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# **LIME/LIMESTONE SCRUBBING IN A PILOT DUSTRACTOR-KEY WEST**



Office of Research and Development  
U.S. Environmental Protection Agency  
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# **LIME/LIMESTONE SCRUBBING IN A PILOT DUSTRACTOR-KEY WEST**

by

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The second phase of the study was carried out at the Shawnee Power Station of the Tennessee Valley Authority (TVA), Paducah, Kentucky. The cooperation of the TVA personnel and on-site EPA personnel played a key part in executing this phase of the study.

Some of the reactants utilized in this study were supplied gratis by interested parties. Special thanks go to Dixie Lime & Stone, Ocala, Florida (aragonite); Fredonia Valley Quarries, Fredonia, Kentucky (limestone); and Frank Toppino & Sons, Key West, Florida (coral marl).

The pilot plant was supplied by Zurn Industries, Inc., Erie, Pennsylvania. The efforts of Zurn's Air System Division, Birmingham, Alabama contributed significantly towards keeping the pilot plant running through certain critical periods.

## SECTION 1

### INTRODUCTION

Abatement of the air pollutants commonly associated with electric power generation--particulates, sulfur dioxide ( $\text{SO}_2$ ), and oxides of nitrogen ( $\text{NO}_x$ )--is a primary goal of the public utility industry and government. Particulates can be controlled through use of commercially available and proven technology; however, processes for control of the oxides of sulfur and nitrogen formed in the combustion process have not reached the same state of commercial availability.  $\text{SO}_2$  is formed when sulfur-bearing fuels, either solid or liquid, are used in the combustion process; hence, two  $\text{SO}_2$  control strategies become immediately apparent: use low-sulfur or sulfur-free fuel or remove the  $\text{SO}_2$  from the flue gas. The use of low-sulfur or sulfur-free fuel depends upon the availability of these fuels. Currently, the demand greatly exceeds the supply. Therefore, removal of  $\text{SO}_2$  from flue gas is receiving considerable attention from both the public utility industry and government.

The major problem in removing  $\text{SO}_2$  from flue gases is not the technical problem of  $\text{SO}_2$  absorption or adsorption, but how to accomplish this economically without adversely affecting the reliability of electrical generation. Handling the required volumes of flue gas by standard chemical engineering unit operations (such as absorption towers) is a complex engineering problem which must be thoroughly investigated in a broad-based research and development program prior to commercial operation of any process. Thus, a primary task of the Control Systems Laboratory, Office of Research and Development, Environmental Protection Agency, has been to investigate a variety of research and development processes potentially amenable to this problem.



Wet scrubbing of flue gases by various aqueous absorbents capable of reacting with  $\text{SO}_2$  has been one of the most vigorously investigated approaches for control of  $\text{SO}_2$ . The obvious technical advantage of wet scrubbing is the well-established unit operation of particulate collection and gas absorption by aqueous scrubbing processes in other industries. The Environmental Protection Agency, therefore, undertook a program to develop the potential of the calcium base or limestone process for  $\text{SO}_2$  control from power plants. The process appeared to be technically simple and the least expensive to install among the potential first-generation wet scrubbing processes.

There are many variations of the lime/limestone wet scrubbing process including:

- Boiler injection with subsequent wet scrubbing
- Open-loop, tail-end lime/limestone wet scrubbing
- Closed-loop, tail-end lime/limestone wet scrubbing

The dry limestone injection into a power plant boiler followed by wet-lime scrubbing has been studied on a large prototype test facility by the Tennessee Valley Authority (TVA). Other studies are currently underway on both open and closed-loop tail-end systems since potential problems related to limestone injection into a boiler are eliminated with these processes.

Initial pilot plant testing of the Zurn Air Systems Division (ZASD) limestone wet scrubbing system on boiler flue gas took place in 1969 with favorable results. Based upon the favorable results from this early effort, Zurn Industries, Inc. proposed a cost-shared pilot plant study with the National Air Pollution Control Administration (now EPA) to investigate certain variables important to the success of the tail-end limestone wet scrubbing process. On April 30, 1970, Engineering-Science, Inc. was awarded a contract for an in-depth study of the tail-end limestone wet scrubbing process utilizing the ZASD scrubber.

The contract included the design, fabrication, and operation of a limestone wet scrubbing pilot plant.

The object of this contract was to evaluate SO<sub>2</sub> removal capabilities of this limestone wet scrubbing system on flue gases from both oil-fired and pulverized coal-fired boilers while utilizing a series of different calcium based reactants.

Several additional goals of the program were the:

- Investigation of potential operating difficulties
- Generation of data for a scaled-up design
- Evaluation of the process economics

The test program was divided into two major segments:

- Key West, Unit No. 3, City Electric System, Key West, Fla.
- TVA Shawnee Units No. 9 and No. 10, Paducah, Ky.

The Key West phase of the study took place while the pilot plant was installed on the No. 3 unit of the Utility Board of the City of Key West, Florida. This unit was a 20 MW base-loaded steam generator firing 1-2% sulfur No. 6 fuel oil. The sulfur content of the fuel was fairly constant due to the method of mixing of fuel shipments; however, on the days that fresh fuel was received, the sulfur concentration in the flue gas varied widely.

The Paducah phase of the study took place while the pilot plant was located between the No. 9 and No. 10 units of the TVA Shawnee generating station. Both of these units were 150 MW steam generators firing 2-4% sulfur pulverized coal. The No. 9 unit was conventional while the No. 10 unit had been modified to allow dry limestone injection for testing under another EPA contract.

A series of statistical design tests was developed to evaluate the effects of design characteristics, reactant properties, catalysts, and operating conditions on primary dependent variables such as:

- SO<sub>2</sub> absorption
- Scale formation
- Corrosion
- Operating problems
- Process costs

The test program resulting from the experimental designs included over 200 tests related to:

- Reactant type
- Stoichiometry
- Particle size
- Slurry concentration
- Catalysts
- Liquid/gas ratio
- Pressure drop

Twelve series of tests were conducted at Key West and Paducah from January 1971 through August 1971 to study the aforementioned variables. Table 1-1 is a list of the test series conducted during this project, the test site location, and test dates.

The initial salt water tests (S-XX series) were performed to determine the operational limitations of the scrubber and monitoring equipment. During this period mechanical and electrical malfunctions were identified and eliminated to produce a reliable system. Once accomplished, the testing program proceeded to the primary test series.

The first series of reactant tests (C-XX series) used a crushed coral slurry to evaluate its potential use as a reactant for the full scale demonstration scrubber. A half factorial statistical design schedule was followed to gain maximum scrubber operation information from a minimum number of test runs. The data generated were later compared with tests which used the more commonly used slurries such as limestone and lime. In addition, specific additives which purportedly enhance SO<sub>2</sub> removal were investigated during this testing period.

Table 1-1. PILOT PLANT TEST SERIES

Test identi- fication	Test series	Dates
S-XX	Key West-Initial Salt Water Shake- down Test Series	1/7/71 - 1/18/71 1/19/71 - 1/28/71
C-XX	Key West - Coral Test Series	1/28/71 - 5/25/71
F-XX	Key West - Fredonia Valley Lime- stone Test Series	2/6/71 - 5/20/71
HL-XX	Key West - Lime Test Series	4/16/71 - 4/20/71
D-XX	Key West - Dolomite Test Series	4/20/71 - 4/28/71
PC-XX	Key West - Precipitated Calcium Carbonate Test Series	4/27/71 - 4/30/71
X-XX	Key West - Recycled Limestone Test Series	4/29/71
P-XX	Paducah - Shawnee No. 9 Limestone Test Series	6/14/71 - 8/6/71
PS-XX	Paducah - Shawnee No. 9 Simulated Key West Limestone Test Series	7/10/71
PA-XX	Paducah - Shawnee No. 9 Aragonite Test Series	7/9/71 - 8/3/71
IPA-XX	Paducah - Shawnee No. 10 Aragonite Injection Test Series	6/23/71 - 6/25/71
IP-XX	Paducah - Shawnee No. 10 Limestone Injection Test Series	7/14/71 - 7/28/71

Fredonia Valley Limestone (FVL), the second principal reactant to be evaluated in this study, was used as a primary standard in gauging the scrubbing efficiency of the coral slurry. As with test series C-XX, a half factorial test design was followed during F-XX series to gain maximum information concerning scrubber operation.

Test series HL-XX was conducted with a lime slurry which normally produced  $\text{SO}_2$  removal efficiencies in the 90% and greater range. The purpose of this test series was to establish the maximum  $\text{SO}_2$  removal efficiency obtainable with the ZASD scrubber design. However, lime slurry was considered a secondary reactant for this program due to the high purchase costs.

A fourth reactant, dolomite, was investigated during test series D-XX as a potential scrubbing agent possessing lower reactivity and high magnesium oxide content.

Precipitated calcium carbonate, considered a secondary reactant for these test purposes, was used as the  $\text{SO}_2$  scrubbing agent during test series PC-XX. This material is a highly reactive calcium carbonate material and was therefore included in this program.

A special test run (X-XX) was performed with a slurry derived from "spent" coral-limestone material that had made one pass through the scrubber. The purpose of this experiment was to investigate the availability of unused calcium carbonate in a "spent" slurry.

The pilot plant was relocated to a coal-fired power plant in Paducah, Kentucky, after the Key West series had been completed. Scrubber operating conditions during these test series duplicated or simulated those during the Key West tests. In addition, several new conditions were investigated.

The first Paducah test series (P-XX) studied the effect of using a Fredonia limestone and untreated river water slurry in removing  $\text{SO}_2$  from flue gases generated by a coal-fired power plant. During the

majority of the test runs, a fly ash collector preceded the scrubbing system; however, several tests were conducted with the dry collector bypassed to investigate its effect on the total system. A half factorial statistical design was followed during this test series.

A secondary test series (PS-XX) was conducted in Paducah to investigate the effects of slurry ionic strength on  $\text{SO}_2$  removal efficiency. An attempt was made to simulate the Key West salt water slurry by adding rock salt to river water.

A new reactant was introduced into the program in Paducah during the PA-XX test series. The use of aragonite during the Key West tests had been considered; however, it was unavailable in sufficient quantities or desired grind size. Aragonite was reported as a highly reactive calcium carbonate material possessing a crystal lattice structure different from the limestone or coral and was therefore included in the Paducah test series.

A brief two day experimental test series (IPA-XX) was designed to investigate the effect of injecting dry aragonite into the boiler while attempting to remove  $\text{SO}_2$  gases in the scrubber with river water. Similar tests were conducted with dry limestone during test series IP-XX.

An addendum to this report, available through EPA's Control Systems Laboratory, contains the raw data collected during the course of this study.



## SECTION 2

### PILOT PLANT EQUIPMENT

The flexible design of the pilot plant allowed for modifications to suit the varied physical configuration expected at each of the plant sites. Discussed in this section are the physical arrangements and specifications of system components as installed in Key West and Paducah, general descriptions of the operation of each component, and descriptions of the sampling and laboratory facilities.

#### PILOT PLANT

A mobile 8 x 40 foot<sup>\*</sup> flat bed trailer supported the entire pilot plant, including a scrubber, a primary particulate collection device, pumps, mixing tanks and a test house. After utilities and duct connections to the flue gas system were provided, the pilot plant became self-sufficient. Figure 2-1 illustrates the mobility of the system and includes pertinent design information.

#### PARTICULATE REMOVAL SYSTEM

Approximately 1% of the flue gas leaving the boiler passed through a primary particulate removal system (Figures 2-1 and 2-2). In theory, the MTSA-3-9 CYTOCD Whirlex collector (Figure 2-2) removed particles by centrifugal action and change in air flow direction. The unit was designed for primary removal of larger size particles generated during the coal-fire phase and was not intended to meet the high removal efficiencies required by stringent emission regulations. Since the Key West tests (dealing with oil-fired flue gas) and the Shawnee No. 10 phase (investigating limestone injection) did not require particulate removal, the collector was bypassed by means of a built-in damper. However, the particulate removal system was utilized during a greater part of the Shawnee No. 9 phase of testing.

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\* Although EPA policy is to use metric units in all of its documentations, certain non-metric units are used in this report for clarity. Readers more familiar with metric units may use the conversion factors in Appendix E.

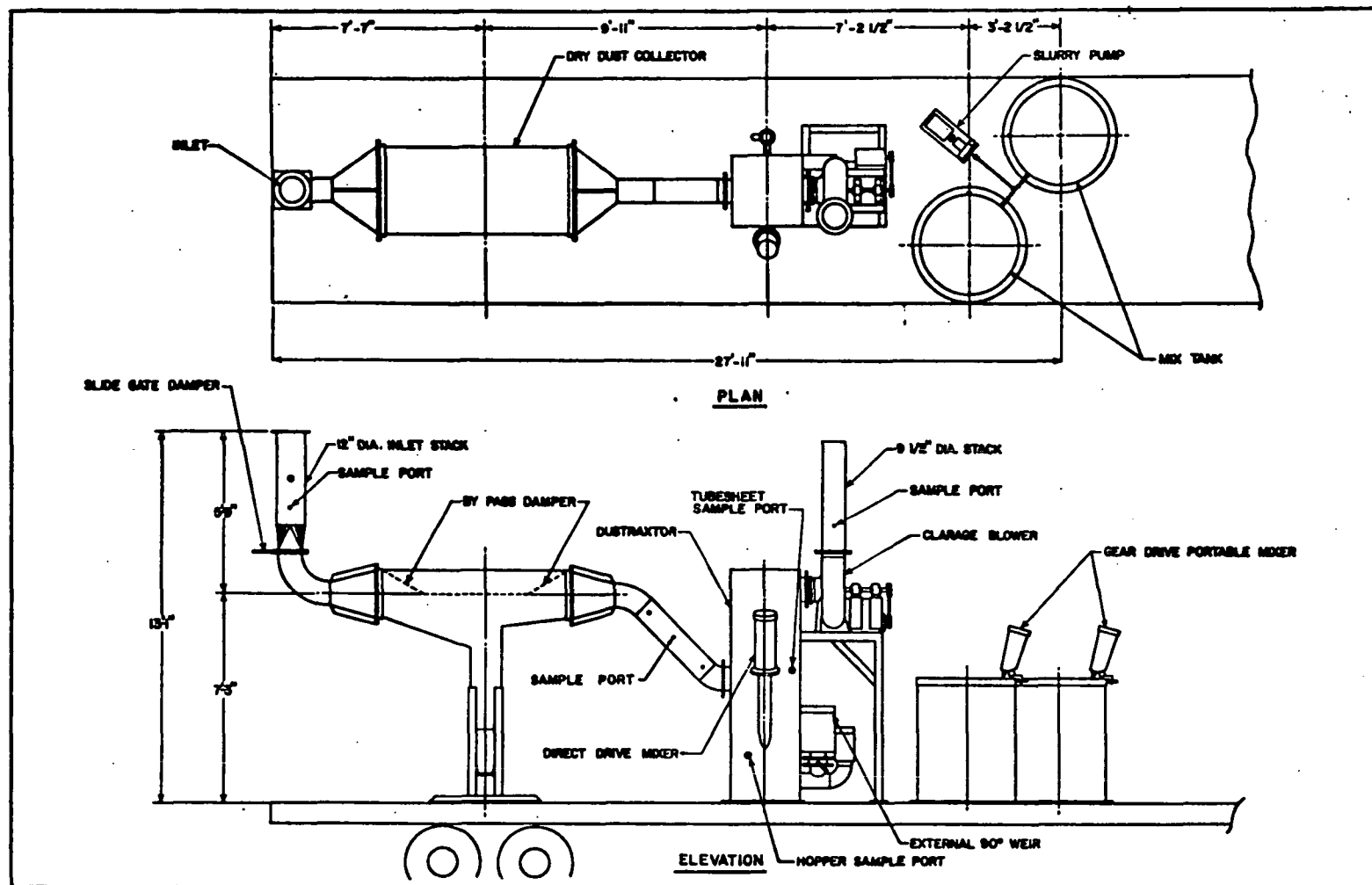


FIG 2-1 MOBILE PILOT PLANT EQUIPMENT ARRANGEMENT

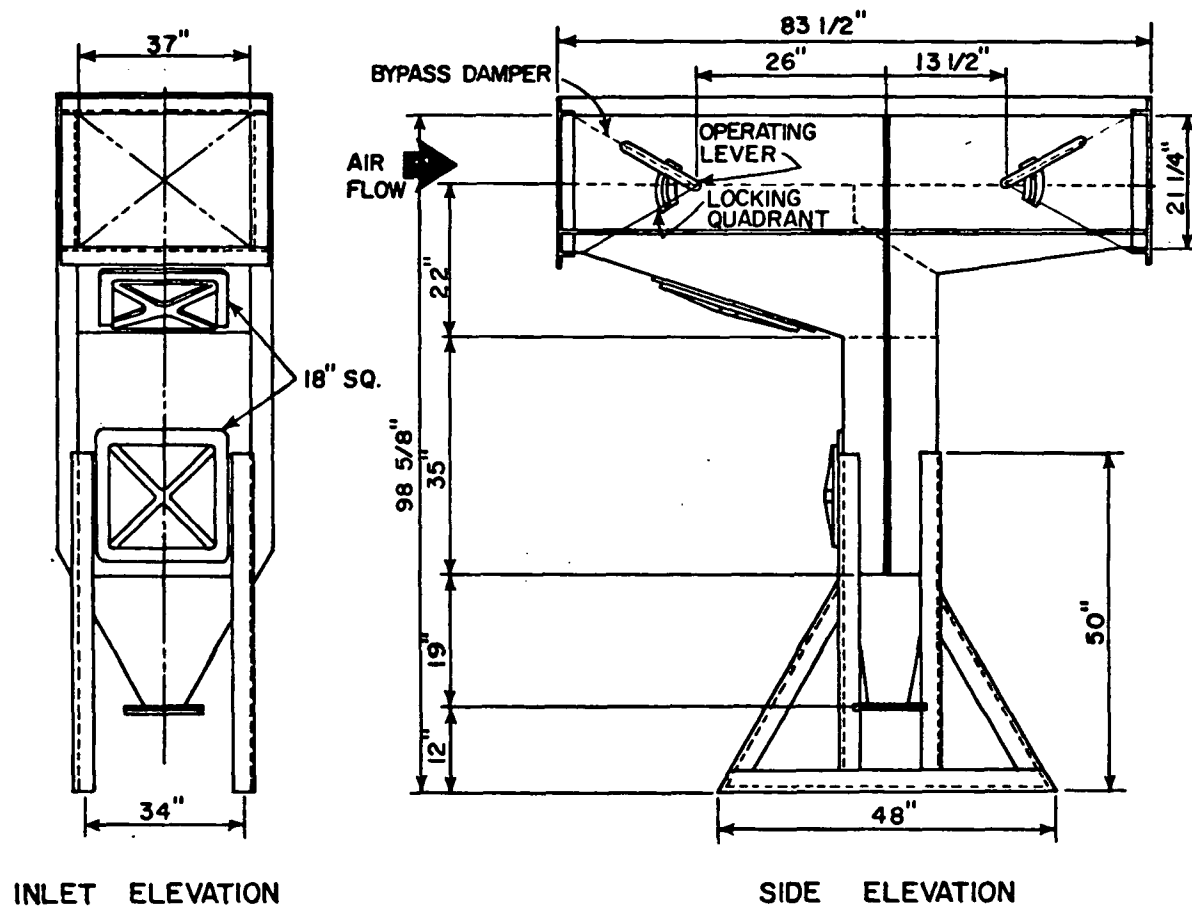


FIG. 2-2 PILOT PLANT DRY DUST COLLECTOR OUTSIDE DETAILS

The material of construction was carbon steel which appeared to be satisfactory for this application. However, the bypass damper for the scrubber system, located on the inlet side of the collector, was made of 304 stainless steel. This damper served an on-off function only and was not used to control the flue gas flow rate to the particulate collector. Sample ports were provided in the connecting ductwork for particulate,  $\text{SO}_2$ , and  $\text{NO}_x$  sampling across the collector as well as across the entire system.

Fly ash removal was accomplished by removing the bolted sealed blind flange while the system was down. Because the collector and ductwork were not insulated, condensate built up in the fly ash hopper. It was necessary to drain this section daily, prior to start-up.

#### DUSTRAXTOR ABSORBER SYSTEM

The objective of scrubbing for  $\text{SO}_2$  was to economically transfer maximum quantities of the contaminant from the flue gas to the scrubbing liquid. Physical and chemical equilibrium and rate relationships all limit the mass transfer of  $\text{SO}_2$  in any system. However, the amount of mass transfer depends not only upon the equilibrium relationships but also upon the contacting scheme. The Dustractor employs many contacting schemes.

Gas leaving the particulate collector passes into a modified single-tube Dustractor. As shown in Figure 2-3, the design is essentially a type of turbulent contact absorber (TCA). The unit consists of a flooded collecting tube through which the flue gas passes. The collecting tube is installed vertically in the inlet plenum chamber directly above the recycle hopper so that the bottom of the tube is a short distance above the liquid level in the hopper.

Flue gas enters the inlet plenum chamber, sweeps over the surface of the scrubbing liquid, and is directed up the collecting tube.

The gas passing between the collecting tube inlet and the collecting bonnet carries scrubbing slurry from the bonnet surface upward into

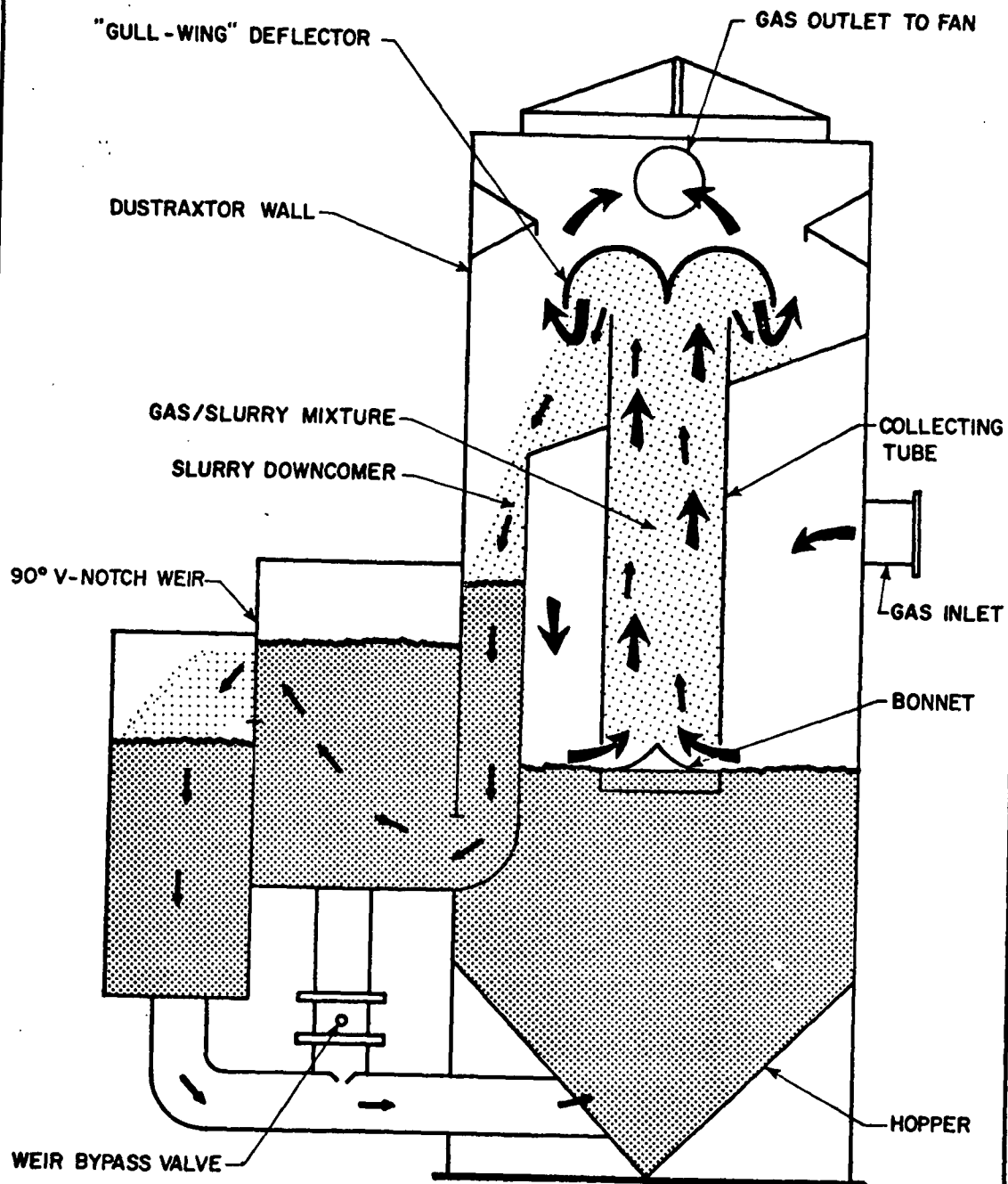


FIG. 2-3 TYPICAL CROSS SECTION OF DUSTRACTOR SCUBBER

the collecting tube. The shearing action of the gas atomizes the scrubbing slurry into a dense spray as the gas slurry mixture continues up the collecting tube. The result of this action is a highly turbulent mixing zone which provides the intimate contact necessary for the chemical reaction to occur. As the gas is discharged from the collecting tube, it is directed into a curved gull-wing deflector which acts as a mechanical separator and forces the slurry downward onto the tube sheet. This shower effect provides an additional mixing zone in which absorption can occur. The cleaned gas is then discharged via the flue gas exhaust system as the scrubbing solution is returned by gravity to the recycle hopper. To summarize, the stages of gas-liquid contact in this type of scrubber are:

1. The initial shearing action as the flue gas passes through the slot between the collecting bonnet and tube.
2. The highly turbulent mixing within the collecting tube where the gas is in intimate contact with slurry droplets.
3. The impingement of the gases and slurry upon the surface of the deflector.
4. The passage of gas through a highly turbulent curtain of slurry being discharged from the deflector.

The contacting schemes found in the Dustraxtor could be categorized as countercurrent, co-current, and crosscurrent flow. Since there were three contacting schemes occurring at the same time in this scrubber, a theoretical analysis of the total mass transfer mechanism would have been extremely difficult and only of academic interest. Therefore, this type of an analysis was not conducted.

The Dustraxtor dimensions are shown in Figure 2-4. This unit differed from the typical Dustraxtor design in the following ways:

1. An exterior weir system was added between the tube sheet downcomer and the hopper.
2. The capability was added to allow alteration of the tube diameter by substitution of different diameter prefabricated tubes.



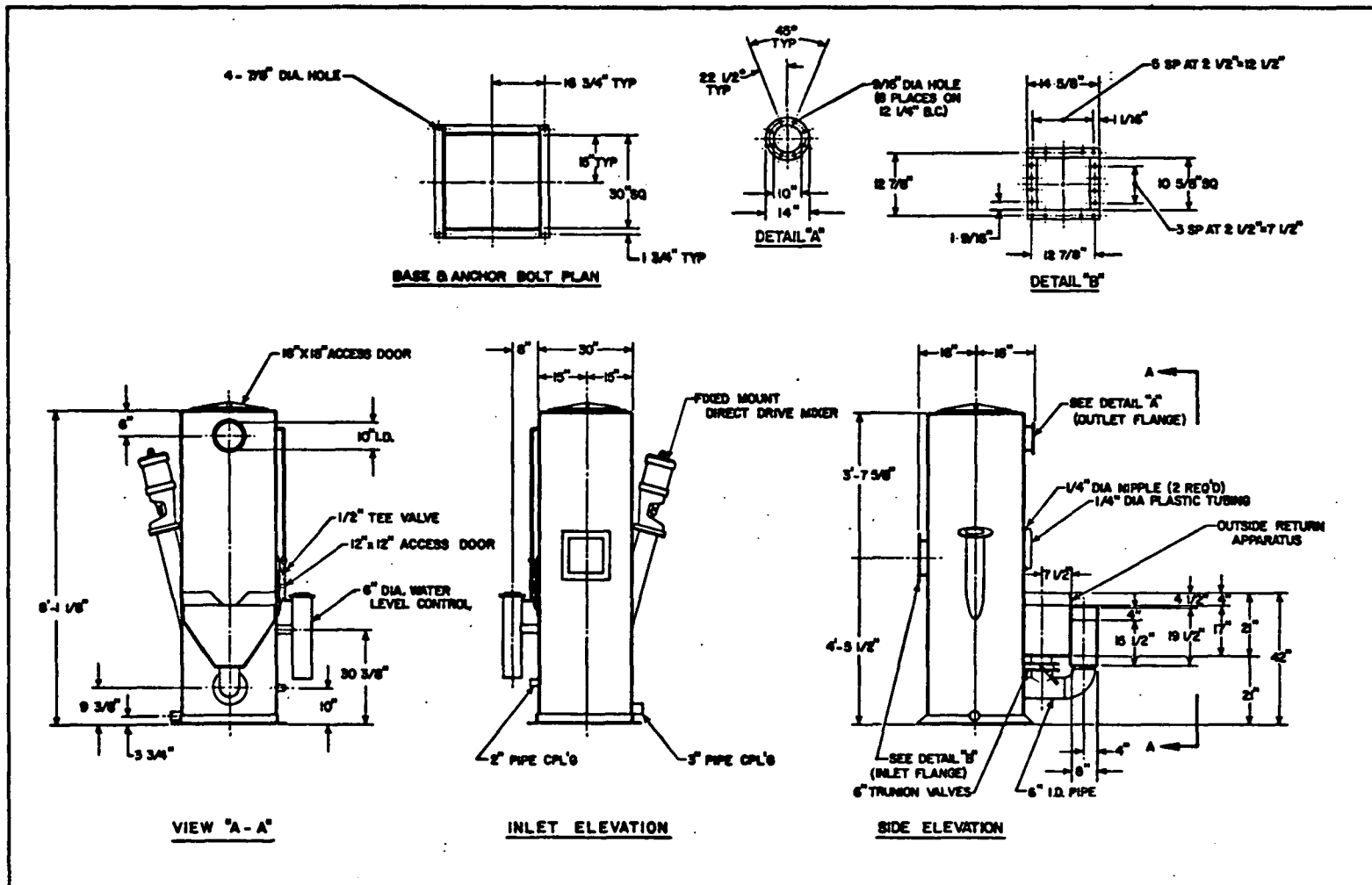


FIG 2-4 PILOT PLANT SCRUBBER (DUSTRAXTON) DETAILS

The exterior weir provided a means of measuring the quantity of slurry taken up into the tube, which was later used to calculate the liquid/gas ratios. The details of the external return apparatus and the 90° "V-notch" weir are shown in Figure 2-5.

The liquid level in the scrubber was maintained by a level controller. The slurry composition was maintained indirectly by adjustment of the spent slurry flow rate. The level controller in turn maintained the desired liquid level by adjusting the fresh slurry feed rate.

In order to avoid problems with solids settling, a mechanical mixer was installed in the Dustractor to help keep solids in suspension. The mixer specifications are listed in Table 2-1. The sump had a 3 inch gravity-feed drain which was used to empty the scrubber after each test.

Table 2-1. DUSTRACTOR MIXER SPECIFICATIONS

---

<u>Trade Name:</u>	Lightnin Mixer
<u>Manufacturer:</u>	Mixing Equipment Company, Inc. 138 Mt. Read Boulevard Rochester, New York 14603
<u>Model:</u>	N33-33, Fixed Mounted Propeller Type
<u>Serial No.:</u>	7012653
<u>Design Specifications:</u>	Motor - 1/3 HP/115V/60 Hz/1 phase/1750 rpm, totally enclosed Shaft - 304 SS, 3/4 inch diameter x 49 inches, 1750 rpm Propeller - 316 SS, 3.8 inch diameter

---

The material of construction for the Dustractor and connecting ductwork to the particulate collector was 304 SS. All piping to and from the scrubber was galvanized steel. All sample ports on the scrubber were either stainless steel or polyvinyl chloride. A stainless steel butterfly valve, located in the bottom of the exterior weir, was used to bypass the measurement system.

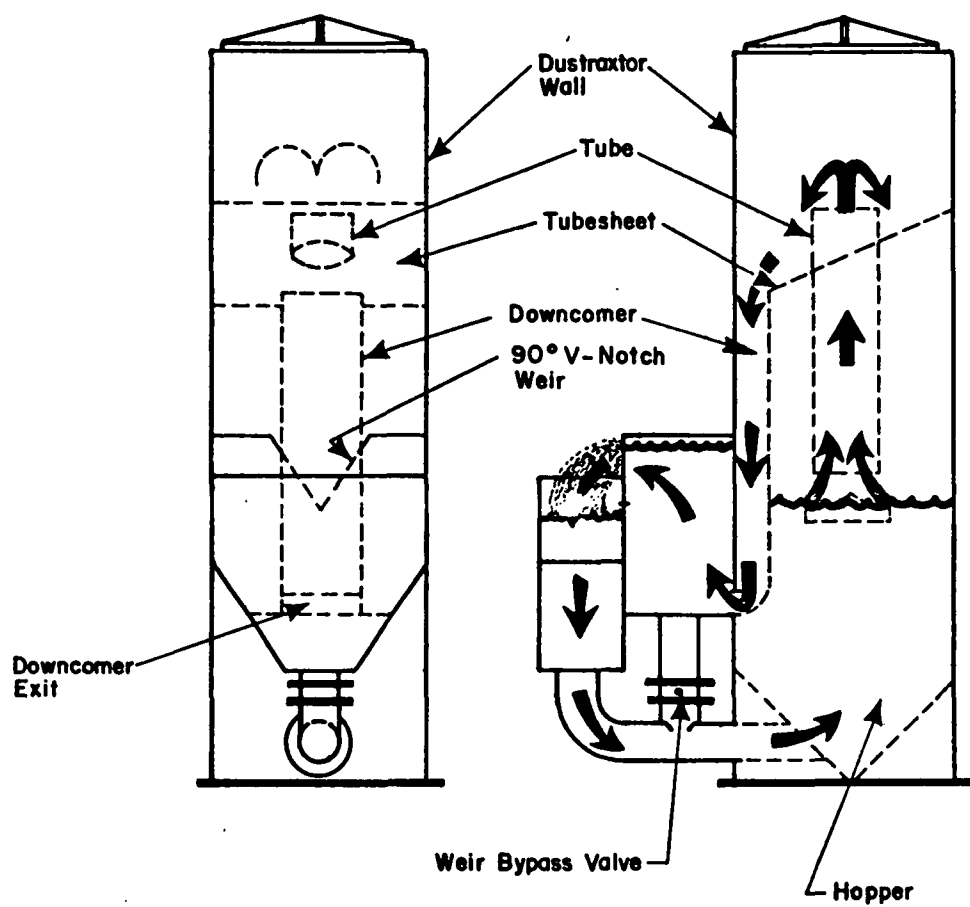


FIG. 2-5 DUSTRACTOR OUTSIDE RETURN APPARATUS AND 90° V-NOTCH WEIR

A slide gate valve, installed on the 10-inch diameter discharge duct was used to control the flue gas flow rate through the entire pilot plant. The pressure drop across the Dustractor was controlled by adjusting the balance vent line control valve. Sample ports were provided in the connecting ductwork for particulate,  $\text{SO}_2$ , and  $\text{NO}_x$  sampling across the Dustractor, as well as across the entire system. Liquid sample ports were provided on the Dustractor body for tube sheet and hopper liquor sampling. A set of spray nozzles for gas saturating purposes was installed in the ductwork between the particulate collector and the Dustractor.

