and units are also given in each table.

Element	Oxide Basis for Reporting	Units
Aluminum	Al ₂ O ₃	% by wt.
Arsenic	As	ppmw
Calcium	CaO	% by wt.
Chromium	Cr ₂ O ₃	ppmw
Iron	Fe ₂ O ₃	% by wt.
Magnesium	MgO	% by wt.
Molybdenum	MoO ₃	% by wt.
Palladium	Pd	% by wt.
Platinum	Pt	% by wt.
Phosphorus	P ₂ O ₅	ppmw
Potassium	K ₂ O	ppmw
Sodium	Na ₂ O	ppmw
Silicon	SiO ₂	% by wt.
Sulfur	SO ₃	% by wt.
Titanium	TiO ₂	% by wt.
Vanadium	V ₂ O ₅	% by wt.
Tungsten	WO ₃	% by wt.

Table 5-4Primary Bulk and Surface Chemical Analytes and Reporting Convention

 Table 5-5

 Additional Discretionary Bulk and Surface Analytes

Element	Oxide Basis for Reporting	Units
Barium	BaO ₃	ppmw
Boron	B ₂ O ₃	ppmw
Chlorine	ClO ₂	ppmw
Nickel	NiO ₂	ppmw
Niobium	Nb ₂ O ₅	ppmw
Thallium	Tl ₂ O	ppmw

The reporting of chemical properties should be as detailed as possible so that data can be properly interpreted, due to underlying differences in test methodology. The report should include the following parameters, at a minimum.

- Measured analytes, with reporting basis and units
- Sample preparation procedure³
- Detailed description of the test procedure
- Detailed description of the instruments utilized⁴
- Specific test conditions employed⁵
- Applicable QA/QC data and analyses

Bulk Chemical Analysis

Conventional bulk chemical analysis of SCR and CO catalyst involves the complete dissolution of the catalyst sample and subsequent determination of the analyte concentrations by spectroscopic and wet chemical methods. This methodology insures that the entire catalyst mass is evaluated. Alternately, a pseudo-bulk analysis can be made using surface analytical techniques applied to finely ground catalyst samples to generate an "average" elemental composition for the catalyst sample. The two methods will not typically yield identical results and direct comparisons of data between the two methods should not be made. Even under the grinding/powder sample preparation scenario in the latter method, data may exhibit an enrichment of surface constituents, since surface analysis techniques will typically not penetrate throughout individual particles. The following sections apply to the conventional determination of bulk catalyst chemistry, while the sections under "Surface Chemical Analysis" will generally apply to the latter method of

³ Detailed sample digestion procedures should be reported, if utilized.

⁴ Description of commercial equipment utilized should include manufacturer and specific model information, as well as minimum detection limit and accuracy specifications for each analyte reported.

⁵ Specific parameters such as accelerating voltage, photon strength, expected depth of penetration, etc. are especially important with surface analysis techniques and should be reported, when applicable.

determining bulk chemistry – only the sample preparation method differs from the surface analysis (although specific instrument settings, such as penetration depth may be altered).

Conventional Bulk Sample Preparation and Digestion

The SCR catalyst matrix can be difficult to digest. Thus, care must be taken to insure the complete dissolution of the samples prior to analysis to avoid erroneous results. No specific digestion method is required under this guideline, but methods such as hydrofluoric acid dissolution and lithium metaborate fusion are commonly used. In general, ASTM standard methods or equivalent should be utilized. In the case of honeycomb and corrugated catalyst samples, grinding of the sample prior to dissolution is common. In the case of metal foil CO catalysts, the active catalyst is typically physically removed from the metallic substrate prior to analysis. Some analytes such as chlorine and sulfur may not be measured via digestion, with the preferred analytical method being at the discretion of the laboratory. In all cases, consideration of the preparation technique, including digestion, on specific analytes should be considered, and reported if interferences are identified.

Conventional Bulk Chemical Analytical Technique

No specific analytical technique is required for the determination of specific analytes under this guideline. Particular laboratories will have preferred analytical methods based on available equipment, general preference, and personnel experience. However, the majority of the analytes will be determined using spectrophotometric methods such atomic absorption (AA) or inductively coupled plasma (ICP), while some species may be determined via wet chemical methods, such as chlorine and sulfur. In all cases, ASTM standard methods or equivalent should be followed when possible.

Surface Chemical Analysis

The surface analysis of catalyst samples provides complementary information to the bulk chemical data. As previously mentioned, surface analyses are generally more sensitive to materials that are deposited on the catalyst surface, such as catalyst poisons, etc. Various surface analysis techniques are discussed below. As mentioned above, a pseudo-bulk chemical analysis can be performed using ground samples and applying the following "surface" analysis techniques.

X-Ray Fluorescence Spectrometry

Currently, X-Ray Fluorescence Spectrometry (XRF) is the most common method of performing surface elemental analyses on SCR catalyst samples. XRF relies on the principle that atoms can release characteristic X-rays as a result of higher energy electrons transferring to lower energy orbital levels. This transference is in response to an electron having been previously ejected from the lower orbital level by the high-energy photon generated by the instrument. Each element will produce these X-rays with strengths characteristic of that particular element, and thus elemental species can be determined by measuring the emitted X-ray strength. Quantitative determination is made by counting the number of X-rays of any particular strength, thus relative counts are proportional to concentration.

A number of variations of XRF exist, and instruments will differ somewhat according to specific design. Furthermore, sample preparation technique, photon strength, data reduction techniques,

etc., will all affect XRF data. Thus, it is suggested that when direct comparisons are made between samples, identical sample preparation procedures, operational conditions and procedures, and instrument specifications and data reduction techniques be used.

XRF is attractive for a number of reasons, including speed, cost, and testing simplicity. Samples may be prepared in a number of ways. By grinding and pelletizing, a sample can be generated that represents an average or bulk composition (see above). Or, by selecting particular geometric surfaces, specific areas of the catalyst can be examined for surface constituents. This technique is of special value when examining specific poisons present to aid in the determination of deactivation mechanisms. In terms of the actual elemental analytes, XRF is attractive because it results in a scan of all elements present (within atomic weights of roughly 23 to 92, sodium to uranium). Thus, a specific list of analytes does not have to be determined prior to testing. This provides an excellent means of "canvassing" the elements present, possibly detecting abnormal species that might not have been specifically identified for quantification prior to testing. Although additional elements may be detected as part of the XRF analysis, the summary section defines the analytes that should be reported routinely.

