NO\(_x\) Removal with Combined Selective Catalytic Reduction and Selective Noncatalytic Reduction: Pilot-Scale Test Results

Brian K. Gullett  
U.S. Environmental Protection Agency  
Research Triangle Park, North Carolina

M. Linda Lin  
Nalco Fuel Tech  
Naperville, Illinois

Paul W. Groff  
Acurex Environmental Corporation  
Research Triangle Park, North Carolina

James M. Chen  
Engelhard Corporation  
iselin, New Jersey

Pilot-scale tests were conducted to develop a combined nitrogen oxide (NO\(_x\)) reduction technology using both selective catalytic reduction (SCR) and selective noncatalytic reduction (SNCR). A commercially available vanadium- and titanium-based composite honeycomb catalyst and enhanced urea (NH\(_2\)CONH\(_2\)) were used with a natural-gas-fired furnace at a NO\(_x\) concentration of 110 ppm. Changes in SNCR chemical injection temperature and stoichiometry led to varying levels of post-furnace ammonia (NH\(_3\)), which acts as the reductant feed to the downstream SCR catalyts. The urea-based chemical could routinely achieve SNCR plus SCR total NO\(_x\) reductions of 85 percent with less than 3 ppm NH\(_3\) slip at reductant/NO\(_x\) stoichiometries ranging from about 1.5 to 2.5 and SCR space velocities of 18,000 to 32,000 h\(^{-1}\). This pilot-scale research has shown that SNCR and SCR can be integrated to achieve high NO\(_x\) removal. SNCR provides high temperature reduction of NO\(_x\) followed by further removal of NO\(_x\) and minimization of NH\(_3\) slip by a significantly downsized (high-space velocity) SCR.

Introduction

The 1990 Clean Air Act Amendment requirements for control of nitrogen oxides (NO\(_x\)) may be met by various control technologies depending on the combustion system application and the level of control needed. These technologies include selective catalytic reduction (SCR), which involves a heterogeneous reaction of NO\(_x\) and ammonia (NH\(_3\)) on the catalyst surface to form nitrogen (N\(_2\)) and water (H\(_2\)O) at temperatures typically ranging from 300 to 450 °C. These catalyst beds are often honeycomb or plate structures formed from metal oxides, zeolites, or noble metals. Oxides of the transition metals, including Co, Fe, Ni, V, and W, are usually most reactive at temperatures above 250 °C. Both pilot- and field-scale results are available for SCR technology, showing NO\(_x\) removals above 90 percent. Most commercial SCR systems are designed to achieve 75 to 90 percent NO\(_x\) conversion.

SCR systems were installed at 250 commercial plants as of 1990, including 40 coal-fired applications totaling 11,000 MW(e) of capacity. In California, more than 75 gas turbine units are equipped with SCR systems. Key performance criteria affecting the design of a commercial SCR system are NO\(_x\) conversion, NH\(_3\) emissions (NH\(_3\)slip), pressure drop, catalyst/system life, sulfur dioxide (SO\(_2\)) oxidation to sulfur trioxide (SO\(_3\)), and operating environments. Because these constraints can vary greatly from one installation to another, SCR catalysts of different physical and chemical properties have been developed to meet these commercial needs. The main concerns with respect to SCR technology include the potential for particulate fouling and catalyst poisoning by trace metals, SO\(_2\), and SO\(_3\). Particulate fouling and catalyst poisoning may reduce catalyst life, which is one of the most important factors in SCR economics. Catalyst poisoning occurs by reaction of solid, liquid, or gaseous matter with the catalyst or through formation of a masking layer over the surface of the catalyst that blocks diffusion of NO\(_x\) and NH\(_3\) to the available reactive sites.

Implications

Continued research, development, and demonstration of the combined SCR/SNCR process will present an alternative technology for use in achieving NO\(_x\) reductions. This will expand the available control options and allow for staged implementation of technologies to achieve the mandates of the 1990 Clean Air Act Amendments. The successfully combined technologies emphasize the importance of structuring the regulations to allow maximum flexibility in the choice of compliance methods without restricting options to specific technologies. This further allows for advances and new developments in technologies to improve the performance of existing systems.

