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## N<sub>2</sub>O FORMATION IN COMBUSTION SYSTEMS

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Program Manager: Douglas Gyorke

Quarterly Technical Progress Report

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## SUMMARY

The objective of this project is to characterize  $N_2O$  emissions from combustion sources emphasizing  $N_2O$  emissions from post-combustion selective gas phase  $NO_x$  reduction processes and reburning. The processes to be evaluated include selective noncatalytic  $NO_x$  control (ammonia, urea and cyanuric acid injection), and reburning. The project includes pilot-scale testing at two facilities supported by chemical kinetic modeling. Testing is being performed on both a gas-fired plug flow combustor and a pulverized-coal fired combustor.

Work performed to date has included the performance of the initial detailed chemical kinetics calculations. These calculations showed that both urea and cyanuric acid produce significant quantities of  $N_2O$ , while  $NH_3$  injection produced negligible amounts. These kinetics data support limited test results reported for cyanuric acid and ammonia injection.

Laboratory work to evaluate the selective gas phase  $NO_x$  reduction processes listed above has begun. Testing to evaluate reburning at the coal-fired facility is being performed in parallel with the testing at the gas-fired facility. Following completion of the test work, additional kinetics calculations will be performed.

## INTRODUCTION

$N_2O$  is a relatively strong absorber of infrared radiation in the troposphere, and can contribute to the Greenhouse Effect. Once transported to the stratosphere,  $N_2O$  is the largest source of stratospheric  $NO$  and the major natural chemical sink establishing the stratospheric  $O_3$  concentration (1). The mean global  $N_2O$  concentration of approximately 330 ppb has been increasing at a rate of 0.2-0.4 percent per year (2,3).

Increases in atmospheric  $N_2O$  concentrations have been attributed to anthropogenic sources, although the dominant man-made source of  $N_2O$  is still uncertain. Previous  $N_2O$  measurements from combustion sources indicated that substantial levels of  $N_2O$  were found in systems fired with residual oil or coals (e.g., fuels containing nitrogen and sulfur). The measurements of Hao et al. (4) and Castaldini et al. (5) suggest that  $N_2O$  levels were 25-40 percent of the  $NO_x$  concentration. Global

N<sub>2</sub>O production from combustion sources was then calculated using the relationship between N<sub>2</sub>O and NO<sub>x</sub>, coupled with known combustion generated NO<sub>x</sub> emissions.

The measurements to establish emission factors for combustion generated N<sub>2</sub>O have been made primarily by gas chromatographic analysis of gas samples collected in glass or stainless steel containers. Recent measurements have shown that these grab samples can undergo chemical reaction in their containers, creating N<sub>2</sub>O concentrations substantially higher than those originating in the combustion process (6,7). Thus, a sampling "artifact" exists, resulting in the measured N<sub>2</sub>O emissions that can exceed actual emissions.

The artifact was discussed by Muzlo, et al. (8), and shown to result from reactions between the NO<sub>x</sub>, SO<sub>2</sub> and N<sub>2</sub>O present in the sample containers. The amount of N<sub>2</sub>O formed depends on the amount of SO<sub>2</sub> and water present and the initial NO level. A continuous infrared analyzer, suitable for characterizing N<sub>2</sub>O emissions from combustion sources has been used to perform continuous on line N<sub>2</sub>O measurements at nine utility boilers. The data indicate that N<sub>2</sub>O levels are generally less than 5 ppm, and were not related to the NO<sub>x</sub> levels in the flue gas. This work indicates that fossil fuel combustion is not a significant source of N<sub>2</sub>O emissions.

Concurrently, Caton and Siebers (9,10) performed laboratory tests characterizing NO<sub>x</sub> removals achievable using cyanuric acid. This work showed that up to 90 percent of the NO<sub>x</sub> removed was converted to N<sub>2</sub>O. Concerns were then raised regarding the performance of currently available post-combustion NO<sub>x</sub> reduction processes, including ammonia, urea or cyanuric acid injection. Each process involves the selective gas phase reduction of NO by one of the nitrogen-containing reagents listed above. The current project is investigating N<sub>2</sub>O emissions from each of these processes.

The specific objectives of this project are aimed at determining:

- The extent to which N<sub>2</sub>O is formed as a product of selective gas phase reduction processes using ammonia, urea and cyanuric acid.
- How N<sub>2</sub>O formation depends on the following process parameters:
  - temperature
  - residence time
  - initial NO<sub>x</sub> level
  - reductant type (NH<sub>3</sub>, urea, cyanuric acid)

- amount of reductant injected
- background gas composition ( $O_2$ , CO,  $SO_2$ )
- Whether coal ash impacts the process.
- In addition to the selective  $NO_x$  reduction processes, the program will investigate the effect of reburning on  $N_2O$  emissions.

The program involves pilot-scale combustion studies in two facilities coupled with chemical kinetic modeling to guide and analyze the experimental data.

The project is divided into five (5) tasks as listed below. Each task is discussed in the following paragraphs.

- Task 1: Chemical Kinetic Modeling of  $N_2O$  Processes
- Task 2: Plug Flow Combustor Tests
- Task 3: Coal Selection
- Task 4: Coal-Fired Combustor Tests
- Task 5: Data Analysis and Reporting

Task 1 includes the chemical kinetic modeling of  $N_2O$  processes using a model initially developed by EER for predictions of gas reburning phenomena. The model includes 201 reactions and rate constants. An initial literature review ensured that the model incorporates potential mechanisms covering the gas phase reduction of  $NO$  with ammonia, urea and cyanuric acid. The basic tool used to model this system is a one-dimensional kinetics code, which is capable of considering both well-stirred and plug-flow reactors.

Task 2 includes a series of plug-flow combustor tests to investigate parameters that can lead to  $N_2O$  formation during the gas phase reduction of  $NO$ . This testing will be performed using the Fossil Energy Research Corp. 250,000 Btu/hr down-fired combustion tunnel. The tunnel is fully instrumented to provide temperature and gaseous composition measurements at a number of locations downstream of the burner.

For the current series of tests, natural gas is being doped with  $NH_3$  to control initial  $NO_x$  levels. The  $NO_x$  reduction reagents are injected at the throat. These tests are investigating the impact of the following parameters on  $N_2O$  formation:

temperature; reducing agent; initial NO levels; N/NO<sub>x</sub> molar ratio; and the effect of additives on N<sub>2</sub>O emissions.

Continuous gas analysis at the combustor exit will be used to determine N<sub>2</sub>O formation at nominal residence times of 1 second, for the majority of the tests. For selected tests, axial gaseous samples will be obtained as well as wet chemical samples for NH<sub>3</sub> determinations.

Coal-fired pilot-scale tests are being performed under Task 4 to confirm the results obtained in the natural gas-fired plug flow combustor tests and to investigate N<sub>2</sub>O formation during reburning. This series of tests is being performed in UCI's pulverized coal-fired pilot-scale facility. The system incorporates a variable swirl burner capable of generating flame characteristics similar to large-scale systems.

