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I am submitting herewith a thesis written by Randal E. Pudelek entitled "The Removal of Sulfur Dioxide from Flue Gas Using Fly Ash in a Spray Dryer/Fabric Filter System." I have examined the final copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Environmental Engineering.

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THE REMOVAL OF SULFUR DIOXIDE FROM FLUE GAS USING FLY ASH IN A SPRAY DRYER/FABRIC FILTER SYSTEM

A Thesis

Presented for the

Master of Science

Degree

The University of Tennessee, Knoxville

Randal E. Pudelek

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ABSTRACT

During the summer of 1981, an investigation on behalf of the Department of Energy was begun at The University of Tennessee, Knoxville. The purpose of this study was to investigate the reactivity of fly ash with SO_2 in the University's pilot plant spray dryer/fabric filter removal system.

The study commenced with the collection of 22 fly ashes (including lignite, subbituminous and bituminous eastern and western ashes) from the Gulf Coast, Rocky Mountains, and Northern Great Plains regions. These ashes were then used to investigate methods of enhancing SO_2 removal capability due to alkaline fly ash placed in a water based slurry and pumped to a spinning disk atomizer located on a slipstream from a stoker-fired boiler.

It was demonstrated that SO_2 removal efficiencies as high as 30 percent could be achieved by several of the fly ashes tested. It was also demonstrated by one fly ash that ball milling, prior to production of the slurry, increased the SO_2 removal efficiency from 18 to 46 percent. It is believed that the 62 percent increase in slurry alkalinity and the 17 percent increase in surface area for the milled ash caused this improvement in efficiency.

Using the concept of total sulfur retention as a function of surface area and slurry alkalinity, four modeling equations were produced. A complete model consisting of all the ashes collected yielded an R^2 of 0.82. In addition, separation of the ashes by rank,

i.e., bituminous, lignite, and subbituminous, yielded R^2 values of 0.96, 0.95, and 0.89, respectively.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

FIGURE PAGE

CHAPTER I

INTRODUCTION

There has developed increased interest in spray dryer usage as a viable economic and technical solution to flue gas desulfurization (FGD). This interest resulted from the Environmental Protection Agency's (EPA) passage of a new standard for sulfur dioxide (SO_2) emissions from fossil-fueled electric utility steam generating units for which construction began after September 18, 1978. This standard, which is based both on the heating value of the fuel as well as the sulfur content permits lower SO_2 removal rates (70 percent) for low-sulfur (less than 1 percent) coal combustion (1).

In the spray drying process, an alkali sorbent liquid is atomized in a spray dryer while reacting with SO_2 using flue gas as the drying medium. The FGD reactions proceed as a function of the drying time, yielding a suspended particulate in the gas stream from which the heavier fraction drops out into the hopper. The remaining reacted sorbent and fly ash proceed to a fabric filter collector (bag house) where further FGD reactions occur with the unreacted alkali and residual moisture in the filter dust cake.

One method used in the design of spray dryers incorporates the use of slaked lime as the alkali sorbent. However, the increased cost of lime has led to the investigation of other sorbent alternatives. One of these is high alkalinity fly ashes resulting from the combustion of low-sulfur western coals. Although fly ash has been

shown successful in removing SO_2, its reactivity potential varies from coal to coal. Consequently, the purpose of this research was to identify the potential reactivity of various types of fly ash and thus determine their capability for SO_2 removal. This knowledge would provide design engineers with the necessary data to determine whether the fly ash from a particular utility could be used as an effective supplement or substitute for slaked lime in the utility's spray dryer system.

CHAPTER II

LITERATURE REVIEW

The energy crisis facing the United States today has forced utilities to increase their capability to burn coal for energy as a solution to rising oil prices. However, as coal combustion increases, so do the levels of SO_2 which result from the virtually complete conversion of sulfur, a natural contaminant of coal, to SO_2 when the coal is burned. Stringent emission requirements have led to the development of various control technologies for SO_2 removal.

Prior to 1980 SO_2 removal was accomplished by absorption using a technique commonly referred to as wet scrubbing. Wet scrubbing utilizes a solution or slurry in a gas-liquid contacting system like a spray or packed tower to remove the SO_2 from the flue gas. In addition to the absorption equipment necessary, further investments are likely to be required for the sulfur to end up as a suitable waste product for a landfill (such criteria are specified by the Resource Recovery and Conservation Act).

The alternative to this is a process known as dry scrubbing or spray dryer absorption. Similar to wet scrubbing the acidic SO_2 gas is removed by contacting a suitable absorbing solution, generally a slurry of alkaline material. This solution is first pumped to an atomizer at a set revolutions per minute (rpm) thus governing droplet size within the spray dryer. As the droplets mix with the incoming flue gas, subsequent absorption and reaction occur resulting

in the conversion of SO_2 to sulfite and sulfate end products (2, 3). Typical solutions used in this process would incorporate, for example, lime or sodium carbonate as a reagent. The overall reaction of lime scrubbing is given by:

$$SO_2\ (g) + CaO\ (s) + 1/2\ H_2O \rightarrow CaSO_3 \cdot 1/2\ H_2O \qquad \text{(Eq. 1)}$$

The scrubbing with a solution of sodium carbonate produces a solution of dissolved sodium/sulfur salts as shown by:

$$Na_2CO_3 + SO_2 \rightarrow Na_2SO_3 + CO_2 \qquad \text{(Eq. 2)}$$

Following scrubbing, the sodium sulfite is oxidized yielding a neutral product of sodium sulfate (with no oxygen demand) which is then discarded (4).

$$Na_2SO_3 + 1/2\ O_2 \rightarrow Na_2SO_4 \qquad \text{(Eq. 3)}$$

Once the products of reaction have been formed, the heavier fraction separates from the flue gas stream by means of a hopper located at the bottom of the spray dryer. The remaining product-laden gas stream then proceeds to the particulate control device, generally a bag house, where additional interaction between the flue gas and the filter dust cake permits further SO_2 removal.

Other FGD removal systems have been developed for application on various industrial processes apart from utility coal combustion for steam generation. Examples of these are chemical, food, offal and fish industries (5). Wark and Warner (6) discuss 10 different processes which are divided into dry processes, and those operating either once through or regenerative, both of which are wet scrubbing processes (Table II-1). Jahnig and Shaw (7) discuss the design

Table II-1. Descriptions of sulfur dioxide removal processes (4).

Process Generics	Process Operations	Active Material	Key Sulfur Product
A. Throwaway scrubbing process			
1. Lime or limestone	Slurry scrubbing	CaO, $CaCO_3$	$CaSO_3/CaSO_4$
2. Sodium	Na_2SO_3 solution	Na_2CO_3	Na_2SO_4
3. Double alkali	Na_2SO_3 solution, regenerated by CaO or $CaCO_3$	$CaCO_3/Na_2SO_3$ or $CaO/NaOH$	$CaSO_3/CaSO_4$
4. Magnesium-promoted lime/limestone	$MgSO_3$ solution, regenerated by CaO or $CaCO_3$	$MgO/MgSO_4$	$CaSO_3/CaSO_4$
B. Regenerative scrubbing processes			
1. Magnesium oxide	$Mg(OH)_2$ slurry	MgO	15% SO_2
2. Sodium	Na_2SO_3 solution	Na_2SO_3	90% SO_2
3. Citrate	Sodium citrate solution	H_2S	Sulfur
4. Ammonia	Ammonia solution, conversion to SO_2	NH_4OH	Sulfur (99.9%)
C. Dry process			
1. Carbon adsorption	Adsorption at 400°K, reaction with H_2S to S, reaction with H_2 to H_2S	Activated carbon/H_2	Sulfur
2. Spray dryer	Absorption by sodium carbonate or slaked lime solutions	$Na_2CO_3/Ca(OH)_2$	Na_2SO_3/Na_2SO_4 or $CaSO_3/CaSO_4$

basis of each of these processes as applied to a modern 800 MW power plant burning Illinois #6 coal.

Advantages and Disadvantages of Dry vs. Wet Scrubbing

The conclusions of research conducted by Basin Electric Power Cooperative (8) and Radian Corporation (9), with regard to the overwhelming merits claimed by vendors of spray dryers, are in agreement. In general, dry FGD is considerably more attractive than wet scrubbing because the product formed by this process is dry and consequently easier to handle compared to a wet sludge. Also, depending on the application, there are potential capital and operating cost savings resulting from reduced equipment requirements, lower energy and water requirements, and relative process simplicity.

The product of dry scrubbers can typically be conveyed by processes analogous to those used for fly ash. This eliminates any special, maintenance intensive, sludge handling equipment. Contrarily, wet scrubbers need considerable equipment like centrifuges, thickeners, or vacuum filters to dewater the sludge rendering it suitable for a landfill.

There are other quantifiable advantages to spray dryer utilization in the area of operation and maintenance. The mode of gas-liquid contacting in wet scrubbers has presented problems of scaling and plugging across packing materials and demisters. The high pressure and volumes required to circulate the abrasive slurry in wet scrubbers has often times caused a considerable decrease in the operational life of pumping equipment. This is evidenced by

liquid-to-gas ratios (L/G) of 0.2 to 0.3 gallons per 1,000 actual cubic feet (acf) for dry systems to about 40-100 for wet scrubbers. Finally, the ability to adjust process operations to variations in the inlet SO_2 concentration and flue gas flow rates give dry scrubbers increased operational flexibility.

A limiting factor in spray dryer application is the large reagent requirements compared to those of wet systems (9). The stoichiometric ratio (SR), moles of sorbent required per mole of SO_2 entering, are greater for increased inlet concentrations which can limit their capabilities for use with high-sulfur coals. Parsons (10) emphasizes the importance of the approach to saturation at the spray dryer outlet in conjunction with SR. The amount of slurry of solution that can be sprayed into the incoming flue gas is limited by the temperature drop that exists between the spray dryer inlet and outlet. Based upon that of the inlet gas, a fixed temperature increment must be maintained above the adiabatic saturation temperature (dew point) to prevent a caustic attack or blinding of the filter bags located downstream. Although, increased reagent costs are a disadvantage, it is unlikely they will circumvent the 25 to 50 percent savings in energy consumption dry FGD systems yield when compared to wet systems.

Economics of Dry vs. Wet Scrubbing

An economic evaluation was conducted by Tennessee Valley Authority (TVA) (11) on flue gas compositions resulting from three different types of coal (Table II-2). Table II-3 gives typical

Table II-2. Coal composition (11).

Origin of Coal	Heating Value, BTU/lb.	Sulfur %	Ash %
I. Western	9700	0.7	9.7
II. Eastern	11700	0.7	16.0
III. Eastern	11700	3.5	16.0

Table II-3. Summary of estimated costs for SO_2 removal processes (11).

Coal Type / Process	Capital Investment M$	Capital Investment $/kW	Annual Revenue Requirements	Annual Revenue Requirements Mills/kWh
I. Low-Sulfur Western Coal				
Lime spray dryer	72-76	144-152	24-25	8.7-9.1
Limestone scrubbing	84-88	168-176	29-30	10.5-10.9
II. Low-Sulfur Eastern Coal				
Lime spray dryer	72-76	144-152	23-24	8.4-8.7
Limestone scrubbing	90-94	180-188	31-32	11.3-11.6
III. High-Sulfur Eastern Coal				
Lime spray dryer	90-94	180-188	40-41	14.5-14.9
Limestone scrubbing	118-122	236-244	45-46	16.4-16.7

Note: Ranges are used to quantify preliminary findings of TVA.

ranges for capital investments for the lime spray dryer process and the limestone scrubbing process. In overall capital investment the limestone scrubbing process is 16 to 31 percent higher than the lime spray dryer process. Of all the features considered in determining capital investment, like slurry preparation, handling costs, waste disposal, and SO_2 absorption, it is the latter that represents one-third of the direct costs in both wet and dry scrubbing. TVA found very slightly higher costs for wet solids separation (thickening and filtering) compared with dry solids handling (pneumatic conveying and solid storage) and slightly lower disposal costs because of the higher bulk density of the gypsum waste from the limestone process.

Also included in Table II-3 are the levelized annual revenue requirements. These levelized costs were first year costs adjusted for 6 percent per year inflation and discounting by 10 percent per year (cost of money) over the estimated 30-year life of the installation.

For the cases of western and eastern low-sulfur coals absorbent costs are minor when compared to maintenance costs. These costs for limestone scrubbing are more than double those of spray dryer processes.

For the high-sulfur eastern coal case the cost advantage of the lime spray dryer process is decreased. Absorbent costs total 24 percent of the annual revenue requirements for the spray dryer process; for the limestone scrubbing process only 3 percent. Generally, all other cost increases remain relatively constant for both processes.

Sorbent Potential of Fly Ash

The intent in the design of spray dryer systems is to meet the federal emission regulations for SO_2 while minimizing the stoichiometric ratio (SR) of reactants, i.e., sorbents.

Data reported by Hurst and Bielawski (12) on an 8,000 actual cubic feet per minute (acfm) spray dryer pilot unit describes the direct link that exists between SO_2 removal efficiency and SR. System efficiencies from 60 to 80 percent were obtained for ranges of SR between 0.8 and 1.2. The findings of other investigators (8, 13, 14) were consistent with these values. However, as SR increases, to produce greater SO_2 reduction, the costs of reagent increases causing a cost/benefit question to be asked of spray dryer manufacturers. Fly ash offers a potential answer to this question of economics by becoming a low cost supplement or even possibly an alternative to traditional sorbents.

Typical flame temperatures achieved by most utility boilers are near 2500° F (15). Meyer (16) has suggested that the sulfur retained by the fly ash is linked to its metal oxide content by the following reaction scheme:

$$MO\ (s) + SO_2\ (g) + 1/2\ O_2\ (g) \rightarrow MSO_4\ (s) \qquad \text{(Eq. 4)}$$

where M is the metal species. The steps involved are (1) adsorption of SO_2 onto the oxide surface and reaction to form the sulfite (M_2SO_3) and (2) further oxidation to the sulfate (MSO_4). The reaction rate is a function of temperature, gas concentration, metal oxide particle size, and surface properties.

Natusch (17) and Keyser (18) have suggested the surface enrichment of fly ash particles is due to volatilization-adsorption (or condensation) that occurs as the ash cools while exiting the high temperature combustion zone. Extraction with both water and dimethyl sulfoxide has shown levels of K, Fe, Na, and S to be higher in the surface layer, possibly as alkali iron sulfates, than in the core of the ash particle. This sulfate formation on the surface substantiates the theory that fly ash may be acting as a catalyst for the oxidation of SO_2.

Many researchers have shown increasing concentrations of sulfur on the surface in conjunction with decreasing particle size (17, 19, 20, 21). Such observations support a surface area-related phenomenon in addition to the chemical reaction between condensing species.

Rothenberg (22) has investigated the specific surface area (SSA) exhibited by fly ashes using nitrogen as an adsorbate and calculated by the method developed by Brunnauer, Emmet, and Teller. Ashes resulting from Stoker fired power plants showed greater SSA compared to pulverized coal power plants. Also a Montana subbituminous and a Texas lignite ash resulting from a fixed bed combustor exhibited values of SSA of 3.7 to 10.5 m^2/g for subbituminous and 12.8 to 20.4 m^2/g for lignite. Sulfur retention was slightly greater for the subbituminous ash due primarily to the fact that (1) the calcium content was 2.75 times greater and (2) fixed bed combustors operate at temperatures substantially less than the

fusion temperature of fly ash. This reduced operating temperature produces an ash with considerable pore volume, as shown by scanning electron microscope, thus permitting physical and/or chemical sorption of the calcium with sulfur.

Fiedler (23) indicated sulfur retention was linked to the metal oxide content of fly ash by analysis of up to 165 fly ash samples. The best coefficients (R^2) were observed for those fly ash samples burned in the same type of combustion system. Improvement in the R^2 value from 0.62 to 0.92 was found when mechanical collector data was removed. A possible explanation lies in the changes in particle size distribution that occur in mechanical collectors which may result in larger particles and less condensed sulfur present. The alkaline metal oxide components investigated by Fiedler (14) included CaO, Na_2O, MgO, and K_2O. The sulfur retained by fly ash had little correlation with the K_2O content of the ash. The magnitude of the data scatter eliminated any trends. The MgO content of the ash did show a contribution to sulfur retention, however, no improvement was found with the removal of mechanical collector data. The CaO and Na_2O content of the ash seemed to be the controlling factors in sulfur retention. Either one considered alone appeared to produce a reasonable correlation. However, the combination of these two metal oxides produced a dramatic improvement in data correlation. Figure II-1 is a plot of sulfur retention as a function of alkaline metal oxides (expressed as g CaO/100 g fly ash) for all data points. Figure II-2 contains only the pulverized

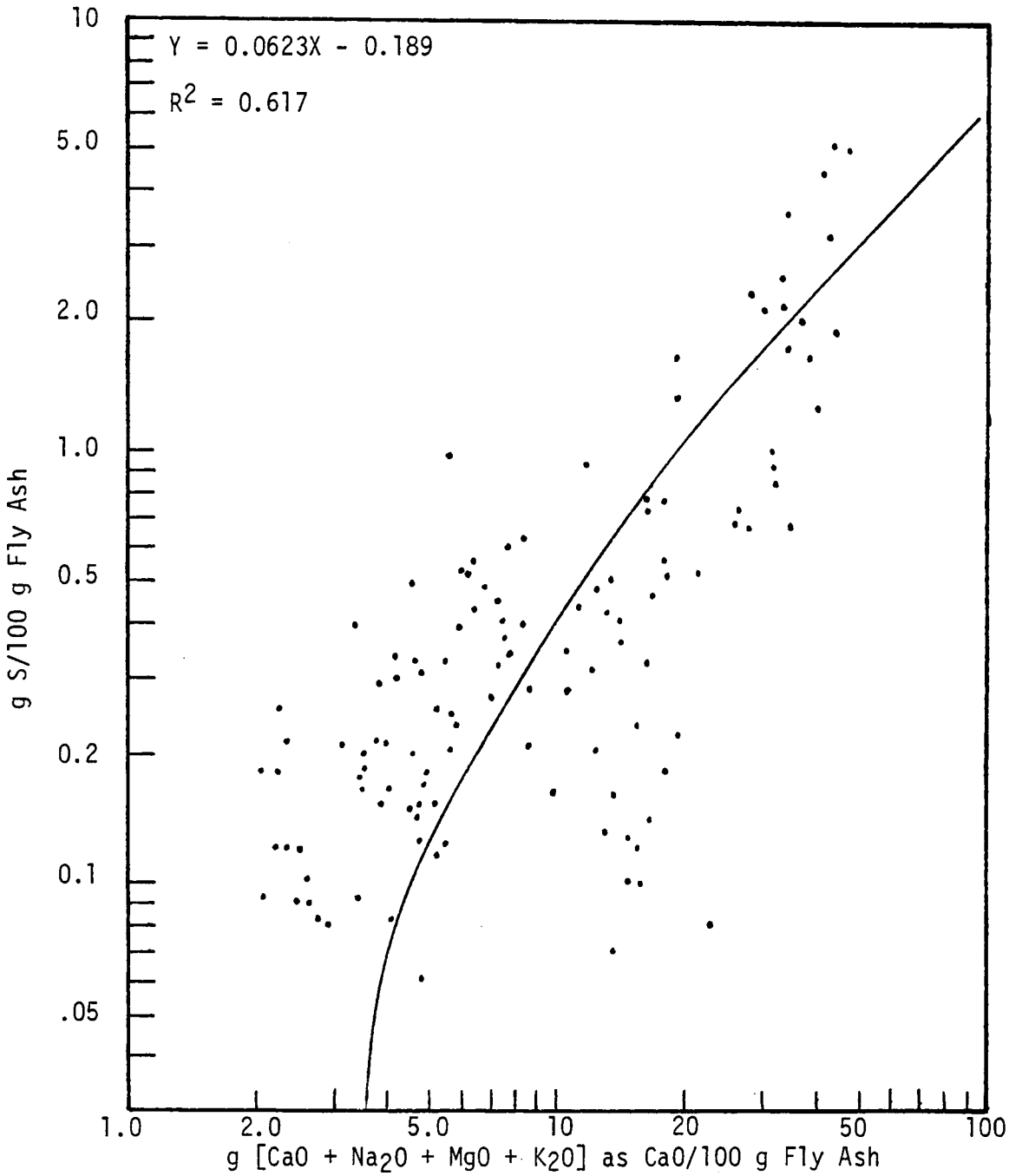

Figure II-1. Grams sulfur retained vs. weighted grams alkaline metal oxides expressed as CaO per 100 grams fly ash (23).

