

Laboratory Testing Guidelines for Gas Turbine Selective Catalytic Reduction (SCR) and CO Catalysts

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ABSTRACT

Selective catalytic reduction (SCR) and CO oxidation catalysts are widely employed on both simple cycle and combined cycle gas turbine systems. Laboratory-based approaches for measuring catalyst activity give end users a means of tracking and forecasting the working life of a catalyst charge. However, current practices for performing activity measurements vary among commercial laboratories offering this service. Catalyst system owners and operators desire standardization of procedures and related terminology to promote consistency in the catalyst life tracking process. These guidelines, developed in collaboration with end users, catalyst vendors, and third-party laboratories, are the first step toward EPRI standard protocols to be accepted and utilized by the industry. The guidelines provide separate recommendations for SCR and CO catalyst testing and cover the following areas: 1) test apparatus, 2) preparation of catalyst samples, 3) activity test methods and procedures, and 4) chemical and physical properties.

Keywords

Gas turbine NO_x CO SCR catalyst CO oxidation catalyst Laboratory testing

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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 gas turbine systems. Since catalyst activity degrades over time, many system owners and operators perform periodic laboratory testing to monitor catalyst performance. Laboratory tests provide a means of gauging remaining activity, and the information can be used 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 prescheduled, plant-wide maintenance outage, and avoids expensive forced outages due to non-compliance.

A number of commercial laboratories, including catalyst vendors and third-party companies, provide gas turbine catalyst testing services. However, these organizations may not use similar testing procedures or terminology. This raises questions about data consistency 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 gas 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 gas catalysts, which can be twice that of coal catalysts. This presents unique challenges for laboratory testing which must be addressed.

Objectives

These laboratory testing guidelines, developed in collaboration with end users, catalyst vendors and third-party laboratories, are an initial step in developing definitive EPRI standards for testing gas turbine SCR and CO catalysts. Specifically, the following areas are addressed:

- Definitions and conventions
- Performance test procedures
- Test apparatus
- Preparation of catalyst samples
- Chemical and physical test procedures

A detailed technical discussion section follows the procedural portion of the guidelines. The intent of this section is to explain the impacts of key factors such as NH_3/NO_x set point, NO_2 fraction, area velocity and others.

Modifications to these guidelines are anticipated over the next few years as the procedures are applied and refined. The ultimate goal is the application of consistent and sound testing methodologies throughout the industry.

2 DEFINITIONS AND CONVENTIONS

There are a number of parameters and conventions associated with gas 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-	1		
Example	es of Sta	andard C	onditions

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 (S_V)

The space velocity (S_V) 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.

$$\boldsymbol{S}_{v}\left(\boldsymbol{h}\boldsymbol{r}^{-1}\right) = \frac{\boldsymbol{Q}_{fq}\left(\boldsymbol{N}\boldsymbol{m}^{3} / \boldsymbol{h}\boldsymbol{r}\right)}{\boldsymbol{V}_{CAT}\left(\boldsymbol{m}^{3}\right)}$$
(2-1)

3. Area Velocity (Av)

The area velocity (A_V) is the flue gas flow rate (expressed at standard conditions) divided by the total catalyst surface area exposed to the flow (the geometric surface area, not the catalyst pore surface area). This quantity is usually expressed in units of m/hr.

$$\mathbf{A}_{v}(\mathbf{m}/\mathbf{hr}) = \frac{\mathbf{Q}_{fq}\left(\mathbf{Nm}^{3}/\mathbf{hr}\right)}{\mathbf{A}_{CAT}\left(\mathbf{m}^{2}\right)}$$
(2-2)

Often the area velocity is expressed in terms of the specific surface area, which is more readily available from the catalyst vendor.