## TECHNICAL PROGRESS TO DATE

This section describes the progress made during the third quarter of the project, on a task basis.

### Task 1 - Chemical Kinetic Modelling of N<sub>2</sub>O Processes.

No work was performed during this quarter on Task 1. The initial kinetic modeling work was completed during the second quarter of the project. The final modeling work is scheduled to be performed following completion of the initial phase of the plug flow combustor tests.

### Task 2 - Plug Flow Combustor Tests

The Task 2 testing was begun during this quarter. Work performed included finalization of a solids injection system and preparation of a detailed laboratory test matrix. This matrix, presented in Table 1, shows the specific measurements to be made at each of the test points required to characterize N<sub>2</sub>O production of selective gas phase NO<sub>x</sub> reduction processes. To date, the work required to complete test days 1 through 6 has been completed. These data should be considered preliminary until the remaining tests in this series (Test Days No. 7 through 14) are completed.

Preliminary results of this work show that the laboratory test results are in good agreement with the chemical kinetics calculations. Figures 1 through 3 present the results of these tests, where NO reduction, N<sub>2</sub>O production, and the ratio of N<sub>2</sub>O production to NO reduction are each plotted as a function of N/NO<sub>x</sub> ratio for temperatures of 1100 K, 1200 K and 1300 K, respectively.

Comparison of the NO reduction data (Figures 1a, 2a and 3a) shows that ammonia injection provided the highest measured reductions at each of the three temperatures evaluated to date. Maximum reduction for ammonia and solid urea were measured at 1200 K, while liquid urea and cyanuric acid performed best at 1300 K. The difference in performance between liquid and solid urea is most likely due to the fact that the liquid solution must become saturated before the urea begins to dissociate while the solid would dissociate almost instantly upon injection.

Evaluation of the N<sub>2</sub>O production data (Figures 1b, 2b and 3b) shows that N<sub>2</sub>O became more sensitive to N/NO<sub>x</sub> ratios as temperatures increased. N<sub>2</sub>O emissions

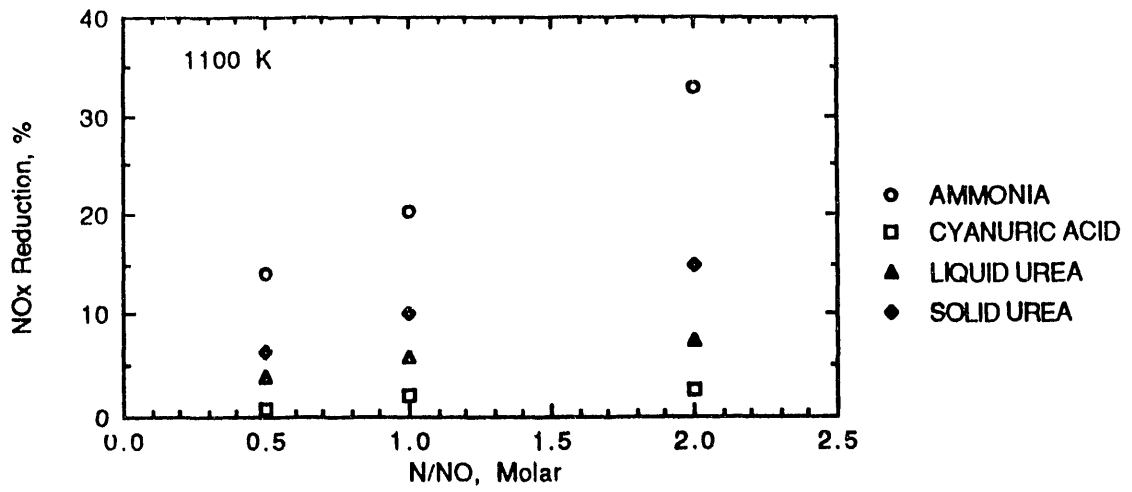
TABLE 1. TASK 2 TEST MATRIX

TEST DAY	REAGENT	NOi ppm	T S1 °C	N/NO molar	NO	NOx	N2O	O2	CO	CO2	NH3
1	NH3	700	927	(1)	x	x	x	x	x	x	-
	UREA(l)				x	x	x	x	x	x	-
2	UREA(s)				x	x	x	x	x	x	-
	CA				x	x	x	x	x	x	-
3	NH3	700	827	(1)	x	x	x	x	x	x	-
	UREA(l)				x	x	x	x	x	x	-
4	UREA(s)				x	x	x	x	x	x	-
	CA				x	x	x	x	x	x	-
5	NH3	700	1027	(1)	x	x	x	x	x	x	-
	UREA(l)				x	x	x	x	x	x	-
6	UREA(s)				x	x	x	x	x	x	-
	CA				x	x	x	x	x	x	-
7	NH3	700	977	(1)	x	x	x	x	x	x	(2)
	UREA(l)				x	x	x	x	x	x	(2)
8	UREA(s)				x	x	x	x	x	x	(2)
	CA				x	x	x	x	x	x	(2)
9	NH3	700	877	(1)	x	x	x	x	x	x	(2)
	UREA(l)				x	x	x	x	x	x	(2)
10	UREA(s)				x	x	x	x	x	x	(2)
	CA				x	x	x	x	x	x	(2)
11	NH3	700	1097	(1)	x	x	x	x	x	x	(2)
	UREA(l)				x	x	x	x	x	x	(2)
12	UREA(s)				x	x	x	x	x	x	(2)
	CA				x	x	x	x	x	x	(2)
13	NH3	300	927	(1)	x	x	x	x	x	x	-
	UREA(3)				x	x	x	x	x	x	-
	CA				x	x	x	x	x	x	-
14	NH3	1000	927	(1)	x	x	x	x	x	x	-
	UREA(3)				x	x	x	x	x	x	-
	CA				x	x	x	x	x	x	-
15	NH3	300	977	(1)	x	x	x	x	x	x	(2)
	UREA(3)				x	x	x	x	x	x	(2)
	CA				x	x	x	x	x	x	(2)

