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Formation of N_2O and NO_2 Across Conventional DeNOx SCR Catalysts

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EPRI Project Manager
A. Jimenez

ELECTRIC POWER RESEARCH INSTITUTE
3420 Hillview Avenue, Palo Alto, California 94304-1395 ▪ PO Box 10412, Palo Alto, California 94303-0813 ▪ USA
800.313.3774 ▪ 650.855.2121 ▪ askepri@epri.com ▪ www.epri.com

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This report was prepared by

Electric Power Research Institute (EPRI)
3420 Hillview Avenue
Palo Alto, California, 94304

Principal Investigator
A. Jimenez

W. S. Hinton and Associates
1612 Smugglers Cove Circle
Gulf Breeze, FL 32563

Principal Investigator
W. Scott Hinton, Ph.D., P.E.

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PRODUCT DESCRIPTION

This project investigated the formation of N_2O and NO_2 across conventional DeNOx selective catalytic reduction (SCR) catalysts. N_2O is a particularly strong greenhouse gas, and both N_2O and NO_2 may adversely impact downstream processes. Additional data related to their formation or reduction across SCR catalysts is desirable.

Results & Findings

Test results showed that the range of NOx activities was consistent with the industry fleet of conventional coal-fired boiler catalysts. SO_2 conversion values also were consistent with industry experience, even though they covered a broad range. As a result, none of the catalysts produced N_2O at levels greater than the system detection limit. The single sample tested for NO_2 production resulted in a net decrease in NO_2 across the catalyst.

Challenges & Objective(s)

This project was designed to determine if N_2O and NO_2 formation should be a concern for the current fleet of conventional SCR catalysts.

Applications, Values & Use

Test results will help evaluate behavior of current commercial SCR catalyst formulations. Because catalyst design has advanced over the years, historical data may not accurately reflect current catalyst behavior.

EPRI Perspective

Project findings determined that N_2O formation should not be a concern for the current fleet of conventional SCR catalysts. The findings also indicated that NO_2 will not be produced across conventional catalysts, but will be reduced. As catalyst formulations change over time or otherwise deviate from the current design, N_2O and NO_2 formation could be a consideration.

Approach

The project team evaluated four samples of conventional SCR catalysts under bench-scale conditions for their behavior with respect to N_2O and NO_2 production. The test program covered a broad range of conventional SCR catalyst from different suppliers. The catalysts were exposed, prior to testing, for varying lengths of time to flue gas from a commercial boiler firing fuels consistent with low-sulfur, eastern bituminous coal. The samples were then tested for N_2O production in a bench-scale reactor. The team tested a single catalyst sample for NO_2 production. Catalyst activity and SO_2 conversion testing was done in accordance with the VGB testing protocol Flue gas conditions and flow rates were typical of low-sulfur coal-fired flue gas.

The project team also examined NO_2 data from a previous EPRI project that evaluated mercury oxidation across new, aged, and regenerated catalysts for both honeycomb and plate catalysts.

Keywords

Selective catalytic reduction (SCR)
SCR catalyst
Nitrogen oxides (NOx)
Nitrogen dioxide (NO_2)
Nitrous oxide (N_2O)

ABSTRACT

This project was designed to determine if N_2O and NO_2 formation should be a concern for the current fleet of conventional SCR catalysts. The project team investigated the formation of N_2O and NO_2 across conventional DeNOx Selective Catalytic Reduction (SCR) catalysts. N_2O is a particularly strong greenhouse gas, and both N_2O and NO_2 may adversely impact downstream processes. The project team evaluated four samples of conventional SCR catalysts under bench-scale conditions for their behavior with respect to N_2O and NO_2 production. The team also examined NO_2 data from a previous EPRI project that evaluated mercury oxidation across new, aged, and regenerated catalysts for both honeycomb and plate catalysts. Test results showed that none of the catalysts produced N_2O at levels greater than the system detection limit (5 ppmvd). The single sample tested for NO_2 production resulted in a net decrease in NO_2 across the catalyst. These findings indicate that N_2O and NO_2 formation should not be a concern for the current fleet of conventional SCR catalysts.