Other Surface Analysis Techniques

A number of other techniques exist for the examination of surface constituents of solid materials. These include Electron Microprobe Analysis, Scanning Electron Microscopy (SEM), X-ray Photoelectron Spectroscopy (XPS), and XRF combined with SEM. Many of these may be especially helpful in troubleshooting scenarios for SCR and CO catalysts. Each technique will have its own particular advantages and disadvantages. Methods such as Electron Microprobe Analysis can provide maps of the catalyst surface with respect to specific elements such as arsenic or calcium, and can be quite useful in evaluation deactivation mechanisms, etc. By preparing cross-sectional samples, microprobe analysis can clearly show the depth of penetration of particular elements, such has catalyst poisons. X-ray diffraction can be used to identify crystalline compounds that may be present on the catalyst surface. SEM is also attractive because it allows a visual inspection of the catalyst surface, and can reveal the presence of fouling deposits, etc. When coupled with companion instruments, the SEM can be used to perform elemental scans similar to XRF. This can be particularly advantageous in troubleshooting scenarios, since particular deposits at the microscopic level can be targeted for analysis in conjunction with the viewing of the sample under the microscope. None of the above methods are required under this guideline but may be used on a discretionary basis according to specific need and preferences.

6 TECHNICAL DISCUSSION

This section contains technical discussions on key issues raised while developing the guidelines, including:

- NH₃/NO_x ratio for laboratory SCR catalyst activity testing
- NO₂/NO_x ratio impact on SCR catalyst activity and catalyst testing
- SO₂ to SO₃ conversion across CO and SCR catalysts
- NO to NO₂ conversion across CO catalyst
- CO Catalyst Testing Temperature and Space Velocity Considerations
- Effect of area velocity, inlet NO_x concentration and moisture on SCR catalyst activity
- Ammonia oxidation across SCR catalyst

NH₃/NO_x Ratio for Laboratory SCR Catalyst Activity Testing

For this discussion, consider the reaction of NO and NH₃ across the SCR catalyst requiring one mole of NH₃ to react with one mole of NO.

$$NO + NH_3 + \frac{1}{4}O_2 \to N_2 + \frac{3}{2}H_2O$$
(6-1)

The catalyst activity, K, is defined using a first-order reaction in terms of NO (i.e., NO reduction is independent of the inlet NO concentration) and zero order in terms of ammonia. The zero order assumption for ammonia essentially assumes full coverage of the active sites⁽⁵⁾ by ammonia (NH₃/NO > 1) such that at NH₃/NO> 1, the NO reduction is given by

$$\Delta \mathbf{NO} = 1 - e^{-K/Av} \tag{6-2}$$

With this definition, ideally the NO reduction will increase as the NH_3/NO ratio increases from 0 to 1.0. Then, ideally there will be no additional NO reduction as the NH_3/NO ratio is increased above 1.0.

For coal SCR catalyst, the laboratory testing protocols utilize $NH_3/NO_x = 1.0$ to determine the catalyst activity. This makes sense in terms of the first-order model introduced above. Also, coal-based SCR systems operate at NH_3/NO_x ratios less than 1.0. With an activity measured at $NH_3/NO_x = 1.0$, most catalyst managers have models that can describe the performance of the catalyst at NH_3/NO_x ratios less than 1.0. However, as discussed in the body of this report, this is not necessarily the case for combustion turbine SCR systems, and care must be taken in setting the NH_3/NO_x ratio for the laboratory testing.

For laboratory testing, the NH₃/NO_x ratio may be determined in a number of ways as follows:

• The preferred approach is to measure inlet NO_x , NO_x reduction across the catalyst sample, and NH_3 slip exiting the sample. A continuous ammonia analyzer is ideal for this approach, although wet chemical techniques are acceptable. The equation to calculate the NH_3/NO_x ratio with these measured values is shown below (all values ppmv), assuming zero NO_2 :

$$\frac{NH_3}{NO_x} = \frac{(NO_{x_{in}} - NO_{x_{out}}) + NH_{3_{slip}}}{NO_{x_{in}}}$$
(6-3)

• An alternate approach is to measure inlet NO_x (ppmv), flue gas flow (lb/hr) and NH₃ injection rate (lb/hr). An example of this calculation is shown below.

$$\frac{NH_3}{NO_x} = \frac{\dot{N}_{NH_3}}{\dot{N}_{NO_x}} \tag{6-4}$$

Where;

$$\begin{split} \dot{N}_{NH_3} &= ammonia \ inlet \ molar \ flow \ rate = \frac{\dot{M}_{NH_3}}{MW_{NH_3}} \\ \dot{M}_{NH_3} &= ammonia \ injection \ mass \ flow \ rate, \ lb/hr \\ MW_{NH_3} &= ammonia \ molecular \ weight \\ \dot{N}_{NO_{x \ in}} &= NOx \ inlet \ molar \ flow \ rate = \ \left(\frac{NO_{x-in} \ (ppmv)}{1 \ x \ 10^6}\right) \ x \ \frac{\dot{M}_{fg}}{MW_{fg}} \\ \dot{M}_{fg} &= flue \ gas \ mass \ flow \ rate, \ lb/hr \\ MW_{fg} &= flue \ gas \ molecular \ weight \end{split}$$

• A direct measurement approach is also possible, where inlet NH₃ (ppmv) and NOx (ppmv) are measured directly, with the NH₃/NOx ratio determined by dividing the inlet NH₃ by the inlet NOx, assuming that they two measurements are on the same basis (i.e. moisture and O₂). Direct measurement of inlet NH₃ may be challenging due to the high levels of NOx, and care must be taken to ensure that NH₃ conversion is not occurring prior to the actual measurement of the NH₃ concentration. Laboratories must perform appropriate QA/QC procedures to ensure the accurate determination of NH₃ under the test conditions.

Selecting the NH₃/NO_x Ratio for Laboratory Testing

The setting of the NH₃/NOx ratio is critical to accurate test measurements and applicability of data to the field application. The following discussions are designed in particular to help the end-user understand the implications of the NH₃/NOx ratio setpoint for testing, as well as how the resulting data correspond to field operation.

As with NOx and area velocity, it is important for both the laboratory and the customer to understand the impact of the NH₃/NOx ratio on resulting activity measurements. In most cases, the NH₃/NOx ratio used for testing will <u>not</u> correspond directly to the field NH₃/NOx ratio, unless coincidentally, since the field NH₃/NOx ratio will vary over time. Thus, even if an NH₃/NOx ratio is selected to match a particular field operating scenario, this will only apply to one specific point in time, and one specific operating condition. When only the <u>relative</u> activity is a concern (i.e., K/Ko) this parameter will be less affected by the selection of the NH₃/NOx ratio than will be the case where absolute deNOx activities are required. Ultimately, however, for

adequate modeling, and in most cases long-term tracking and life estimating, absolute deNOx activities must be determined, and thus an understanding of the effect of NH₃/NOx ratio on deNOx activity is required. Regardless of the selected NH₃/NOx ratio, laboratories should provide customers with the expected effect that this has on the activity measurements, disclosing quantitatively, if possible, what the expected deNOx activity would be at alternate NH₃/NOx ratios that may be appliable to specific operating scenarios, such as end-of-life. This allows for appropriate deNOx activities to be used which correspond directly to field performance.