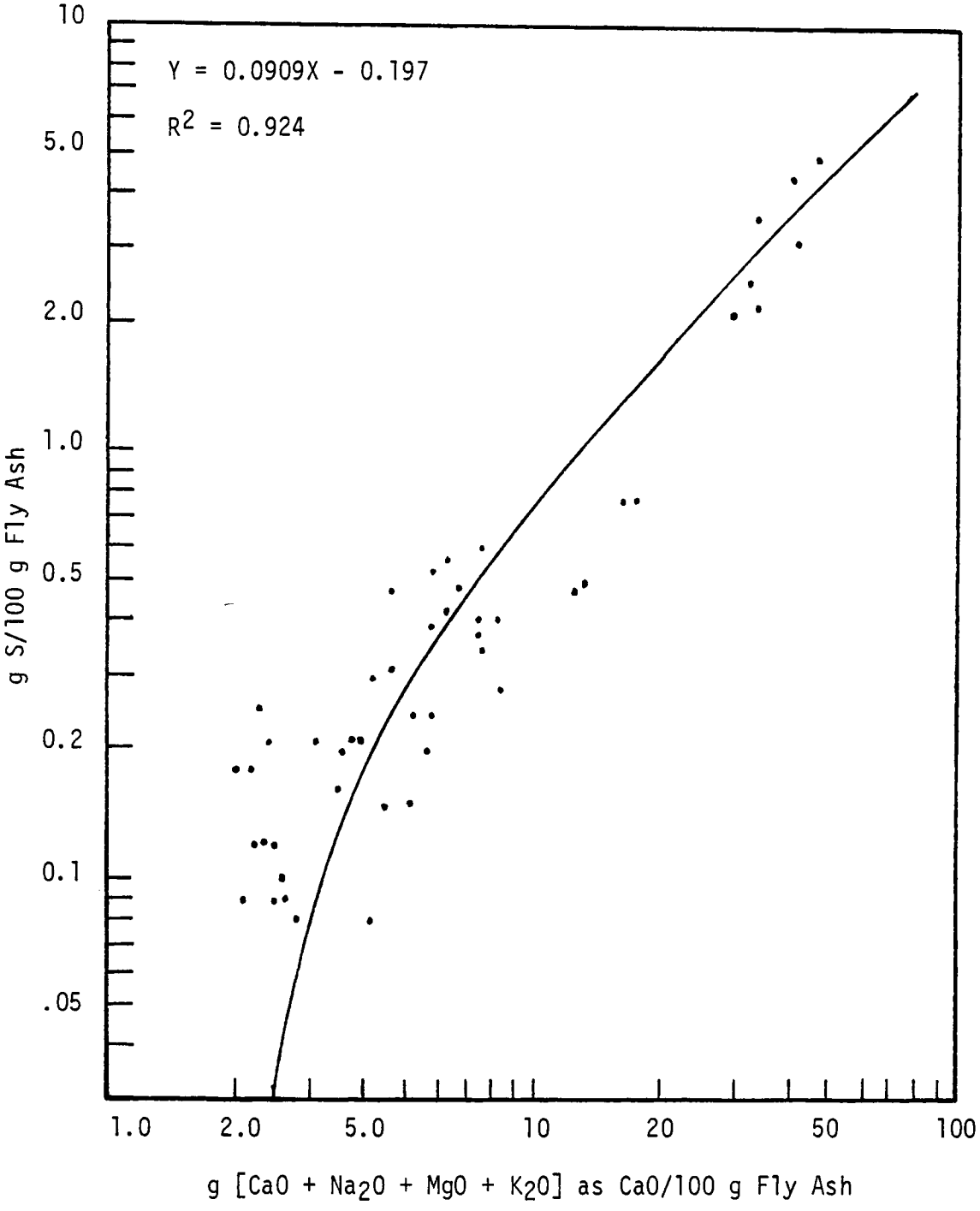

Figure II-2. Sulfur retained vs. alkaline metal oxide content for pulverized coal-fired boilers.

coal-fired fly ashes. The first and second order linear equations developed by Fiedler (23) are:

$$\%S \text{ retained} = 9.09 \times 10^{-2} X - 0.197 \qquad R^2 = 0.924 \quad \text{(Eq. 5)}$$

$$\%S \text{ retained} = 1.72 \times 10^{-3} X^2 + 1.77 \times 10^{-2} X + 0.127$$

$$R^2 = 0.958 \quad \text{(Eq. 6)}$$

The fly ashes examined by Fiedler (23) showed the level of alkalinity to vary from 2-40 g as CaO/100 g fly ash. These data suggest that certain ashes still have the potential for even greater sulfur retention.

Ness and Selle (24) have studied the effect of leaching calcium oxide from North Dakota lignite fly ashes. Utilizing a fly ash-water slurry in batch leach tests indicated an increase in the amount of calcium oxide in solution as pH was decreased. Available calcium oxide in the slurry ranged from 10 to 40 percent at pH 7 for three different ashes (Figure II-3). Diversity in chemical composition and combustion of the original coal are the most plausible explanation for this wide range.

This ability to produce significant levels of alkalinity in a fly ash slurry suggests potential application for SO_2 removal in spray dryer/fabric filter systems. It has been shown by Davis (25) that SO_2 removal efficiencies of 10-30 percent across a fabric filter can be achieved due to the presence of a fly ash cake on the filter.

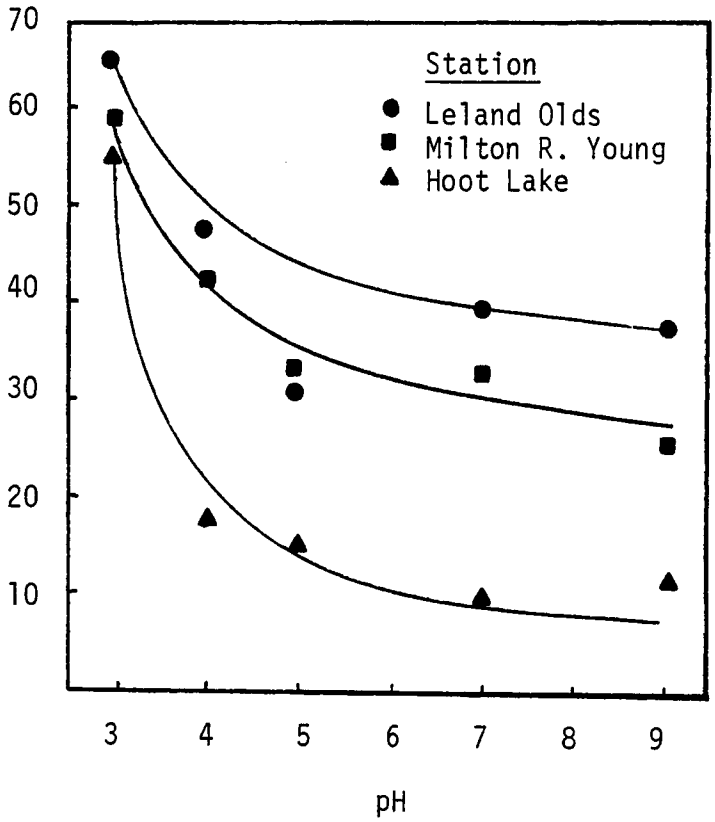

Figure II-3. Effect of pH on calcium oxide availability for three Western lignite fly ashes (24).

CHAPTER III

EXPERIMENTAL DESIGN

Approach

The purpose of this research effort, as stated earlier was to identify the potential reactivity of various types of fly ash and thus determine their capability for SO_2 removal. The ashes tested originated from three different rank coals: bituminous, subbituminous, and lignite. Significant variability in physical and chemical characteristics was insured by obtaining the ashes from different coal-bearing regions in the United States (Table III-1).

Description of the Test Facility

The spray dryer/fabric filter pilot plant was installed on a Riley spreader-stoker coal-fired boiler at The University of Tennessee, Knoxville, steam plant. A slipstream of flue gas with a nominal flow of 1,000 acfm, taken from the main ductwork, enters the system which consists of a spray dryer with a spinning disc atomizer followed by a fabric filter collector. The slipstream is pulled through the pilot system by an induced-draft fan. A layout of the system is shown in Figure III-1.

The ductwork leading to the spray dryer contains ports for the injection of ambient air and SO_2. Other ports labeled one, two and three, respectively, enable the monitoring of temperature, static pressure, and SO_2 concentration (Figure III-2).

Table III-1. Origin of the fly ashes investigated.

Fly Ash I.D. No.[a]	Power Plant of Origin	Type of Fuel (Approximate)	Source
6	Belews Creek Steam Station	bituminous	Low sulfur eastern coal
10[b]	Bowen Steam Plant	bituminous	eastern Kentucky
13	Clay Boswell Unit 4	subbituminous	Big Sky Mine - Colstrip Montana
14	Nebraska Public Power Station Gerald Gentleman Station	subbituminous	Black Thunder Mine Campbell County, Wyoming
18A	Pacific Power and Light Wyodak Plant	subbituminous	Wyodak Resources - Wyodak Mine
18B	Hunter Steam Plant	bituminous	Wilberg Mine - Emery County, Utah
23B	Cherokee #4	bituminous	Colorado western slope
24	Gallagher Station II Public Service of Indiana	bituminous	Amax Aryshire
27	Texas Utilities Generating Co. Big Brown	lignite	Freestone County
34	New Madrid Power Plant #2	bituminous	Southern Illinois, Seam #6
36	Ohio Edison - Gorge	bituminous	Ohio strip mine
37	Harrington Station: Southwestern Public Service Co.	subbituminous	Black Thunder Mine near Gillette, Wyoming
38B	Laramie River Station	subbituminous	Cordero Mine
38C	Basin Electric Power Cooperative - Unit 1	lignite	Consolidated Coal Co. Stanton, North Dakota
41	Milton R. Young Station Center Unit 1	lignite	Bankol-Noonan Mine Center, North Dakota
42	United Power Assoc. Cooperative Power Assoc.	lignite	Falkirk Mine Underwood, North Dakota
43	Black Hills Power and Light Company	subbituminous	Wyodak Mine - Wyoming
44	Otter Tail Power Hoot Lake Station Unit #2	lignite	Knife River Coal Mining Co. Beulah, North Dakota
45[b]	Minnesota Power and Light Co. Clay Boswell Station	subbituminous	Big Sky Mine Colstrip, Montana
46	Monifer Resources San Antonio Public Service	subbituminous	Cordero Mine, Wyoming
49	University of Tennessee Steam Plant	bituminous	eastern Kentucky
50	Marshall Steam Station	bituminous	low sulfur eastern coal

[a]I.D. No. = Identification number.

[b]These fly ashes were obtained from the same source, however, their production resulted from different combustion conditions.

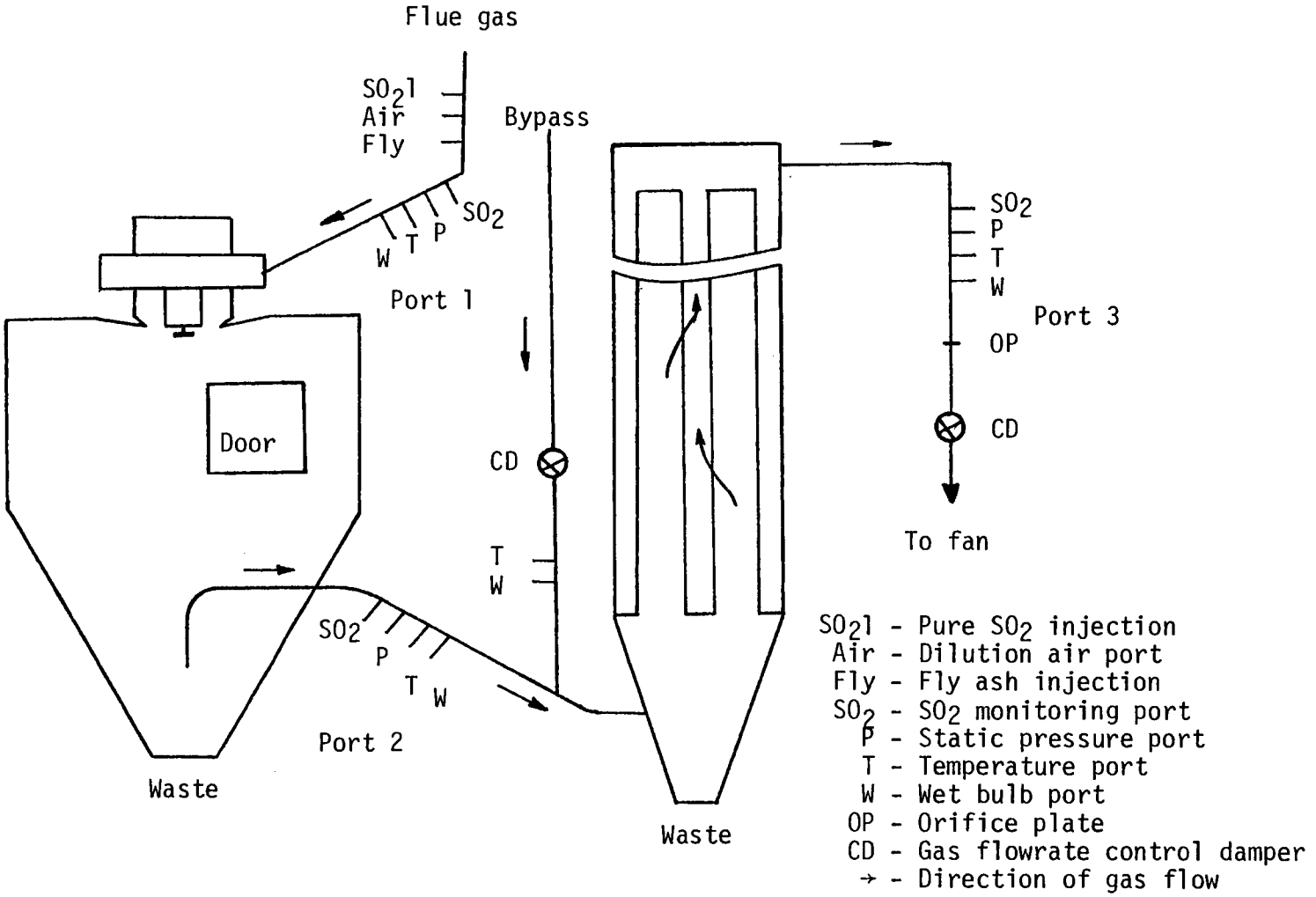

Figure III-1. Pilot plant schematic.

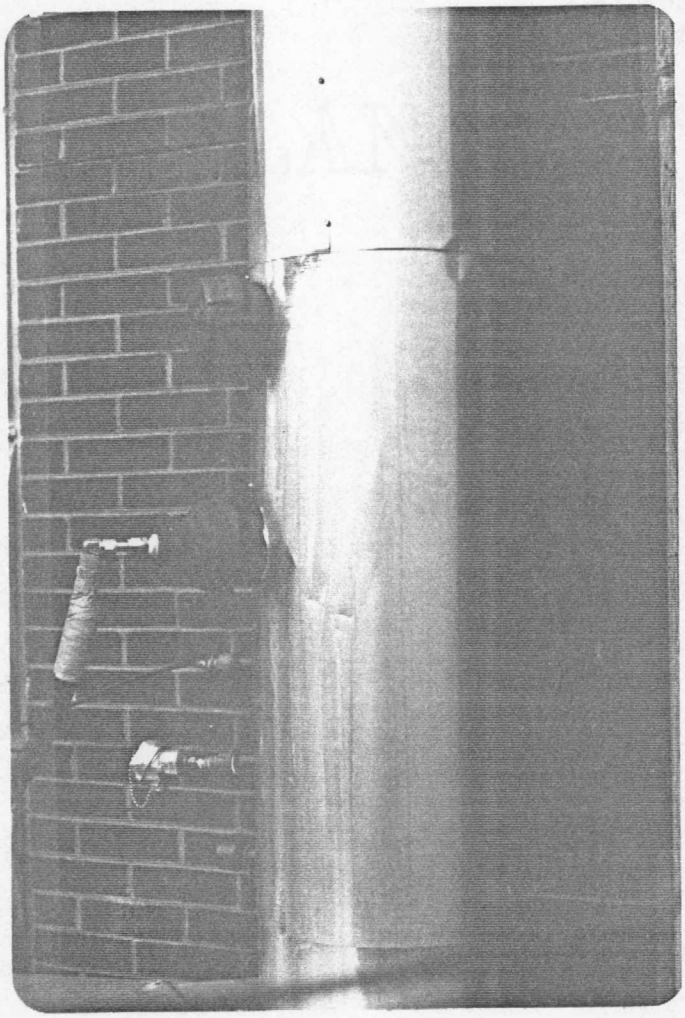

Figure III-2. Port I--Inlet to spray dryer.