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Background

Coal-fired flue gases generally contain a mixture of nitrogen oxides (NOx), in the form of nitric oxide (NO), nitrogen dioxide (NO_2) and nitrous oxide (N_2O). The vast majority of the total NOx is typically present as NO (>95%), with NO_2 and N_2O levels usually in the low ppm range. The relative amounts of NO, NO_2 and N_2O for any particular operating unit are strongly affected by boiler operating conditions, especially as a function of fuel type, temperature profile, and oxygen concentrations.

Historically, selective catalytic reduction (SCR) technology has focused on the removal of NO, since this is the principal NOx component. Early industry data showed that NO_2 was reduced across the vanadium-titania based catalyst, similar to the NO, and also showed that N_2O levels were consistently low throughout the system. Studies from Muzio[1] and Blanco[2] have demonstrated that N_2O does not form across vanadium-titania catalyst. They also demonstrated that components such as platinum and copper-nickel can generate N_2O. Conventional SCR catalyst formulations will vary depending on make, but the main components are titania (TiO_2), silica, tungsten, molybdenum, and vanadium.

Given that N_2O is a particularly strong greenhouse gas, and given that both N_2O and NO_2 may adversely impact downstream processes, additional data related to the formation or reduction across SCR catalysts is desirable. It is of special interest to evaluate the behavior of current commercial SCR catalsyt formulations, since catalyst design has advanced over time, and therefore historical data may not accurately reflect the current catalyst behavior.

Objectives

The primary objective of the program was to determine the behavior of conventional SCR catalysts in terms of N_2O and NO_2 production/reduction under normal operating conditions.

Catalyst Design

Four catalyst samples of conventional design were selected for testing in this program, all from different suppliers. These catalysts were utilized in alternate EPRI projects, and sampled for laboratory testing. All catalysts were designed for application to coal-fired boilers, and thus had a relatively large pitch. Latitude was given to the catalyst suppliers in terms of the activity and SO_2 conversion of the formulations provided, thus a relatively wide range of catalyst characteristics was obtained. The catalyst samples consisted of two (2) plate type catalysts, one (1) honeycomb

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[1] Muzio, L. Evaluation of Hybrid SNCR/SCR System Performance. Laguna Hills, California. 1992

[2] Blanco, J. Alumina- and titania-based monolithic catalysts for low temperature selective catalytic reduction of nitrogen oxides. Instituto de Catálisis y Petroleoquímica. Madrid, Spain. 2000.

catalyst, and one (1) corrugated type catalyst. The catalysts were designed based on the SCR reactor parameters shown in Table 1-1.

Table 1-1
SCR and Catalyst Design Parameters

Parameter	Value
Reactor Catalyst Layers	3
Reagent Type	Anhydrous Ammonia
Cleaning System	Sonic horns (one per layer)
Temperature (nominal baseline)	700°F (371°C)
Reactor NOx Removal Level	90%
Ammonia Slip	< 2 ppmv
SO_2 Conversion	Not specified
Fuel Type	Bituminous

Catalyst Exposure

Prior to N_2O and NO_2 testing, catalysts were used as part of a different research program where catalyst samples were exposed to flue gas in a slip-stream reactor attached to a 75 MW, T-fired unit firing pulverized coal. Flue gas was extracted from the host boiler prior to any ash collection. Catalysts were exposed for various lengths of time, as shown in Table 1-2. Both Catalysts C and D were relatively new; while Catalyst A was aged (catalyst life is approximately 20,000 hours, depending on fuel).

Table 1-2
Catalyst Exposure Hours

Catalyst	A	B	C	D
Approximate Exposure Hours	16,000	8,000	2,000	2,000

The host unit fuel was sourced from various locations, and included various fuel blends, but in all cases the fuels were similar to eastern bituminous coal, with low to mid-sulfur and low to mid-chlorine. Representative coal analyses which approximate the range of coals fired over the catalyst's exposure history are shown in Table 1-3.

