Aftertreatment Protocols for Catalyst Characterization and Performance Evaluation:
Low-Temperature NH$_3$-SCR Catalyst Test Protocol

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SUMMARY

Catalyst testing protocols are being developed to address the need for consistent and realistic metrics for aftertreatment catalyst evaluation. Catalyst testing protocols will consist of a set of standardized requirements and test procedures that sufficiently capture performance of a catalyst technology that is adaptable to various laboratories. This document details a testing protocol applicable to catalysts to be used for the selective catalytic reduction (SCR) of oxides of nitrogen (NOx) using ammonia as a reducing agent, a.k.a., NH₃-SCR catalysts.
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BACKGROUND

Low-temperature aftertreatment catalyst test protocols have been developed to provide consistent and realistic standardized catalyst test procedures and aging conditions that sufficiently capture a catalyst technology’s performance capability within an applicable temperature window. The protocols are intended as a means to share the results of catalyst research with the technical community. They are meant solely to enable the accurate evaluation and benchmarking of the performance of candidate catalyst technologies in an open public forum; they are not meant to replace or dictate individual research institute protocols or methodologies of investigation. The protocols have been developed with the aspirations of (1) having general community consensus; (2) being consistent with anticipated and emerging catalyst technologies; (3) being confidently reproducible and adaptable in various laboratories with varying test capabilities around the nation; (4) being practical to the end user and having utility to the research; and (5) being ultimately captured in the literature citations of the procedures employed for emerging catalyst research.

Catalytic aftertreatment can include singular functionalities such as oxidation, reduction, adsorption, desorption, and physical filtration. However, current and future aftertreatment strategies often integrate multiple functionalities. “Device” is the term used to describe an aftertreatment component and strategy that may employ one or multiple functionalities. With this in mind, test protocols will likely take unique forms depending on multiple factors, including (i) the research activity the protocol is supporting, (ii) the nature and complexity of the aftertreatment strategy (e.g., conversion versus simple adsorption, singular versus multi-functionality), and (iii) the combustion platform of interest (e.g. diesel vs. gasoline, lean burn vs. stoichiometric combustion).

Research activities can be broadly characterized as catalyst discovery and performance screening, reaction engineering, or development to achieve a specific performance target. The protocols presented here are performance-based protocols for screening candidate catalysts, and thus they are global in nature assessing only the critical overall efficiency of the catalytic reaction(s)/under application(s) of interest. The protocols are intended to be simple to execute in a timely manner, as it is desirable to maximize the pace of catalyst development. This is in contrast to more detailed protocols intended to probe individual reaction steps of a global process (e.g., for advanced catalyst development or for supporting reaction engineering & modeling efforts). In those instances, the focus is placed on isolating and characterizing each contributing reaction that supports the overall reaction scheme. Such protocols will be more complex and will likely place additional demands on the testing requirements needed. An example of this is the CLEERS SCR protocol focused on data-set generation for calibrating and validating device models, or the Cummins 4-step SCR protocol developed to elucidate the multiple catalytic functions, both steady-state and transient, that are practically relevant to mobile SCR applications [2].

The relative complexity of the aftertreatment process being studied is expected to affect the form of the protocol employed as well. The level of detail is primarily determined by the focus of the protocol being either a single functionality or a multi-functional device. Aftertreatment process that require only a single functionality, e.g., oxidation catalysis, can be evaluated with a fairly simple protocol. However, multiple functionalities often dictate overall device performance, e.g., the three-way catalyst with oxygen storage. In this instance, a test protocol requires multiple steps to adequately characterize both conversion and storage performance. In the protocols presented here, the method of performance testing differs depending on the relative complexity required to adequately quantify the performance of the catalyst system of interest.

Three (3) catalyst test protocols have been completed to date:
As the test protocols are reviewed and approved by the Advanced Combustion and Emission Control (ACEC) Technical Team and the Advanced Powertrain Technology Leadership Council (APTLC), they will be released to the technical community at https://cleers.org/low-temperature-protocols/. This document represents a low-temperature catalyst testing protocol applicable to NH\textsubscript{3}-SCR catalysts, i.e., selective catalytic reduction (SCR) catalysts for the reduction of oxides of nitrogen (NOx) using ammonia (NH\textsubscript{3}) as a reducing agent.

OVERVIEW

This protocol is developed as a guide for conducting continuous flow reactor studies for evaluating and comparing performance of candidate aftertreatment catalyst technologies. The goal is to efficiently characterize applicable performance parameters of candidate catalysts. Specifically, this protocol will address the performance evaluation of NH\textsubscript{3}-SCR catalysts for low temperature aftertreatment systems intended for advanced lean-combustion strategies. Description of the protocol provided below is separated into five (5) sections: (i) reactor description and best practices, (ii) exhaust simulation, (iii) protocol execution, (iv) de-greening and aging, and (v) reporting.

The catalyst aging protocol in section (iv) is staged into two phases of testing. These two phases of testing together form a complete durability assessment that is necessary to determine the viability of candidate NH\textsubscript{3}-SCR catalysts for application to vehicles. Phase 1 testing employs a sub-set of the complete aging protocol to expeditiously screen candidate NH\textsubscript{3}-SCR catalysts in high-throughput fashion for consideration of further testing. Then, Phase 2 testing is performed on candidate NH\textsubscript{3}-SCR catalysts that demonstrate promise in Phase 1 testing. It employs the balance of the aging protocol to form a complete and necessary evaluation of NH\textsubscript{3}-SCR catalyst durability.