Once the desired inlet SO_2 concentration and temperature is achieved the flue gas then proceeds to the spray dryer inlet where it encounters the atomization machinery. Atomization is accomplished by a 13.5 KVA solid state frequency inverter, a Stork-Bowen high speed AA-6 Spray Machine and a centrifugal atomizer. The frequency inverter steps up the "house current" from 60 hertz to the required frequency and delivers this power to the spray machine (Figure III-3). Attached to the spray machine is a specially designed dynamically balanced atomizer (Figure III-4). The fly ash and water slurry passes from the pumping system through the spray machine housing into the atomizer. Rotating at a high speed, the disc atomizes and distributes the slurry as a uniform, fine mist into the hot flue gas entering the drying chamber.

Flue gas exiting the boiler enters the drying chamber through a set of vanes concentric with the spray machine (Figure III-5). These vanes impart an angular downward swirling motion to the air. The rotation of the swirling air is opposite to that of the feed leaving the atomizer thus insuring intimate mixing of the hot air and the fine particulate mist.

The drying chamber is a 7 foot diameter vessel with a standard conical bottom which contains a rotary valve for the removal of the spray dryer product (Figure III-6).

The particulate-laden gas stream upon leaving the spray dryer enters the fabric filter collector. This collector removes from the flue gas both the unsettled fly ash-SO_2 product and any other type

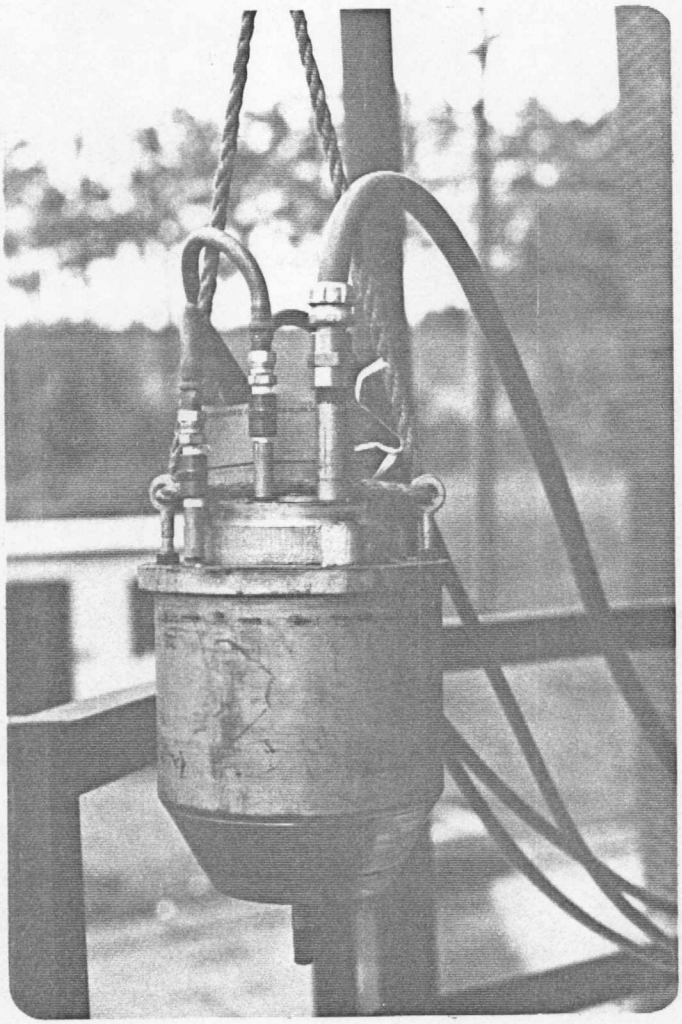

Figure III-3. Stork-Bowen spray machine.

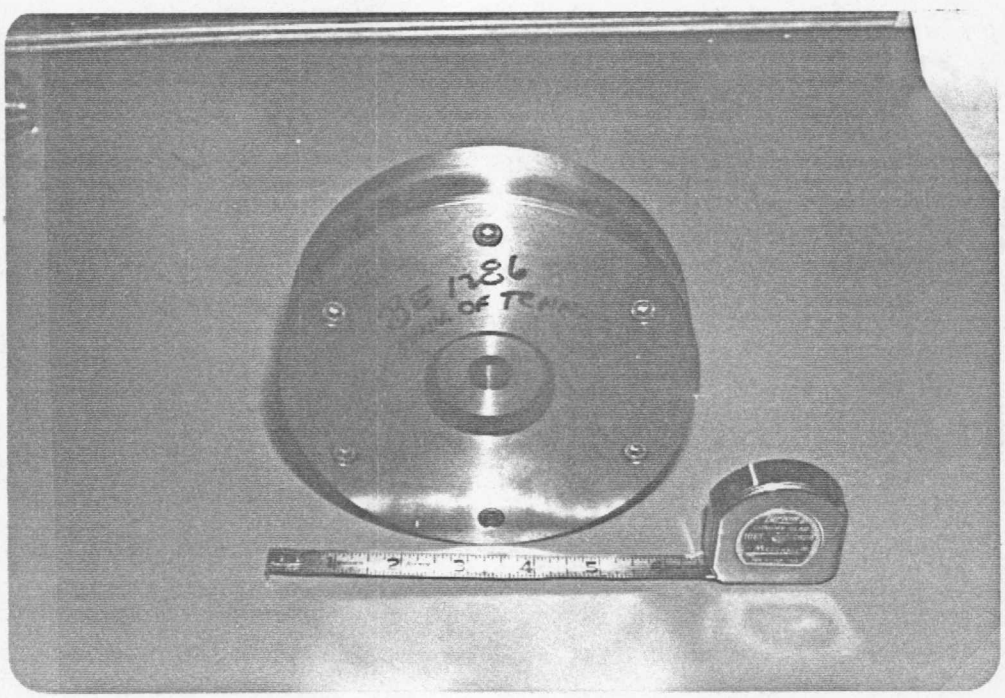

Figure III-4. Centrifugal atomizer.

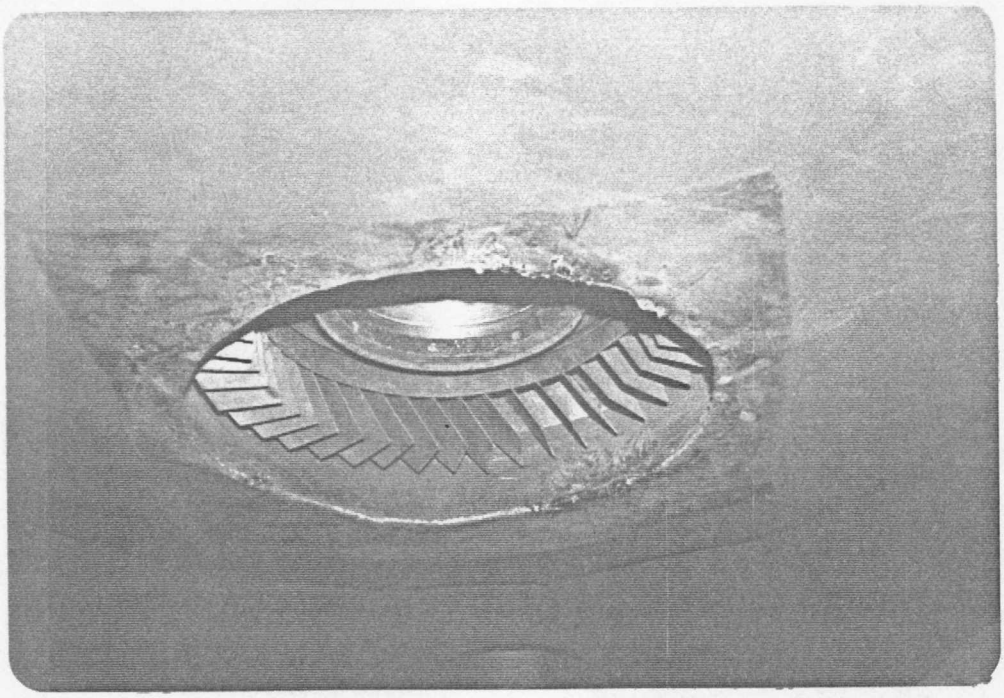

Figure III-5. Spray dryer inlet vanes.

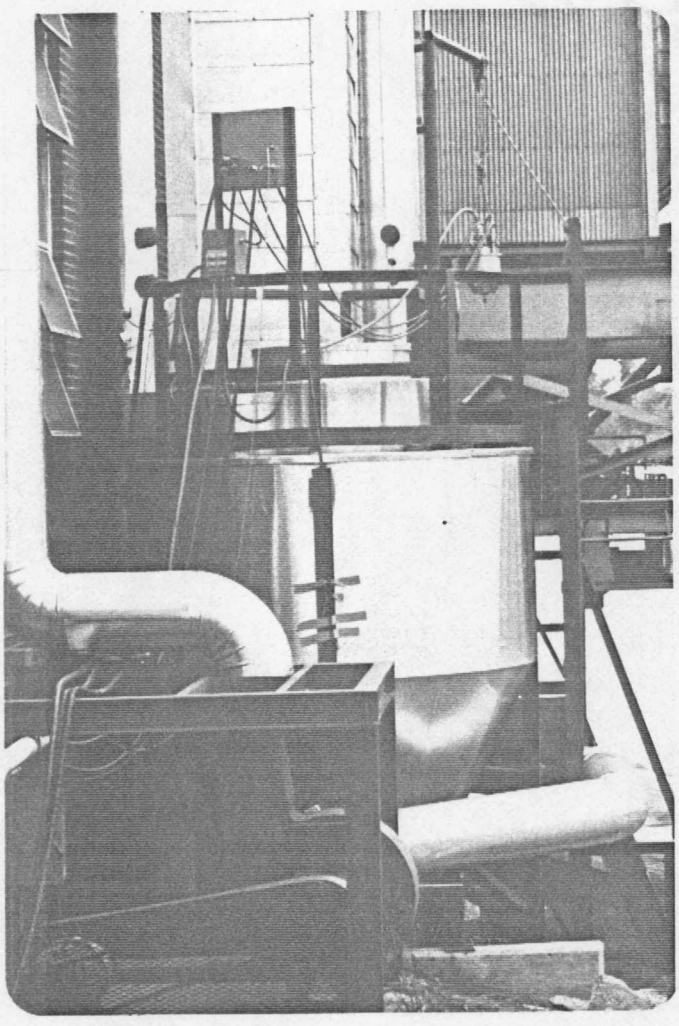

Figure III-6. Spray dryer unit.

boiler ash contained in the stream. The bag house consists of one filtration compartment containing four 32 ft x 12 inch fiberglass bags (14.5 oz/sq yd). A low energy shaker mechanism is used to clean the bags. It is operated manually at the completion or prior to a test. While in the automatic mode which is used at the end of the day, cleaning occurs once per hour. A rotary valve located beneath the hopper facilitates the removal of the settled out dust (see Figure III-7 for photograph of complete system).

The operation of the spray dryer/bag house facility is from a central control room located about 40 feet from the facility (Figure III-8). The control room contains a panel for the control of atomizer rpm and other devices for the monitoring of the system flow rate and slurry feed rate. Also, monitored in the control room are the following:

1. SO_2 concentration (3 ports) Lear-Siegler SM800
2. Temperature (thermocouples)
    a. Inlet/outlet spray dryer
    b. Inlet bag house
    c. Ambient temperature
    d. Slurry feed temperature
3. Slurry feed rate
4. Concentration of additive (i.e., fly ash percent)
5. Static pressure inlet/outlet of spray dryer/bag house

When the desired range of SO_2 concentration is not achieved, a manually controlled supplemental SO_2 injection system is used.

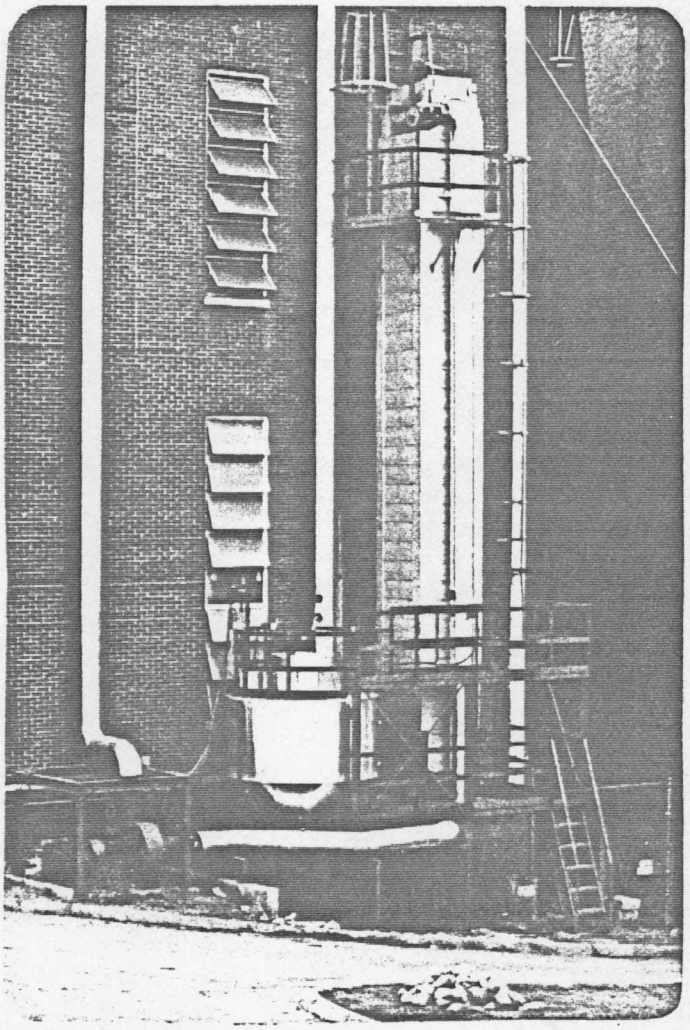

Figure III-7. Spray dryer/fabric filter pilot facility.

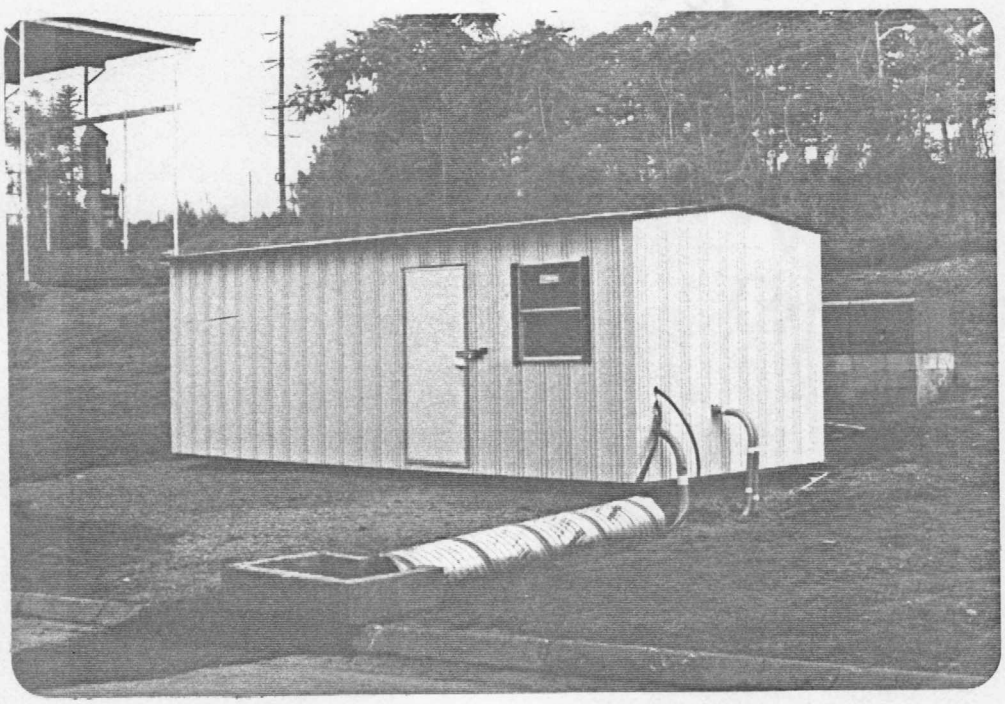

Figure III-8. Control room.

This system is located about 80 feet upstream of the spray dryer to allow for sufficient mixing.

Inlet temperature control is maintained by a manually operated ambient air dilution damper located upstream of the SO_2 injection port.

Monitoring of SO_2 is accomplished by extracting samples through heated trace lines into the control room where the flue gas sample is drawn through the sampling cell of a Lear-Siegler SM800. The concentration of SO_2 is measured on a wet basis by this instrument. A dry basis concentration is calculated later after the moisture content is determined.

The shed housing the control room (10 ft x 24 ft) is divided into two equal sections, one section housing the office/control room, the other housing the feed mixing tanks and transfer tank. Figure II-9 shows the three tanks, however, only the two on the right were necessary for the preparation of the fly ash and water slurry. Figure II-10 is a photograph of the final transfer tank.