Technically, testing at the field operating NH₃/NOx ratio, for which the activity is being determined, would give catalyst performance information that is most directly applicable to field operations. However, when the field NH₃/NOx ratio is high, and/or when the catalyst is fresh, there are concerns over data accuracy due to high NOx conversion during testing, which makes the accurate determination of the K-value difficult. Further, most facilities need to assess performance under a variety of operating conditions, each with their own actual field NH₃/NOx ratio due to differences in inlet and outlet NOx and expected slip according to those actual field operating conditions. In addition, the NH₃/NOx ratio will change over time as the catalyst ages for any specific operating condition. As a result, it is not obvious which NH₃/NOx ratio is most appropriate for testing, even if the target is the "field" NH₃/NOx ratio. In other words, there is no single NH₃/NOx ratio test condition which will serve all purposes. However, in almost all cases, the activity measurement can be "corrected" to other NH₃/NOx ratio values, <u>if</u> enough is known about the performance of the particular catalyst. Unfortunately, there does not appear to be a standard "correction curve" that would apply to all catalysts and all operating conditions that could be used to confidently translate activity measured at one NH₃/NOx ratio to another.

Table 6-1 shows some common combustion turbine SCR NH₃/NOx ratios based on various inlet NOx, outlet NOx, and NH₃ slip operating conditions (assuming zero NO₂ and no ammonia oxidation). The slip conditions shown generally correspond to common end-of-life slip limits and would be lower at earlier stages of the catalyst's life. For the vast majority of applications, the catalyst will operate in an NH₃/NOx ratio range of 1.0 to 2.0. Note that fresh catalyst will operate at a lower NH₃/NOx ratio, all other factors being equal, than when near the end of life, due to the increase in slip as the catalyst ages. Again, this indicates that the actual field NH₃/NOx ratio will change as a function of time, thus no single NH₃/NOx ratio applies to any particular catalyst and operating condition throughout its installed life.

Equation 6-5 can be used to calculate the expected field NH_3/NO_x ratio for specific NOx and ammonia slip conditions, assuming zero NO_2 and no ammonia oxidation. All values are in ppmv and should be on the same moisture and O_2 basis.

$$\frac{NH_3}{NO_x} = \frac{(NO_{x_{in}} - NO_{x_{out}}) + NH_{3_{slip}}}{NO_{x_{in}}}$$
(6-5)

Inlet NOx (ppm)	Outlet NOx (ppm)	NH3 Slip (ppm)	NH3/NOx (ppm)
	10	10	1.00
40		5	0.88
40	5	10	1.13
		5	1.00
	5	10	1.20
25		5	1.00
25	2	10	1.32
		5	1.12
15	2	10	1.53
		5	1.20
12	2	10	1.67
		5	1.25
10	2	10	1.80
		5	1.30
8	2	10	2.00
		5	1.38

Table 6-1 Common Combustion Turbine SCR NH₃/NO_x Ratios

Figure 6-1 shows a <u>conceptual</u> plot of how measured deNOx, and the resulting calculated activity, vary as a function of NH₃/NOx ratio. In practice each catalyst will have a slightly different relative response, which may change according to catalyst design, age, and test conditions (e.g., temperature, flow rate, etc.). For this example, the catalyst achieves 90% deNOx at an NH₃/NOx ratio of 1.2. This corresponds to a calculated activity of 80 m/hr (for an assumed AV of 35 m/hr). Again, this is a hypothetical example with nominal deNOx and activity values and should not be applied to any particular field installation.

As indicated in the plot, deNOx typically responds linearly to increases in NH₃/NOx ratio up to roughly 0.75 or above (for a typical combustion turbine catalyst formulation and potential), then levels off at higher NH₃/NOx ratios, usually demonstrating a prominent "knee" around an NH_3/NOx ratio of roughly 1.0. At higher NH_3/NOx ratios, the response tends to be relatively linear above $NH_3/NOx = 1.2$. The corresponding activity, which is usually measured/calculated at $NH_3/NOx \ge 1.0$ responds similarly. In any event, there is typically a significant difference in measured activity across the range of $NH_3/NOx = 1.0$ to 2.0. For example, based on the hypothetical data in Figure 6-1, one can determine that an activity value measured at NH₃/NOx ratio of 2.0 (87 m/hr) will be approximately 15% higher than the activity measured at NH $_3$ /NOx ratio = 1.0 (72 m/hr). Again, this is a hypothetical example and is not necessarily applicable to any particular test data or field application. Consequently, if the purpose of testing is to predict the life of a particular catalyst installation, if the activity tests are performed at an NH₃/NOx ratio = 1.0, but the field installation operates at an NH₃/NOx ratio = 2.0 at the end of life, then the measured activity will underestimate the actual field activity. Conversely, for example, if the activity is being estimated real-time when the actual field NH₃/NOx ratio is relatively low, measuring the activity at $NH_3/NOx = 1.2$ will overstate the activity. It is also important to note that according to Figure 6-1, the activity response to NH₃/NOx ratio is much more sensitive at

values close to $NH_3/NOx = 1.0$ than at higher values. For field systems that actually operate at NH_3/NOx ratios near 1.0, end-users should be aware that even small deviations in NH_3/NOx ratio between the test conditions and the field conditions can lead to substantial differences in the determined K-value.

Considering the intricacies of selecting the NH₃/NOx ratio, and typical laboratory preferred "standard" NH₃/NOx ratio values for testing, an NH₃/NOx ratio of 1.2 has been selected as the standard condition for this protocol. Alternate values are allowed, however, and laboratories and customers may choose values based upon their needs. In particular, if data accuracy is not a concern, customers may choose to test at the NH₃/NOx ratio consistent with the end-of-life "design" condition for the SCR. Typically this design condition is the worst-case, or "controlling" condition for the SCR, and testing at an NH₃/NOx ratio consistent with this design condition can provide very useful information, especially if the focus of the testing is to predict the end-of-life for the catalyst. Use of alternate NH₃/NOx ratios should meet the required criteria for the utilization of alternate test conditions as set forth in Chapter 3. And in all cases, even when the standard NH₃/NOx ratio on test results to help the end-user understand the applicability of the test data to field operations.