#### FLUE GAS EXHAUST SYSTEM

The scrubbed flue gas leaving the Zurn scrubber passed through an induced-draft fan with an inlet damper control, to a vertical stack containing sampling ports. The induced-draft fan was a No. 10 Clarage Blower; the fan curve is shown in Figure 2-6. The I-D fan had a paddle-blade impeller which, because of particulate carry-over and subsequent "plating-out" on the blades, became unbalanced and failed twice during the study. A field review of the I-D fan failures indicated that the cast stainless steel spider, which secures the blade, fractured due to fatigue caused by the fan imbalance.

#### PILOT PLANT SCHEMATIC

Figure 2-7 shows the overall flow diagram of the pilot plant. The location of thermometers and flow measuring devices are also shown in this Figure. Additional information concerning the monitoring system is included in Section 3: Test Instrumentation and Procedures.

#### REACTANT HANDLING AND WASTE DISPOSAL SYSTEMS

The calcium based scrubbing slurry was prepared in mix tanks. The mix tanks were equipped with steam coils and gear driven portable mixers.

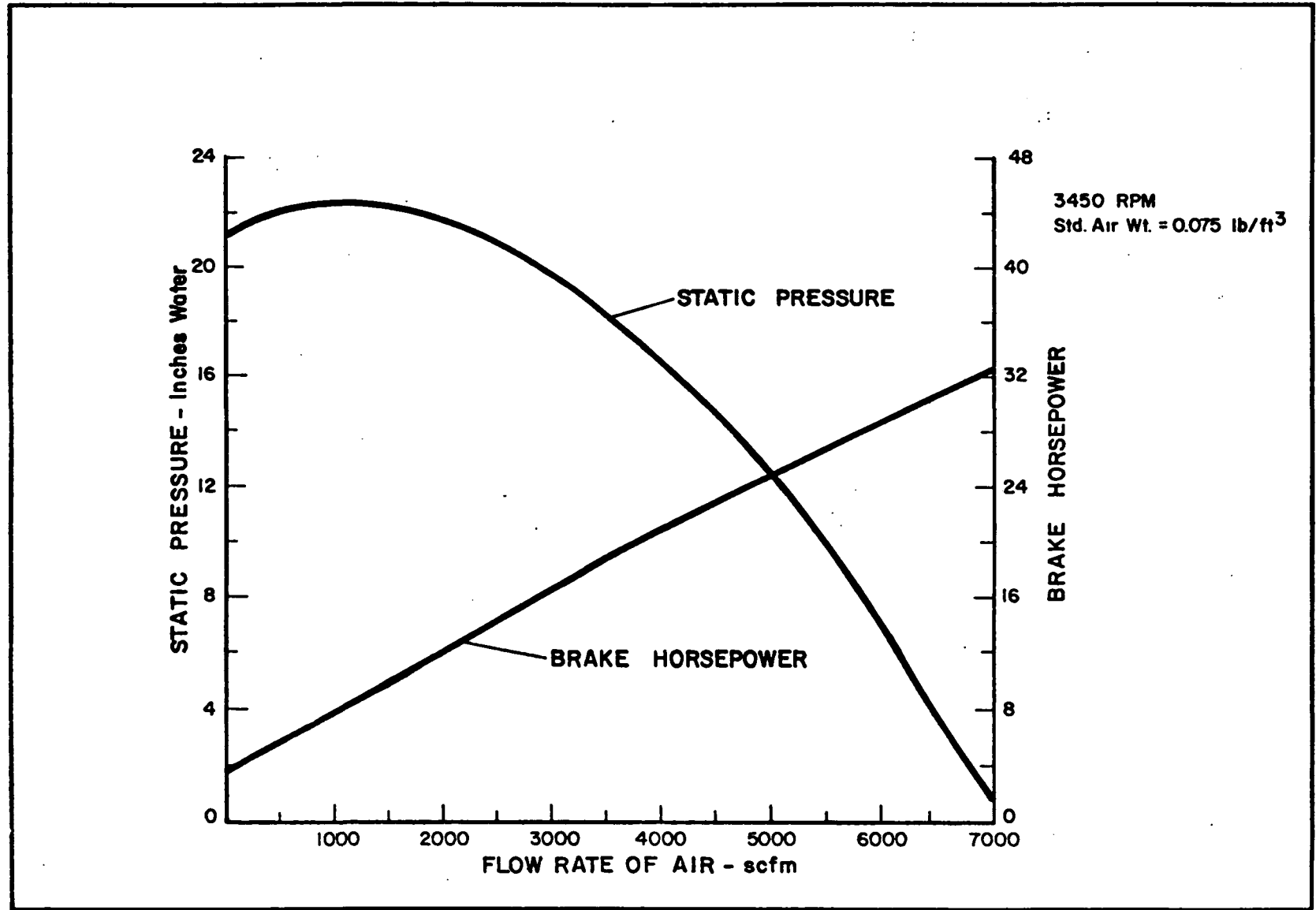


FIG. 2-6 NO. 10 CLARAGE BLOWER INDUCED DRAFT FAN CURVE

The steam coils were not used during the study; however, they were available for use if desired. The mixers had a 304 stainless steel shaft, a 316 stainless steel 10 inch diameter propeller, and were driven by a 1/3 HP, 115 volt, 1750 rpm, totally enclosed electric motor. Detailed tank mixer specifications are shown in Table 2-2.

Table 2-2. TANK MIXER SPECIFICATIONS

<u>Trade Name:</u>	Lightnin Mixer
<u>Manufacturer:</u>	Mixing Equipment Company, Inc. 138 Mt. Read Boulevard Rochester, New York 14603
<u>Model:</u>	ND-1, Portable Mixer
<u>Serial No.:</u>	None, Mixco Order No. M315836
<u>Design Specifications:</u>	Motor - 1/3 HP/115V/60Hz/1 phase/1750 rpm, totally enclosed  Shaft - 304 SS, 5/8 inch diameter x 48 inches, 1750 rpm  Propeller - 316 SS, 10 inch diameter

The scrubbing slurry from the mix tanks was fed by gravity to a high silicon iron Mark II Durcopump manufactured by the Duriron Company, Inc. The pump specifications are noted in Table 2-3. The slurry feed rate from the pump to the scrubber was measured by a flowmeter located near the scrubber. Excess slurry was returned to the mix tanks through a recirculation line shown in Figure 2-7.

Table 2-3. SLURRY PUMP SPECIFICATIONS

<u>Manufacturer:</u>	Duriron Company, Inc. N. Findlay and Thomas Streets Dayton, Ohio 45401
<u>Pump Design:</u>	Series - 1-1/2 x 1 H - 6/60; Size - Mark II GPI Packing - Standard; Alloy - Superchlor Impeller Diameter - 6 inches; Shaft Wet End - Superchlor
<u>Performance:</u>	20 gallons per minute 36 feet of water total differential head NPSH - 1.5 feet of water net positive suction head 0.9 maximum brake horsepower

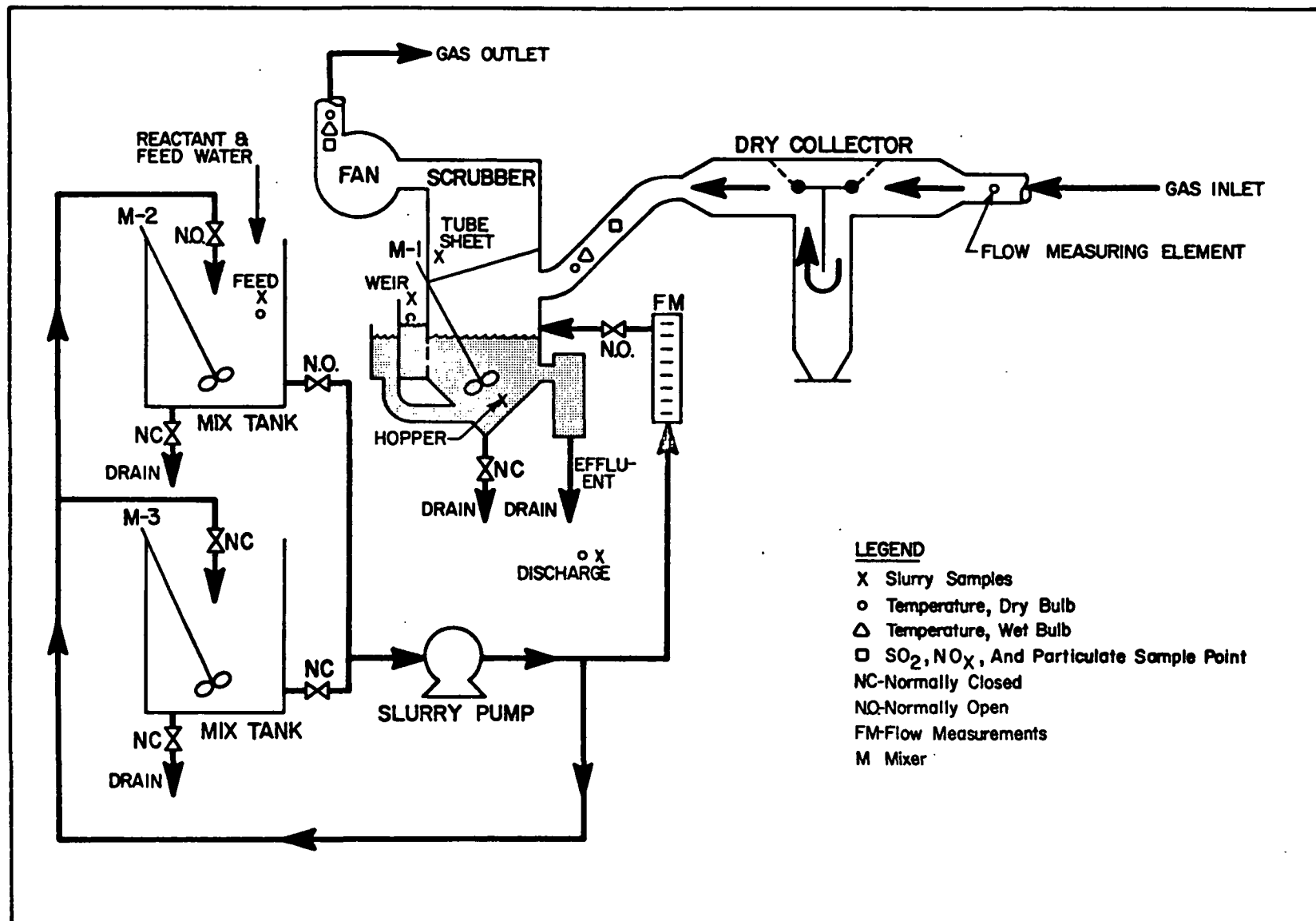


FIG. 2-7 SCHEMATIC FLOW DIAGRAM OF FLUE GAS AND SLURRY SOLUTIONS WITH LOCATIONS OF SAMPLING POINTS

The required quantity of the calcium-based reactant was weighed out and dumped into each mix tank containing fresh or salt water depending upon the location of the pilot plant. The amount of reactant was set by the required slurry concentration. Adequate time was allowed for proper mixing of the calcium-based reactant and the water before pumping into the scrubber system. Fresh slurry was fed to the scrubber through the flowmeter while the spent slurry, from the scrubber system, passed out the level controller.

During the Key West testing, the spent effluent was deposited in a holding pond where the liquid was allowed to evaporate or permeate the coral soil. During the Shawnee testing, the effluent was deposited in a storm sewer, mixed with other plant waters, and pumped to the Mississippi River. Each disposal technique was satisfactory for the pilot scale system.

Several operating problems concerning the reactant handling system were noted during the study. Due to infrequent maintenance checks, the feed pump packing developed a leak (a marked reduction of pump pressure was experienced) and became progressively worse as the project continued. A more serious problem was encountered at the Shawnee site. Settleable solids in the waste material often clogged the flexible hoses. This necessitated frequent flushing with a fresh water stream to prevent back-up into the scrubber.

#### CHEMICAL ANALYSIS LABORATORY

During the Key West testing, the City Electric System power plant laboratory was utilized for all chemical analyses. The laboratory was equipped with instrumentation necessary for conducting the analyses noted in Appendix A.



### SECTION 3

#### TEST INSTRUMENTATION AND PROCEDURES

In order to facilitate movement of the pilot plant, basic but highly reliable instrumentation was utilized for this study. The  $\text{SO}_2$  concentrations were determined with an electrochemical type sensor which was calibrated daily with guaranteed-analysis calibration gas. All temperatures were determined with properly calibrated thermometers, and gas flow rates were determined with a calibrated sharp-edge orifice. Standard sampling techniques were employed for particulates,  $\text{SO}_2$ , and  $\text{NO}_x$ . Details of the analytical techniques are in Appendix A.

Test procedures were established based upon the experimental design required for each phase of the test program. Day-to-day operation of the pilot plant was held as constant as possible, with the only variation occurring in the level of the variable set for each test. The test procedures employed at the two sites, Key West and Shawnee, were varied only where necessary in order to comply with necessary conditions.

#### $\text{SO}_2$ MONITORING

One of the most important analyses of the test program was the measurement of the  $\text{SO}_2$  concentration in the flue gas.  $\text{SO}_2$  sampling probes, fabricated from 6 inch lengths of 1/4 inch diameter SS tubing, were located in the entrance and discharge ducts of the Dustractor. Analyses were performed for the most part by a Dynasciences Model SS-330 monitor. Selection of this monitor was based in part on the results of limited test work conducted earlier.

The Dynasciences monitor operated on the principle of a fuel cell.  $\text{SO}_2$  was absorbed on a sensing electrode to form activated species capable of undergoing electro-oxidation. The resulting current was directly proportional to the partial pressure of  $\text{SO}_2$  in the gas mixture. The current was amplified and the output recorded on a meter and a 10 mV recorder.

The instrument specifications indicated linear response over the entire range of  $\text{SO}_2$  concentration. Overall accuracy was specified at  $\pm 1\%$  full scale with the use of an external potentiometric recorder. Response time was specified as 90% of full scale in 1 minute. The instrument was to exhibit no response to  $\text{N}_2$ ,  $\text{O}_2$ ,  $\text{NO}_x$ ,  $\text{CO}$ ,  $\text{CO}_2$ , water vapor or hydrocarbons. In addition, the instrument was very portable and well suited to the field environment experienced in pilot plant operations.

In operation, the Dynasciences Model SS-330 did not meet all the specifications. Response time was not as rapid as claimed; the instrument was sensitive to changes in sampling gas flow rate (slight changes in operating pressure), and changes in ambient temperature. Also, the instrument stability was considerably less than specified. Electrochemical cell life was better than claimed by the manufacturer and once the initial operating problems were corrected,  $\text{SO}_2$  monitoring operations were very reliable. In general, the instrument proved satisfactory for the conditions experienced during the pilot plant operation.

#### $\text{NO}_x$ SAMPLING AND ANALYSIS

$\text{NO}_x$  samples were taken randomly during certain test periods.  $\text{NO}_x$  probes were located at the entrance and discharge ducts of the Dustraxtor virtually in line with the  $\text{SO}_2$  sampling probes. The phenoldisulfonic acid method was used for analysis.

A typical  $\text{NO}_x$  sampling and analysis consisted of adding 25 ml of an absorbing solution (hydrogen peroxide and dilute sulfuric acid) to a 250 ml (nominal) evacuation flask. The flask was evacuated and attached to a purged sample line; the stop cock was opened; and the gas sample was drawn into the flask.

$\text{NO}_x$  was converted to nitric acid by the absorbent solution and reacted with phenoldisulfonic acid to produce a yellow compound which was measured colorimetrically. Color was measured with a photometer and compared with calibration curves from solutions containing a known quantity of nitrate.

A more complete and descriptive explanation of the phenoldisulfonic acid method used in these tests is given in the American Society for Testing and Materials (ASTM), Standard Method of Test for Oxides of Nitrogen in Gaseous Combustion Products (Phenoldisulfonic Acid Procedure), ASTM Designation: D1608-60 (1967). The analytical technique associated with this test procedure was very tedious and time consuming. However, since other procedures had not been adequately demonstrated on power plant flue gases, it was necessary to use this method to obtain reproducible data.

#### PARTICULATE SAMPLING

Particulate sampling was conducted at three points in the pilot plant: the entrance to the dry dust collector, the entrance duct to the Dust-ractor, and the Dustractor exit duct. The sampling train consisted of a stainless steel probe and nozzle, a glass fiber filter and a series of three Greenberg-Smith impingers. During the first attempts at particulate sampling, the glass fiber filter clogged as a result of the high moisture content present in the flue gas (the filter was not heated). The filter medium was therefore eliminated from the train during the remaining tests. The remainder of the particulate sampling system included a gas meter and vacuum pump.

Isokinetic sampling was performed by regulating the sample flow rate to correspond with the calculated velocity at the sampling point. This method was justified over the null balance procedure for this study to conform with suggested EPA test method. Uniform flow rates were demonstrated by observing the flue gas orifice over an extended period of time.

#### SLURRY SAMPLING AND ANALYSIS

During each test, representative slurry samples were taken at the following locations:

- Slurry feed
- Scrubber discharge

- External weir
- Hopper sump
- Tube sheet

Figure 2-6 illustrates the various sampling points. During every test at least one 100 ml slurry sample was taken for analysis of the following parameters:

- pH
- Calcium
- Magnesium
- Chloride
- Nitrate
- Nitrite
- Sulfite
- Sulfate

Sampling was performed in a manner to avoid oxidation; the samples were placed in an ice water bath for transportation to the laboratory. pH was measured in the field as well as in the laboratory. Standard gravimetric, titrimetric, and colorimetric methods were used by the chemist following procedures outlined in American Society for Testing Materials (ASTM) 1970 Annual Book of ASTM Standards, Part 23, Water; Atmospheric Analysis, and the American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 12th Edition. The slurry samples which contained solids and liquid were not separated prior to the analysis.

#### OTHER MEASUREMENTS

Field measurements for pH of the slurry samples were made with a Sargent-Welch Model PBX pH meter and laboratory pH measurements were made with a Sargent-Welch Model LSX pH meter.

Flue gas flow rate was measured by an Ellison Instrument Division, 12 inch "Annubar," type 740. The Annubar is a primary flow element utilizing a form of the classical Bernoulli energy balance equation to determine flow rate. The equation used was:

$$Q_n = 7.9 \text{ SND}^2 \frac{\sqrt{\gamma_f}}{\gamma_1} \sqrt{h_n} \quad (3-1)$$

A full description of the symbols of this equation, and other equations available for use with this flow element, were supplied by Ellison Instrument Division, Boulder, Colorado, and are reproduced in Appendix B. The Annubar included an interpolating tube with equal annuli segments, an equalizing element, and a downstream element for measuring the downstream pressure (static pressure less the impact pressure of the flow). The system had a non-clog design which was desirable for operation in the pilot plant. The unit was calibrated against standard pitot tube traverses prior to initial testing in Key West. Detailed drawings and specifications of the Annubar were supplied by Ellison Instrument Division and are also reproduced in Appendix B.

#### GENERALIZED TEST PROCEDURE

A typical testing day in which three tests were usually completed is described below. The experimental design established the operating levels for the variables under study. These variables were set during the test period.

In preparation for a run with a cold start (that is, the scrubber had been idle long enough to allow the ducts to accumulate condensation), the unit was first filled with salt or fresh water and allowed to run for a minimum of 30 minutes to attain operating temperatures and to flush the accumulated condensate out of the ductwork. Normal conditions for this warm-up and flushing were a gas flow rate of 1000-1450 scfm, liquid feed rate of 10-15 gpm and a  $\Delta P$  of 9-12 in.  $H_2O$ .

While waiting for the system to reach operating conditions, the two mix tanks were flushed and cleaned; the proper quantity of reactant for

the test was calculated, weighed out, and mixed in one tank; and the liquid for the warm-up operation was pumped from the remaining tank. After warm-up, the system was shut-down (fan off, pump off, reactant feed valve off, inlet damper closed) and drained immediately. While the Dustractor was draining, the proper quantity of reactant was weighed out and mixed in the second feed tank forming the scrubbing slurry.

When the scrubber was drained, the drain valve and weir butterfly shut-off valves were closed. Slurry was then pumped into the scrubber at maximum flow rate until the Dustractor was full. The sequence of events concerning the controls were as follows:

1. Hopper mixer - on
2. Reactant flow - 2-5 gpm
3. Level control - 50% open
4. Inlet damper - open
5. Fan - on

Once the system was in operation, the levels were set for the various operating parameters. During the first hour of a test, the gas flow rate, slurry flow rate, and pressure drop were monitored to assure that the unit would approach equilibrium at the predetermined conditions. Based upon preliminary test results, steady state was assumed after the system had been in operation for 2 hours and the  $\text{SO}_2$  concentration at the pilot plant exit remained constant.

Prior to final data acquisition, the  $\text{SO}_2$  monitor was calibrated and the inlet  $\text{SO}_2$  concentration was determined. During this time, the inlet gas conditions were recorded (temperature, flow rate, pressures and  $\text{SO}_2$  concentration). The required stoichiometric ratio based on the inlet  $\text{SO}_2$ , was calculated and adjusted as necessary. The  $\text{SO}_2$  monitor was then used to analyze the Dustractor outlet gas. When the effluent  $\text{SO}_2$  concentration stabilized, outlet conditions were recorded (temperature, flow rate, pressure, and  $\text{SO}_2$  concentration).

During a test, several slurry samples were collected and placed in an ice water bath for storage prior to laboratory analyses. The samples collected and stored in 8-ounce plastic bottles included the following: slurry feed, scrubber discharge, weir overflow, tube sheet, and Dust-raxtor hopper samples.

After a test was completed, operating conditions were changed for the next test. The unit was shut down, drained, and refilled with reactant for another test. There was usually a working period of about 1/2 hour before the system was ready for another test. Following this procedure, about three 2 1/2 hour tests were completed every day.

#### TEST PROCEDURE FOR EXTENDED OPERATION

The start-up for extended operation did not differ from that of a normal test day. Test conditions were specified to be held constant for approximately an 8 hour period. No variables were changed during this test period.

#### TEST PROCEDURE FOR ADDITIVE OPERATION

The start-up for additive experiments (catalyst-- $\text{FeCl}_3$  or inhibitor--hydroquinone) did not differ from that of a normal test day.

All experimental procedures outlined above were followed with the following additions and modifications:

1. Two identical reactant tanks were mixed: one contained the test additive, the other did not.
2. The scrubber was filled and started up on reactant without the test additive.
3. Inlet and outlet conditions were recorded as outlined earlier. During some experiments, scrubber effluent samples were not taken at this time.
4. After stabilization of the outlet conditions, the correct amount of additive was added to the scrubber weir simultaneously with changing the reactant feed to the tank with additive. The feed rates remained constant.

5. The outlet conditions were allowed to re-stabilize after additive addition; they were recorded again and scrubber effluent samples were collected.



## SECTION 4

### SUMMARY OF RESULTS

Results of both the short term and extended tests were consolidated into one set of results for each test site. Because of the great volume of data gathered during the test program, only a summary of the results is presented in this report. An addendum, available through EPA's Control Systems Laboratory, contains all test data taken during the 10 months of field work. Appendices C and D contain scrubber operating conditions and data summary tables, respectively.

#### SYSTEM OPERATING CONDITIONS AND CHARACTERISTICS

A summary of the important system operating conditions and characteristics including data important to future design and optimization of the Dustraxtor absorber is given below.

##### Liquid Entrainment Relationships

The relationships of the parameters governing liquid entrainment in the Dustraxtor unit are presented in Figures 4-1 and 4-2. Approximately 170 data points are represented showing the association between the liquid entrainment and gas flow rate at selected pressure drops ( $\Delta P$ ) from 6.5-14.0 in.  $H_2O$ .

Figure 4-1 applies to a single 12-inch diameter Dustraxtor tube. Each curve, representing a constant pressure drop condition, exhibits a point of maximum liquid entrainment which becomes more apparent as pressure drop increases. Thus, at a  $\Delta P$  of 6.5 inches, a  $\pm 350$  scfm variation in gas flow rate about the maximum entrainment point results in a 15 gpm reduction in liquid entrainment. Compared to this, at a  $\Delta P$  of 12.0 inches, a decrease in entrainment by 15 gpm is caused by a gas flow variation of only  $\pm 170$  scfm. Another characteristic of this maximum point is its tendency to occur at progressively lower gas flow

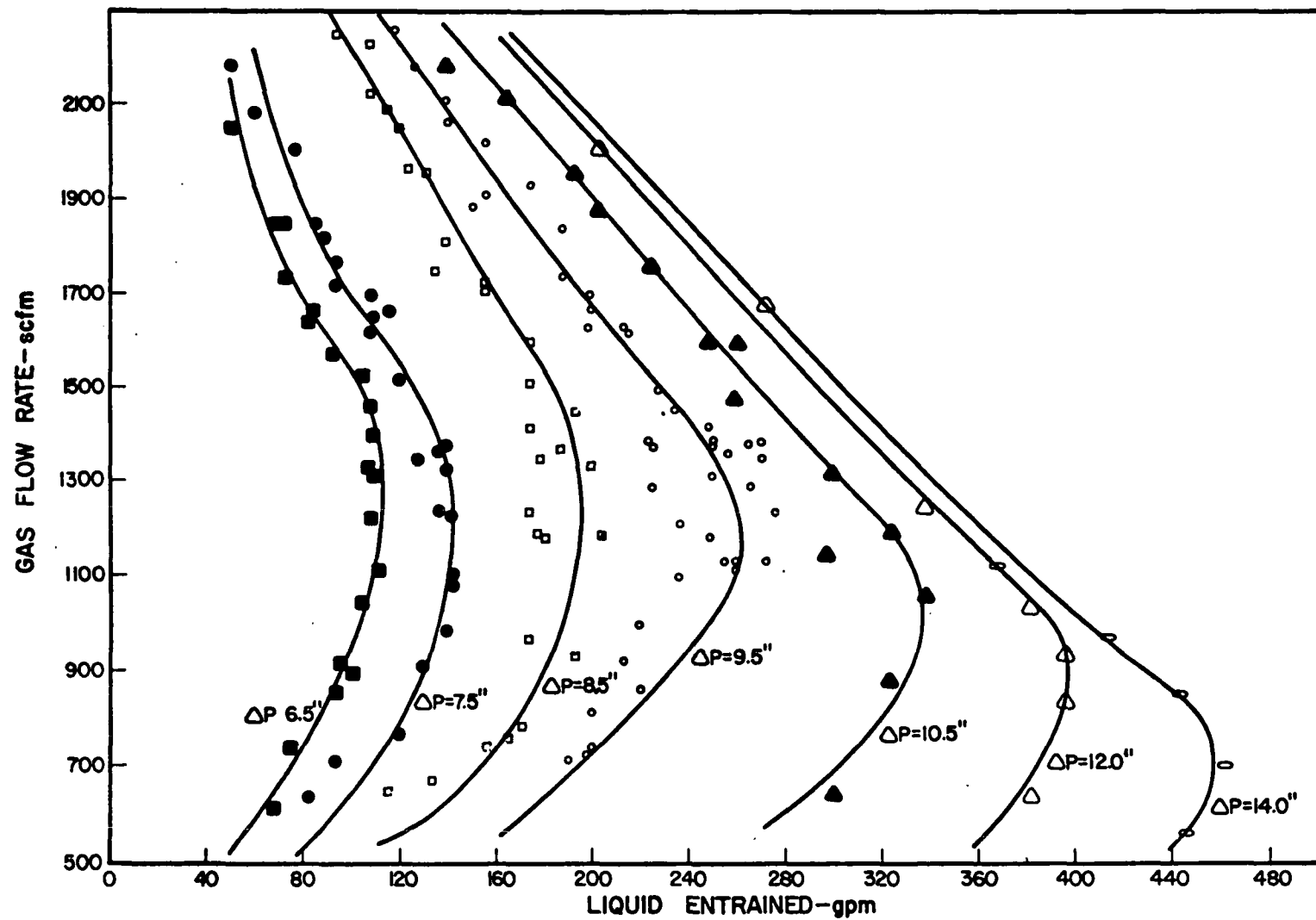


FIG. 4-1. SALT WATER ENTRAINED IN THE 12" TUBE AT SELECTED PRESSURE DROPS AND GAS FLOW RATES

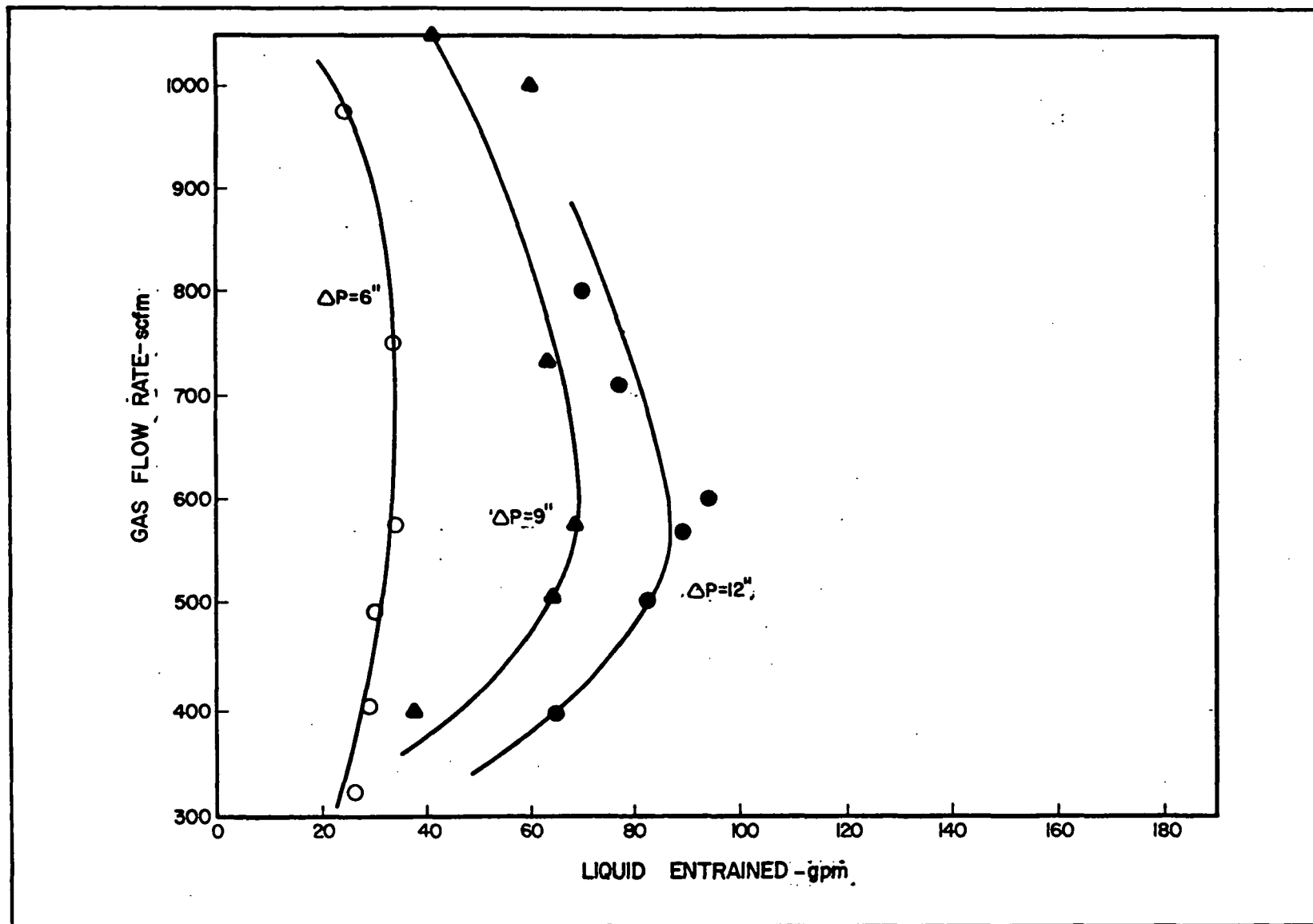


FIG. 4-2. RIVER WATER ENTRAINED IN THE 8" TUBE AT SELECTED PRESSURE DROPS AND GAS FLOW RATES

rates as pressure drop increases. As an illustration, a maximum entrainment of 112 gpm is realized at about 1300 scfm for a 6.5 inch  $\Delta P$ . At a 12 inch  $\Delta P$  the maximum entrainment (396 gpm) has dropped to 900 scfm and at a 14 inch pressure drop the maximum has fallen to 700 scfm, nearly half of the flow rate and velocity of the gas stream for the 6.5 inch  $\Delta P$  maximum.

As gas flow rate and corresponding gas velocity increase above the maximum entrainment point, all of the curves converge upward toward a greatly reduced range of entrainment volumes. This trend is so pronounced that if assumed to continue at the same rate, extrapolation of the data would indicate a minimal gain of 50 gpm in liquid entrainment at a 2700 scfm flow rate when the  $\Delta P$  changed from 6.5 to 14.5 in.  $H_2O$ .

Figure 4-2 is a corresponding plot of the scrubber characteristics utilizing a single 8 inch diameter Dustractor tube. The same general shape and trends appear for the 8 inch tube as were exhibited for the 12 inch tube. However, the maximum entrainment points occurred at lower gas flow rates and higher gas velocities and were 1/2 to 1/4 the order of magnitude found for the 12 inch Dustractor tube.

No attempt has been made to analyze the actual physical mechanism of entraining and lifting the slurry from the surface within the hopper or its transportation through the scrubber; hence, a complete understanding of the dynamic principles illustrated by the data of Figures 4-1 and 4-2 is not possible. The development of a model to establish the analytical relationship of liquid entrainment as a function of  $\Delta P$  and gas flow was not attempted.