Table 1-3
Representative Coal Analyses – Ultimate Analysis

Coal ID	**A**	**B**	**C**	**D**	**E**	**F**	**G**
State	Colombia, SA	Colombia, SA	UT	WV	WV	IL	IL
ULTIMATE ANALYSIS (%, dry basis)							
Total Moisture	12.39	9.77	6.54	5.63	5.75	11.89	11.39
Ash	7.03	6.92	10.05	16.03	14.46	8.2	7.26
HOC (Btu/lb)	13003	13366	13325	12612	12785	13570	13656
Total Sulfur	0.65	0.59	1.3	0.66	0.94	1.75	1.18
Carbon	73.63	77.94	76.95	72.01	74.12	76.87	78.49
Hydrogen	4.94	4.94	4.78	4.49	4.62	4.98	4.85
Nitrogen	1.49	1.56	1.56	1.3	1.31	1.75	1.84
Oxygen	12.26	8.05	5.36	5.51	4.55	6.45	6.38
Volatiles	37.47	37.12	35.22	31.22	30.12	34.32	32.32
Fixed Carbon	55.5	55.96	54.73	52.75	55.42	57.48	60.42
Ash Fusion IT (^{o}F)	2196	2368	2060	2541	2800	2046	2154
Ash Fusion ST(^{o}F)	2378	2574	2134	2510	2800	2134	2260
Ash Fusion HT(^{o}F)	2438	2652	2158	2645	2800	2238	2238
Ash Fusion FT(^{o}F)	2532	2728	2260	2662	2800	2340	2534
Grindability Index	40	44	45	46	44	53	52
ASH CONSTITUENTS (%, as ashed in laboratory)							
Al_2O_3	20.94	20.31	18.28	29.4	29.46	19.88	22.42
Fe_2O_3	7.13	7.48	4.37	3.75	4.4	17.13	11.85
CaO	2.49	1.23	10.51	1	0.67	2.07	1.91
MgO	1.32	1.01	3.26	1.1	0.82	0.93	1.07
MnO_2	0.03152	0.0461	0.03018		0.0164	0.03108	0.03288
P_2O_5	0.32	0.28	0.16		0.17	0.55	0.36
K_2O	1.81	1.52	1.25	2.98	2.77	2.48	2.66
SiO_2	59.62	64.72	43.63	63	59.14	52.67	55.26
Na_2O	1.49	0.56	4.57	0.346	0.2	1.06	1.28
SO_3	3.21	1.55	11.69	0.636	0.59	1.81	1.82
TiO_2	1	1	1.08	1.75	1.66	1.25	1.31

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TESTING APPROACH

Test Facility Design and Experimental Conditions

The catalysts were tested in a bench-scale facility at an independent laboratory. The testing apparatus and experimental design was consistent with typical testing for deNOx and SO_2 conversion activity.[3] Figure 2-1 shows the flow diagram for the test facility. A propane fired boiler (1) was used to generate the flue gas. The gas passes through an electric reheater (2) to assure a stable and precise temperature. The flue gas conditioning unit (3) adjusts the required SO_2 and NH_3 levels upstream (4) and downstream (6) of the catalyst chamber (5) for O_2, SO_2, and nitric oxide (NOx) and nitrous oxide (N_2O) measurement in the flue gas. Figure 2-2 shows two photographs of the laboratory test reactor, with catalyst sample being installed.

