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U.S. Army Toxic and Hazardous Materials Agency

Laboratory Testing of a Fluidized-Bed Dry-Scrubbing Process for the Removal of Acidic Gases from a Simulated Incinerator Flue Gas

Final Report to the U.S. Army Toxic and Hazardous Materials Agency

Prepared by

W. M. Bradshaw
R. P. Krishnan
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April 1989

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Engineering Technology Division

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Research sponsored by the U.S. Army Toxic and Hazardous Materials Agency through the U. S. Department of Energy under contract No. DE-AC05-84OR21400 with the Martin Marietta Energy Systems, Inc.

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ABSTRACT

A series of bench-scale tests was conducted to evaluate a dry, fluidized-bed, scrubbing process for removing acidic gases from incinerator flue gas. The acidic gases studied were sulfur dioxide, hydrogen chloride, and phosphorus pentoxide. These gases were found to react readily with lime in a bubbling bed operating at 540°C (1000°F). Superficial gas velocity, bed temperature, bed depth, sorbent type, and sorbent utilization strongly affected the degree of acidic gas removal. Sorbent utilization was inhibited by reaction product occlusion of the particle surface.

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1. INTRODUCTION

The Oak Ridge National Laboratory (ORNL) has been investigating dry fluidized-bed scrubbing of acidic compounds from incinerator flue gas under the sponsorship of the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA). The U.S. Army is pursuing incineration as a means of disposing of contaminated sludges, soils, and liquids. These wastes frequently contain chemical compounds that produce acids when incinerated; in most cases, the acidic compounds must be removed from the flue gas to meet environmental standards. These acidic gases include sulfur dioxide (SO_2), phosphorus pentoxide (P_2O_5), hydrogen chloride (HCl), and nitrogen oxides (NO_x).

This report documents experimental work done in FY 1988 at ORNL on a bench-scale, dry-scrubbing process to remove acidic gases from a simulated incinerator flue gas, using a fluidized bed of lime {primarily calcium hydroxide [$Ca(OH)_2$]} operating in the bubbling-bed regime at 427 to 540°C (800 to 1000°F). The experimental program was a 2-year, two-phase effort. Phase I (FY 1987) focused on determining the feasibility of dry scrubbing P_2O_5 and SO_2 in a fluidized bed.[1] Phase II of the program, covered in this report, focused on (1) dry scrubbing HCl at the same process conditions as P_2O_5 and SO_2, (2) scrubber performance with a mixture of acidic gases representative of a typical incinerator flue gas, and (3) sorbent utilization. The results of the experimental work were used to determine the economics of fluidized-bed scrubbing relative to other flue gas treatment technologies.

2. EXPERIMENTAL PROGRAM

Dry scrubbing has several advantages that make it an attractive alternative to wet scrubbing.[1] The experimental program described in this report is part of an effort by USATHAMA to develop and test a flue gas dry-scrubbing system that can be used as a replacement for wet scrubbers for Army incinerators.

The first phase of the experimental program investigated the basic feasibility of dry scrubbing, using two acid gases (P_2O_5 and SO_2) frequently found in Army waste incinerator flue gas.[1] The second phase, summarized in this report, produced additional data needed to design a pilot-scale dry-scrubbing system. The data were also used to determine the economics of such a system for a large incinerator.

2.1 Scope

The FY 1988 (Phase II) portion of the experimental program focused on (1) dry scrubbing HCl from a nitrogen (N_2) carrier gas stream in a bed of lime, (2) dry scrubbing a mixture of P_2O_5, HCl, and SO_2 from a simulated incinerator flue gas, and (3) determining the ultimate utilization of the sorbent. Lime was chosen as the baseline sorbent for reasons cited in the report covering the Phase I activities.[1] The lime used for the majority of the tests was 87% $Ca(OH)_2$ and 13% calcium carbonate ($CaCO_3$).

2.2 Test Conditions

Phase II consisted of 11 tests. The first three verified that HCl could be removed from a carrier gas stream at conditions favorable for the removal of P_2O_5 and SO_2. Next, the validity of using lime as the sorbent of choice was demonstrated with one short-term test using a mixture of all three acidic gases along with carbon dioxide (CO_2), oxygen (O_2), and N_2, and a bed of limestone (calcium carbonate) as the sorbent.

Two additional tests provided baseline performance data using lime as the sorbent by scrubbing HCl and SO_2 from a gas stream of CO_2, O_2, and N_2. Four subsequent tests using lime with a mixture of all the gases (acidic and nonacidic) were carried out at different temperatures, flow rates, and static bed depths. Finally, one long-term test (>100 h) was run with all gases to determine the removal efficiency as a function of sorbent conversion.

The test conditions for the short-term experiments were based on the findings of the work done in Phase I. The conditions for the long-term test were based on the results of the Phase II short-term tests. The parameters studied included bed temperature, gas velocity, and bed depth. The range of interest is shown in Table 2.1. Test conditions for the individual runs are in Appendix D.

Table 2.1. Test conditions

Parameter	Range tested
Bed temperature	
HCl tests	540°C (1000°F)
Acidic gas mixtures	427-540°F (800-1000°F)
Inlet concentration	
HCl tests	<138-2510 ppm
Acidic gas mixtures	
P_2O_5	<1.0-170 ppm
HCl	200-6900 ppm
SO_2	<18-11900 ppm
Gas velocity	
HCl tests	6.8 cm/s
Acidic gas mixtures	2.6-5.7 cm/s
Static bed depth	
HCl tests	20 cm (8 in.)
Acidic gas mixtures	2.5-10 cm (1-4 in.)
Sorbent particle size (all tests)	212-425 μm

3. EXPERIMENTAL EQUIPMENT AND PROCEDURES

All of the bench-scale tests were conducted in the scrubber system shown in Fig. 3.1.

3.1 System Description

A schematic diagram of the dry scrubber is shown in Fig. 3.2. The system was the same as that used in the Phase I experimental work with a few modifications.

3.1.1 Fluidized-Bed Scrubber

The fluidized bed was contained in a 2-in., Schedule 40, 316L stainless steel pipe 46 cm (18 in.) long. A 4-in., Schedule 40, 304L stainless steel pipe 76 cm (30 in.) long was provided immediately above the 2-in. section to reduce solids entrainment.

Three changes were made to the scrubber section. Pressure taps were installed to allow direct measurement of the pressure drop across both the bed and the distributor plate. A seamless, porous metal tube (20 μm) was incorporated into the scrubber outlet line to act as a filter for any particles carried over from the bed. Finally, the inner wall of the scrubber was plated with nickel to inhibit corrosion that could bias the component material balances.

3.1.2 Gas Streams

Solid P_2O_5 was sublimed into a nitrogen stream, and the gaseous mixture entered the scrubber via a porous metal (316L stainless steel) tuyere plate designed to enhance gas distribution and support the lime. Both SO_2 and HCl were introduced through a port immediately below the tuyere plate. Another line carried a mixture of N_2, CO_2, and O_2 to the same port.

The HCl, O_2, and CO_2 gas streams were new to the system. All gases (with the exception of P_2O_5) were controlled with in-line rotameters. The amount of P_2O_5 fed into the scrubber was a function of the sublimation chamber temperature and the N_2 flow rate through the chamber.

3.2 Operating Procedures

The operating procedures for this phase of the dry-scrubbing experimental work were similar to those of Phase I.[1] A detailed description of the procedures and equipment is given in Appendix E. With the exception of run 11, which was a long-term test (112 h), the duration of all the tests was between 1 and 16 h.

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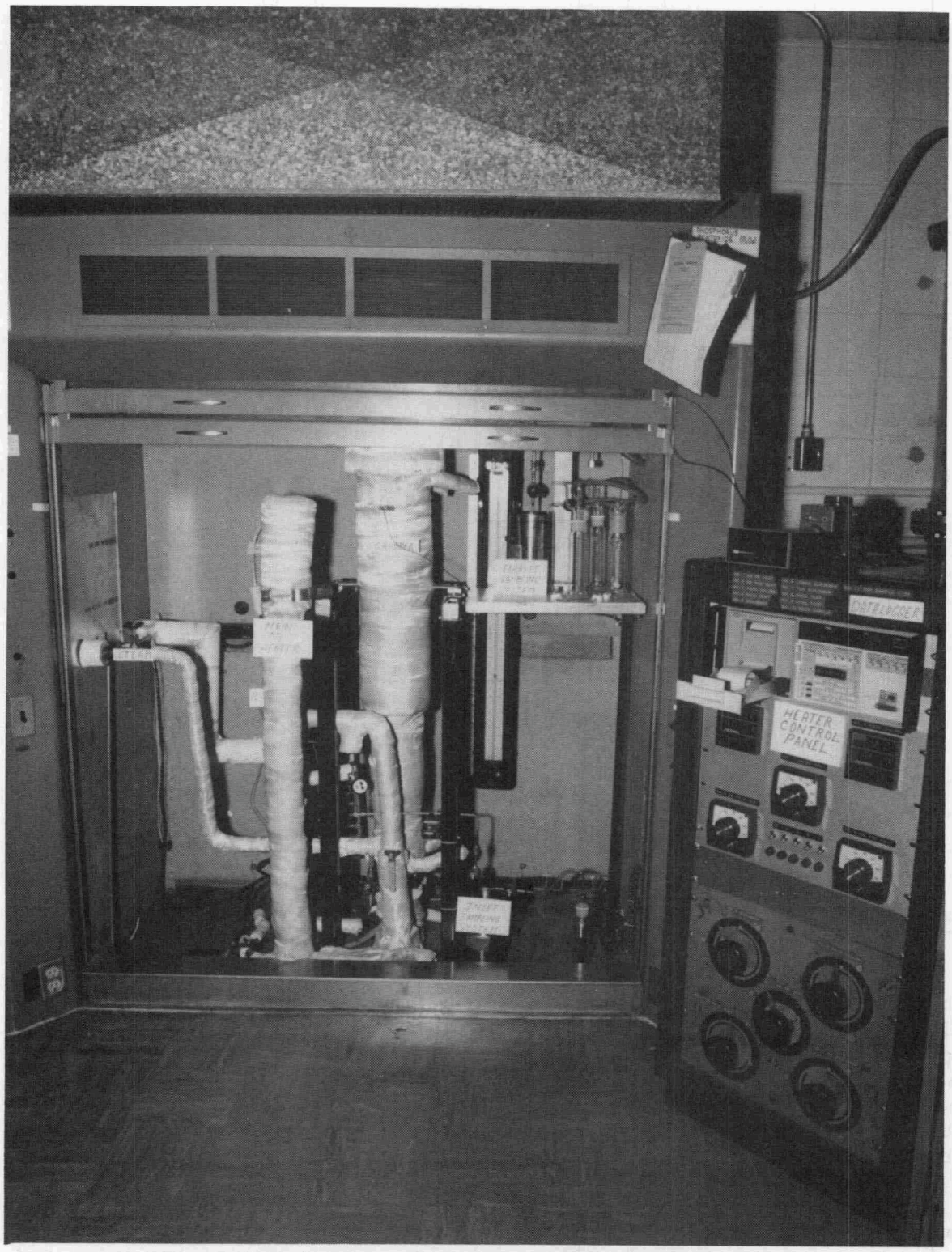

Fig. 3.1. Bench-scale, dry-scrubbing system.