#### Liquid/Gas Ratios

Figure 4-3 is a plot of liquid/gas ratios versus gas flow rates for both the 8 inch and 12 inch Dustractor tubes. Two corresponding pressure drops are depicted for each size tube (9 and 12 inches for the 8 inch

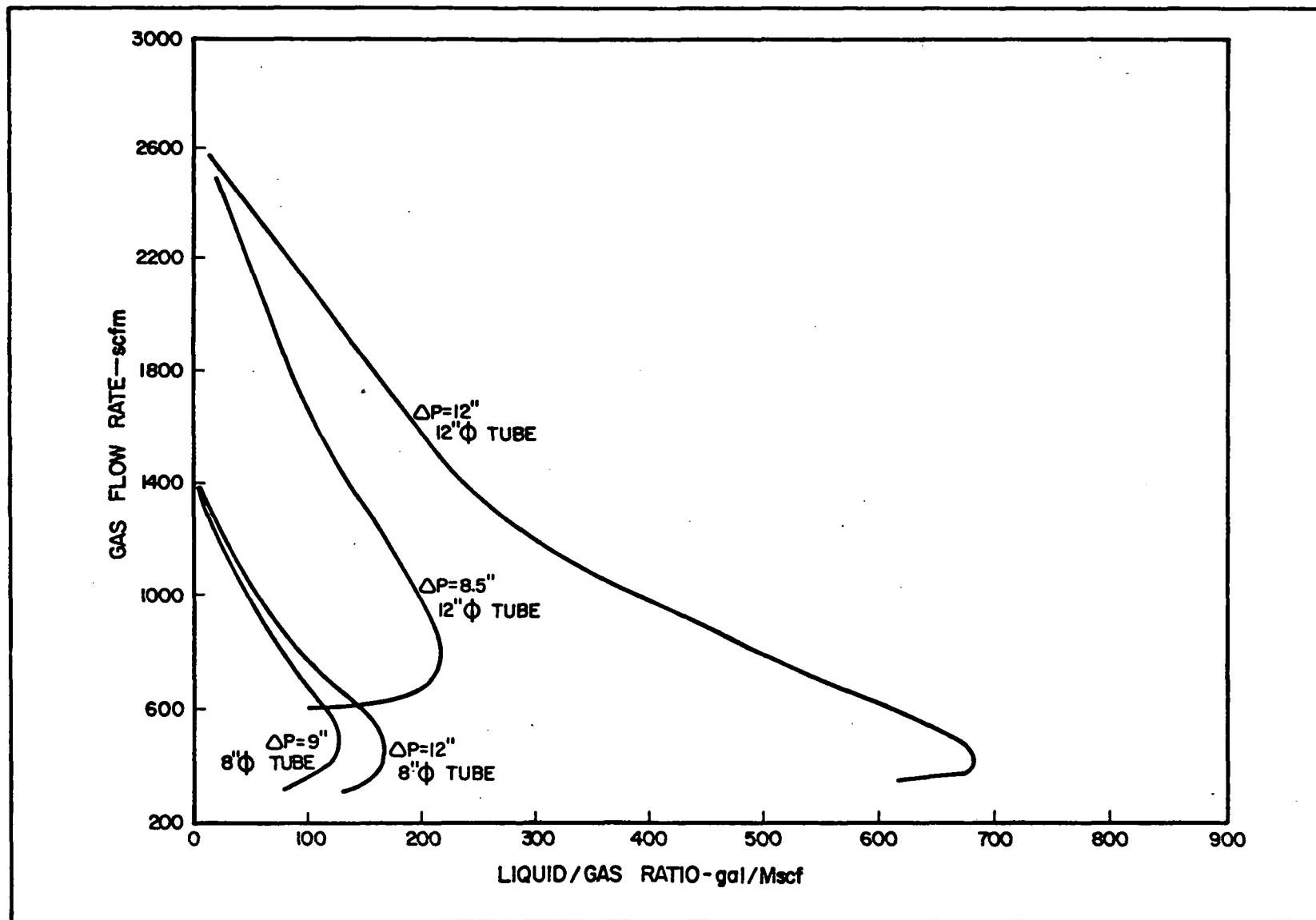


FIG. 4-3. LIQUID/GAS RATIOS FOR 8" & 12" DIA. DUSTRACTOR TUBES AT SELECTED PRESSURE DROPS AND GAS FLOW RATES

diameter tube; 8.5 and 12 inches for the 12 inch diameter tube). These curves were wholly derived from the data points shown in Figures 4-1 and 4-2. For the sake of clarity, the actual data points have been eliminated. All of the curves show an expected maximum liquid/gas ratio at some relatively low gas flow rate. The magnitude of this maximum increases with increasing values of  $\Delta P$ ; however, the point of maximum liquid/gas ratio occurs at a slightly lower gas flow rate than the maximum entrainment volume (Figure 4-1 and 4-2). As an example: maximum entrainment volume for a 12 inch  $\Delta P$  and a 12 inch Dustraxtor tube occurs at about 850 scfm while the maximum liquid/gas ratio, 680 gallons per thousand cubic feet (gal./Mcf), occurs at a significantly lower gas flow rate of about 420 scfm. Also at high flow rates the liquid/gas ratio seems to fall off very rapidly, approaching zero for the 12 inch tube at 2700 scfm and at 1400 scfm for the 8 inch tube. Therefore, from Figure 4-3, it would appear that liquid/gas ratio is independent of pressure drop at higher gas flow rates.

Finally, Figure 4-3 exhibits the same trend as Figures 4-1 and 4-2 regarding tube diameter; that is, increasing tube diameter results in an increased liquid/gas ratio.

#### Characteristics and Analysis of Feed Reactants and Discharge

While lack of sufficient instrumentation and funds prevented a detailed study of the chemical reaction kinetics and mechanisms occurring during the scrubbing process, analyses were made of the chemical constituents important to the understanding of the chemical processes occurring in the Dustraxtor absorber. These analyses were made for pH,  $\text{Ca}^{++}$ ,  $\text{Mg}^{++}$ ,  $\text{Cl}^-$ ,  $\text{NO}_3^-$ ,  $\text{NO}_2^-$ ,  $\text{SO}_3^{--}$ , and  $\text{SO}_4^{--}$ . A summary of the chemical analyses results for the scrubber feed and discharge streams of each reactant tested during the test program are shown in Tables 4-1 through 4-11.

#### Characteristics and $\text{SO}_2$ Analysis of Influent Flue Gases

A summary of the influent flue gas characteristics for both the oil-fired Key West unit No. 3 and coal-fired Shawnee units No. 9 and 10 is

Table 4-1. SUMMARY OF SALT WATER TESTS

## CHEMICAL ANALYSES

(Key West)

	pH	Ca (mg/l)	Mg (mg/l)	Cl (mg/l)	NO <sub>3</sub> (mg/l)	NO <sub>2</sub> (mg/l)	SO <sub>3</sub> (mg/l)	SO <sub>4</sub> (mg/l)
<u>Scrubber Feed</u>								
Average	7.6	475	1385	23,207	0.40	<0.01	1	2640
Maximum	7.8	1040	1510	25,000	0.60	<0.01	1	2740
Minimum	7.4	410	1215	19,800	0.02	<0.01	1	2125
<u>Scrubber Discharge</u>								
Average	2.6	432	1375	21,667	0.20	<0.01	32	3171
Maximum	2.9	530	1440	23,500	0.80	<0.01	127	3840
Minimum	2.3	420	1360	20,000	<0.01	<0.01	3	2800

Table 4-2. SUMMARY OF CORAL MARL

## CHEMICAL ANALYSES

(Key West)

	pH	Ca (mg/l)	Mg (mg/l)	Cl (mg/l)	NO <sub>3</sub> (mg/l)	NO <sub>2</sub> (mg/l)	SO <sub>3</sub> (mg/l)	SO <sub>4</sub> (mg/l)
<u>Scrubber Feed</u>								
Average	7.7	541	1431	22,400	0.9	0.14	2	2872
Maximum	8.0	2180	1920	30,000	16.0	0.40	7	3840
Minimum	7.5	400	1340	19,000	0	0	1	2220
<u>Scrubber Discharge</u>								
Average	5.5	1614	1471	23,180	3.0	2.27	6419	4641
Maximum	6.4	2800	1875	30,000	15.2	10.40	40,000	6100
Minimum	4.7	880	584	12,500	0	0	1110	3500

Table 4-3. SUMMARY OF FREDONIA LIMESTONE

## CHEMICAL ANALYSES

(Key West)

	pH	Ca (mg/l)	Mg (mg/l)	Cl (mg/l)	NO <sub>3</sub> (mg/l)	NO <sub>2</sub> (mg/l)	SO <sub>3</sub> (mg/l)	SO <sub>4</sub> (mg/l)
<u>Scrubber Feed</u>								
Average	7.6	492	1401	22,520	0.50	0.20	1	2810
Maximum	7.9	600	1460	26,500	2.80	0.60	2	3040
Minimum	7.1	400	1340	20,000	0	0	1	2750
<u>Scrubber Discharge</u>								
Average	5.6	1551	1498	22,900	1.60	2.20	5601	5183
Maximum	6.1	1960	1940	31,500	5.80	7.92	23300	5400
Minimum	4.5	1120	875	15,000	0	0	900	3700

Table 4-4. SUMMARY OF HYDRATED LIME

## CHEMICAL ANALYSES

(Key West)

	pH	Ca (mg/l)	Mg (mg/l)	Cl (mg/l)	NO <sub>3</sub> (mg/l)	NO <sub>2</sub> (mg/l)	SO <sub>3</sub> (mg/l)	SO <sub>4</sub> (mg/l)
<u>Scrubber Feed</u>								
Average	11.4	2800	158	20,900	0.70	0.08	2	2815
Maximum	11.6	2960	267	21,500	0.80	0.15	3	2840
Minimum	11.1	2680	97	20,500	0.60	0.01	1	2760
<u>Scrubber Discharge</u>								
Average	8.3	80	2697	21,200	0.70	0.22	25,400	4203
Maximum	8.7	100	3340	22,000	1.20	0.35	37,300	4850
Minimum	7.7	56	2330	20,500	0.20	0.13	11,900	3660



Table 4-5. SUMMARY OF DOLOMITE

## CHEMICAL ANALYSES

(Key West)

	pH	Ca (mg/l)	Mg (mg/l)	Cl (mg/l)	NO <sub>3</sub> (mg/l)	NO <sub>2</sub> (mg/l)	SO <sub>3</sub> (mg/l)	SO <sub>4</sub> (mg/l)
<u>Scrubber Feed</u>								
Average	7.6	485	1386	21,500	1.20	0.50	2	2963
Maximum	7.7	560	1410	21,500	1.60	0.90	3	3000
Minimum	7.5	440	1340	21,500	0.80	0.10	1	2920
<u>Scrubber Discharge</u>								
Average	5.2	599	1563	20,100	2.70	0.03	515	4740
Maximum	5.4	840	1850	21,500	3.20	0.09	600	6740
Minimum	4.9	116	1410	18,000	1.80	0.01	450	4050

Table 4-6. SUMMARY OF PRECIPITATED CALCIUM CARBONATE

## CHEMICAL ANALYSES

(Key West)

	pH	Ca (mg/l)	Mg (mg/l)	Cl (mg/l)	NO <sub>3</sub> (mg/l)	NO <sub>2</sub> (mg/l)	SO <sub>3</sub> (mg/l)	SO <sub>4</sub> (mg/l)
<u>Scrubber Feed</u>								
Average	7.6	467	1355	21,300	1.10	0.10	2	2800
Maximum	7.6	480	1385	22,500	1.20	0.14	2	2840
Minimum	7.5	440	1340	20,500	1.00	0.07	2	2760
<u>Scrubber Discharge</u>								
Average	5.9	1133	1360	16,200	0.90	0.18	1919	3677
Maximum	6.2	1640	804	20,500	1.40	0.27	3500	41200
Minimum	6.0	880	655	13,000	0.40	0.09	1000	3250

Table 4-7. SUMMARY OF SHAWNEE NO. 9 FREDONIA  
LIMESTONE CHEMICAL ANALYSES  
(Paducah)

	pH	Ca (mg/l)	Mg (mg/l)	Cl (mg/l)	NO <sub>3</sub> (mg/l)	NO <sub>2</sub> (mg/l)	SO <sub>3</sub> (mg/l)	SO <sub>4</sub> (mg/l)
<u>Scrubber Feed</u>								
Average	8.2	24	4.9	14	3.30	0.27	4	43
Maximum	8.5	32	7.4	15	5.50	0.35	5	131
Minimum	8.0	12	3.5	13	1.50	0.16	2	0
<u>Scrubber Discharge</u>								
Average	5.3	1063	61.9	373	7.50	0.10	1585	1654
Maximum	5.6	1440	97.0	800	15.50	0.16	2600	2479
Minimum	5.0	600	48.5	100	3.50	0.09	950	910

Table 4-8. SUMMARY OF SHAWNEE NO. 9 ARAGONITE  
CHEMICAL ANALYSES  
(Paducah)

	pH	Ca (mg/l)	Mg (mg/l)	Cl (mg/l)	NO <sub>3</sub> (mg/l)	NO <sub>2</sub> (mg/l)	SO <sub>3</sub> (mg/l)	SO <sub>4</sub> (mg/l)
<u>Scrubber Feed</u>								
Average	8.1	36	11.3	60	3.9	0.20	2	108
Maximum	8.1	38	12.0	90	5.0	0.22	2	156
Minimum	8.1	34	9.5	30	2.5	0.18	1	41
<u>Scrubber Discharge</u>								
Average	6.1	570	48.6	211	3.8	0.34	478	1061
Maximum	6.3	1000	48.6	350	5.5	0.55	780	2060
Minimum	5.8	360	48.5	150	3.0	0.16	300	580

Table 4-9. SUMMARY OF SHAWNEE NO. 9 SALTWATER/FREDONIA  
LIMESTONE CHEMICAL ANALYSES  
(Paducah)

	pH	Ca (mg/l)	Mg (mg/l)	Cl (mg/l)	NO <sub>3</sub> (mg/l)	NO <sub>2</sub> (mg/l)	SO <sub>3</sub> (mg/l)	SO <sub>4</sub> (mg/l)
<u>Scrubber Feed</u>								
Average	8.2	60	8.5	12,000	2.50	0.40	1	58
Maximum	8.2	60	8.5	12,000	2.50	0.40	1	58
Minimum	8.1	60	8.5	12,000	2.50	0.40	1	58
<u>Scrubber Discharge</u>								
Average	5.6	1040	48.5	9,500	3.30	0.17	865	2120
Maximum	5.7	1280	48.5	10,000	3.50	0.17	1150	2880
Minimum	5.4	800	48.5	9,000	2.00	0.16	580	1360

Table 4-10. SUMMARY OF SHAWNEE NO. 10 ARAGONITE INJECTION  
CHEMICAL ANALYSES  
(Paducah)

	pH	Ca (mg/l)	Mg (mg/l)	Cl (mg/l)	NO <sub>3</sub> (mg/l)	NO <sub>2</sub> (mg/l)	SO <sub>3</sub> (mg/l)	SO <sub>4</sub> (mg/l)
<u>Scrubber Feed</u>								
Average	7.9	58	2.7	10.8	35.20	0.17	4	125
Maximum	8.1	92	3.5	13	52.00	0.18	8	205
Minimum	7.8	24	2.4	10	1.60	0.16	2	24
<u>Scrubber Discharge</u>								
Average	7.8	520	60.7	200	3.90	4.20	37	1021
Maximum	7.9	600	72.9	250	8.00	6.00	80	1230
Minimum	7.4	440	48.5	150	1.60	3.00	15	740

Table 4-11. SUMMARY OF SHAWNEE NO. 10 FREDONIA LIMESTONE  
INJECTION CHEMICAL ANALYSES  
(Paducah)

	pH	Ca (mg/l)	Mg (mg/l)	Cl (mg/l)	NO <sub>3</sub> (mg/l)	NO <sub>2</sub> (mg/l)	SO <sub>3</sub> (mg/l)	SO <sub>4</sub> (mg/l)
<u>Scrubber Feed</u>								
Average	7.9	37	5.7	15	2.00	0.39	2	61
Maximum	8.1	64	6.0	15	3.10	1.50	2	80
Minimum	7.4	28	5.0	15	0.20	0.20	1	24
<u>Scrubber Discharge</u>								
Average	5.8	643	33.6	135	5.20	0.61	557	1200
Maximum	6.9	1320	48.6	200	7.50	3.60	1700	2080
Minimum	4.9	320	24.3	100	2.40	0.18	100	310

presented in Tables 4-12 and 4-13, respectively. In addition, plots of the variation in entering SO<sub>2</sub> concentration with time at both test sites are shown in Figures 4-4 and 4-5. The major variations in inlet SO<sub>2</sub> concentration at the Key West site were attributed to a change in sulfur content of the fuel oil. This was either due to stratification of different sulfur content fuels in the fuel storage tank or a change in the total sulfur content in the tank, the latter resulting from the mixing of fuel oil shipments in the tank. Similarly, SO<sub>2</sub> variations at the Shawnee test site were attributed to the wide variation of sulfur present in the coal supplied to the plant. In addition, during the month of July, TVA was conducting precipitator efficiency tests on Shawnee No. 10. These tests frequently increased dilution air leakages into the flue gas upstream of the pilot plant entrance.

#### Scale Deposition

Deposition of scale in piping, fans, pumps, tanks, sumps, and on the absorber internals varied from nondetectable to moderately heavy during the Key West and Shawnee No. 9 test programs. The degree and rate of

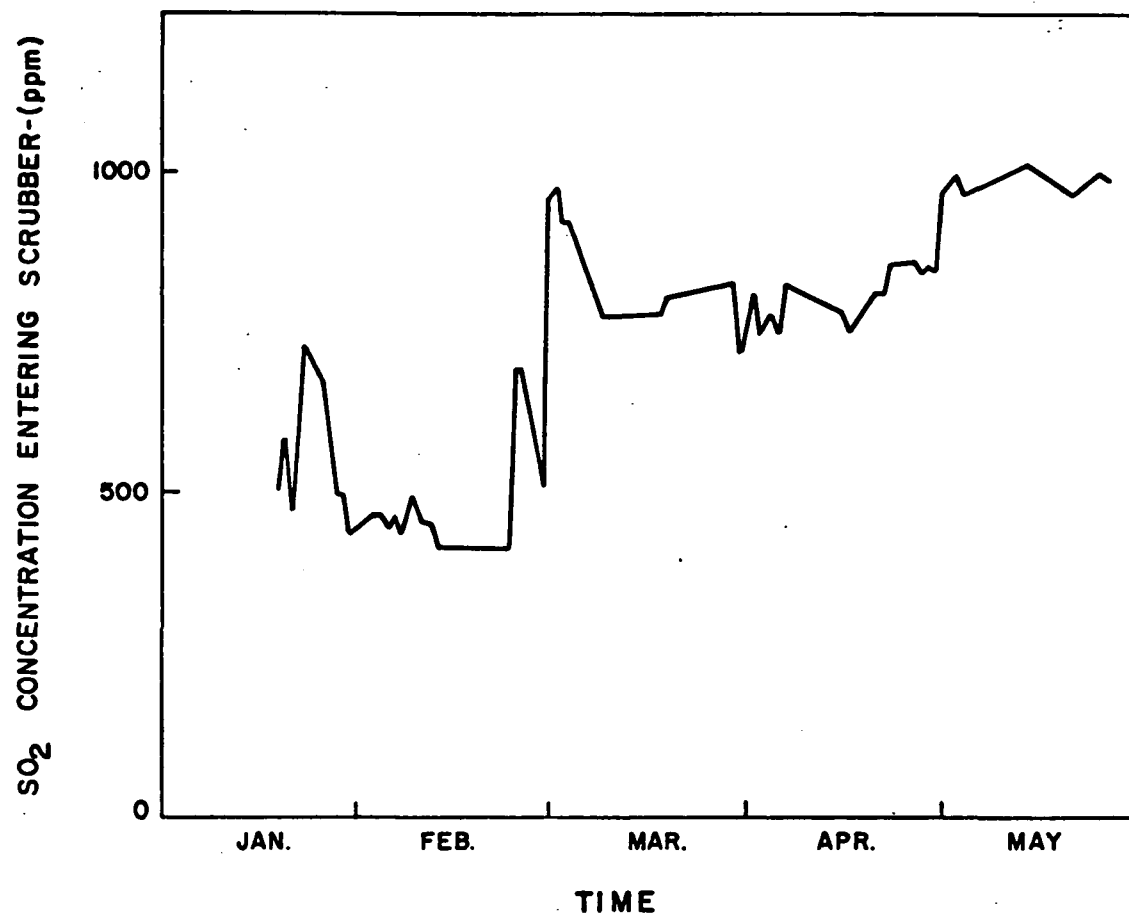


FIG. 4-4. VARIATION OF INLET  $\text{SO}_2$  CONCENTRATION, KEY WEST - 1971

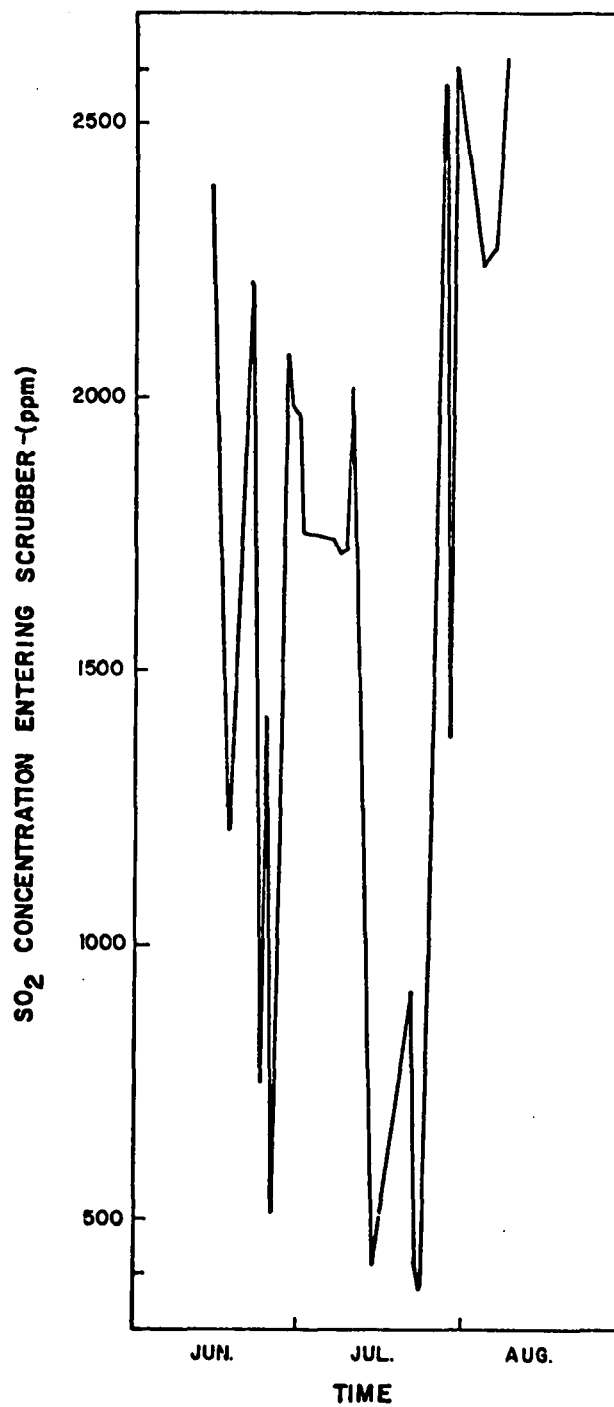


FIG. 4-5. VARIATION OF INLET SO<sub>2</sub> CONCENTRATION, PADUCAH - 1971

Table 4-12. SUMMARY OF INLET FLUE GAS CONDITIONS

(Key West Unit No. 3)

Average Dry Bulb Temperature	231°F
Average Wet Bulb Temperature	130°F
Average SO <sub>2</sub> Concentration	703 ppm

Table 4-13. SUMMARY OF INLET FLUE GAS CONDITIONS

(Shawnee Units No. 9 and 10)

Average Dry Bulb Temperature	191°F
Average Wet Bulb Temperature	119°F
Average SO <sub>2</sub> Concentration	1699 ppm

deposition was not quantified except in terms of how rapidly the scrubbing system was affected. While scale deposits did occur, the only interference with the system operation was a fan failure caused by build-up on the blades.

The deposits resulted primarily from precipitation of calcium salts and deposition of the reactant material. The composition of the deposit on the tube was identified by x-ray diffraction and is presented in Table 4-14. Deposits were not found in the mix tank, pumps, or related piping. Deposits were found on the weir hopper and inside scrubber wall at the water line, the tube at the liquid/gas interface, the mist elimination section, and in the fan housing. It should be noted that all deposits except those found on the tube and in the fan were very minor and did not interfere with the system performance over a 6 month period. The deposits on the tube occurred moderately fast and the rate of formation appeared to increase with slurry concentration. The tube deposits took the form of stalactites which grew directly into the gas path. If allowed to grow for an extended time period, these deposits would interfere with system performance. Table 4-15 summarizes the degree of deposition at various locations in the pilot plant during both the Key West and TVA Shawnee Unit No. 9 tests.

Table 4-14. X-RAY DIFFRACTION ANALYSES OF SCALE  
FORMATION ON THE TUBE  
(Key West)

Compound	Quantity (wt%)
$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	30.5
$\text{CaCO}_3$	68.0
$\text{MgCO}_3$	1.5



Table 4-15. DEGREE OF DEPOSITION AT VARIOUS SYSTEM LOCATIONS  
(Key West Unit No. 3 and Shawnee Unit No. 9)

Location	Degree	Rate of Formation
Mix Tanks	negligible	-
Pumps & Related Piping	negligible	-
Weir Hopper (water line)	slight	slow
Absorber Body (water line)	slight	slow
Tube Before <sup>a</sup>	moderately heavy	moderate
After	negligible	-
Mist Eliminator	slight	slow
Fan	slight	very slow

<sup>a</sup>Note the change when wash water was added to control scale formation at this location.

The deposits at the tube lip were controlled later in the test program by continually washing the tube with a small spray of fresh water in the vicinity of the tube lip. Fresh water was used because it was readily available at the pressure required. It is assumed that salt water would be entirely suitable for this purpose. The design requirements to control scale at the tube lip necessitated a ring attached to the tube approximately 6 inches above the bottom of the lip. The ring had a series of 1/16 inch diameter holes drilled in such a manner that the tube was continually wetted down to the lip. Once this modification was installed and adjusted, stalactite-type growth into the gas stream was eliminated. However, during the limestone injection testing on Unit No. 10 the scale deposition was so heavy that the spray ring proved ineffective. Therefore, operation in this manner for extended periods of time (16-24 hours) was not possible.

The deposits during testing on Unit No. 10 resulted primarily from impaction of calcined limestone on the exterior surface of the tube. This resulted from the heavy particulate loading to the absorber and an inadequately designed entrance for this heavy loading. This deposition interfered with the operation because calcined limestone impacted on the tube at the entrance, built up, and fell in large cakes into the hopper. Table 4-16 summarizes the scale and sludge deposition locations in the pilot plant while scrubbing flue gas from TVA Shawnee Unit No. 10.

#### Settling Characteristics

Table 4-17 summarizes the results of one test to determine the settling characteristics of the slurry effluent. This was done by measuring the time required for the turbid portion of a slurry sample (interface between slurry and clarified liquor) to settle in an undisturbed graduated cylinder. The exact location of the interface was defined by the graduations on the side of the cylinder. The majority of the settling appeared to occur during the first 10 minutes. These data were collected to aid in the design of a clarifier or other solid-waste handling equipment which might be required on a larger sized unit.

Table 4-16. DEGREE OF DEPOSITION AT VARIOUS SYSTEM LOCATIONS  
(TVA Shawnee Unit No. 10)

Location	Degree	Rate of Formation
Mix Tanks	negligible	-
Pumps & Related Piping	negligible	-
Weir Hopper (water line)	slight	slow
Absorber Body (water line)	slight	slow
Tube <sup>a</sup>	severe	rapid
Mist Eliminator	slight	slow
Fan	slight	very slow

<sup>a</sup>Scale at tube lip controlled by wash water. Major depositing occurred by impaction of calcined limestone carried in gas stream from Unit No. 10 during limestone injection test program.

Table 4-17. SETTLING RATE OF LIMESTONE SLURRY EFFLUENT

Interface Location (ml in 500 ml graduated cylinder)	Time (Minutes)
500	0
400	3.5
350	4.6
300	5.8
250	7.3
200	8.5
150	9.7
100	10.7
48	144.0

Note: sample taken from scrubber discharge during test P-32 which utilized 325 mesh Fredonia Valley limestone in a 3% slurry concentration.

### Dry Collector

No attempt was made at quantifying the operating parameters or the performance of the dry collector regarding SO<sub>2</sub> or NO<sub>x</sub> removal. While an attempt was made to quantify the particulate removal efficiency of the dry collector during the Shawnee No. 9 tests, procedural errors and equipment malfunctions caused the tests to be voided. Since sufficient data had been obtained concerning particulate removal across the entire pilot plant (dry collector and Dustraxtor scrubber) no further attempt was made to measure the removal in the dry collector separately.

The dry collector was bypassed, when not in use, by means of built-in dampers and gas ducts at both the Key West and Shawnee No. 10 test sites. The fly ash collected from Shawnee No. 9 flue gas had the characteristics expected of coal fly ash; however, the ash was not chemically analyzed.

### SO<sub>2</sub> ABSORPTION: KEY WEST PROGRAM

In this section some of the more general results of the Key West program are summarized. An addendum to this report, available through EPA's Control Systems Laboratory, contains all the data collected. Section 5 is a more complete analysis of the data collected during the factorial design experiments.

### Salt Water Tests

A total of forty three preliminary tests were conducted with no reactant in the salt water feed using a 12 inch diameter tube size. The purpose of this series was to determine the operating characteristics of the equipment, to establish base line data and to calibrate the instrument system. Thirty of the tests provided useful data from which SO<sub>2</sub> removal efficiencies could be calculated. Two levels of gas flow and pressure drop were used, these being the same as would later be utilized in the experimental design tests. The arithmetic average SO<sub>2</sub> removal efficiency was 39.6% with sea water alone. Figure 4-6 summarizes the effects of the two gas flow and pressure drop levels.

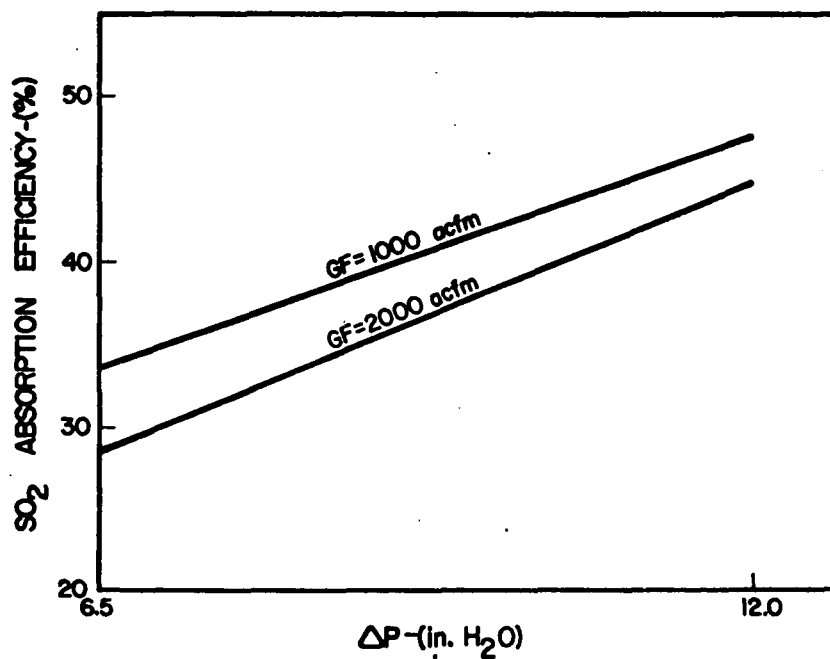


FIG. 4-6. ABSORPTION EFFICIENCY AS A FUNCTION OF GAS FLOW AND PRESSURE  
DROP: SALT WATER

Three levels of salt water feed flow rate were tested during the preliminary shakedown period; these were 2, 5, and 10 gpm. Little change in absorption efficiency was measured between the two lower rates. Figure 4-7 summarizes the effects of the 2 and 10 gpm levels.

#### Coral Marl Tests

A total of thirty six tests were conducted with coral as the reactant of which thirty four provided useful data. All tests were conducted with a 12 inch diameter tube. Sixteen of these were the factorial experiment, ten were independent data for validating the derived prediction equation, and eight were centerpoint and additive tests. Except for the last eight tests, each of the five independent variable factors was tested at two levels. The average  $\text{SO}_2$  removal efficiency using coral was 74.3%.

Figure 4-8 illustrates the absorption (expressed as  $\text{SO}_2$  absorption efficiency) as a function of the independent variables studied during the experiments with the coral reactant. The independent variables were: gas flow rate, stoichiometry, reactant particle size, slurry concentration, and scrubber pressure drop. An increase in stoichiometric ratio or a decrease in gas flow rate at a fixed pressure drop, produced a significant increase in  $\text{SO}_2$  absorption efficiency in the Dustraxtor. It should be noted that the pilot plant system was operated in a once-through configuration. These results should not be extrapolated to a closed-loop, recycle system with different ionic strength scrubbing slurries.

Four centerpoint experiments were conducted in which four of the variables were at the midpoint of the high and low level. The fifth variable, particle size, was not centered because of the unavailability of the proper size reactant. The average  $\text{SO}_2$  removal efficiency for the centerpoint experiments was 71.1%. This compares closely with the 74.3% average  $\text{SO}_2$  removal for all Key West coral tests and indicates there is a linear response to all variable factors within the range tested. Extrapolation beyond this range, however, is not recommended.