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

4. Specific Surface Area (A_{sp})

The specific surface area is the total catalyst surface area (A_{CAT}) per unit volume of catalyst (V_{CAT}). This is usually expressed as m^2/m^3 . Specific surface area should be obtained from the catalyst vendor and is used predominately in the calculation of SCR catalyst activity. Note, this specific surface area is not to be confused with the catalyst pore surface area.

$$\mathbf{A}_{sp}\left(\mathbf{m}^{2}/\mathbf{m}^{3}\right) = \frac{\mathbf{A}_{CAT}\left(\mathbf{m}^{2}\right)}{\mathbf{V}_{CAT}\left(\mathbf{m}^{3}\right)}$$
(2-4)

5. Activity (K)

Catalyst activity (K) represents the efficiency of a catalyst in reducing NO_x concentrations. 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 activity for SCR catalyst is as follows:

$$\mathbf{K}(\mathbf{m/hr}) = -\mathbf{A}_{\mathbf{v}} \times \ln(1 - \Delta \mathbf{NO}_{\mathbf{x}})$$
(2-5)

where ΔNO_x is the NO_x reduction measured in a laboratory at A_v with NH₃/NO_x = 1.

For a CO catalyst, the activity is defined in terms of the space velocity, rather than the area velocity.

$$\mathbf{K}(\mathbf{1/hr}) = -\mathbf{S}_{v} x \ln(\mathbf{1} - \Delta \mathbf{CO})$$
(2-6)

where ΔCO is the CO oxidation measured in the laboratory at S_v .

The activity is not a fundamental property of a particular catalyst material but depends on the catalyst composition, geometry and flow conditions.

6. Reactor Potential (RP)

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

$\mathbf{RP}_{clean} = \mathbf{K}/\mathbf{A}_{v}$

The reactor potential is the catalyst activity times the total catalyst surface area per unit of flue gas flow.

7. 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, plate and corrugated. (Note, some early gas turbine SCR systems utilized plate catalysts. However, most new installations use honeycomb or corrugated configurations.) Descriptions of the various terms typically used to document catalyst dimensions are provided below:

<u>Honeycomb</u>



<u>Plate</u>



Corrugated



CO catalysts are available in honeycomb, metal foil, and corrugated varieties, but differ in composition from SCR catalysts. The active metals in SCR catalysts are homogeneous within the substrate material, whereas CO catalyst substrates (metal or ceramic) are wash-coated with alumina and precious metals. Metal foil CO catalysts are constructed into elements, or cassettes, similar to corrugated SCR catalyst.

3 SCR CATALYST LABORATORY TESTING

There are two approaches for laboratory SCR catalyst performance testing presented in this document:

- 1. Measure catalyst activity, K (m/hr);
- 2. Measure NO_x reduction achievable at an NH₃ slip limit.

Either may be used, although it is important to use the same method consistently over time when tracking catalyst performance. Background and procedures are provided below.

SCR Catalyst Activity Measurement

Catalyst activity (K) 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 activity is then calculated as

 $K = -A_V \ln (1 - \Delta NOx/100)$

(3-1)

Where ΔNO_x is the percent NO_x reduction measured across the sample and A_V is the area velocity used for the test.

The required operating conditions for measuring catalyst activity are shown in Table 3-1 and may be applied to all categories of laboratory apparatus described in this document (micro and bench-type reactors, defined later in this section). 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.

Impact of Area Velocity and NO_x Level on Activity Measurement Accuracy

For new gas 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 1ppm, which may be difficult to measure accurately.

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 outlet

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

Parameter ¹	Target Level	Accuracy ²	Maximum Drift ³
Temperature	Field value	$\pm 4.5^{\circ} F (\pm 2.5^{\circ} C)$	4.5°F (2.5°C)
Area Velocity	Field value or 35 m/hr ⁴	\pm 5% of target	$\pm 2\%$ of value
Linear Velocity	As needed to achieve desired A_V	\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 ⁵ or field value (nominally 7%)	\pm 1% absolute of target	$\pm 0.5\%$ absolute of value
CO ₂	As generated ⁵ or field value (nominally 3% dry)	\pm 1% absolute of target	$\pm 0.5\%$ absolute of value
NO _x	20 - 100 ppm dry at 15% O ₂ ⁶	\pm 1% of target	$\pm 0.5\%$ of value
NO ₂ /NO _x	Less than 5%		
SO_2	None added		
NH ₃ /NO _x ratio	1.20	$\pm 2\%$ of target	$\pm 2\%$ 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. Bench/semi-bench apparatus use field A_V value if measured NO_x reduction < 95%; otherwise increase A_V to 35 m/hr by increasing flow or reducing sample length. Micro apparatus use $A_V = 35$ m/hr.

5. "As generated" assumes a natural gas-fired combustion source.