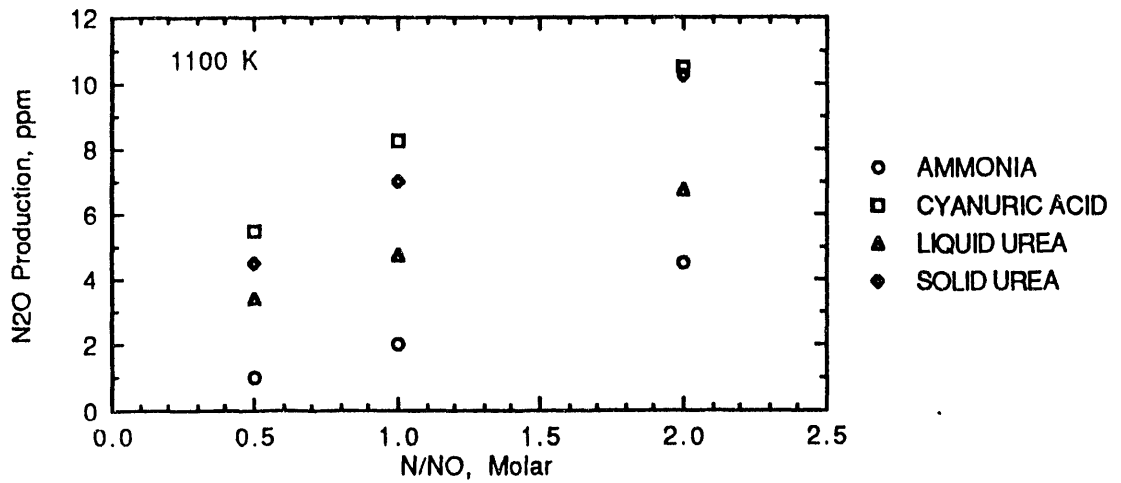
TABLE 1. TASK 2 TEST MATRIX

TEST DAY	REAGENT	NOi ppm	T S1 °C	N/NO molar	NO	NOx	N2O	O2	CO	CO2	NH3
16	NH3	1000	977	(1)	x	x	x	x	x	x	(2)
	UREA(3)				x	x	x	x	x	x	(2)
	CA				x	x	x	x	x	x	(2)
17	NH3	300	877	(1)	x	x	x	x	x	x	(2)
	UREA(3)				x	x	x	x	x	x	(2)
	CA				x	x	x	x	x	x	(2)
18	NH3	1000	877	(1)	x	x	x	x	x	x	(2)
	UREA(3)				x	x	x	x	x	x	(2)
	CA				x	x	x	x	x	x	(2)
19	NH3	300	1097	(1)	x	x	x	x	x	x	(2)
	UREA(3)				x	x	x	x	x	x	(2)
	CA				x	x	x	x	x	x	(2)
20	NH3	1000	1097	(1)	x	x	x	x	x	x	(2)
	UREA(3)				x	x	x	x	x	x	(2)
	CA				x	x	x	x	x	x	(2)
	NOTES										
	(1) N/NO= 0,0.5,1.0,2.0										
	(2) NH3 TESTS @ N/NO= 0.5,1.0,2.0										
	(3) EITHER LIQUID OR SLID UREA BASED ON PREVIOUS TEST RESULTS										