NH\textsubscript{3}-SCR catalysts are materials that have been developed for the reduction of NO\textsubscript{x} to N\textsubscript{2} and O\textsubscript{2} in lean exhaust that use NH\textsubscript{3} as a reducing agent. Although NH\textsubscript{3} is currently derived from the decomposition of urea [(NH\textsubscript{2})\textsubscript{2}CO] and is limited to ~190°C and higher, the decomposition of urea or the derivation of NH\textsubscript{3} from alternative sources is outside the scope of this protocol. Thus, NH\textsubscript{3} is used in this protocol. NH\textsubscript{3}-SCR catalysts are designed to function in lean-burn engine exhaust, and thus stoichiometric GDI (S-GDI, diluted with EGR) exhaust is considered only as it relates to aging of NH\textsubscript{3}-SCR catalysts when deployed in multi-mode combustion applications.

1. REACTOR DESCRIPTION AND BEST PRACTICES

A reactor description is provided as a set of minimum instrumentation, sample, and reactor performance requirements for execution of the protocol. Best practices for reactor design, derived from industry, university, and national laboratory reactor experience, are provided as a guide to achieve accurate and reproducible results that can be easily shared throughout the aftertreatment R&D community. As an example, NH\textsubscript{3}-SCR catalyst testing has unique requirements to prevent unwanted deposits from forming in the reactor system; this is elaborated upon below under Reactor Performance Requirements.

1.1 Instrumentation Requirements

A minimum of two thermocouples should be employed in the reactor configuration for inlet gas and catalyst bed temperature measurement, as shown in Figure 2. An inlet thermocouple should be
located less than ½-inch upstream of the catalyst (packed bed or core) for measuring the temperature of the simulated exhaust stream entering the catalyst. A catalyst thermocouple should be placed inside of the catalyst for measuring its temperature (embedded in the packed-bed for powders, or inserted into a monolith channel for cores), and should be located downstream axially of mid-bed and radially close to centerline of the catalyst. The maximum thermocouple diameter should be 0.032 inch (~0.8 mm) to minimize adverse effects of the thermocouple on catalyst powder flow dynamics, or to avoid channel plugging as a result of thermocouple insertion in monoliths. For core testing, it is best practice to place an inert monolith core just upstream of the catalyst, with the inlet thermocouple embedded within the inert core. This serves two functions: it provides a higher confidence in accuracy of the inlet temperature measurement, and it ensures that the inlet thermocouple is located radially close to centerline and not in contact with the reactor wall.

![Image of thermocouple placement](image)

**Figure 1** – Inlet and catalyst thermocouple (TC) placement

Chemical analysis capability should include, at a minimum, quantitative analysis of total NOx and NH₃. It is preferable to have quantitative analysis of NO, NO₂, and N₂O separately, but this is not required. Analytical capability less than the minimum stated here will limit the usefulness and applicability of results. A point of emphasis is N₂O byproduct formation. Although N₂O analysis is not required, it is highly encouraged due its importance as a regulated greenhouse gas with a 298-times CO₂-equivalent 100-year global warming potential impact. Additionally, not all analytical methods provide the necessary measurement or resolution for emissions analysis. The level of reproducible detection must be equal or greater to the relative conversion reported, e.g. reporting of 95% conversion of a NOx at 200 ppm feed dictates that reproducible detection of 10 ppm of NOx is feasible. Please note that the preferable resolution of an emission measurement is ≤2% of feed concentration, and capability less than this will limit the use and applicability of results.

The frequency of NOx and NH₃ gas sample analysis is also important. The frequency of sample analysis will dictate the relative fidelity of the data used to determine light-off characteristics and NH₃ storage properties of the catalyst. Therefore, best practice is a sampling frequency of 1 Hz. If sampling frequency is less than this, it should be reported along with the data. This level of frequency will exclude chromatography-based analysis techniques.
1.2 Catalyst Sample Requirements

**Powder**
- Bed diameter (i.e. reactor ID) > 3 mm and < 13 mm
- Catalyst particle size ≤ 0.25 mm (60 mesh)
- Catalyst bed aspect ratio (length/diameter) ≥ 1

**Monolith Core**
- Core diameter > 12 mm, preferably > 19 mm
- Cordierite core substrate
- 400 CPSI or as appropriate to the application
- Catalyst bed aspect ratio (length/diameter) ≥ 1

1.3 Reactor Performance Requirements

NH3-SCR testing has unique requirements due to the potential formation of ammonium nitrate (NH4NO3) solid deposits, which can occur in the presence of NO2 and NH3 at temperatures below ~190°C. NH4NO3 formation must be avoided as it can plug fittings and flow lines impeding flow, and it is a potential sink or source of NH3 and NO2 that can artificially affect test results. Figure 2 is provided for additional clarity. As shown in Figure 2, the surfaces of the reactor system, sampling lines, and analytical system that will be exposed to NH3 and/or NO2 must be heated to 200°C. Additionally, it is highly recommended to preheat the constant components of the simulated exhaust stream [i.e., O2, H2O, CO2 and diluent (N2)] to 200°C prior to mixing with NO2 and NH3. And finally, NO2 and NH3 must be mixed with the hot simulated exhaust individually (i.e., separately with individual feed lines) and not mixed together prior. In certain test scenarios, the catalyst and reactor will be cooled to <200°C and may incur NH4NO3 deposit formation. However, all reasonable efforts should be made to ensure that NH4NO3 formation does not occur unexpectedly outside of the catalyst reactor (i.e., in other areas of the feed or sampling system). Periodic checks should be performed for NH4NO3 deposits in process and sample lines.

![Figure 2 – Heating and flow strategy for preventing NH4NO3 formation](image-url)
It is best practice to try to minimize any thermal gradients within the catalyst sample during protocol testing or aging. Therefore, in nitrogen-only flow, the allowable temperature gradient measured between the inlet thermocouple and catalyst thermocouple is ≤ 5°C at 100°C feed and ≤ 20°C at 500°C feed. If a dedicated pre-heating furnace is employed (in addition to a catalyst furnace), this is generally easily accomplished. However, if only a single catalyst furnace is employed, it is best practice to place the catalyst immediately downstream of the actively heated zone.