6. Use field value if it falls within this range. Use the same value consistently when tracking activity, since activity is sensitive to inlet NOx concentration.



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



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

 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.

To improve accuracy of the activity measurement under high activity conditions (see Table 3-1 notes), the area velocity shall be set at 35 m/hr by shortening the sample length or increasing flow. The inlet NO_x level shall be increased above the field value if needed (20 ppm minimum to a maximum of 100ppm) to more accurately calculate the NO_x reduction.

Test data provided in the technical discussion section of this document (Section 6) show that catalyst activity is relatively insensitive to changes in area velocity (i.e., changes in sample length or flow). In fact, this sensitivity may be assumed to be within the activity measurement error for area velocity ranges analyzed in Section 6. Conversely, test data show that activity may be significantly sensitive to variations in the inlet NOx concentration. *When tracking deactivation of a given sample or performing comparative studies, it is crucial to use the same inlet NOx concentration during each test.*

A formal error analysis of the catalyst activity calculation is provided in the appendix. For the assumed conditions and measurement uncertainties shown in Table 3-2, the overall activity uncertainty is nominally 2.7%.

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	$NO_{x \text{ out}}$	0.1 ppm
Uncertainty	K	2.3 m/hr 2.7%

Table 3-2 Assumed Uncertainties (K = 85 m/hr, NO_x-in=100 ppm, Av=20-45 m/hr)

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

NH₃/NO_x Ratio

The activity test shall be conducted at $NH_3/NO_x = 1.20$, and set in the test apparatus with an accuracy of ±0.02. If the NH_3/NO_x ratio setting does not have the required precision, a series of tests (4 minimum) is recommended varying the NH_3/NO_x ratio over a range of nominally 0.95 to 1.50 and the data interpolated to $NH_3/NO_x = 1.20$.

The NH₃/NO_x ratio shall be determined one of two ways:

• The preferred approach is to measure inlet NO_x, NO_x reduction across the catalyst sample, and NH₃ 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 :

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

• 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.

$$\frac{NH_3}{NO_x} = \frac{\dot{N}_{NH_3}}{\dot{N}_{NO_x}}$$

Where

$$\dot{N}_{NH_3}$$
 = ammonia inlet molar flow rate = $\frac{\dot{M}_{NH_3}}{MW_{NH_3}}$

 \dot{M}_{NH_3} = ammonia injection mass flow rate, lb/hr

 $MW_{NH_3} = ammonia molecular weight$

$$\dot{N}_{NO_{x\,in}} = NOx \text{ inlet molar flow rate} = \left(\frac{NO_{x-in} (ppmv)}{1 \, x \, 10^6}\right) x \, \frac{\dot{M}_{fg}}{MW_{fg}}$$

 $\dot{M}_{\rm fg}$ = flue gas mass flow rate, lb/hr MW $_{\rm fg}$ = flue gas molecular weight

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. No conditioning is needed for samples previously exposed to flue gas.

Number of Activity Tests

At stable conditions, three (3) tests shall be performed and the activity calculated at $NH_3/NO_x = 1.20$. These tests may be performed either at a single point representing $NH_3/NO_x = 1.20$, in which case data for each test should be collected and averaged over a period of 30 minutes of steady operation; or, three separate curves of NO_x reduction versus NH_3/NO_x (0.95 – 1.50) may be generated to allow interpolation of the NO_x reduction at $NH_3/NO_x = 1.20$. For this latter approach, the exit NO_x should be stable at each injection rate and at least 5 minutes of data averaged at each point.

Activity shall be determined within ± 2.0 m/hr. If the three tests differ by more than ± 2 m/hr additional tests shall be conducted.

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 NO₂ 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 3-2.

$$\mathbf{K} = -\mathbf{A}\mathbf{v} \ln \left(1 - \Delta \mathbf{NO}_{\mathbf{x}} / 100\right)$$

$$\mathbf{A}\mathbf{v} = \frac{\mathbf{Q}(\mathbf{0}^{\circ}\mathbf{C}, \mathbf{1} \operatorname{atm})}{\mathbf{A}_{\operatorname{cat}}}$$
(3-2)

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
- Number of total cells
- Number of blocked cells
- Specific surface area (m²/m³)
- Amount of blockage for each sample, if any

- 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 (O_2, H_2O, CO_2, NO_x)
 - Area velocity for the test (including calculation procedure)
 - Linear velocity at the face of the catalyst (not the cell velocity)
 - 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

Measure NO_x Reduction at NH₃ Slip Limit (Alternate Test Approach)

For gas turbine SCR systems, the performance requirement is usually an outlet NO_x level (or NO_x reduction) 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 the desired NH_3 slip limit.