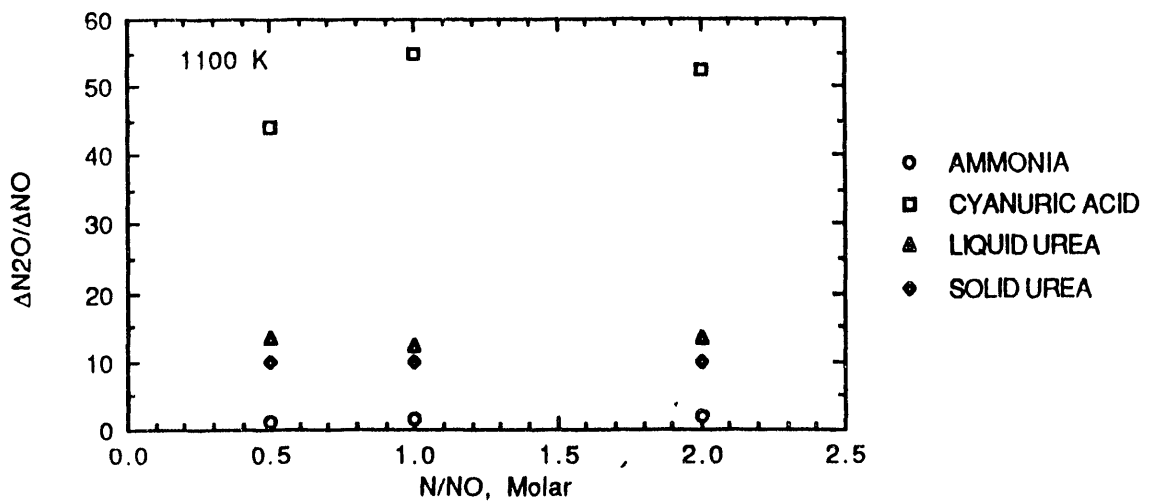




1a. NOx reduction vs. [N/NO] ratio

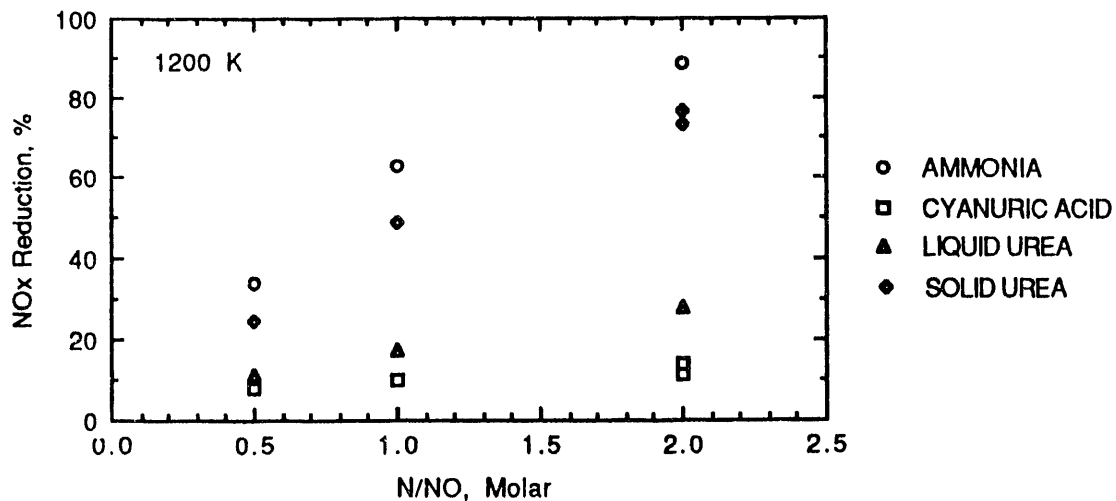


1b. N2O production vs. [N/NO] ratio

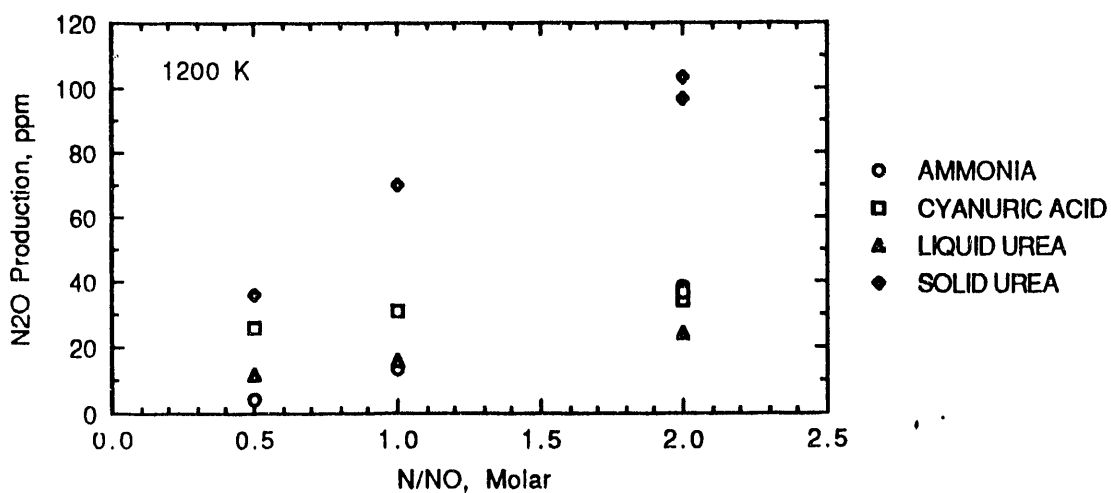


1c. N2O production/NOx reduction ratio vs. [N/NO] ratio

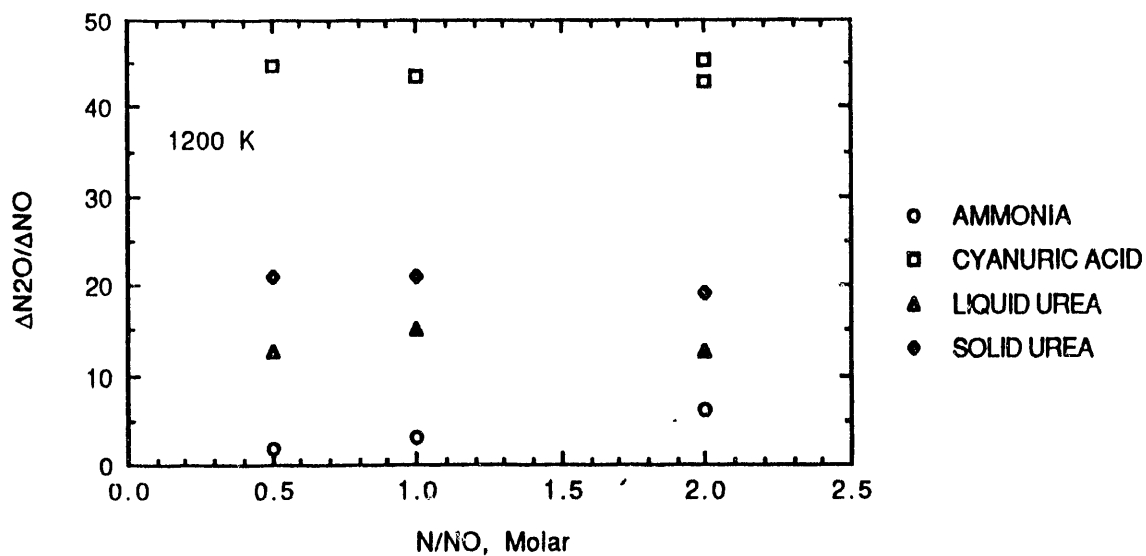
Figure 1. Effect of N/NO molar ratio on NOx reduction and N2O production, laboratory test data at 1100 K with 700 ppm initial NO.