In order to confidently attribute conversion efficiency to catalysis during the test, the test protocol must first be run without a catalyst in place, i.e. with an empty reactor tube or inert sample. This is called reactor “base-lining” and is necessary to ensure that the reported performance is solely attributed to catalyst behavior and does not contain artifacts originating from the apparatus. This is extremely important for NH3-SCR performance to accurately understand and isolate the impact of the catalyst to the fate of NH3. Reactor “base-lining” is to be performed once per specific application and per unique apparatus configuration and should be repeated following any significant hardware repair, replacement, or reconfiguration. The recovery of reactants over the entire temperature ramp of the protocol during reactor “base-lining” should be ≥ 95%. Please note that at very high temperatures (>550°C), gas phase reactions may occur that are not of concern and can be ignored.

1.4 Additional Reactor Configuration and Operation Requirements and Recommendations
- System leak checked daily and following each sample change or any hardware re-configuration.
- Analyzer calibration at least once daily (e.g. zero gases and span gases)
- Inlet gas composition characterized each experiment.
- If employing dedicated pre-heating, suggested pre-heating of inert species only (i.e. air, N2, H2O, CO2).
- Passivated stainless steel tubing for gas transport to and from the reactor (recommended).
- Calibrate mass flow controllers at least once per year or according to manufacturer guidelines (recommended).
- IMPORTANT: Periodically check for NH4NO3 deposits in process and sample lines.

2. EXHAUST SIMULATION

This protocol is intended to characterize the NOx SCR performance of catalysts for a specific combustion mode by using a suitable gas composition for the simulated exhaust. The recommended gas compositions for the NH3-SCR catalyst test protocol are detailed in Table 1 for clean diesel combustion (CDC), low temperature combustion of diesel (LTC-D), low temperature combustion of gasoline (LTC-G), and lean gasoline direct injection (L-GDI). The difference between measured and targeted gas concentrations (from Table 1) should be ≤ 10%. If the user does not have a targeted application for their development efforts, then it is suggested that they employ CDC conditions as a default.

The protocol considers certain gas concentrations and parameters as constants during testing, including % O2, % H2O, % CO2, the NH3-to-NOx ratio (ANR), space velocity (SV), and catalyst aged state. Each of these be held constant during testing. The test components in the protocol are NOx, NOx make-up (i.e., fraction of NO and NO2), and NH3. The protocol excludes CO, H2, and hydrocarbons in the simulated exhaust as it assumes that an oxidation function is upstream of the NH3-SCR catalyst. Hydrocarbon content in the exhaust is considered as it relates to catalyst durability and/or poisoning (i.e., coking) and is discussed in more detail in Section 4.5.
Table 1 – Simulated exhaust parameters: NH₃-SCR catalysis [a]

<table>
<thead>
<tr>
<th>Constant components</th>
<th>CDC</th>
<th>L-GDI</th>
<th>LTC-G</th>
<th>LTC-D</th>
</tr>
</thead>
<tbody>
<tr>
<td>[O₂]</td>
<td>12%</td>
<td>9%</td>
<td>12%</td>
<td>12%</td>
</tr>
<tr>
<td>[H₂O]</td>
<td>6%</td>
<td>8%</td>
<td>6%</td>
<td>6%</td>
</tr>
<tr>
<td>[CO₂]</td>
<td>6%</td>
<td>8%</td>
<td>6%</td>
<td>6%</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Variable components</th>
<th>all in [ppm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total [NOₓ]</td>
<td>200</td>
</tr>
<tr>
<td>[NH₃]</td>
<td>200</td>
</tr>
</tbody>
</table>

(a) Balance N₂

The standard space velocity to be employed during testing is 30,000 hr⁻¹ for monolith catalysts and 200 L/g-hr for powder catalysts across all combustion modes. Optionally, a higher 60,000 hr⁻¹ space velocity for monolith catalysts and 400 L/g-hr for powder catalysts is encouraged if more appropriate for the application.

The gas concentrations shown in Table 1 were selected by a team of experts in the field to reflect the actual exhaust compositions for the various combustion strategies listed. Thus, it is important that these gas compositions as well as the testing conditions described above are followed when evaluating new catalyst materials. Omitting or significantly altering the components and concentrations identified in Table 1, or employing a smaller space velocity than what is identified above, will cast significant doubt on the ability of the catalyst to perform analogously to the data presented when evaluated on actual engines.

3. PROTOCOL EXECUTION

The protocol should only be executed on a catalyst that has been, at a minimum, fully calcined and de-greened. It is requested that the user conduct catalyst testing after each of the following sequence: (i) de-greening, (ii) thermal aging, and (iii) sulfur exposure and desulfation attempt(s). Then, only those catalysts that show promise will proceed to additional durability testing, including (iv) low-temperature HC exposure, and (v) low-temperature water exposure event. For additional detail on each of these, see Section 4.

As shown in Figure 3, protocol execution consists of pretreatment and then catalyst performance testing under standard SCR reaction conditions (i.e. NOx is NO only), followed by a second pretreatment and then performance testing under fast SCR reaction conditions (i.e. NOx is equimolar NO & NO₂). Pretreatments are employed to ensure that common conditions prevail prior to activity characterization, and each should occur for 20 minutes at 600°C with the constant components ([O₂], [CO₂] and [H₂O]) from Table 1. Light-off tests are performed with a temperature ramp (rather than isothermal test conditions) to efficiently and quickly assess global SCR performance of candidate catalysts as a function of temperature. Although not required as part of the protocol, the user is encouraged to perform an NH₃ temperature programmed desorption (TPD) measurement at the end of the performance testing. An NH₃ TPD analysis can be valuable for assessing catalyst state, and is discussed as a potential modification to the protocol in Appendix C. Additional detail is provided in the discussion below and in Tables 2 & 3 below for the light-off tests. Additionally, Figure 3 is provided as a reference for temperature control during NH₃-SCR catalyst testing.
3.1 Standard SCR Light-off Test