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Selective noncatalytic reduction (SNCR), which involves high-temperature furnace injection of a solid, gaseous, or liquid N-based reducing agent such as urea (NH₂CONH₂) or NH₃, reacts homogeneously to convert NOₓ to N₂. SNCR is a proven technology that has application to utility and industrial boilers burning a variety of fuels and waste-to-energy facilities. With this type of reduction, reaction temperatures range from 800 to 1100 °C. SNCR has achieved about 60 percent NOₓ reduction on a coal-fired 150 MW(e) boiler at a molar ratio of reductant N to initial NOₓ concentration (normalized stoichiometric ratio or NSR) of 2.5 to 7 percent NOₓ reduction on a 15 MW(e) coal-fired boiler at an NSR of 1.2 to 1.6.² and has been the subject of numerous laboratory or pilot-scale studies.² Pilot-scale laboratory studies have shown NOₓ reduction around 60 to 80 percent depending on initial NOₓ and chemical stoichiometry with levels of NH₃SLIP and nitrous oxide (N₂O) below 5 and 20 ppm, respectively.²

The potential problems of using SNCR center around byproduct emissions of NH₃ (NH₃SLIP), which result from incomplete reaction of the injected reductant. NH₃SLIP is generally caused by operation outside of the effective temperature range. NH₃SLIP can react with SO₂ to form ammonium bisulfate (NH₃₂HSO₄) around 275 °C and ammonium sulfate ([NH₄]₂SO₄) around 150 °C. The ammonium salts can form deposits on heat-transfer surfaces causing reduced heat transfer and increased pressure drop. NH₃SLIP can also react with hydrogen chloride (HCl) or chlorine (Cl₂) to form ammonium chloride (NH₄Cl) around 100 °C resulting in a visible white plume in the stack emissions.

This research investigated the combination of SNCR and SCR technologies with the aim of demonstrating a more economical method of high NOₓ removal. This "hybrid" process aims to achieve combined NOₓ removal from the SNCR process initially and then further NOₓ removal from the SCR downstream. Because significant NOₓ reductions can be achieved by the upstream SNCR, downsizing of the SCR for only supplemental NOₓ reduction becomes possible. The SCR catalyst utilizes the normally undesired NH₃SLIP as the SCR reactant feed to further reduce NOₓ as it minimizes NH₃SLIP problems. Additional NH₃ can be added upstream of the catalyst, if necessary. The smaller catalyst bed (higher space velocity) and elimination of reheat requirements will result in considerable cost savings through minimizing catalyst costs and equipment modification costs such as, reactor size, ductwork expansion, and fan size. The smaller catalyst volume may also reduce the potential for oxidation of SO₂ to SO₃ and subsequent formation of catalyst-plugging ammonium salts.

An adaptation of this technology has undergone full-scale testing by others on a 110 MW gas/oil-fired boiler using in-duct and air heater catalysts. SNCR-urea injection and SCR-ammonia injection resulted in NOₓ reductions varying from 72 to 91 percent, depending on boiler load.

Pilot-scale work on a natural gas combustor examined the optimum combination of SNCR and SCR by varying SNCR injection temperature and chemical injection rate along with SCR space velocity and NH₃ feed rate. Emissions were measured for NOₓ, NH₃, N₂O, and combustion gases. The limited scope of work reported here discusses only one SNCR reductant chemical and SCR catalyst, although other chemicals and catalysts also were tested.

Experimental

SNCR/SCR tests were run on the U.S. Environmental Protection Agency's Innovative Furnace Reactor (IFR), a pilot-scale 14.7 kW (50,000 Btu/h), refractory lined, natural-gas-fired cylindrical furnace (see Figure 1). The IFR has an inner diameter of 15.2 cm and an overall vertical length of about 4 m. Several ports,

![Figure 1. Schematic of the innovative furnace reactor (IFR) with the hybrid system.](image)

spaced approximately 30 cm apart, are located along the vertical length of the furnace and can be used for sampling probes or chemical injection probes. The furnace simulates the gaseous combustion environment and quench-rate conditions anticipated in utility and industrial boilers. Typical operating concentrations were 110 ppm NOₓ dry measured values are adjusted for actual in-furnace water concentrations. The IFR total gas flow is 391 L/min. Reported values are at standard temperature, 0 °C, and pressure, 1 atm (STP), including an excess air of 50 percent. All monitored gases are adjusted to account for air in-leakage, most of which occurs near the elbow, resulting in typical operating oxygen (O₂) concentrations ranging from 5.1 to 6.5 percent and a NOₓ concentration of about 95 ppm at the SCR.