2a. NOx reduction vs. [N/NO] ratio

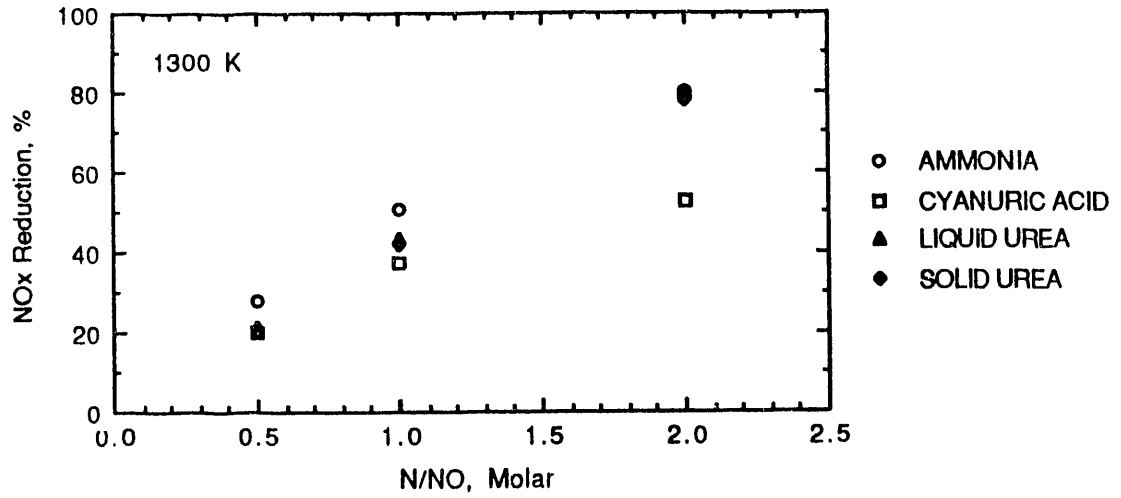


2b. N2O production vs. [N/NO] ratio

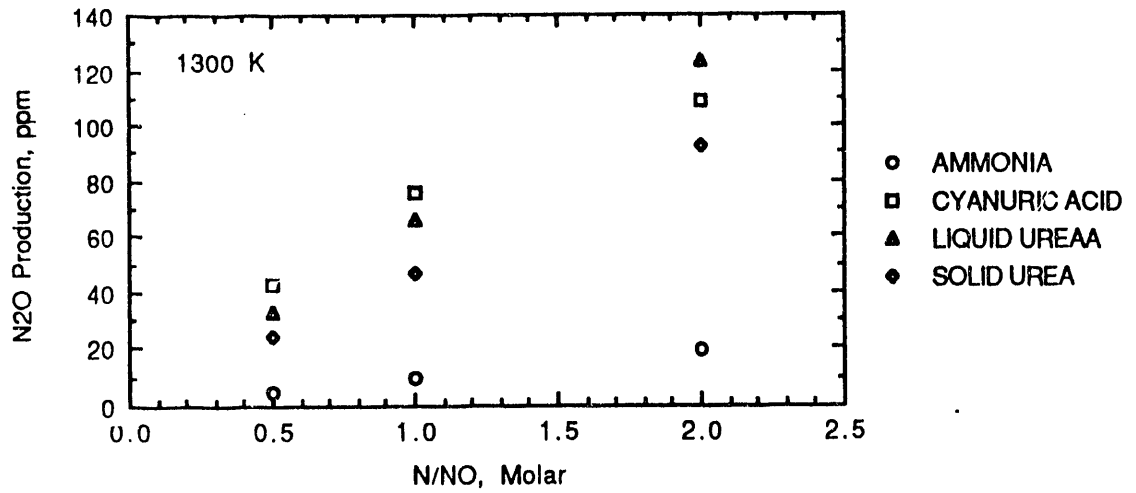


2c. N2O production/NOx reduction ratio vs. [N/NO] ratio

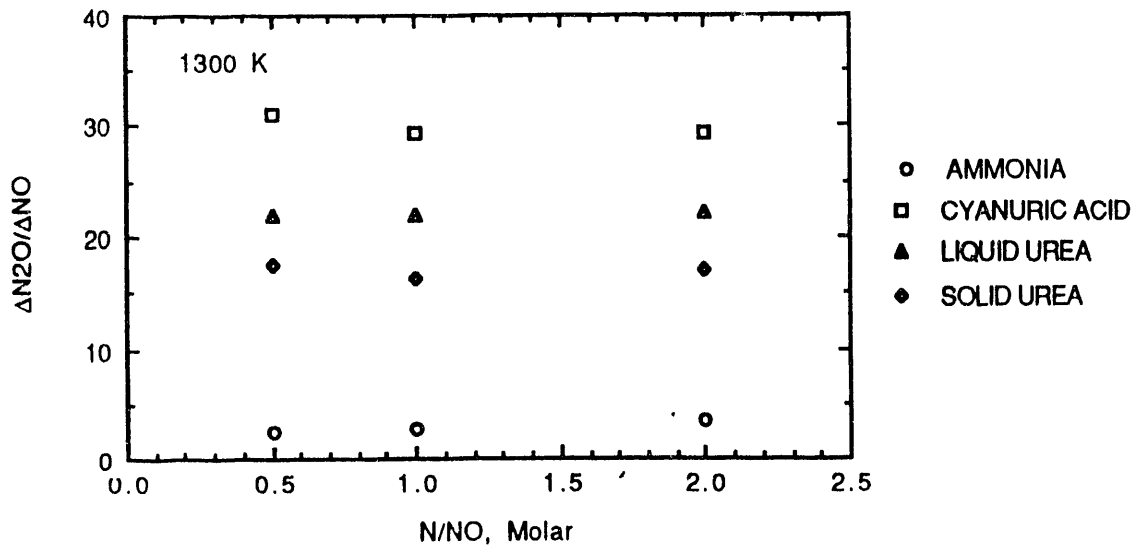
Figure 2. Effect of N/NO molar ratio on NOx reduction and N2O production, laboratory test data at 1200 K with 700 ppm initial NO.



3a. NOx reduction vs. [N/NO] ratio



3b. N2O production vs. [N/NO] ratio



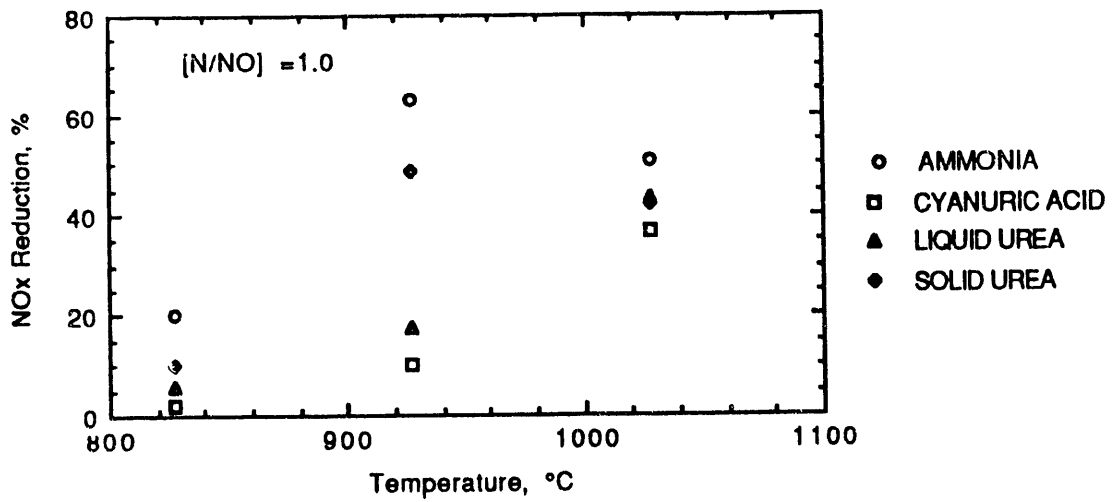
3c. N2O production/NOx reduction ratio vs. [N/NO] ratio

Figure 3. Effect of N/NO molar ratio on NOx reduction and N2O production, laboratory test data at 1300 K with 700 ppm Initial NO.

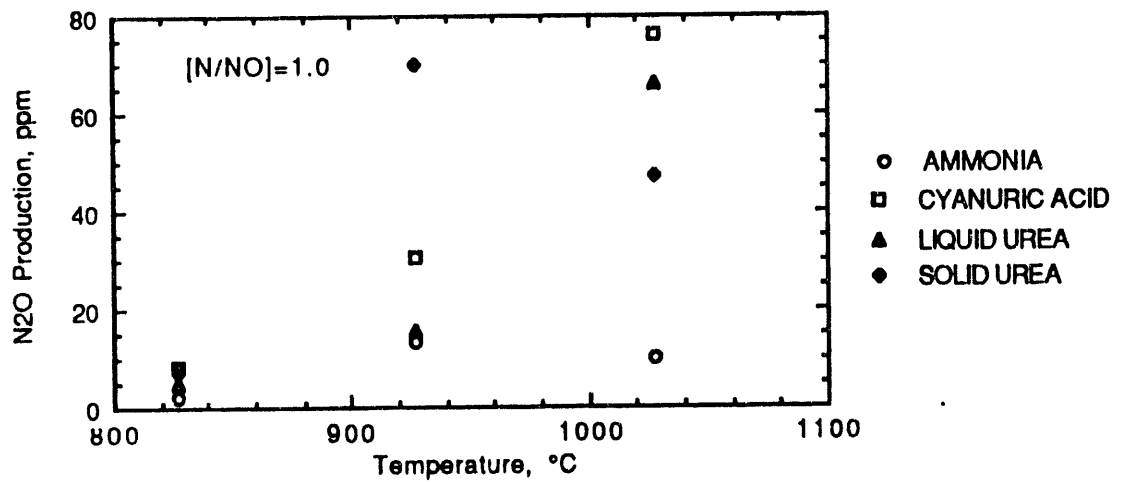
appeared to peak at the same conditions as the NO reductions. Maximum N<sub>2</sub>O production, for ammonia and solid urea, was measured at 1200 K, while maximum N<sub>2</sub>O production levels, at N/NO ratios of 1.0, varied from 13 ppm with ammonia to 76 ppm with cyanuric acid.