The required operating conditions for the alternate test measurement are shown in Table 3-3 and may be applied to all categories of laboratory apparatus described in this document (micro and bench-type reactors, defined later in this section). If a laboratory chooses to deviate from these conditions or procedures, the changes must be identified and discussed with the end user.

An accurate method for measuring ammonia slip is required for this approach. A continuous ammonia analyzer (i.e., TDL or FTIR) is preferred, but not required. Other measurement procedures are similar to the activity test procedures described earlier.

The test shall proceed as follows:

- 1. Set up the operating conditions shown in Table 3-3.
- 2. Condition the sample as described in the catalyst activity test section.
- 3. Slowly increase the ammonia injection rate until the NH_3 slip from the catalyst stabilizes at the target NH_3 slip concentration (±0.2 ppm).
- 4. Once stable, record the inlet and outlet NO_x concentrations over a period of 20 minutes.

Table 3-3 Alternate Test Conditions; NO_{x} (or NO_{x} Reduction) at NH_{3} Slip Limit

Parameter ¹	Target Level	Accuracy ²	Maximum Drift ³
Temperature	Field value	± 4.5°F (±2.5°C)	4.5°F (2.5°C)
Linear Velocity	As needed to achieve desired A_V	\pm 5% of target	$\pm 2\%$ of value
Area Velocity	Field value	\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 (nominally 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%		
SO_2	None added		
NH ₃ /NO _x ratio	As measured at NH ₃ slip permit level	$\pm 2\%$ of target	$\pm 2\%$ of value
NH ₃ Slip	Permit level	± 0.2 ppm	± 0.2 ppm
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

- 5. Calculate the NO_x reduction achieved at the NH_3 slip level, or note the average outlet NO_x level.
- 6. Turn off ammonia injection and allow the outlet NO_x to return to the inlet concentration.
- 7. Repeat Steps 3 6 two more times for a total of 3 tests.

If calculated NO_x reductions vary by more than $\pm 2\%$, or the outlet NO_x level varies by greater than ± 0.5 ppm, discard the test and repeat.

Following the tests, a report shall 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
- Catalyst type, honeycomb/corrugated
- Number of total cells
- Number of blocked cells
- Specific surface area (m^2/m^3)
- Amount of blockage for each sample, if any
- 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 velocity within a catalyst channel)
 - NO_x reduction for each test
 - Documentation of sample conditioning
 - NH₃ flow rate
 - NH₃ slip
 - NO_x Reduction and outlet NO_x
 - Start and stop time of each test

Catalyst Test Apparatus

Micro and bench-type reactors are generally used in a laboratory to measure catalyst performance. These categories are defined below.

Micro Reactors

Micro reactors use small "core" catalyst samples which typically do not represent the full catalyst block length. For instance, a micro reactor might accommodate a core-drilled sample 25mm in diameter and 150mm long, cut from a catalyst bed 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. This is illustrated in Figure 3-3, showing the relationship between linear velocity and area velocity for catalyst with a specific surface area of $1200 \text{ m}^2/\text{m}^3$. A 150mm micro reactor core sample will have a higher area velocity at a given linear velocity compared to a full-length 300 mm block. However, test data (provided in Section 6) show that area velocity variations imposed by changes in flow or sample length have only a minor impact on the measured activity (within the measurement error). As a result, activity determinations using micro reactors are acceptable.





Bench or Semi-bench Reactors

Bench reactors are designed to hold larger catalyst samples. For instance, most are able to accommodate a full 150 mm x 150 mm block of honeycomb or corrugated catalyst, one meter in length. Semi-bench reactors are similar, but may have a smaller cross section (e.g., 75 mm x 75 mm). The distinguishing feature is the ability to accommodate a full-length catalyst block, allowing the field linear velocity and area velocity to be simultaneously matched. This also allows the deactivation profile across the entire catalyst length to be accurately simulated.