Figure 6-1 Conceptual Laboratory NO_x Reduction vs. NH₃/NO_x and Calculated Catalyst Activity

NO₂/NO_x Ratio Impact on SCR Catalyst Activity and Catalyst Testing

NO_x entering combustion turbine SCR catalyst may have a significant fraction of NO₂. The reactions between NH₃, NO, and NO₂ are shown below:

$$NO + NH_3 + \frac{1}{4}O_2 \rightarrow N_2 + \frac{3}{2}H_2O$$
(6-6)

$$6NO_2 + 8NH_3 \rightarrow 7N_2 + 12H_2O$$
 (6-7)

$$NO_2 + 2NH_3 + \frac{1}{2}O_2 \rightarrow \frac{3}{2}N_2 + \frac{3}{4}O$$
(6-8)

$$NO + NO_2 + 2NH_3 \rightarrow 2N_2 + 2H_2O \tag{6-9}$$

Reaction (6-9), referred to as the "Fast SCR Reaction," occurs when there is an equimolar ratio of NO and NO₂ (i.e., NO₂/NO_x = 0.5) entering the catalyst.

There are two issues associated with these higher fractions of NO_2 in the flue gas. First, the overall stoichiometry for the NO_x reduction across the catalyst may vary from 1.0 to 2.0, depending on the amount of NO_2 present and the reaction that is dominant.

Second, the catalyst activity depends on the NO₂/NO_x ratio entering the catalyst ⁽⁶⁻¹⁰⁾. This is illustrated in Figure 6-2⁽¹⁴⁾ which shows that up to a 50% proportion of NO₂ (i.e., NO₂/NO_x = 0.5) the activity will increase with increasing NO₂ proportion. Above 50% proportion of NO₂, however, the activity will decline markedly. The exact nature of the activity response to NO₂ proportion is temperature-dependent, as demonstrated in the plot, especially at high NO₂ proportions.



Figure 6-2 Effect of NO₂/NO_x Ratio on Catalyst Performance

The above two issues raise the following questions regarding laboratory tests:

- Should NO₂ be included in the laboratory flue gas?
- If so, how much? (field level, or a reference level?)
- If NO₂ is present, what NH₃/NO_x ratio should be used to measure catalyst activity? Figure 6-3 shows how the stoichiometry varies for Equations 6-7 and 6-8 as the proportion of NO₂ in the flue gas varies.
- For a given combustion turbine SCR, is the NO₂/NO_x ratio entering the SCR catalyst known?

Since it is unlikely the actual field NO_2/NO_x ratio at the catalyst inlet is known for a given unit, and considering the ambiguity of the NH_3/NO_x stoichiometry for the reactions with a mixture of NO and NO_2 , the current guidelines do not require simulation of the actual inlet NO_2/NO_x ratio, and laboratory NO_2 fractions less than 5% are acceptable. However, if the NO_2/NO_x ratio for a particular field installation is known to be high, it is recommended at the field ratio to improve the applicability of the data to the field installation. This is especially true in cases where the field NO_2/NO_x ratio is > 50%, since in these cases, measured catalyst performance can be significantly overstated if the test conditions do not include NO_2 .



Figure 6-3 Effect of NO₂ on the SCR Stoichiometry

SO₂ to SO₃ Conversion Across CO and SCR Catalysts

Both CO and SCR catalysts oxidize SO_2 to SO_3 . The measurement of SO_2 to SO_3 across CO or SCR catalyst is not a formal part of these guidelines. However, some discussion is provided to outline the issues and provide guidance if desired.

While sulfur content in natural gas is typically low, even low levels over long time periods have caused salt deposition issues in some HRSGs. Sulfur levels in natural gas will vary regionally, but SO₂ levels leaving the combustion turbine will typically be less than 1 ppm (by volume). Assuming an oxidation rate of 20%, measuring the change in SO₂ or SO₃ across a catalyst sample would require resolution better than 0.2 ppm. With current instrumentation used by most laboratories (continuous SO₂ analyzer or EPA Method 8A, Controlled Condensate), this is not possible. If the oxidation rate must be measured, SO₂ levels may be increased entering the catalyst sample to nominally 100 ppm, thereby increasing SO₃ concentrations at the outlet. However, it is important to first consult with the catalyst supplier, since 1) the oxidation rate may

be dependent on the SO₂ level entering the catalyst, and 2) this test may poison the CO catalyst sample.

NO to NO₂ Oxidation across CO Catalyst

CO catalyst may oxidize some fraction of NO exiting the combustion turbine to NO_2 . While not a formal part of this guideline, it is a relatively easy measurement to make in the laboratory if desired (as described in an earlier section). Most laboratories will have a chemiluminescent NO_x analyzer available which incorporates a NO_2 to NO converter. The analyzer may be used to measure the NO and NO_x at the inlet and outlet of the CO catalyst and the level of oxidation may be calculated. Other instrumentation is also available such as an FTIR where the NO and NO_2 components are measured separately. At present, the behavior of typical CO catalysts with respect to NO to NO_2 oxidation is not well understood. This has been included as an area for future work in Chapter 7.

CO Catalyst Testing Temperature and Space Velocity Considerations

Two key concerns for CO catalyst testing involve the ability of the laboratory to achieve either 1) a full range of temperatures, or 2) the field space velocity.

Temperature

Temperature range tests require careful consideration due to CO catalyst deactivation behavior. Figure 6-4⁽¹¹⁾ shows typical examples of CO deactivation. The light-off curve may vary markedly depending on the nature of deactivation. Using the curves shown in the figure, if the field operating temperature is 1000°F (538°C) and a particular laboratory can only achieve a temperature of 800°F (427°C), there may be a small amount of error in the results if deactivation follows the "deactivated" (red), or "fouled" (green) data. However, there may be a larger error if deactivation followed the "dotted" sulfur contamination curve. The end-user and laboratory must be aware of this possible problem.


Figure 6-4 Deactivation Characteristics of CO Catalyst

The above is an extreme case. If the catalyst is operating in the field at 800-850°F (427-454°C) and the laboratory can only achieve 750°F (399°C), there may only be a few percentage points difference. These issues must be addressed on a case-by-case basis.