To more clearly see the effect of selective non-catalytic NO<sub>x</sub> reduction processes on N<sub>2</sub>O formation, the ratio of N<sub>2</sub>O production to NO reduction was plotted versus N/NO<sub>x</sub> ratio. These data, presented in Figures 1c, 2c and 3c, show that cyanuric acid produced the highest relative amount of N<sub>2</sub>O of the reagents evaluated. Ratios of N<sub>2</sub>O production to NO reduction were on the order of 30 to 60 percent, depending on temperature. Maximum relative N<sub>2</sub>O production, with cyanuric acid injection, was measured at 1100 K. This compares favorably to the chemical kinetics calculations, which predicted peak N<sub>2</sub>O production at temperatures of 1200 K. Ammonia injection resulted in low levels of N<sub>2</sub>O production. The levels were, however, higher than those predicted by the kinetics calculations, which suggested that essentially no N<sub>2</sub>O would be formed. Maximum levels of N<sub>2</sub>O emissions were measured at 1200 K. Urea injection resulted in relative N<sub>2</sub>O emissions of 10 to 25 percent of the measured NO reductions. The difference between liquid and solid urea injection is not yet clear; in two cases, liquid injection resulted in higher relative N<sub>2</sub>O emissions. The maximum difference was seen at 1200 K, where the difference between liquid and solid injection was nominally 7 percentage points. At the remaining injection temperatures, relative differences between solid and liquid injection were 3 to 5 percentage points. This difference may be related to differences in the time temperature histories experienced by the aqueous urea and solid urea in the combustor. The combustor has a quench rate of nominally 250°C/sec. In this environment, the aqueous solution will have to evaporate to saturation before it becomes available to react with NO<sub>x</sub>, whereas the solid material should decompose in a shorter period of time following injection.

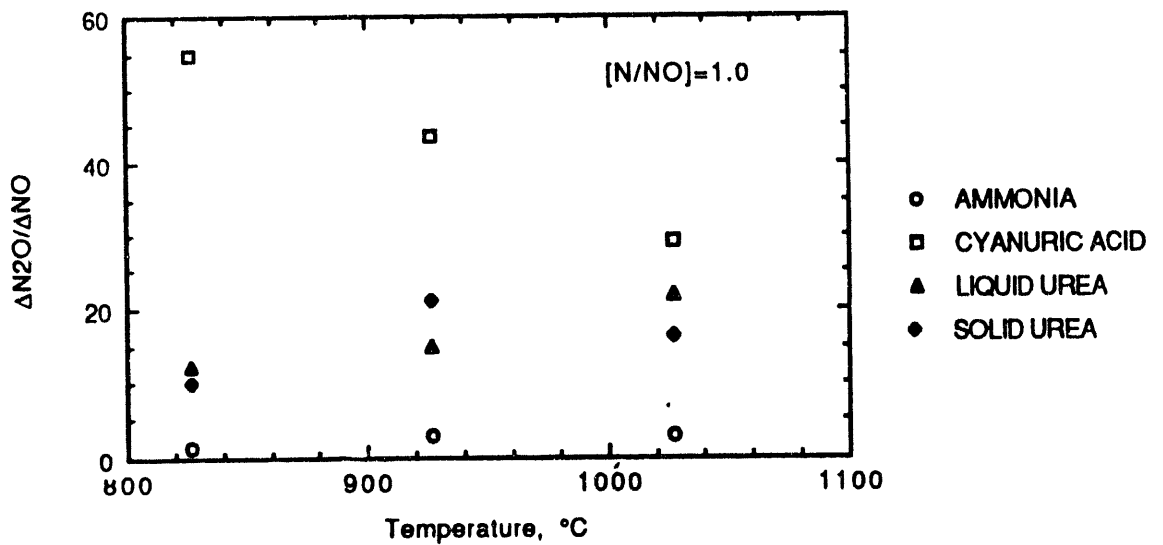
NO reduction and N<sub>2</sub>O production data are plotted as a function of temperature in Figure 4 along with the ratio of N<sub>2</sub>O production to NO reduction for data obtained at a N/NO<sub>x</sub> ratio of 1.0. The NO reduction data presented in Figure 4a show that peak removals with ammonia and solid urea were measured at nominally 1200°K. The data obtained to date have not shown a peak in the NO removal versus temperature curves for other liquid urea or cyanuric acid. It is anticipated that tests scheduled to be performed at high temperatures will better define where those peaks



4a. NOx reduction vs. injection temperature



4b. N2O production vs. [N/NO] ratio



4c. N2O production/NOx reduction ratio vs. [N/NO] ratio

Figure 4. Effect of temperature on NOx reduction and N2O production, laboratory test data at [N/NO]=1.0 with 700 ppm initial NO.

occur. The  $N_2O$  production data presented in Figure 4b show that  $N_2O$  production resulting from both ammonia and solid urea injection peaked at 1200°K.  $N_2O$  production from both cyanuric acid and solid urea injection exhibited no peaks over the range of temperatures evaluated to date. The cyanuric acid data are in agreement with those reported in Reference 10, which showed that  $N_2O$  production peaked at a temperature of about 1370°K. Figure 4c, which illustrates the ratio of  $N_2O$  production versus NO reduction as a function of temperature, shows that  $N_2O$  production increased with temperature for both ammonia and liquid urea injection. When injecting solid urea,  $N_2O$  emissions appeared to peak at a temperature of 1200 K. When injecting cyanuric acid or liquid urea,  $N_2O$  emissions increased as injection temperature decreased. These data reflect the trends seen above, showing that both NO reduction and  $N_2O$  production peak at similar temperatures.

### Task 3 - Coal Selection

Coal for the Task 4 coal-fired combustor tests was selected during this quarter. A drum of bituminous coal was shipped to the coal-fired facility for use in the Task 4 tests.

### Task 4 - Coal-Fired Combustor Tests

The coal-fired combustor tests, evaluating the  $N_2O$  production potential of reburning with coal, were nearly completed during this quarter. The data should, however, be considered preliminary until the validation of the  $N_2O$  sampling system is completed. The matrix for these tests is presented in Table 2. This table shows that seven test series were performed. Principal variables were firing rate, initial NO levels, and reburn zone stoichiometry.

TABLE 2. TEST MATRIX, COAL-FIRED COMBUSTOR

Test No.	Firing Rate Btu/hr	NO <sub>i</sub> ppm	SR <sub>r</sub>	NO <sub>x</sub>	O <sub>2</sub>	N <sub>2</sub> O
1	77,000	735	(1)	x	x	x
2	89,800	715	(2)	x	x	x
3	89,800	890	(2)	x	x	x
4	89,800	530	(2)	x	x	x
5	102,700	530	(3)	x	x	x
6	76,400	405	(1)	x	x	x
7	77,000	900	(1)	x	x	x

- (1) SR<sub>r</sub> = 1.2, 1.0, 0.9, 0.8, 0.7, 0.6  
 (2) SR<sub>r</sub> = 1.3, 1.1, 1.0, 0.9, 0.8, 0.7  
 (3) SR<sub>r</sub> = 1.4, 1.2, 1.1, 1.0, 0.8, 0.7

Figures 5 through 7 illustrate the results of the tests performed at a nominal firing rate of 90,000 Btu/hr. In these figures, both the NO<sub>x</sub> reduction, expressed as a percentage, and the change in N<sub>2</sub>O emissions in ppm are plotted as a function of reburn zone stoichiometry for initial NO<sub>x</sub> levels of 550, 730 and 900 ppm, respectively. The data show that NO<sub>x</sub> reductions increased as the reburn zone stoichiometry decreased, as expected. Maximum reductions varied from nominally 30 percent to 45 percent, and were shown to increase with initial NO<sub>x</sub> levels. Measured changes in N<sub>2</sub>O emissions were generally less than 10 ppm.