Two 4.72 cm inner diameter stainless steel arms were added in parallel to the IFR horizontal sampling stack to hold the SCR catalysts. The tested catalyst was loaded into the catalyst arms in three 20.32 cm sections with about 2.5 cm spacing between each pair of sections. The flow from the furnace could be run through one or both of the catalyst arms, yielding two space velocities. Both catalyst arms were heat-traced and insulated to maintain a constant, monitored temperature of 315 °C.

Gas sampled from the horizontal sampling stack of the IFR was passed through sample lines to continuous-emission monitors (CEMs). Dry NOₓ measurements were determined by a chemiluminescent method and reported as wet values at actual O₂ concentration. The reported NOₓ concentrations do not include NO, because earlier tests showed that the NO₂ concentrations were below 5 percent of the total NO concentration.

Gas samples for carbon dioxide (CO₂), carbon monoxide (CO), and O₂ were first passed through a Hankinson gas dryer and a dessicant carister of anhydrous calcium sulfate (CaSO₄). The desiccant was not used with the NOₓ analyzer because CaSO₄ absorbs NO₂. All of the above on-line CEMs were zeroed and spanned with gases of known concentration both before and after each daily trial. N₂O concentrations were monitored by on-line gas chromatography (GC). The GC was used for analysis of grab samples taken before and during testing.

The concentrations of stack gas NH₃ were determined by two wet methods. The first was a batch method using a Fisher Accumet ion-selective electrode with a temperature-compensating probe. The second was a continuous-sampling system by Severins Science Instruments (SSI) that operates under the same principle. Both methods remove water for dry and humidity measurements, respectively. The first uses dual impingers in ice, and the second uses a room temperature condensing impinger. A comparison of
results between both methods showed excellent agreement when the SSL system was calibrated within a limited range of interest. The stack gas was drawn through an impinger system containing 0.02N H2SO4, and the pH was then adjusted with 10M NaOH solution. The NH4+ ion selective electrode, coupled with a pH meter, determines the NH3 concentration. The meter and electrode were calibrated prior to analyses with known standards and checked throughout the testing to ensure that the values fell within the manufacturer’s limits. NH3 values, from both methods, were reported at actual H2O and O2 concentrations.

Urea Solution Injection

Testing during this work used injection of a proprietary urea-based solution with the trade name NO3OUT A PLUS (Nalco Fuel Tech). NO3OUT A PLUS, hereafter referred to as “enhanced urea,” is a urea-based solution with antiscalants, dispersants, and more reactive ammonium compounds that shift the equilibrium point of the N2O formation mechanism away from the N2O product.

Different ratios of urea/NO3 were provided to the furnace by varying the concentration of the enhanced urea solution while maintaining a constant volumetric injection rate. A peristaltic pump was used to deliver low concentrations of reagent to the furnace. All tests proceeded with determination of stable baseline emissions during 100 percent H2O injection to simulate the SNCR’s supply of chemical. When baseline emissions stabilized, the chemical solution was injected at the desired urea/NO3 stoichiometry. After completing emissions analyses, H2O only was injected until the IFR returned to baseline emission concentrations.

Solutions were injected through a water-cooled probe that injected coaxially and coflowing to the process gas. The injection probe uses air (15 percent of the total IFR air flow) to effect droplet atomization. The droplet-size distribution exiting the nozzle was determined through the use of a Malvern particle size analyzer, which determines droplet size by measuring diffraction of laser light. These droplet sizes were measured outside of the IFR using H2O flow rates and pressures identical to in-furnace operation. The nozzle had a droplet-size distribution with a D50 of 13 µm and a D90 of 88 µm.

A commercial V2O5-based SCR catalyst, NO3Cat™ VNX™, was used to test the catalytic reduction efficiency of NO3 at 300 to 400 °C. This catalyst, supplied by Engelhard Corporation, is a composite formulation of V2O5/NO3, materials bonded to a ceramic honeycomb substrate. The catalyst specific area can be varied by using honeycombs of different cell density, ranging from 4 to 31 cells per cm2 (25 to 200 cells per inch), for most industrial applications. Increasing cell density, which increases catalyst specific area, improves the volumetric rate for NO3 reduction since the reaction is diffusion limited. However, it also increases the pressure drop at the same space velocity. Thus, a compact SCR system may be designed using catalyst honeycombs with high cell density. For this study, a 9,9 cells per cm2 (64 cells per inch) VNX™ catalyst was chosen for measuring SCR catalyst effectiveness at removing NO3 with enhanced-urea solution. Measured pressure drops were 1.8, 2.2, and 2.9 cm of water for 18,000, 24,000, and 32,000 hr−1 space velocity, respectively. The reductant was supplied solely by residual, unreacted NH3 from the SNCR process. This NH3 was measured prior to the catalyst inlet (see Figure 1) and termed NH3RES.