Space Velocity

As discussed previously, if a laboratory is not able to duplicate field space velocity conditions, an allowable approach is to assume that the CO oxidation is a first order reaction, similar to the SCR reaction. This leads to an equation for the CO oxidation as follows:

$$\Delta \mathbf{CO}_{\mathbf{lab}} = 1 - e^{-K_{\mathbf{CO}}/SV_{\mathbf{lab}}}$$
(6-10)

where SV_{lab} is the space velocity in the laboratory and K_{CO} is a "CO activity" that can be derived from the laboratory test. Note, in this case, the activity is based on space velocity, not area velocity. The field CO oxidation may then be calculated using the field space velocity and the K_{CO} . The above equation should be used to correct for small deviations in space velocity between the laboratory and full-scale for a particular catalyst geometry. This equation is not intended to compare different CO catalysts.

Effect of Area Velocity, Inlet NO_x Concentration, and Moisture on SCR Catalyst Activity

During the development of the first iteration of these guidelines, EPRI performed laboratory studies to investigate SCR catalyst activity sensitivities to area velocity, inlet NOx and moisture. The background and conclusions from the studies are discussed below.

Area Velocity

For combustion turbine catalyst samples, high activity values may lead to measured NO_x reductions greater than 95% at field area velocity conditions. This generates a large uncertainty in the calculated activity, which may be mitigated by requiring laboratory tests at an area velocity higher than the field value. However, a key question emerges: Does a change in area velocity introduce a bias in the measured activity, since the activity is dependent on flow conditions within the catalyst channel as well as the material?

To answer the question, it is important to consider fluid dynamics in the catalyst cell. When flue gas enters the catalyst, the flow is turbulent and will then transition to laminar along the length of the channel. The mass transfer in the turbulent region will be higher than in the laminar region, and higher mass transfer rates may lead to higher activity. Thus, if a catalyst sample is shortened to increase the area velocity, the flow over a larger fraction of the sample length will be turbulent. This may bias the activity to a higher value. Likewise, if the full-length element is used and the flow rate is increased to adjust area velocity, velocity in the catalyst cells will increase. This will lead to higher mass transfer rates and a possible increase in activity. As discussed below, laboratory tests were performed to determine if these potential biases are significant.

Area Velocity Test Results

Tests were conducted in a micro reactor and a bench reactor to investigate area velocity effects on measured activity. In both facilities, the area velocity was adjusted by changing the flow through a fixed-length catalyst sample, and/or changing the length of the sample at a fixed flow. The results are also plotted in Figure 6-5, along with predictive model data. Tables 6-2 and 6-3 provide the conditions and results for the bench and micro reactor tests, respectively. Three different catalyst blocks (new OEM catalyst, similar catalyst types and geometries) were tested in the bench reactor (sample ID numbers 306, 351 and 360). Two core samples were tested in the micro reactor, both taken from a single catalyst block (new OEM catalyst, same catalyst type and geometry as the bench samples). Tests 1-3 in the micro reactor were performed with the first core sample, and Tests 4A-4C and 5A-5C using the second sample.

The model predictions in Figure 6-5 utilize the mass transfer correlations suggested by Tranconi and Beretta⁽¹²⁾. The predictions indicate an increase in activity with increasing area velocity, either by increasing the flow or shortening the sample length. (Note that shortening of the catalyst is not recommended in this guideline as a means for adjusting AV for test accuracy improvements.) The activity increase is small (nominally 3%) over an area velocity range of 25 to 45 m/hr. As discussed earlier, if a catalyst sample is shortened to increase the area velocity, the flow over a larger fraction of the sample length will be turbulent and will result in higher mass transfer rates and activity. Likewise, if the flow rate is increased, the mass transfer rate will also be higher due to the increased velocity in the catalyst cells.

The experimental results, however, did not consistently follow the model predictions. It was apparent that uncertainty in the measured activity values overshadowed any measurable sensitivities caused by area velocity variations. As discussed in the appendix, an uncertainty on the order of ± 2.3 m/hr (2.7%) is not unreasonable for activity measurements at these test conditions. This relative uncertainty is comparable to the activity sensitivity discussed earlier. Ultimately, the experimental results demonstrated the relative insensitivity of the activity







Table 6	5-2		
Bench	Reactor Area	Velocity	Tests

Test		1	2	3	1B	4B	4C	5B	5C
Date		4/27/2015	4/28/2015	4/28/2015	7/22/2015	7/22/2015	8/12/2015	8/14/2015	8/12/2015
Sample ID	-	306	306	306	351	360	360	351	360
Sample Length	mm	283.5	283.5	283.5	285.0	192.0	192.0	151.4	147.3
Temperature	F	644	645	645	647	643	643	644	644
Flow	N m3/h	156.4	231.0	297.0	156.5	164.9	164.9	156.4	165.0
LV	m/s	5.00	7.38	9.49	5.01	5.00	5.00	5.01	5.01
AV	m/h	23.68	34.97	44.96	23.59	34.99	34.98	44.38	45.62
02	vol%, dry	12.1	12.2	12.1	12.1	12.0	12.1	11.9	12.3
H2O	vol%	7.3	7.0	7.0	6.9	7.1	7.0	6.8	7.0
NOx	ppmv, wet	93.9	93.2	94.4	92.8	93.8	93.6	91.6	92.8
NH3	ppmv, wet	112.8	112.2	113.4	113.9	112.5	112.2	111.6	111.5
NH3/NOx	-	1.201	1.204	1.201	1.227	1.199	1.199	1.218	1.202
SO2	ppmv, wet	0.0	0.0	0.0	0.7	0.1	0.4	0.0	0.2
deNOx	%	97.50	91.75	85.40	97.30	91.61	92.50	85.31	86.43
activity, k	m/h	87.4	87.3	86.5	85.2	86.7	90.6	85.1	91.1
dP	in WC	1.13	1.73	2.28	1.01	0.84	0.84	0.66	0.66

Table 6-3Micro Reactor Area Velocity Tests

Test		1	2	3	4A	4B	4C	5A	5B	5C
Date		7/28/2015	7/29/2015	7/30/2015	7/31/2015	7/31/2015	7/31/2015	8/3/2015	8/3/2015	8/3/2015
Sample Length	mm	165.1	152.4	127	152.4	152.4	152.4	152.4	152.4	152.4
Temperature	F	646	643	637	642	645	647	644	645	645
Flow	scfm	0.832	0.840	0.843	1.107	0.980	0.828	0.838	0.988	1.116
AV	m/h	30.2	32.9	39.7	43.6	38.5	32.6	33.0	38.9	43.9
02	vol%, dry	15.1	15.1	15.0	14.9	15.0	15.0	15.0	15.0	14.9
H2O	vol%	7.0	7.1	7.0	6.8	7.0	7.3	7.0	6.8	6.7
NOx	ppmv, dry	101.5	101.3	102.7	97.6	99.2	102.1	100.3	99.2	98.7
NH3	ppmv, dry	121.8	120.9	123.1	119.0	119.6	122.1	120.5	119.0	118.8
NH3/NOx	-	1.200	1.193	1.199	1.220	1.205	1.196	1.201	1.200	1.203
deNOx	%	94.9	93.3	89.2	86.8	89.1	92.4	92.4	89.20	86.8
Activity, K	m/h	89.9	88.9	88.4	88.3	85.3	84.0	85.0	86.6	88.9

Inlet NO_x Level

Another key question is whether NO_x reduction is always first order in NO_x and zero order in NH_3 . Laboratory tests were performed to determine potential impacts of varying laboratory inlet NOx levels for the purpose of improving NOx measurement accuracy.