Figure 8 shows the relationship between the change in N<sub>2</sub>O emissions, relative to the change in NO<sub>x</sub> emissions, and reburn zone stoichiometry for tests performed at a firing rate of 90,000 Btu/hr. The data show that N<sub>2</sub>O emissions were a constant fraction of NO<sub>x</sub> reductions for tests performed at initial NO<sub>x</sub> levels of 715 ppm and 890 ppm. The ratio of N<sub>2</sub>O production to NO<sub>x</sub> emission reduction was nominally 2 percent (0.02) at these test conditions. Tests performed at initial NO<sub>x</sub> levels of 530 ppm showed that the N<sub>2</sub>O/NO<sub>x</sub> ratio decreased as reburn zone stoichiometry. These data show that the ratio of N<sub>2</sub>O production to NO<sub>x</sub> emission reduction decreased from about 6 percent to 3 percent as reburn zone stoichiometry was decreased from 1.26 to 0.69. Thus, the preliminary analysis of the data indicates that N<sub>2</sub>O is not a major product when reburning with coal.

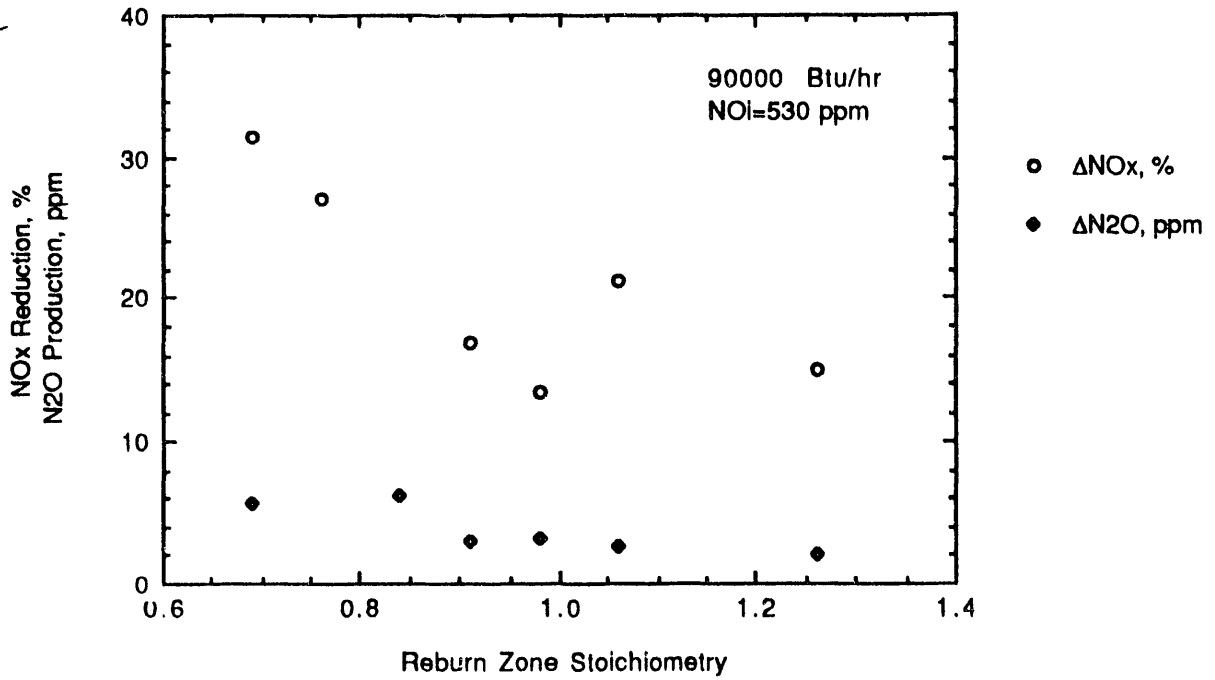


Figure 5. Effect of reburn zone stoichiometry on NO<sub>x</sub> reduction and N<sub>2</sub>O emissions.

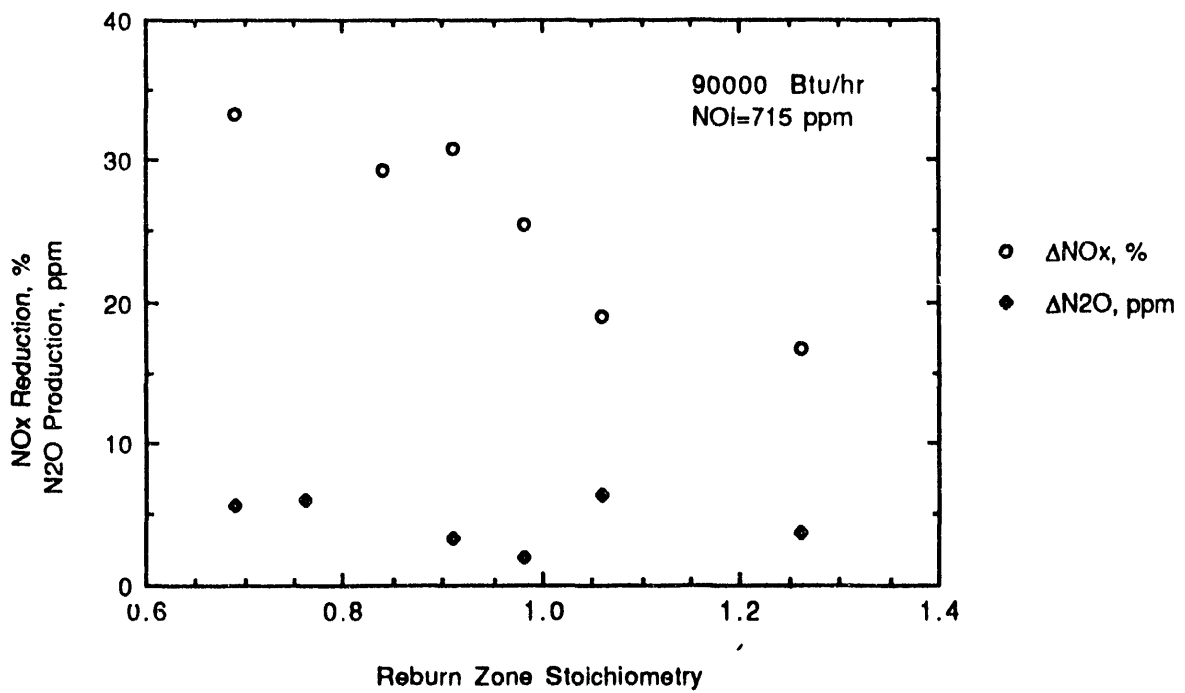


Figure 6. Effect of reburn zone stoichiometry on NO<sub>x</sub> reductions and N<sub>2</sub>O emissions.