Temperature Profiles

The temperature profiles through the IFR were determined using a suction pyrometer and a type R thermocouple. Temperatures were determined during injection of air and H2O to mimic the conditions expected during injection of the SNCR and SCR reagents. The temperature at the point of the injection nozzle was calculated by linear extrapolation of the temperature values from downstream ports. The quench rate for natural-gas-firing with injection was nominally 114 and 240 °C/s at the low- and high-injection ports, respectively.

Tests were conducted at a limited range of operating conditions by varying the SNCR injection port level (TNO3), the SCR gas-flow rate or space velocity (SV), and the amount of chemical injection. Operating conditions were chosen based on earlier work that had defined the general system performance over a broad range of TNO3, SV, and NSR. The enhanced urea feed rate into the SNCR system is called the Normalized Stoichiometric Ratio (NSR) and is a ratio of the available N reductant groups to the baseline NO3 concentration NO3. Urea-based chemicals decompose into 2 moles of reductant N per mole of urea. The value of NSR is calculated by multiplying the concentration of enhanced urea solution (moles N/mL) by the solution feed rate (10 mL/min) then dividing by the measured NO3. The NH3 feed into the SCR system is supplied solely by the unreacted NH3 from the SNCR process, NH3RES. The rate of feed is termed SRRES, where

\[
SR_{RES} = \frac{NH_3{RES}}{NO_3}.
\]

Here, NH3RES refers to the amount of residual NH3 reductant prior to the catalyst, and NO3RES is the amount of NO3 residual after SNCR and prior to the catalyst. SRRES is determined from the sampled NH3 concentration prior to the catalyst.

SCR tests were first conducted with enhanced urea injection to establish the baseline performance characteristics. These tests were rerun at the end of the test matrix and ensured that no degradation in catalyst performance had occurred.

Results and Discussion

Figure 2 shows the general optimum operating range of an SNCR system for NO3 removal (XSNCR) as a function of TNO3 and NSR. The contour plot fit to the data shows that between 850 and 950 °C, NO3 removals of 60 to 75 percent were achieved at an NSR of greater than 1.5 (NO3 = 110 ppm). A few chemical injections were made beyond the optimum SNCR injection-temperature window, simulating sudden load swings in a full-scale plant. At higher TNO3 cases, the enhanced-urea chemical will undergo partial oxidation, creating NO2. This would create more
of a NOx burden on the downstream SCR. At injection temperatures below the optimum, considerable reaction inefficiency is expected, leading to lower values of NOx reduction and high levels of NH3RES. In either the high- or low-injection temperature case, the XSCR values shown on Figure 2 are considerably lower than at the middle injection temperatures.

Figure 3 illustrates the NSR and TINJ conditions under which NH3RES from the SNCR operation provides the reductant for the downstream SCR operation. NH3RES is expressed here as a ratio with NOxRES (the residual NOx prior to the SCR that was unreduced during SNCR) or the SCR stoichiometry, SrES. Figures 2 and 3 are printed with the same ranges to facilitate direct comparison. Because NH3 measurements were not made for all of the tests, Figure 3 will have less data points than Figure 2. The majority of the tested SNCR conditions provided a functional reductant to NOx ratio, approximately 6.4 to 1.2, into the SCR system without the necessity of additional precatalyst reductant feed.

The NOx removal performance of the SCR system (XSCR) is shown in Figure 4 plotted against SrRES. The reductant chemical for the SCR, NH3RES, is supplied solely from the enhanced urea injected with the SNCR operation. Prior SCR-only testing with enhanced urea fed into the endplate, without SNCR injection, showed no distinguishable performance differences from when the reductant was supplied solely by NH3RES from SNCR operation. This implies that the residual reductant species from the SNCR enhanced urea chemical performs similarly to NH3 injected ahead of the catalyst bed, regardless of the SCR TINJ.