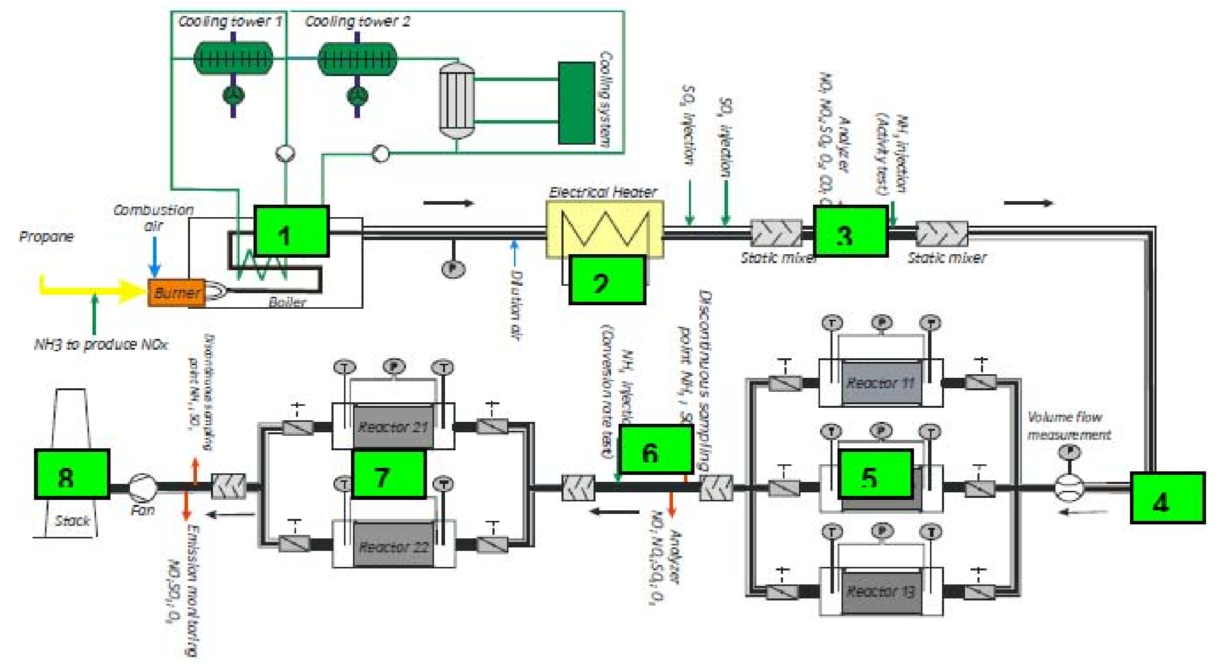

Figure 2-1
Flow Diagram for Bench-Scale Test Facility

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[3] See "Protocol for Laboratory Testing of SR Catalyst: 2nd Edition," EPRI Report No. 1014256, 12/21/2007; and "VGB Guideline for the Testing of DeNOx Catalytic Converters, VGB-R 302He, 2nd Revised Version, Published by VGB Kraftwerkstechnik, Klinkestrasse 27-31, 45136 Essen, Germany.

Figure 2-2
Photographs of Laboratory Test Reactor with Catalyst Sample

Gas Measurement

The continuous gas analysis system uses a heated filter element and sample line. A chiller removes excess moisture to a dewpoint of 4^{o}C (39ºF). After the pump and filter unit, the gas path splits to the SO_2 detector and the SO_2 scrubber. A 3% hydrogen peroxide solution removes more than 90% of the SO_2 before entering the NOx, N_2O, and O_2 analyzer. Figure 2-1 shows the gas detectors that are utilized with the bench-scale apparatus.

Table 2-1
Gas Measurement Devices

Gas	Device	Measurement Method
O_2	ABB AO2000	Paramagnetic
SO_2	ABB AO2000	UV
NOx	Ecophysics 800	CLD
N_2O	Siemens 5E	NDIR

DeNOx and SO_2 Conversion Reporting Basis

NOx Activity

The values for NOx activity (K-value) are calculated using Equation 1 below:

$$K = -Av * \ln(1-\eta_{NOx}) \qquad \text{Equation 1}$$

Where Av is the area velocity and η_{NOx} is the catalyst deNOx efficiency at an ammonia to NOx ration equal to 1.

Av is ratio of the flue gas volumetric flow rate (m^3/h) over the actual catalyst surface area (m^2) and is defined by:

$$Av = \frac{V}{A_{act}} \qquad \text{Equation 2}$$

The NOx efficiency is denoted by:

$$\eta_{NOx} = \frac{NO_{x\text{-inlet}} - NO_{x\text{-outlet}}}{NO_{x\text{-Inlet}}} \qquad \text{Equation 3}$$

where NOx-inlet is the NOx inlet concentration (ppm) and NOx-outlet is the NOx outlet concentration (ppm).