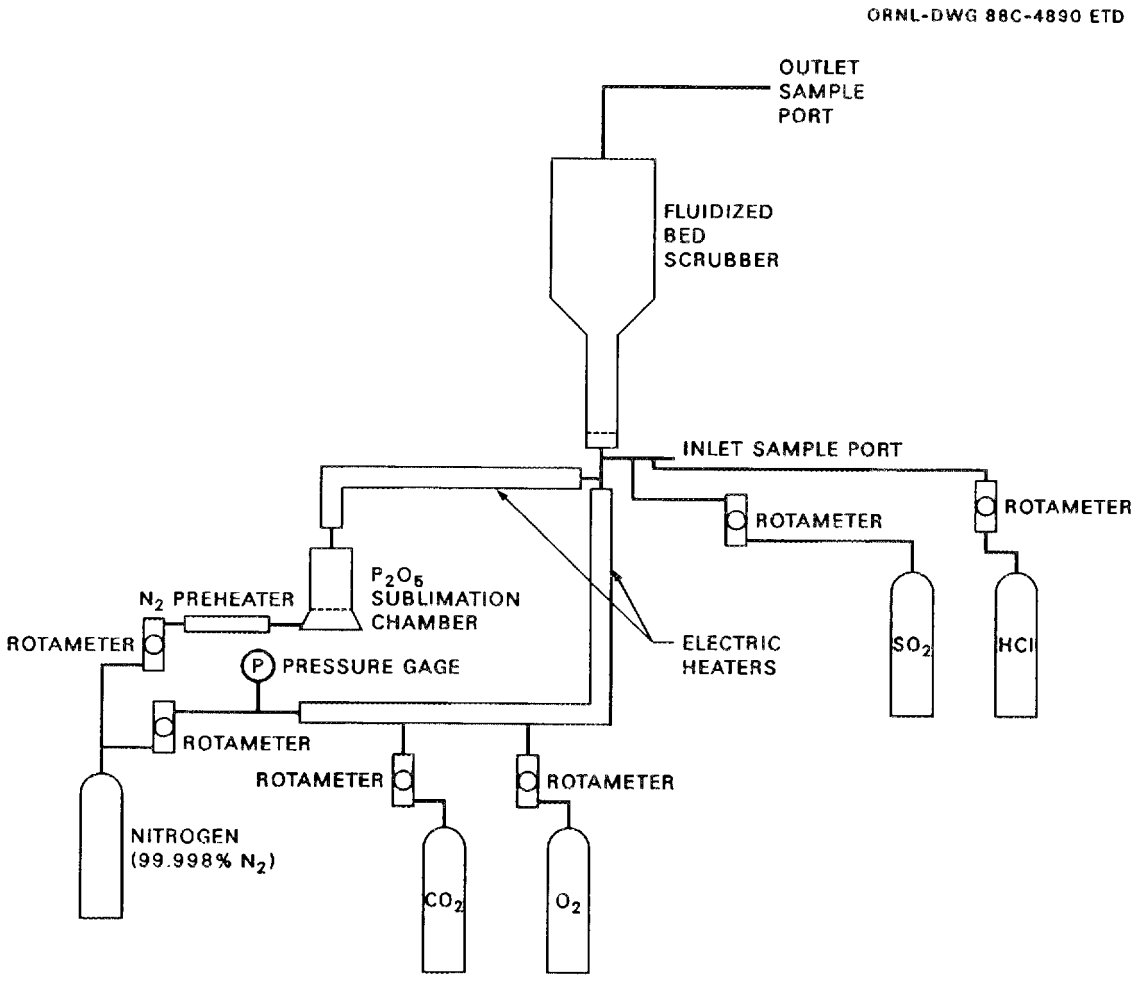

Fig. 3.2. Schematic diagram of experimental dry scrubber.

3.2.1 Sampling and data collection

The inlet and effluent gas streams were bubbled into gas-washing bottles containing either distilled water or sodium hydroxide (NaOH). The sample train was tailored to the specific gas being sampled. Phosphorus pentoxide and HCl are both soluble in water, so a single gas-washing bottle filled with distilled water was used to trap these gases when they were tested individually. Scrubbing all three acid gases in the same test necessitated using a 1-*N* NaOH solution in the gas-washing bottles because of the limited solubility of SO_2 in water.

The sample bottles were changed at regular intervals to provide time-weighted averages of inlet and outlet concentrations. The inlet gas was sampled before and after each run, and samples of the inlet and outlet gas streams were obtained periodically throughout each run.

3.3 Sample Analysis

The liquid samples were analyzed for total phosphorus with a colorimetric procedure (based on Beer's law) at 470 μm. Total sulfate and chloride were determined by ion chromatography (IC). Total chloride in the solids was also determined by IC. The carbonate, sulfate, and water content of the sorbent were determined by X-ray powder diffractometry.

4. DISCUSSION AND RESULTS

Phase II of the dry-scrubbing testing program focused on (1) determining if HCl could be removed from an N_2 carrier gas stream at the same process conditions as P_2O_5 and SO_2, (2) ascertaining which parameters affect the removal efficiency of a mixture of P_2O_5, HCl, and SO_2 from a simulated incinerator flue gas, and (3) establishing the ultimate sorbent utilization through a long-term test.

4.1 Short-Term Tests

A summary of the results from the short-term (1-d) tests is given in Table 4.1.

Table 4.1 Results of short-term dry scrubbing tests

Text No.	Acid	Temperature (°C)	Gas flow rate (cm/s)	u/u_{mf}[a]	Sorbent (g)	Concentration (ppm)	
						Inlet	Outlet
1	HCl	540	6.8	2.6	400 Lime	230	<36
2	HCl	540	6.8	2.6	400 Lime	1800	<100
3	HCl	540	6.8	2.6	400 Dolomite	1900	<80
4	P_2O_5	540	6.9	2.6	50 Limestone	7.3	<0.4
	HCl					2160	2140
	SO_2					1080	1060
5	HCl	540	6.9	2.6	50 Lime	680	68
	SO_2					670	190
6	HCl	540	6.9	2.6	100 Lime	680	12
	SO_2					700	<4
7	P_2O_5	540	6.9	2.6	50 Lime	<1.8	<0.4
	HCl					1400	100
	SO_2					900	380
8	P_2O_5	540	3.2	1.2	50 Lime	12	<0.3
	HCl					1300	250
	SO_2					1000	220
9	P_2O_5	427	2.8	1.1	50 Lime	<0.8	<0.2
	HCl					930	850
	SO_2					980	770
10	P_2O_5	540	3.2	1.2	200 Lime	73	<0.2
	HCl					1840	82
	SO_2					654	<3

[a]Superficial gas velocity/minimum fluidization velocity.

4.1.1 HCl removal

Three tests were conducted to determine if HCl could be removed from a N_2 stream at conditions favorable for removing P_2O_5 and SO_2. The first test showed a >84% removal; the second and third tests showed >94% and >96% removal, respectively, thus confirming that HCl can be removed at the same conditions as P_2O_5 and SO_2. In each test, HCl was not detected in the outlet stream so the actual removal was probably higher than the minimum valves given above. (For tests 1–3, the HCl detection limit was ~35 ppm.)

4.1.2 Removal of P_2O_5, SO_2, and HCl from simulated flue gas

Seven tests were run to establish the parameters that have the greatest effect on acid removal from a mixture of gases.

4.1.2.1 Superficial gas velocity. Superficial gas velocity had a pronounced effect on the HCl and P_2O_5 removal, but not on the SO_2 removal. HCl and SO_2 removal data from tests 5, 7, and 8 are shown in Fig. 4.1 as a function of gas velocity. Similar data for P_2O_5 removal

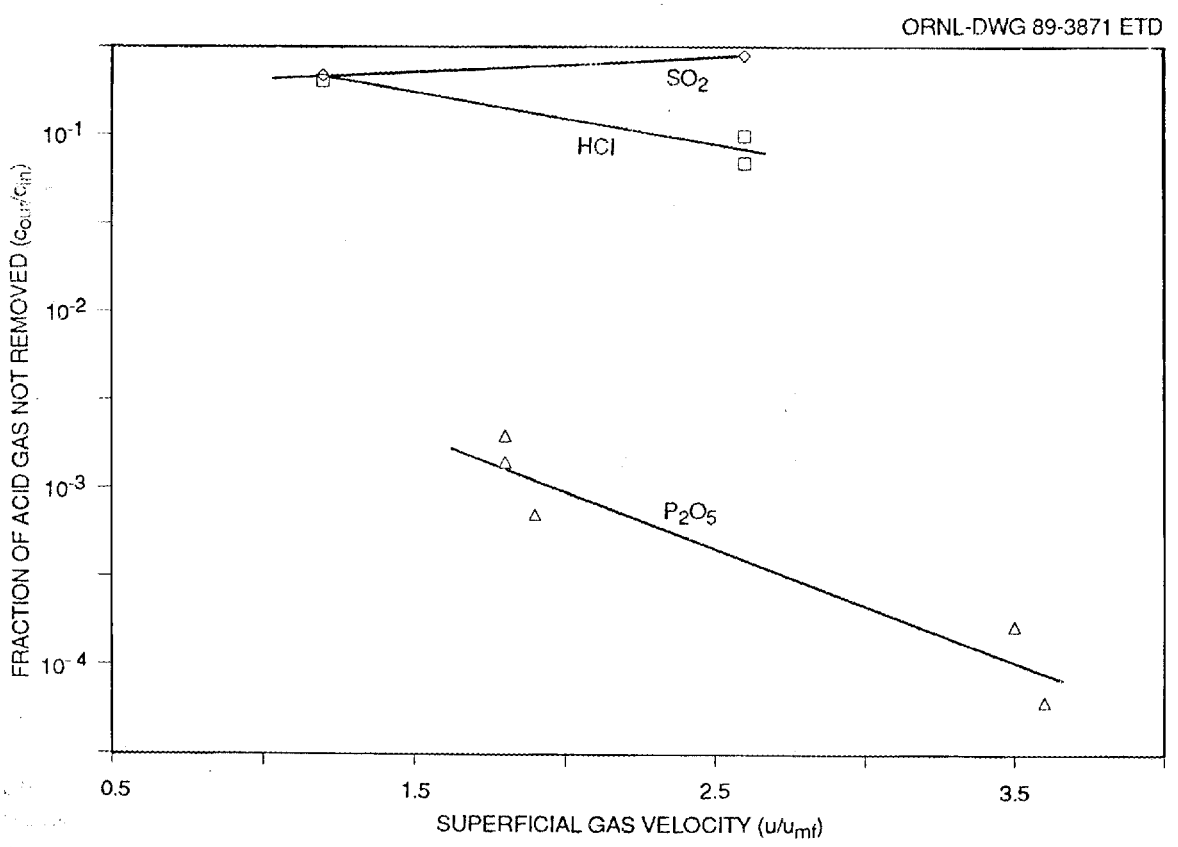

Fig. 4.1. Acid Gas removal vs superficial gas velocity.

are also included in Fig. 4.1.[1] Both HCl and P_2O_5 removal increased with gas velocity, while the SO_2 removal decreased slightly with velocity.

This phenomenon might be explained on the basis of the rate-limiting step involved in the gas-sorbent reaction. Both HCl and P_2O_5 are significantly stronger acids than SO_2 so it follows that the rate-limiting step may be different. In general, mass transfer between the bulk gas and any particular sorbent particle increases with an increase in gas velocity. It is possible that the HCl and P_2O_5 removal is limited by external mass transfer, while SO_2 removal is limited by internal mass transfer or reaction with the sorbent.

External mass transfer is also increased as sorbent particle size decreases. Because particle attrition increases with gas velocity, this could also contribute to the observed trend. (Data on the attrition rate for the lime used in this test are given in Appendix B.)

4.1.2.2 Bed temperature. Bed temperature was found to also affect the acid removal efficiency. The P_2O_5 removal increased slightly with temperature between 427 and 540°C (99.7% and 99.9%, respectively).[1] Both HCl and SO_2 showed a much more distinct temperature dependence. At 427°C (test 9), SO_2 removal was 21%, and the HCl removal was 9%. At the same gas velocity and 540°C (test 8), the HCl removal increased to 80% and the SO_2 removal increased to 78%.

All of the processes involved in acidic gas removal – external mass transfer, internal mass transfer, and chemical reaction – are temperature dependent. It is not certain that increasing the bed temperature beyond 540°C will increase removal. For example, >90% SO_2 removal has been reported at 75 to 165°C at very high humidity.[2] "Temperature windows" have been reported for lime-SO_2 processes, and there is no reason to doubt that "windows" exist for the lime-HCl reaction also.

The removal efficiency for HCl and SO_2 at 427°C in test 9 is very low. Lurgi has reported HCl and SO_2 removal of 95% and 90%, respectively, in a circulating fluidized bed (CFB) of hydrated lime at about 200°C.[3] Because the acid gas-lime reaction mechanism is a complex function of temperature, humidity, sorbent characteristics, and a host of other parameters, the temperature dependence can not be reliably predicted at this point.