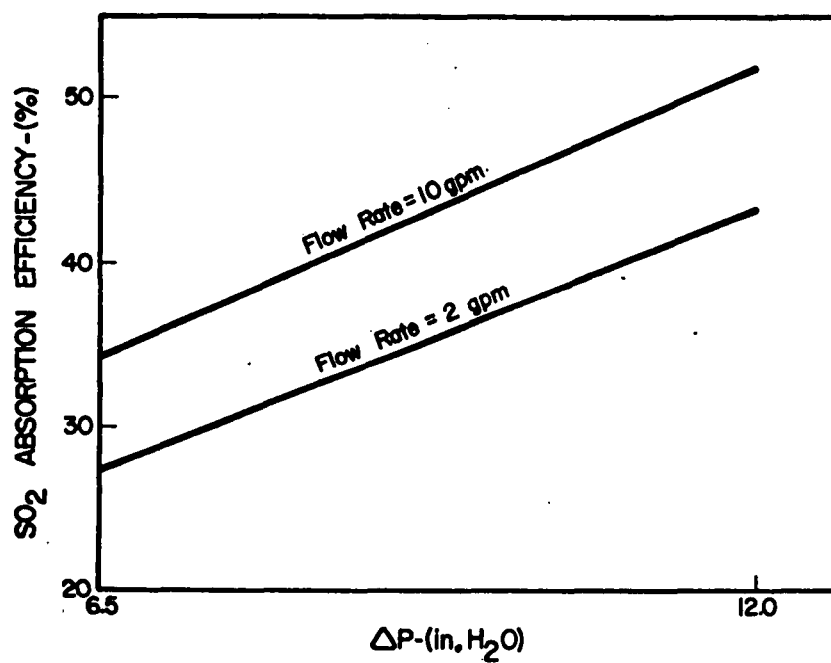


FIG. 4-7. ABSORPTION EFFICIENCY AS A FUNCTION OF FEED FLOW RATE AND PRESSURE DROP: SALT WATER

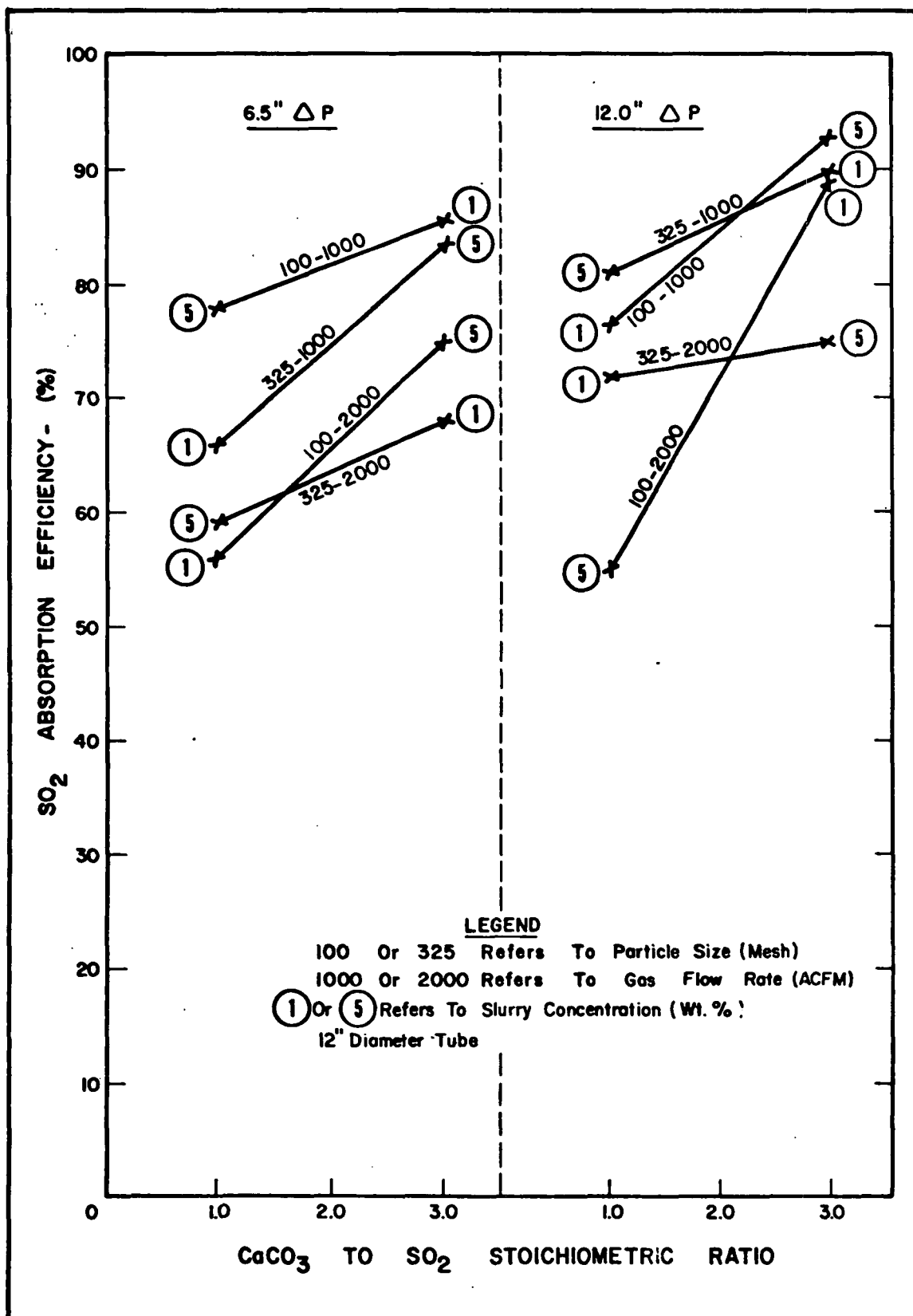


FIG.4-8. SUMMARY OF CORAL SALT WATER EXPERIMENTAL DESIGN POINTS



Three tests were conducted in which additions were made to the slurry to test the effect of  $\text{FeCl}_3$ , as a catalyst, and hydroquinone, as an inhibitor. These compounds were recommended by EPA because they have been reported to affect  $\text{SO}_2$  absorption by influencing the oxidation of sulfite to sulfate in the scrubber medium. Results were inconclusive concerning this claim since there did not appear to be a significant change in the  $\text{SO}_2$  absorption capability with the additives tested. When the  $\text{FeCl}_3$  was added directly to the mixture in the weir, an immediate decrease in  $\text{SO}_2$  outlet concentration was observed on the  $\text{SO}_2$  monitor. This response, however, was only temporary and the outlet concentration soon returned to its original level.

No noticeable results were detected in the case of the addition of hydroquinone nor in the case of the addition of both catalyst and inhibitor.

#### Fredonia Valley Limestone Tests

A total of forty three tests were conducted with limestone as the reactant of which forty one provided useful data. Sixteen of those were the factorial experiment, nine were independent data for validating the derived prediction equation, four were centerpoint tests, and twelve were comparative tests of the 8 inch and 12 inch scrubber tubes. The average  $\text{SO}_2$  removal efficiency using Fredonia Valley limestone was 73.7%.

Figure 4-9 illustrates the absorption efficiency as a function of the five independent variables studied during the experiments with Fredonia Valley limestone. An increase in stoichiometry, a decrease in gas flow rate, or an increase in pressure drop each resulted in an increase in  $\text{SO}_2$  absorption during the limestone tests. These same variables caused similar effects on  $\text{SO}_2$  absorption during the coral reactant tests.

Four centerpoint experiments were conducted in which four of the variables were at the midpoint of the high and low level. As before, particle size was not centered. Three of the tests resulted in high  $\text{SO}_2$

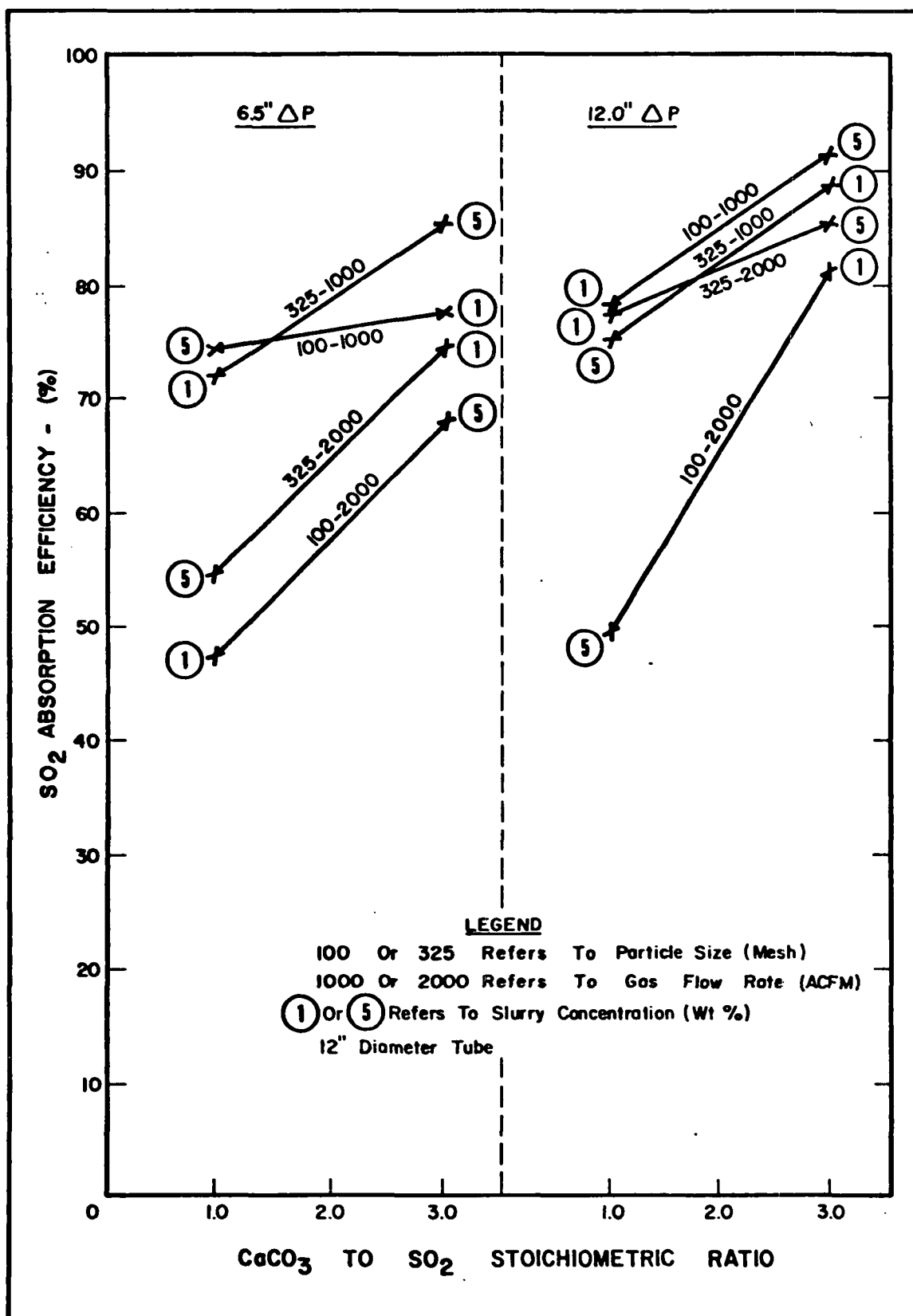


FIG.4-9 SUMMARY OF FREDONIA VALLEY LIMESTONE  
SALT WATER EXPERIMENTAL DESIGN POINTS

removal efficiencies, ranging between 84 to 86%. These values are considerably higher than expected results. It is suspected that a malfunction of the SO<sub>2</sub> monitor occurred. The fourth value was 70.6% which is much closer to the average value from all of the tests in this series.

Twelve tests with limestone demonstrated the increased absorption efficiency of the 12 inch scrubber tube. This series consisted of seven tests with the 12 inch tubes at various gas flow rates, all other variables held constant, and five similar tests with the 8 inch tube. The results of the experiment are shown in Figure 4-10.

#### Secondary Reactant Tests

Four tests with each of three secondary reactants were conducted. These were designed for factorial experiments, but one test for one reactant was voided due to carry-over into the sampling line rendering that test series inadequate for analysis. One final test was conducted with spent reactant. All tests were conducted with a 12 inch tube diameter. The average SO<sub>2</sub> removal efficiencies for the four reactants are shown in Table 4-18:

Table 4-18. SO<sub>2</sub> REMOVAL EFFICIENCY FOR SECONDARY  
REACTANT TESTS  
(Key West No. 3)

Reactant	Per Cent Removal (%)
Hydrated Lime	93.0
Dolomite	46.8
Precipitated Calcium Carbonate	78.3
Spent Reactant	69.9

Figure 4-11 summarizes the results of the hydrated lime and dolomite for which adequate data were available.

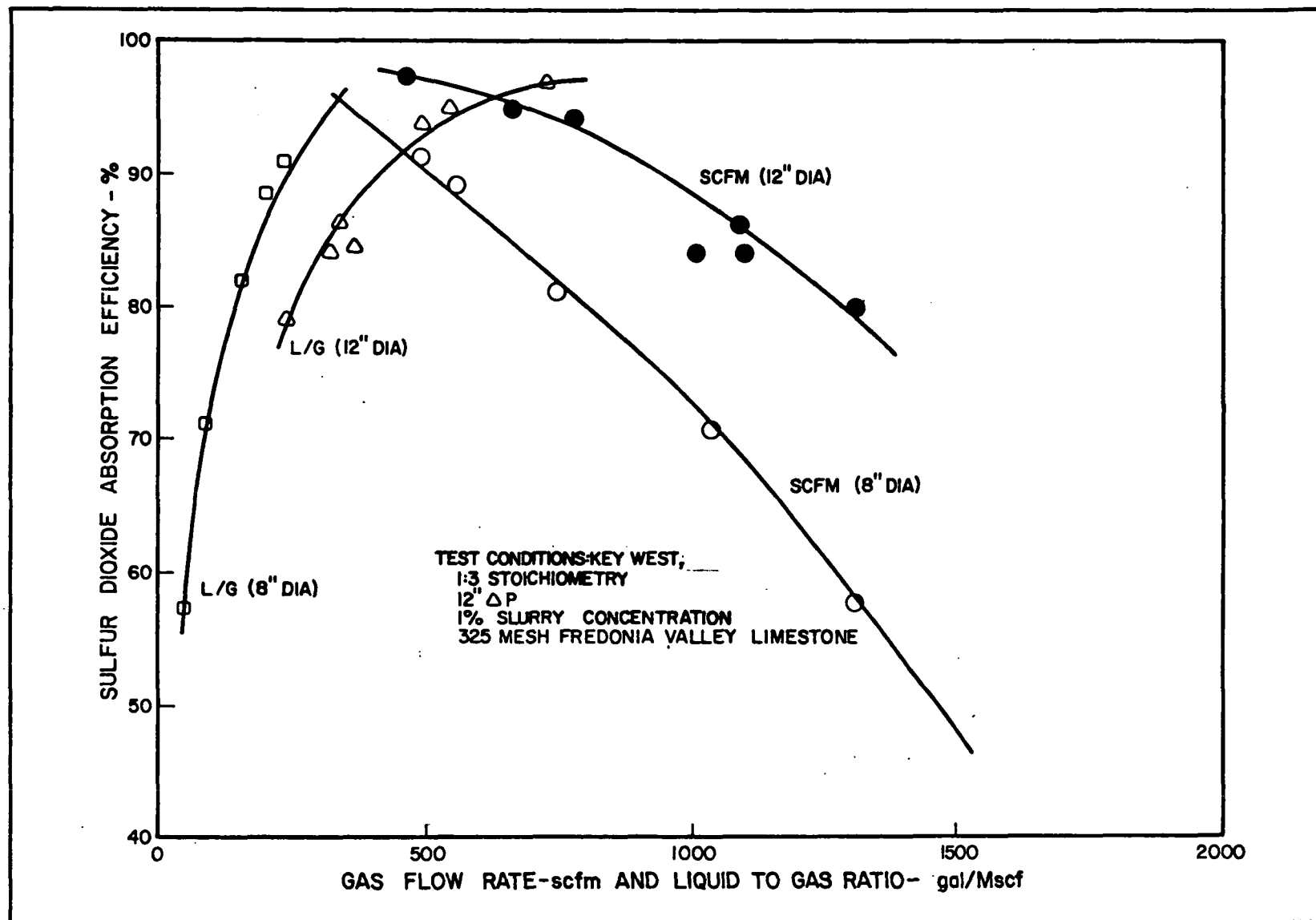


FIG. 4-10. SO<sub>2</sub> ABSORPTION EFFICIENCY AS A FUNCTION OF GAS FLOW RATE AND LIQUID/GAS RATIO

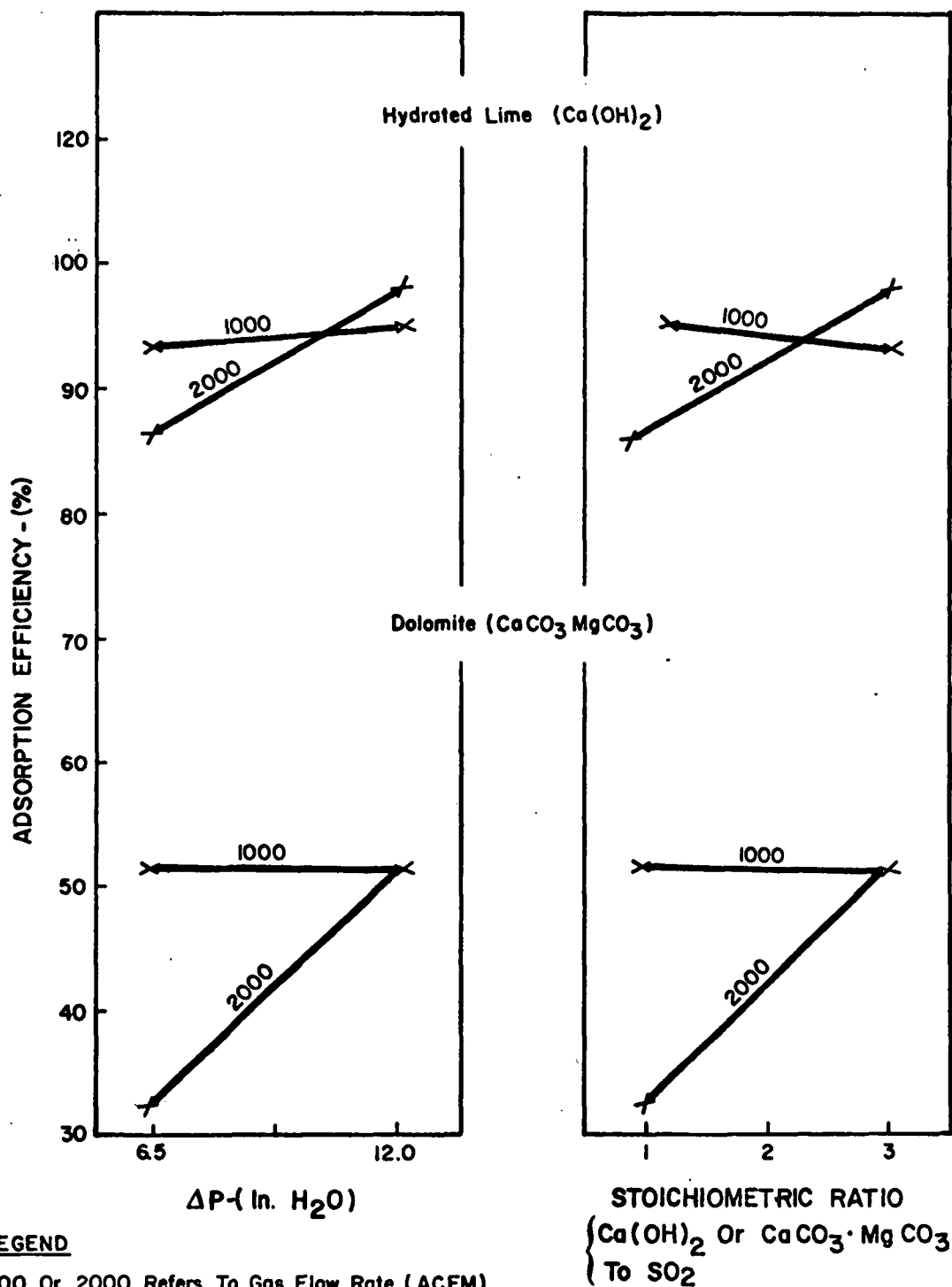


FIG. 4-II SUMMARY OF SECONDARY REACTANTS SALT WATER EXPERIMENTAL DESIGN POINTS

## SO<sub>2</sub>. ABSORPTION: PADUCAH PROGRAM

In this section some of the more general results of the Paducah program are summarized. An addendum to this report, available through EPA's Control Systems Laboratory, contains all the data collected. Section 5 is a more complete analysis of the data collected during the factorial design experiments.

### Fredonia Valley Limestone Tests

A total of thirty five tests were conducted with limestone reactant in a fresh water medium at the TVA Shawnee No. 9 unit. Thirty three provided useful data. Of these, sixteen were the factorial experiment, eight were centerpoint tests, five were additive tests, and four were tests with the dry collector bypassed. No tests were run for independent validating purposes.

The average SO<sub>2</sub> removal efficiency for the sixteen factorial experiment tests was 57.1%. This compares badly with the 73.8% average removal efficiency obtained in the sixteen factorial experimental tests conducted at Key West using Fredonia Valley limestone with a salt water medium. The variation in results is not entirely unexpected since there were many differences in the two test series, namely: tube size, stoichiometric ratio, slurry concentration, slurry medium and gas flow rate.

Figure 4-12 illustrates the absorption (expressed as SO<sub>2</sub> absorption efficiency) as a function of some of the more important parameters studied during the experiments on Unit No. 9. An increase in stoichiometric ratio or decrease in gas flow rate for each pressure drop produced a statistically significant increase in SO<sub>2</sub> absorption efficiency in the Dustractor. Again, it should be noted that the pilot plant was operated in a one-through configuration. The results should not be extrapolated to a closed-loop, recycle system with different ionic strength scrubbing slurries.

Five additive tests were conducted. In three tests 200 ppm of FeCl<sub>3</sub> were added at the weir to study the catalytic effects (see Coral Marl Tests paragraph above). There were none: the average SO<sub>2</sub> removal efficiency was 67.9% before the addition of the catalyst and 65.2% after. Similarly.

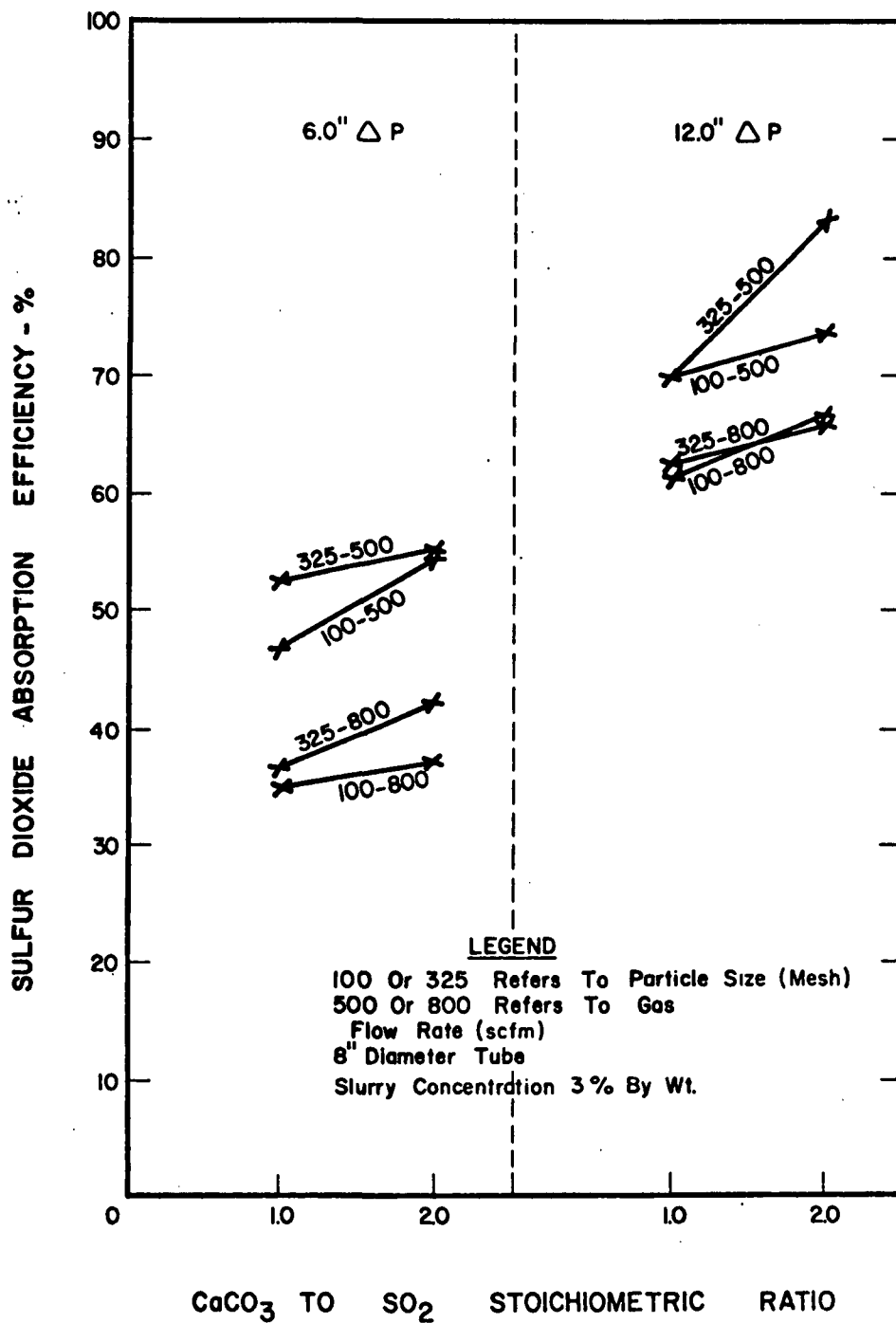


FIG. 4-12 SUMMARY OF FREDONIA VALLEY LIMESTONE RIVER WATER EXPERIMENTAL DESIGN POINTS

hydroquinone was tested in two cases as an inhibitor. Before addition the mean removal efficiency was 69.4%. After addition it was 66.4%. Based on knowledge of the SO<sub>2</sub> monitor's accuracy, drift characteristics, and response time, it was decided that effects on removal efficiencies amounting to less than 5% could not be separated from instrument and experimental error.

A total of twelve centerpoint tests were conducted. Four tests were performed at centerpoint conditions with the dry collector bypassed. This was done to evaluate the effects of coal fly ash on SO<sub>2</sub> absorption efficiencies. The average SO<sub>2</sub> removal efficiency for the four tests was 65.7%. The remaining eight centerpoint tests were conducted with the dry collector in line and resulted in an average SO<sub>2</sub> removal of 63.4%. The difference in average SO<sub>2</sub> removal for the two centerpoint test series is within instrument and experimental error. Therefore, no conclusion can be made concerning the effect of fly ash on SO<sub>2</sub> removal efficiencies.

#### Aragonite Tests

Eight tests were conducted with aragonite using an 8 inch diameter tube. Four of these were the factorial experiment, two provided independent data at centerpoint, and two were dry collector bypass tests. All were at a 3% slurry concentration. The average SO<sub>2</sub> removal efficiency was 76.2%. The two tests with the dry collector bypassed, at centerpoint, showed an average of 67.7%.

#### Simulated Key West Salt Water Tests

Three tests were conducted with Fredonia Valley Limestone in simulated salt water using an 8 inch tube diameter. An average SO<sub>2</sub> removal efficiency of 76.5% was obtained at centerpoint

#### Injection Tests

In this test series conducted on TVA Shawnee Unit No. 10, the mobile pilot plant received flue gas laden with calcined limestone or aragonite,



as supplied from the boiler injection system. A total of seventeen injection tests were conducted; four with aragonite injection, thirteen with limestone injection. During all injection tests only river water was used as the scrubbing medium. The recorded stoichiometry is the injection stoichiometry of dry reactant to  $\text{SO}_2$  as determined by TVA equipment operators. The tests conducted with aragonite injection resulted in an average  $\text{SO}_2$  efficiency of 85.9%. The limestone tests gave an average  $\text{SO}_2$  efficiency of 77.1%.

#### PARTICULATE COLLECTION IN THE SCRUBBER SYSTEM

Since the city of Key West was using fuel oil exclusively, no particulate testing was conducted at this site, and no quantitative results were available. From deposits observed above the scrubber mist eliminator section and in the fan and fan ductwork, it was concluded that particulates were generated during the scrubbing processes.

A number of tests utilizing the apparatus described in Section 3 Particulate Sampling paragraph, were conducted to quantify the particulate removal at the Paducah test site (Shawnee No.9). The results of the successful tests are presented in Table 4-19. The average result was dry collector and Dustraxtor. No attempt was made to determine the size range or composition of the particulates.

#### $\text{NO}_x$ ABSORPTION

Tables 4-20 through 4-23 present  $\text{NO}_x$  absorption data. Tables 4-21 and 4-23 present individual test results. Tables 4-20 and 4-22 show the average inlet and outlet measurements. Although these averages show a reduction in  $\text{NO}_x$  across the scrubber, the conclusion that significant absorption occurred is unwarranted because of the large standard deviations of the measured concentrations and of the experimental error associated with the analytical techniques.

#### SULFUR BALANCE

Table 4-24 shows sulfur balances from selected factorial design tests. These were selected as representative of both the Key West (coral and limestone) and the Paducah (limestone) tests. The consistently low exit

Table 4-19. PILOT PLANT PARTICULATE REMOVAL EFFICIENCY

(PADUCAH UNIT NO. 9)

Test Number	$\Delta P$ (in. H <sub>2</sub> O)	Gas Flow (scfm)	Particulate Loading (gr/scf)		Particulate Removal Efficiency(%)
			Influent	Effluent	
P-2	6.0	422	5.83	0.13	97.77
P-5	12.0	620	4.77	0.08	98.32
P-7	6.0	617	5.21	0.11	97.89
P-12	12.0	417	5.16	0.05	99.03
P-17	9.0	520	5.62	0.07	98.75
PA-5	9.0	520	4.92	0.08	98.37
PA-6	9.0	520	5.76	0.09	98.44
Average Efficiency.....					98.37%

Table 4-20. AVERAGE OF NO<sub>x</sub> MEASUREMENTS

(KEY WEST UNIT NO. 3)

	Average NO <sub>x</sub> (ppm)	Standard Deviation (ppm)
Key West		
Pilot Plant Entrance	440.9	97.0
Pilot Plant Exit	390.6	85.4

Table 4-21. TEST RESULTS OF NO<sub>x</sub> DURING THE KEY WEST TEST SERIES

Test No.	Influent Concentration (ppm)	Effluent Concentration (ppm)	NO <sub>x</sub> Absorption (ppm)	Removal (%)
S-38	357	310	47	13.2
C-19	425	401	24	5.6
C-26	387	362	25	6.5
C-27	195	404	-	-
C-28	392	356	36	9.2
C-29	645	596	49	7.6
C-30	475	452	23	4.8
F-3	392	307	85	23.2
F-4	475	504	-	-
F-5	417	398	19	4.6
F-16	547	415	132	24.1
F-17	388	300	88	22.7
F-20	195	228	33	-
F-21	310	247	63	20.3
F-22	416	374	42	10.1
F-23	407	408	-	-
F-24	536	402	134	25.0
F-25	512	400	112	21.9
F-26	501	472	29	5.8
F-27	187	325	-	-
F-28	462	551	-	-
HL-1	462	475	-	-
HL-2	425	376	49	11.5
D-3	510	472	38	7.5
D-4	175	365	-	-
PC-1	416	392	24	5.8
PC-2	489	471	18	3.7
Average NO <sub>x</sub> Absorption (ppm) . . . 54				

Table 4-22. AVERAGE NO<sub>x</sub> MEASUREMENTS  
(PADUCAH TEST SERIES)

	Average NO <sub>x</sub> (ppm)	Standard Deviation (ppm)
Unit No. 9		
Pilot Plant Entrance	710.8	176.6
Pilot Plant Exit	672.0	173.4
Unit No. 10		
Pilot Plant Entrance	764.0	101.8
Pilot Plant Exit	723.5	108.2

Table 4-23. TEST RESULTS OF NO<sub>x</sub> DURING THE PADUCAH TEST SERIES

Test No.	Influent Concentration (ppm)	Effluent Concentration (ppm)	NO <sub>x</sub> Absorption (ppm)	Removal (%)
P-5	437	402	35	4.2
P-7	178	811	-	-
P-8	782	741	41	5.2
P-16	645	608	37	5.7
PA-5	892	846	46	5.2
PA-6	798	763	35	4.4
IP-6	836	800	36	4.3
IP-7	700	845	-	-
IP-8	692	647	45	6.5
Average NO <sub>x</sub> Absorption (ppm) . . . . .			39	

Table 4-24. SULFUR BALANCES FOR SELECTED TESTS AT

## KEY WEST AND PADUCAH

Test Number	Liquid Stream		Gas Stream		Total Liquid & Gas Stream		Sulfur Accounted For (%)
	Sulfur Flow (lbs/min)		Sulfur Flow (lbs/min)		Sulfur Flow (lbs/min)		
	Feed	Disch	Influent	Effluent	Influent	Effluent	
C-20	0.0432	0.0600	0.0486	0.0071	0.0918	0.0671	73.1
C-21	0.0140	0.0200	0.0486	0.0131	0.0626	0.0331	52.9
C-22	0.0288	0.0445	0.0989	0.0431	0.1277	0.0876	68.6
C-23	0.0173	0.0245	0.0988	0.0249	0.1161	0.0494	42.5
C-34A	0.0200	0.0242	0.0932	0.0247	0.1132	0.0489	43.2
F-8	0.0080	0.0143	0.0267	0.0072	0.0347	0.0215	62.0
F-18	0.0320	0.0489	0.1027	0.0532	0.1347	0.1021	75.8
F-20	0.0064	0.0104	0.1004	0.0508	0.1068	0.0612	57.3
F-26	0.0033	0.0053	0.0513	0.0128	0.0546	0.0181	33.2
F-42	0.0189	0.0222	0.0912	0.0268	0.1101	0.4900	44.5
P-1	0.0002	0.0055	0.0902	0.0432	0.0904	0.0487	53.9
P-2	0.0004	0.0085	0.0826	0.0379	0.0830	0.4640	55.9
P-3	0.0000	0.0065	0.0822	0.0245	0.0822	0.0310	37.7
P-5	0.0001	0.0067	0.1030	0.0395	0.1031	0.0462	44.8
P-8	0.0005	0.0084	0.0896	0.0558	0.0901	0.0642	71.3
P-13	0.0002	0.0066	0.1146	0.0729	0.1148	0.0789	68.7
P-20	0.0001	0.0074	0.0084	0.0345	0.0885	0.0419	47.3
P-21	0.0001	0.0104	0.0861	0.0323	0.0861	0.0427	49.6
P-22	0.0003	0.0198	0.0877	0.0303	0.0878	0.0413	47.0
P-23	0.0003	0.0082	0.0812	0.0303	0.0813	0.0342	42.1

## Key West:

Average Sulfur Accounted for ..... 55.3%

Maximum Sulfur Accounted for ..... 75.8%

## Paducah:

Average Sulfur Accounted for ..... 51.8%

Maximum Sulfur Accounted for ..... 71.3%

Total Average Sulfur Accounted for ..... 53.57%

sulfur values might be attributed to the analytical technique used on the liquid discharge stream. Slurry samples were sometimes allowed to stand up to 24 hours before the total sulfur analysis (as  $\text{SO}_4^{--}$ ) was performed.