The first performance test is a standard SCR light-off test performed from 100°C to 550°C at a ramp rate of 2°C/min. This test employs NO only as the NOx in the feed at the concentration from Table 1. It is preferred that the user conduct testing that includes both a ramp down in temperature (i.e., reverse light-off) and a ramp up in temperature (i.e., forward light-off) as shown in Figure 4; the reasons for this include capturing NH₃ storage effects and the potential effects of deposit formation at <200°C on SCR performance. As detailed in Table 2, following pretreatment and cooling the catalyst from 600°C to 550°C, NH₃ and NO are added to the feed and held at 550°C to reach steady-state; this should occur for at least 5 minutes, but no more than 20 minutes. Performance testing is then continued by cooling the catalyst from 550°C to 100°C at 2°C/min, and then heating from 100°C to 550°C at 2°C/min.

Although not recommended, if the user chooses they may perform an alternative form of the protocol that includes only the temperature ramp-up portion of the test, thus omitting the controlled ramp-down. To accomplish this, following pretreatment, the user first cools the catalyst from 600°C to 100°C, NH₃ and NO are added and held at 100°C for 20 minutes, and then the catalyst is heated from 100°C to 550°C at 2°C/min.

Table 2 – Standard SCR activity testing \(^{(a)}\)

<table>
<thead>
<tr>
<th>Test Mode</th>
<th>Action</th>
<th>Temperature</th>
<th>Time</th>
<th>Exhaust make-up (balance N₂) (^{(a)})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pretreatment</td>
<td>Hold</td>
<td>600°C, 20 min</td>
<td></td>
<td>[O₂] [CO₂] [H₂O]</td>
</tr>
<tr>
<td>OPTION #1 – Preferred test strategy: ramp down and ramp up</td>
<td>-</td>
<td>Cool</td>
<td>550°C, 5 min</td>
<td>[O₂] [CO₂] [H₂O]</td>
</tr>
<tr>
<td>STD SCR test</td>
<td>Hold</td>
<td>550°C, 5 – 20 min</td>
<td></td>
<td>[O₂] [CO₂] [H₂O] [NO] [NH₃]</td>
</tr>
<tr>
<td>STD SCR test</td>
<td>Ramp down</td>
<td>550-100°C @ 2°C/min</td>
<td></td>
<td>[O₂] [CO₂] [H₂O] [NO] [NH₃]</td>
</tr>
<tr>
<td>STD SCR test</td>
<td>Ramp up</td>
<td>100-550°C @ 2°C/min</td>
<td></td>
<td>[O₂] [CO₂] [H₂O] [NO] [NH₃]</td>
</tr>
<tr>
<td>OPTION #1 – Alternative test strategy: ramp up only</td>
<td>-</td>
<td>Cool</td>
<td>100°C, 5 min</td>
<td>[O₂] [CO₂] [H₂O]</td>
</tr>
<tr>
<td>STD SCR test</td>
<td>Ramp up</td>
<td>100-550°C @ 5°C/min</td>
<td></td>
<td>[O₂] [CO₂] [H₂O] [NO] [NH₃]</td>
</tr>
</tbody>
</table>

\(^{(a)}\) The procedures are the same for all combustion modes and vary only be the exhaust make-up used.
\(^{(b)}\) The bracketed concentration values are combustion-mode dependent and found in Table 14.
3.2 Fast SCR Light-off Test

The second performance test is a fast SCR light-off test performed similarly from 100°C to 550°C. This test employs equimolar amounts of NO and NO₂ for the total NOx concentration from Table 1. As with the standard SCR test, it is strongly encouraged that the user conduct both a temperature ramp down and ramp up light-off test as shown in Figure 4. To accomplish this, referencing Table 3, following pretreatment and cooling to 550°C, NH₃, NO, and NO₂ are added to the feed and held at 550°C to reach steady-state. Then the catalyst is ramped down from 550°C to 100°C at 2°C/min, and then ramped up from 100°C to 550°C at 2°C/min. Alternatively, for performing only a temperature ramp up fast SCR light-off test, following pretreatment the catalyst is cooled from 600°C to 100°C and then NH₃, NO and NO₂ are added followed by immediately heating the catalyst from 100°C to 550°C at 2°C/min.

As mentioned above, the test protocol employs a 2°C/min ramp rate. Cooling at a rate of 2°C/min at temperatures approaching 100°C may not be achievable on all reactor systems. It is requested that the user provide indication of this, and some understanding of the cooling ramp rates that were feasible. This is especially important for consideration due to the potential formation of NH₄NO₃ deposits on the catalyst at low temperatures.

![Figure 4 – NH₃-SCR protocol temperature control: pretreatment plus ramp-down and ramp-up light-off tests for standard and fast SCR reaction characterization (illustrative only; actual test times may vary)](image-url)
### Table 3 – Fast SCR activity testing (a)

| Test Mode       | Action      | Temperature | Time     | Exhaust make-up (balance N₂)  
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Pretreatment</td>
<td>Hold</td>
<td>600°C, 20 min</td>
<td></td>
<td>[O₂] [CO₂] [H₂O]</td>
</tr>
<tr>
<td>OPTION #1 – Preferred test strategy: ramp down and ramp up</td>
<td>Cool</td>
<td>600–550°C</td>
<td></td>
<td>[O₂] [CO₂] [H₂O]</td>
</tr>
<tr>
<td>Fast SCR test</td>
<td>Hold</td>
<td>550°C, 5–20 min</td>
<td>[O₂] [CO₂] [H₂O] [NO] [NO₂] [NH₃]</td>
<td></td>
</tr>
<tr>
<td>Fast SCR test</td>
<td>Ramp down</td>
<td>550-100°C @ 2°C/min</td>
<td>[O₂] [CO₂] [H₂O] [NO] [NO₂] [NH₃]</td>
<td></td>
</tr>
<tr>
<td>Fast SCR test</td>
<td>Ramp up</td>
<td>100-550°C @ 2°C/min</td>
<td>[O₂] [CO₂] [H₂O] [NO] [NO₂] [NH₃]</td>
<td></td>
</tr>
<tr>
<td>OPTION #1 – Alternative test strategy: ramp up only</td>
<td>Cool</td>
<td>600–100°C</td>
<td></td>
<td>[O₂] [CO₂] [H₂O]</td>
</tr>
<tr>
<td>Fast SCR test</td>
<td>Ramp up</td>
<td>100-550°C @ 5°C/min</td>
<td>[O₂] [CO₂] [H₂O] [NO] [NO₂] [NH₃]</td>
<td></td>
</tr>
</tbody>
</table>