4.1.2.3 Sorbent type. Lime, limestone, and pulverized dolomitic quicklime (57% CaO, 40% MgO) were tested for their effectiveness in removing acid gases. As expected, uncalcined limestone was a very poor sorbent in the chosen temperature range. In test 4, only 1% of the HCl and 3% of the SO_2 reacted with the limestone. These percentages are within the experimental error; thus, no removal could be claimed for HCl or SO_2. Test 3 was conducted with HCl in N_2 using dolomitic quicklime at 540°C and a gas velocity of 6.8 cm/s (u/u_{mf} = 2.6). The removal efficiency exceeded 96%, which is comparable to results obtained with calcined lime at the same conditions. Lime was selected as the sorbent for the remaining tests.

4.1.2.4 Residence time. In general, higher acid gas removal was observed as the gas residence time increased. For example, in test 8 (2.5-cm static bed depth at minimum fluidization and 540°C) HCl removal averaged 80%. Increasing the static bed depth to 10-cm at the same temperature and gas flow rate resulted in an average HCl removal of 96% (test 10). Similar results were obtained for P_2O_5 and SO_2. Bed expansion at room temperature was observed to be ≈10% at minimum fluidization for the sorbent; similar expansion would be expected at higher temperatures.

4.1.2.5 Inlet gas concentration. The acid gas removal efficiency was independent of inlet gas concentration at the conditions studied. Similar results have been reported for the lime-SO_2 reaction in an entrained bed.[2] However, for a given set of conditions, the removal efficiency was higher for a single acidic gas in an inert carrier gas than when other acidic gases were also present.

As shown in Fig. 4.2, HCl removal in test 11 was consistently higher when SO_2 was not present. A comparison of tests 5 and 7 shows that SO_2 removal decreases when the inlet HCl concentration is increased. The relationship between removal and presence of other acidic gases probably results from competition for reaction sites on the

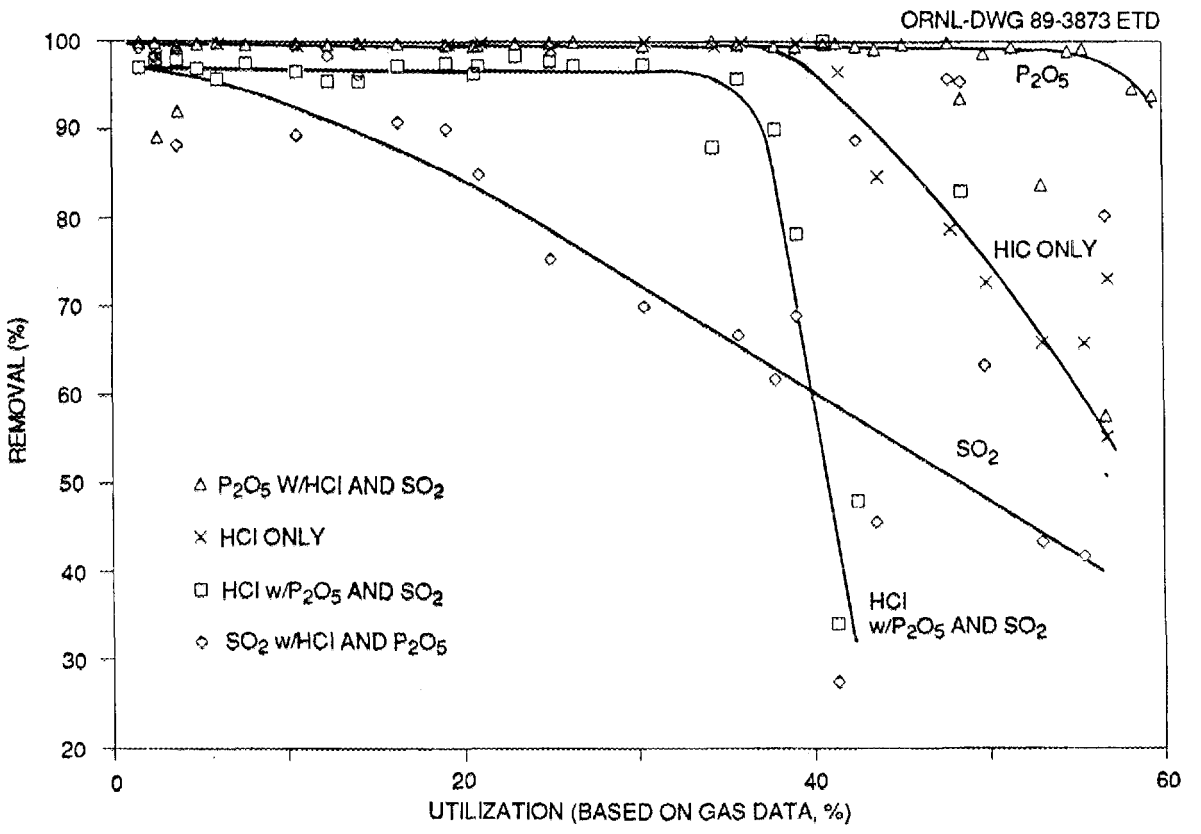

Fig. 4.2. Acidic gas removal efficiency vs sorbent utilization.

surface of sorbent particles. This phenomena is addressed in more detail in Sect. 4.2.4.

4.2 Long-Term Test

One long-term test (112 h) was conducted to determine the relationship between sorbent conversion and gas removal.

4.2.1 Sorbent conversion

The long-term test included all three acidic gases plus N_2, O_2, and CO_2, in ratios representative of an incinerator flue gas. The sorbent conversion at the conclusion of the test was 71% (based on solids analysis). Acidic gas removal was strongly dependent on sorbent conversion. The relationship between removal efficiency and sorbent conversion (using the gas data) is shown in Fig. 4.2.

4.2.2 Attrition and elutriation

During the 112 h of testing, 32% of the lime escaped from the bed (based on a calcium balance). The particles collected in the outlet filter were very fine, with >99% passing a 44-μm sieve. For the most part, the loss resulted from elutriation of fines from the bed. A 24-h blank run was conducted with an unreacted lime test bed using N_2 to fluidize the bed at the same flow rate and temperature as the long-term test. About 15% elutriation was observed for the unreacted lime.

4.2.3 Sorbent/gas material balance

The major products formed from the reaction of the lime and the acidic gases are calcium chloride ($CaCl_2$), calcium sulfate ($CaSO_4$) and calcium phosphate [$Ca_3(PO_4)_2$], which are all solids. At the conclusion of the long-term test, the sorbent contained slightly more $CaCl_2$ and $CaSO_4$ than predicted. A material balance using the gas data indicated that 36.8 g of chloride and 30.3 g sulfate should have been captured by the sorbent. A similiar balance based on solids data showed that the sorbent actually contained 39.4 g chloride and 36.0 g sulfate at the end of the test. These differences fall within the ±10% accuracy associated with the procedures used to determine the amount of Cl^- and SO_4^{-2} in liquid and solid samples.

4.2.4 Particle surface occlusion

As sorbent conversion increased, the pores on the particle surface became increasingly occluded with reaction product. This is clearly shown in scanning electron micrographs of particles from the long-term test. Figure 4.3 shows a typical lime particle (at 750X) before reacting with the sorbent. Figure 4.4 shows a spent particle from the long-

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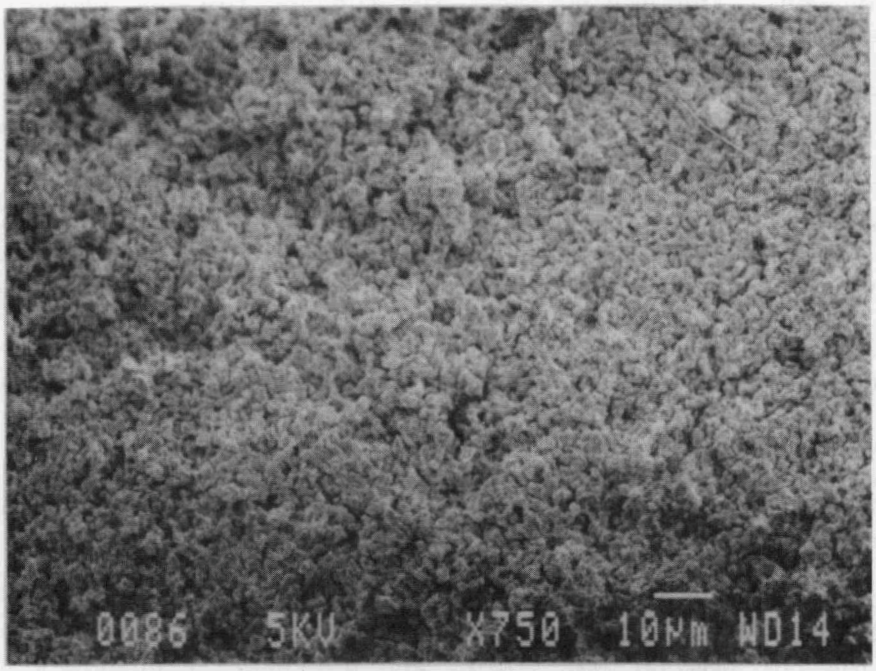

Fig. 4.3. Lime particle before test.

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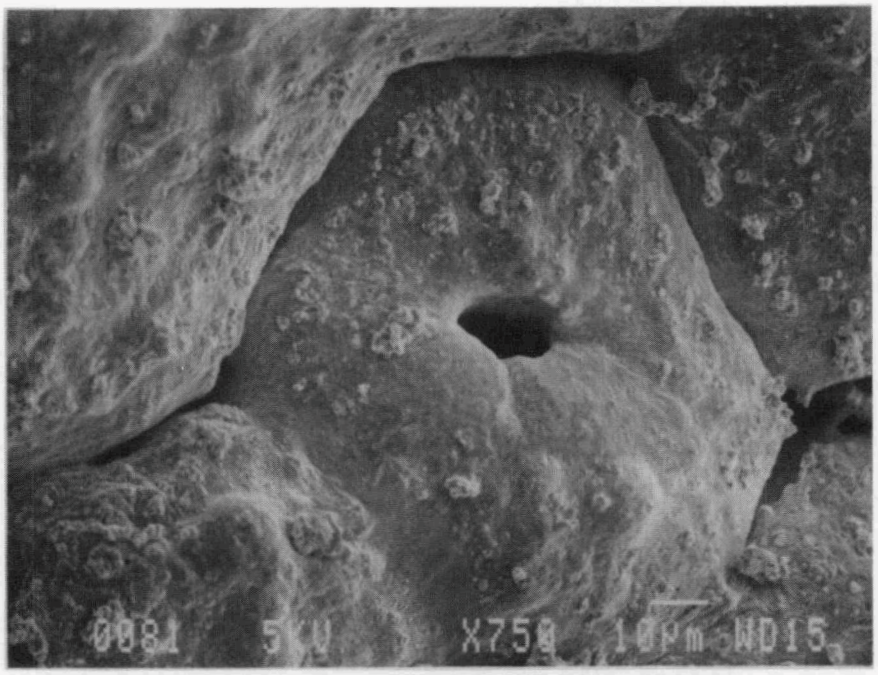

Fig. 4.4. Lime particle after test.

term test. The micrographs strongly suggest that, as the reaction proceeded, spent particles became coated on the surface and further reaction was impeded. The BET surface area for the raw and spent lime was 3.93 m^2/g and 1.87 m^2/g, respectively.

Surface occlusion is typically the Achilles' heel of dry-scrubbing processes that use lime or limestone. Pore plugging is inevitable because the volume per mole of calcium for CaO, $Ca_3(PO_4)_2$, $CaCl_2$, and $CaSO_4$ is 17, 33, 52, and 63 cm^3, respectively. The high molar volume of $CaSO_4$ makes SO_2 a particularly difficult gas to remove. Electron microprobe analysis of sorbent particles showed that many particles were almost completely coated with $CaSO_4$; it was virtually impossible to detect any other compounds on the surface.

A scan of the particle surface of the spent sorbent from the long-term test in Phase I, in which P_2O_5 was the only acidic gas, showed no tendency toward plugging. The scan showed <1% phosphorus (below detectable limits) although the bulk sorbent contained 1.8% phosphorus. It is believed that the bulk of the $Ca_3(PO_4)_2$ was formed within the pores of the lime particles and did not expand to the outer surface.