This time period allowed sulfur to crystallize out of solution as the dihydrate,  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ . Although slurry samples were thoroughly mixed prior to aliquot removal, the rapid settling that occurred made representative sampling difficult. In addition, it is suspected that the  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  may not have been completely digested in the time allotted. Either of these two situations could account for the low sulfur results shown in Table 4-24.

## SECTION 5

### DATA ANALYSIS

#### INTRODUCTION AND SUMMARY

Five factorial experiments were conducted with the reactants to determine the effects of the several independent variables. The five factors investigated were: gas flow rate (GF); pressure drop ( $\Delta P$ ); stoichiometric ratio (SR); particle size (PS); and slurry concentration (SC). These experiments are summarized in Table 5-1.

TABLE 5-1. SUMMARY OF FACTORIAL EXPERIMENTS

	<u>Key West</u>				<u>Key West</u>				<u>Paducah</u>			
Type of Design	Half Replicate of a $2^5 = 16$ Runs				Half Replicate of a $2^3 = 4$ runs				Full Replicate of a $2^4 = 16$ runs			
Reactants Studied	Coral (C) and Fredonia Valley Lime-Stone (FVL)				Dolomite (D) and Hydrated Lime (HL)				Fredonia Valley Limestone (FVL)			
Factors Studied and Their Levels	GF(scfm)	1450	775		GF(acfm)	2000	1000		GF(scfm)	600	400	
	$\Delta P$ (in. H <sub>2</sub> O)	12.5	6.5		$\Delta P$	12.0	6.5		$\Delta P$	12.0	6.0	
	SR(mole/mole)	1:3	1:1		SR	1:3	1:1		SR	1:2	1:1	
	PS(mesh)	325	100		PS	Uncontrolled			PS	325	200	
	SC(%)	5	1		SC	1	1		SC	3	3	
Factors found Statistically Significant at 95% level	GF, $\Delta P$ , SR ) for C GFSC, PSC)				GF, $\Delta P$ , SR for D  $\Delta P$ , SR for HL				GF, $\Delta P$ , SR for FVL			

A factorial experiment is designed to test the significance of a number of different operating factors simultaneously. The proper application of the method permits a rapid and inexpensive method for determining the effects of each of the factors over the specified range. The interpretation of the results is difficult, however, and sound engineering judgement must be used in assigning statistical significance to the conclusions. This judgement

Table 5-2. KEY WEST TEST PROGRAM EXPERIMENTAL DESIGN

REACTANTS:

Coral (C)

Fredonia Valley Limestone (FVL)

Run No.	Factors					Responses (% SO <sub>2</sub> Removal)	
	GF	ΔP	SR	PS	SC	C	FVL
1	-	-	-	+	-	64.6	71.1
2	+	-	-	-	-	56.5	48.3
3	-	+	-	-	-	73.1	76.6
4	+	+	-	+	-	70.6	76.4
5	-	-	+	-	-	85.3	78.9
6	+	-	+	+	-	67.6	74.4
7	-	+	+	+	-	90.1	90.4
8	+	+	+	-	-	88.8	82.6
9	-	-	-	-	+	77.5	73.7
10	+	-	-	+	+	58.2	50.0
11	-	+	-	+	+	80.5	75.0
12	+	+	-	-	+	55.6	49.4
13	-	-	+	+	+	83.3	88.5
14	+	-	+	-	+	74.7	68.7
15	-	+	+	-	+	93.3	90.5
16	+	+	+	+	+	75.0	86.0

Total Response. . . . 1195.2 1180.5

Average Response. . . 74.7 73.8



may be based on knowledge of and experience with the physical and chemical processes involved and the methods in which the factors act; comparison of results with other similar experiments; or conducting additional tests of factors whose significance is questionable.

In treatment of the data, classical analysis of the variance techniques are used, details of which may be found in any elementary statistics text. Important assumptions involved in the use of these techniques include:

- o Independence of the tests
- o Normal distribution of the experimental errors
- o Linearity of response (percent SO<sub>2</sub> removal) to each of the factors in the range tested.

In general the analysis showed that in these experiments the three factors GF, ΔP and SR were statistically significant at the 95% confidence level. This means that one can be 95% confident that SO<sub>2</sub> removal efficiency is different for the two levels tested for each factor. No conclusion can be reached concerning the effect of levels outside the range tested nor can it be concluded that one factor is more effective than another.

#### KEY WEST TESTS

The main test series at Key West consisted of half-replicate 2<sup>5</sup> factorial experiments for each of two reactants, coral and Fredonia Valley limestone (FVL). The factors and levels are listed below:

<u>Factor</u>	<u>Design Levels</u>	
	High (+)	Low (-)
GF - Gas flow (scfm)	1450	775
ΔP - Pressure drop (in.H <sub>2</sub> O)	12.0	6.5
SR - Stoichiometric ratio	1:3	1:1
PS - Particle size (mesh)	325	100
SC - Slurry concentration (%)	5	1

Table 5-2 shows the experimental design and the response measured as percent SO<sub>2</sub> removal for the main Key West test program.

A half replicate  $2^5$  factorial experiment permits determination of all main effects and two-factor interactions. Each main effect is aliased with a four-factor interaction and each two-factor interaction is aliased with a three-factor interaction. If all third order and above interactions are considered small (to be shown in the analysis of the Paducah tests, following), the design permits independent estimates of the main effects and two-factor interactions. However, if all two factor interactions are considered important, there remain no degrees of freedom to estimate the error. Thus, the investigator must estimate the error mean square by the engineering judgement mentioned above or by assuming, a priori, that some specific two factor interactions are negligible.

The means of the response from the two reactants, 74.7% for coral and 73.8% for Fredonia Valley limestone, were tested for significant difference. There was found to be no significant difference at the 99% confidence level. Furthermore, the correlation coefficient between the sixteen pairs of data points was calculated to be 0.89. These facts might indicate that the physical and chemical processes involved were similar for each of the two reactants and that any main effect or interaction that is assigned to experimental error in the one case should be so assigned in the other case. In view of the lack of substantiating information concerning the estimation of the error, an a priori judgement was made that differences in average effect of less than 2% are within the range of experimental error.

Tables 5-3 and 5-4 show the analysis of the variance for the Key West coral and limestone tests respectively. If the calculated effect is below this value in either test, the values are used for the estimate of the error. With this scheme only three main effects and two interactions would remain. An exception to this scheme is made in the main effect of particle size. It has been shown that coral undergoes certain physical changes in the presence of sea water in which the particles are retained in a colloidal suspension, regardless of the original particle size distribution. For this reason, particle size main effect is assigned to the error sum in the case of coral but not in the case of limestone.

Table 5-3. CORAL REACTANT---ANALYSIS OF THE VARIANCE

## KEY WEST TEST PROGRAM

	Total Effects	Average Effects	Sum of Squares	Mean Square	F Ratio
Total	1195.2	74.7			
GF	-101.2	-12.7	640.1	640.1	37.7
$\Delta P$	58.8	7.4	216.1	216.1	12.7
SR	122.0	15.3	930.2	930.2	54.7
PS	-14.4	-1.8	13.0	Error	
SC	2.0	0.3	0.3	Error	
GF $\Delta P$	7.2	0.9	3.2	Error	
GF $\Delta P$ SR	8.4	1.1	4.4	Error	
GF $\Delta P$ PS	6.0	0.8	2.3	Error	
GF $\Delta P$ SC	-42.0	-5.3	110.3	110.3	6.5
$\Delta P$ SR	12.8	1.6	10.2	Error	
$\Delta P$ PS	25.2	3.2	39.7	Error	
$\Delta P$ SC	-38.4	-4.8	92.2	92.2	5.5
SRPS	-36.8	-4.6	84.6	Error	
SRSC	-12.0	-1.5	9.0	Error	
PSSC	7.2	0.9	3.2	Error	
			TOTAL ....	2158.8	

Error sum of squares - 169.9

Error mean square - 17.0

 $F_{0.95} (1,10) = 4.96$  $F_{0.99} (1,10) = 10.04$

Table 5-4. FVL REACTANT---ANALYSIS OF THE VARIANCE

## KEY WEST TEST PROGRAM

	Total Effects	Average Effects	Sum of Squares	Mean Square	F Ratio
Total	1180.5	73.8			
GF	108.9	-13.6	741.2	741.2	21.2
ΔP	73.3	9.2	335.8	335.8	9.6
SR	139.5	17.4	1216.3	1216.3	34.8
PS	43.1	5.4	116.1	116.1	3.3
SC	-16.9	-2.1	17.8	Error	
GFΔP	32.7	4.1	66.8	Error	
GFPSR	35.7	4.5	79.7	Error	
GFPS	32.5	4.1	66.0	Error	
GFSC	-38.3	-4.8	91.7	91.7	2.6
ΔPSR	4.7	0.6	1.4	Error	
ΔPPS	14.3	1.8	12.8	Error	
ΔPSC	-33.3	-4.2	69.3	69.3	2.0
SRPS	-5.9	-0.7	2.2	Error	
SRSC	31.7	4.0	62.8	Error	
PSSC	-8.7	-1.1	4.7	Error	
TOTAL .....			2884.6		

Error sum of squares - 314.2

Error mean square - 34.9

 $F_{0.95} (1,9) = 5.12$  $F_{0.99} (1,9) = 10.60$

Some further justification for the 2% cut-off value is provided by tests conducted with limestone in Paducah. In this test series, where aliasing does not occur, all interaction effects are small. A comparison of the average effects for the two Key West and the one Paducah test series shows considerable stability from one test to another.

As can be noted in the case of coral, three main effects and two interactions are statistically significant at a confidence level of 95% or greater. In the case of limestone, only the three main effects are significant. This is determined by comparing the F ratio, defined as

$$F = \frac{\text{Factor Mean Square}}{\text{Error Mean Square}}$$

with the significance values in the tables. For instance in Table 5-3 the F ratio for GF is

$$F = \frac{640.1}{17.0} = 37.7$$

which is greater than  $F_{0.99} = 10.04$ . Therefore, gas flow rate has a statistically significant effect on percent  $\text{SO}_2$  removal by coral.

Prediction equations have been developed for relating response to the significant factors:

$$Y_C = 74.7 + [-12.7GF + 7.4\Delta P + 15.3SR - 5.3(GF\delta SC) - 4.8(P\delta SC)] \quad (5-1)$$

$$Y_{FVL} = 73.8 + [-13.6GF + 9.2\Delta P + 17.4SR] \quad (5-2)$$

where  $Y_C$  and  $Y_{FVL}$  are percent  $\text{SO}_2$  removal for coral reactant and limestone reactant, respectively.

$$GF = \frac{\text{gas flow (scfm)} - 1112}{675}$$

$$\Delta P = \frac{\text{pressure drop (H}_2\text{O)} - 9.25}{5.5}$$

$$SR = \frac{\text{stoichiometric ratio} - 2}{2}$$

$$SC = \frac{\text{slurry concentration} - 3}{4}$$

These equations are given in this way to show their deviation. As can be

seen, the basic equations include the average total response, Table 5-2, and the average effects of each of the significant factors and interactions, Tables 5-3 and 5-4. The constants in the definitions of the variables are the average of the test design levels and the range of test design levels.

There were nineteen additional tests in the coral and limestone series at Key West which were not part of the factorial experiment. These tests provided independent data to examine the accuracy of the prediction equations. Figure 5-1 is a plot of predicted response versus actual response for these nineteen data points.

Figure 5-2 illustrates the response expected from the two significant interactions in the coral reactant series. It appears that high slurry concentration increases the change in response due to changes in gas flow rate and decreases the change in response due to pressure drop changes.

Two additional reactants, dolomite and hydrated lime were tested in half-replicate  $2^3$  experimental design programs at Key West. Table 5-5 shows the design criteria and the results of the tests. In this experimental design each main factorial effect is aliased with a two-factor interaction. Further, the second order interaction cannot be estimated nor can the error mean square. A partial analysis of the variance is given in Table 5-6.

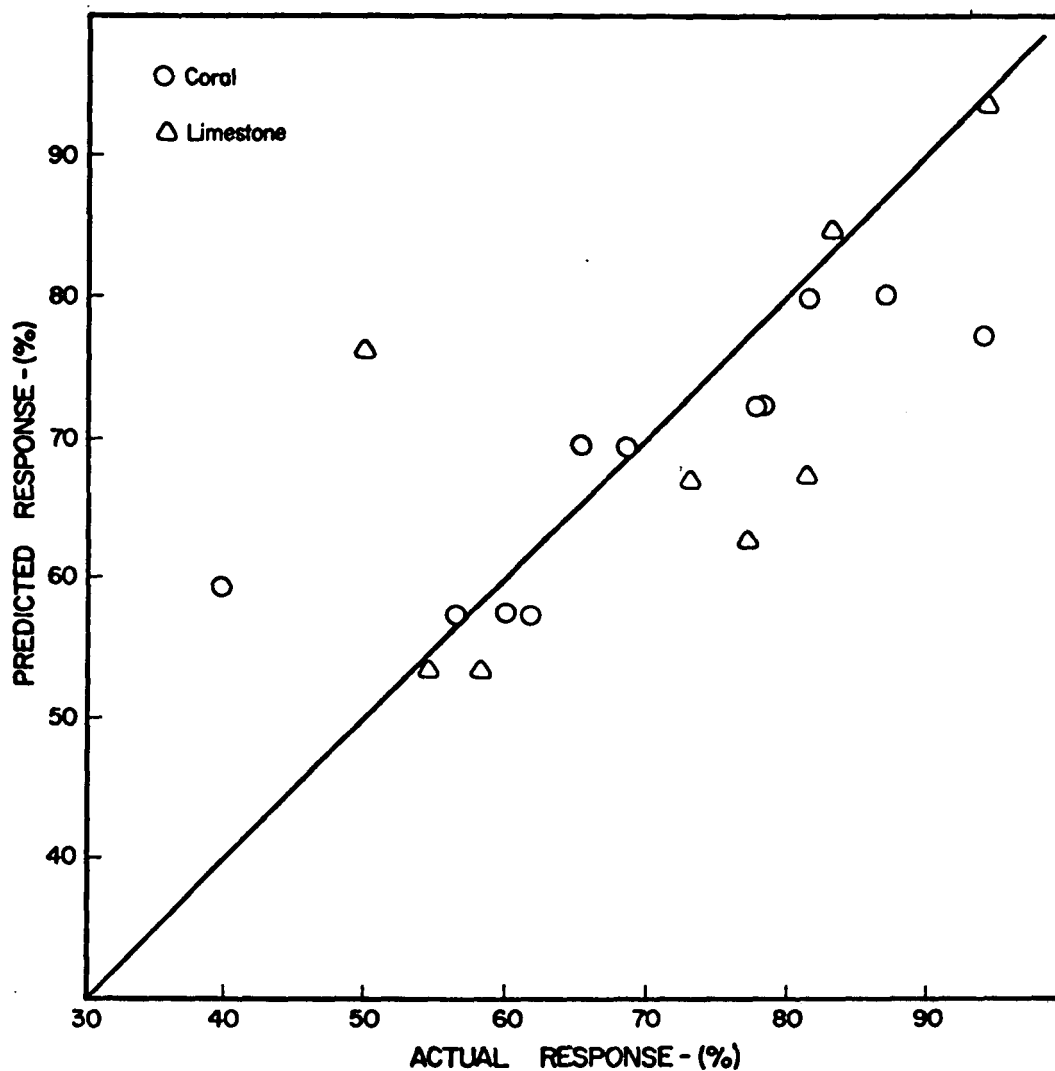


FIG. 5-1. PREDICTED RESPONSE VS. ACTUAL RESPONSE FOR INDEPENDENT DATA FROM THE MULTIPLE LINEAR REGRESSION FORMULA

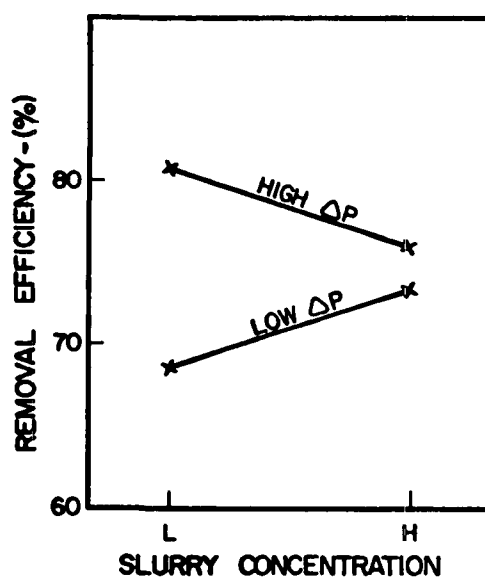
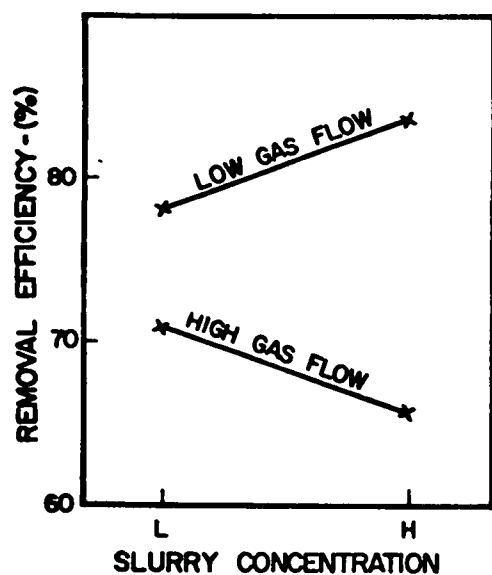


FIG. 5-2. SUMMARY OF SIGNIFICANT INTERACTIONS IN CORAL EXPERIMENTS



Table 5-5. SECONDARY REACTANT TEST PROGRAM

## AT KEY WEST TEST SITE

## SECONDARY REACTANTS:

Dolomite (D)

Hydrated Lime (HL)

TEST PROGRAM					
Test No.	$\Delta P$	Stoichiometry	Gas Flow Rate	Response (% SO <sub>2</sub> Removal)	
				D	HL
1	6.5"	1:1	2000 acfm	32.6	86.4
2	12.0"	1:1	1000 acfm	51.8	94.9
3	6.5"	1:3	1000 acfm	51.7	93.4
4	12.0"	1:3	2000 acfm	51.2	97.5

Slurry concentration - 1% by wt.

Particle size - no control due to use of commercially available materials.

Table 5-6. SECONDARY REACTANTS-- ANALYSIS OF THE VARIANCE

## KEY WEST TEST PROGRAM

Effect	Dolomite			Hydrated Lime		
	Total Effect	Mean Effect	Mean Square	Total Effect	Mean Effect	Mean Square
Total	187.3	46.8		372.2	93.0	
Gas Flow (GF)	-19.7	-9.8	97.0	-4.4	-2.2	4.8
Pressure Drop ( $\Delta P$ )	18.7	9.4	87.4	12.6	6.3	39.7
Stoichiometric Ratio (SR)	18.5	9.2	85.6	9.6	4.8	23.0
Total			270.0			67.5

Prediction equations for the secondary reactants are:

$$Y_D = 46.8 + [-9.8GF + 9.4\Delta P + 9.2SR] \quad (5-3)$$

and

$$Y_{HL} = 93.0 + [-2.2GF + 6.3\Delta P + 4.8SR] \quad (5-4)$$

where  $Y_D$  and  $Y_{HL}$  are percent  $SO_2$  removal for dolomite reactant and hydrated lime reactant respectively.

$$GF = \frac{\text{gas flow (acfm)} - 1500}{1000}$$

$$\Delta P = \frac{\text{pressure drop (in. H}_2\text{O)} - 9.25}{5.5}$$

$$SR = \frac{\text{stoichiometric ratio} - 2}{2}$$

An analysis of the variance in response to the two different reactants showed a significant difference of the means at the 99.5% confidence level.

#### PADUCAH TEST PROGRAM

A full replicate  $2^4$  factorial test program was conducted on the No. 9 unit at Paducah, using FVL as a reactant. The factors and levels are listed below. Slurry concentration was constant at 3 in this test series.

<u>Factor</u>	<u>Design Level</u>	
	High (+)	Low (-)
GF - Gas Flow (scfm)	600	400
$\Delta P$ - Pressure drop (in. H <sub>2</sub> O)	12.0	6.0
SR - Stoichiometric ratio	1:2	1:1
PS - Particle size (mesh)	325	200

Table 5-7 shows the experimental design and the response measured as percent  $SO_2$  removal. The full replicate design permits determination of all main effects and interactions. With the criteria established earlier, i.e. that effect values less than 2% in any test are considered error, all two-factor interactions would be used to estimate the error mean square. Further, if all two-factor interactions are so considered, certainly higher

Table 5-7. PADUCAH TEST PROGRAM EXPERIMENTAL DESIGN

REACTANT: Fredonia Valley Limestone

SLURRY CONCENTRATION: 3% wt.

Run No.	Factors				Response (% SO <sub>2</sub> Removal)
	GF	ΔP	SR	PS	
1	-	-	-	+	52.0
2	-	-	+	+	54.0
3	-	+	-	+	70.2
4	-	+	+	+	83.7
5	+	+	-	+	61.7
6	+	+	+	+	67.3
7	+	-	-	+	34.7
8	+	-	+	+	37.8
9	-	-	+	-	53.2
10	-	-	-	-	46.3
11	-	+	+	-	73.9
12	-	+	-	-	70.0
13	+	-	-	-	36.3
14	+	-	+	-	42.1
15	+	+	-	-	62.6
16	+	+	+	-	67.7

Total Response 913.5

Average Response 57.1

order interactions should be as well. Table 5-8 shows the analysis of the variance for the Paducah limestone test program. It is seen that the three main effects, gas flow, pressure drop, and stoichiometric ratio, are significant at the 95% confidence level. A prediction equation for this test series is given below:

$$Y_p = 57.1 + [-11.6GF + 25.1\Delta P + 5.7SR]$$

where  $Y_p$  is percent  $SO_2$  removal.

$$GF = \frac{\text{gas flow rate (scfm)} - 500}{200}$$

$$\Delta P = \frac{\text{pressure drop (in. H}_2\text{O)} - 9}{6}$$

$$SR = \text{stoichiometric ratio} - 1.5$$

No independent data are available for testing the accuracy of the prediction equation.

#### LIQUID/GAS RATIO

Some evidence has been seen that only three factors are significant in determining the  $SO_2$  removal efficiency. Two of these factors, gas flow and pressure drop (for a given size scrubber tube), uniquely determine the liquid/gas ratio. Figure 5-3 is a plot of absorption efficiency versus liquid/gas ratio for five of the reactants. The efficiencies for high and low levels of stoichiometric ratios are shown as envelopes around the mean response for each reactant. As can be noted, the efficiency increases rapidly with liquid/gas ratio up to about 140 gal/Mcf. After that point, little additional increase is achieved. Furthermore, it would appear that increased efficiency is gained by increased reactivity of the slurry above that from increased stoichiometric ratio.

The three Paducah salt water simulation test points lie on the Key West Limestone curve indicating that the difference in this curve and the Paducah curve results from the different media (salt water and river water) rather than the different size tubes.

Table 5-8. FVL REACTANT - ANALYSIS OF THE VARIANCE  
PADUCAH TEST PROGRAM

	Total Effects	Average Effects	Sum of Square	Mean Square	F Ratio
Total	913.5	57.1			
GF	-93.1	-11.6	541.7	541.7	62.3
$\Delta$ P	200.7	25.1	2517.5	2517.5	289.4
SR	45.9	5.7	131.7	131.7	15.1
PS	9.3	1.2	5.4	Error	
GF $\Delta$ P	16.1	2.0	16.2	Error	
GFSR	-6.7	-0.8	2.8	Error	
GFPS	-23.7	-3.0	35.1	Error	
$\Delta$ PSR	10.3	1.3	6.6	Error	
$\Delta$ PPS	9.1	1.0	4.1	Error	
SRPS	2.5	0.3	0.4	Error	
GF $\Delta$ PSR	-6.7	-0.8	2.8	Error	
GF $\Delta$ PPS	1.1	0.1	0.1	Error	
GFSRPS	-6.9	0.9	3.0	Error	
$\Delta$ PSRPS	17.7	2.2	19.6	Error	
GF $\Delta$ PSRPS	-11.3	-1.4	8.0	Error	
TOTAL .....			3295.0		

Error sum of squares - 104.1

Error mean square - 8.7

$F_{0.95} (1,12) = 4.75$

$F_{0.99} (1,12) = 9.33$

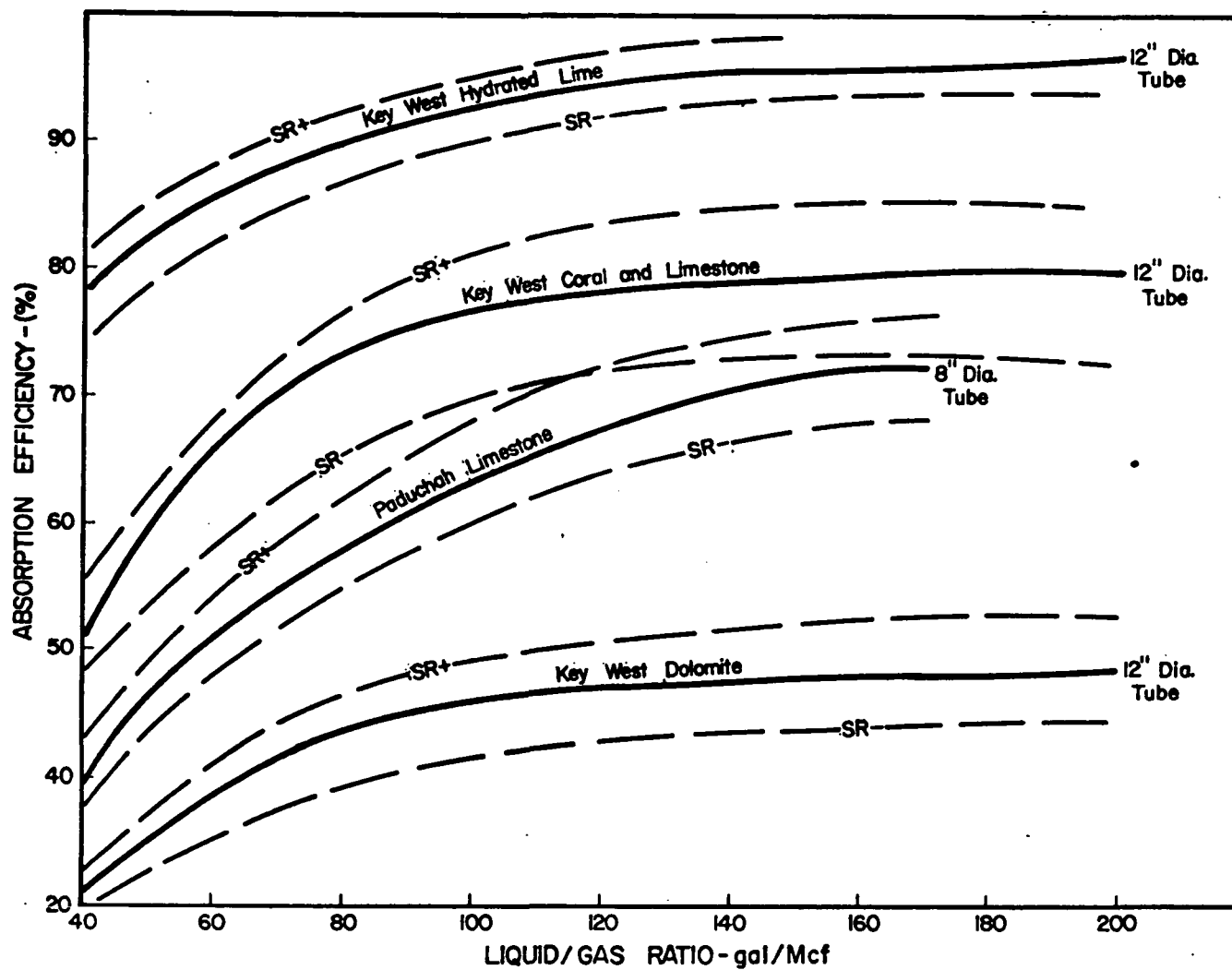


FIG.5-3. ABSORPTION EFFICIENCY AS A FUNCTION OF LIQUID/GAS RATIO

Further, at a given liquid/gas ratio, residence time in the 8 inch tube is longer than in the 12 inch tube (Figures 4-1 and 4-2).

## SECTION 6

### CONCLUSIONS

The following conclusions are based upon field observations and analysis of the data.

#### PILOT PLANT EQUIPMENT

- (1) Locating the scrubber fan at the outlet of the system caused solids build up on the blades which ultimately resulted in fan failure due to imbalance.
- (2) Corrosion and pitting were experienced on the interior walls of the 316-SS scrubber body and the non-PVC piping and valving. This corrosion and pitting appeared to be more severe, both in degree and rate, when using salt water. However, this corrosion and pitting did not result in failure of the scrubber body during the ten months of pilot plant operation.
- (3) Condensation was experienced in the non-insulated dry collector and ductwork.
- (4) The long hoses used to transport scrubber waste discharge frequently clogged due to low flow rates.
- (5) The pilot plant spray nozzles, intended for precontacting the inlet flue gas, were rendered useless due to clogging and corrosion before an evaluation could be made.

#### SAMPLING AND ANALYSIS

- (1) An "ice-trap" condenser immediately following the SO<sub>2</sub> sampling probe operated in a satisfactory manner with the Dynasciences SS-330 monitor.
- (2) The Dynasciences SO<sub>2</sub> monitor performed satisfactorily.
- (3) The use of one SO<sub>2</sub> monitor to measure both inlet and outlet flue gas proved cumbersome and reduced the accuracy of the calculated scrubber efficiency.



- (4) The phenoldisulfonic acid analysis for determining concentrations of  $\text{NO}_x$  proved limited.
- (5) The Annubar velocity measuring device performed very well, producing accurate readings in a minimal length of time.
- (6) The use of a non-heated probe and filter in the particulate sampling train caused condensation and ultimate clogging of the filter.

#### $\text{SO}_2$ ABSORPTION EFFICIENCY

- (1) Absorption efficiency increased significantly with increased pressure drop across the scrubber.
- (2) Absorption efficiency increased significantly with decreased gas flow rate through the tube.
- (3) Absorption efficiency increased significantly with increased stoichiometric ratio.
- (4) There was no significant change in absorption efficiency resulting from a change in reactant particle size.
- (5) There was no significant change in absorption efficiency resulting from a change in slurry concentration.
- (6) The analysis of the factorial experiments showed no significant interaction between or among the five factors listed above.
- (7) Absorption efficiency is an increasing function of liquid to gas ratio.
- (8) For a given tube size and gas flow rate, liquid to gas ratio increased with increasing pressure drop.
- (9) For a given tube size and pressure drop, liquid to gas ratio was a function of gas flow rate with maximum value near 600 scfm.
- (10) For a given pressure drop and gas flow rate, liquid to gas ratio increased with tube size.

- (11) Absorption efficiency was different with different reactants with decreasing efficiency as follows:

- Hydrated Lime
- Precipitated Calcium Carbonate
- Limestone
- Coral
- Spent Coral
- Dolomite

- (12) In the tests conducted, residence time within the tube had no apparent effect on absorption efficiency.
- (13) Addition of the catalyst ( $\text{FeCl}_3$ ) and/or the inhibitor (hydroquinone) to the slurry produced no significant effect on absorption efficiency.

#### $\text{NO}_x$ AND PARTICULATE REMOVAL

- (1)  $\text{NO}_x$  removal in the Dustraxtor scrubber utilizing limestone type reactants was negligible.
- (2) Particulate removal by the total pilot plant system (dry collector and Dustraxtor) was excellent though evidence of re-entrainment of slurry solids was noted.

#### SCALE FORMATION

- (1) Except during the injection tests, all scale formation that threatened the operation of the pilot plant was eliminated by the installation of an annular fresh water spray ring around the lower few inches of the Dustraxtor tube.

#### FULL SCALE SYSTEM

As indicated by results of the pilot test program on a single-tube open-loop Dustraxtor, scale-up to a full size unit (capable of handling 50,000 to 60,000 scfm) should not present any problems other than those already encountered and discussed on the pilot scale level. However, to handle larger quantities of boiler stack gases, the designer should consider the potential unequal gas distribution in the scrubber as the

number of tubes is increased. One solution might be to investigate the possibility of using fewer tubes of larger diameter (e.g., 16 or 18 inch) to increase scrubber capacity.

## APPENDIX A

### DETAILS OF ANALYSIS TECHNIQUES

#### SO<sub>2</sub> APPARATUS AND PROCEDURE

Sampling of flue gas for SO<sub>2</sub> determination was accomplished by passing the sample gas through a Dynasciences Model SS-330 SO<sub>2</sub> monitor, the output of which was continuously recorded on a strip chart recorder. Once the instrumentation was "debugged", operation of the equipment was simple and required little operator time. Based upon wet chemical analyses, it was found to be very accurate.

The complete sampling train is shown in Figure A-1. The sampling probe used at all three sample locations consisted of stainless steel tubing 6 inches long x 1/4 inch O.D. A 500 ml flask was placed after the sampling probe to trap any moisture which may have condensed in the sampling tube. The dry trap was completely immersed in an ice bath to cool the sample gas to approximately 32°F. A single acting diaphragm pump drew the gas sample through approximately 15 feet of Tygon tubing. A tee located in the sample line was fitted with a 1/4 inch needle valve. Excess gas, pumped by the diaphragm pump, which did not flow through the monitor was vented.

The Dynasciences SO<sub>2</sub> monitor is an electrochemical gas analyzer. The sample gas is passed over a selective permeable membrane, where the SO<sub>2</sub> is absorbed on a sensing electrode to form activated species capable of undergoing electro-oxidation. The resulting current is directly proportional to the partial pressure of SO<sub>2</sub> in the gas mixture. The current is amplified and the output of the amplifier recorded.

The sampling procedure is as follows:

1. Connect the sampling train to zero gas (nitrogen) and zero the SO<sub>2</sub> monitor.
2. Connect the sampling train to the calibration gas (guaranteed analysis gas - sulfur dioxide and nitrogen) and calibrate the SO<sub>2</sub> monitor.