SO_2 to SO_3 Conversion

In the bench-scale reactor, the SO_2 to SO_3 conversion rate is measured without ammonia injection. This testing protocol was postulated by VGB-R302He2[4] in order to represent worst case operating conditions, given that the DeNOx reaction competes with SO_2 to SO_3 conversion in the catalyst. As a result, SO_2 to SO_3 conversion usually decreases significantly within the SCR reactor when ammonia is added to the flue gas. Thus measuring SO_2 to SO_3 conversion without ammonia ensures reproducible results and yields the highest "worse case" conversion rate possible. SO_2 to SO_3 conversion is given by Equation 4 below.

$$K_{\mathrm{SO}2} = \frac{SO_3\,out - SO_3\,in}{SO_2} * 100\,[\%] \qquad \text{Equation 4}$$

The SO_2 conversion rate (K_{SO2}) is defined as follows:

SO_3 out: SO_3 outlet concentration (ppmvd)

SO_3 in: SO_3 inlet concentration (ppmvd)

SO_2: SO_2 flue gas concentration (ppmvd)

A sufficient catalyst conditioning time with flue gas without NH_3 is important in order to generate reproducible value for the SO_2/SO_3 conversion rate.

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[4] Guideline For The Testing Of DeNOx Catalytic Converters, VGB R-302He, 2nd revised version, published by VGB Kraftwerkstechnik, Klinkestrasse 27-31, 45136 Essen, Germany, 1998.

Simulated Flue Gas

Table 2-2 shows the simulated flue gas concentrations and flow rate that were utilized for the testing. These conditions were consistent with conventional catalyst applications on coal-fired boilers, firing low sulfur coal, and having a moderate level of NOx.

Table 2-2
Simulated Flue Gas Composition and Flow Rate

Parameter	Units	Value
Flue Gas Flow Rate (for 150 mm X 150 mm test element)	m^3/h (STP, wet, actual O_2)	120
Temperature	^oF	700
NOx Inlet	ppmv (STP, dry, actual O_2)	200
SO_2 Inlet	ppmv (STP)	500
SO_3 Inlet	ppmv (STP)	TBD
NH_3/NOx Ratio (α) for activity	ppm/ppm	1.000
NH_3/NOx Ratio (α) for SO_2 Conversion	ppm/ppm	0.000
O_2	% by volume (dry)	4.0
H_2O	% by volume	10.0

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TEST RESULTS

Table 3-1 shows the results of the bench-scale testing. The actual linear and area velocities are given, along with NOx removal efficiency, activity, SO_2 conversion, N_2O production, and the pressure drop of the sample as tested. Since the tests were conducted at constant volumetric flow rate, the linear and area velocities differed according to the geometry of the particular catalyst sample being tested.

The data show that the catalysts ranged in activity from roughly 30 to 45 m/h. These activity values are consistent with conventional commercial SCR catalysts applied to coal-fired boilers. The SO_2 conversion rates ranged from a low of 0.12 to a high of 0.86. Although this is a relatively broad range, it is well within the industry experience where different requirements and operating philosophies allow for a relatively broad range of SO_2 conversions to be utilized.

In terms of N_2O production, none of the catalysts produced a significant amount of N_2O, since all test data showed outlet N_2O levels of < 5 ppmvd. Note that N_2O testing included three NH_3/NOx ratio (α) conditions. The single catalyst tested for NO_2 production showed a reduction in NO_2 from 2.7 ppmvd at the inlet to <1 ppmvd (the detection limit) at the outlet. Thus, no net NO_2 was produced by the catalyst. The exact rate of NO_2 reduction could not be determined, given the low inlet level and the comparatively high detection limit. The data do show, however, that the NO_2 reduction rate was at least 63%. Note that the overall NOx reduction for the NO_2 test was roughly 83%.

Table 3-1
Test Results

Parameter	Catalyst A	Catalyst B	Catalyst C	Catalyst D
Linear Velocity (actual m/s)	3.52	3.36	3.45	3.53
Area Velocity (actual m/s)	25.0	21.1	23.9	31.1
NOx Removal Eff. (%)	69.1	84.1	84.6	72.5
Activity (m/h)	29.4	38.7	44.7	40.1
SO_2 Conversion Rate, as tested at α=0 (%)*	0.86	0.12	0.33	0.33
N_2O production (ppmvd)				
α = 0.00	<LDL**	<LDL	<LDL	<LDL
α = 0.66	<LDL	<LDL	<LDL	<LDL
α = 1.00	<LDL	<LDL	<LDL	<LDL
NO/NO_2 Data (ppmvd)				
NO_{in}		197.9		
NO_{2in}		2.7		
NO_{out}		33.7		
NO_{2out}		<1		
Pressure drop, as tested (mbar)	0.35	0.59	0.69	0.31