The $CaSO_4$ coating clearly impedes HCl removal. During the course of test 11, daily HCl-only removal data were taken to monitor breakthrough. The HCl removal was lower, and HCl breakthrough was observed earlier, for samples taken when HCl and SO_2 were scrubbed simultaneously. The data indicate that alternative SO_2 control strategies (e.g., operating an incinerator with low-sulfur fuel) will enhance HCl removal and increase the fraction of sorbent that is effectively used.

5. ECONOMIC COMPARISON OF SCRUBBING TECHNOLOGIES

Dry scrubbing is emerging as the method of choice for controlling acidic gas emissions from hazardous waste incinerators. Incineration facilities being built for Trade Waste Incineration, Inc. and Chemical Waste Management, Inc. use spray drying for controlling acidic gases.[4] So, too, does a new mobile system being developed by International Waste Energy Systems.[4] In all three cases, dry scrubbing was chosen over the more conventional wet scrubbing because of its lower life-cycle cost.

5.1 Cost Data for Flue Gas Desulfurization

In 1982, the Electric Power Research Institute (EPRI) evaluated 17 flue gas desulfurization processes for a hypothetical 1000-MW, coal-fired steam plant.[5,6] EPRI found the dry processes to be consistently less expensive than the wet processes. However, the dry processes were limited to low sulfur coal.

The scope of the EPRI report was limited to processes that were in use or had been demonstrated in a large-scale facility (>100 MW). Cost data for two conventional wet processes (limestone and wet lime) are shown in Table 5.1.

Table 5.1. Costs for flue gas desulfurization systems[a] ($\$ \times 10^6$)

	Conventional limestone[b]	Wet lime[b]	Spray dryer[c]	Nahcolite injection[c]	CFB[d] (lime)
Net capital cost	175.0	163.0	111.0	27.0	60.4
Fixed operating cost	10.5	9.6	7.0	1.7	4.5
Sorbent	7.1	14.7	2.0	15.3	1.9
Other operating costs	18.7	17.0	5.6	5.0	5.2
Total annual operating cost	36.3	41.3	14.6	22.0	11.6

[a]Based on a hypothetical 1000-MW steam plant, December 1982 dollars.

[b]Data from EPRI report CS-3342 for high-sulfur coal plant.

[c]Data from EPRI report CS-3342 for low-sulfur coal plant.

[d]Estimated based on similar equipment included in EPRI report CS-3342 for low-sulfur coal plant.

Dry systems have seen limited application at the plant scale. Spray drying is used in the electric power industry as well as the three hazardous waste incinerators cited above. Nahcolite ($NaHCO_3$) injection

has been demonstrated on a 223-MW unit that burns low-sulfur western coal. In the latter case, SO_2 is removed by contacting the flue gas with dry sodium-based sorbent in an entrained fluidized bed.[7]

Cost data for the CFB, the fifth process shown in Table 5.1, have been estimated from cost data on several processes in the EPRI report. Although a CFB system was not analyzed as part of the EPRI study, the assumptions used are consistent with those of the other four processes.

5.2 Cost Estimates for Hazardous Waste Incineration Scrubbing Systems

A large hazardous waste incinerator has a gross heating rate on the order of 44 MW (150 × 10^6 Btu/h). Scaling factors were applied to the EPRI data to estimate scrubbing costs for an incinerator of this size. The results are shown in Fig. 5.1. Cost data, inflation factors, and capacity scaling factors used to estimate the capital and fixed operating costs are included in Appendix C. The sorbent disposal cost is not included in this analysis because it varies widely depending on waste characteristics. In general, waste disposal will cost significantly less for processes that produce dry wastes (i.e., spray dryer, nahcolite injection, and CFB).

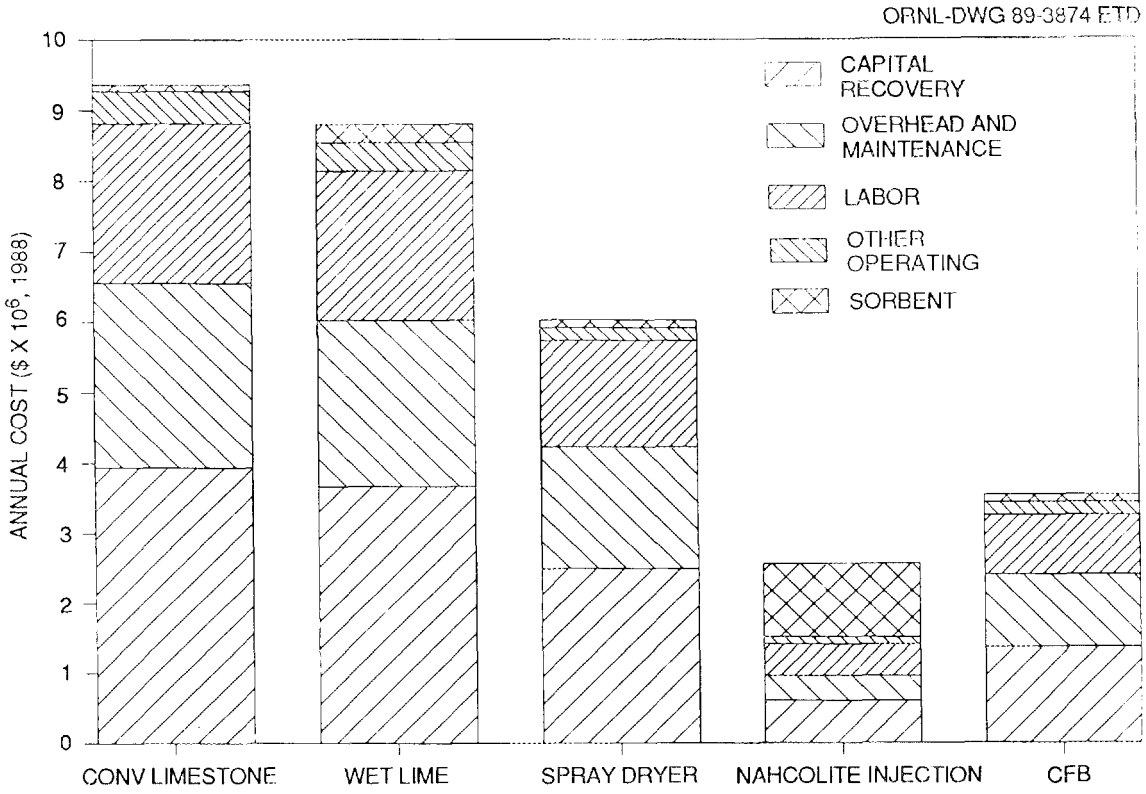

Fig. 5.1. Annual flue gas treatment cost for a 44-MW incinerator.

Figure 5.1 shows that nahcolite injection is the least expensive option for the non-site-specific case. This process is not capable of removing P_2O_5 because the sorbent sinters well below the P_2O_5 sublimation temperature (400°C). Furthermore, the residence time is limited for this process because of the high gas velocity. Both residence time and temperature were found to be important in removing HCl. Thus, nahcolite injection is not suitable for P_2O_5 and may not work for HCl, but it has been included for comparison.

5.3 Uncertainty of Cost Data

All of the cost data are based on EPRI Report CS-3342.[5,6] EPRI estimates that its data are ± 30% (in absolute terms) but are internally consistent to ± 15%. Scaling factors and capital cost indices (to adjust for inflation) add to the uncertainty. However, the data shown in Fig. 5.1 indicate that in general, CFB scrubbing is clearly less expensive than conventional wet processes.

6. SUMMARY AND CONCLUSIONS

Bench-scale tests support the feasibility of dry, fluidized-bed scrubbing of flue gas containing HCl, P_2O_5, and SO_2. The information obtained in Phases I and II of the bench-scale studies provides a basis for designing and testing a pilot-scale system.

Limestone is not an adequate sorbent for HCl and SO_2 at the temperature range of interest (450 to 650°C). A calcined sorbent such as hydrated lime or dolomitic lime is superior to limestone.

Maintaining accessible particle surface area is a key to maximizing sorbent utilization. Increased particle abrasion and attrition at higher gas velocity is beneficial to removal efficiency, if the loss caused by elutriation is controlled. A CFB would likely be a better candidate from this standpoint. High gas velocity and extensive particle attrition in the CFB would serve to minimize particle coating and therefore increase the availability of the sorbent.

Non-site-specific cost estimates for several acidic gas control technologies show that a CFB is less expensive than conventional scrubbing methods. The economic advantage probably increases when the waste disposal cost is factored into the analysis. However, the technical and cost uncertainty of CFB flue gas scrubbing is currently high due to lack of operational data at the pilot and production scale.

7. RECOMMENDATIONS

The feasibility of fluidized bed scrubbing has been demonstrated at the bench scale. A CFB is predicted to be less expensive than conventional wet scrubbing. The Army has a widespread need for hazardous waste incineration, and hence flue gas scrubbing, to support ongoing production activities and the installation restoration program. The cost of flue gas scrubbing will be a significant fraction of the total incineration cost.

The next step is pilot-scale testing, which should be performed with an actual flue gas stream from an incinerator operating on the order of 0.03 to 3 MW (0.1 to 10 $\times$ 10^6 Btu/h). The objectives of the pilot tests should include determining the operating characteristics of a CFB, confirming that bench-scale observations hold at the pilot-scale, and evaluating sorbent materials and characteristics.

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1. W. M. Bradshaw, *Laboratory Testing of a Fluidized Bed Dry Scrubbing Process for Removal of Sulfur Dioxide and Phosphorus Pentoxide from an Inert Carrier Gas*, AMXTH-TE-CR-88008, ORNL/TM-10753, Martin Marietta Energy Systems, Inc., Oak Ridge Natl. Lab., March 1988.

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6. R. J. Keeth et al., *Economic Evaluation of FGD Systems*, Vol. 5, EPRI CS-3342, Electric Power Research Institute, October 1986.

7. D. W. Albin et al., "100 + MW Demonstration of Dry Sodium Injection Flue Gas Desulfurization," pp. 7-37 through 7-42, *Proceedings: Tenth Symposium on Flue Gas Desulfurization*, Vol. 2, Atlanta, Georgia, June 1986.

Appendix A

CHARACTERIZATION OF BED MATERIAL

The majority of the Phase II tests used lime as the sorbent. This lime was 87% $Ca(OH)_2$ and 13% $CaCO_3$, and it was supplied by the Tenn-Luttrell Company in Luttrell, Tennessee.

The dolomitic quicklime used in Test No. 3 was nominally 57% CaO, 40% MgO, 3% other, and was supplied from National Lime & Stone Co. of Findlay, Ohio. The granular limestone used in Test No. 4 was received from the Calcium Carbonate Company of Quincy, Illinois.

All the sorbents were screened to delineate the size fractions. The 212- to 425-μm fraction was used in all the tests because it was less prone to channeling than the finer material, had a larger specific surface area than the large-size ranges, and required a lower gas flow rate for minimum fluidization than the large-size particles.[1]

Detailed data on the Tenn-Luttrell lime that was used in all of the tests except 3 and 4 is included in Table A.1.

Table A.1. Detailed description of the Tenn-Luttrell lime

Composition, (mass fraction)	87% $Ca(OH)_2$ 13% $CaCO_3$
Size distribution of sorbent, as supplied wt%[a]	
+1400 μm	1.4
1400 to 850 μm	11.9
850 to 600 μm	15.3
600 to 500 μm	6.4
500 to 425 μm	8.2
425 to 300 μm	11.2
300 to 250 μm	4.4
250 to 212 μm	5.2
212 to 180 μm	3.5
180 to 150 μm	5.2
150 to 90 μm	10.8
90 to 45 μm	13.5
-45 μm	3.2
Apparent loose density of 212- to 425-μm fraction, g/cm^3[a]	0.92
Apparent packed density of 212- to 425-μm fraction, g/cm^3[a]	0.94
Particle density of 212- to 425 μm fraction, g/cm^3	
displaced volume of varsol	2.94
displaced volume of diesel oil	3.15
Mean particle diameter for 212- to 425-μm fraction, μm	
weight mean	311
harmonic mean (Kuni and Levenspiel[3])	300
Geldart classification[4]	Group B

[a]Procedure described in ASTM Standard C 110-87.[2]

REFERENCES

1. W. M. Bradshaw, *Laboratory Testing of a Fluidized Bed Dry Scrubbing Process for Removal of Sulfur Dioxide and Phosphorus Pentoxide from an Inert Carrier Gas*, AMXTH-TE-CR-88008, ORNL/TM-10753, Martin Marietta Energy Systems, Inc., Oak Ridge Natl., Lab., March 1988.