Obtaining Catalyst Samples

The first task associated with laboratory SCR catalyst testing is obtaining an appropriate sample. The nature of this task will depend on whether the unit contains "built-in" samples, or if samples must be cut from the larger catalyst bed.

Built-in Test Elements

If the unit has built-in test elements, these are located within specific catalyst modules in the bed. 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. Catalyst modules in newer units may last over 200,000 operating hours before they need to be replaced. Thus, early in the life of the catalyst, samples may be taken less frequently, leaving more samples for later when "end of life" approaches. As the catalyst ages, the NH₃ slip versus time will become steeper, as illustrated in Figure 3-4. For instance, if the unit has 18 test elements and an expected life of 150,000 hours, an element could be taken every two years for the first six to eight years, then annually thereafter.



Figure 3-4 Example Relationship between SCR Catalyst Activity and NH₃ Slip

When a test element is removed, the location must be documented and the inlet/outlet orientation of the element noted. Another test element should be used to replace the element just removed. Care must be taken to ensure a replacement element is not sampled at a later date as it will not have the correct operating time.

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.

Core Samples from the Catalyst Bed

If the SCR catalyst bed does not have built-in test elements, or if there is reason to believe the test elements are not representative, then core samples may be taken from any location in the bed. The key issues are a) location, b) frequency, c) number of samples, and d) how the samples are obtained:

a) Location

There is no concrete data to suggest one region of a catalyst bed will deactivate at a different rate than another, unless there are large temperature non-uniformities or other abnormalities across the catalyst bed. If no large temperature gradients exist, take core samples from reasonably accessible locations. If there are significant gradients, take core samples from an area of the catalyst with average temperatures, and also from an area with high temperatures.

b) Frequency

Since core samples can be taken from the entire bed, it is recommended that samples be taken annually when the catalyst is new. As the catalyst approaches end-of-life, consider sampling more often.

c) Number of Samples

Two locations shall be sampled if they are cored in situ. Also, because the entire length of the catalyst is not tested, samples shall be taken from both the inlet and exit sides of the catalyst block to determine if there is a significant activity gradient. If the entire catalyst block is removed and sent to the laboratory for sample preparation, then inlet and outlet samples shall be taken at a single location in the block.

d) Obtaining Samples

For core samples, the entire block, or cassette, may be removed and sent to the laboratory for preparation. Or, core samples may be obtained from the larger bed in situ. 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 3-5.

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.



Figure 3-5 Examples of Catalyst Coring Tools

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 vendor $(\underline{m^2/m^3})$. 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.

$$A_{cat} = \begin{bmatrix} A_{SP} \ x \ a \ x \ b \ x \ L \end{bmatrix} \begin{bmatrix} \frac{C_{Tot} - C_{Pl}}{C_{Tot}} \end{bmatrix}$$

$$A_{SP} = \text{specific surface area, m}^2/\text{m}^3$$

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 venier (Figure 3-6). The surface area of the sample may then be calculated as follows:

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

 $d_{Ave} =$ average cell opening



Figure 3-6 Example of Microscope Used to Document Catalyst Cell Dimensions

Corrugated Catalyst

Because of the complex geometry of the corrugated catalyst, the specific surface area (m^2/m^3) should be obtained from the vendor. The catalyst surface area is then determined by multiplying the volume of the corrugated test sample by the specific surface area.

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:

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

$$\tag{4-1}$$

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 all categories of laboratory apparatus described in this document (micro, bench, and full size elements, described later in this section). 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.

Test Conditions

Space Velocity

Figure 4-1 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:

$$K_{CO-Lab} = -SV_{\nu-Lab} \times \ln(1 - \Delta CO) \tag{4-2}$$

CO reductions at field conditions shall then be calculated from the laboratory measurements using K_{CO} :

$$\Delta CO_{full-scale} = 1 - e^{\left(-K_{CO,lab}/S_{V-fullscale}\right)}$$
(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.

Table 4-1CO 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)	5º 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 (nominally 7%)	± 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)


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

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, 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 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 two (2) hours. 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 shall range from nominally 200°F (93°C) up 100°F (56°C) higher than the full-load field condition). 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 $\pm 2^{\circ}F$ ($\pm 1^{\circ}C$).