Figure 4 indicates that SCR NOX removal is a function of stoichiometry, SrRES; values of SrRES approaching 1.0 led to NOx removals above 90 percent, in most cases. While space velocity was varied from 18,000 to 32,000 hr−1 during the testing, no obvious effect was observed. This likely results because under these high-conversion, low-SrRES conditions, the catalyst NOx conversion is limited by available NH3 (or SrRES) not by residence time in the SCR. However, as the SV increases further, the NOx conversion will begin to decrease with increasing flow rate as the NH3 has insufficient time to diffuse and react.

The SCR NOX removal can be compared with its NH3SLIP results, Figure 5. In the SrRES range from 0.9 to 1.0, Figure 5 shows that NH3SLIP starts to increase above 5 ppm. Figure 4 shows that XSCR has leveled off at above 90 percent removal. The data show that the NH3SLIP performance of the SCR, at least below a stoichiometry of unity, is apparently independent of both space velocity (SV) and the input NOx value, NOxRES.

**Hybrid SCR/SNCR**

The NOx and NH3SLIP performance results of the combined SNCR and SCR systems are shown in Figure 6. Because of the large amount of NH3SLIP values less than 1 ppm, the ordinate is plotted on a logarithmic scale for clarity. All of the data at ≤ 85 percent total NOx reduction had less than 1 ppm NH3SLIP. Because the data represent varying test conditions of NSR and TINJ, both optimal and suboptimal operating conditions are shown. This illustrates the importance of proper operation, monitoring, and control of the SNCR system toward achieving consistently optimum hybrid process NOx and NH3SLIP Performance. Although Figure 6 also indicates variation of SV (18,000 to 32,000 hr−1), no relation to NOx reduction or NH3SLIP results was apparent with the limited number of tests conducted.

Figure 7 plots the percentage of unconverted NH3 against SrRES where the former is defined by

\[
\text{NH3 UNC} (\%) = \frac{\text{NH3 SLIP}}{\text{NH3 RES}} \times 100.
\]

(2)

This is similar to Figure 5, but now also discriminates the data by hybrid versus SCR-only operation. All of the results show less
than 5 percent unconverted ammonia until SR\textsubscript{RES} values, exceeding about 0.9, are reached. No distinguishable dependency on NO\textsubscript{RES} can be noted. No apparent difference in catalyst function is noted between the SCR-only results and the hybrid results.

While not the main focus of this work, measurements of pre- and post-SCR N\textsubscript{2}O showed concentrations that were always below 7 ppm. Baseline CO was always below 1.5 ppm, increasing to 27 ppm during testing.

Optimization of the hybrid SCR/SNCR system performance must take into account a balance between NO\textsubscript{x} reduction and NH\textsubscript{3}SLIP. The latter can be illustrated by combining Equations 1 and 2 into a definition of NH\textsubscript{3}SLIP:

\[ \text{NH}_{3}\text{SLIP} = \text{NH}_{3}\text{UNC} \ast \text{SR}_{\text{RES}} \ast \text{NO}_{\text{RES}} \]

where

\[ \text{NH}_{3}\text{UNC} = f(\text{cat.type}, \text{SV}, \text{cat. T}). \]

Equation 3 shows that NH\textsubscript{3}SLIP can be minimized by reducing either the SR\textsubscript{RES} or NO\textsubscript{RES} term. Thus, if either lower NO\textsubscript{RES} reaches the SCR or the SCR can be operated at a lower SR\textsubscript{RES} value — still achieving the target overall NO\textsubscript{x} reduction — then the amount of NH\textsubscript{3}SLIP will be reduced.

Minimization of both NO\textsubscript{RES} and SR\textsubscript{RES} depends on high levels of SNCR reagent utilization achieving significant reductions in NO\textsubscript{x} (lowering NO\textsubscript{RES}) with sufficient, yet not excessive, amounts of unconverted reductant (NH\textsubscript{3}UNC) passed on to the catalyst. Optimization of the hybrid system is achieved when the SNCR technology is operated at peak performance, maximizing NO\textsubscript{x} reduction while achieving a NH\textsubscript{3}UNC value that results in an SR\textsubscript{RES} value into the catalyst that will not result in excessive NH\textsubscript{3}SLIP.