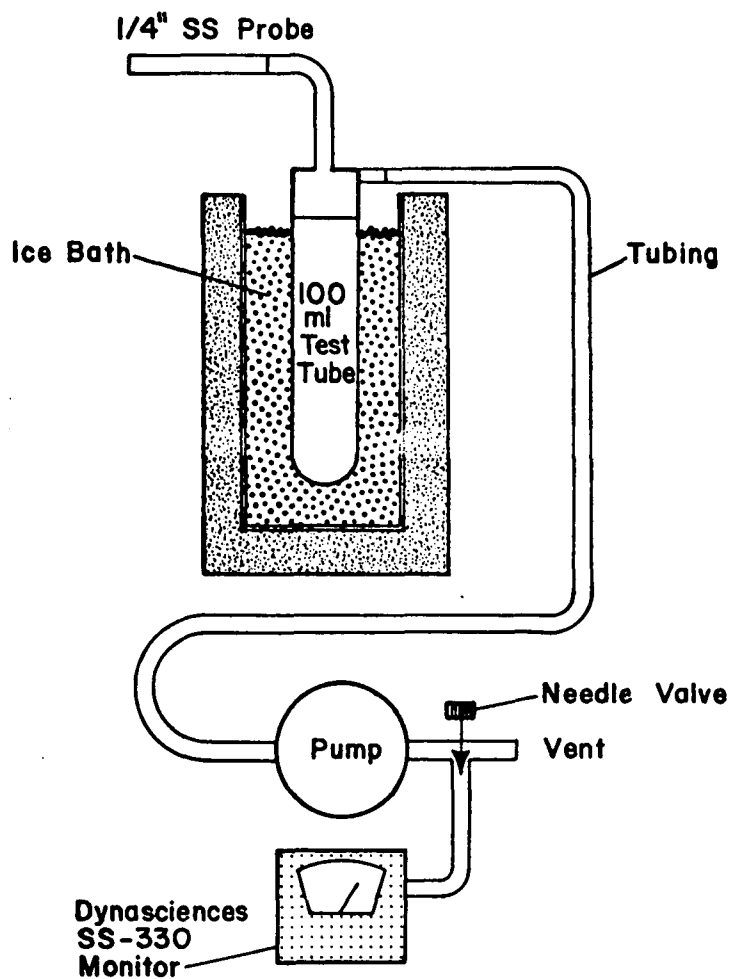


FIG. A-1.  $\text{SO}_2$  SAMPLING APPARATUS

3. Connect the sampling train to the pilot plant making sure that all joints are gas-tight. Make sure the appropriate sampling line is open and connected with the sampling train.
4. Turn on the pump and adjust the gas flow rate through the SO<sub>2</sub> monitor (approximately 1 scfm).

#### DUST LOADING APPARATUS AND PROCEDURE

The particulate sampling train is shown in Figure A-2. The probe, consisting of 1/4 inch stainless steel tubing, was connected to the first Greenberg-Smith impinger by a 4 foot length of Tygon tubing. Impingers No. 1 and No. 2 were each filled with 200 ml of distilled water. Impinger No. 3 was dry and used as a water trap.

In operation, the gas velocity through the duct was calculated before the start of the particulate sampling by means of the Annubar flow measuring element. The apparatus was assembled and the probe inserted into the center of the duct, open end upstream. Initial dry gas meter readings were recorded and the vacuum pump started. The flow rate through the dry gas meter was determined with a stop watch by measuring the time required for 0.1 ft<sup>3</sup> of gas to be pulled through the meter. The flow was then regulated by means of a gas control valve until the flow rate through the probe equalled the flow rate through the duct and an isokinetic condition existed. Between 10 and 20 standard cubic feet of gas was drawn through the train. The temperature and pressure inside the dry gas meter were recorded from the attached thermometer and vacuum gauge; the vacuum pump was shut off and the final dry gas meter reading was recorded.

#### NITROGEN OXIDE APPARATUS AND PROCEDURE

##### Principle

Stack gas samples are collected in evacuated flasks containing an absorbent consisting of hydrogen peroxide in dilute sulfuric acid. The nitric acid formed by the oxidation and absorption of nitrogen oxides is used to nitrate phenoldisulfonic acid which, when

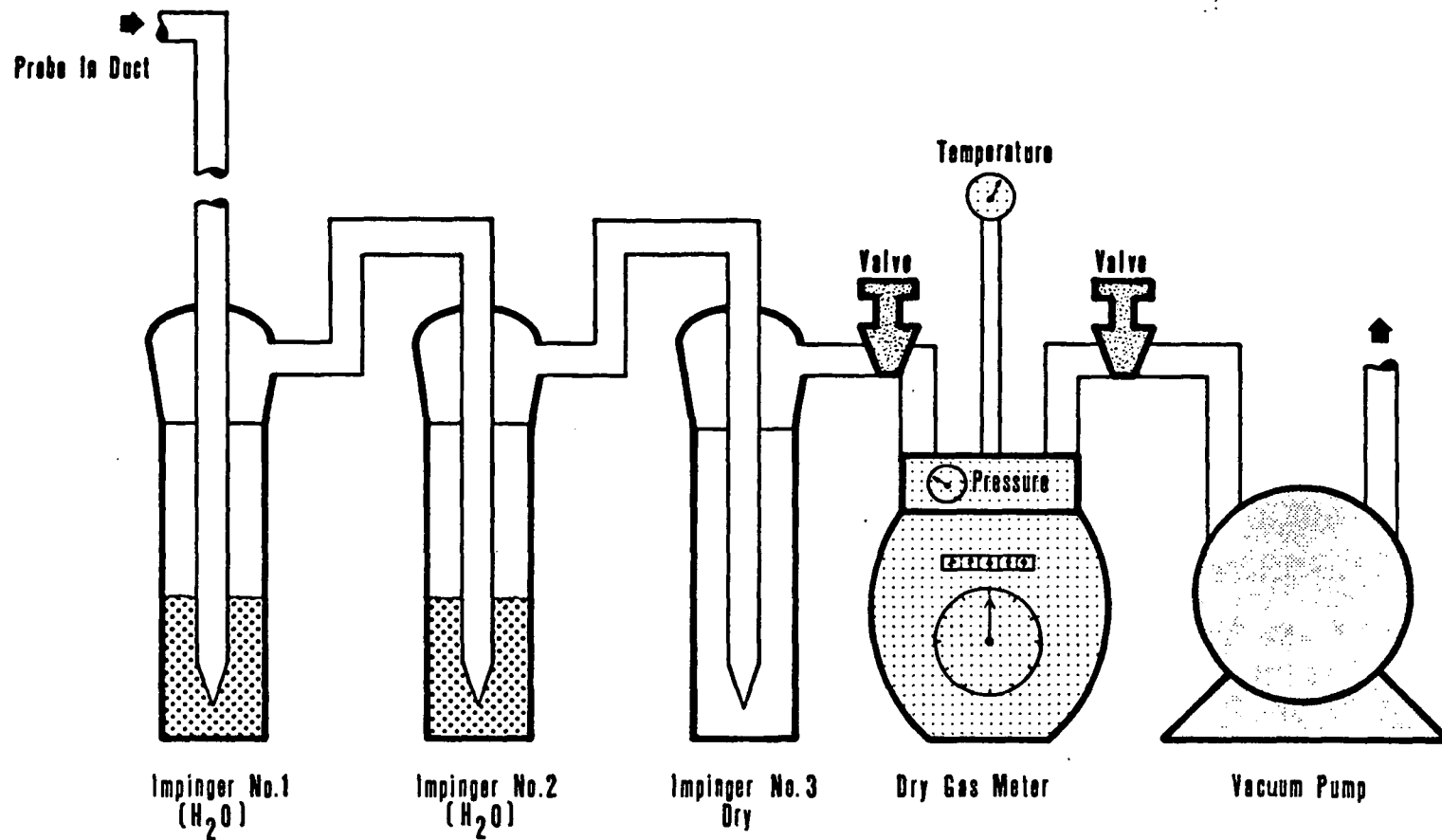


FIG. A-2. PARTICULATE SAMPLING TRAIN

reacted with ammonium hydroxide, forms a yellow compound (5-nitro, 6 hydroxy, 1, 3-benzenedisulfonic acid, triammonium salt). The intensity of the color produced is proportional to the concentration of nitrogen oxides in the sample and is measured spectrophotometrically at 420 mμ.

### Interference

Inorganic nitrates, nitrites, or organic bearing compounds easily oxidized to nitrates and interfere with this method. Reducing agents, such as  $\text{SO}_2$ , when present in high concentrations, may interfere by reacting with the hydrogen peroxide in the absorbing reagent to leave an insufficient amount for reaction with the nitrogen oxides. Halides tend to interfere, if present, and give lower results.

### Reagents

Hydrogen peroxide solution - Dilute 10 ml of 30 percent

$\text{H}_2\text{O}_2$  to 100 ml in a 100 ml volumetric flask with water.

Sulfuric acid (0.1 N) - Dilute 2.8 ml of concentrated  $\text{H}_2\text{SO}_4$  to 1 liter with water.

Absorbing reagent - Dilute 6 ml of 3 percent  $\text{H}_2\text{O}_2$  to 1 liter with 0.1 N  $\text{H}_2\text{SO}_4$ . This solution is stable and may be used for at least 30 days. Analyses in this laboratory have shown that the percent  $\text{H}_2\text{O}_2$  in the absorbing reagent remained constant over a 49 day period.

Sodium hydroxide (1 N) - Dissolve 40 gm of NaOH pellets in water and dilute to 1 liter.

Ammonium hydroxide (concentrated)

Sulfuric acid (fuming)



Phenoldisulfonic acid solution - Dissolve 25 grams of pure white phenol in 150 ml of concentrated  $\text{H}_2\text{SO}_4$  on a steam bath. Cool and add 75 ml fuming sulfuric acid. Heat to  $100^\circ\text{C}$  for 2 hours. Store in a dark stoppered reagent bottle.

Potassium nitrate solution (standard) - Dissolve 0.5495 grams of  $\text{KNO}_3$  in 1 liter of water in a volumetric flask. Dilute 100 ml of this solution to 1 liter in a volumetric flask. One ml of the final solution is equivalent to 0.025 mg  $\text{NO}_2$ .

### Collection of Samples

Emission sources containing oxides of nitrogen are sampled by a grab sampling technique using an evacuated 250 ml flask.

The following procedure is used for the collection of samples: Add 25 ml of absorbing solution to the sample flask. Evacuate the flask to the vapor pressure of the absorbing solution (approximately 20 mm Hg). Disconnect the vacuum pump line and accurately measure the vacuum in the flask. Connect the flask to the sample line and allow the flask to fill with a sample of stack gas until there is very little or no vacuum left. Measure precisely the final vacuum in the flask and record the flask temperature. Shake the flask for 15 minutes and allow to stand overnight to ensure complete reaction and absorption of the nitrogen oxides.

### Analysis

Transfer the contents of the collection flask to a 250 ml beaker. Wash the flask three times with 10 ml of water and add to the beaker. For a blank, add 25 ml of absorbing solution and 30 ml of water to a 250 ml beaker. Proceed as follows for both the sample and blank:

Add 1 N NaOH dropwise to the beaker until the solution is alkaline to litmus paper. Evaporate to dryness on a steam bath and allow to cool. Add 2 ml of phenoldisulfonic acid solution to the residue and triturate thoroughly with a glass stirring rod. Make sure all the residue comes in

contact with the solution. Add 1 ml H<sub>2</sub>O and 4 drops of concentrated H<sub>2</sub>SO<sub>4</sub>. Heat the solution on the steam bath for 3 minutes with occasional stirring.

Allow the solution to cool, add 20 ml H<sub>2</sub>O, mix well, and add 10 ml of concentrated NH<sub>4</sub>OH, dropwise, with constant stirring. Transfer the solution to a 50 ml volumetric flask. Wash the beaker three times with 5 ml portions of water. Dilute to 50 ml and mix thoroughly. Transfer a portion of the solution to a centrifuge tube and centrifuge for several minutes. If no centrifuge is available, filter the solution.

Determine the absorbency of each sample at 420 mμ. If the absorbency is outside the range of the calibration curve (e.g., absorbency >0.6), make a suitable dilution of the sample and blank and determine the absorbency. Obtain the number of milligrams of NO<sub>2</sub> present in the sample from a previously prepared calibration curve, where absorbency was plotted versus concentration.

#### Calculations

Calculate the concentration of oxides of nitrogen as NO<sub>2</sub> in parts per million by volume as follows:

$$\text{ppm NO}_2 = \frac{(5.24 \times 10^2)(C)}{V_S}$$

where C = concentration of NO<sub>2</sub>, mg

V<sub>S</sub> = gas sample volume at 70°F and 29.92 in. Hg, liters

Calculate the volume of gas sampled at standard conditions of 70°F, 29.92 in. Hg.

$$\text{Volume of gas sampled} = \frac{V_f(P_f - P_i) \times 530^\circ\text{R}}{T_f \times 29.92 \text{ in. Hg}}$$

where V<sub>f</sub> = flask volume, liters

P<sub>f</sub> = final flask pressure, in. Hg

P<sub>i</sub> = initial flask pressure, in. Hg

T<sub>f</sub> = flask temperature, °R

## CALCIUM--EDTA TITRIMETRIC METHOD

### Principle

When EDTA (ethylenediamine tetraacetic acid or its salts) is added to water containing both calcium and magnesium, it combines first with the calcium that is present. Calcium can be determined directly using EDTA when the pH is made sufficiently high so that the magnesium is largely precipitated as the hydroxide and an indicator is used which combines with the calcium only. Several indicators are available that will give a color change at the point where all of the calcium has been complexed by the EDTA at a pH of 12-13.

### Interference

Under conditions of this test, the following concentrations of ions cause no interference with the calcium hardness determination: copper, 2 mg/l; ferrous iron, 20 mg/l; ferric iron, 20 mg/l; manganese, 10 mg/l; zinc, 5 mg/l; lead, 5 mg/l; aluminum, 5 mg/l; tin, 5 mg/l. Orthophosphate will precipitate calcium at the pH of the test. Strontium and barium interfere with the calcium determination and alkalinity in excess of 30 mg/l may cause an indistinct endpoint with hard waters.

### Reagents

Sodium hydroxide, 1 N - Dissolve 40 g NaOH and dilute to 1 liter with distilled water.

Murexide (ammonium purpurate) was prepared by mixing it with NaCl.

a. Analytical reagent grade disodium ethylenediamine tetraacetate dihydrate, also called (ethylenedinitrilo) tetraacetic acid disodium salt (EDTA),  $\text{Na}_2\text{H}_2\text{C}_{10}\text{H}_{12}\text{O}_8\text{N}_2 \cdot 2\text{H}_2\text{O}$ , is commercially available. Weigh 3.723 g of the dry reagent, dissolve in distilled water, and dilute to 1,000 ml. Check the titer by standardizing against standard calcium solution.

b. The technical grade of the disodium salt of EDTA dihydrate may also be used if the titrant is allowed to stand for several days and is then filtered. Dissolve 4.0 g of such material in 800 ml distilled water. Standardize against standard calcium solution. Adjust the titrant so that 1.00 ml = 1.00 mg  $\text{CaCO}_3$ .

Because the titrant extracts hardness-producing cations from soft glass containers, store preferably in polyethylene and secondarily in Pyrex bottles. Compensate for gradual deterioration by periodic restandardization and a suitable correction factor.

### Procedure

Because of the high pH used in this procedure, the titration should be performed immediately after the addition of the alkali.

Use 1 ml of sample diluted to 50 ml with water.

Add 2.0 ml NaOH solution, or a volume sufficient to produce a pH of 12-13. Stir. Add 0.1-0.2 g of the indicator mixture selected (or 1-2 drops if a solution is used). Add EDTA titrant slowly with continuous stirring to the proper endpoint. When using murexide, the endpoint may be checked by adding 1 or 2 drops of titrant in excess to make certain that no further color change occurs.

$$\begin{aligned}\text{Calculation: } 1 \text{ ml EDTA} &= 0.4008 \text{ mg Ca}^{++} \\ \text{Ca}^{++} \text{ mg/l or ppm} &= \text{Vol. EDTA} \times 0.4008 \times 1000\end{aligned}$$

### MAGNESIUM--EDTA TITRIMETRIC METHOD

#### Principle

EDTA and its sodium salts form a chelated soluble complex when added to a solution of certain metal cations. If a small amount of a dye such as Eriochrome Black T is added to an aqueous solution containing calcium and magnesium ions at a pH of  $10.0 \pm 0.1$ , the solution will become wine red.

If EDTA is then added as a titrant, the calcium and magnesium will be complexed. After sufficient EDTA has been added to complex all the magnesium and calcium, the solution will turn from wine red to blue. This is the endpoint of the titration. Magnesium ion must be present to yield a satisfactory endpoint in the titration. A small amount of complexometrically neutral magnesium salt of EDTA is therefore added to the buffer, a step which automatically introduces sufficient magnesium and at the same time obviates a blank correction.

The sharpness of the endpoint increases with increasing pH. The pH, however, cannot be increased indefinitely because of the danger of precipitating  $\text{CaCO}_3$  or  $\text{Mg(OH)}_2$ , and because the dye changes color at high pH values. The pH value of  $10.0 \pm 0.1$  recommended in this procedure is a satisfactory compromise. A limit of 5 minutes is set for the duration of the titration in order to minimize the tendency toward  $\text{CaCO}_3$  precipitation.

### Interference

Some metal ions interfere with this procedure by causing fading or indistinct endpoints. This interference is reduced by the addition of certain inhibitors to the water sample prior to titration with EDTA.

Suspended or colloidal organic matter in the sample may also interfere with the endpoint but may be overcome by evaporating the aliquot to dryness on a steam bath, followed by heating in a muffle furnace at  $600^\circ\text{C}$  until the organic matter is completely oxidized. Dissolve the residue in 20 ml 1 N HCl, neutralize to pH 7 with 1 N NaOH, and make up to 50 ml with distilled water; cool to room temperature and continue according to the general procedure.

### Titration Precautions

Titrations are best conducted at or near normal room temperatures. The color change becomes impractically slow as the sample approaches freezing temperature. Indicator decomposition presents a problem in hot water.

The pH specified in the recommended procedure may result in an environment conducive to  $\text{CaCO}_3$  precipitation. Although the titrant can slowly redissolve such precipitates, a drifting endpoint will often yield low results. A time limit of 5 minutes for the overall procedure minimizes the tendency for  $\text{CaCO}_3$  to precipitate. The following three methods also combat precipitation loss:

- a. The samples can be diluted with distilled water to reduce the  $\text{CaCO}_3$  concentration. The simple expedient of diluting a 25 ml aliquot to 50 ml has been incorporated in the recommended procedure. If precipitation occurs at this dilution, modification b or c can be followed. Reliance upon too small an aliquot contributes a systematic error originating from the buret-reading error.
- b. If the approximate hardness of a sample is known or is ascertained by a preliminary titration, 90 per cent or more of the titrant can be added to the sample before the pH is adjusted with the buffer.
- c. The sample can be acidified and stirred for 2 minutes to expel  $\text{CO}_2$  before pH adjustment with the buffer. A prior alkalinity determination can indicate the amount of acid to be added to the sample for this purpose.

### Reagents

Buffer solution - Dissolve 1.179 g disodium salt of EDTA dihydrate and 0.644 g  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  in 50 ml distilled water. Add this solution to 16.9 g  $\text{NH}_4\text{Cl}$  and 143 ml conc.  $\text{NH}_4\text{OH}$  with mixing and dilute to 250 ml with distilled water.

Eriochrome Black T is mixed with NaCl and used as an indicator.

Standard EDTA titrant, 0.01M. Analytical reagent grade disodium ethylenediamine tetraacetate dihydrate, also called (ethylenedinitrilo) tetraacetic acid disodium salt (EDTA),  $\text{Na}_2\text{H}_2\text{C}_{10}\text{H}_{12}\text{O}_8 \cdot 2\text{H}_2\text{O}$ , is commercially available. Weigh 3.723 g of the dry reagent, dissolve in distilled water, and dilute to 1,000 ml. Check the titer by standardizing against standard calcium solution.

### Procedure

The aliquot of sample taken for the titration should require less than 15 ml of EDTA titrant. The duration of titration should not exceed 5 minutes measured from the time of the buffer addition.

Dilute 1 ml of the sample to 50 ml with distilled water. Add 1-2 ml of buffer solution. Add an appropriate amount of dry-powder indicator. Add EDTA to the endpoint.

### Calculation

1 ml EDTA = 0.2431 mg  $Mg^{++}$  (The calcium concentration, determination by the EDTA method, is required for this calculation since the volume of EDTA used in the above titration is consumed by both  $Ca^{++}$  and  $Mg^{++}$  ions.)

$$\text{Vol. EDTA } Mg^{++} = \text{Total EDTA Vol.} - \text{Vol. EDTA } Ca^{++} \text{ (from prior } Ca^{++} \text{ titration)}$$

$$\text{mg } Mg^{++}/1 = \text{Vol. EDTA } Mg^{++} \times 0.2431 \times 1000$$

### CHLORIDE--ARGENTOMETRIC METHOD

#### Principle

In a neutral or slightly alkaline solution, potassium chromate can be used to indicate the endpoint of the silver nitrate titration of chloride. Silver chloride is quantitatively precipitated before red silver chromate is formed.

#### Interference

Substances in amounts normally found in potable waters will not interfere. Bromide, iodide, and cyanide register as equivalent chloride concentrations. Sulfide, thiosulfate, and sulfite ions interfere. However, sulfite can be removed by treatment with hydrogen peroxide in a neutral solution, while sulfite and thiosulfate can be removed by treatment with hydrogen peroxide in alkaline solution. Orthophosphate in excess of 25 mg/l

interferes by precipitation as silver phosphate. Iron in excess of 10 mg/l will interfere by masking the endpoint.

### Reagents

Chloride-free water - If necessary, remove any chloride impurity from distilled water by redistillation from an all-pyrex apparatus or passage through a mixed bed of ion-exchange resins.

Potassium chromate indicator solution - Dissolve 50 g  $K_2CrO_4$  in a little distilled water. Add silver nitrate solution until a definite red precipitate is formed. Allow to stand 12 hrs, filter, and dilute filtrate to 1 liter with distilled water.

Standard silver nitrate titrant, 0.0141 N - Dissolve 2.396 g  $AgNO_3$  in distilled water and dilute to 1,000 ml. Standardize against 0.0141 N NaCl. Store in a brown bottle. Standard silver nitrate solution, exactly 0.0141 N, is equivalent to 0.500 mg Cl per 1.00 ml.

## NITRITE

### Principle

Diazotized sulfanilic acid, formed by the reaction between sulfanilic acid and  $NO_2$ , forms a reddish-purple azo dye by coupling with naphthylamine hydrochloride at pH 2 to 2.5. The nitrite concentration is determined by spectrophotometrically measuring this dye at 520 m.

### Interference

This method is not interfered with by relatively large amounts, up to 1,000 times, of the alkaline earths, zinc, nickel, arsenate, benzoate, borate, bromide, chloride, fluoride, iodate, molybdate, nitrate, phosphate,



sulfate, and thiocyanate. Numerous heavy metals such as gold, lead, bismuth, iron, or mercury interfere by precipitation and others because of colored salts. Aliphatic amines react with nitrites to liberate gaseous nitrogen. Ammonia does not interfere in the small concentrations usually encountered. Strong reducing or oxidizing agents should be absent.

### Reagents

Sulfanilic acid solution - Dissolve 0.60 g sulfanilic acid in 70 ml hot distilled water, cool, add 20 ml conc. HCl, dilute to 100 ml with distilled water, and mix thoroughly.

Naphthylamine hydrochloride solution - Dissolve 0.60 g 1-naphthylamine hydrochloride and 1 ml conc. HCl in distilled water and dilute to 100 ml.

Sodium acetate solution, 2M - Dissolve 16.4 g  $\text{NaC}_2\text{H}_3\text{O}_2$  or 27.2 g  $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$  in distilled water and dilute to 100 ml. Filter if the solution is not clear.

Stock sodium nitrite solution - Dissolve 0.492 g  $\text{NaNO}_2$  in 1,000 ml nitrite-free distilled water.

Standard sodium nitrite solution - Dilute 100.0 ml stock sodium nitrite solution to 1,000 ml; then dilute 50.0 ml of this solution to 1,000 ml with sterilized nitrite-free distilled water, add 1 ml chloroform, and preserve in a sterilized bottle; 1.0 ml = 0.5  $\mu\text{g}$  [N] or 1.6  $\mu\text{g}$   $\text{NO}_2^-$ .

Manganese sulfate solution - Dissolve 480 g  $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$  or 400 g  $\text{MnSO}_4 \cdot 2\text{H}_2\text{O}$  or 364 g  $\text{MnSO}_4 \cdot \text{H}_2\text{O}$  in distilled water, filter, and dilute to 1 liter.

Potassium permanganate solution - Dissolve 0.4 g  $\text{KMnO}_4$  in 1 liter distilled water.

**Ammonium oxalate solution** - Dissolve 0.9 g  $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$  in 1 liter distilled water.

**Nitrite-free water** - Add 1 ml conc.  $\text{H}_2\text{SO}_4$  and 0.2 ml manganous sulfate solution to 1 liter distilled water and make pink with 1 to 3 ml potassium permanganate solution. After 15 minutes decolorize with ammonium oxalate solution.

### Procedure

Using appropriate dilutions of standard sodium nitrite solution, prepare a curve for various  $\text{NO}_2^-$  concentrations ranging from 0.05 to 1.4 mg  $\text{NO}_2^-$ /l and plot against absorption at 520 m on a spectrophotometer using a light path of 2 cm.

Place a 10 ml sample in the tube. Measure 1.0 ml sulfanilic acid solution into the diluted sample, mix, and allow to stand at least 3 minutes and not more than 10 minutes for diazotization. The pH of this solution should be about 1.4

Add 1.0 ml naphthylamine hydrochloride solution and 1 ml sodium acetate solution. This should buffer the system to a pH of 2.5. Dilute to 50 ml and mix well. After 10 minutes, but before 20 minutes, measure the intensity of the reddish-purple color in a spectrophotometer, a filter photometer, or by comparison in Nessler tubes.

### Calculation

Read  $\text{NO}_2^-$  mg/l directly from the calibration curve.

### **NITRATE—BRUCINE METHOD**

#### Principle

The reaction between nitrate and brucine yields a sulfur yellow color employed for colorimetric estimation. The color system does not obey Beer's law, although in plotting transmittance against nitrate concentration

a smooth curve is produced. It is necessary to develop color simultaneously in a series of standards and samples. The intensity of the color is measured at 410 m .

The intensity of the maximum color produced varies more or less inversely with the temperature, while the rate of color development varies more or less directly with the temperature. The temperature generated upon mixing sulfuric acid with water can be controlled by adjusting the acid concentration. Both the acid concentration and the reaction time have been selected to yield optimum results and to compensate for any normal variations in room temperature.

### Interference

All strong oxidizing or reducing agents interfere. The presence of oxidizing agents may be determined by the addition of orthotolidine reagent as in the measurement of residual chlorine. The interference by residual chlorine may be eliminated by the addition of sodium arsenite, provided that the residual chlorine does not exceed 5 mg/l. A slight excess of sodium arsenite will affect the determination. Ferrous and ferric iron and quadrivalent manganese give slight positive interferences, but in concentrations less than 1 mg/l these interferences are minimized by the use of sulfanilic acid. Chlorides do not interfere.

### Reagents

Stock nitrate solution - Dissolve 0.7218 g anhydrous potassium nitrate,  $\text{KNO}_3$ , and dilute to 1,000 ml with distilled water. This solution contains 100 mg/l N.

Standard nitrate solution - Dilute 100.0 ml stock nitrate solution to 1,000 ml with distilled water; 1.00 ml = 10.0  $\mu\text{g}$  N.

Sodium arsenite solution - Dissolve 5.0 g  $\text{NaAsO}_2$  and dilute to 1 liter with distilled water. (CAUTION: Toxic; take care to avoid ingestion.)

Brucine-sulfanilic acid - Dissolve 1 g brucine sulfate and 0.1 g sulfanilic acid in approximately 70 ml hot distilled water. Add 3 ml conc.  $\text{HCl}$ , cool and make up to 100 ml. This solution is stable for several months. The pink color that develops slowly does not affect its usefulness. (CAUTION: Brucine is toxic; take care to avoid ingestion.)

Sulfuric acid solution - Carefully add 500 ml of conc.  $\text{H}_2\text{SO}_4$  to 74 ml distilled water. Cool to room temperature before use. Keep tightly stoppered to prevent absorption of atmospheric moisture.

### Procedure

Prepare a calibration curve by plotting concentrations from 0.1 to 2.6 mg/l  $\text{NO}_3^-$  against absorption at 410 m on a spectrophotometer using a light path of 2 cm.

### Color Development

Carefully pipet 2.00 ml of sample containing not more than 10 mg/l nitrogen into a 50 ml beaker. Add 1.0 ml brucine-sulfanilic acid reagent, using a safety pipet. Into a second 50 ml beaker measure 10 ml  $\text{H}_2\text{SO}_4$ . (An automatic buret is convenient for this purpose. The intensity of color is affected slightly by the heat capacity of the containers. The concentration of  $\text{H}_2\text{SO}_4$  has been chosen so that normal variations in heat capacities of beakers will not affect the result. It is important, however, that only 50 ml beakers be used.) Mix the contents of the two beakers by carefully adding the sample with brucine-sulfanilic acid reagent to the beaker containing acid. Pour from one beaker to the other four to six times to ensure mixing. Allow the treated sample to remain in the dark for  $10 \pm 1$  minutes. (The beaker may conveniently be covered

with a cardboard carton during this period.) While the sample is standing for color development, measure 10 ml distilled water into the empty beaker. After 10 minutes, add the water to the sample and mix as before. Allow to cool in the dark for 20-30 minutes. Set the blank at 100 per cent transmittance at a wave-length of 410 m $\mu$ . It is advisable to run a series of standards with each set of samples. With a proper arrangement of work, as many as twelve samples may be determined in a batch along with eight standards.

### Calculation

Read NO<sub>3</sub><sup>-</sup>mg/l directly from the calibration curve.

## SULFITE

### Principle

An acidified water sample containing sulfite is titrated with a standardized potassium iodide-iodate titrant. Free iodine is released when the sulfite has been completely oxidized, resulting in the formation of a blue color in the presence of starch indicator.

### Interference

The presence of other oxidizable substances in the water such as organic matter and sulfide will result in higher titration values for sulfite than are actually present. Nitrite, on the other hand, will combine with sulfite in the acid medium to destroy both, leading to low results. No interference occurs with the Dual-Purpose Dry Starch Indicator Powder because the sulfonic acid in this proprietary compound destroys the nitrite. Copper ion rapidly accelerates the oxidation of sulfite solution. Certain heavy metals may also react in a manner similar to copper. Proper sampling and immediate fixing by acid addition should minimize those difficulties.

## Reagents

Sulfuric acid, 1 + 1.

Starch solution - To 5 g starch (potato, arrowroot, or soluble) in a mortar, add a little cold distilled water and grind to a paste. Pour into 1 liter of boiling distilled water, stir, and allow to settle overnight. Use the clear supernant. Preserve by adding either 1.3 g salicylic acid, 4 g zinc chloride, or a combination of 4 g sodium propionate and 2 g sodium azide to 1 liter of starch solution.

Standard potassium iodide-iodate titrant, 0.0125 N - Dissolve 0.4458 g anhydrous potassium iodate,  $\text{KIO}_3$  (primary standard grade dried for several hours at  $120^\circ\text{C}$ ), 4.35 g potassium iodide,  $\text{KI}$ , and 0.31 g sodium bicarbonate,  $\text{NaHCO}_3$ , in distilled water, and dilute to 1,000 ml. This titrant is equivalent to 0.500 mg  $\text{SO}_3^{2-}$  per 1.00 ml.

## Procedure

Collect a fresh water sample with as little contact with air as possible. Cool hot samples to  $50^\circ\text{C}$  or below in the cooling apparatus. Do not filter the samples.

Add 1 ml  $\text{H}_2\text{SO}_4$  (or 1 g dual-purpose starch indicator) to a 250 ml Erlenmeyer flask or other titrating vessel, then measure 50 ml water sample in a graduated cylinder, and transfer to the flask. Add 1 ml starch indicator solution or 0.1 g starch powder. Titrate with potassium iodide-iodate titrant until a faint permanent blue color develops in the sample, signaling the end of the titration. View the color changes against a white background.

### Calculation

$$\text{mg/l SO}_3^{\equiv} \text{ equals } \frac{A \times N \times 40,000}{\text{ml sample}}$$

where: A = ml of titrant used for sample

N = normality of KI-KIO<sub>3</sub>.

### SULFATE -- GRAVIMETRIC METHOD

#### Principle

Sulfate is precipitated in a hydrochloric acid medium as barium sulfate by the addition of barium chloride. The precipitation is carried out near the boiling temperature and, after a period of digestion, the precipitate is filtered, washed with water until free of chlorides, ignited or dried, and weighed as BaSO<sub>4</sub>.

#### Interference

The gravimetric determination of sulfate is subject to many errors, both positive and negative. In potable waters where the mineral concentration is low, these may be of minor importance. The analyst should be familiar with the more common interferences, however, so that he may apply corrective measures when necessary.

#### Reagents

Methyl red indicator solution - Dissolve 0.1 g methyl red sodium salt in distilled water and dilute to 100 ml.

Hydrochloric acid- 1 + 1.

Barium chloride solution - Dissolve 100 g BaCl<sub>2</sub> · 2H<sub>2</sub>O in 1 liter distilled water. Filter through a membrane filter or hard-finish filter paper before use; 1 ml of this reagent is capable of precipitating approximately 40 mg SO<sub>4</sub><sup>=</sup>.

Asbestos cream - Add 15 g acid-washed medium-fiber asbestos, which is prepared especially for Gooch crucible determinations, to 1 liter distilled water. Remove the fine material from the asbestos before use by repeated decantations.

Silver nitrate-nitric acid - Dissolve 8.5 g  $\text{AgNO}_3$  and 0.5 ml conc.  $\text{HNO}_3$  in 500 ml distilled water.

### Procedure

Adjust the clarified sample -- treated if necessary to remove interfering agents -- to contain approximately 50 mg of sulfate ion in a 250 ml volume. Adjust the acidity with  $\text{HCl}$  to pH 4.5-5.0 using a pH meter or the orange color of methyl red indicator. Then add on additional 1 to 2 ml  $\text{HCl}$ . Lower concentrations of sulfate ion may be tolerated if it is impractical to concentrate the sample to the optimum level, but in such cases it is better to fix the total volume at 150 ml. Heat the solution to boiling and, while stirring gently, add warm barium chloride solution slowly until precipitation appears to be complete; then add about 2 ml in excess. If the amount of precipitate is small, add a total of 5 ml barium chloride solution. Digest the precipitate at  $80^\circ\text{--}90^\circ\text{C}$ , preferably overnight but for not less than 2 hours.

Prepare an asbestos filter mat in a Gooch crucible by using suitable suction apparatus. Wash with several portions of hot distilled water, dry, and ignite at  $800^\circ\text{C}$  for 1 hour. Cool the crucible in a desiccator and weigh.

Mix a small amount of ashless filter paper pulp with the barium sulfate, and filter at room temperature. The pulp aids filtration and reduces the tendency of the precipitate to creep. Wash the precipitate with small portions of warm distilled water until the washings are free of chlorides, as indicated by testing with silver nitrate-



nitric acid reagent. Dry the filter and precipitate and ignite at 800°C for 1 hour. Do not allow the filter paper to flame. Cool in a desiccator and weight.