Inlet NO_x Level Test Results

The activity equation assumes the SCR reaction is first order in terms of inlet NO_x (i.e., independent) and zero order in terms of NH_3 . This was investigated previously for the original guideline using a micro-reactor. The tests were conducted using the following nominal conditions:

Temperature:	342°C (647°F)
O ₂ :	15%
NO _x :	5 – 100 ppm
Catalyst Length:	152mm (6 inches)
Area Velocity:	45 m/hr
H_2O :	7.8%

The area velocity was set at a relatively high value (45 m/hr) to avoid high levels of NO_x reduction (and thus higher uncertainty). The results are plotted in Figure 6-6, and data are shown in Table 6-4. Three sets of tests were performed with the same catalyst sample on three separate days. The results show the activity was not independent of the inlet NO_x level. Reducing the NOx inlet from 100 ppm to 20 ppm decreased the activity by about 14%. Below 20 ppm, the effect was more pronounced. This shows the first-order NO_x and zero-order NH_3 assumptions leading to the definition of activity are overly simplified⁽⁵⁾. If tests are conducted at NO_x levels markedly different than field levels, the measured activity may be impacted.



Figure 6-6 Micro Reactor Tests: Effect of Inlet NO_x Level

Table 6-4 Micro Reactor Inlet NO_x Tests

Test No.	O ₂ (%)	H ₂ O (%)	NH ₃ /NO	Temp (°F)	Inlet NO (ppmv @15% O ₂)	Outlet NO (ppmv @15% O ₂)	dNOx (%)	Area Velocity (m/hr)	Activity (m/hr)
1	15.0	7.8	1.18	648	4.9	1.1	77.8	45.0	67.8
	15.1	7.9	1.20	647	10.0	2.0	80.3	45.0	73.0
	15.0	8.2	1.20	646	19.7	3.5	82.1	45.0	77.3
	14.8	7.4	1.18	648	99.3	13.4	86.5	45.0	90.0
2	14.9	7.0	1.20	649	6.1	1.6	73.7	45.4	60.5
	14.9	7.3	1.19	647	11.1	2.6	76.3	45.4	65.3
	14.9	7.2	1.21	648	21.9	4.1	81.2	45.4	75.9
	14.9	7.1	1.20	648	43.7	6.8	84.4	45.4	84.2
	14.9	7.1	1.20	649	86.8	12.5	85.6	45.4	88.0
3	14.8	7.2	1.20	647	6.0	1.6	74.4	44.9	61.1
	14.8	7.2	1.20	647	11.1	2.3	79.0	44.9	70.0
	14.8	7.2	1.20	648	21.6	3.8	82.6	44.9	78.6
	14.8	7.2	1.20	648	43.0	6.5	84.8	44.9	84.7
	14.9	7.1	1.21	649	86.7	12.0	86.2	44.9	89.0

Flue Gas Water Vapor Content

In the EPRI coal catalyst testing protocol⁽³⁾, a correction curve is provided if the laboratory and field water vapor contents differ. This curve is shown in Figure 6-7. The equation for the curve (normalized to 1.0 at 0% H₂O) is shown below. Laboratory tests were performed to validate this curve as discussed below.





Figure 6-7 Moisture Corrective Curve⁽³⁾

Flue Gas Moisture Test Results

A series of tests were conducted in a micro reactor using a new combustion turbine SCR catalyst sample. These tests were conducted at the following conditions:

Temperature:	340°C (643°F)
O ₂ :	15% dry
NO _x :	100 ppm dry
Catalyst Length:	152mm (6 inches)
Area Velocity:	35 m/hr
H_2O :	3 – 15%

The results are shown in Figure 6-8 and show good agreement with the water correction curve shown above. An increase in water content from 3% to 15% decreases the activity by 7 m/hr (approximately 8%).



Figure 6-8 Micro-scale Tests: Effect of H₂O Content on Activity

Ammonia Oxidation Across SCR Catalysts

SCR catalysts can promote ammonia oxidation as a function of operating conditions and catalyst design.⁽¹³⁾ This guideline does not require the measurement of ammonia oxidation, or require a testing approach which would necessarily detect ammonia oxidation. However, it is important for laboratories and end-users to understand the implications of ammonia oxidation, and to take steps to evaluate it if needed. Ammonia oxidation by a catalyst is not necessarily considered a catalyst failure or detractor, but it can influence catalyst performance, and the impacts should be understood if appreciable oxidation is occurring.

For catalysts that do promote ammonia oxidation, this is most appreciable at temperatures >800 °F. If ammonia oxidation is occurring during a laboratory test, it can cause misinterpretation of the laboratory data, as well as inconsistencies between the laboratory test conditions and the target field conditions. For instance, if laboratory testing is designed to simulate a target field NH₃/NOx ratio (at end of life, for instance), but ammonia oxidation is occurring and is not accounted for, the laboratory test will be operated at an NH₃/NOx ratio that is actually lower than the field, resulting in an activity measurement that is lower than the practical field operating activity, resulting in a predicted end of life that is actually sooner than is actually the case. For example, Table 6-5 shows the differences between two hypothetical cases, one with ammonia oxidation, and one without ammonia oxidation. For this example, it is assumed that the laboratory testing will be performed at the calculated end-of-life NH₃/NOx ratio for a unit operating at 16 ppm inlet NOx, 2 ppm outlet NOx, and an end of life ammonia slip of 10 ppm. For Case A, where it is assumed that no ammonia oxidation is occurring, the calculated

NH₃/NOx ratio would be 1.5, calculated simply as the ammonia demand for deNOx plus the ammonia slip, divided by the inlet NOx ((14+10)/16=1.5). For Case B, it is assumed that 20% of the inlet ammonia is lost to oxidation, and thus the NH₃/NOx ratio must be higher to compensate for this loss. For this case, the inlet NH₃/NOx ratio would be the deNOx demand, plus the ammonia slip, <u>plus</u> the oxidized ammonia, divided by the inlet NOx ((14+10+6)/16=1.88). Assuming that the NH₃/NOx ratio for this example test is set directly (by either measuring inlet NH₃ as ppm, or computing the ppm by known flow rates), if ammonia oxidation is assumed to be zero then the test NH₃/NOx ratio would be set to 1.5. However, if in practice the catalyst was actually oxidizing 20% of the available inlet ammonia (Case B) then the actual field NH₃/NOx ratio would be performed at an NH₃/NOx ratio substantially lower than the actual field value and the deNOx activity generated by the test would be underestimated as compared to field performance. This would cause the predicted life to be shorter than would actually be the case.