* α is the ammonia to NOx ration
** LDL: Lower than detection limit – 5 ppmvd N_2O

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SUPPLEMENTAL NO_2 DATA

Background

A previous EPRI project examined the mercury oxidation behavior of new, aged, and regenerated SCR catalyst in a three-train pilot facility for their behavior with respect to mercury oxidation.[5] Although NO_2 behavior was not a focus of the project, NO_2 data were acquired as a matter of course during the project testing.[6] These data has been reviewed and summarized in support of this project.

Catalyst Characteristics

Two groups of catalyst types were evaluated in the above referenced project, one including honeycombs, and one other including plates. New, aged, and regenerated versions of each of these catalysts were evaluated in the pilot facility, for a total of six (6) distinct catalyst versions. Both catalyst types were consistent with current commercial SCR catalysts for application to high-dust coal fired boiler flue gas. The exposure conditions of the aged catalsyt samples are summarized below in Table 4-1. New versions of the catalysts were sourced from the manufacturer and were of identical formulation to that of the aged catalyst. Regenerated versions of the catalysts were obtained by regenerating samples of the aged catalysts following full-scale regeneration practices. Additional information on the catalysts can be found in the above referenced reports.

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[5] See "Mercury Oxidation Behavior of New, Aged, and regenerated SCR Catalysts," EPRI Report No. 1014252, 12/20/2007; and "Regenerated Plate Type SCR Catalsyt Performance," EPRI Report No. 1018487. 1/26/2009.

[6] NOx and other gaseous constituents were measured via continuous FTIR spectroscopy, which gave a wide range of potential analytes.

Table 4-1
Exposure Conditions of Characteristics of Aged Catalyst Samples

Parameter	Honeycomb Catalyst	Plate Catalyst
Exposure Hours	20,000	22,000
Boiler Configuration	T-Fired	T-Fired
Coal	Central Appalachian	Central Appalachian
Coal Sulfur Content	1.2 %	1.4 %
Inlet NOx	0.45 %	0.48 %
Design DeNOx	90 %	90 %
Activity (m/hr)		
New	48.1	48.3
Aged	35.1	28.0
Regen	42.0[7]	36.9
SO_2 Conversion (%)		
New	0.31	0.17
Aged	0.36	0.16
Regen	0.43[5]	0.40

Pilot Plant Operating Conditions

The pilot plant operated as a slip stream facility attached to a 150 MW T-fired boiler, firing low-chlorine central Appalachian coal. Figures 4-1 and 4-2 depict the pilot-scale reactor and a catalyst test module respectively. Table 4-2 gives the nominal operating conditions for the tests. Two layers of catalsyt were utilized in the pilot reactors, making the overall configuration similar to a full-scale SCR application. Additional data related to the pilot plant design, operation, and host boiler can be found in the previously referenced EPRI reports.

Table 4-2
Nominal Pilot Plant Operating Conditions

Parameter	Value
Space Velocity	4,000 hr^{-1} @ 68°F (20°C)
Temperature	650°F (343°C)
Reactor DeNOx	90 %
Inlet NOx	225 ppm
SO_2	850 ppm
O_2	6 %
CO_2	12 %
CO	7 ppm
Chlorine	5 ppm

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[7] The regeneration process resulted in two distinct groups of regenerated catalyst being produced, due to some exposure differences between the various catalysts elements that were being treated. The values for activity and SO_2 conversion represent the average of these two batches of regenerated catalyst, and are representative of the activity and SO_2 conversion for the reactor as a whole, as installed in the pilot plant.