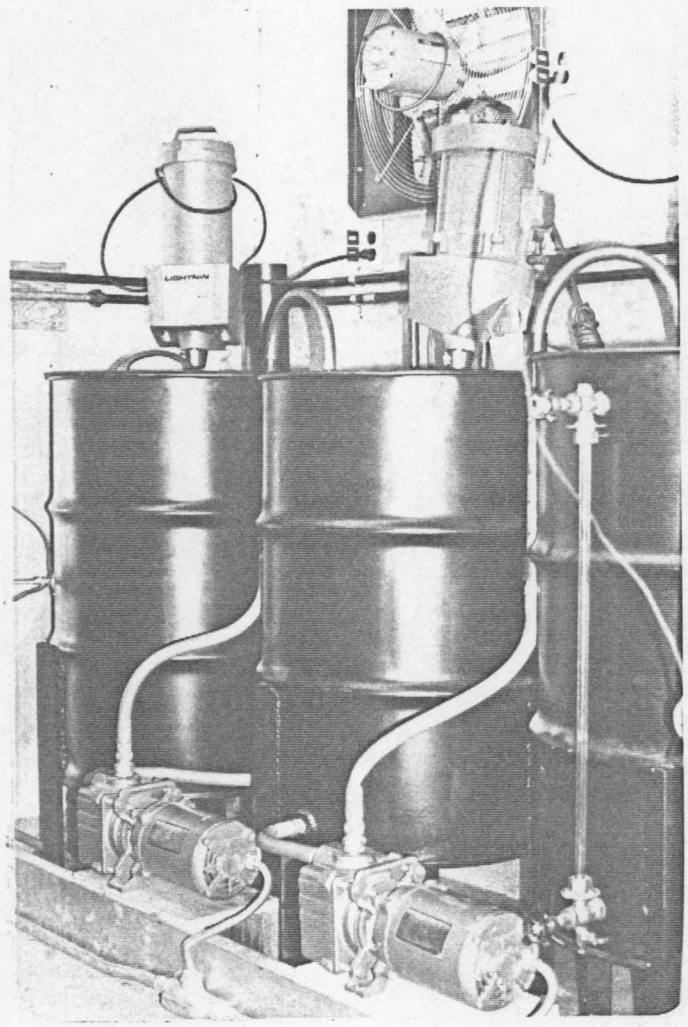

Figure III-9. Slurry preparation tanks.

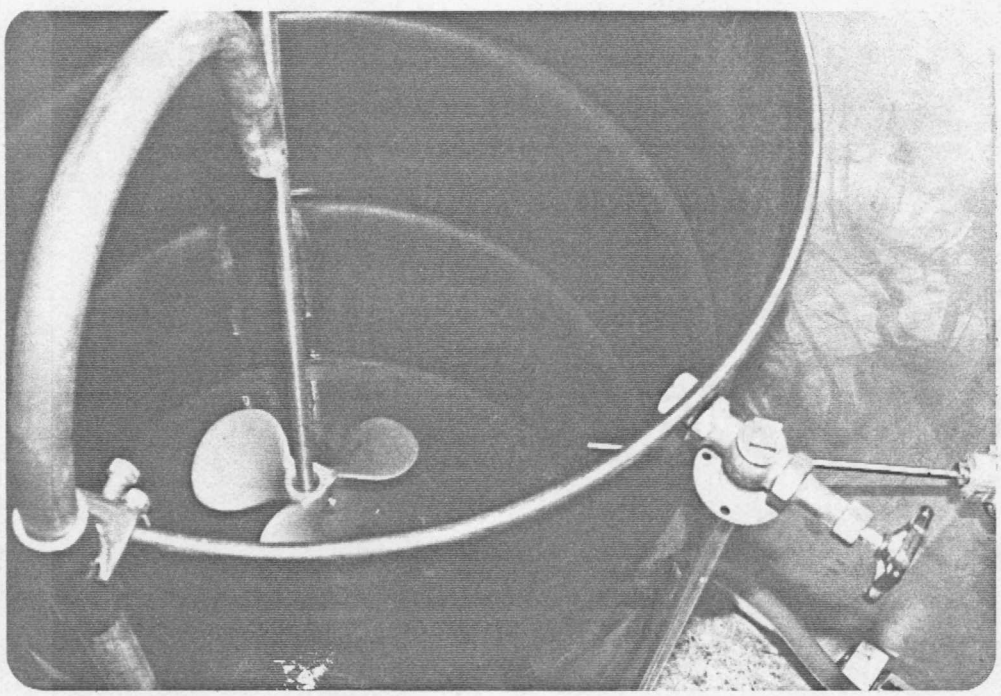

Figure III-10. Final transfer tank.

CHAPTER IV

RESULTS AND DISCUSSION

Results of Laboratory Analyses

Each fly ash was analyzed, prior to testing at the spray dryer/fabric filter facility, to determine both its physical and chemical characteristics. Table IV-1 shows the properties measured and the instruments that were used. A summary of these results can be found in Table IV-2. Chemical analyses conducted for total alkalinity, calcium and sodium content, pH, and total hardness yielded information regarding the behavior of fly ash in a water based slurry (26). These analyses were conducted at a temperature of 140°F and a fly ash concentration of 1.25 pounds per gallon of water. These conditions were chosen in order to simulate slurry preparation at the spray dryer facility. Table IV-3 contains the results of this testing for each of the fly ashes.

In addition, laboratory preparation of the fly ashes, according to ASTM procedures (27), followed by analysis with an atomic absorption spectrometer determined the levels of calcium oxide (CaO), sodium oxide (Na_2O), magnesium oxide (MgO), and potassium oxide (K_2O). An estimate of total available alkaline metal oxides (TAMO-S) was then determined by a weighting scheme (see Equation 7). The metal oxides were summed on a molar basis less the amount that had reacted with sulfur after combustion. This sulfur

Table IV-1. Fly ash characteristics.

Physical Property	Measuring Instrument[a]
Particle Size Distribution	Coulter Counter Model TaII
Particle Density	Micromeritics Model 1302 Helium-Air Pycnometer
Sulfur Content	Fisher Total Sulfur Analyzer
Surface Area	Micromeritics Surface Area Analyzer

[a]A brief description of each instrument can be found in Appendix A.

Table IV-2. Fly ash size and density data summary.

Fly Ash I.D. No.	MMD[a]	Geometric Deviation	Particle Density, g/cm^3	Specific Surface Area, m^2/g
6	12.0	2.3	2.7	1.31
10	14.8	2.2	2.1	2.14
13	9.4	1.9	1.5	0.21
14	9.5	1.8	2.7	2.09
18A	15.0	1.9	2.5	0.73
18B	7.8	1.9	2.3	2.14
23B	14.9	2.8	2.2	2.85
24	12.3	2.2	2.7	2.88
27	9.5	2.5	2.4	0.74
34	9.2	1.7	2.5	1.23
36	15.3	2.0	2.6	0.22
37	7.4	2.5	2.7	1.15
38B	11.5	2.2	2.6	1.66
38C	15.6	2.6	3.2	0.76
41	12.0	1.8	2.7	3.27
42	9.4	1.9	2.6	0.29
43	18.0	2.4	2.5	1.03
44	11.0	1.9	2.7	2.37
45	10.0	2.0	2.5	3.39
46	8.5	1.8	2.5	1.48
49	12.9	1.9	2.1	12.52
50	14.0	2.1	2.2	1.47

[a]MMD = Mass mean diameter, micrometers (10^{-6} meters).

Table IV-3. Fly ash slurry data summary.[a]

I.D. No.	pH	Alkalinity,[b] mg/l as $CaCO_3$	Ca, mg/l	Na. mg/l	Total Hardness, mg/l as Ca
6	5.2	200	472	40	547
10	6.7	500	265	26	
13	11.2	3800	962	48	1094
14	11.0	2500	132	200	210
18A	10.6	1200	130	54	160
18B	11.2	3400	849	88	1000
23B	11.3	1800	264	51	340
24	11.4	2900	1151	91	1208
27	10.3	1700	415	300	547
34	4.4	0	604	300	717
36	10.5	1400	245	49	283
37	11.3	1800	85	200	170
38B	11.2	1500	123	54	245
38C	10.8	1600	274	4200	368
41	10.7	1100	321	8000	396
42	11.4	3700	887	62	981
43	10.9	2100	179	75	274
44	10.9	1300	340	8200	396
44M[c]	10.9	2100	302	8500	472
45	10.7	1600	302	58	396
46	10.9	1600	128	100	170
49	3.6	0	175	150	
50	4.8	0	321	52	358

[a]The slurry parameters shown in this table were obtained by analysis of the filtered supernatant.

[b]Alkalinity is an indirect estimate of the soluble cations by measurement of the hydroxide (OH), carbonate (CO_3), and bicarbonate (HCO_3) species.

[c]This ash was obtained by grinding fly ash number 44 in a ball mill.

retained in either sulfite or sulfate forms was measured, upon receipt of the ash, by a Fisher Total Sulfur Analyzer (see Appendix A).

$$\text{TAMO-S, g moles/100 g} = \frac{\text{g CaO/100 g}}{56.1\ \text{g/g mole}} + \frac{\text{g } Na_2O\text{/100 g}}{62.0\ \text{g/g mole}} + \frac{\text{g MgO/100 g}}{40.3\ \text{g/g mole}} + \frac{\text{g } K_2O\text{/100 g}}{94.2\ \text{g/g mole}} - \frac{\text{g S/100 g}}{32.1\ \text{g/g mole}} \quad \text{(Eq. 7)}$$

This approach is similar to those used by References (12, 23) for predicting the effect of fly ash alkalinity on SO_2 removal and also sulfur retention in fly ash. Table IV-4 contains both the percent of each compound in the ash (wet basis) and the estimate of total alkalinity for each fly ash investigated at the pilot plant.

Fly ash testing, at the spray dryer facility, was initiated by the production of a slurry in the mixing tanks described earlier. This was accomplished by mixing a predetermined mass of fly ash into a known quantity of water with sufficient time (greater than 30 minutes) to allow the soluble metal oxides to disperse within the slurry. Figures IV-1 and IV-2 are typical examples of the dissolution kinetics exhibited by fly ash. Such information was used to determine the required duration of mixing and the effect of slurry preparation temperature. After mixing, the slurry was pumped to the final dilution tank where it awaited final transfer into the spray dryer. Prior to and during the slurry production procedure, the following conditions were maintained for all fly ashes tested at the spray dryer/fabric filter facility:

Table IV-4. Fly ash mineral analysis summary.

Fly Ash I.D. No.	%S	%CaO	%Na_2O	%MgO	%K_2O	TAMO-S[a]
6	0.43	7.79	10.71	0.97	2.53	0.35
10	0.32	5.44	9.42	0.91	2.36	0.29
13	0.49	13.96	10.96	2.79	1.79	0.50
14	0.73	22.91	10.96	3.49	1.29	0.65
18A	0.70	18.96	10.78	3.79	1.50	0.60
18B	0.45	9.72	10.89	2.14	1.94	0.41
23B	1.87	7.88	10.25	1.18	1.68	0.35
24	1.22	8.60	9.94	1.91	0.76	0.31
27	0.66	14.99	11.19	2.40	1.70	0.50
34	1.04	6.76	11.79	0.97	2.90	0.33
36	0.36	0.98	12.69	0.39	5.56	0.28
37	0.78	24.95	10.98	3.59	1.00	0.70
38B	0.72	21.81	13.48	2.97	1.29	0.67
38C	0.00	13.95	35.88	5.58	3.69	1.01
41	4.06	18.01	16.68	3.98	1.52	0.58
42	1.02	18.96	10.97	3.79	1.90	0.60
43	0.66	6.97	10.76	1.00	2.49	0.33
44	0.75	23.80	17.71	4.57	1.14	0.81
44M	0.75	23.80	17.71	4.57	1.14	0.81
45	0.68	11.63	10.66	2.71	1.45	0.44
46	0.75	28.90	14.94	3.98	1.39	0.84
49	2.19	4.02	6.97	0.94	1.94	0.16
50	0.41	5.75	12.47	1.44	2.78	0.36

[a]TAMO-S can be obtained from (Eq. 7) whereas total alkaline metal oxides are solely a measure of the CaO, Na_2O, MgO, and K_2O content of the ash.

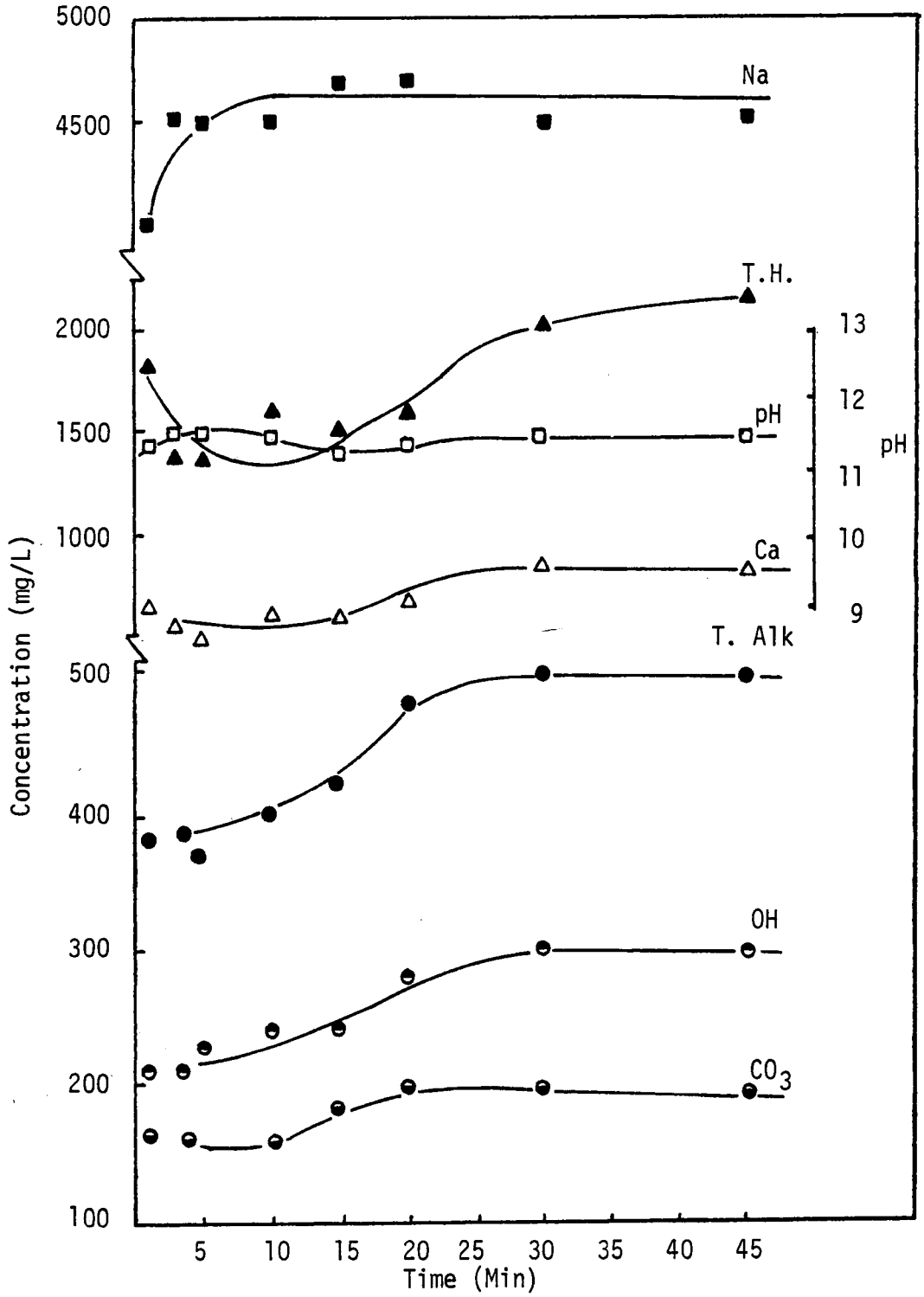

Figure IV-1. Dissolution kinetics of Hoot Lake fly ash (44) in distilled water at 66°F.

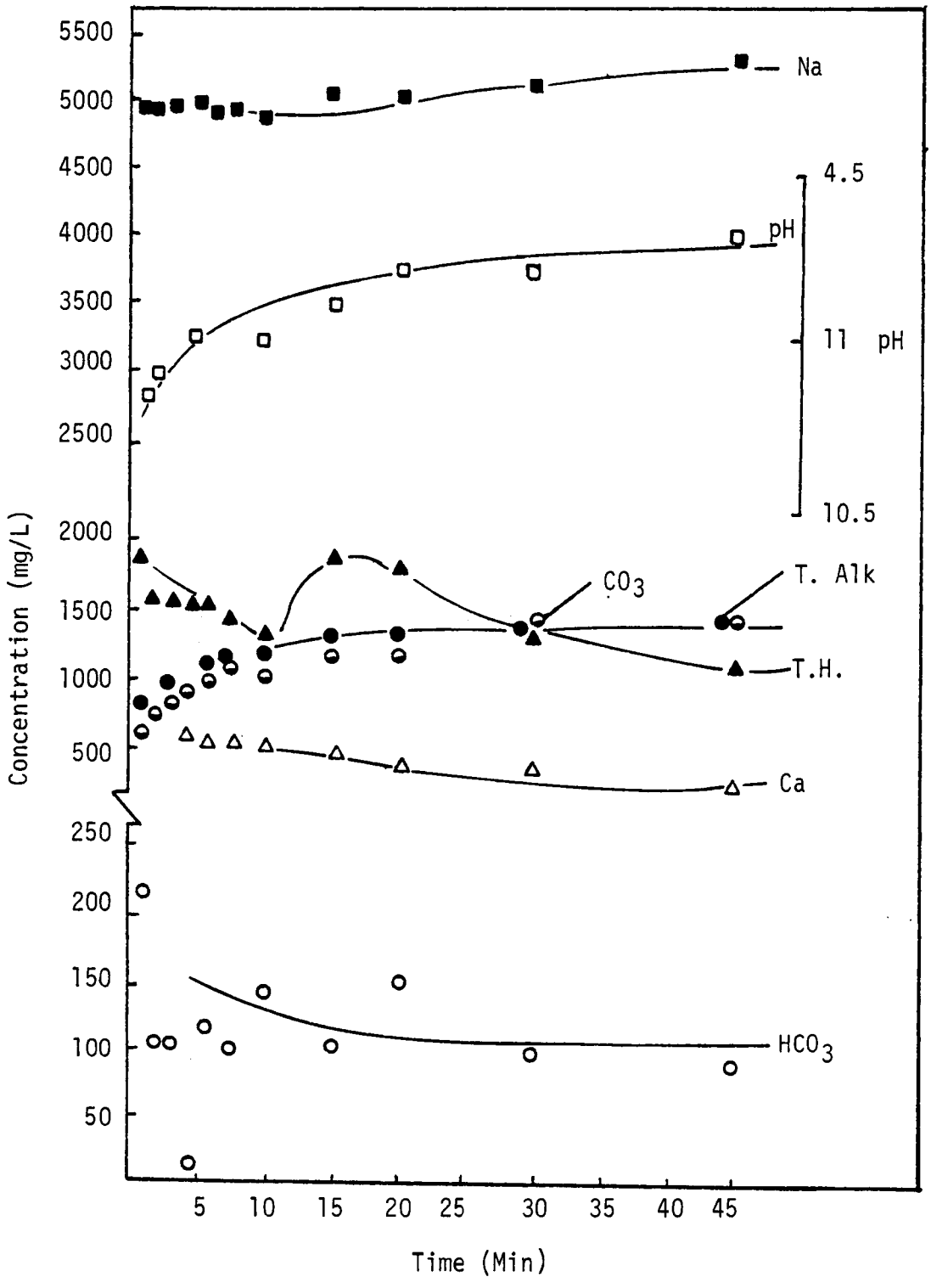

Figure IV-2. Dissolution kinetics of Hoot Lake fly ash (44) in distilled water at 165°F.