This concept is developed as follows for a facility with a hypothetical limit of 3 ppm NH\textsubscript{3}SLIP and a NO\textsubscript{x} = 110 ppm. First, for a range of assumed SNCR NO\textsubscript{x} reductions, the maximum allowable unconverted ammonia, NH\textsubscript{3}UNC, or 3 ppm * 100 / NO\textsubscript{RES}, is associated with its experimentally determined SR\textsubscript{RES} from Figure 7. This value of SR\textsubscript{RES} is the highest stoichiometry at which the SCR can operate without exceeding 3 ppm NH\textsubscript{3}SLIP. The anticipated SCR NO\textsubscript{x} reduction at this SR\textsubscript{RES} value is determined from previous experimental results reported in Figure 4.

Finally, these results are plotted in Figure 8 showing the optimal performance of the SCR/SNCR system given an assumed effectiveness of the SNCR process and a limit of 3 ppm NH\textsubscript{3}SLIP shown using the solid line on Figure 8. Of course, these results are a strong function of the SCR’s performance (see Equation 4) and the initial NO\textsubscript{x} level in the facility. The latter is an issue because, for a given SNCR percentage NO\textsubscript{x} reduction, as NO\textsubscript{x} increases the fraction of allowable unconverted ammonia decreases. This results in lower tolerable levels of SR\textsubscript{RES} and, therefore, lower SCR NO\textsubscript{x} reduction. Full-scale applications may achieve lower performance because of reactant-mixing limitations causing non-uniform reagent distributions. Use of computational fluid dynamics and kinetic modeling, coupled with appropriate injector and flow design criteria, can ameliorate these limitations.

Our results can be extrapolated to higher NO\textsubscript{x} systems, given the assumptions:

1) SCR NO\textsubscript{x} reduction performance, at a given value of SR\textsubscript{RES}, is not sensitive to NO\textsubscript{RES} and can therefore be approximated by a curve fit to Figure 4; and
2) SCR NH\textsubscript{3}UNC performance, at a given value of SR\textsubscript{RES}, is not sensitive to NO\textsubscript{RES} and can be approximated by a curve fit to Figure 7.

Both of these assumptions are validated by the results of this research over the range 23 ppm < NO\textsubscript{x} < 662 ppm. The SCR

![Figure 6. Combined hybrid system performance: total NO\textsubscript{x} removal and NH\textsubscript{3} slip. (756 °C < T\textsubscript{INJ} < 952 °C, NO\textsubscript{INJ} = 103 to 126 ppm).](image)

![Figure 7. Effect of SR\textsubscript{RES} on unconverted NH\textsubscript{3}, NH\textsubscript{3}UNC, for the ranges of NO\textsubscript{RES}.](image)

![Figure 8. Optimal-operating conditions and performance for the hybrid SCR/ SNCR system at NO\textsubscript{x} = 110 ppm based on the maximum allowable SR\textsubscript{RES} that will not exceed 3 ppm NH\textsubscript{3}SLIP. From results presented in Figures 4 and 7.](image)
system's NO\textsubscript{2} reduction performance had no apparent dependency on NO\textsubscript{2} which is not shown, and Figure 7 indicates that NH\textsubscript{3} performance was insensitive to NO\textsubscript{2} over the same range.

For a system with NO\textsubscript{2} = 500 ppm, the results in Figure 9 show a higher incremental benefit of the hybrid system (versus SCR alone) than in Figure 8. NO\textsubscript{2} reduction increases from about 50 percent with the SCR alone to over 90 percent when SNCR achieves 65 percent reduction.

Figure 10 shows the total NO\textsubscript{2} reduction results for NO\textsubscript{2} ranging from 110 to 500 ppm. Higher NO\textsubscript{2} values result in lower X\textsubscript{TOT} at a given value of X\textsubscript{SNCR}. At X\textsubscript{SNCR} = 0, the maximum NO\textsubscript{2} reduction performance of the SCR system (3 ppm NH\textsubscript{3}SLIP) is observed. This observed NO\textsubscript{2} reduction can be improved by adding the SNCR process, especially at higher NO\textsubscript{2} values where the slopes of the curves are steeper.

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About the Authors
B. K. Gullett is a research engineer in the Gas Cleaning Technology Branch at the U.S. Environmental Protection Agency/AEERL. P. W. Groff is a project scientist for Acurex Environmental Corporation working on the EPA’s in-house contractor support. M. L. Lin is the manager of research at Nalco Fuel Tech. J. M. Chen is a research associate in environmental catalyst research and development at England Corporation. The work presented in this paper was performed in the laboratories at EPA/AEERL.