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, O₂) 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, l)
 - 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
 - Temperature (in/out)
 - Gas composition (O₂, H₂O, CO)
 - Space velocity for the test (including calculation procedure)
 - CO oxidation rate for each test
 - Documentation of sample conditioning
 - Start and stop time of each test

CO Catalyst Testing Apparatus and Samples

CO oxidation tests shall 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. Samples are discussed in more detail below.

Types of CO Catalyst Samples

CO catalyst samples shall be classified as follows:

Core Samples (Figure 4-3a)

Core samples are generally 1" diameter and full field length. Core samples are sized for testing in micro reactors. Since micro reactors use simulated flue gas, gas compositions and temperatures may be carefully controlled and monitored. Micro reactors are typically capable of producing temperatures high enough to develop a light-off curve.

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). Test buttons are used in bench or semi-bench reactors. As a bench reactor traditionally uses actual flue gas from a combustor, it is generally more difficult to maintain flows while varying temperatures to develop a light-off curve.

Full-Scale Blocks (Figure 4-3c)

In certain instances, full-scale (24" x 24") or half-scale (12"x 12") catalyst blocks may be sent to the laboratory for CO oxidation testing. Tested at this size, an apparatus larger than a typical bench rig is required.

Samples from the Catalyst Bed

General guidance for obtaining CO catalyst samples is provided below. CO catalysts are relatively thin in the direction of flow compared to SCR samples, making it practical to test full field length samples in micro or bench-style reactors.

Obtaining Samples

Some metal foil catalyst vendors install test buttons within the catalyst beds. In these cases, obtaining samples for testing is relatively easy, as the button(s) merely need 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 in a micro or bench-scale reactor. For ceramic honeycomb catalyst, either a core sample is drilled for testing in a micro 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.

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.



a) Core: 25mm dia.



b) Button: 76mm dia.



c) Full Scale Module: 600 x 600mm

Figure 4-3 Examples of CO Catalysts

Documenting Test Sample Dimensions

A core sample or test button shall be round in cross-section. The length of the sample (1) 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.

$$V_{cat} = \left[l x \pi x \frac{d^2}{4} \right] \left[\frac{C_{Tot} - C_{Pl}}{C_{Tot}} \right]$$
(4-6)

 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.

$$V_{cat} = ((C_{Tot} - C_{Pl})*L) / (CPSI)$$
(4-7)

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, or sand, 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 vendor 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.

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

Table 5-1 SCR Catalyst Sampling Guideline – Minimum Requirements

Parameter	New Catalyst	Aged/Regenerated
Sample Location – Corrugated and Honeycomb*	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

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

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.

^{*} It is assumed that with a honeycomb element, or portion of element, that the entire catalyst bed height is represented.

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 apparatus[†]
- 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 which is 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.

[†] Description of commercial equipment utilized should include manufacturer and specific model information.

Bulk Chemical Analysis

The most rigorous method of performing 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 and thus a true bulk chemical determination is made. 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 more rigorous determination of bulk catalyst chemistry, while the sections under "Surface Analysis" will generally apply to the latter method of determining bulk chemistry – only the sample preparation method differs from the surface analysis (although specific instrument settings, such as penetration depth may be altered).

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.

Analytical Technique and Specific Analytes

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. Table 6-3 gives the preferred elemental species for routine bulk chemical testing, along with oxide compound to be used as the reporting convention, and appropriate units. In addition to the preferred analytes, several other analytes may also be of interest as shown in Table 6-4, and can be determined at discretion.

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.

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, that 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), 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 poison. X-ray diffraction can be used to identify crystalline compounds that may be present on the catalyst surface. Scanning Electron Microscopy (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.

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.
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	SO3	% 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

Element	Oxide Basis for Reporting	Units
Barium	BaO₃	ppmw
Boron	B2O3	ppmw
Chlorine	CIO ₂	ppmw
Chromium	Cr ₂ O ₃	ppmw
Nickel	NiO ₂	ppmw
Niobium	Nb ₂ O ₅	ppmw
Thallium	Tl ₂ O	ppmw

Table 5-5Additional Discretionary Bulk and Surface Analytes

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 procedure*
- Detailed description of the test procedure
- Detailed description of the instruments utilized[†]
- Specific test conditions employed[‡]
- Applicable QA/QC data and analyses

^{*} Detailed sample digestion procedures should be reported, if utilized.