1. Inlet temperature range (300 ± 10°F)
2. Inlet SO_2 concentration (600 - 800 ppm)
3. Gas volumetric flow rate (1,000 acfm)
4. Slurry preparation temperature (130 - 140°F)
5. Slurry concentration (1.45 lbs fly ash/gallon H_2O)
6. Temperature at port two (20°F above dew point)
7. Atomizer disc speed (17,000 rpm)

Results of Spray Dryer/Fabric Filter Testing

The total amount of SO_2 removed by the system, spray dryer plus bag house, was determined from inlet and outlet mass flow rates of SO_2 by the following relationship:

$$\frac{MSO_2(1) - MSO_2(3)}{64.04 \text{ lbs/lb mole}} \times \frac{453.6 \text{ g}}{\text{lb}} \times \frac{\text{min}}{60 \text{ sec}} \qquad \text{(Eq. 8)}$$

$$= \frac{\text{g moles } SO_2 \text{ removed}}{\text{sec}}$$

where

MSO_2 (1) = mass flow SO_2 at port 1 (lbs/min)

MSO_2 (3) = mass flow SO_2 at port 3 (lbs/min)

By defining the quantity of SO_2 removed in this manner, the existence of any significant correlation with alkaline metal oxide content of the ash (also expressed in g moles/sec, see calculations in Appendix B) could be identified.

The percent efficiency of SO_2 removal was determined by Equation 9. Concentration data were obtained as follows: (1) the inlet value was measured by the analyzer, (2) an average concentration

at the outlet was achieved by allowing the analyzer to purge to a stable value and re-enumerate the level of SO_2 in the gas stream, (3) the inlet was verified with the value obtained in Step (1) and if necessary averaged. The inlet and outlet concentrations were measured at ports one, two, and three, located as shown in the schematic in Figure III-1, page 20.

$$\% \ SO_2 \text{ efficiency} = \left(1 - \frac{C_{out}}{C_{in}}\right) x \ 100\% \qquad \text{(Eq. 9)}$$

where

C_{out} = outlet gas concentration (ppm)*

C_{in} = inlet gas concentration (ppm)*

Table IV-5 contains the efficiency data for both the spray dryer and bag house in addition to the net SO_2 removal achieved by the complete system.

Two important checks were conducted on the system during the course of this investigation to minimize the errors involved. First, SO_2 balances were conducted at test conditions by measuring concentrations at all three ports without any fly ash injection. A 4 percent reduction in the level of SO_2 was observed at the system's outlet and was thus accounted for in all calculations. Secondly, water injection (no fly ash), via the spinning disk atomizer, produced a 2 percent SO_2 efficiency which was subtracted from both the total system efficiency and the molar SO_2 removal rate.

Finally, statistical analyses performed on both the laboratory and the spray dryer data were done using the statistical analysis

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*On a dry basis (see Appendix B).

Table IV-5. SO_2 removal efficiency by a fly ash slurry.

Fly Ash I.D. No.	EFFSD	EFFBH	EFFSYS
6	0	4	1
10	10	-2	7
13	5	14	17
14	33	-2	30
18A	20	2	21
18B	24	-1	23
23B	2	0	2
24	5	1	6
27	1	4	5
34	4	2	6
36	0	-1	0
37	25	1	26
38B	17	11	26
38C	11	5	15
41	17	5	21
42	8	1	9
43	21	6	26
44	15	1	16
44M	15	38	46
45	11	15	24
46	12	1	13
49	11	21	29
50	0	1	1

[a]EFFSD = Efficiency of spray dryer.

[b]EFFBH = Efficiency of bag house.

[c]EFFSYS = Efficiency of system corrected for water effect.

system developed by SAS Inc. (28). The routines used included PLOT (a data plotting routine), RSQUARE (an all possible regressions routine) and GLM (a general linear models procedure). The calculations performed on both sets of data to obtain the desired units for statistical correlation purposes are detailed in Appendix B.

Effect of Fly Ash Properties on SO_2 Removal

The ability of fly ash to remove SO_2 in a spray dryer system was confirmed by the percent efficiencies shown in Table IV-5. However, attempts to develop linear relationships between SO_2 removal, in g moles/sec, and the laboratory data obtained on each ash, also expressed in g moles/sec, yielded extremely poor correlations. Table IV-6 contains the correlation coefficients, R^2, for the first order models based on both fly ash and fly ash slurry analyses. Figure IV-3 shows the inability of total available alkaline metals injected, via the fly ash slurry, to remove the level of SO_2 that theoretical utilization would suggest. Additional plots of the laboratory data versus SO_2 removal rates can be found in Appendix C.

A possible limiting factor in the reaction of SO_2 with alkaline metal oxides is the inability of these compounds within the ash to dissolve into the slurry. Figure IV-4 documents this effect for calcium contained in the fly ash. A similar problem has developed as a result of new control strategies for sulfuric acid produced by the oxidation of pyrite, FeS_2, in acid mine waters (29). One approach under investigation for elimination of excess

Table IV-6. Summary of regression equation coefficients and correlation coefficients.

SO_2 Removed vs.	No. of Fly Ashes in Model	Coefficient Term[a] 0	1	R^2
TAMO-S	23	4.84×10^{-2}	0.001	0.08
Lignite ash	6	3.17×10^{-4}	0.063	0.06
Subbituminous ash	8	2.50×10^{-3}	-0.044	0.14
Bituminous ash	9	2.91×10^{-3}	-0.270	0.30
Slurry Alkalinity	23	1.08×10^{-3}	1.012	0.03
Lignite ash	6	2.13×10^{-3}	-2.014	0.05
Subbituminous ash	8	2.27×10^{-3}	-1.440	0.13
Bituminous ash	9	5.75×10^{-4}	0.908	0.04
Slurry Calcium	23	1.70×10^{-3}	-2.600	0.09
Lignite ash	6	2.74×10^{-3}	-7.429	0.26
Subbituminous ash	8	2.07×10^{-3}	-2.336	0.27
Bituminous ash	9	5.53×10^{-4}	0.959	0.02
Slurry Sodium	23	1.14×10^{-3}	0.215	0.16
Lignite ash	6	3.34×10^{-4}	0.379	0.61
Subbituminous ash	8	1.55×10^{-3}	4.929	0.14
Bituminous ash	9	5.79×10^{-4}	2.318	0.03
Slurry (Calcium + Sodium)	23	1.14×10^{-3}	0.430	0.16
Lignite ash	6	3.34×10^{-4}	0.758	0.61
Subbituminous ash	8	1.55×10^{-3}	9.859	0.14
Bituminous ash	9	5.79×10^{-4}	4.637	0.03
Slurry Total Hardness	23	1.68×10^{-3}	-1.980	0.06
Lignite ash	6	-6.049	0.003	0.18
Subbituminous ash	8	2.09×10^{-3}	-1.971	0.22
Bituminous ash	9	-2.84×10^{-4}	3.453	0.40

Table IV-6 (Continued)

SO_2 Removed vs.	No. of Fly Ashes in Model	Coefficient Term[a] 0	1	R^2
Slurry (Total Hardness + Sodium)	23	1.05×10^{-3}	0.413	0.16
Lignite ash	6	7.19×10^{-5}	0.818	0.64
Subbituminous ash	8	2.14×10^{-3}	-1.900	0.18
Bituminous ash	9	-2.23×10^{-4}	2.806	0.33
Surface Area	23	1.856	0.143	0.28
Lignite ash	6	0.268	0.620	0.85
Subbituminous ash	8	2.801	0.088	0.08
Bituminous ash	9	0.697	0.158	0.73

[a]Coefficient terms of the form: $Y = (0) + (1)X$

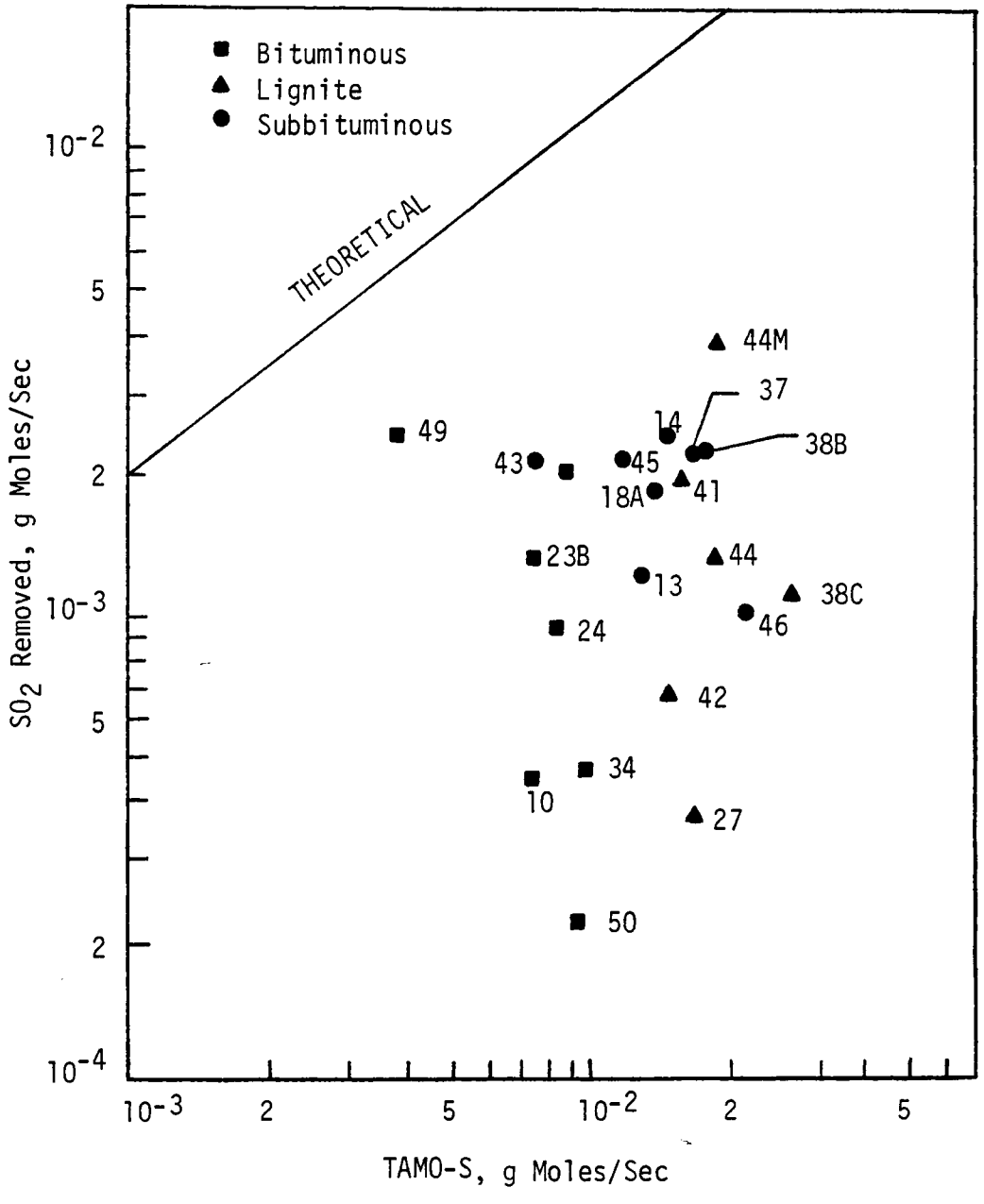

Figure IV-3. SO_2 removed as a function of total available alkaline metal oxides (TAMO-S) injected.

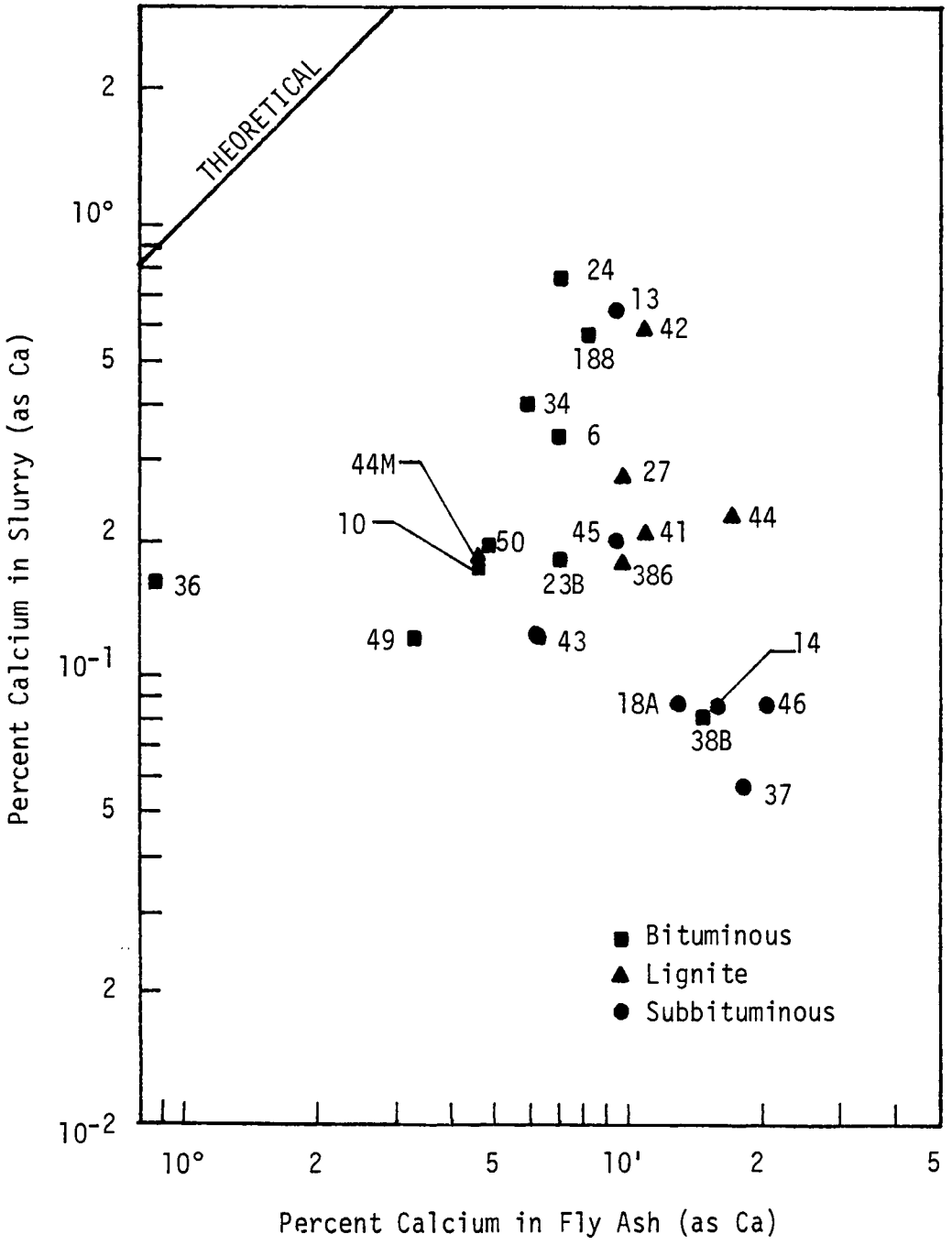

Figure IV-4. Calcium in slurry as a function of calcium content of the fly ash.

acidity involves the use of carbonate rocks, e.g., limestone. However, a difficulty encountered in neutralizing the iron-rich waters is the precipitation of insoluble ferric hydroxide on reacting surfaces. This armoring effect prevents further reaction of the limestone with the acid, and the neutralization reaction stops after a short time. Similarly, the elemental sulfur retained by the fly ash after combustion in various sulfite and sulfate forms may be providing an impermeable barrier to further reaction with SO_2. If this is true, destruction of such a barrier should increase the reaction potential of the ash. For the case of carbonate rocks, Barnes (29) suggests the problem of armoring can be minimized by grinding and stirring during reaction.

In order to determine whether grinding fly ash could increase its SO_2 reaction potential, a ball mill was used. A highly alkaline fly ash, number 44, which had demonstrated 16 percent system efficiency was ground and retested at the spray dryer facility as ash number 44M. A significant increase to 46 percent efficiency was observed with this ash. Also, laboratory analyses of this ash (44M) showed a 62 percent increase in slurry alkalinity and a 17 percent increase in surface area.

The importance of fly ash surface area was demonstrated by the lignite and bituminous fly ashes in particular. The specific surface area measured in square meters per gram was expressed in terms of square meters per second of fly ash injected and correlated with SO_2 removal in g moles per second (Table IV-6). The resulting

correlation coefficients were, R^2 = 0.85, for the lignite ashes and R^2 = 0.73 for the bituminous ashes. It was also shown, in one instance, that a fly ash, number 49, with considerable surface area (12.5 m^2/g) but negligible alkaline metal content removed nearly 30 percent SO_2. This large amount of surface area was due primarily to rather incomplete combustion within the stoker-fired boiler; thus producing an ash with a high carbon content. Consequently, physical adsorption of the SO_2 in the internal pores was considered to be the mechanism responsible for this removal.

It is evident from the data that the reactivity of a fly ash slurry can be influenced by physical and/or chemical parameters. The effect of ball milling, mentioned earlier, does increase the surface area (a parameter which demonstrated a high degree of correlation with SO_2 removal). Slurry alkalinity was also increased by this procedure. In addition, all fly ash slurry testing at the spray dryer facility used the same mass of fly ash in water. Determination of this quantity was based both on the combustion conditions maintained at the boiler and gas flow rate through the system. The mass concentration of the slurry was 1.45 pounds of fly ash per gallon of water (approximately 3 grains/actual cubic feet of flue gas). However, if the ash concentration was to be increased to 5 or even 10 pounds per gallon, a significantly greater efficiency improvement might be produced. Consequently, further study is needed to determine the optimum fly ash-water mixture.