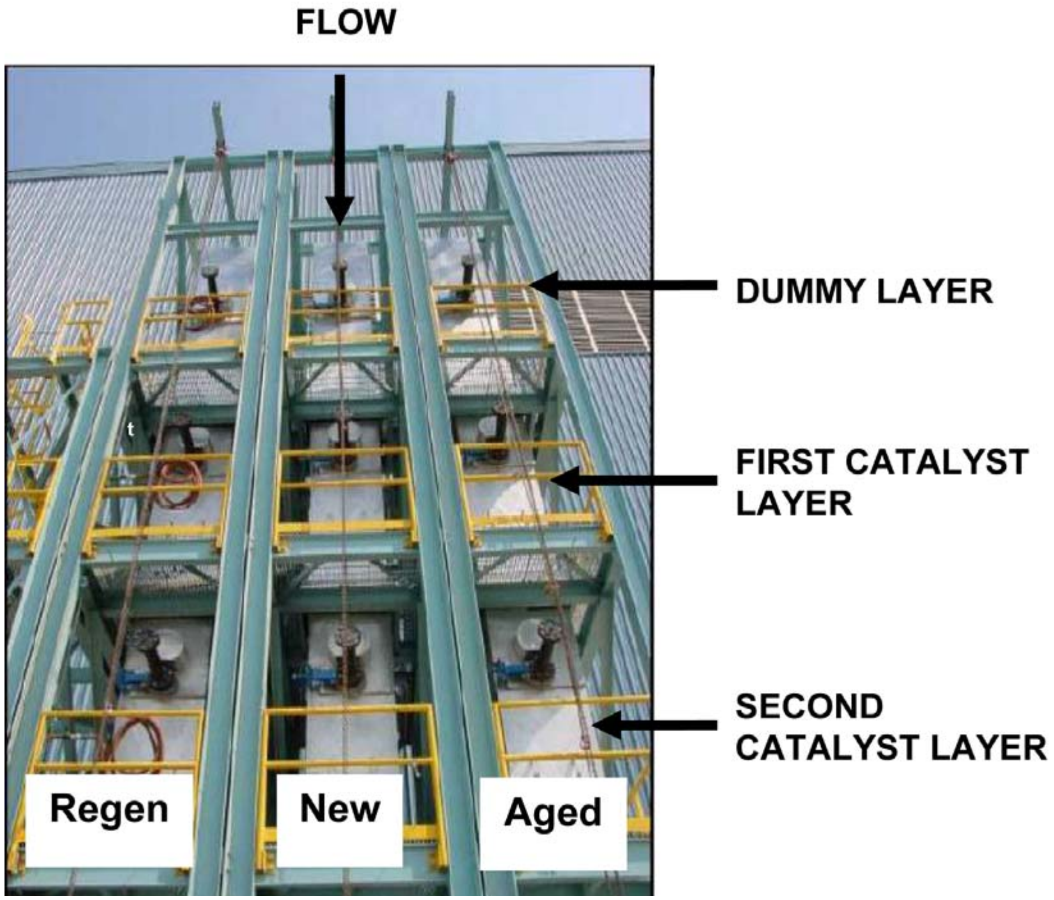

Figure 4-1
Three Train Pilot Scale Reactor

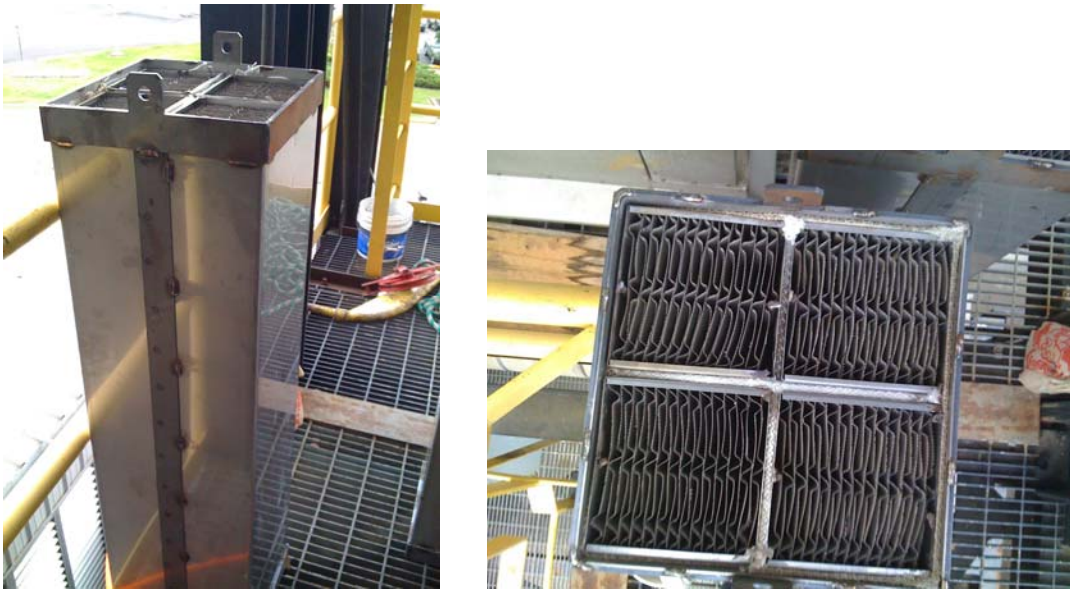

Figure 4-2
Photograph of Pilot Catalyst Module (side and top views)

Reference Flue Gas Constituents

Reference flue gas constituents, including H_2O, NO_2, NO, SO_2, HCl, NH_3, CO_2, and CO were measured using a Fourier Transform Infrared (FTIR) Spectrometer. FTIR technology allows for the simultaneous measurement of target flue gas constituents based upon infrared spectra. The low detection limits of the instrument along with near real-time response, made this measurement technique highly suitable for the application. A MKS Multigas™ 2030 FTIR instrument[8] was utilized. The sample taken from the QSIS probe was transported to the FTIR through a heated stainless steel sample line. The line was heated to approximately 450ºF (232ºC) to ensure no loss of ammonia during transport. The sample flow through the FTIR was induced by a vacuum pump at a flow rate of about 2 liters per minute. The oxygen content of the flue gas exiting the FTIR was measured periodically with a hand-held oxygen analyzer. A background zero check was conducted each day using nitrogen from gas bottles. Calibration spectra for each gas were held internally in the FTIR. This provided for accurate real-time display and data logging of actual measured gas concentrations (wet basis) in the sampled flue gas.

Test Results

The NO_2 data from the pilot plant testing is shown below in Table 4-3. In all cases, the measured NO_2 level was below the detection limit of approximately 1 ppm. This indicates that there was very little formation of NO_2 in the boiler itself, and that the catalyst did not produce any additional NO_2 (at least within the sensitivity of the measurement method). Since these data were acquired for new, aged, and regenerated catalysts of both honeycomb and plate type, there is some confidence that catalyst aging or regeneration does not adversely affect the NO_2 behavior of the catalysts.