(a) The procedures are the same for all combustion modes and vary only by the exhaust make-up used.
(b) The bracketed concentration values are combustion-mode dependent and found in Table 14.

### 4. DE-GREENING AND AGING

The catalyst aged state is considered a constant parameter in the protocol. Procedures for providing a realistic and representative aged catalyst are described in this section. If it is the user’s intent to characterize activity of an aged catalyst, then the procedures for providing the aged state should be performed prior to the test protocol.

Fresh catalysts should be “de-greened” prior to initial catalyst testing to ensure a common and stabilized initial state of performance. Catalyst de-greening is not included as part of the protocol test strategy, but should be done prior to activity characterization. It is important to note that the pretreatment portion of the protocol is not intended to replace adequate de-greening. Subsequent to initial catalyst testing, catalyst aging evaluation should occur. A single catalyst should be used for the different aging tests. However, if SCR activity is significantly reduced due to impact from one of the aging evaluations and it is desired to proceed with subsequent aging evaluation, then a new catalyst should be used and de-greened prior to use.

Catalyst aging for NH₃-SCR catalysts consists of a two-phase staged approach intended to accelerate the pace of catalyst evaluation. Phase 1 focuses on more conventional on-cycle aging mechanisms, including thermal aging and fuel-derived sulfur poisoning. Phase 2 focuses more on less conventional off-cycle aging mechanisms primarily surrounding extended low-temperature exhaust conditions and includes both low temperature HC exposure (and subsequent combustion) and low temperature water exposure. The staged approach is employed to quickly eliminate catalyst materials from consideration in Phase 1 that do not meet the minimum requirements associated with hydrothermal and sulfur durability. Then Phase 2 testing, which is comparatively more time consuming, can focus solely on those catalysts that show promise in Phase 1. Activity testing should be conducted following each aging event, and it is suggested to conduct the aging tests in the sequence described here.

It is important to emphasize that the staged durability testing should NOT BE misinterpreted as Phase 2 being optional, but rather solely a mechanism to expeditiously screen out candidate materials that show inferior thermal or chemical durability. These durability evaluations are critical to ensuring successful use of a candidate NH₃-SCR catalyst technology on vehicles, and have been included to replicate
aspects of vehicle use that are uniquely challenging to NH₃-SCR catalysts. The user is encouraged to perform Phase 2 testing early in the development process for any new catalyst technology to minimize time spent fruitlessly on a technology that does not exhibit the necessary durability.

4.1 Catalyst De-greening

De-greening is not intended to replace full and complete catalyst calcination; all catalysts should be fully calcined prior to de-greening. The de-greening conditions are shown in Table 4, and consist of neutral conditions (10% CO₂, 10% H₂O, balance N₂) for all gasoline applications (L-GDI, LTC-G), and lean conditions (10% O₂, 5% CO₂, 5% H₂O, balance N₂) for all diesel applications (CDC, LTC-D). The de-greening procedure consists of exposing the catalyst to the most applicable mixture for the application (Table 4), ramping from room temperature to 700°C and holding for 4 hours. The user should perform only a single de-greening per catalyst sample. The temperatures noted above for catalyst de-greening refer to the catalyst inlet temperature.

<table>
<thead>
<tr>
<th>Combustion Mode</th>
<th>Action</th>
<th>Temperature Time</th>
<th>Exhaust make-up (balance N₂)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>[O₂]</td>
</tr>
<tr>
<td>L-GDI</td>
<td>De-greening (neutral)</td>
<td>700°C/4 hours</td>
<td>-</td>
</tr>
<tr>
<td>LTC-G</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CDC</td>
<td>De-greening (lean)</td>
<td>700°C/4 hours</td>
<td>10%</td>
</tr>
<tr>
<td>LTC-D</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

4.2 Phase 1 Durability – Catalyst Thermal Aging

The thermal aging protocol is intended to be representative of anticipated in-use durability requirements, and is to consist of 16 hours continuous operation at 800°C inlet temperature. The gas composition during thermal aging is dependent on the combustion mode that the aftertreatment technology will address. It is currently expected that lean gasoline applications (L-GDI, LTC-G) will be deployed in multi-mode fashion employing SI combustion for portions of the operating profile. This dictates that de-greening and aging must defer to the most severe conditions which is SI. Thus, all gasoline combustion applications are aged in a three-cycle neutral-rich-lean strategy analogous to SI combustion.