Predicting SO_2 Removal by Total Sulfur Retention

It was discussed earlier in Chapter II that various sorbents containing one or all of the alkaline metal oxide compounds (CaO, Na_2O, MgO and K_2O) have demonstrated success in the removal of SO_2. These metals have been suggested as the primary constituents that occur in the formation of sulfate or sulfite end products (2, 3). Fiedler (23) used this information to quantify the ability of fly ash to retain sulfur in the boiler after the coal was burned. Figure IV-5 shows the curve developed by Fiedler (23) with the fly ashes used in this study plotted in the same manner. The sulfur retained after combustion was measured once the ashes were received from the utilities using a Fisher Total Sulfur Analyzer (Appendix A). However, the ashes of this investigation exhibited less sulfur retention than one would expect from the best fit line based on alkaline metal content. This indicates that the curve by Fiedler (23) may be demonstrating the upper limit for sulfur retention by higher alkaline metal content fly ashes.

It was this concept that provided the basis for the modeling of total sulfur retention. Expressing the amount of SO_2 removed, by the spray dryer/fabric filter system, as grams of sulfur retained per 100 grams of ash and summing that with the quantity of sulfur retained after combustion (see Table IV-4, page 38) determined the total sulfur retained.

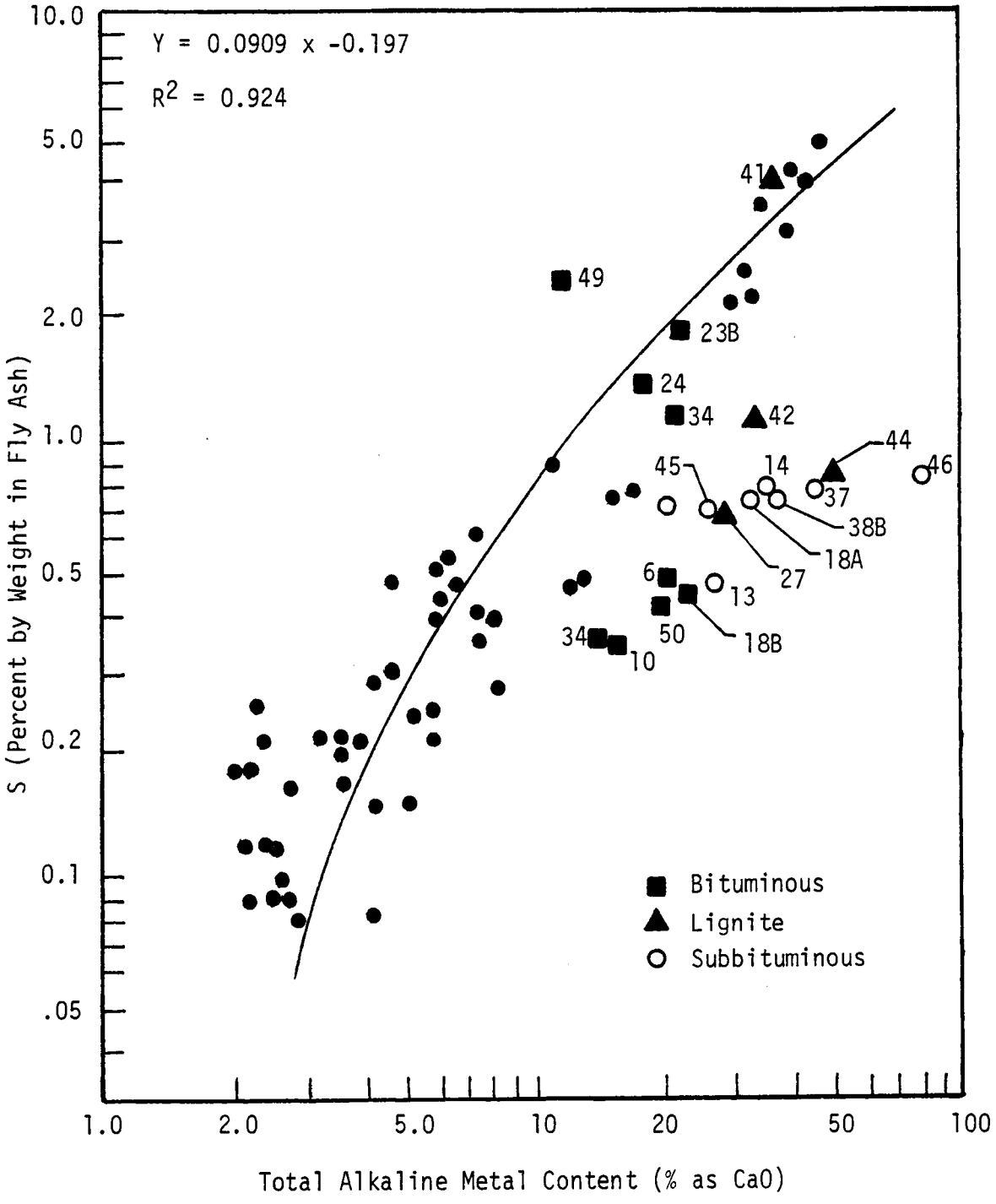

Figure IV-5. Sulfur retention versus total alkaline metal content (reported as CaO).

$$\frac{\text{g Total Sulfur Retained}}{\text{100 g of Fly Ash}} =$$

$$\frac{MSO_2\ (1) - MSO_2\ (3)}{K \times GPM} \times 100 + \%S \qquad \text{(Eq. 9)}$$

where

$$K = 1.45\ \frac{\text{lbs fly ash}}{\text{gallon } H_2O} \times \frac{64\ \text{lbs } SO_2/\text{lb mole}}{32\ \text{lbs } S/\text{lb mole}}$$

GPM = slurry feed rate (gallons/min)

%S = sulfur retained after combustion (g/100 g fly ash)

Table IV-7 contains the sulfur retention data as received and for the fly ash slurry injected into the system.

This new dependent variable, total sulfur retention, in conjunction with the concept described by Barnes (29), provided a new modeling approach. It was hypothesized that armoring of the slurried fly ash was limiting its reaction potential. Thus high alkalinity fly ashes did not necessarily produce highly alkaline slurries. Instead, reaction potential might be due to the slurry alkalinity as governed by the surface area of the ash.

To substantiate this theory, both surface area and slurry alkalinity were used as independent variables in this model to predict total sulfur retention. The resulting statistics showed correlation coefficients of 0.90 for the lignite and bituminous fly ashes. The subbituminous fly ash showed virtually no correlation with sulfur retention (R^2 = 0.11). Table IV-8 contains the regression summary for the lignite and bituminous fly ashes. It is evident from the level of significance that both surface area and

Table IV-7. Sulfur retention by fly ash.

Fly Ash I.D. No.	Sulfur Retained by Boiler g/100g Fly Ash	Sulfur Retained by System g/100g Fly Ash	Total Sulfur Retention g/100g Fly Ash
Lignite			
27	0.66	0.35	1.01
38C	0.00	1.33	1.33
41	4.06	2.23	6.29
42	1.03	0.88	1.91
44	0.75	1.81	2.56
44M	0.75	4.61	5.36
Subbituminous			
13	0.49	1.44	1.93
14	0.73	3.35	4.08
18A	0.70	2.46	3.16
37	0.78	2.76	3.54
38B	0.72	2.87	3.59
43	0.66	2.89	3.55
45	0.68	2.48	3.16
46	0.75	1.21	1.96
Bituminous			
6	0.43	0.11	0.54
10	0.32	0.53	0.85
18B	0.45	2.95	3.40
23B	1.87	0.15	2.02
24	1.22	1.05	2.27
34	1.04	0.49	1.53
36	0.36	0.00	0.36
49	2.19	3.12	5.31
50	0.41	0.25	0.66

Table IV-8. Multiple regression summary intercept model.

Independent Variable	Regression Coefficient	Standard Error[a]	T Value[b]	Significance Level[c]
Lignite Model				
Intercept	- 1.906	1.591	-1.20	68%
SA	0.019	0.004	4.84	98%
ALKAL	129.187	81.172	1.59	79%
Bituminous Model				
Intercept	0.070	0.344	0.20	15.5%
SA	0.004	0.001	7.11	100%
ALKAL	74.464	24.544	3.03	98%

[a]Standard Error = Square root of the estimated variance of the regression coefficient.

[b]T-Value = Regression Coefficient ÷ Standard Error.

[c]Significance Level = 1 - α, with α = the probability of rejecting the existence of a relation when one exists.

slurry alkalinity are critical to the models. However, the contribution of the intercept to the models exhibited only minimal significance. Consequently, when the equations were forced through a zero intercept, a dramatic improvement in the correlation efficient resulted. Values of R^2 of 0.96, 0.95, and 0.89 for bituminous, lignite, and subbituminous ashes, respectively, were calculated. Placing all 23 fly ashes into one model yielded an R^2 of 0.82. Equations 10-13 shown below are the resulting models for total sulfur retention both independent and dependent of fly ash rank.

$$TSR = 0.005\ (SA) + 121.137\ (ALKAL) \qquad \text{(Eq. 10)}$$

$$TSB = 0.004\ (SA) + 77.692\ (ALKAL) \qquad \text{(Eq. 11)}$$

$$TSL = 0.015\ (SA) + 41.043\ (ALKAL) \qquad \text{(Eq. 12)}$$

$$TSSB = 0.009\ (SA) + 117.516\ (ALKAL) \qquad \text{(Eq. 13)}$$

where

TSR = total sulfur retention by fly ash (g/100 g ash)

TSB = total sulfur retention by bituminous fly ash (g/100 g ash)

TSL = total sulfur retention by lignite fly ash (g/100 g ash)

TSSB = total sulfur retention by subbituminous fly ash (g/100 g ash)

SA = surface area of fly ash (m^2/100 g ash)

ALKAL = slurry alkalinity (g moles/100 g ash)

The summary of regression statistics for the modeling equations are contained in Table IV-9.

Table IV-9. Multiple regression summary zero-intercept model.

Independent Variable	Regression Coefficient	Standard Error[a]	T Value[b]	Significance Level[c]
Complete Model				
SA	0.005	0.001	5.40	100%
ALKAL	121.137	24.026	5.04	100%
Bituminous Model				
SA	0.004	0.0004	9.92	100%
ALKAL	77.692	17.443	4.45	100%
Lignite Model				
SA	0.015	6.33	6.33	100%
ALKAL	41.043	1.14	1.14	68%
Subbituminous Model				
SA	0.009	0.004	2.57	96%
ALKAL	117.516	42.713	2.75	97%

[a]Standard Error = Square root of the estimated variance of the regression coefficient.

[b]T-Value = Regression Coefficient ÷ Standard Error.

[c]Significance Level = 1 - α, with α = the probability of rejecting the existence of a relation when one exists.

Both the T-values and the correlation coefficients for these modeling equations confirm their validity. However, to advocate modeling total sulfur retention dependent on fly ash rank incorporates several risks. For example, the reduction in sample size for each of the 3 rank specific models provides a greater chance for error. Also, the variability in slurry alkalinity, determined from the filtered substrate, within the bituminous rank ashes indicates a weakness in the scheme for coal classification. Hence, improperly ranked ashes may yield an inaccurate estimate of total sulfur retention. It is recommended that the complete fly ash model based on 23 ashes be used to minimize these risks.

CHAPTER V

CONCLUSION

The major conclusions of this research effort are summarized below:

1. It was demonstrated that SO_2 efficiencies of 30 percent can be attained by a fly ash water slurry (at 1.45 lbs. ash/gallon) in a spray dryer/fabric filter system.
2. The high levels of total alkaline metal oxides, contained in several of the fly ashes tested, were not a reliable predictor of SO_2 removal efficiency alone. Laboratory analysis of the 23 fly ashes show an inability of metal oxides to dissolve in the slurry thereby limiting reaction with SO_2.
3. Grinding fly ash number 44 in a ball mill produced an ash (44M) that when slurried and injected into the flue gas increased the SO_2 removal 156 percent. Ash number 44M showed a 17 percent increase in surface area in addition to a 62 percent enhancement in the alkalinity of the slurry.
4. Calculating the amount of SO_2 removed as grams of sulfur retained per 100 grams of fly ash and adding the percent sulfur in the ash after combustion produced an estimate of total sulfur retention. Four modeling equations using fly ash surface area and slurry alkalinity were produced

based on this concept. A complete model (using all of the ashes collected for this study) for fly ash total sulfur retention yielded an R^2 of 0.82. Separating the fly ashes by rank bituminous, lignite, and subbituminous yielded R^2 values of 0.96, 0.95, and 0.89, respectively.

CHAPTER VI

RECOMMENDATIONS FOR ADDITIONAL RESEARCH

The potential of fly ash as a sorbent in spray dryer systems was shown to be significant for SO_2 removal. Further investigations to more clearly define and increase the range of application are suggested below:

1. The optimum fly ash slurry concentration for SO_2 removal in a spray dryer should be determined.
2. Extraction methods for increasing the alkalinity of the fly ash slurry need to be developed to increase the reaction potential of the slurry.
3. Further studies documenting the advantages of grinding the fly ash in a ball mill and thus producing corresponding increases in SO_2 removal.
4. Development and study of high surface area sorbents and their potential contribution to traditional lime based slurries.

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APPENDICES

APPENDIX A

INSTRUMENTATION

Listed below is a brief description regarding operation of instruments used in this study that were not discussed in Chapter II.

1. Coulter Counter Model TaII--The Coulter Counter was used to determine particle size distributions for the fly ash used in this study. A suspension of ash in electrolyte was forced through a charged apperture. Particles passing through the apperture increase the resistance between the electrode immersed in the conductive fluid and the apperture. Resulting current pulse heights are scaled and counted thereby determining a quantity proportional to the particle volume. With this information, the analyzer delineates the size distribution.

2. Helium-Air Pycnometer Model 1302/1303--The pycnometer uses pressure and volume relationships for pure gases to determine the volume of solids in a vessel. The density is then obtained by dividing the solid's weight, determined separately, by its volume.

3. Fisher Sulfur Analyzer Model 470--The weight percent of sulfur contained in the fly ash as a result of combustion was measured. An amperometric titration technique is used by this analyzer.

4. Surface Area Analyzer--Surface area is measured by determining the amount of liquid nitrogen necessary to form a single layer of gas molecules on a solid material.

5. Lear Siegler Model SM800 SO_2/NO Analyzer--This instrument was used to monitor SO_2 concentrations at three ports. The analyzer enumerates the absorption of ultraviolet light by molecules of SO_2 and NO (nitric oxide).

6. Fisher U.S. Signal Corps Mercury Barometer--Local barometric pressure was obtained from this instrument.

APPENDIX B

CALCULATIONS

In the following relationships, machine notation is used, for example 1 x 10-6 is expressed as 1E - 6.

1. Vapor Pressure of Water

$$PWBn = \exp (0.027\ TWn - 2.006) \qquad \text{(Eq. 14)}$$

where

n = ports 1, 2, and 3 calculated

PWBn = vapor pressure of water (in Hg)

TWn = wet bulb temperature (°F)

2. Static Pressure Determination

$$PSn = \frac{SPn}{13.6 + PBAR} \qquad \text{(Eq. 15)}$$

where

n = ports 1, 2, and 3 calculated

PSn = static pressure (in Hg)

SPn = static pressure (in H_2O)

PBAR = barometric pressure (in Hg)

3. Actual Vapor Pressure Exerted by Water in Duct

$$PWn = PWBn - \frac{(PSn - PWBn)\ (Tn - TWn)}{2830 - 1.3\ TWn} \qquad \text{(Eq. 16)}$$

where

n = ports 1, 2, and 3 calculated

PWn = vapor pressure exerted in duct (in Hg)

Tn = duct temperature (°F)

4. Moisture Fraction

$$BWn = \frac{PWn}{PSn} \qquad \text{(Eq. 17)}$$

where

n = ports 1, 2, and 3 calculated

BWn = moisture fraction in duct

The following constants were determined by system calibration:

Static Pressure at port 3 = PS3C = 28.59 in Hg.