---

[8] Manufactured by MKS Instruments , 90 Industrial Way, Wilmington, Massachusetts 01887

Table 4-3
Pilot Plant NO_2 Test Results

Catalyst	**Honeycomb**			**Plate**		
	New	Aged	Regen.	New	Aged	Regen.
NO_2 In	< 1 ppm	< 1 ppm	< 1 ppm	< 1 ppm	< 1 ppm	< 1 ppm
NO_2 Out	< 1 ppm	< 1 ppm	< 1 ppm	< 1 ppm	< 1 ppm	< 1 ppm

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CONCLUSIONS

The test program covered a relatively broad range of conventional SCR catalysts, with designs consistent with coal-fired applications. The activity and SO_2 conversions values of the catalysts were consistent with industry experience. Within the sensitivity of the experimental design, none of the catalysts produced detectible amounts of N_2O. Assuming that these results apply to the commercial conventional SCR catalyst fleet as a whole, it does not appear that N_2O formation will be of concern for conventional catalysts. However, as catalyst formulations change over time or otherwise deviate from the current normal conventional catalyst design, N_2O formation may be a consideration. Similar to the findings for N_2O, the data indicate that the conventional catalysts tested do not have the propensity to produce NO_2. Limited data indicate that NO_2 is reduced across the catalyst similar to NO, but the exact rate of NO_2 reduction could not be determined with the available data. As with N_2O formation, changes in catalyst design may result in NO_2 formation/reduction characteristics which differ from the current findings.

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Electric Power Research Institute
3420 Hillview Avenue, Palo Alto, California 94304-1338 • PO Box 10412, Palo Alto, California 94303-0813 • USA
800.313.3774 • 650.855.2121 • askepri@epri.com • www.epri.com