As detailed in Table 5 and Figure 5, gasoline applications will employ a 1-minute cycle consisting of 40 seconds neutral, 10 seconds rich, and 10 seconds lean operation conducted in continuous sequential fashion for 16 hours at 800°C. Diesel applications will employ continuous lean operation over the entire 16 hours at 800°C. Users should conduct only a single 16-hour aging cycle per catalyst.
### Table 5 – Catalyst aging cycle parameters for Phase 1 durability testing

<table>
<thead>
<tr>
<th>Combustion Mode</th>
<th>Cycle Mode</th>
<th>Cycle Duration</th>
<th>Exhaust make-up (balance N\textsubscript{2})</th>
<th>[O\textsubscript{2}]</th>
<th>[CO\textsubscript{2}]</th>
<th>[H\textsubscript{2}O]</th>
<th>[CO]</th>
<th>[H\textsubscript{2}]</th>
</tr>
</thead>
<tbody>
<tr>
<td>L-GDI LTC-G</td>
<td>Neutral</td>
<td>40 seconds</td>
<td>-</td>
<td>10%</td>
<td>10%</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Rich</td>
<td>10 seconds</td>
<td>-</td>
<td>10%</td>
<td>10%</td>
<td>3%</td>
<td>1%</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Lean</td>
<td>10 seconds</td>
<td>5%</td>
<td>10%</td>
<td>10%</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>CDC LTC-D</td>
<td>Lean</td>
<td>Continuous</td>
<td>10%</td>
<td>5%</td>
<td>5%</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

**Figure 5 – Catalyst aging cycles for Phase 1 durability testing**

#### 4.3 Temperature Control during Thermal Aging

Accurately controlling the catalyst temperature during thermal aging is very important, and significant deviations in aging temperature will impact test results. Thus, adequate and repeatable temperature control during catalyst aging procedures is important, especially when sequential tests will be used to compare different catalyst formulations or designs. Although the physical and chemical changes of a catalyst due to thermal exposure are unique to different catalyst systems, the necessity for accurate and repeatable temperature control during thermal aging is universal. Typically, temperature deviation should not exceed +/- 10°C during aging.

#### 4.4 Phase 1 Durability – Sulfur Exposure

Chemical poisoning will initially focus only on transitory poisons in the form of SO\textsubscript{2} derived from in-use fuels, and should be performed as shown in Figure 6. Poisoning is to occur after catalyst pretreatment and cooling to 300°C; additional details are shown in Table 6. Note that the 5 hour poisoning step at 300°C is the only step in which SO\textsubscript{2} should be present. For core samples, a total exposure level of approximately 1 g sulfur per liter of catalyst is to be achieved by exposing the catalyst to 5 ppm SO\textsubscript{2} for 5 hours at 300°C inlet temperature (in full simulated exhaust at 30,000 hr\textsuperscript{-1} SV). For powder samples, sulfur exposure is to be performed at 200 L/g-hr under the same conditions,
resulting in approximately 7 mg sulfur exposure per gram of catalyst. After sulfur exposure, the catalyst should be tested in ‘sulfated’ form for standard SCR activity in ramp-up only fashion according to the procedures in Section 3.1. It is important to note that the catalyst should not be inadvertently desulfated by exposure to high temperature (i.e., ≥ 500°C) following sulfur exposure until after the activity has been characterized, at which point desulfation can be performed.

Once the catalyst is sulfated and tested for activity, it should then be desulfated at 600°C for 20 minutes and then re-tested. Desulfating for gasoline applications should occur in a rich exhaust mixture and catalysts for diesel applications should be desulfated in lean exhaust. If activity is not fully recovered after desulfating, then the user can choose to desulfate at progressively higher temperature with subsequent activity testing.

Table 6 – Sulfur poisoning for Phase 1 durability testing (a)

<table>
<thead>
<tr>
<th>Action</th>
<th>Temperature Time</th>
<th>Exhaust make-up (balance N₂) (a)</th>
<th>Diesel combustion modes (CDC, LTC-D)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pretreatment</td>
<td>600°C/20 min</td>
<td>[O₂] [H₂O] [CO₂]</td>
<td>[O₂] [H₂O] [CO₂]</td>
</tr>
<tr>
<td>Cool</td>
<td>600°C – 300°C</td>
<td>[O₂] [H₂O] [CO₂]</td>
<td>[O₂] [H₂O] [CO₂]</td>
</tr>
<tr>
<td>Poison</td>
<td>300°C/5 hours</td>
<td>[O₂] [CO₂] [H₂O] [NO] [NH₃] + 5ppm SO₂</td>
<td>[O₂] [CO₂] [H₂O] [NO] [NH₃] + 5ppm SO₂</td>
</tr>
<tr>
<td>Activity testing</td>
<td></td>
<td>Standard SCR activity testing (ramp up only) – Table 2</td>
<td></td>
</tr>
<tr>
<td>Desulfate</td>
<td>600°C/20 min</td>
<td>[H₂O] [CO₂] 1% CO, 0.33% H₂</td>
<td>[O₂] [H₂O] [CO₂] (b)</td>
</tr>
<tr>
<td>Cool</td>
<td>600°C – 100°C</td>
<td>[O₂] [H₂O] [CO₂]</td>
<td>[O₂] [H₂O] [CO₂]</td>
</tr>
<tr>
<td>Activity testing</td>
<td></td>
<td>Standard SCR activity testing (ramp up only) – Table 2</td>
<td></td>
</tr>
<tr>
<td>Desulfate &amp; test (if necessary)</td>
<td>650°C/20 min</td>
<td>[H₂O] [CO₂] 1% CO, 0.33% H₂</td>
<td>[O₂] [H₂O] [CO₂] (b)</td>
</tr>
<tr>
<td>Standard SCR activity testing (ramp up only) – Table 2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Repeat the above procedure as necessary to progressively higher temperature (e.g., 700°C &amp; up) for more aggressive desulfating</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(a) The procedures are the same for all combustion modes and only vary by exhaust make-up
(b) The bracketed concentrations are combustion-mode dependent and found in Table 1.