Orifice Constant = KOR = 1400.00

Temperature at port 3 = T3C = 250°F

5. Gas Flow Through System at Standard Conditions

$$T4 = \frac{PS3C\ (T3 + 460)}{PS3\ (T3C + 460)} \qquad \text{(Eq. 18)}$$

$$ACFM3 = KOR\ (T4)^{1/2}\ (DELTP)^{1/2} \qquad \text{(Eq. 19)}$$

$$TC = \frac{530}{T3 + 460} \qquad \text{(Eq. 20)}$$

$$PC = \frac{PS3}{29.92} \qquad \text{(Eq. 21)}$$

$$SCFM3 = ACFM3\ (TC)\ (PC) \qquad \text{(Eq. 22)}$$

where

T4 = pressure and temperature corrected to standard conditions

PS3C = pressure at port 3 corrected to standard conditions (in Hg)

T3C = temperature at port 3 corrected to standard conditions (°R)

ACFM3 = actual gas flow rate at port 3 (acfm)

DELTP = pressure across orifice (in H_2O)

TC = temperature corrected to standard conditions (°R)

PC = pressure corrected to standard conditions (in Hg)

SCFM3 = gas flow rate at port 3 at standard conditions (scfm)

6. Gas Flow and Moisture Standard Conditions

$$SCFM2 = SCFM3\ (1 - (BW3 + BW2)) \qquad \text{(Eq. 23)}$$

$$SCFM1 = SCFM2\ (0.957)\ (1 - (BW2 + BW1)) \qquad \text{(Eq. 24)}$$

$$SCFM1C = SCFM1 + 0.043\ (SCFM2) \qquad \text{(Eq. 25)}$$

$$BW1C = \frac{(SCFM1\ (BW1)) + (0.043\ (SCFM2)\ (0.005))}{SCFM1 + 0.043\ (SCFM2)} \qquad \text{(Eq. 26)}$$

where

SCFM2 = gas flow rate at port 2 at standard conditions (scfm)

SCFM1 = gas flow rate at port 1 at standard conditions (scfm)

SCFM1C = SCFM1 corrected for air leakage into the system (scfm)

BW1C = BW1 corrected for air leakage into the system

7. Temperature Correction

$$TEMP1C = \frac{((SCFM1\ (TEMP1)) + (0.043\ (SCFM2)\ (TAMB)))}{SCFM1 + 0.043\ (SCFM2)} \qquad \text{(Eq. 27)}$$

where

TEMP1 = temperature measured at port 1 (°F)

TAMB = ambient temperature (°F)

TEMP1C = TEMP1 corrected for air leakage into the system (°F)

8. SO_2 Concentration

$$SO2ONEC = \frac{SCFM1\ (SO2ONE)}{SCFM1C} \qquad \text{(Eq. 28)}$$

where

SO2ONEC = SO_2 concentration at port 1 at standard conditions (ppm)

SO2ONE = SO_2 concentration measured at port 1 (ppm, wet)

9. SO_2 Concentration Corrected for Moisture

$$SO_2\ (1) = \frac{SO2ONEC}{1-BW1C} \quad \text{(Eq. 29)}$$

$$SO_2\ (2) = \frac{SO2TWO}{1-BW2} \quad \text{(Eq. 30)}$$

$$SO_2\ (3) = \frac{SO2THR}{1-BW3} \quad \text{(Eq. 31)}$$

where

n = ports 1, 2, and 3 calculated

$SO_2(n)$ = SO_2 concentration at port indicated (ppm, dry)

SO2TWO = SO_2 concentration measured at port 2 (ppm, wet)

SO2THR = SO_2 concentration measured at port 3 (ppm, wet)

10. Efficiency Determination

$$EFFSD = \frac{SO_2\ (1) - SO_2\ (2) \times 100}{SO_2\ (1)} \quad \text{(Eq. 32)}$$

$$EFFBH = \frac{SO_2\ (2) - SO_2\ (3) \times 100}{SO_2\ (2)} \quad \text{(Eq. 33)}$$

$$EFFSYS = \frac{SO_2\ (1) - SO_2\ (3) \times 100}{SO_2\ (1)} \quad \text{(Eq. 34)}$$

$$EFFSYSC = EFFSYS - 2.2447 \quad \text{(Eq. 35)}$$

where

EFFSD = efficiency of spray dryer

EFFBH = efficiency of bag house

EFFSYS = efficiency of system

EFFSYSC = efficiency of system minus water effect

11. Temperature Differences

$$DELTASD = TEMPIC - TEMP2 \quad \text{(Eq. 36)}$$

$$DELTABH = TEMP2 - TEMP3 \quad \text{(Eq. 37)}$$

where

DELTASD = temperature drop across spray dryer (°F)

DELTABH = temperature drop across bag house (°F)

TEMP2 and TEMP3 = temperature in duct at ports 2 and 3 (°F)

12. Mass Flow Rate of SO_2

MSO2 (1) = 2.56E-9 (SO2ONE) (SCFMn) (64.04) (Eq. 38)

MSO2 (2) = 2.56E-9 (SO2TWO) (SCFMn) (64.04) (Eq. 39)

MSO2 (3) = 2.56E-9 (SO2THR) (SCFMn) (64.04) (Eq. 40)

where

MSO2(n) = mass flow rate of SO_2 (lbs/min)

n = ports 1, 2, and 3

13. System SO_2 Removal Minus Water Effect

$$SO2REMC = \left(\frac{(MSO2\ (1) - MSO2\ (3))}{64.04}\right) \times 7.56 - 2.38E\text{-}4 \quad \text{(Eq. 41)}$$

where

SO2REMC = SO_2 removed by system (moles/sec)

14. Approach to Saturation

DELTA = TEMP2 - TW2 (Eq. 42)

where

DELTA = degrees above dew point (°F)

15. Flue Gas Corrected to Actual Conditions

$$TACT = \frac{460 + TEMP1C}{530} \quad \text{(Eq. 43)}$$

$$ACFMC = \frac{SCFMC\ (TACT)\ (29.92)}{PSn} \quad \text{(Eq. 44)}$$

where

TACT = actual gas temperature at port 1 (°F)

ACFMC = actual gas flow rate through system (acfm)

16. Calculations on Fly Ash Slurry Data

ALKALI = ALKALM (GPM) (6.308E-7) (Eq. 45)

ALKAL = ALKALM (6.677E-6) (Eq. 46)

CASLUR = CASLURM (6.677E-4) (Eq. 47)

NASLUR = NASLURM (6.677E-4) (Eq. 48)

CAINJ = CASLURM (GPM) (1.57E-6) (Eq. 49)

NAINJ = NASLURM (GPM) (2.74E-6) (Eq. 50)

NACA = NASLURM (GPM) (1.37E-6) (Eq. 51)

CANA = CAINJ + NACA (Eq. 52)

THINJ = THM (GPM) (1.57E-6) (Eq. 53)

THNAINJ = THINJ + NACA (Eq. 54)

where

GPM = slurry flow rate to spray dryer (gallons/min)

ALKALI = total alkalinity injected (moles/sec as $CaCO_3$)

ALKAL = total alkalinity (moles $CaCO_3$/100 g Fly Ash)

ALKALM = total alkalinity measured (mg/L as $CaCO_3$)

CASLUR = calcium in slurry (g Ca/100 g Fly Ash)

CASLURM = calcium in slurry measured (mg/L as Ca)

NASLUR = sodium in slurry (g Na/100 g Fly Ash)

NASLURM = sodium in slurry measured (mg/L as Na)

CAINJ = calcium in slurry injected (moles Ca/sec)

NAINJ = sodium in slurry injected (moles Na/sec)

NACA = sodium expressed as calcium (moles Na/sec as Ca)

CANA = sum of calcium and sodium injected (moles/sec as Ca)

THINJ = total hardness injected (moles/sec as Ca)

THM = total hardness measured (mg/L as Ca)

THNAINJ = total hardness plus sodium injected (moles/sec as Ca)

17. Calculations on Fly Ash ASTM Analysis

$$TAMO\text{-}S = TAMO\text{-}S'(GPM)(0.11) \quad \text{(Eq. 55)}$$

where

TAMO-S = total alkaline metals injected (moles/sec)

TAMO-S' = found in Table IV-4, page 38

18. Surface Area Conversion

$$SAI = SAM\ (GPM)\ (10.96) \quad \text{(Eq. 56)}$$

$$SA = SAM\ (100) \quad \text{(Eq. 57)}$$

where

SAI = surface area injected (m^2/sec)

SAM = specific surface area measured (m^2/g)

SA = surface area (m^2/100 g fly ash)

APPENDIX C

FIGURES

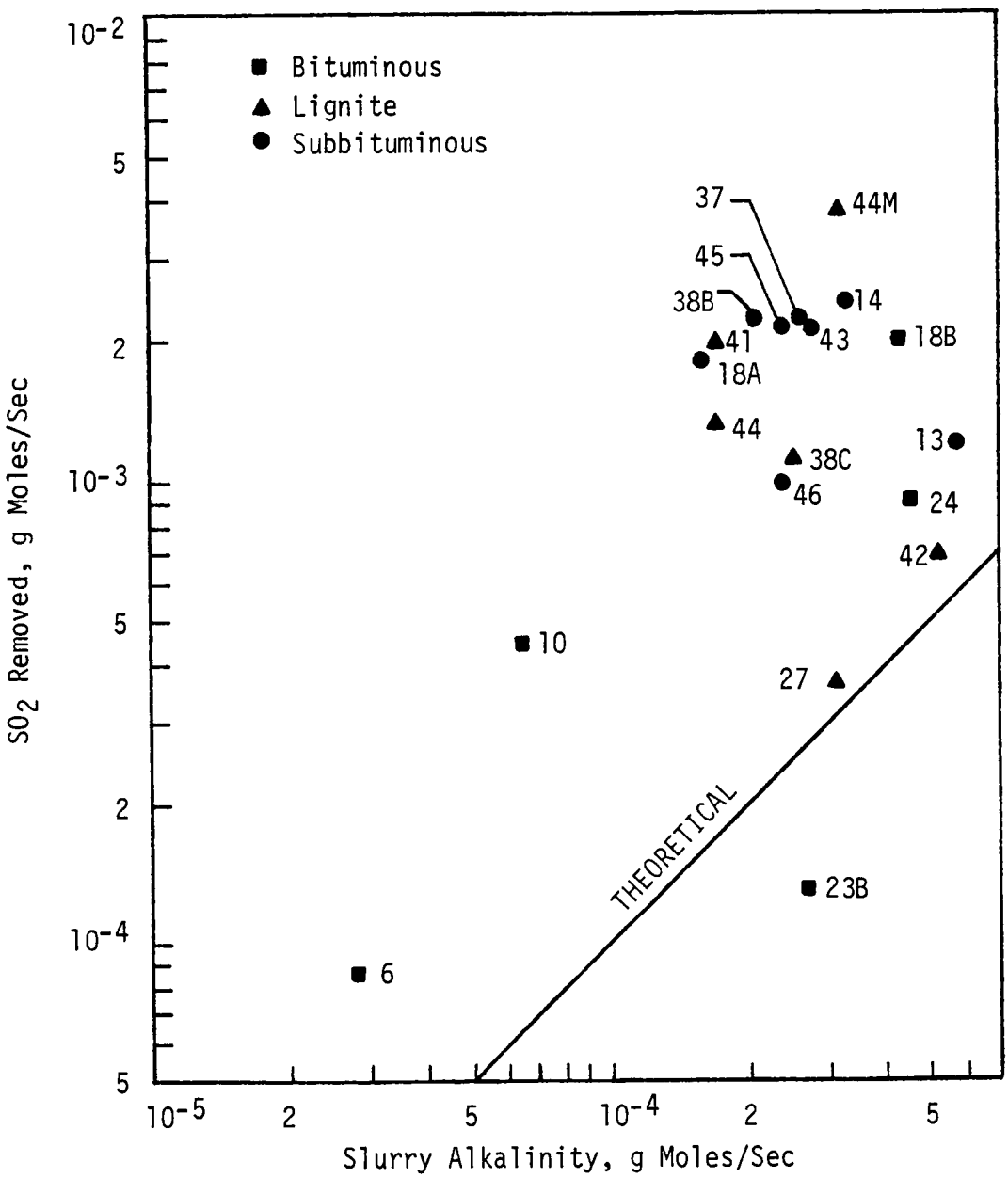

Figure C-1. SO_2 removed as a function of the total slurry alkalinity.

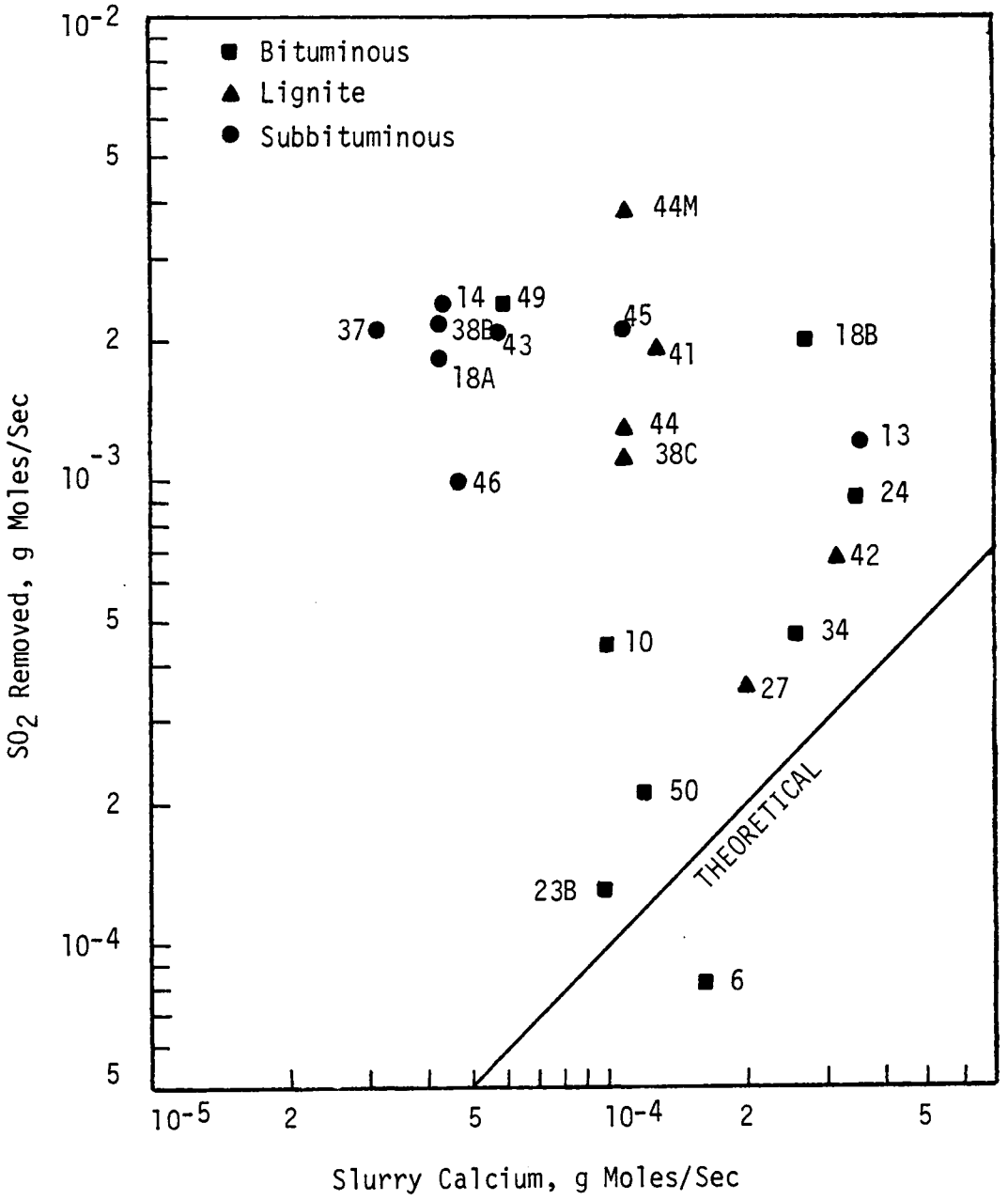

Figure C-2. SO_2 removed as a function of calcium in the slurry.

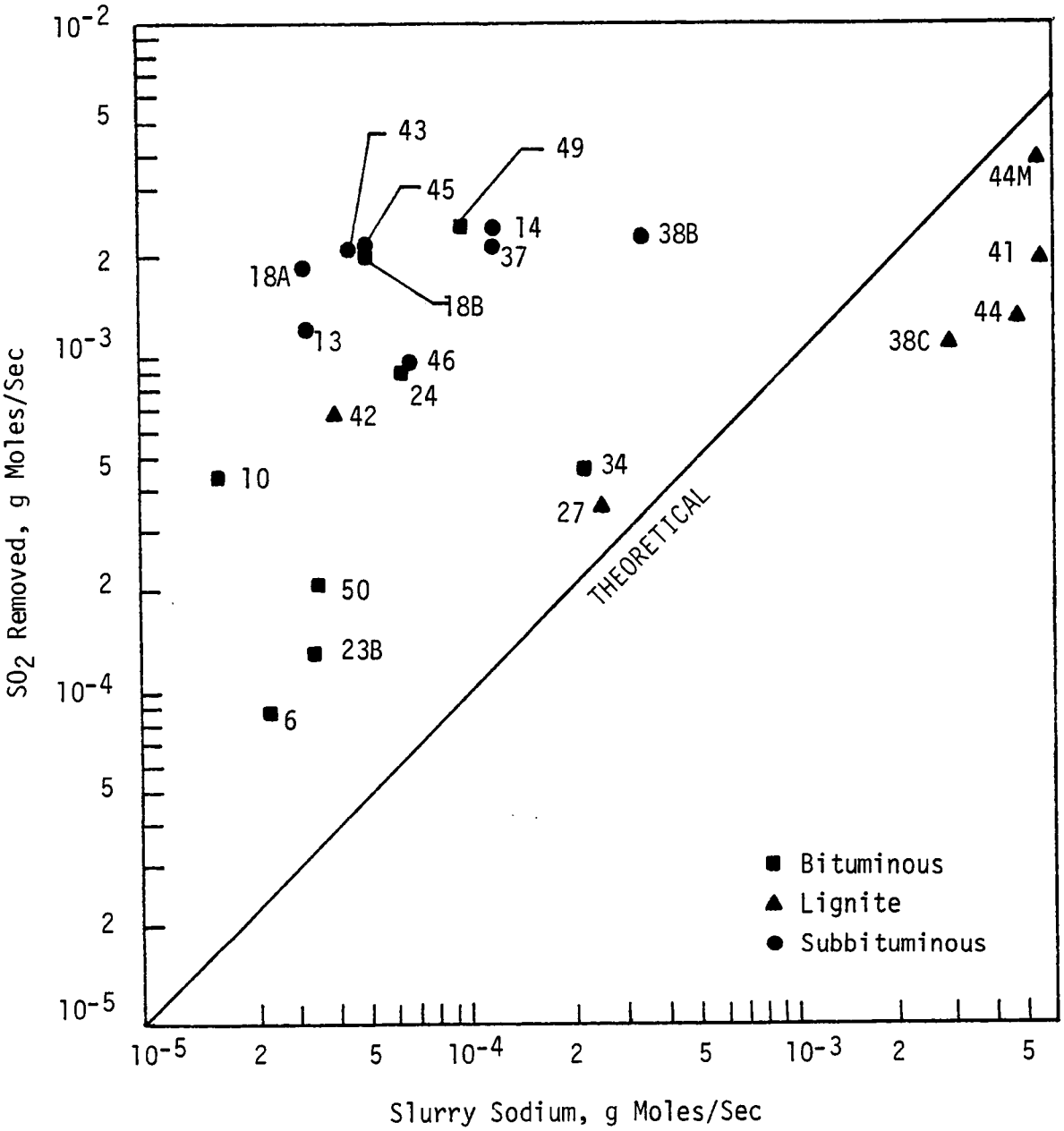

Figure C-3. SO_2 removed as a function of sodium in the slurry.