2. *1988 Annual Book of ASTM Standards*, Volume 4.01 Cement; Lime; Gypsum, American Society for Testing and Materials, Philadelphia, Pennsylvania, 1988.

3. D. Kuni and O. Levenspiel, *Fluidization Engineering*, Robert E. Krieger Publishing Company, Inc., Huntington, N.Y., 1977.

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Appendix B

SORBENT ATTRITION TESTS

Two tests were run to determine the degree of sorbent attrition caused by bed fluidization. In each test, 100 g of 212- to -425 μm lime was placed in the bed, the bed and all pertinent lines were heated to 540°C (1000°F), and the bed was fluidized with dry N_2 for ~20 h. At the end of the test, the bed material was removed and weighed. It was then passed through the largest screen (425 μm), and the amount remaining on the screen was weighed and recorded. This process was repeated for all the screens.

Table B.1 shows how the particle size distribution changed as a result of fluidization. In general, the larger particles suffered more attrition than the smaller particles. The elutriation increased from 5% at minimum fluidization to 19% at twice minimum fluidization.

Table B.1. Particle size distribution of solids remaining in bed after fluidization for 20 h

Particle size (μm)	Before test (wt %)	$u/u_{mf}=1$ (wt %)	$u/u_{mf}=2$ (wt %)
>425		<0.5	<0.6
300–425	45.1	28.6	37.1
212–300	54.9	52.9	43.0
180–212		10.6	12.3
150–180		3.2	4.2
125–150		2.1	2.2
106–125		1.1	0.4
90–106		1.1	0.3

The fraction of particles in the various size ranges is shown graphically in Fig. B.1. At the conditions studied, the particle size distribution for lime remaining in the bed is independent of fluidization velocity for small particles (<180 μm). The small particles probably accounted for almost all of the elutriated material. (This is valid because u/u_{mf} never exceeded 2.) The quantity of small particles in this range was 13 g at minimum fluidization and 26 g at twice minimum fluidization (13% and 26%, respectively). The production of fines

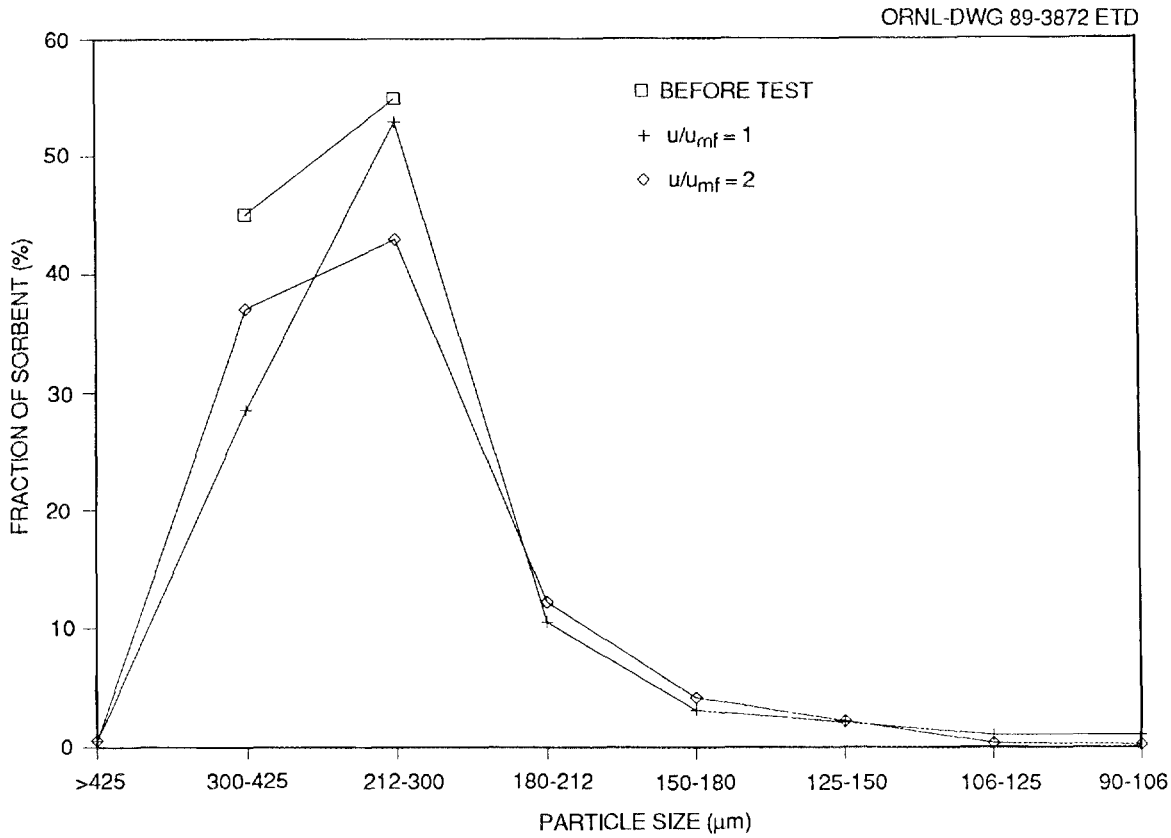

Fig. B.1. Lime particle size frequency distribution.

doubled between minimum fluidization velocity and twice minimum fluidization velocity.

The particle size distribution for large particles is dependent on gas velocity. In both tests, the fraction of larger (300 to 425 μm) particles decreased by ≈1/3. There was a marked difference in the distribution of intermediate particles. At minimum fluidization, the fraction of 212- to 300-μm particles was essentially the same as the starting material. At twice minimum fluidization, however, more of these particles became fines.

The relationship between particle size and fluidization velocity is important in evaluating the relationship between acid gas removal, sorbent conversion, and superficial gas velocity. A high attrition rate (resulting in smaller particles) will increase acid gas removal at a given conversion because smaller particles have a higher specific surface area (ratio of surface area to mass). However, for a given bed height, increasing gas velocity results in less gas residence time, which is another parameter that is positively related to acid gas removal.

Optimum operating conditions are a function of the sorbent, the acidic gas(es), and the type of fluidized bed. A circulating fluidized bed (CFB) could be operated over a fairly wide range of conditions, which would be necessary to select optimum conditions for maximizing removal efficiency. Experimental data from a CFB are necessary to pinpoint optimum conditions.

Appendix C

ASSUMPTIONS AND METHODS USED IN ECONOMIC ANALYSIS

The economic comparison of alternative scrubbing technologies in Sect. 5 is based largely on a study done for the Electric Power Research Institute (EPRI) in 1982 that compared the cost of various flue-gas-desulfurization technologies for a hypothetical 1000-MW coal-fired boiler station.[1] Cost data provided in that study, coupled with similar data in a follow-on study concerning dry injection flue gas desulfurization, were analyzed using EPRI guidelines and standard engineering economics methods to compare the cost of five flue gas treatment technologies.

C.1 Accuracy

The accuracy of data presented in the EPRI study is estimated by the authors to be ±30%. Because identical methodology was used to evaluate each technology, the EPRI authors estimate the relative accuracy between processes to be ±15%.

Incinerators are normally <10% of the capacity (in terms of heat load) of the boiler plant used for the EPRI study. Scaling capital and fixed operating costs for a process that is an order of magnitude smaller can result in significant error. Until a specific incinerator is identified at a specific site, scaling factors are just one of many sources of error that could skew the results. However, it is clear from the analysis that fluidized-bed technology is economically competitive and should be considered in a detailed site-specific economic analysis when the need for acidic gas control arises.

C.2 Assumptions

The assumptions used in the economic analysis and their sources are included in Table C.1.

C.3 Capital Cost

The capital costs for the limestone, wet lime, spray drying, and nahcolite injection processes were taken directly from the EPRI report.[1] The capital cost for the fluidized-bed process was based on data provided for similar unit operations in the EPRI report. A flow sheet was developed for the fluidized bed that paralleled the nahcolite injection process. The cost of the fluidized bed per se was estimated from cost data provided for similar process equipment in the EPRI report that is part of the NOXSO™ flue gas desulfurization process.

A scaling factor was used to adjust for plant capacity. The Marshall and Stevens index was used to adjust from 1982 to 1988 construction costs.[2]

Table C.1. Cost and process assumptions used in economic analysis

Element	Value used	Source[a]
	Economic factors	
Interest rate	10%	AMC-P 11-28 (Ref. 3)
Inflation rate	0%	AMC-P 11-28
Economic life	10 years	Peters and Timmerhaus[4]
Capital scaling factor	0.6	Peters and Timmerhaus
Labor scaling factor	0.2	Peters and Timmerhaus
Construction cost index	1.14	Marshall & Stevens Index 1988/1982 (Ref. 3)
	Process conditions	
Capacity	150 MW	Medium to large incinerator
O_2 concentration	5.1%	Typical hazardous waste incinerator (vol basis)
HCl concentration	1250 ppm	Typical hazardous waste incinerator (vol basis)
SO_2 concentration	310 ppm	Typical hazardous waste incinerator (vol basis)
	Variable costs	
Limestone	$7.5/ton	Tenn-Luttrell FOB cost, Nov. 1988 (Ref. 5)
Lime	$42/ton	*Chemical Marketing Reporter*[6]
Soda ash	$93/ton	*Chemical Marketing Reporter*
Labor cost index	394/325	*Chemical Engineering*[2]
Power cost	7¢/kW-h	Typical power rate, 1988
No. 6 fuel oil	55¢/gal	1988 GSA price

[a]See Sect. C.5 for a list of references.

C.4 Operating Cost

The sorbent requirement was adjusted for the quantity of acid that will be scrubbed. The ratio of moles of alkaline per mole of acid was assumed to be independent of throughput. The sorbent requirement for the fluidized bed was based on data obtained in test 11.

The EPRI estimates for sorbent transportation costs were used directly. It is impossible to evaluate transportation costs without specifying a site. Note that the transportation cost can exceed the sorbent cost, so this cost cannot be ignored in the site-specific case.

For a hazardous waste incinerator, sorbent disposal costs will be significant. The variability in this cost is so great that it has not

been included in the analysis. Disposal costs are probably lower for the dry products produced by spray drying, nahcolite injection, and fluidized-bed scrubbing because solids are easier to handle than sludge. This generalization is borne out in the EPRI data; disposal costs for solids are consistently lower.[1] However, wastes from boiler plant acid gas treatment are not normally hazardous wastes, as is the case with a hazardous waste incinerator, so no reliable prediction can be made for a non-site-specific hazardous waste incinerator based on EPRI cost data.

A scaling factor of 0.2 was also used to estimate labor requirements.[4] The hourly earnings index for chemical and allied products was used to adjust from 1982 to 1988 labor costs.[2]

C.5 References

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6. *Chemical Marketing Reporter*, October 28, 1988.

Appendix D

EXPERIMENTAL DATA

The data obtained in each of the tests are presented in Table D.1.