Figure 6 – Sulfur poisoning for Phase 1 durability testing
4.5 Phase 2 Durability – Low-Temperature Hydrocarbon Exposure

Low-temperature hydrocarbon (HC) exposure is used to assess the damaging effect that certain HCs may have on zeolite-based catalysts by storage and combustion. This assessment is confined to diesel combustion modes in the protocol, i.e., CDC or LTC-D. The mechanism involves low-temperature HC storage and subsequent combustion of the stored HC during a fast heat-up of the catalyst resulting in damage to the catalyst structure. In deployment, this is encountered when NH3-SCR catalysts are exposed high levels of unreacted HCs during an extended idle or cold start condition and subsequently heated quickly during vehicle operation. To assess this, the user should perform the steps described in Table 7. This consists of low-temperature HC exposure at 100°C for 1-hour with the HC mix in Table 8 immediately followed by heating of the catalyst as quickly as possible to 500°C. This should be repeated twice for a total of three (3) HC exposures and ramps to 500°C. When that is complete, the user should re-characterize performance via a standard SCR light-off test as described in Section 3.1. The reported results should include the temperature ramp rate achieved following HC exposure.

**Table 7 – Low-temperature HC exposure for Phase 2 durability testing**

<table>
<thead>
<tr>
<th>Test Mode</th>
<th>Action</th>
<th>Temperature Time</th>
<th>Exhaust make-up (balance N&lt;sub&gt;2&lt;/sub&gt;)&lt;sup&gt;(a)&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>HC exposure</td>
<td>Hold</td>
<td>100°C, 60 min</td>
<td>[O&lt;sub&gt;2&lt;/sub&gt;] [CO&lt;sub&gt;2&lt;/sub&gt;] [H&lt;sub&gt;2&lt;/sub&gt;O] [NO] [NH&lt;sub&gt;3&lt;/sub&gt;] [HC]&lt;sup&gt;(b)&lt;/sup&gt;</td>
</tr>
<tr>
<td>HC exposure</td>
<td>Heat</td>
<td>100-500°C&lt;sup&gt;(c)&lt;/sup&gt;</td>
<td>[O&lt;sub&gt;2&lt;/sub&gt;] [CO&lt;sub&gt;2&lt;/sub&gt;] [H&lt;sub&gt;2&lt;/sub&gt;O] [NO] [NH&lt;sub&gt;3&lt;/sub&gt;] [HC]&lt;sup&gt;(b)&lt;/sup&gt;</td>
</tr>
<tr>
<td>-</td>
<td>Cool</td>
<td>500-100°C</td>
<td>[O&lt;sub&gt;2&lt;/sub&gt;] [CO&lt;sub&gt;2&lt;/sub&gt;] [H&lt;sub&gt;2&lt;/sub&gt;O]</td>
</tr>
<tr>
<td>HC exposure</td>
<td>Hold</td>
<td>100°C, 60 min</td>
<td>[O&lt;sub&gt;2&lt;/sub&gt;] [CO&lt;sub&gt;2&lt;/sub&gt;] [H&lt;sub&gt;2&lt;/sub&gt;O] [NO] [NH&lt;sub&gt;3&lt;/sub&gt;] [HC]&lt;sup&gt;(b)&lt;/sup&gt;</td>
</tr>
<tr>
<td>-</td>
<td>Cool</td>
<td>500-100°C</td>
<td>[O&lt;sub&gt;2&lt;/sub&gt;] [CO&lt;sub&gt;2&lt;/sub&gt;] [H&lt;sub&gt;2&lt;/sub&gt;O]</td>
</tr>
<tr>
<td>HC exposure</td>
<td>Hold</td>
<td>100°C, 60 min</td>
<td>[O&lt;sub&gt;2&lt;/sub&gt;] [CO&lt;sub&gt;2&lt;/sub&gt;] [H&lt;sub&gt;2&lt;/sub&gt;O] [NO] [NH&lt;sub&gt;3&lt;/sub&gt;] [HC]&lt;sup&gt;(b)&lt;/sup&gt;</td>
</tr>
<tr>
<td>HC exposure</td>
<td>Heat</td>
<td>100-500°C&lt;sup&gt;(c)&lt;/sup&gt;</td>
<td>[O&lt;sub&gt;2&lt;/sub&gt;] [CO&lt;sub&gt;2&lt;/sub&gt;] [H&lt;sub&gt;2&lt;/sub&gt;O] [NO] [NH&lt;sub&gt;3&lt;/sub&gt;] [HC]&lt;sup&gt;(b)&lt;/sup&gt;</td>
</tr>
<tr>
<td>STD SCR test</td>
<td>Perform STD SCR activity testing – see Section 3.1</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<sup>(a)</sup> The bracketed concentration values are found in Table 1.

<sup>(b)</sup> See Table 8 for the exact HC blend.

<sup>(c)</sup> The catalyst should be heated to the target temperature as quickly as possible.

**Figure 7 – Low temperature HC exposure for Phase 2 durability testing**
4.6 Phase 2 Durability – Low-Temperature Water Exposure

Low-temperature water exposure is used to assess the damaging effect that condensed water may have on certain NH3-SCR catalyst formulations during prolonged exposure. Similar to low-temperature HC exposure, this is encountered during extended vehicle idling which results in exposure to high water concentrations at low exhaust temperatures. In practice, researchers commonly investigate this either by exposure of the catalyst to high water concentrations at low temperature (<100°C) for a number of hours, by immersing the catalyst in liquid water for a longer duration (e.g., 24 hours or longer), or by simply exposing the catalyst to ambient air conditions for an extended duration (e.g., weeks or months). This protocol, as described in Table 9, involves exposing the catalyst to the simulated exhaust without NOx or NH3 for 16 hours. Following the exposure, the subsequent damage is assessed by performing a standard SCR light-off test as described in Section 3.1.