[†] 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.

[‡] 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.

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 approaches;
- Effect of area velocity, inlet NO_x concentration and moisture on SCR catalyst activity.

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 initial 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

$$\Delta NO = 1 - e^{-K/Av}$$
(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.

This is not necessarily the case for gas turbine SCR systems. Table 6-1 shows examples of gas turbine SCR permit conditions and the resulting NH_3/NO_x ratio required to meet these conditions. As the table illustrates, gas turbine SCR systems may operate at NH_3/NO_x ratios greater than 1.0.

Table 6-1 Typical GT SCR NH₃/NO_x Ratios

Unit	NO _x -in ppm	NO _x -out ppm	NH₃ slip ppm	NH ₃ /NO _x
1	25	2	5	1.12
2	20	2	2	1.00
3	3 28		5	1.12
4	11	2	2	1.00
5	5 14		7	1.36
6	6 25		5	1.12
7	15	2	5	1.20
8	11	2	5	1.27
9	13	2	5	1.23

Further, laboratory NO_x reduction versus NH₃/NO_x ratio curves frequently show increasing NO_x reduction as the NH₃/NO_x ratio is increased above 1.0. This would suggest that at NH₃/NO_x = 1.0, there may not be full coverage of the active sites by ammonia. Figure 6-1 shows an example of this behavior. The impact on calculated catalyst activity is also shown in this figure. Since typical gas turbine SCR systems may operate at NH₃/NO_x > 1.0, it makes sense to determine the catalyst activity at NH₃/NO_x > 1.0, rather than 1.0 as is done with coal SCR catalyst. As discussed earlier, these guidelines require activity be measured at NH₃/NO_x = 1.2. To distinguish this from an activity measured at NH₃/NO_x = 1.0, this value may be designated by laboratories as K_{1.2}.

Figure 6-2 shows how activity may vary between a test conducted at $NH_3/NO_x = 1.2$ versus 1.0. If the activity is determined at $NH_3/NO_x = 1.2$, it will over-predict conditions at $NH_3/NO_x = 1.0$, and vice versa. However, since most gas turbine SCR systems operate at $NH_3/NO_x > 1.0$, testing at $NH_3/NO_x = 1.2$ is more appropriate.

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

 NO_x entering gas turbine SCR catalyst may have a significant fraction of NO_2 . The reactions between NH_3 , NO, and NO_2 are shown below:

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

$$6NO_2 + 8NH_3 \rightarrow 7N_2 + 12H_2O$$
 (6-4)

$$NO_2 + 2NH_3 + \frac{1}{2}O_2 \rightarrow \frac{3}{2}N_2 + \frac{3}{2}H_2O$$
(6-5)

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



Figure 6-1 Typical Laboratory NO_x Reduction vs. NH_3/NO_x and Calculated Catalyst Activity, K



Figure 6-2 Activity (K_{1.0 and} K_{1.2}) Determination

Reaction (6), 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 6-3 which shows a 50% mixture of NO₂ and NO (i.e., NO₂/NO_x = 0.5) exhibits higher NO_x reduction than NO or NO₂ alone.

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 NH_3/NO_x ratio should be used to measure catalyst activity? Figure 6-4 shows how the stoichiometry varies for Equations 6-4 and 6-5 as the amount of NO₂ in the flue gas varies.
- For a given gas turbine SCR, is the NO_2/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 ratio 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. Laboratory NO_2 fractions less than 5% are acceptable. NO_2 may be increased, but consideration of the required stoichiometry must be addressed.

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 gas 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 vendor, 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.



Figure 6-3 Effect of NO₂/NO_x Ratio on Catalyst Performance (data from Ref. 10)



Figure 6-4 Effect of NO₂ on the SCR Stoichiometry

NO to NO₂ Oxidation across CO Catalyst

CO catalyst will oxidize some fraction of NO exiting the gas turbine to NO₂. 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 NO₂ 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 NO₂ components are measured separately. Figure 6-5 shows an example of NO to NO₂ oxidation across a CO catalyst over a wide temperature range.