Parameter	Case A (no ammonia oxidation)	Case B (20% ammonia oxidation)
Inlet NOx (ppm)	16	16
Outlet NOx (ppm)	2	2
NH₃ Slip (ppm)	10	10
NH ₃ Demand for DeNOx	14	14
NH ₃ Oxidation Rate	0%	20%
NH ₃ Oxidized (ppm)	0	6
Total NH₃ In (ppm)	24	30
NH₃/NOx Ratio (inlet)	1.50	1.88

Table 6-5

Example Calculation of NH ₃ /NOx Ratio with and without Ammonia Oxidatio

In practice, the ability of a laboratory test to detect ammonia oxidation will be dependent on the specific test approach and the data acquired. Specifically, approaches that do not fully close the ammonia-NOx material balance will not be able to detect ammonia oxidation. For example, if the inlet NH₃/NOx ratio is set directly to some target value by directly measuring the inlet NH₃ concentration or calculating it based on flow rates, and slip is not measured for the test then the material balance will not be fully closed (i.e., the actual ammonia consumption cannot be detected. However, if the ammonia slip is measured). In this case, ammonia oxidation will not be detected, and this can then be compared to the ammonia demand from deNOx. Any discrepancy would be attributed to ammonia oxidation, assuming there is confidence in the measurements.

Note that as discussed in Chapter 3, testing at a low NH₃/NOx ratio (e.g. 0.5) can alleviate the need to measure ammonia slip to fully close the material balance, since at low NH₃/NOx ratios the ammonia slip can be assumed to be at or near zero for a typical installation. Using this low NH₃/NOx ratio testing approach as part of a routine QA/QC procedure is highly recommended and can help to identify if significant ammonia oxidation is occurring. In cases where deNOx is measured (the alternate catalyst testing approach where inlet ammonia is adjusted to attain the target slip limit at a given inlet NOx value), if the ammonia at the inlet is not determined, the material balance cannot be closed, and ammonia oxidation cannot be detected.

In addition to potential discrepancies between test and field conditions, one practical implication of ammonia oxidation is that the ammonia consumption for a given installation at a given operating condition will increase if ammonia oxidation is occurring. This is due to the loss of ammonia to oxidation, which must be compensated for by increasing the ammonia flow to the reactor. This has implications in terms of both cost and maximum design ammonia flow rates for the unit.

7 FUTURE WORK

The development of this guideline has highlighted the need for future work in several areas as discussed below. The completion of this work would add greatly to the body of knowledge and aid end-users in accurately assessing combustion turbine catalysts using laboratory data.

Round-Robin Testing

Round-robin testing allows for data produced at different laboratories to be compared for accuracy and consistency. Previously, EPRI performed a round-robin study on coal-fired boiler catalysts which provided important information related to the accuracy of test results and comparability of data between several different commercial laboratories using several different coal-fired SCR catalysts.¹⁵ A similar test program for combustion turbine SCR and CO catalysts would be helpful in determining the typical variability between laboratories for various catalyst samples at the same conditions. This would help to establish the "error" band associated with laboratory tests, and help to determine if any inherent differences between test reactors/facilities are present. This would add confidence for the end-user when applying laboratory data to field installations, especially in terms of accurately predicting catalyst end of life.

Effect of Inlet NOx on Measured DeNOx Activity

The discussions and data in this guideline clearly show that laboratory data for SCR catalysts will be sensitive to the inlet NOx value used in testing. Although increases in inlet NOx compared to the field value are commonly employed in laboratory testing, at present it is unclear how consistent this sensitivity is between different catalysts (e.g., differences in geometry, formulation, etc.) and if other operating conditions affect this sensitivity and to what degree (e.g., NH₃/NOx ratio, temperature, etc.). Data for multiple catalysts and possibly multiple operating conditions would help to establish, 1) what the expected range of sensitivity is, and 2) if a "standard" correction curve could be used for multiple catalysts/conditions. These findings would help to reduce the current unknown impacts of using higher inlet NOx values during testing for specific catalysts at specific test conditions, and therefore add to the confidence for which laboratory data can be applied to the field.

Effect of NH₃/NOx Ratio on Measured DeNOx Activity

Similar to the above, this guideline demonstrates that there will be an impact on deNOx activity measurements for SCR catalysts as a function of the NH₃/NOx ratio used during testing. The industry consensus is that this sensitivity will vary according to the specific catalyst, and possibly other test conditions. A better understanding of this sensitivity for multiple catalysts, and at multiple operating conditions, would help end-users in several ways, including, 1) determining what NH₃/NOx ratio setpoint is most appropriate for laboratory testing, and 2) if a standard "correction" curve could be used that would provide reasonable accuracy when adjusting data from one NH₃/NOx ratio to another. If a standard curve (or set of curves) can be applied with reasonable accuracy, this would be extremely beneficial to end-users, increasing the

applicability of laboratory to the field for various purposes, as well as potentially reducing testing costs.

NO to NO₂ Oxidation Across SCR and CO Catalysts

CO catalysts, and to a lesser degree SCR catalysts, may oxidize some fraction of NO exiting the combustion turbine to NO_2 . Data indicate that this can be substantial, as much as 60-70% for some CO catalysts. In the case of SCR, some data have also shown the chemical reduction in NO_2 to NO. At present it is not well understood how different catalysts behave with respect to NO and NO_2 oxidation/reduction, or how the operating conditions affect this behavior. Testing with multiple catalysts and at multiple test conditions would help to better understand the behavior of common catalysts with respect to NO to NO_2 oxidation.