Table D.1 Experimental data

Test No. 1

Acid: HCl
Bed temperature: 540°C
N_2 flow: 7.71 mol/h
Superficial gas velocity: 6.8 cm/s
Total sorbent: 400 g lime

Sample No.	Location	Elapsed time (min)	HCl flow (10^{-6} mol/h)	HCl concentration (ppm)
1-1	Inlet	15	4,140	537
1-2	Outlet	60	<284	<37
1-3	Inlet	15	1,205	156
1-4	Outlet	60	<296	<38
1-5	Inlet	15	2,090	271
1-6	Outlet	60	<260	<34
1-7	Inlet	15	<1,111	<144
1-8	Outlet	60	<282	<37
1-9	Inlet	15	<1,066	<138
1-10	Outlet	60	<276	<36
1-11	Inlet	15	<1,106	<143

Table D.1 (continued)

Test No. 2

Acid: HCl
Bed temperature: 540°C
N_2 flow: 7.71 mol/h
Superficial gas velocity: 6.8 cm/s
Total sorbent: 400 g lime

Sample No.	Location	Elapsed time (min)	HCl flow (10^{-6} mol/h)	HCl concentration (ppm)
2-1	Inlet	15	13,416	1,740
2-2	Outlet	60	<265	<34
2-3	Inlet	15	16,192	2,100
2-4	Outlet	60	2,833	368
2-5	Inlet	15	12,670	1,640
2-6	Outlet	60	278	<36
2-7	Inlet	15	17,061	2,210
2-8	Outlet	60	289	<38
2-9	Inlet	15	13,743	1,780
2-10	Outlet	60	274	<36
2-11	Inlet	15	11,627	1,500

Test No. 3

Acid: HCl
Bed temperature: 540°C
N_2 flow: 7.71 mol/h
Superficial gas velocity: 6.8 cm/s
Total sorbent: 400 g dolomitic quicklime

Sample No.	Location	Elapsed time (min)	HCl flow (10^{-6} mol/h)	HCl concentration (ppm)
3-1	Inlet	15	7,662	994
3-2	Outlet	60	2,004	260
3-3	Inlet	15	11,821	1,530
3-4	Outlet	60	276	<36
3-5	Inlet	15	14,804	1,920
3-6	Outlet	60	291	<38
3-7	Inlet	15	18,620	2,420
3-8	Outlet	60	268	<35
3-9	Inlet	15	17,250	2,240
3-10	Outlet	60	286	<37
3-11	Inlet	15	19,351	2,510

Table D.1 (continued)

Test No. 4

Acid: P_2O_5, HCl, SO_2
Bed temperature: 540°C
N_2 flow: 6.46 mol/h
O_2 flow: 0.50 mol/h
CO_2 flow: 0.90 mol/h
Superficial gas velocity: 6.9 cm/s
Total Sorbent: 50 g limestone

Sample No.	Location	Elapsed time (min)	P_2O_5 flow (10^{-6} mol/h)	P_2O_5 concentration (ppm)	HCl flow (10^{-6} mol/h)	HCl concentration (ppm)	SO_2 flow (10^{-6} mol/h)	SO_2 concentration (ppm)
4-1	Inlet	15	50	8.1	NA[a]	NA	6,700	1,000
4-1	Outlet	30	2.1	0.33	NA	NA	6,200	960
4-2	Inlet	15	100	15.0	NA	NA	6,300	1,000
4-2	Outlet	30	4.7	0.72	NA	NA	6,100	940
4-3	Inlet	15	50	7.1	43,900	6,800	4,500	690
4-3	Outlet	30	1.1	0.17	11,000	1,700	4,000	620
4-4	Inlet	15	50	8.1	17,500	2,700	NA	NA
4-4	Outlet	30	1.5	0.23	14,300	2,200	NA	NA
4-5	Inlet	15	60	9.9	15,400	2,400	16,600	2,600
4-5	Outlet	30	3.2	0.49	15,000	2,300	10,100	1,600
4-6	Inlet	15	30	5.0	9,100	1,400	7,600	1,200
4-6	Outlet	30	6.0	0.93	7,600	1,200	5,400	840

[a]NA: Not available.

Table D.1 (continued)

Test No. 5

Acid: HCl, SO_2
Bed temperature: 540°C
N_2 flow: 6.46 mol/h
O_2 flow: 0.50 mol/h
CO_2 flow: 0.90 mol/h
Superficial gas velocity: 6.9 cm/s
Total sorbent: 50 g lime

Sample No.	Location	Elapsed time (min)	HCl flow (10^{-6} mol/h)	HCl concen-tration (ppm)	SO_2 flow (10^{-6} mol/h)	SO_2 concen-tration (ppm)
5-1	Inlet	30	3,800	590	4,300	670
5-1	Outlet	30	760	120	660	100
5-2	Inlet	30	6,100	950	7,000	1,100
5-2	Outlet	30	650	100	1,400	220
5-3	Inlet	30	6,100	940	5,300	810
5-3	Outlet	30	460	71	1,400	210
5-4	Inlet	30	5,900	910	6,000	940
5-4	Outlet	30	380	59	1,600	250
5-5	Inlet	30	4,800	740	3,900	600
5-5	Outlet	30	420	65	2,400	370

Test No. 6

Acid: HCl, SO_2
Bed temperature: 540°C
N_2 flow: 6.46 mol/h
O_2 flow: 0.50 mol/h
CO_2 flow: 0.90 mol/h
Superficial gas velocity: 6.9 cm/s
Total sorbent: 100 g lime

Sample No.	Location	Elapsed time (min)	HCl flow (10^{-6} mol/h)	HCl concen-tration (ppm)	SO_2 flow (10^{-6} mol/h)	SO_2 concen-tration (ppm)
6-1	Inlet	30	5,300	820	5,500	850
6-1	Outlet	30	100	15	<30	<5.0

Table D.1 (continued)

Test No. 7

Acid: P_2O_5, HCl, SO_2
Bed temperature: 540°C
N_2 flow: 6.46 mol/h
O_2 flow: 0.50 mol/h
CO_2 flow: 0.90 mol/h
Superficial gas velocity: 6.9 cm/s
Total sorbent: 50 g lime

Sample No.	Location	Elapsed time (min)	P_2O_5 flow (10^{-6} mol/h)	P_2O_5 concentration (ppm)	HCl flow (10^{-6} mol/h)	HCl concentration (ppm)	SO_2 flow (10^{-6} mol/h)	SO_2 concentration (ppm)
7-1	Inlet	30	<5.0	<1.0	5,500	850	4,700	720
7-1	Outlet	30	<5.0	<1.0	290	44	2,300	350
7-2	Inlet	30	<5.0	<1.0	5,900	920	5,000	770
7-2	Outlet	30	<5.0	<1.0	250	39	2,300	350
7-3	Inlet	30	<5.0	<1.0	3,900	600	2,800	440
7-3	Outlet	30	<5.0	<1.0	240	36	1,700	270
7-4	Inlet	15	<5.0	<1.0	6,800	1,000	4,000	620
7-4	Outlet	30	<5.0	<1.0	170	27	680	110
7-5	Inlet	15	<5.0	<1.0	17,800	2,800	10,100	1,600
7-5	Outlet	30	<5.0	<1.0	300	46	2,800	430
7-6	Inlet	15	120	19.0	12,200	1,900	8,100	1,300
7-6	Outlet	30	<5.0	<1.0	1,200	180	80	13
7-7	Inlet	15	14	2.1	10,100	1,600	NA[a]	NA
7-7	Outlet	30	5.3	0.83	<110	<16	NA	NA
7-8	Inlet	15	<5.0	<1.0	12,200	1,900	9,500	1,500
7-8	Outlet	30	<5.0	<1.0	220	33	300	47
7-9	Inlet	15	<5.0	<1.0	11,600	1,800	9,300	1,400
7-9	Outlet	30	<5.0	<1.0	370	58	1,200	180
7-10	Inlet	15	<5.0	<1.0	10,100	1,600	6,800	1,100
7-10	Outlet	30	<5.0	<1.0	590	92	3,200	500
7-11	Inlet	15	40	5.5	10,000	1,500	7,800	1,200
7-11	Outlet	30	<5.0	<1.0	580	89	1,300	200
7-12	Inlet	15	<5.0	<1.0	17,800	2,800	NA	NA
7-12	Outlet	30	<5.0	<1.0	2,000	300	NA	NA
7-13	Inlet	15	<5.0	<1.0	20,400	3,200	10,000	1,600
7-13	Outlet	30	<5.0	<1.0	3,800	590	6,800	1,100

[a]NA: Not available.

Table D.1 (continued)

Test No. 8

Acid: P_2O_5, HCl, SO_2
Bed temperature: 540°C
N_2 flow: 3.05 mol/h
O_2 flow: 0.22 mol/h
CO_2 flow: 0.40 mol/h
Superficial gas velocity: 3.2 cm/s
Total sorbent: 50 g lime

Sample No.	Location	Elapsed time (min)	P_2O_5 flow (10^{-6} mol/h)	P_2O_5 concentration (ppm)	HCl flow (10^{-6} mol/h)	HCl concentration (ppm)	SO_2 flow (10^{-6} mol/h)	SO_2 concentration (ppm)
8-1	Inlet	30	33	11	1,600	510	300	100
8-1	Outlet	30	2.4	0.78	110	36	39	13
8-2	Inlet	30	29	9.6	2,500	830	2,300	750
8-2	Outlet	30	1.6	0.51	260	85	74	24
8-3	Inlet	30	13	4.3	5,600	1,800	4,100	1,400
8-3	Outlet	30	2.9	0.96	330	110	310	100
8-4	Inlet	30	20	6.4	4,000	1,300	3,100	1,000
8-4	Outlet	30	0.97	0.32	320	100	450	150
8-5	Inlet	30	6.1	2.0	3,600	1,200	2,500	820
8-5	Outlet	30	0.97	0.32	270	89	490	160
8-6	Inlet	30	7.6	2.5	1,900	620	720	240
8-6	Outlet	30	0.75	0.25	260	85	400	130
8-7	Inlet	30	7.2	2.4	2,700	900	3,200	1,000
8-7	Outlet	30	0.92	0.30	300	98	560	190
8-8	Inlet	30	3.6	1.2	2,800	900	2,600	900
8-8	Outlet	30	0.70	0.23	310	100	540	180
8-9	Inlet	30	28	9.3	5,200	1,700	4,200	1,400
8-9	Outlet	30	1.4	0.45	260	85	350	120
8-10	Inlet	30	1.9	0.61	3,400	1,100	3,000	1,000
8-10	Outlet	30	0.65	0.21	270	89	320	110
8-11	Inlet	30	33	11	3,200	1,100	3,100	1,000
8-11	Outlet	30	1.1	0.37	1,000	330	1,600	530
8-12	Inlet	30	NA[a]	NA	20,900	6,900	13,300	4,400
8-12	Outlet	30	NA	NA	1,600	530	1,300	430
8-13	Inlet	30	76	25	3,500	1,100	3,600	1,200
8-13	Outlet	30	0.76	0.25	2,400	790	1,500	480
8-14	Inlet	30	160	53	4,100	1,300	3,200	1,000
8-14	Outlet	30	1.0	0.33	2,500	820	1,600	510
8-15	Inlet	30	180	60	3,500	1,100	4,000	1,300
8-15	Outlet	30	1.1	0.37	1,700	550	1,200	400
8-16	Inlet	30	64	21	3,300	1,100	4,000	1,304
8-16	Outlet	30	0.94	0.31	2,300	750	1,600	530

[a]NA: Not available.

Table D.1 (continued)

Test No. 9

Acid: P_2O_5, HCl, SO_2
Bed temperature: 427°C
N_2 flow: 3.05 mol/h
O_2 flow: 0.22 mol/h
CO_2 flow: 0.40 mol/h
Superficial gas velocity: 2.8 cm/s
Total sorbent: 50 g lime

Sample No.	Location	Elapsed time (min)	P_2O_5 flow (10^{-6} mol/h)	P_2O_5 concentration (ppm)	HCl flow (10^{-6} mol/h)	HCl concentration (ppm)	SO_2 flow (10^{-6} mol/h)	SO_2 concentration (ppm)
9-1	Inlet	30	2.7	0.90	3,700	1,200	3,800	1,300
9-1	Outlet	30	0.68	0.22	3,200	1,000	2,200	730
9-2	Inlet	30	3.5	1.1	3,100	1,000	3,800	1,200
9-2	Outlet	30	0.86	0.28	2,700	880	520	170
9-3	Inlet	30	5.8	1.9	3,400	1,100	3,600	1,200
9-3	Outlet	30	0.75	0.25	3,300	1,100	3,000	1,000
9-4	Inlet	30	1.9	0.61	3,200	1,000	3,700	1,200
9-4	Outlet	30	0.50	0.16	2,800	930	3,100	1,000
9-5	Inlet	30	3.3	1.1	4,000	1,300	4,300	1,400
9-5	Outlet	30	0.69	0.23	2,600	840	4,000	1,300
9-6	Inlet	30	1.3	0.44	NA[a]	NA	NA	NA
9-6	Outlet	30	0.56	0.18	NA	NA	NA	NA

[a]NA: Not available.