<table>
<thead>
<tr>
<th>Test Mode</th>
<th>Action</th>
<th>Temperature</th>
<th>Time</th>
<th>Exhaust make-up (balance N2) (b)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pretreatment</td>
<td>Hold</td>
<td>600°C, 20 min</td>
<td>[O2] [CO2] [H2O]</td>
<td></td>
</tr>
<tr>
<td>-</td>
<td>Cool</td>
<td>600-80°C</td>
<td>[O2] [CO2] [H2O]</td>
<td></td>
</tr>
<tr>
<td>HC exposure</td>
<td>Hold</td>
<td>80°C, 16 hours</td>
<td>[O2] [CO2] [H2O]</td>
<td></td>
</tr>
<tr>
<td>STD SCR test</td>
<td>Perform STD SCR activity testing – see Section 3.2</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

It is suggested the user repeat the above steps multiple times (ideally ≥ 3 times total)

(a) The procedures are the same for all combustion modes and vary only in the exhaust make-up used.
(b) The bracketed concentration values are combustion-mode dependent and found in Table 14.
5. REPORTING

When reporting on catalyst performance, it is important to ensure that all applicable details of the catalyst sample, reactor configuration, and test conditions are adequately reported along with the catalyst performance data.

5.1 Catalyst Sample

For monolith core samples, details that should accompany catalyst test results include:
- Core length and diameter
- Cell density, i.e., cells per square inch (CPSI)
- Substrate wall thickness
- Substrate composition, e.g., cordierite, aluminum titanate
- Washcoat loading density, e.g., grams/in³

For powder samples, details that should accompany catalyst test results include:
- Mass of the catalyst sample tested
- Catalyst bed dimensions, i.e., bed length and diameter
- If possible, catalyst particle size range, i.e., mesh-size

For all catalyst samples (monolith and powder), general information is required regarding the catalyst composition to assess the manufacturability and cost of the catalyst.

5.2 Reactor Configuration

A minimum level of reactor and test configuration detail should accompany catalyst test results. This includes:
- Reactor tube (e.g., catalyst housing) description and dimensions
- Catalyst heating method (e.g., furnace description and configuration, pre-heater description)
- Location and orientation of catalyst sample within the heating apparatus
- Thermocouple description and location
- Chemical analysis technique(s) and instrumentation used (e.g., MKS MultiGas™ 2030 FTIR with 190°C heated gas cell)
- Pertinent chemical analysis sampling details, such as temperature of sampling lines and any sample conditioning performed
- Water vaporization hardware for wetting the simulated exhaust
- Liquid hydrocarbon vaporization hardware and technique, if used, for inclusion of liquid hydrocarbons (i.e., iso-octane or dodecane) in the simulated exhaust

5.3 Test Conditions

Full details of the conditions employed for testing catalyst performance should accompany the reported results, including, at a minimum, the items listed below.
- The intended engine application/combustion strategy
- The procedures used for pre-treating and measuring performance of the catalyst(s), including gas compositions, flowrate, temperatures, hold times, and ramp rates, if they are different than those specified in Tables 1, 2, and 3. If they are not, then referencing the applicable tables and engine application is sufficient
The procedures used for de-greening, hydrothermal aging, low-temperature HC and water exposure, and sulfur poisoning the catalyst(s), including gas composition, flowrate, temperature, and hold times. Again, if they are different than those specified in Tables 4 through 9 then please provide detail. Otherwise, referencing the applicable table(s) and engine application (i.e., combustion strategy) is sufficient.

5.4 Test Results/Performance Data

Reported test results should include:

- NOx and NH3 concentration [ppm] versus temperature.
- NOx and NH3 conversion [%] versus temperature.
- Preferably, byproduct formation (primarily N2O) as concentration [ppm] versus temperature.

Test results should be provided for each of the following procedures performed on the catalyst:

1. De-greening
2. Phase 1 aging evaluation
   a. Hydrothermal aging
   b. Sulfur exposure & de-sulfating
3. Phase 2 aging evaluation
   a. Low temperature HC exposure
   b. Low temperature water exposure

As mentioned in prior, a single catalyst should be used with each of the above procedures and activity testing. If a new catalyst is used for a portion of them due to detrimental impact of one of the aging evaluations, then this should be indicated along with detail of how that catalyst was processed.
APPENDIX A: PROTOCOL FLOW CHART

It is suggested that the user conduct catalyst testing in the order as it appears in Figure A1 below. An appropriate application should be selected based on the engine type (diesel vs. gasoline) and combustion mode (CDC vs. LTC-D, or LTC-G vs. L-GDI) that the catalyst is expected to support. If multiple engine types are to be investigated, they should be done so with separate samples. If the user wishes to test aged catalysts only, then aging can be conducted without requiring an initial de-greening.

Figure A1 – Test strategy flow chart for NH₃-SCR catalyst test protocol
APPENDIX B: PERFORMANCE DATA

Figure B1 – Illustrative example of NH₃ and NOx concentrations versus temperature

Figure B2 – Illustrative example of NH₃ & NOx conversion and N₂O selectivity versus temperature
APPENDIX C: POTENTIAL MODIFICATIONS

Analytical Challenges

When using a mass spec, the user must omit CO₂ from the feed to allow determination of selectivity to N₂O.

Nitrogen Balancing

Nitrogen balancing and direct determination of SCR selectivity is only feasible with a mass spec in an inert balance other than nitrogen, e.g., argon. FTIR will not allow for measurement of diatomic products (i.e., N₂). Similarly, an alternative (to N₂) inert balance is required to allow for accurate determination of selectivity to N₂.

NH₃ Temperature Programmed Desorption (TPD) Measurement

An NH₃ TPD measurement consists of (i) exposing the catalyst to NH₃ at the concentration in Table 1 without NOx or O₂ at 100°C until the catalyst NH₃ storage is saturated, i.e., waiting until [NH₃]₀UT = [NH₃]ᵢN, and then (ii) omitting NH₃ and conducting a 10°C/min temperature ramp to 550°C. The NH₃ TPD measurement will inform on strongly- and weakly-bound NH₃ and their relative distribution in the catalyst, which can provide further insight into the nature and behavior of the active NH₃-SCR catalyst sites at different temperatures.