Calculation

$$\text{mg/l SO}_4^{\text{--}} \text{ equals } \frac{\text{mgBaSO}_4 \times 411.5}{\text{ml sample}}$$

APPENDIX B  
ANNUBAR FLOW ELEMENT, CALCULATION EQUATIONS,  
AND ENGINEERING SPECIFICATIONS

Supplied by  
Ellison Instrument Division  
Boulder, Colorado



# ELLISON

## ANNUBAR FLOW CALCULATION REPORT

VERSON STANDARD CORP. • DRAWER M • BOULDER COLORADO 80302 USA • TEL 303/448-8000 • TLX 45-903 • CABLE DISTAN BOR

Annubar Primary Elements, like other differential measuring flow elements, utilize a form of the classical Bernoulli energy balance equation to determine flow rate. But, unlike most other flow elements, Annubar's differential pressure signal is consistent and uniform for a given pipe size and flow condition. Since it is uniform, only the operating range of the instrument or control system needs to be sized. This feature reduces

calculations and also allows future changes to a system's flow rate without the necessity of resizing and changing primary elements.

Sizing the operating range of the instrument to be used with an Annubar element is made easy with the charts and formulas shown below. If you prefer to have Ellison compute your instrument's operating range there is a small charge for this engineering service.

### HELPFUL HINTS FOR SIZING

1. Select one of the calculation methods shown below according to your accuracy requirements.
2. When flow is liquid, check your result against the "Quick-Size" chart in the Annubar catalogue, E-100. This is a fast way to double check calculations.
3. If your system's flow rates are extremely high or extremely low, it may be desirable to change the pipe size for the metered section. Changing diameters of

the metered section will not affect accuracy providing sufficient upstream and downstream lengths of pipe are provided . . . . see Table II.

4. Contact your local Ellison Engineering Representative or our factory if you need any further information or help in sizing your instrument's operating range . . . . we are here to serve you.

### EASY INSTRUMENT SIZING

Select the calculation method that meets your needs:

- A. QUICK-SIZE CHART — fastest sizing for water flows — see chart on the fold-out section of the Annubar catalogue, E-100.
- B. STANDARD "PLANT" EQUATIONS — for general industrial metering needs . . . . see "B" below.

C. PRECISE "THEORETICAL" EQUATIONS — for highest accuracy requirements and laboratory work . . . . see "C" on next page.

D. SLIDE RULE CALCULATOR — convenient for fast sizing of gases, liquids, and steam. Request form E-87 from your Ellison Representative.

### "B" STANDARD "PLANT" EQUATIONS

- 1a. Liquid volume flow rate,

$$Q_v = \text{SND}^2 \frac{\sqrt{G_f}}{G_f} \sqrt{h_a} \quad , \text{ or } \quad h_a = \left( \frac{Q_v G_f}{\text{SND}^2 \sqrt{G_f}} \right)^2$$

- 2a. Liquid weight flow rate,

$$W_s = \text{SND}^2 \sqrt{G_f} \sqrt{h_a} \quad , \text{ or } \quad h_a = \left( \frac{W_s}{\text{SND}^2 \sqrt{G_f}} \right)^2$$

- 3a. Gas volume flow rate at standard condition,\*

$$Q_v = 7.9 \text{ SND}^2 \frac{\sqrt{\gamma_f}}{\gamma_f} \sqrt{h_a} \quad , \text{ or } \quad h_a = \left( \frac{Q_v \gamma_f}{7.9 \text{ SND}^2 \sqrt{\gamma_f}} \right)^2$$

- 4a. Gas or steam weight flow rate,

$$W_s = .127 \text{ SND}^2 \sqrt{\gamma_f} \sqrt{h_a} \quad , \text{ or } \quad h_a = \left( \frac{W_s}{.127 \text{ SND}^2 \sqrt{\gamma_f}} \right)^2$$

## "C" PRECISE "THEORETICAL" EQUATIONS

### 1c. Liquid volume flow rate,

$$Q_v = \text{SND}^2 F_s F_m F_v \frac{\sqrt{G_t}}{G_t} \sqrt{h_n} \quad \text{or} \quad h_n = \left( \frac{Q_v G_t}{\text{SND}^2 F_s F_m F_v \sqrt{G_t}} \right)^2$$

### 2c. Liquid weight flow rate,

$$W_s = \text{SND}^2 F_s F_m F_v \sqrt{G_t} \sqrt{h_n} \quad \text{or} \quad h_n = \left( \frac{W_s}{\text{SND}^2 F_s F_m F_v \sqrt{G_t}} \right)^2$$

### 3c. Gas volume flow rate at standard condition,\*

$$Q_v = 7.897 \text{ SND}^2 F_s F_m V_s \frac{\sqrt{\gamma_t}}{\gamma_t} \sqrt{h_n} \quad \text{or} \quad h_n = \left( \frac{Q_v \gamma_t}{7.897 \text{ SND}^2 F_s F_m V_s \sqrt{\gamma_t}} \right)^2$$

### 4c. Gas or steam weight flow rate,

$$W_s = .1266 \text{ SND}^2 F_s F_m V_s \sqrt{\gamma_t} \sqrt{h_n} \quad \text{or} \quad h_n = \left( \frac{W_s}{.1266 \text{ SND}^2 F_s F_m V_s \sqrt{\gamma_t}} \right)^2$$

\*To determine flow at flowing conditions set  $\gamma_t = \gamma$ .

## EQUATION SYMBOLS

For convenience the symbols shown below are of equal value to those listed in leading handbooks. Page reference numbers are provided after most of the symbol definitions. These numbers refer to the reference pages

in "Principles and Practices of Flow Meter Engineering", handbook by L. K. Spink which is available from the Foxboro Company, Foxboro, Massachusetts.

D = Inside diameter of pipe in inches (exact). (p. 155)

$F_s$  = Expansion-contraction factor for inside area of pipe at flowing temperature to that at 68°F. Although " $F_s$ " in this definition is not the same as found in handbooks, its numerical value is the same.  $F_s = 1.000$  between +31 and +106°F. on steel pipe. (p. 156)

$F_m$  = Manometer correction factor. The value to correct for effect of density of the fluid that displaces the float or the manometer liquid in the measuring instrument. Do not use this factor if instrument's scale has correction factor included. (p. 157)

$F_v$  = Correction for compressibility of flowing liquid. When negligible use 1.00. (p. 271)

$G_t$  = Specific gravity of liquid at flowing temperature as compared to water at 60°F. which is 1.00. See Figure 2. (p. 158, 162, 163, 165, 262, 267)

$G_s$  = Specific gravity of liquid at 60°F. as compared to water at 60°F. which is 1.00. (p. 162, 264, 269)

$h_n$  = Differential pressure output of Annubar element in convenient units... see Table I.

N = Grouped constant including  $\sqrt{2g}$  (gravity acceleration),  $\pi/4$  (circular area), and conversion constants which depend on units chosen for  $Q_v$ ,  $W_s$ , and  $h_n$ .... see Table I. (p. 154)

$Q_v$  = Volume flow rate in convenient units. See Table I. (p. 148)

S = Constant factor for element at specific flow,  $S = K_s F_v$ . If  $K_s$  or  $F_v$  factors are other than those shown below, they will be supplied with individual Annubar elements or are available on request.

$K_s$  = Geometrical constant:

Element Types 710 & 720	Element Types 730 & 740
1/2" Pipe - $K_g = 0.722$	2" to 2 1/2" Pipe - $K_g = 0.878$
3/4" Pipe - $K_g = 0.732$	3" to 4" Pipe - $K_g = 0.877$
1" Pipe - $K_g = 0.755$	5" to 6" Pipe - $K_g = 0.878$
1 1/4" Pipe - $K_g = 0.776$	8" Pipe - $K_g = 0.887$
1 1/2" Pipe - $K_g = 0.812$	10" Pipe - $K_g = 0.898$
2" Pipe - $K_g = 0.846$	12" Pipe - $K_g = 0.908$
2 1/4" Pipe - $K_g = 0.863$	14" Pipe - $K_g = 0.917$
	16" Pipe - $K_g = 0.928$
	18" Pipe - $K_g = 0.938$
	20" Pipe - $K_g = 0.949$
	24" Pipe - $K_g = 0.969$

$F_v$  = Velocity distribution factor:

$F_v = 0.83$  for transition and turbulent flow.

$V_s$  = Gas adiabatic compression factor. Velocities below 12,000 ft./min. use 1.00.

$$V_s = \sqrt{\frac{k}{k-1} \frac{P_s}{P_t - P_s} \left( \left( \frac{P_t}{P_s} \right)^{\frac{k-1}{k}} - 1 \right)}$$

$k$  = Ratio of specific heats, or isentropic exponent. Gases which follow the perfect gas law (Diatomic gases such as Oxygen, Hydrogen, Nitrogen, Air and etc.) have  $k \approx 1.40$  under normal conditions). (264)

$P_s$  = Upstream static pressure in consistent absolute units.

$P_t$  = Total or Impact pressure at element in consistent absolute units.

\* Solving with digital equipment is recommended.

$W_s$  = Weight flow rate in convenient units. See Table I. (p. 148)

$\gamma_f$  = Specific weight at flowing conditions in pounds per cubic foot including compressibility.

$$\gamma_f = \frac{14.7 + \text{PSIG of line}}{14.7} \times \frac{520}{460 + \text{line temp } (^{\circ}\text{F})} \times \gamma_b$$

Also,  $\gamma_f$  = specific gravity of gas at flowing conditions times the weight of air (#/ft.<sup>3</sup>) at conditions equal to

flowing conditions. See Figure 1 and 3. (p. 333,372)

$\gamma_b$  = Specific weight of gas at base conditions in pounds per cubic foot.  $\gamma_b$  = Specific Gravity of gas at base conditions times the weight of air (#/ft.<sup>3</sup>) at base conditions. Air = .0765#/ft.<sup>3</sup> at standard (60°F./14.73 psia) base conditions. (p. 78)

TABLE I — Factor "N" Values for Various "n" Units

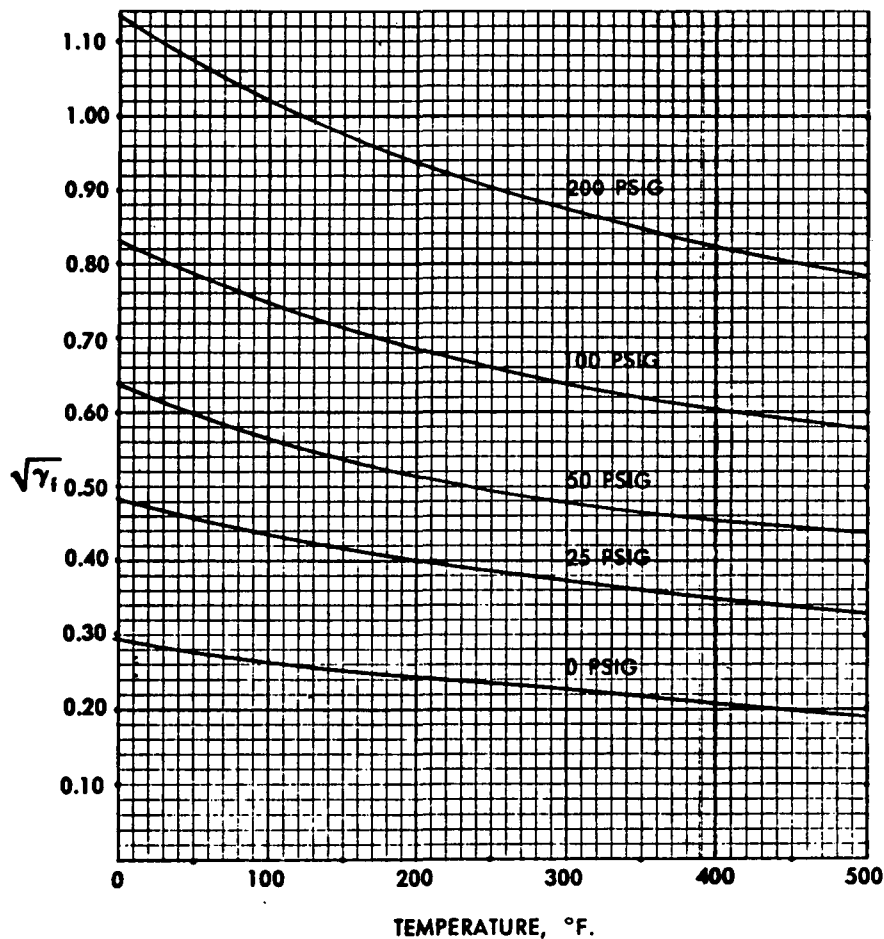
Q <sub>n</sub> , Volume	"h <sub>n</sub> " Units of Differential Pressure, Dry Calibration			
	IN. of H <sub>2</sub> O *	IN. of Hg. *	Kg./cm	P.S.I.
GPM	5.667	20.88	112.5	29.84
GPH	340.0	1252.0	6750.	1790.
CFM	0.7576	2.791	15.04	3.990
CFH	45.46	167.5	902.5	239.4
LPM	21.45	79.02	425.8	113.0

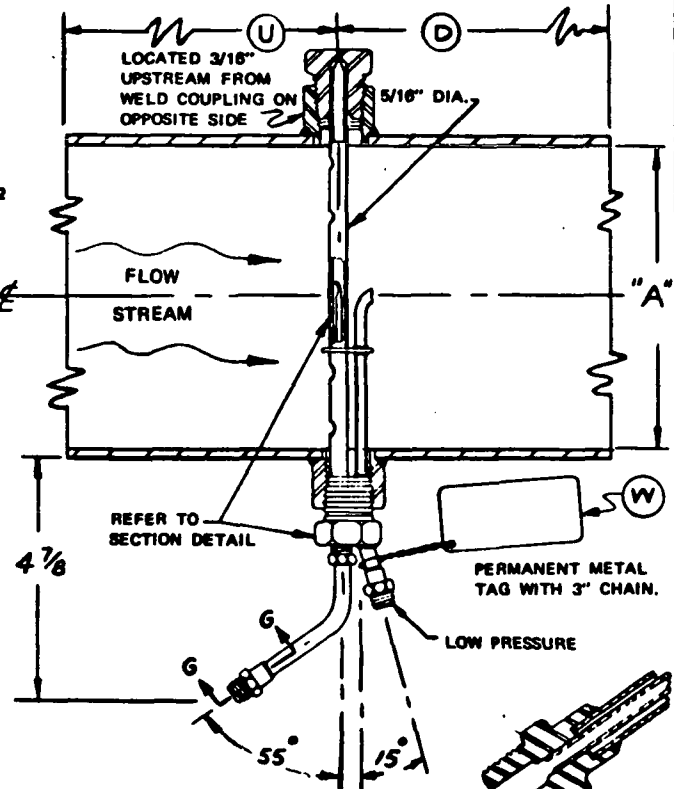
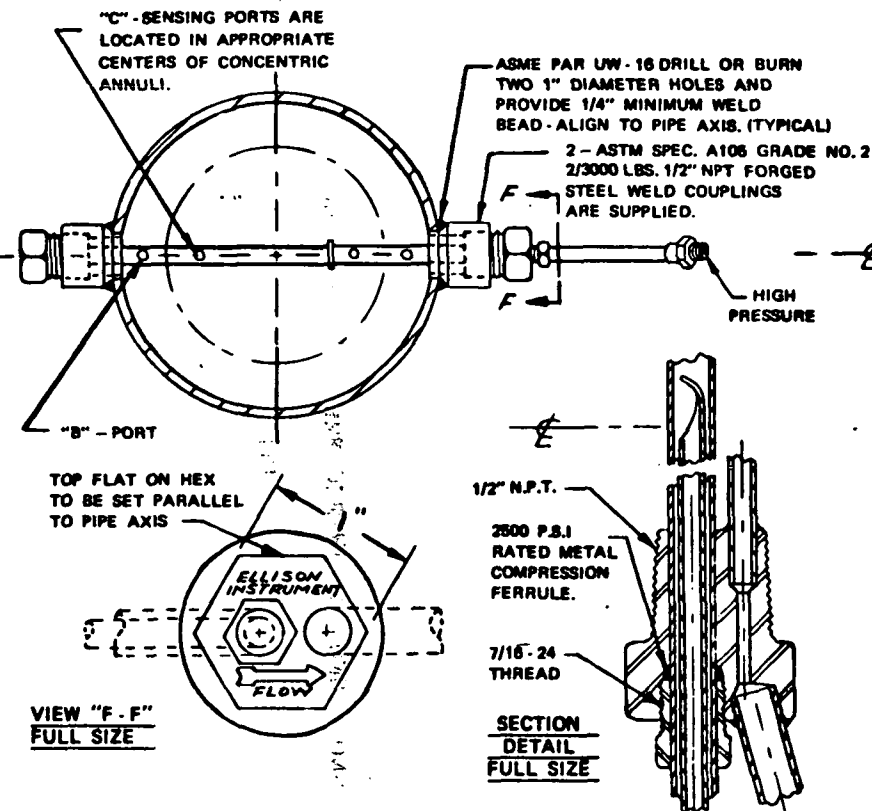
W<sub>n</sub>, Weight

PPM	47.25	174.1	938.0	248.8
PPH	2835.	10440.	56280.	14930.

\*Column readings corrected to 68° F.

FIGURE 1. VALUES OF  $\sqrt{\gamma_f}$  FOR AIR





PROJECT \_\_\_\_\_  
 LOCATION \_\_\_\_\_  
 MATERIAL \_\_\_\_\_  
 APPROVED BY \_\_\_\_\_

SECTION  
 "G-G"  
 FULL SIZE

- (D) 3 TO 4 PIPE DIAMETERS IS RECOMMENDED FOR DOWNSTREAM SIDE.
- (U) 8 OR MORE PIPE DIAMETERS IS RECOMMENDED FOR UPSTREAM SIDE AFTER VALVES, ELBOWS & ETC. - SEE FORM E-78.
- (W) PERMANENT TAG SHOWING MIN., NORM. & MAX. DESIGNED FLOWS, METER READINGS FOR DESIGNED FLOWS, TAG NO., LINE SIZE, SER. NO. & METERED FLUID.

ANOM. PIPE SIZE *	4	5	6	8	10	12	14	16	18	20	24
B HOLE DIA. - ALL	5/32	5/32	5/32	5/32	5/32	5/32	5/32	5/32	5/32	5/32	1/8
C NO. OF PORTS	4	4	4	4	4	4	4	4	4	4	4

\* SPECIFY PIPE SCHEDULE OR I.D. & O.D.

**DIETERICH STANDARD CORPORATION**  
**ELLISON INSTRUMENT DIVISION**  
 DRAWER M BOULDER COLORADO 80308 USA

**741 TO 744 ANNULAR FLOW ELEMENTS**

Date 2-5-69	Revisions A	Date 6-4-68	Revisions A
Drawn HRH	Checked GJD	Scale 3/8" = 1"	Part No.



SECTION D

**APPENDIX C**  
**SUMMARY OF SCRUBBER OPERATING CONDITIONS**

TABLE C-1. SCRUBBER OPERATING CONDITIONS--SERIES S-XX--KEY WEST INITIAL SALT WATER TESTS

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO ( $\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}$ )	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
S1	Preliminary	12	2000	8.5	-	-	-
S2	Preliminary	12	1013	6.5	-	-	-
S3	Preliminary	12	1013	6.5	-	-	-
S4	Preliminary	12	935	6.5	1:1	325	3
S5	-	-	-	-	V O I D	-	-
S6	-	-	-	-	V O I D	-	-
S7	-	-	-	-	V O I D	-	-
S8	-	-	-	-	V O I D	-	-
S9	-	-	-	-	V O I D	-	-
S10	-	-	-	-	V O I D	-	-
S11	Break in	12	1572	12.0	-	-	-
S12	Break in	12	788	6.5	-	-	-
S13	Break in	12	783	6.5	-	-	-
S14	Break in	12	789	6.5	-	-	-
S15	Break in	12	1538	12.0	-	-	-
S16	Break in	12	1538	12.0	-	-	-
S17	Break in	12	1652	12.0	-	-	-
S18	-	-	-	-	V O I D	-	-
S19	Break in	12	784	6.5	-	-	-



TABLE C-1 (contd). SCRUBBER OPERATING CONDITIONS--SERIES S-XX--KEY WEST INITIAL SALT  
WATER TESTS

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO ( $\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}$ )	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
S20	Break in	12	778	6.5	-	-	-
S21	-	-	-	- V O I D	-	-	-
S22	-	-	-	- V O I D	-	-	-
S23	Break in	12	795	12.0	-	-	-
S24	Break in	12	795	12.0	-	-	-
S25	Break in	12	1496	6.5	-	-	-
S26	Break in	12	1478	6.5	-	-	-
S27	Break in	12	1483	6.5	-	-	-
S28	Break in	12	1495	6.5	-	-	-
S29	Break in	12	1488	6.5	-	-	-
S30	Break in	12	1480	6.5	-	-	-
S31	Break in	12	1492	12.0	-	-	-
S32	Break in	12	1507	12.0	-	-	-
S33	Break in	12	1486	12.0	-	-	-
S34	Break in	12	810	12.0	-	-	-
S35	Break in	12	776	12.0	-	-	-
S36	Break in	12	776	12.0	-	-	-
S37	Break in	12	1500	12.0	-	-	-

TABLE C-1 (contd). SCRUBBER OPERATING CONDITIONS--SERIES S-XX--KEY WEST INITIAL SALT

## WATER TESTS

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO ( $\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}$ )	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
S38	Break in	12	1468	6.5	-	-	-
S39	Break in	12	784	12.0	-	-	-
S40	Break in	12	782	12.0	-	-	-
S41	Break in	12	781	12.0	-	-	-
S42	Break in	12	780	12.0	-	-	-
S43	Break in	12	778	6.5	-	-	-

TABLE C-2. SCRUBBER OPERATING CONDITIONS--SERIES C-XX--KEY WEST CORAL

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO ( $\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}$ )	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
C1	-	-	-	- V O I D	-	-	-
C2	Shakedown	12	781	6.5	1:1.9	325	1
C3	Shakedown	12	779	6.5	1:1.8	325	1
C4	Shakedown	12	1468	6.5	1:1	325	1
C5	Shakedown	12	1467	6.5	1:1	325	1
C6	Statistical	12	1463	12.0	1:0.9	325	1
C7	Duplicate C6	12	1478	12.0	1:1	325	1
C8	Statistical	12	775	6.5	1:1	325	1
C9	Extra	12	779	6.5	1:3	325	1
C10	Extra	12	1477	6.5	1:1	325	1
C11	-	-	-	- V O I D	-	-	-
C12	Duplicate C6	12	1370	12.0	1:1	325	1
C13	Statistical	12	721	12.0	1:3	325	1
C14	Statistical	12	1470	12.0	1:3	325	5
C15	Statistical	12	771	6.5	1:3	325	5
C16	Statistical	12	1452	6.5	1:1	325	5
C17	Statistical	12	772	12.0	1:1	325	5
C18	Statistical	12	1498	6.5	1:3	325	1
C19	Statistical	12	1460	12.0	1:3	100	1

TABLE C-2 (Cont.) SCRUBBER OPERATING CONDITIONS--SERIES C-XX--KEY WEST CORAL

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO ( $\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}$ )	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
C20	Statistical	12	794	6.5	1:3	100	1
C21	Statistical	12	794	12.0	1:1	100	1
C22	Statistical	12	1457	6.5	1:1	100	1
C23	Statistical	12	1483	6.5	1:3	100	1
C24	Statistical	12	788	12.0	1:3	100	5
C25	Statistical	12	1482	12.0	1:1	100	5
C26	Centerpoint	12	1146	9.0	1:2	325	3
C27	Centerpoint	12	1152	9.0	1:2	325	5
C28	Statistical	12	788	6.5	1:1	100	5
C29	-	-	-	-	V O I D	-	-
C30	Duplicate C17	12	782	12.0	1:1	325	5
C31	Centerpoint	12	1120	9.0	1:2	325	3
C32	Catalyst	12	1120	9.0	1:2	325	3
C33A	Centerpoint	12	1120	9.0	1:2	325	3
C33B	Inhibitor	12	1120	9.0	1:2	325	3
C34A	Inhibitor	12	1120	9.0	1:2	325	3
C34B	Inhibitor + Catalyst	12	1120	9.0	1:2	325	3

TABLE C-3. SCRUBBER OPERATING CONDITIONS--SERIES F-XX--KEY WEST FREDONIA VALLEY LIMESTONE

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO ( $\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}$ )	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
F1	-	-	-	- V O I D	-	-	-
F2	-	-	-	- V O I D	-	-	-
F3	Statistical	12	776	6.5	1:1	325	1
F4	Extra	12	766	6.5	1:3	325	1
F5	Extra	12	1462	6.5	1:1	325	1
F6	Extra	12	1476	6.5	1:1	325	1
F7	Statistical	12	1475	12.0	1:1	325	1
F8	Duplicate F3	12	777	6.5	1:1	325	1
F9	Statistical	12	1468	12.0	1:3	325	5
F10	Statistical	12	769	6.5	1:3	325	5
F11	Statistical	12	1497	6.5	1:1	325	5
F12	Statistical	12	1469	6.5	1:3	325	1
F13	Statistical	12	694	12.0	1:3.3	325	1
F14	Extra	12	781	12.0	1:1	325	5
F15	Statistical	12	1480	12.0	1:3	100	1
F16	Statistical	12	798	6.5	1:3	100	1
F17	Statistical	12	796	12.0	1:1	100	1
F18	Statistical	12	1468	6.5	1:1	100	1

TABLE C-3 (Cont.). SCRUBBER OPERATING CONDITIONS—SERIES F-XX—KEY WEST FREDONIA VALLEY LIMESTONE

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO $\left(\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}\right)$	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
F19	Statistical	12	800	6.5	1:1	100	5
F20	Statistical	12	1453	12.0	1:1	100	5
F21	Statistical	12	1460	6.5	1:3	100	5
F22	Statistical	12	765	12.0	1:3	100	5
F23	Centerpoint	12	1107	9.0	1:2	100	3
F24	Centerpoint	12	1140	9.0	1:2	325	3
F25	Centerpoint	12	1152	9.0	1:2	325	3
F26	Statistical	12	790	12.0	1:1	325	5
F27	Duplicate F7	12	1440	12.0	1:1	325	1
F28	Duplicate F9	12	1445	12.0	1:3	325	5
F29	Duplicate F22	12	778	12.0	1:3	100	5
F30	Duplicate F19	12	778	6.5	1:1	100	5
F31	Special	12	1317	12.0	1:3	325	1
F32	Special	12	1110	12.0	1:3	325	1
F33	Special	12	678	12.0	1:3	325	1
F34	Special	12	784	12.0	1:3	325	1
F35	Special	12	455	12.0	1:3	325	1
F35A	Special	12	1452	12.0	1:3	325	1

TABLE C-3 (Cont.). SCRUBBER OPERATING CONDITIONS--SERIES F-XX--KEY WEST FREDONIA VALLEY LIMESTONE

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO ( $\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}$ )	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
F36	Special	12	1093	12.0	1:3	325	1
F37	Special	8	1052	12.0	1:3	325	1
F38	Special	8	755	12.0	1:3	325	1
F39	Special	8	557	12.0	1:3	325	1
F40	Special	8	486	12.0	1:3	325	1
F41	Special	8	1320	12.0	1:3	325	1
F42	Centerpoint	12	1129	9.0	1:2	100	3

TABLE C-4. SCRUBBER OPERATING CONDITIONS--SERIES HL-XX--KEY WEST LIME, DOLOMITE, PRECIPITATED  
CaCO<sub>3</sub>, AND RECYCLED LIMESTONE

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO ( $\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}$ )	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
HL1	Statistical	12	1445	6.5	1:1	-	1
HL2	Statistical	12	790	12.0	1:1	-	1
HL3	Statistical	12	782	6.5	1:3	-	1
HL4	Statistical	12	1455	12.0	1:3	-	1
D1	Statistical	12	1430	6.5	1:1	-	1
D2	Statistical	12	1430	12.0	1:3	-	1
D3	Statistical	12	772	6.5	1:3	-	1
D4	Statistical	12	772	12.0	1:1	-	1
PC1	Statistical	12	1431	6.5	1:1	-	1
PC2	Statistical	12	778	12.0	1:1	-	1
PC3	Statistical	12	773	6.5	1:3	-	1
PC4	-	-	-	- V O I D	-	-	-
X1	Recycle	12	1110	-	-	-	3



TABLE C-5. SCRUBBER OPERATING CONDITIONS--SERIES P-XX--PADUCAH-SHAWNEE NO. 9 LIMESTONE

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO ( $\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}$ )	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
P1	Statistical	8	442	6.0	1:1	325	3
P2	Statistical	8	422	6.0	1:2	325	3
P3	Statistical	8	420	12.0	1:1	325	3
P4	Statistical	8	426	3	1:2	325	3
P5	Statistical	8	620	12.0	1:1	325	3
P6	Statistical	8	620	12.0	1:2	325	3
P7	Statistical	8	617	6.0	1:1	325	3
P8	Statistical	8	615	6.0	1:2	325	3
P9	Statistical	8	415	6.0	1:2	325	3
P10	Statistical	8	415	6.0	1:1	200	3
P11	Statistical	8	417	12.0	1:2	200	3
P12	Statistical	8	417	12.0	1:1	200	3
P13	Statistical	8	617	6.0	1:1	200	3
P14	Statistical	8	618	6.0	1:2	200	3
P15	Statistical	8	619	12.0	1:1	200	3
P16	Statistical	8	619	12.0	1:2	200	3
P17	-	-	-	- V O I D	-	-	-
P18	-	-	-	- V O I D	-	-	-

TABLE C-5. (Cont.) SCRUBBER OPERATING CONDITIONS—SERIES P-XX- --PADUCAH-SHAWNEE NO.9 LIMESTONE

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO ( $\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}$ )	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
P19	Centerpoint	8	518	9.0	1:1.5	325	3
P20	Centerpoint	8	518	9.0	1:1.5	325	3
P21	Centerpoint	8	517	9.0	1:1.5	325	3
P22	Centerpoint	8	520	9.0	1:1.5	325	3
P23	Centerpoint	8	520	9.0	1:1.5	200	3
P24	Centerpoint	8	520	9.0	1:1.5	200	3
P25	Centerpoint	8	520	9.0	1:1.5	200	3
P26	Centerpoint	8	520	9.0	1:1.5	200	3
P27	Catalyst	8	522	9.0	1:1.5	325	3
P28	Catalyst	8	522	9.0	1:1.5	325	3
P29	Catalyst	8	522	9.0	1:1.5	200	3
P30	Inhibitor	8	517	9.0	1:1.5	325	3
P31	Inhibitor	8	517	9.0	1:1.5	325	3
P32	Dry collector Bypassed	8	523	9.0	1:1.5	325	3
P33	Dry Collector Bypassed	8	620	6.0	1:1	325	3
P34	Dry Collector Bypassed	8	619	12.0	1:1	325	3

TABLE C-5. (Cont.) SCRUBBER OPERATING CONDITIONS--SERIES P-XX- --PADUCAH-SHAWNEE NO.9 LIMESTONE

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO ( $\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}$ )	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
P35	Dry Collector Bypassed	8	524	9.0	1:1.5	325	1

TABLE C-6. SCRUBBER OPERATING CONDITIONS--SERIES PA-XX, and PS-XX--PADUCAH-SHAWNEE, NO. 9  
ARAGONITE AND SIMULATED KEY WEST LIMESTONE

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO ( $\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}$ )	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
PA1	Statistical	8	620	6.0	1:1	325	3
PA2	Statistical	8	620	6.0	1:2	325	3
PA3	Statistical	8	386	12.0	1:1	325	3
PA4	Statistical	8	386	12.0	1:2	325	3
PA5	Dry Collector Bypassed	8	520	9.0	1:1.5	325	3
PA6	Dry Collector Bypassed	8	520	9.0	1:1.5	325	3
PA7	Centerpoint	8	522	9.0	1:1.5	325	3
PA8	Centerpoint	8	522	9.0	1:1.5	325	3
PS1	Simulated Salt Water	8	525	9.0	1:1.5	325	3
PS2	Simulated Salt Water	8	525	9.0	1:1.5	325	3
PS3	Simulated Salt Water	8	525	9.0	1:2	325	3

TABLE C-7 SCRUBBER OPERATING CONDITIONS--SERIES IPA-XX AND IP-XX--PADUCHA-SHAWNEE NO. 10  
ARAGONITE AND LIMESTONE INJECTION

Test Number	PURPOSE	TUBE SIZE (inches)	GAS FLOW RATE (scfm)	PRESSURE DROP (inches H <sub>2</sub> O)	STOICHIOMETRIC RATIO ( $\frac{\text{lb-mole reactant}}{\text{lb-mole entering SO}_2}$ )	PARTICLE SIZE (mesh)	SLURRY CONCENTRATION (weight %)
IPA1	Statistical	8	615	6.0	1:1.4	-	-
IPA2	Statistical	8	622	12.0	1:2	-	-
IPA3	Statistical	8	419	6.0	1:2	-	-
IPA4	Statistical	8	421	12.0	1:1	-	-
IP1	Centerpoint	8	516	9.0	1:1.85	-	-
IP2	Centerpoint	8	525	9.0	1:3.0	-	-
IP3	Centerpoint	8	523	9.0	1:0.68	-	-
IP4	Centerpoint	8	513	9.0	1:1.11	-	-
IP5	Centerpoint	8	522	9.0	1:2.46	-	-
IP6	Centerpoint	8	526	9.0	1:1.31	-	-
IP7	Centerpoint	8	512	9.0	1:1.47	-	-
IP8	Centerpoint	8	513	9.0	1:1.47	-	-
IP9	Special	8	618	12.0	1:0.95	-	-
IP10	Special	8	618	12.0	1:0.95	-	-
IP11	Special	8	614	12.0	1:1.45	-	-
IP12	Special	8	617	6.0	1:0.99	-	-
IP13	Special	8	368	6.0	1:0.99	-	-

**APPENDIX D**  
**SUMMARY OF TEST DATA**

Table D-1. DATA CONSOLIDATION -- SERIES S-XX -- KEY WEST INITIAL SALT WATER TESTS

Test Number	GAS HUMIDITY (lb/lb dry air)		DEW POINT (°F)		DRY GAS FLOW RATE (lb/min)	LIQUID FLOW RATE (lb/min)	GAS VELOCITY (ft/sec)	LIQUID/GAS RATIO		ABSORPTION PARAMETERS		
										Entering SO <sub>2</sub> Concentration	Leaving SO <sub>2</sub> Concentration	SO <sub>2</sub> Removal Efficiency
	IN	OUT	IN	OUT	(lb/min)	(lb/min)	(ft/sec)	(gal/1000ft <sup>3</sup> )	(lb/lb)	(ppm)	(ppm)	(%)
S5	-	-	-	-	-	V O I D	-	-	-	-	-	-
S6	-	-	-	-	-	V O I D	-	-	-	-	-	-
S7	-	-	-	-	-	V O I D	-	-	-	-	-	-
S8	-	-	-	-	-	V O I D	-	-	-	-	-	-
S9	-	-	-	-	-	V O I D	-	-	-	-	-	-
S10	-	-	-	-	-	V O I D	-	-	-	-	-	-
S11	0.130	0.072	138.0	116.0	107.12	2644.9	46.26	142.20	21.85	505	245	51.5
S12	0.078	0.071	118.8	115.7	56.29	656.9	21.58	75.71	10.83	470	350	25.6
S13	0.085	0.063	121.5	112.2	55.57	656.9	21.67	75.42	10.90	510	360	29.4
S14	0.085	0.050	121.3	104.5	55.99	656.9	21.71	75.27	10.81	495	305	38.4
S15	0.086	0.092	121.8	124.0	109.05	2644.9	44.56	147.62	22.33	520	320	38.4
S16	0.069	0.084	115.0	121.0	110.78	2644.9	44.56	147.62	22.33	510	290	43.2
S17	0.089	0.076	123.0	118.0	116.81	2644.9	46.90	140.27	20.79	460	190	58.7
S18	-	-	-	-	-	V O I D	-	-	-	-	-	-
S19	0.106	0.078	128.6	118.5	54.58	656.9	21.81	74.90	10.88	655	485	26.0
S20	0.084	0.066	120.8	113.5	55.26	656.9	21.88	74.68	10.96	805	540	32.9
S21	-	-	-	-	-	V O I D	-	-	-	-	-	-
S22	-	-	-	-	-	V O I D	-	-	-	-	-	-
S23	0.093	0.059	124.2	110.0	56.01	3764.8	22.88	340.45	61.50	672	375	44.2

NOTE: Tests S1 through S4 were preliminary tests. No calculations were performed.