Table D.1 (continued)

Test No. 10

Acid: P_2O_5, HCl, SO_2
Bed temperature: 540°C
N_2 flow: 3.05 mol/h
O_2 flow: 0.22 mol/h
CO_2 flow: 0.40 mol/h
Superficial gas velocity: 3.2 cm/s
Total sorbent: 200 g lime

Sample No.	Location	Elapsed time (min)	P_2O_5 flow (10^{-6} mol/h)	P_2O_5 concentration (ppm)	HCl flow (10^{-6} mol/h)	HCl concentration (ppm)	SO_2 flow (10^{-6} mol/h)	SO_2 concentration (ppm)
10-1	Inlet	30	520	170.0	4,400	1,400	220	72
10-1	Outlet	30	1.0	0.33	1,000	330	<40	<13
10-2	Inlet	30	530	170.0	4,400	1,400	380	120
10-2	Outlet	30	0.51	0.17	340	110	<3.3	<1.1
10-3	Inlet	30	420	140	3,800	1,200	2,600	870
10-3	Outlet	30	0.38	0.12	250	83	<3.7	<1.2
10-4	Inlet	30	53	18	8,200	2,700	5,300	1,700
10-4	Outlet	30	0.56	0.18	90	30	<3.7	<1.2
10-5	Inlet	30	20	6.6	8,300	2,700	2,700	870
10-5	Outlet	30	0.75	0.25	61	20	<3.7	<1.2
10-6	Inlet	30	55	18	11,400	3,700	3,200	1,100
10-6	Outlet	30	0.57	0.19	61	20	<3.7	<1.2

Table D.1 (continued)

Test No. 11

Acid: P_2O_5, HCl, SO_2
Bed temperature: 540°C
N_2 flow: 6.46 mol/h
O_2 flow 0.50 mol/h
CO_2 flow 0.90 mol/h
Superficial gas velocity: 6.9 cm/s
Total sorbent: 100 g lime

Sample No.	Location	Elapsed time (min)	P_2O_5 flow (10^{-6} mol/h)	P_2O_5 concentration (ppm)	HCl flow (10^{-6} mol/h)	HCl concentration (ppm)	SO_2 flow (10^{-6} mol/h)	SO_2 concentration (ppm)
11-1	Inlet	15	8.1	1.2	12,600	2,000	<720	<110
11-1	Outlet	180	<0.02	<0.0025	280	44	<6.3	<0.97
11-2	Inlet	15	15	2.3	6,600	1,000	<780	<120
11-2	Outlet	180	0.02	0.0032	120	19	<14	<2.1
11-3	Inlet	15	3.6	0.56	5,200	800		
11-3	Outlet	15	0.65	0.10	<170	<27		
11-4	Inlet	15	8.3	1.3	14,500	2,200	<150	<23
11-4	Outlet	120	0.03	0.0041	180	27	<18	<2.7
11-5	Inlet	15	2.5	0.39	2,000	310		
11-5	Outlet	15	0.20	0.031	36	5.6		
11-6	Inlet	15	2.6	0.40	9,200	1,400	1,300	200
11-6	Outlet	180	0.02	0.0027	290	45	860	130
11-7	Inlet	15	7.4	1.2	9,500	1,500	730	110
11-7	Outlet	180	0.02	0.0026	300	46	450	69
11-8	Inlet	15			4,300	660		
11-8	Outlet	15			24	3.7		
11-9	Inlet	15	5.7	0.88	9,100	1,400	NA[a]	NA
11-9	Outlet	180	0.04	0.0069	290	46	NA	NA
11-10	Inlet	15	16	2.5	14,900	2,300	5,900	910
11-10	Outlet	180	0.05	0.0071	350	54	620	96
11-11	Inlet	20			5,300	830		
11-11	Outlet	20			43	6.7		
11-12	Inlet	15	1.2	0.19	8,700	1,300	3,200	490
11-12	Outlet	180	0.02	0.0029	370	58	57	8.7
11-13	Inlet	15	8.6	1.3	8,000	1,200	3,900	600
11-13	Outlet	180	0.02	0.0030	360	55	150	23
11-14	Inlet	15			7,700	1,200		
11-14	Outlet	15			32	5.0		
11-15	Inlet	15	4.8	0.74	10,700	1,700	4,400	690
11-15	Outlet	180	0.01	0.0022	300	46	520	81
11-16	Inlet	15	5.2	0.80	11,100	1,700	7,100	1,100
11-16	Outlet	180	0.02	0.0027	280	44	700	110

[a]NA: Not available.

Table D.1 (continued)

Test No. 11, continued

Acid: P_2O_5, HCl, SO_2
Bed temperature: 540°C
N_2 flow: 6.46 mol/h
O_2 flow: 0.50 mol/h
CO_2 flow: 0.90 mol/h
Superficial gas velocity: 6.9 cm/s
Total sorbent: 100 g lime

Sample No.	Location	Elapsed time (min)	P_2O_5 flow (10^{-6} mol/h)	P_2O_5 concentration (ppm)	HCl flow (10^{-6} mol/h)	HCl concentration (ppm)	SO_2 flow (10^{-6} mol/h)	SO_2 concentration (ppm)
11-17	Inlet	15			12,100	1,900		
11-17	Outlet	15			34	5.3		
11-18	Inlet	15	2.5	0.39	11,300	1,800	1,600	240
11-18	Outlet	180	0.02	0.0026	230	36	950	150
11-19	Inlet	15	3.5	0.55	1,300	200	640[b]	100[b]
11-19	Outlet	180	0.02	0.0028	200	31	97	15
11-20	Inlet	15			13,200	2,000		
11-20	Outlet	15			32	4.9		
11-21	Inlet	15	1.4	0.22	12,900	2,000	350	54
11-21	Outlet	180	0.03	0.0042	230	35	58	9.0
11-22	Inlet	15	19	2.9	14,300	2,200	2,000	300
11-22	Outlet	180	0.03	0.0040	220	34	480	75
11-23	Inlet	15			4,900	750		
11-23	Outlet	15			50	7.7		
11-24	Inlet	15	2.6	0.40	7,200	1,100	NA[a]	NA
11-24	Outlet	180	0.03	0.0043	450	69	NA	NA
11-25	Inlet	15	110	17	25,900	4,000	5,800[b]	910[b]
11-25	Outlet	180	0.53	0.082	440	69	1,800	270
11-26	Inlet	15			8,100	1,300		
11-26	Outlet	15			19	2.9		
11-27	Inlet	15	60	9.3	30,200	4,700	NA	NA
11-27	Outlet	180	0.02	0.0023	2,100	330	NA	NA
11-28	Inlet	20			5,300	820		
11-28	Outlet	20			39	6.1		
11-29	Inlet	15	4.7	0.73	6,600	1,000	3,900	600
11-29	Outlet	180	<0.02	<0.0026	630	97	1,300	200
11-30	Inlet	15			23,000	3,600		
11-30	Outlet	15			32	4.9		
11-31	Inlet	15	2.5	0.39	10,800	1,700	5,000	770
11-31	Outlet	180	<0.02	<0.0023	830	130	1,800	280

[a]NA: Not available.

[b]Estimated SO_2 input.

Table D.1 (continued)

Test No. 11, continued

Acid: P_2O_5, HCl, SO_2
Bed temperature: 540°C
N_2 flow: 6.46 mol/h
O_2 flow: 0.50 mol/h
CO_2 flow: 0.90 mol/h
Superficial gas velocity: 6.9 cm/s
Total sorbent: 100 g lime

Sample No.	Location	Elapsed time (min)	P_2O_5 flow (10^{-6} mol/h)	P_2O_5 concentration (ppm)	HCl flow (10^{-6} mol/h)	HCl concentration (ppm)	SO_2 flow (10^{-6} mol/h)	SO_2 concentration (ppm)
11-32	Inlet	15	2.8	0.44	5,900	910	4,500	700
11-32	Outlet	180	<0.02	<0.0027	1,700	260	1,400	220
11-33	Inlet	15			9,300	1,400		
11-33	Outlet	15			21	3.3		
11-34	Inlet	15	4.4	0.68	NA[a]	NA	NA	NA
11-34	Outlet	180	<0.02	<0.0026	NA	NA	NA	NA
11-35	Inlet	15	7.0	1.1	8,000	1,200	8,300	1,300
11-35	Outlet	180	<0.02	<0.0025	6,300	980	6,000	930
11-36	Inlet	15			11,100	1,700		
11-36	Outlet	15			370	57		
11-37	Inlet	15	2.7	0.42	10,100	1,600	2,100	320
11-37	Outlet	180	<0.01	<0.0021	5,200	810	170	26
11-38	Inlet	15	2.1	0.32	NA	NA	880	140
11-38	Outlet	180	<0.02	<0.0027	NA	NA	480	74
11-39	Inlet	15			6,200	960		
11-39	Outlet	15			940	150		
11-40	Inlet	15	7.1	1.1	NA	NA	NA	NA
11-40	Outlet	180	0.03	0.0039	NA	NA	NA	NA
11-41	Inlet	15	6.9	1.1	NA	NA	7,100	1,100
11-41	Outlet	180	<0.02	<0.0024	NA	NA	3,000	460
11-42	Inlet	15			10,200	1,600		
11-42	Outlet	15			1,600	250		
11-43	Inlet	15	2.1	0.33	4,800	740	680	100
11-43	Outlet	180	0.10	0.016	810	130	65	10
11-44	Inlet	15	<1.0	<0.16	NA	NA	2,200	340
11-44	Outlet	180	<0.01	<0.0023	NA	NA	800	120
11-45	Inlet	15			4,700	730		
11-45	Outlet	15			1,300	200		
11-46	Inlet	15	9.0	1.4	NA	NA	NA	NA
11-46	Outlet	180	0.05	0.0076	NA	NA	NA	NA
11-47	Inlet	15	6.1	0.94	NA	NA	6,400	1,000
11-47	Outlet	180	0.98	0.15	NA	NA	3,600	560

[a]NA: Not available.

Table D.1 (continued)

Test No. 11, continued

Acid: P_2O_5, HCl, SO_2
Bed temperature: 540°C
N_2 flow: 6.46 mol/h
O_2 flow: 0.50 mol/h
CO_2 flow: 0.90 mol/h
Superficial gas velocity: 6.9 cm/s
Total sorbent: 100 g lime

Sample No.	Location	Elapsed time (min)	P_2O_5 flow (10^{-6} mol/h)	P_2O_5 concen-tration (ppm)	HCl flow (10^{-6} mol/h)	HCl concen-tration (ppm)	SO_2 flow (10^{-6} mol/h)	SO_2 concen-tration (ppm)
11-48	Inlet	15			4,100	640		
11-48	Outlet	15			1,400	220		
11-49	Inlet	15			NA	NA	NA	NA
11-49	Outlet	180	<0.02	<0.0023	NA	NA	NA	NA
11-50	Inlet	15	1.6	0.25	6,800	1,100	7,400	1,100
11-50	Outlet	180	<0.01	<0.0022	5,800	890	4,300	670
11-51	Inlet	15			2,600	400		
11-51	Outlet	15			890	140		
11-52	Inlet	15	2.7	0.41	NA[a]	NA	1,100	170
11-52	Outlet	180	1.1	0.17	NA	NA	220	34
11-53	Inlet	15			4,800	750		
11-53	Outlet	15			2,700	420		
11-54	Inlet	15			7,200	1,100		
11-54	Outlet	15			1,900	300		
11-55	Inlet	15	2.2	0.33	NA	NA	NA	NA
11-55	Outlet	180	0.12	0.02	NA	NA	NA	NA
11-56	Inlet	15	2.1	0.32	NA	NA	6,900	1,100
11-56	Outlet	180	0.13	0.02	NA	NA	6,200	960

[a]NA: Not available.

APPENDIX E

DETAILED OPERATING PROCEDURES, SAMPLE CALCULATIONS, AND EQUIPMENT SPECIFICATIONS

E.1 Operating Procedures

The procedures used during the course of the test program are outlined below.