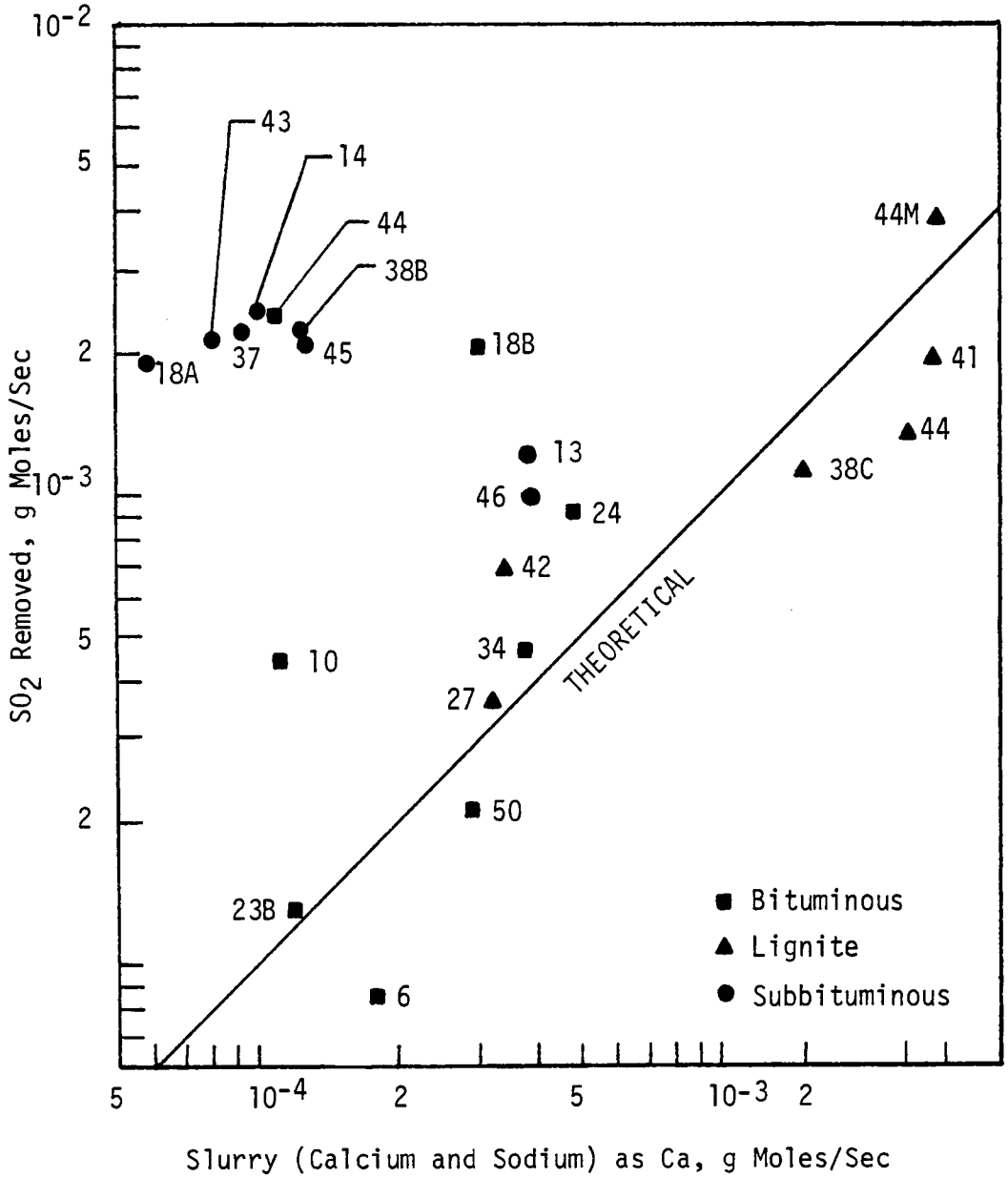

Figure C-4. SO_2 removed as a function of both calcium and sodium in the slurry expressed as calcium.

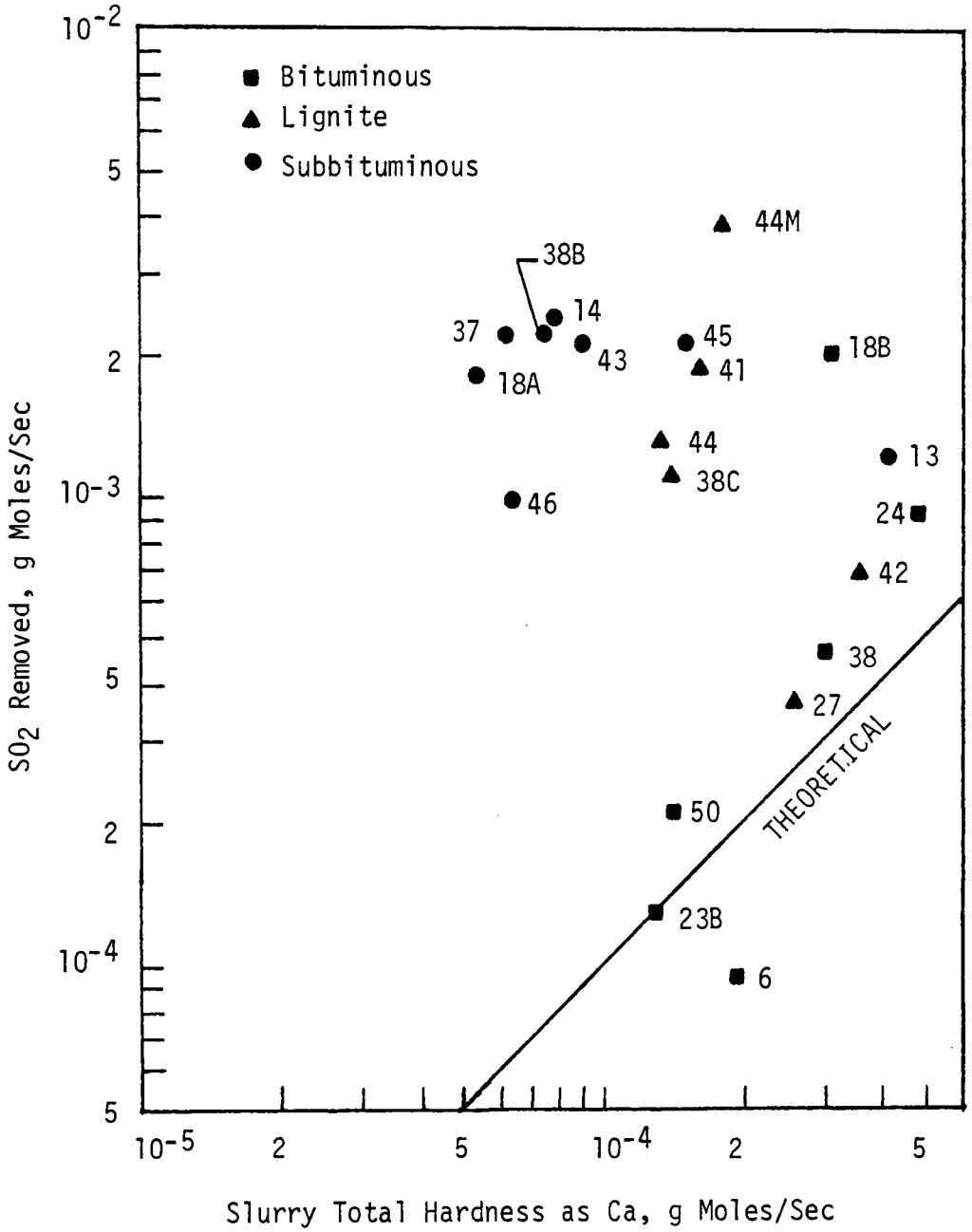

Figure C-5. SO_2 removed as a function of total hardness content of the slurry expressed as calcium.

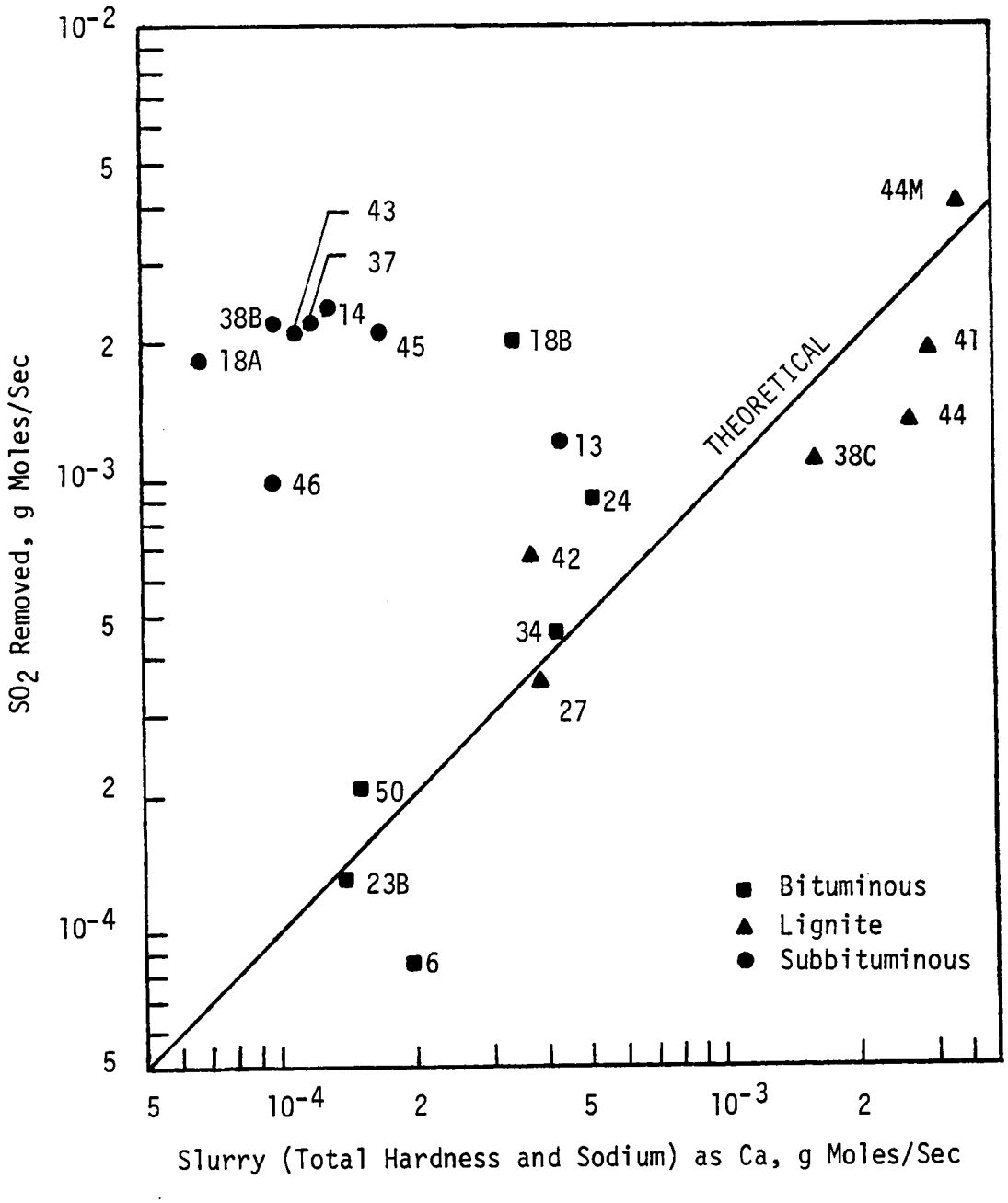

Figure C-6. SO_2 removed as a function of total hardness and sodium in the slurry expressed as calcium.

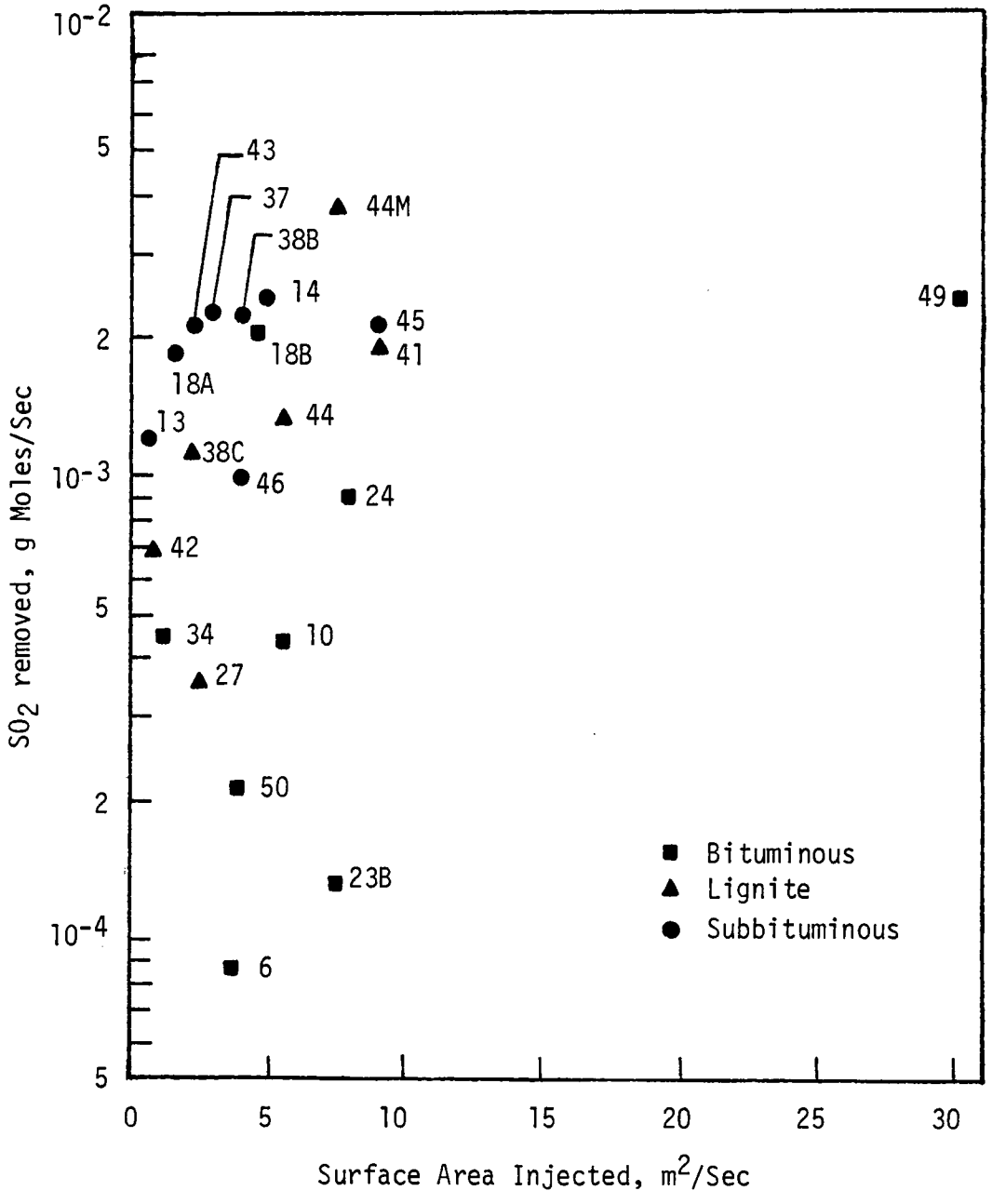

Figure C-7. SO_2 removed as a function of surface area injected into flue gas.

APPENDIX D

SPRAY DRYER TEST DATA

Table D-1. Spray dryer test data.

Fly Ash I.D. No.	Test Date	Port 1 Temp °F	Port 2 Temp °F	Port 3 Temp °F	Orifice P in H_2O	Static Press Port 1 in H_2O	Static Press Port 2 in H_2O	Static Press Port 3 in H_2O	Barometric Press in Hg	Slurry Feed Rate GPM	Wet Bulb Temp Port 1 °F	Wet Bulb Temp Port 2 °F	Wet Bulb Temp Port 3 °F	SO_2 Port 1 (Wet Basis) ppm	SO_2 Port 2 (Wet Basis) ppm	SO_2 Port 3 (Wet Basis) ppm
6	121081	304	138	144	0.43	5.2	7.2	7.3	29.33	0.22	120	118	117	670	610	590
10	100781	311	136	138	0.38	3.5	6.0	6.5	29.01	0.24	120	117	114	578	460	480
13	100781	310	136	151	0.38	3.5	6.0	6.5	29.01	0.24	120	116	112	592	500	440
14	111081	310	139	138	0.41	4.0	6.6	9.0	29.40	0.21	120	118	117	665	390	410
18A	111081	307	137	140	0.40	4.1	7.0	9.2	29.40	0.21	119	117	116	680	480	470
18B	100681	316	138	132	0.41	3.5	5.9	6.5	29.13	0.20	119	117	116	705	475	480
23B	111181	314	137	138	0.40	4.2	7.2	9.7	29.40	0.24	119	117	116	635	550	550
24	110481	310	135	146	0.39	3.9	6.4	7.0	29.16	0.25	120	117	114	1015	850	850
27	111381	307	137	141	0.41	4.4	9.8	12.3	29.43	0.30	119	117	116	630	550	530
34	111381	305	136	143	0.41	4.4	9.5	12.2	29.43	0.27	119	117	116	670	510	560
36	111181	302	135	152	0.39	4.3	8.9	11.5	29.40	0.26	119	117	116	670	590	600
37	121081	303	139	136	0.42	5.3	7.2	7.3	29.33	0.23	119	117	116	680	450	445
38B	110681	304	137	133	0.44	4.1	6.8	9.3	29.42	0.22	119	115	114	655	480	430
38C	111181	302	137	162	0.36	3.9	7.5	9.9	29.40	0.25	119	117	116	675	530	510
41	111881	303	137	159	0.44	4.2	7.3	9.8	29.33	0.25	120	118	117	725	530	510
42	100581	312	138	132	0.42	3.5	5.9	6.5	29.39	0.23	120	116	112	625	510	510
43	100581	310	138	130	0.42	3.5	6.0	6.2	29.39	0.21	120	116	112	633	440	417
44	100581	298	138	139	0.42	3.5	5.0	6.2	29.39	0.21	120	116	112	658	500	500
44M	121181	291	137	136	0.43	6.0	8.3	8.1	29.13	0.24	119	117	116	650	490	305
45	100681	310	138	132	0.41	3.7	5.5	6.2	29.13	0.24	120	118	114	678	530	455
46	100581	315	138	131	0.42	3.5	6.0	6.5	29.39	0.24	120	116	113	630	490	490
49	121181	292	138	145	0.43	6.0	8.1	8.2	29.13	0.22	120	118	117	640	505	400
50	100181	315	138	135	0.41	3.8	5.5	6.5	28.96	0.24	120	119	119	1000	880	870

VITA

Randal E. Pudelek was born in Chicago, Illinois on November 11, 1958. He attended Catholic schools in Chicago and was graduated from Gordon Technical High School in June 1976. He entered Illinois Institute of Technology and received a Bachelor of Science degree in Engineering Science with a major in Environmental Engineering in December 1980. In January of 1980 he accepted a graduate assistantship at The University of Tennessee, Knoxville, and began study toward a Master's degree in Environmental Engineering. This degree was received in December 1982.

The author is a member of the Western Society of Engineers and the Air Pollution Control Association.

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