Table D-1 (cont) DATA CONSOLIDATION -- SERIES S-XX -- KEY WEST INITIAL SALT WATER TESTS

Test Number	GAS HUMIDITY (lb/lb dry air)		DEW POINT (°F)		DRY GAS FLOW RATE (lb/min)	LIQUID FLOW RATE (lb/min)	GAS VELOCITY (ft/sec)	LIQUID/GAS RATIO		ABSORPTION PARAMETERS		
	IN	OUT	IN	OUT				(gal/1000ft <sup>3</sup> )	(lb/lb)	Entering SO <sub>2</sub> Concentration (ppm)	Leaving SO <sub>2</sub> Concentration (ppm)	SO <sub>2</sub> Removal Efficiency (%)
S24	0.094	0.078	124.7	118.5	55.96	3764.8	22.88	340.45	61.50	690	405	41.3
S25	0.089	0.100	122.8	126.5	105.78	750.8	43.40	43.03	6.52	565	408	27.8
S26	0.086	0.090	121.8	123.3	104.79	750.8	43.71	42.72	6.60	622	438	29.6
S27	0.093	0.077	124.3	118.1	104.47	750.8	43.61	42.82	6.58	620	430	30.6
S28	0.091	0.090	123.7	123.1	105.51	750.8	43.33	43.10	6.52	630	462	26.7
S29	0.092	0.088	124.0	122.3	104.92	750.8	43.71	42.72	6.55	630	450	28.6
S30	0.088	0.079	122.5	119.2	104.76	750.8	42.95	39.29	6.59	625	445	28.8
S31	0.086	0.087	121.8	122.0	105.79	2883.8	42.65	167.33	25.10	518	325	37.3
S32	0.092	0.077	124.0	118.0	106.26	2883.8	43.12	166.34	24.85	505	290	42.6
S33	0.078	0.068	118.7	114.0	106.14	2883.8	42.87	167.33	25.20	500	260	48.0
S34	0.074	0.046	117.0	102.0	58.07	3131.2	22.07	352.88	50.20	482	225	53.4
S35	0.085	0.060	121.4	110.5	55.07	2883.8	21.88	327.84	48.26	465	270	41.9
S36	0.085	0.068	121.3	114.0	55.07	2883.8	21.94	326.89	48.26	510	254	50.2
S37	0.085	0.087	121.3	122.0	106.45	2644.9	43.18	152.33	22.90	465	257	44.7
S38	0.089	0.083	122.8	120.5	103.80	802.0	43.29	46.08	7.09	450	322	28.5
S39	0.081	0.069	119.8	114.8	55.84	3131.2	21.75	358.05	51.87	443	236	46.7
S40	0.086	0.061	121.7	110.7	55.45	3131.2	21.77	357.70	52.00	445	242	45.6
S41	0.082	0.045	120.1	101.9	55.58	3131.2	21.79	357.35	52.07	465	210	54.8
S42	0.085	0.076	121.5	117.8	55.35	3131.2	21.71	358.75	52.13	480	242	49.6
S43	0.084	0.055	121.0	107.7	55.26	802.0	21.84	91.35	13.39	410	220	46.3



Table D-2. DATA CONSOLIDATION-- SERIES C-XX-- KEY WEST CORAL

Test Number	GAS HUMIDITY (lb/lb dry air)		DEW POINT (°F)		DRY GAS FLOW RATE (lb/min)	LIQUID FLOW RATE (lb/min)	GAS VELOCITY (ft/sec)	LIQUID/GAS RATIO		ABSORPTION PARAMETERS		
	IN	OUT	IN	OUT				(gal/1000ft <sup>3</sup> )	(lb/lb)	Entering SO <sub>2</sub> Concentration (ppm)	Leaving SO <sub>2</sub> Concentration (ppm)	SO <sub>2</sub> Removal Efficiency (%)
C1	-	-	-	-	-	V O I D	-	-	-	-	-	-
C2	0.076	0.073	117.9	116.5	54.87	704.4	21.75	80.00	11.71	438	97	77.9
C3	0.087	0.083	122.0	120.5	55.18	704.4	21.81	79.77	11.74	465	100	78.5
C4	0.029	0.094	122.7	124.6	104.47	807.5	43.29	46.08	7.14	454	173	61.9
C5	0.087	0.097	122.3	125.5	104.69	807.5	43.39	45.97	7.15	436	174	60.1
C6	0.089	0.103	122.6	127.8	104.40	2439.6	43.39	138.88	21.66	492	154	68.7
C7	0.091	0.103	123.5	128.0	104.31	2439.6	43.93	137.20	21.44	436	128	70.7
C8	0.094	0.091	124.8	123.6	54.75	704.4	22.05	78.92	11.80	415	147	64.6
C9	0.085	0.077	121.2	118.0	54.93	704.4	21.84	79.69	11.74	414	75	81.9
C10	0.087	0.093	122.2	124.4	104.82	807.5	43.18	46.19	7.10	415	180	56.6
C11	-	-	-	-	-	V O I D	-	-	-	-	-	-
C12	0.095	0.103	124.9	127.8	95.99	2903.5	46.26	155.05	27.52	695	240	65.5
C13	0.089	0.077	123.0	118.2	51.03	2663.0	23.58	279.03	47.97	690	67.5	90.2
C14	0.094	0.103	124.5	127.8	103.46	2983.4	44.14	162.50	7.08	960	240	75.0
C15	0.079	0.093	119.0	124.2	55.02	723.6	22.11	78.69	12.19	960	155	83.8
C16	0.092	0.106	124.0	128.8	102.57	829.4	42.44	47.00	7.42	980	410	58.2
C17	0.091	0.097	123.5	125.6	54.39	2982.4	21.35	335.98	50.17	980	191	80.5
C18	0.075	0.068	117.5	114.1	107.30	807.5	41.97	47.52	7.00	920	298	67.6
C19	0.078	0.072	118.7	116.0	104.29	2903.5	40.95	175.13	25.83	715	80	88.8

Table D-2 (cont). DATA CONSOLIDATION -- SERIES C-XX -- KEY WEST CORAL

Test Number	GAS HUMIDITY (lb/lb dry air)		DEW POINT (°F)		DRY GAS FLOW RATE (lb/min)	LIQUID FLOW RATE (lb/min)	GAS VELOCITY (ft/sec)	LIQUID/GAS RATIO		ABSORPTION PARAMETERS		
	IN	OUT	IN	OUT				(gal/1000ft <sup>3</sup> )	(lb/lb)	Entering SO <sub>2</sub> Concentration (ppm)	Leaving SO <sub>2</sub> Concentration (ppm)	SO <sub>2</sub> Removal Efficiency (%)
C20	0.074	0.061	117.0	110.6	56.92	704.4	21.50	80.95	11.52	735	108	85.3
C21	0.075	0.069	117.5	114.8	56.87	2903.5	21.43	334.65	47.49	735	198	73.1
C22	0.081	0.084	119.6	121.0	103.78	807.5	41.59	47.96	7.20	815	355	56.4
C23	0.081	0.088	119.6	122.4	104.95	829.4	42.55	46.88	7.26	800	202	74.7
C24	0.081	0.089	119.6	122.9	56.13	2735.4	22.28	295.24	45.08	750	48	93.6
C25	0.082	0.103	120.2	128.0	105.47	2982.4	42.29	169.59	26.14	750	128	82.9
C26	0.088	0.101	122.4	126.9	81.10	1689.2	32.25	127.63	19.14	780	225	71.2
C27	0.084	0.101	121.0	127.0	81.83	1689.2	32.47	126.80	19.04	825	243	70.5
C28	0.081	0.088	119.6	122.4	56.13	723.6	21.75	80.00	11.92	770	166	78.4
C29	-	-	-	-	-	V O I D	-	-	-	-	-	-
C30	0.083	0.095	120.5	124.8	55.60	2982.4	21.75	329.76	49.53	810	45	94.4
C31	0.083	0.094	120.5	124.5	79.63	1436.7	32.36	108.20	16.66	1000	262	73.8
C32	0.083	0.094	120.5	124.5	79.63	1436.7	32.36	108.20	16.66	1000	230	77.0
C33A	0.085	0.101	121.3	126.9	79.48	1436.7	32.57	107.49	16.66	1000	312	68.8
C33B	0.085	0.101	121.3	126.9	79.48	1436.7	32.57	107.49	16.66	1000	265	73.5
C34A	0.085	0.101	121.3	126.9	79.48	1436.7	32.57	107.49	16.66	1000	265	73.5
C34B	0.085	0.101	121.3	126.9	79.48	1436.7	32.57	107.49	16.66	1000	255	74.5

Table D-3. DATA CONSOLIDATION -- SERIES F-XX -- KEY WEST FREDONIA VALLEY LIMESTONE

Test Number	GAS HUMIDITY (lb/lb dry air)		DEW POINT (°F)		DRY GAS FLOW RATE (lb/min)	LIQUID FLOW RATE (lb/min)	GAS VELOCITY (ft/sec)	LIQUID/GAS RATIO		ABSORPTION PARAMETERS		
	IN	OUT	IN	OUT				(gal/1000ft <sup>3</sup> )	(lb/lb)	Entering SO <sub>2</sub> Concentration (ppm)	Leaving SO <sub>2</sub> Concentration (ppm)	SO <sub>2</sub> Removal Efficiency (%)
F1	-	-	-	-	-	V O I D	-	-	-	-	-	-
F2	-	-	-	-	-	V O I D	-	-	-	-	-	-
F3	0.092	0.099	123.9	126.3	54.72	704.4	22.49	77.36	11.79	491	142	71.1
F4	0.077	0.088	118.2	122.5	54.77	704.4	22.22	78.32	11.94	494	83	83.2
F5	0.091	0.094	123.5	124.6	103.18	704.4	43.46	40.04	6.26	465	210	54.8
F6	0.090	0.093	123.2	124.2	104.27	807.5	43.08	46.31	7.10	415	172	58.6
F7	0.085	0.098	121.4	126.0	104.68	2903.5	43.25	165.85	25.56	415	97.5	76.5
F8	0.079	0.083	119.0	120.6	55.45	704.4	21.94	79.30	11.77	413	111	73.1
F9	0.092	0.103	123.9	127.5	103.51	2982.4	43.12	166.30	26.38	414	52	87.4
F10	0.087	0.093	122.1	124.2	54.22	723.6	22.07	78.85	12.22	414	48	88.4
F11	0.093	0.110	124.3	130.0	105.46	829.4	45.16	44.17	7.20	414	207	50.0
F12	0.094	0.099	124.7	126.3	103.39	807.5	44.77	44.55	7.14	414	116	71.9
F13	0.092	0.082	124.0	120.5	48.94	2903.5	20.39	351.72	54.33	510	49	90.4
F14	0.079	0.075	119.0	117.5	55.79	2735.4	20.99	313.45	45.49	920	460	50.0
F15	0.080	0.072	119.5	116.0	105.52	2903.5	41.06	174.68	25.48	775	135	82.6
F16	0.075	0.061	117.5	111.0	57.16	704.4	21.43	81.19	11.46	780	165	78.8
F17	0.075	0.070	117.5	115.5	57.02	2903.5	21.43	334.70	47.37	780	183	76.5
F18	0.082	0.091	120.5	123.5	104.47	807.5	43.08	46.31	7.14	840	435	48.2
F19	0.077	0.088	118.2	122.7	57.20	723.6	22.13	78.62	11.75	775	143	81.5

Table D-3 (cont). DATA CONSOLIDATION -- SERIES F-XX -- KEY WEST FREDONIA VALLEY LIMESTONE

Test Number	GAS HUMIDITY (lb/lb dry air)		DEW POINT (°F)		DRY GAS FLOW RATE (lb/min)	LIQUID FLOW RATE (lb/min)	GAS VELOCITY (ft/sec)	LIQUID/GAS RATIO		ABSORPTION PARAMETERS		
	IN	OUT	IN	OUT				(gal/1000ft <sup>3</sup> )	(lb/lb)	Entering SO <sub>2</sub> Concentration (ppm)	Leaving SO <sub>2</sub> Concentration (ppm)	SO <sub>2</sub> Removal Efficiency (%)
F20	0.086	0.110	121.6	130.0	103.02	2982.4	42.06	170.53	26.66	830	420	49.4
F21	0.088	0.10	122.5	126.5	103.33	829.4	42.76	46.65	7.38	830	260	68.7
F22	0.078	0.073	118.7	116.5	54.64	2735.6	21.16	310.93	46.44	720	30	95.8
F23	0.085	0.088	121.5	122.5	78.56	1689.2	31.41	131.08	19.82	780	120	84.6
F24	0.085	0.088	121.3	122.5	80.90	1689.2	32.04	128.48	19.24	780	110	85.9
F25	0.084	0.088	121.0	122.6	81.83	1689.2	32.57	126.38	19.04	820	122	85.1
F26	0.083	0.089	120.5	122.8	56.17	2982.4	21.60	332.02	49.03	780	195	75.0
F27	0.088	0.095	122.5	125.0	101.91	2903.5	42.23	169.85	26.19	810	185	77.2
F28	0.081	0.103	120.0	127.7	102.93	2982.4	42.23	169.85	26.80	800	112	86.0
F29	0.090	0.094	123.4	124.6	54.96	2982.4	21.96	326.57	49.78	855	82.5	90.4
F30	0.089	0.103	123.0	127.6	55.01	723.6	22.17	78.47	12.08	855	225	73.7
F31	0.077	0.080	118.5	119.5	94.16	2663.0	38.51	170.80	26.26	922	204	79.4
F32	0.086	0.070	121.5	115.5	78.77	2783.2	31.62	217.45	32.56	965	155	83.9
F33	0.092	0.069	123.8	115.0	47.81	2439.6	18.67	322.73	46.73	1015	57	94.4
F34	0.092	0.069	123.8	115.0	55.28	3152.6	21.75	358.05	52.22	1015	70	93.1
F35	0.089	0.069	123.0	114.7	32.17	1846.9	12.43	366.89	52.72	1015	38	96.3
F35A	0.089	0.069	123.0	114.7	102.66	2293.6	42.95	131.92	20.51	1015	180	82.3
F36	0.082	0.076	120.0	118.0	77.78	2663.0	31.83	206.67	31.64	1000	144	85.6
F37	0.088	0.062	122.5	111.5	74.45	755.9	68.05	61.75	9.33	965	286	70.4

Table D-3 (cont). DATA CONSOLIDATION --SERIES F-XX --KEY WEST FREDONIA VALLEY LIMESTONE

Test Number	GAS HUMIDITY (lb/lb dry air)		DEW POINT (°F)		DRY GAS FLOW RATE (lb/min)	LIQUID FLOW RATE (lb/min)	GAS VELOCITY (ft/sec)	LIQUID/GAS RATIO		ABSORPTION PARAMETERS		
	IN	OUT	IN	OUT				(gal/1000ft <sup>3</sup> )	(lb/lb)	Entering SO <sub>2</sub>	Leaving SO <sub>2</sub>	SO <sub>2</sub> Removal
										Concentration (ppm)	Concentration (ppm)	Efficiency (%)
F38	0.088	0.058	122.3	109.0	53.43	953.5	47.56	111.45	16.40	965	183	81.0
F39	0.086	0.054	121.7	107.0	39.49	919.2	34.34	149.23	21.43	975	116	88.1
F40	-	-	-	-	-	859.0	30.66	155.76	22.95	975	94.5	90.3
F41	0.086	0.070	121.8	115.3	93.33	438.1	85.96	28.33	4.34	965	415	57.0
F42	0.086	0.101	121.5	126.8	80.05	1436.7	33.74	103.77	16.53	970	285	70.6

Table D-4. DATA CONSOLIDATION-- SERIES HL-XX, D-XX, PC-XX, AND X-XX-- KEY WEST LIME,  
DOLOMITE, PRECIPITATED CaCO<sub>3</sub>, AND RECYCLED LIMESTONE

Test Number	GAS HUMIDITY (lb/lb dry air)		DEW POINT (°F)		DRY GAS FLOW RATE (lb/min)	LIQUID FLOW RATE (lb/min)	GAS VELOCITY (ft/sec)	LIQUID/GAS RATIO		ABSORPTION PARAMETERS		
	IN	OUT	IN	OUT				(gal/1000ft <sup>3</sup> )	(lb/lb)	Entering SO <sub>2</sub> Concentration (ppm)	Leaving SO <sub>2</sub> Concentration (ppm)	SO <sub>2</sub> Removal Efficiency (%)
HL1	0.080	0.096	119.5	125.5	103.02	807.5	41.59	47.96	7.26	750	102	86.4
HL2	0.088	0.077	122.5	118.1	55.91	2903.5	21.81	328.79	47.73	216	40	94.9
HL3	0.085	0.077	121.6	118.1	55.50	704.4	21.75	80.00	11.70	775	51	93.4
HL4	0.087	0.089	122.1	123.0	102.36	2903.5	41.91	171.14	25.92	815	20	97.5
D1	0.089	0.094	123.0	124.5	101.11	807.5	42.44	47.00	7.33	860	580	32.6
D2	0.089	0.086	122.7	121.7	101.11	2903.5	42.44	169.00	26.37	860	420	51.2
D3	0.085	0.081	121.2	120.0	54.79	704.4	22.03	79.00	11.85	880	425	51.7
D4	0.085	0.094	121.2	124.6	54.79	2903.5	22.03	325.63	48.84	830	400	51.8
PC1	0.085	0.094	121.5	124.5	101.55	807.48	42.44	47.00	7.33	860	280	67.4
PC2	0.100	0.092	126.5	124.0	55.42	2903.5	21.90	327.52	48.47	825	120	85.5
PC3	0.083	0.087	120.4	122.3	54.96	704.4	21.92	79.38	11.83	830	150	81.9
PC4	-	-	-	-	-	V O I D	-	-	-	-	-	-
X1	0.088	0.097	122.5	125.5	78.56	1689.2	31.62	130.20	19.76	860	258	70.0

Table D-5. DATA CONSOLIDATION -- SERIES P-XX -- PADUCAH-SHAWNEE NO. 9 LIMESTONE

Test Number	GAS HUMIDITY (lb/lb dry air)		DEW POINT (°F)		DRY GAS FLOW RATE (lb/min)	LIQUID FLOW RATE (lb/min)	GAS VELOCITY (ft/sec)	LIQUID/GAS RATIO		ABSORPTION PARAMETERS		
										Entering SO <sub>2</sub> Concentration	Leaving SO <sub>2</sub> Concentration	SO <sub>2</sub> Removal Efficiency
	IN	OUT	IN	OUT	(lb/min)	(lb/min)	(ft/sec)	(gal/1000ft <sup>3</sup> )	(lb/lb)	(ppm)	(ppm)	(%)
P1	0.071	0.054	115.9	107.0	31.78	281.0	26.02	60.55	8.26	2450	1175	52.0
P2	0.074	0.049	117.0	104.5	30.256	281.0	24.83	63.46	8.65	2350	1080	54.0
P3	0.072	0.051	116.0	105.0	30.17	800.4	24.59	182.52	24.75	2350	700	70.2
P4	0.048	0.040	103.0	97.5	31.30	698.2	24.11	162.38	21.29	1900	310	83.7
P5	0.059	0.065	110.0	113.0	45.08	698.2	36.76	106.49	14.63	1995	765	61.7
P6	0.051	0.063	105.1	112.0	45.42	698.2	37.62	104.06	14.63	1500	490	67.3
P7	0.061	0.060	111.0	110.5	44.78	289.5	37.25	43.59	6.09	1500	980	34.7
P8	0.077	0.066	118.3	113.1	43.97	289.5	37.58	43.20	6.11	1750	1090	37.7
P9	0.059	0.033	110.0	92.0	30.17	289.5	24.64	65.89	9.06	940	440	53.2
P10	0.061	0.053	110.8	106.3	30.12	289.5	24.64	65.89	9.06	940	505	46.3
P11	0.054	0.059	107.0	110.0	30.46	698.2	24.02	163.02	21.75	2200	575	73.9
P12	0.054	0.059	107.0	110.0	30.46	698.2	24.02	163.02	21.75	2200	660	70.0
P13	0.060	0.066	110.5	113.5	44.82	289.5	37.58	43.20	6.09	2230	1420	36.3
P14	0.051	0.058	105.5	109.5	45.15	289.5	37.24	43.59	6.08	1450	840	42.1
P15	0.051	0.068	105.5	114.0	45.22	698.2	37.24	105.13	14.65	1470	550	62.6
P16	0.051	0.068	105.5	114.0	45.22	698.2	37.24	105.13	14.65	1470	475	67.7
P17	-	-	-	-	-	V O I D	-	-	-	-	-	-
P18	-	-	-	-	-	V O I D	-	-	-	-	-	-
P19	0.059	0.068	110.0	114.0	37.66	604.6	31.13	108.90	15.16	1900	730	61.6

Table D-5 (cont). DATA CONSOLIDATION -- SERIES P-XX -- PADUCAH-SHAWNEE NO. 9 LIMESTONE

Test Number	GAS HUMIDITY (lb/lb dry air)		DEW POINT (°F)		DRY GAS FLOW RATE (lb/min)	LIQUID FLOW RATE (lb/min)	GAS VELOCITY (ft/sec)	LIQUID/GAS RATIO		ABSORPTION PARAMETERS		
	IN	OUT	IN	OUT				(gal/1000ft <sup>3</sup> )	(lb/lb)	Entering SO <sub>2</sub> Concentration (ppm)	Leaving SO <sub>2</sub> Concentration (ppm)	SO <sub>2</sub> Removal Efficiency (%)
P20	0.061	0.068	111.0	114.0	37.59	604.6	31.27	108.40	15.16	2050	800	61.0
P21	0.060	0.068	110.5	114.0	37.56	604.6	31.42	107.90	15.19	2000	750	62.5
P22	0.053	0.058	106.6	109.3	38.02	604.6	30.94	109.57	15.10	2025	700	65.4
P23	0.056	0.056	108.1	107.9	37.92	604.6	31.42	107.90	15.10	1875	700	62.7
P24	0.060	0.056	108.0	107.9	37.77	604.6	31.61	107.25	15.10	2025	775	64.2
P25	0.055	0.061	107.0	110.7	37.84	604.6	31.61	107.25	15.10	1650	525	68.2
P26	0.054	0.061	107.0	110.7	37.99	604.6	31.61	107.25	15.10	1850	715	61.4
P27	0.059	0.065	109.7	113.0	37.95	604.6	31.27	108.40	15.04	1650	500	69.7
P28	0.059	0.065	109.7	113.0	37.95	604.6	31.27	108.40	15.04	1700	550	67.6
P29	0.054	0.063	107.0	111.7	37.66	604.6	30.84	109.91	15.04	1850	775	58.1
P30	0.059	0.068	110.0	114.4	37.59	604.6	30.80	110.08	15.19	1730	560	67.6
P31	0.062	0.065	111.5	113.0	37.48	604.6	30.80	110.08	15.19	1700	590	65.3
P32	0.069	0.072	114.5	116.0	37.60	604.6	31.23	108.56	15.01	2100	760	63.8
P33	0.077	0.074	118.1	117.0	44.33	289.5	37.91	42.82	6.06	2300	1125	51.1
P34	0.076	0.072	117.7	116.0	43.30	698.2	38.10	102.76	14.65	2420	975	59.7
P35	0.069	0.072	114.6	116.3	37.74	596.3	31.27	108.40	14.78	2620	1040	60.3



Table D-6. DATA CONSOLIDATION -- SERIES PA-XX, AND PS-XX -- PADUCAH-SHAWNEE

## NO. 9 ARAGONITE AND SIMULATED KEY WEST LIMESTONE

Test Number	GAS HUMIDITY (lb/lb dry air)		DEW POINT (°F)		DRY GAS FLOW RATE (lb/min)	LIQUID FLOW RATE (lb/min)	GAS VELOCITY (ft/sec)	LIQUID/GAS RATIO		ABSORPTION PARAMETERS		
	IN	OUT	IN	OUT				(gal/1000ft <sup>3</sup> )	(lb/lb)	Entering SO <sub>2</sub> Concentration (ppm)	Leaving SO <sub>2</sub> Concentration (ppm)	SO <sub>2</sub> Removal Efficiency (%)
PA1	0.060	0.081	110.5	120.0	45.04	289.5	37.05	43.81	6.06	2050	780	62.0
PA2	0.060	0.078	110.5	118.7	45.04	289.5	37.05	43.81	6.06	2050	700	65.9
PA3	0.056	0.051	108.0	105.0	28.15	698.2	21.25	184.27	23.49	1600	160	90.0
PA4	0.056	0.049	108.0	103.7	28.15	698.2	21.25	184.27	23.49	1200	84	93.0
PA5	0.087	0.064	122.2	112.5	36.84	698.2	31.75	123.31	17.44	2500	820	67.2
PA6	0.086	0.069	122.0	115.0	36.87	698.2	31.89	122.75	17.44	2700	860	68.1
PA7	0.088	0.064	122.3	112.0	36.94	698.2	30.56	128.13	17.37	2350	600	74.5
PA8	0.086	0.064	121.7	112.3	37.01	698.2	30.84	126.93	17.37	2100	590	71.9
PS1	0.058	0.063	109.0	112.0	38.21	604.6	30.37	111.64	14.96	2000	450	77.5
PS2	0.058	0.068	109.0	114.2	38.21	604.6	30.37	111.64	14.96	2020	525	74.0
PS3	0.059	0.057	110.0	108.3	38.17	604.6	30.37	111.64	14.96	2020	525	74.0

Table D-7. DATA CONSOLIDATION --SERIES IPA-XX AND IP-XX-- PADUCAH-SHAWNEE

## NO. 10 ARAGONITE AND LIMESTONE INJECTION

Test Number	GAS HUMIDITY (lb/lb dry air)		DEW POINT (°F)		DRY GAS FLOW RATE (lb/min)	LIQUID FLOW RATE (lb/min)	GAS VELOCITY (ft/sec)	LIQUID/GAS RATIO		ABSORPTION PARAMETERS		
	IN	OUT	IN	OUT				(gal/1000ft <sup>3</sup> )	(lb/lb)	Entering SO <sub>2</sub> Concentration (ppm)	Leaving SO <sub>2</sub> Concentration (ppm)	SO <sub>2</sub> Removal Efficiency (%)
IPA1	0.052	0.061	106.0	111.0	45.01	289.5	39.25	41.36	6.11	750	180	76.0
IPA2	0.055	0.067	108.0	113.8	45.40	698.2	37.62	104.06	14.58	415	31.5	92.4
IPA3	0.053	0.064	106.5	112.3	30.64	698.2	25.73	152.13	21.64	415	78	81.2
IPA4	0.049	0.052	104.0	105.5	30.90	698.2	24.92	157.09	21.54	510	30	94.1
IP1	0.051	0.054	105.0	106.8	37.80	698.2	31.18	125.57	17.57	420	37.5	91.1
IP2	0.063	0.051	111.7	105.3	38.03	698.2	30.37	128.93	17.27	700	46.5	93.4
IP3	0.061	0.51	111.0	105.0	37.96	698.2	30.56	128.13	17.34	975	262	73.1
IP4	0.051	0.059	105.3	109.5	37.58	698.2	31.42	124.62	17.68	1080	285	73.6
IP5	0.053	0.057	106.2	108.7	38.10	698.2	31.18	125.57	17.37	420	40	90.5
IP6	0.052	0.055	106.0	107.8	38.50	698.2	30.51	128.33	17.24	495	75	84.8
IP7	0.052	0.051	105.7	105.5	37.48	698.2	31.23	125.38	17.71	250	30	88.0
IP8	0.075	0.089	117.5	122.7	36.75	698.2	33.42	117.14	17.68	2000	600	70.0
IP9	0.063	0.068	111.9	114.3	44.77	800.4	38.05	117.94	16.82	2625	1050	60.0
IP10	0.063	0.068	111.9	114.3	44.77	800.4	38.05	117.94	16.82	2625	1050	60.0
IP11	0.085	0.070	121.3	115.4	43.57	800.4	38.58	116.34	16.93	2450	460	81.2
IP12	0.087	0.071	122.2	115.5	43.71	289.5	38.48	42.18	6.09	1520	540	64.5
IP13	0.085	0.071	121.3	115.5	26.12	272.5	22.30	68.52	9.62	1250	350	72.0

## APPENDIX E

### UNITS OF MEASURE

It is EPA policy to express all measurements in metric units. If undue costs or difficulty in clarity result from implementing this practice, British units may be employed and a conversion table provided. Such is the case with this document, the first draft of which was submitted before promulgation of the EPA policy.

	<u>British</u>	<u>Metric</u>
Length	1 inch (in.)	2.54 centimeters
	1 foot (ft)	0.3048 meters
Power	1 horsepower (HP)	746 watts
Volume	1 cubic foot (cf) (ft <sup>3</sup> )	28.3161 liters
	1 gallon (gal)	3.7853 liters
Pressure	1 inch of water (in.H <sub>2</sub> O)	2419 dynes per square centimeter
	1 inch of mercury (in.Hg)	338639 " "
	1 pound per square inch (psi)	689476 " "
Density	1 pound per cubic foot (lb ft <sup>-3</sup> )	0.0160 grams per cubic centimeter
Mass	1 ounce (oz)	28.3495 grams
	1 pound (lb)	453.5923 grams
	1 ton (T)	907.1846 kilograms
	1 grain (gr)	0.0648 grams
Temperature	1° Fahrenheit (°F)	(5/9)(°F-32) °Centigrade
	1° Rankine (°R)	(5/9)(°R) °Kelvin

# **TECHNICAL REPORT DATA**

*(Please read instructions on the reverse before completing)*

<b>1. REPORT NO.</b> A-650/2-74-077		<b>2.</b>	<b>3. RECIPIENT'S ACCESSION NO.</b>
<b>4. TITLE AND SUBTITLE</b> Limestone Scrubbing in a Pilot Dustractor-- Key West		<b>5. REPORT DATE</b> September 1974	
		<b>6. PERFORMING ORGANIZATION CODE</b>	
<b>7. AUTHOR(S)</b> Burke A. Bell; Terrence A. LiPuma; J. M. Craig, Ph.D.; and J. K. Allison		<b>8. PERFORMING ORGANIZATION REPORT NO.</b>	
<b>9. PERFORMING ORGANIZATION NAME AND ADDRESS</b> Engineering Science, Inc. 13 Westpark Drive Leans, Virginia 22101		<b>10. PROGRAM ELEMENT NO.</b> LAB013; ROAP 21AQO-001	
		<b>11. CONTRACT/GRANT NO.</b> CPA 70-61	
<b>12. SPONSORING AGENCY NAME AND ADDRESS</b> A, Office of Research and Development RC-RTP, Control Systems Laboratory Research Triangle Park, NC 27711		<b>13. TYPE OF REPORT AND PERIOD COVERED</b> Final; January-July 1971	
		<b>14. SPONSORING AGENCY CODE</b>	

**15. SUPPLEMENTARY NOTES**

**ABSTRACT** The report gives results of a 7-month series of nearly 200 tests of the Dustractor limestone wet scrubbing system in 1971, both in Key West, Florida, and at Shawnee's Shawnee Plant in Kentucky. At Key West, No. 6 fuel oil containing 1-2.2% sulfur was burned; at Shawnee, 2-4% sulfur pulverized coal was burned. The tests included systematic variation of stoichiometry, reactant particle size, slurry concentration, pressure drop, and gas flow rate. Reactants tested included coral marl, Fredonia limestone, dolomite, lime, aragonite, and precipitated calcium carbonate. Tests also included evaluation of spent reactant material, boiler injection of dry aragonite, addition of an inhibitor and catalyst, and effects on particulate and NOx removal. After installing an annular fresh water spray ring to reduce scale formation, the dustractor worked satisfactorily. SO2 removal efficiencies varied up to 90-plus %, depending on the reactant used. Absorption efficiency increased significantly with increased pressure drop, decreased gas flow rate, increased stoichiometric ratio, increased liquid-to-gas ratio. Other variables and an inhibitor and catalyst had no significant effect. NOx removal in the scrubber was negligible. Particulate removal in the total pilot plant system was excellent.

## **KEY WORDS AND DOCUMENT ANALYSIS**

<b>a. DESCRIPTORS</b>			<b>b. IDENTIFIERS/OPEN ENDED TERMS</b>	<b>c. COSATI Field/Group</b>
Pollution	Limestone	Desulfurization	Air Pollution Control	13B , 07D
Scrubbers	Marls	Nitrogen	Stationary Sources	07A
Shawnee	Dolomite	Oxides	Dustractor	13H
1 Oil	Aragonite		Particulates	21D, 11H
1	Calcium			07B
Calcium Oxides	Carbonates			
<b>16. DISTRIBUTION STATEMENT</b> Limited			<b>19. SECURITY CLASS (This Report)</b> Unclassified	<b>21. NO. OF PAGES</b> 157
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