1. Insert sorbent and warm up scrubber in preparation for testing the following day.
   a. Sieve sorbent according to ASTM Standard C 110-87[1] to obtain the required quantity of sorbent in the specified size range.
   b. Weigh sorbent (to the nearest 0.1 g) and pour it into the column through the top flange using a funnel to ensure that the sorbent is not caught on the transition section. Tap the bottom flange several times with a hammer to insure that the sorbent settles evenly across the bed.
   c. Assemble the top flange, outlet filter, and outlet sampling system and leak check the system at 20 psig.
   d. Adjust the flow on the main N_2 line to ≈1 mol/h. Allow the N_2 to flow through the column overnight.
   e. Set the main N_2 heater, outlet sample line heaters, and the column heaters at the specified temperature and allow the system to come to thermal steady state overnight.
   f. Log the bed and sample line temperatures every hour overnight.

2. Start up the P_2O_5 column.
   a. Adjust the flow of dry N_2 through the P_2O_5 column to ≈1.5 mol/h. Set the valves at the inlet to the bed to insure that all of the flow is exiting through the inlet sample port.
   b. Turn on the sample line heaters and wait for them to reach 400°C.
   c. Set the temperature of the P_2O_5 column heater to obtain the desired P_2O_5 flow rate based on previous data for the column.
   d. Allow the column to come to steady state by visually monitoring the rate that P_2O_5 condenses in the inlet sample bottle (this normally takes 1 h).
   e. Ensure that the P_2O_5 flow has reached steady state by titrating two samples taken 15 min apart with 0.1 or 0.01-*N* NaOH (depending on P_2O_5 flow rate).

3. Adjust HCl and SO_2 flow.
   a. Increase the N_2 flow to the value to be used in the test. Ensure that all of the gases are still exiting the scrubber at the inlet sample port.
   b. Set the needle valves on the HCl and SO_2 rotameters to achieve the specified flows. The relationship between mass flow rate and rotameter setting is based on calibration curves.
   c. Wait 15 min for the HCl and SO_2 flow to reach steady state. Check the rotameters every 5 min to ensure that they are not drifting.

4. Take inlet and outlet samples.
   a. Replace the inlet sample bottle with a gas washing bottle containing the wet scrubbing solution. In general, distilled water is used for HCl and P_2O_5; aqueous NaOH solutions are used to trap SO_2.
   b. During the last 5 minutes of the first inlet sample, set the flow of O_2 and CO_2 to the specified levels.
   c. Place an similar sample bottle on the outlet sample port. Wait the specified time for the inlet sample. Simultaneously close the inlet sample valve and open the outlet sample valve to route the flow to the scrubber.
   d. Prepare a similar sample bottle for the inlet and switch back to sampling at the inlet after the specified time.
   e. Repeat the procedure of sampling at the inlet and outlet of the scrubber during the course of the test.

5. Process samples and collect data.
   a. Transfer each sample from the gas washing bottle to a sample bottle. Thoroughly rinse the gas washing bottle with distilled water and add the rinse solution to the sample bottle. Measure and record the total volume of the sample. Note that the rinsing process is particularly necessary for samples containing P_2O_5 because it tends to condense on the glass tube leading to the fritted glass plate.
   b. Transfer a portion of the aqueous sample to a second bottle and submit it to the analytical laboratory for testing.
   c. Record all mass flow rates and pressures while each sample is being taken. Ensure that the data logger is recording temperatures.

6. Estimate total acid gas flow and control process.
   a. Titrate a portion of each inlet and outlet sample to insure that the process stays at steady state and that the total inlet acidic gas flow rate remains constant throughout the test (within the accuracy of the titration technique).
   b. Monitor all rotameters to ensure that gas flows do not change during the course of the test.
   c. Monitor the pressure drop across the distributor plate and across the bed to ensure that the distributor is not plugging and that the bed is not channeling.

7. Shut the system down.
   a. At the conclusion of testing, turn off the O_2, CO_2, HCl, and SO_2 flow (at the bottle) and turn off the heater for the P_2O_5 sublimer. Wait until the P_2O_5 sublimer cools at least 100°C before shutting the N_2 flow off.
   b. Close all valves and pressurize the system to 25 psig with dry N_2. Take particular care to maintain positive pressure on the P_2O_5 sublimer and to tightly close the Nupro™ valves leading to and from the sublimer.

c. Let the system cool over night. The pressure in the scrubber will drop as it cools. Once the system reaches room temperature, add dry N_2 as necessary to bring the system back to 0 psig.

d. Remove the sorbent from the system by removing the inlet header (immediately below the distributor plate) and catching the distributor plate and spent sorbent in a plastic bag. Tap the scrubber with a hammer to ensure that no sorbent particles remain on the walls.

E.2 Sample Calculations

Sample calculations for the different quantities reported in this report are included below.

Gas flow rate – The gas flow rate was measured with calibrated rotameters. The mass flow was adjusted for fluid density as described in the *Chemical Engineers' Handbook*[2]

Example:

If the calibration curve indicated that "2 scfh" on the rotameter equated to 2.65 mol/h for dry air at 25°C and 101 kPa, the mass flow rate for dry N_2 at 22°C and 94 kPa at the same rotameter setting was estimated by:

$$\frac{\dot{M}_{N_2}}{\dot{M}_{Air}} \approx \sqrt{\frac{\rho_{N_2}}{\rho_{Air}}} \text{ for } \rho_f >> \rho_{N_2};\ \rho_f >> \rho_{Air};\ \text{and } \rho_{N_2} \approx \rho_{Air}$$

where

$\dot{M}_{N_2}$ = mass flow rate of N_2,
$\dot{M}_{Air}$ = mass flow rate of air,
ρ_{N_2} = density of N_2,
ρ_{Air} = density of air, and
ρ_f = density of rotameter float.

For this case

$$\rho_{N_2} = \frac{(94{,}000\ N/m^2)(28 g/mol)}{8.314\ \frac{Nm}{k\ mol}\ (298\ k)} = 1060\ g/m^3$$

$$\rho_{Air} = \frac{(101{,}000\ N/m^2)(28.84 g/mol)}{8.314\ \frac{Nm}{k\ mol}\ (295\ k)} = 1190\ g/m^3$$

$$\dot{M}_{Air} = (2.65\ mol/h)(28.8\ g/mol) = 76.3\ g/h$$

$$\dot{M}_{N_2} = 76.3\ g/h \sqrt{\frac{1060\ g/m^3}{1190\ g/m^3}}$$

$$\dot{M}_{N_2} = 72.0\ g/h \text{ or } 2.57\ mol/h$$

Superficial gas velocity – The superficial gas velocity was determined by summing the molar gas flow rates and using the ideal gas law to estimate the average flow of gas through the bed. Changes to the inside diameter of the bed due to thermal expansion were neglected.

Example:

If the flow rate of N_2, O_2, and CO_2 were 6.46, 0.50, and 0.90 mol/h, respectively, and the flow of P_2O_5, HCl, and SO_2 were 0.05, 10, and 8 mmol/h respectively; and the bed was operating at 540°C and 98.9 kPa, then the superficial gas velocity was estimated to be:

$$\text{Total gas flow} = [(6.46 + 0.50 + 0.90) + (0.05 + 10 + 8) \times 10^{-3}]\ mol/h = 7.88\ mol/h$$

$$\text{Area of bed} = \frac{\pi}{4}[(2.067\ in.)^2(2.54\ cm/in.)^2] = 21.65\ cm^2$$

$$\text{Volumetric gas flow} = (7.88\ mol/h)\left[\frac{(8.314\ \frac{Nm}{mol\ k})(813\ k)}{98{,}900\ N/m^2}\right]$$

$$= 0.539\ m^3/h$$

$$(0.539\ m^3/h)\left(\frac{100\ cm}{m}\right)^3\left(\frac{1h}{3600s}\right)$$

$$= 150\ cm^3/s$$

$$\text{Superficial gas velocity} = \frac{150\ cm^3/s}{21.65\ cm^2} = 6.91\ cm/s$$

Acidic gas flow rate – Rotameters were used to control the acidic gas flow rate, but the actual flow was determined by analyzing an aqueous solution for the anion associated with the particular acid. For example, HCl flow was determined by the amount of Cl^- ions trapped in an aqueous solution over a specified length of time.

Example:

If the total volume from a gas washing bottle was 180 mL, the sample was collected over a period of 1 h, and the analytical laboratory reported that the concentration of Cl^- ions was 1800 mg/L, then the flow rate of

HCl was determined to be:

$$\text{HCl flow} = \frac{(0.180\ \text{L})(1.8\ \text{g Cl}^-/\text{L})(10^6\ \mu\text{mol/mol})}{(1.00\ \text{h collection time})(35.45\ \text{g Cl}^-/\text{mol HCl})}$$

$$= 9100\ \mu\text{mol/h}$$

Acidic gas removal – The acidic gas removal rate was simply determined by comparing the total mass flow for the particular gas at the exit of the scrubber with the total mass flow for the same gas at the inlet to the scrubber.

For example, using data from Test 9, the HCl removal rate is estimated by:

Sample #	HCl flow (mmol/h)	
	Inlet	Outlet
1	3.7	3.2
2	3.1	2.7
3	3.4	3.3
4	3.2	2.8
5	4.0	2.6
average	3.48	2.92

$$\text{average removal} = 1 - \frac{2.92\ \mu\text{mol/L}}{3.48\ \mu\text{mol/h}}$$

$$= 0.16 \text{ or } 16\%$$

Since all samples were taken for the same length of time, this method equates to a "time-weighted average" removal rate. If different sample times had been used, appropriate corrections would have been required to obtain the time-weighted average flow rate.

Sorbent utilization – The sorbent utilization is defined as the ratio of the equivalents of acidic gas neutralized to the equivalents of base contained in the sorbent.

For example, analytical tests showed that the sorbent used in Test 11 can neutralize 0.026 equivalents/g. The total acidic gas removed by 100 g of sorbent (based on gas sampling data) was 1.04, 0.51,

and 0.01 equivalents for HCl, SO_2 and P_2O_5, respectively. Therefore, the sorbent utilization at the conclusion of testing (based on gas data) was:

$$\text{Utilization} = \frac{(1.04 + 0.51 + 0.01 \text{ equivalents removed})}{(0.026 \text{ equivalents/g sorbent})(100 \text{ g sorbent})}$$

$$= 0.60 \text{ or } 60\% \text{ sorbent utilization.}$$

E.3 Equipment Specifications and Measurements

The equipment used to measure the various parameters studied is listed in Table E-1. A brief description of how those parameters were measured is included below.

Gas flow – All gas flows were measured with variable area rotameters. They were calibrated according to ORNL I&C Procedure M100432.

Table E.1. Equipment used to measure inert gas flow, temperature, pressure, sorbent particle size, and acidic gas flow

Item	Manufacturer	Model Number
Gas flow measurement		
N_2(main line)	Fisher-Porter	Tube 8701A0761A1
N_2(P_2O_5 sublimer)	Fisher-Porter	Tube 7908A0780A1
O_2	Fisher-Porter	Model 10A6130
CO_2	Fisher-Porter	Model 10A6130
HCl	Fisher-Porter	Tube FP 1/8-038-G-6
SO_2	Matheson Scientific	Tube FM-1050-VI
Temperature measurement		
Thermocouples	Omega Engineering	Type K-CASS-18G-12
Temperature controllers	Omega Engineering	4001 KC
Data logger	Fluke	2200 B
Pressure measurement		
Differential pressure transducer	MKS, Inc.	223 BD
Power supply/vendout	MKS, Inc.	PDR C5B
Absolute pressure measurement	Cenco Scientific	Hg barometer
Particle size measurement		
U.S. Standard Sieve Scale	W. S. Tyler, Inc.	20-325 Mesh
Gas sample collection		
Gas washing bottle	Fisher Scientific	3-038A

The appropriate corrections were made for fluid density, as described in Sect. E.2.

Temperature – All temperature measurements were made with 1/8 in. type K thermocouples that were sheathed in 304L stainless steel. The bed temperature was measured 2 cm above the distributor plate at the centerline of the bed.

Pressure – The pressure drop across the distributor plate and bed was measured directly with capacitance manometers. A mechanical pressure gage was used to measure the gage pressure in the header for the N_2, O_2, and CO_2 (so that appropriate corrections could be made to the gas flows measured by the rotameters). The absolute pressure in the lab was determined with a standard mercury barometer.

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2. R. H. Perry and C. H. Chilton, *Chemical Engineers' Handbook*, 5th ed., McGraw-Hill Book Company, New York, 1973, p 5-16.

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