Figure 6-5 Example of NO Oxidation Across a CO Catalyst

CO Catalyst Testing

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 6-6⁽¹¹⁾ shows typical examples of CO deactivation. The light-off curve of may vary markedly depending on the nature of deactivation. Using the curves shown in Figure 6-6, 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 6-6 Deactivation Characteristics of CO Catalyst ⁽¹¹⁾

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:

$$\Delta CO_{lab} = 1 - e^{-K_{CO}/_{Sv_{lab}}} \tag{6-7}$$

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 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 gas turbine catalyst samples, high activity values may lead to measured NO_x reductions greater than 97% 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.

Laboratory tests were performed to determine if these potential biases are significant.

Initial 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.

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:

$$\binom{K}{K(@0\%H_20)} = -1.222 x 10^{-6} (H_20)^5 + 6.403 x 10^{-5} (H_20)^4 - 1.269 x 10^{-3} (H_20)^3 + 1.199 x 10^{-2} (H_20)^2 - 6.161 x 10^{-2} (H_20) + 1.0$$

Laboratory tests were performed to validate this curve.

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. Tables 6-2 and 6-3 provide the conditions and results for the bench and micro reactor tests, respectively. The results are also plotted in Figure 6-8, along with predictive model data. 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.



Figure 6-7 Moisture Corrective Curve⁽³⁾

The model predictions in Figure 6-8 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. 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 measurement to area velocity variations, and validated the approach of using higher area velocity values (i.e., 35 m/hr) in micro or bench reactors.

Table 6-2Bench 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 6-3 Micro 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



Figure 6-8 Micro-scale and Bench-scale Tests: Effect of Area Velocity on Activity

Flue Gas Moisture Test Results

A series of tests were conducted in a micro reactor using a new gas 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 ₂ O:	3-15%

The results are shown in Figure 6-9, and show good agreement with the water correction curve in Reference 3 (Figure 6-7). An increase in water content from 3% to 15% decreases the activity by 7 m/hr (approximately 8%).



Figure 6-9 Micro-scale Tests: Effect of H₂O Content on Activity

Initial 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 during the current project in 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 ₂ O:	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 given in Table 6-4 and plotted in Figure 6-10. 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 initial NO_x level. Reducing inlet NO_x 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.

Table 6-4 Micro Reactor Initial 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



Figure 6-10 Micro Reactor Tests: Effect of Initial NO_x Level

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A UNCERTAINTY ANALYSIS

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

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

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

K = catalyst activity, m/hr

 $Q = gas flow rate Nm^3/hr$

d = sample cross section dimension, m

L = sample length, m

Asp = sample specific surface area, m^2/m^3

 NO_{xo} = 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 1 shows the partial derivatives of equation (4-7).

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 vendor 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	$\partial K = 1 \left(NO_{x_{o}} \right)$
	$\overline{\partial Q} = \frac{1}{d^2 L A s p} \ln \left(\frac{1}{N O_{x_i}} \right)$
Asp	$\partial K \qquad Q \qquad (NO_{x_0})$
	$\frac{\partial}{\partial Asp} = -\frac{\partial}{\partial L A_{sp}^2} \ln\left(\frac{\partial}{\partial NO_{x_i}}\right)$
L	∂K 1 (NO_{x_0})
	$\frac{\partial L}{\partial L} = -\frac{\partial^2 L^2 A sp}{\partial L^2 A sp} \ln\left(\frac{\partial V}{NO_{x_i}}\right)$
d	$\partial K = 2Q \left(NO_{x_0} \right)$
	$\frac{\partial d}{\partial d} = -\frac{\partial^2 L}{d^2 L A s p} \ln\left(\frac{\partial^2 V}{N O_{x_i}}\right)$
NO_{x_0}	∂K Q
0	$\frac{\partial NO_{x_0}}{\partial L A sp NO_{x_0}} = \frac{\partial C}{\partial L A sp NO_{x_0}}$
NO_{x_i}	∂K Q
Ľ	$\frac{\partial NO_{x_i}}{\partial NO_{x_i}} = \frac{\partial^2 L Asp NO_{x_i}}{\partial VO_{x_i}}$

Table A-2 Assumed Uncertainties (K = 85 m/hr, NOx-in=100 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	$NO_{x \text{ out}}$	0.1 ppm
Uncertainty	K	2.3 m/hr 2.7%

^(b)Assumed zero as this parameter provided by the catalyst vendor



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