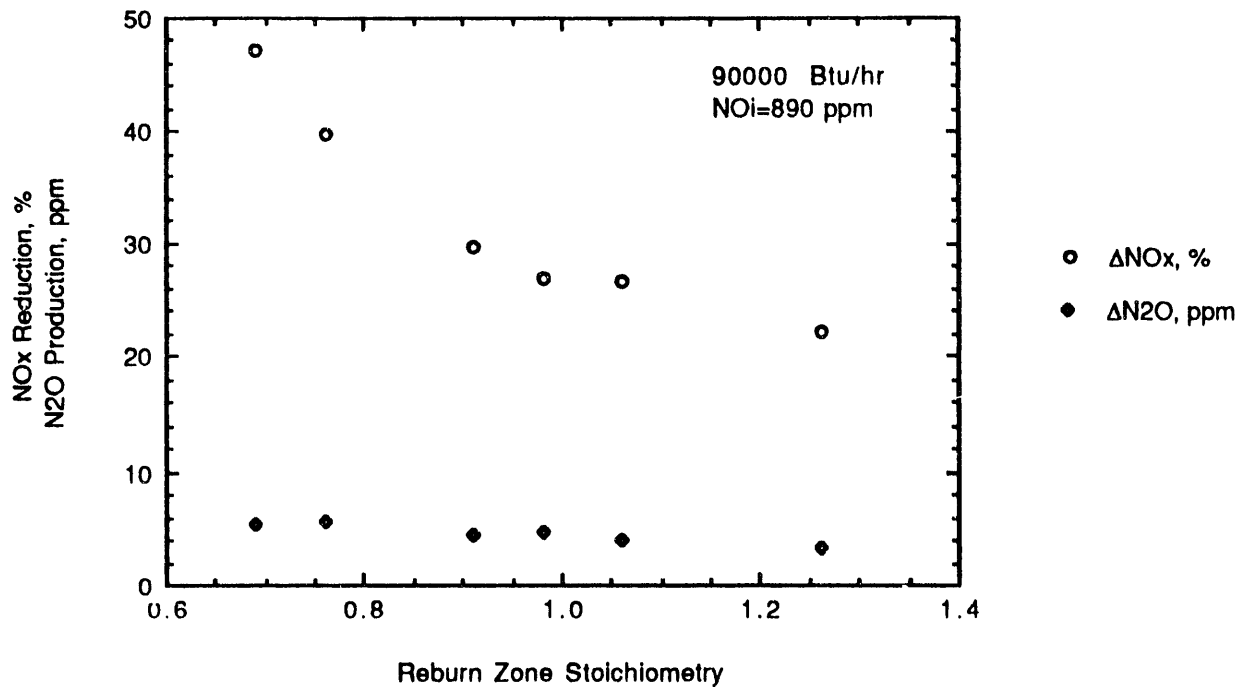


Figure 7. Effect of reburn zone stoichiometry on NO<sub>x</sub> reduction and N<sub>2</sub>O emissions.

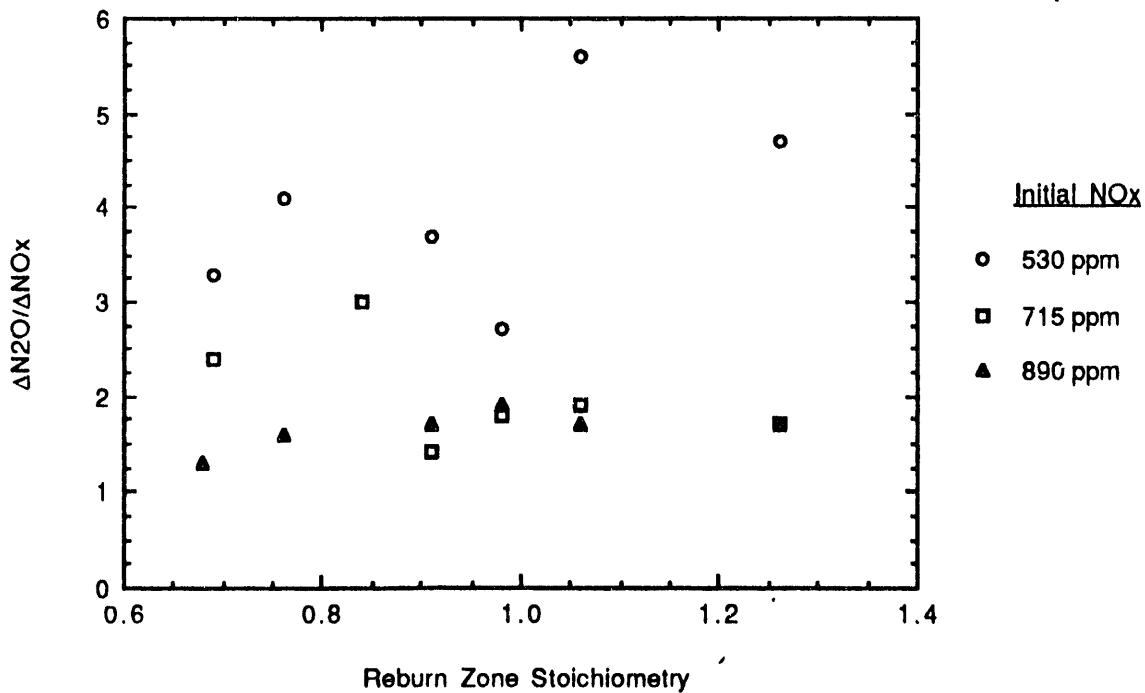


Figure 8. Effect of reburn zone stoichiometry on ratio of N<sub>2</sub>O production to NO<sub>x</sub> reduction.

### Task 5 - Data Analysis and Reporting

Task 5 work performed during this quarter has included the preparation of required monthly progress reports, and preparation of the quarterly progress report.

## OUTLINE OF WORK PLANNED FOR NEXT REPORTING PERIOD

### Task 1 - Chemical Kinetic Modelling of N<sub>2</sub>O Processes

It is anticipated that Task 1 activities will include performance of the final chemical kinetic modeling. This work will be initiated following completion of the first phase of the Task 2 testing.

### Task 2 - Plug Flow Combustor Tests

Task 2 work planned for this quarter includes completion of the majority of the testing. The evaluation of selective gas phase NO<sub>x</sub> reduction processes should be completed during this quarter. Evaluation of potential N<sub>2</sub>O reduction processes should be underway during this quarter. This work will likely be completed during the final quarter of the project.

### Task 3 - Coal Selection

Task 3 work has been completed. The coal required for the Task 4 testing has been delivered to the coal-fired combustor facility.

### Task 4 - Coal-Fired Combustor Tests

Task 4 work will include validation of the N<sub>2</sub>O sampling system. Following this, the results of this task will be documented.

### Task 5 - Data Analysis and Reporting

Preparation of monthly and quarterly reports will continue during the upcoming quarter. It is anticipated that preparation of the final report will commence in conjunction with the testing activities of Tasks 2 and 4. Results from the planned Task 2 testing should allow for finalization of the Task 1 work.

## REFERENCES

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