Formaldehyde and Other Volatile Organic Compounds (VOCs) Conversion Across CO Catalysts

VOCs, and formaldehyde in particular, are a significant source of hazardous air pollutants (HAPs) associated with combustion turbines. Generally applicable current federal limits for formaldehyde emissions associated with utility combustion turbines are 91 ppbvd (15% O₂).¹⁶ The current guideline does not provide a methodology for VOC testing (including formaldehyde), although many of the principles applied to CO oxidation testing will also apply to VOCs. Historically, it has been assumed that CO oxidation would be a reasonable surrogate for assessing VOC conversion. However, this assumption has been challenged,^{17,18} and ultimately VOC conversion is not well understood, especially as it relates to the specific VOC being converted (e.g., formaldehyde, methane, ethane, etc.). In addition, catalyst specifics and operating conditions will affect the conversion. In particular, the VOC conversion behavior of the catalysts as they age, and with respect to CO oxidation, is of importance. Further testing to better quantify VOC conversion behavior, especially with aged catalysts, would greatly improve the industry's knowledge base, and help to ensure that VOC regulatory limits are consistently met.

Multi-Function Catalyst and Catalysts in Series

This guideline provides for testing of SCR and CO catalysts independently. However, multifunction catalysts combine the deNOx and CO oxidation functionality into one integral catalyst. As a result, it is not clear if the independent testing approach prescribed in the guideline would be adequate for multifunction catalysts. For instance, in this guideline the independent testing of CO catalyst does not require the presence of ammonia (this is consistent with the field conditions for a CO catalyst placed upstream of ammonia injection and the SCR catalyst). However, in a multifunction catalyst application, ammonia will be present at the catalyst inlet, since it is required for the deNOx reaction. As a result, if the ammonia has an impact on the CO oxidation, testing without it would provide inaccurate results. To some degree, a similar concern arises when independent catalysts are applied in series. For instance, in a typical field installation which includes both CO catalyst and SCR catalyst, the CO catalyst will be placed upstream of the SCR catalyst, with ammonia injection occurring between the two catalysts. However, if the CO catalyst is affecting flue gas constituents such that they influence the behavior of the SCR catalyst, lab testing would not likely take this into account unless the "field" reference conditions specially considered this. For instance, if an upstream CO catalyst is converting a high proportion of NO to NO₂, this may have an effect on the resulting performance of the

downstream deNOx catalyst. Thus, in an independent testing scenario, where the upstream catalyst's behavior is not considered, this could result in data that are not applicable to the actual field operations. Further work is needed to better understand the appropriate testing approach for multifunction catalysts, as well as to better understand the impacts of catalyst placed in series.

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A UNCERTAINTY ANALYSIS

An uncertainty analysis was performed on the calculation of combustion turbine SCR catalyst activity. SCR activity is calculated from the following equation:

$$K = \frac{Q}{V_{cat}Asp} \ln(1 - \Delta NO_{\chi})$$
(A-1)

$$K = \frac{Q}{d^2 L A s p} \ln\left(\frac{N O_{Xo}}{N O_{Xin}}\right) \tag{A-2}$$

K = catalyst activity, m/hr Q = gas flow rate Nm³/hr d = sample cross section dimension, m L = sample length, m Asp = sample specific surface area, m²/m³ NO_{x0} = outlet NO_x measurement, ppm NO_{xi} = inlet NO_x measurement, ppm

The uncertainty in the measurement of K is given by the following expression

$$W_{K} = \left[\sum_{i} \left(\frac{\partial K}{\partial X_{i}} W_{i}\right)^{2}\right]^{1/2}$$
(A-3)

Table A-1 shows the partial derivatives of equation (A-2).

Equation (A-3) and the partial derivatives shown in Table A-1 were used to assess the importance of each parameter in determining K. The results are shown in Figure A-1 for an activity of 85 m/hr and three area velocities; 20 m/hr, 35 m/hr, and 45 m/hr. The following can be seen in Figure A-1:

- With the exception of the outlet NO_x levels, the uncertainty associated with the other parameters is relatively independent of the area velocity. This is due to the high NO_x reduction and low outlet NO_x at the low area velocity.
- Reasonable levels of uncertainty with the flow (Q), cell opening (d), length (L), and inlet NO_x impact the activity by nominally 1-2 m/hr.
- An uncertainty in the specific surface area can have a large impact on the activity. However, in most cases, laboratories will be using a supplier supplied value so this, in effect, is not an experimental uncertainty.

If the uncertainties in each parameter are as shown in Table A-2, the overall uncertainty in K would be nominally 2.5-3%, (2.2 - 2.5 m/hr) with the higher value corresponding to the low area velocity, 20 m/hr.

Table A-1 Partial Derivatives for Determining the Uncertainty in K

Parameter	∂K
	$\overline{\partial X_i}$
Q	$\partial K = 1 \left(NO_{x_o} \right)$
	$\frac{\partial Q}{\partial Q} = \frac{\partial Q}{\partial a^2 L A s p} \ln \left(\frac{\partial N O_{x_i}}{\partial x_i} \right)$
Asp	$\partial K = Q = (NO_{x_o})$
	$\frac{\partial Asp}{\partial Asp} = -\frac{\partial^2 L A_{sp}^2}{\partial^2 L A_{sp}^2} \ln\left(\frac{\partial O_{x_i}}{\partial N O_{x_i}}\right)$
L	$\partial K = 1 \left(NO_{x_0} \right)$
	$\frac{\partial L}{\partial L} = -\frac{1}{d^2 L^2 A s p} \ln\left(\frac{1}{NO_{x_i}}\right)$
d	$\partial K = 2Q (NO_{x_0})$
	$\frac{\partial d}{\partial d} = -\frac{\partial^2 L}{d^2 L A s p} \ln\left(\frac{\partial O_{x_i}}{\partial x_i}\right)$
NO_{x_0}	$\partial K \qquad Q$
	$\frac{\partial NO_{x_0}}{\partial L Asp NO_{x_0}} = \frac{\partial^2 L Asp NO_{x_0}}{\partial L Asp NO_{x_0}}$
NO_{x_i}	$\partial K = Q$
L	$\frac{\partial NO_{x_i}}{\partial IO_{x_i}} = \frac{\partial LASP NO_{x_i}}{\partial I}$

Table A-2				
Assumed Uncertainties	(K = 85 m/hr	, NOx-in=100 j	ppm, Av=20)-45 m/hr)

Parameter		Uncertainty
Flow	Q	2%
Sample X-Section Dimension	d	1 mm
Sample Length	L	1 mm
Specific Surface Area	Asp	$0 \ m^2/m^3$ (a)
Inlet NO _x	NO _{x in}	0.5 ppm
Outlet NO _x	NO _{x out}	0.1 ppm
Uncertainty	K	2.3 m/hr 2.7%

^(a)Assumed zero as this parameter provided by the catalyst supplier



Figure A-1 Impact of Individual Parameter Uncertainties of the Uncertainty in K

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