

# AMAX Chemical Corporation

A SUBSIDIARY OF AMAX INC.

402 SOUTH KENTUCKY AVENUE • LAKELAND, FLORIDA 33801 • (813) 687-2561

May 11, 1984

DER

MAY 17 1984

BAQM

Mr. Willard Hanks  
Florida Department of  
Environmental Regulation  
Central Air Permitting Section  
2600 Blair Stone Road  
Tallahassee, FL 32301

Dear Mr. Hanks:

Attached is the air point source test results for the AMAX Big Four Mine Dryer COWM (Coal-Oil-Water-Mixture) test results. These tests were run for AMAX on February 16 and 17 by Sholtes and Koogler Environmental Consultants.

The test results indicate that the dryer was well within the allowable standards established by your staff.

If you have any questions about this point source test, please let me know.

Sincerely,



Fred G. Mullins, III  
Regulatory Compliance Manager

FGM/ds  
Attachment

cc: G.P. Uebelhoer  
R.F. Crabill

HILLSBOROUGH COUNTY  
ENVIRONMENTAL PROTECTION COMMISSION

CONVERSATION RECORD

Date May 2, 1984

Subject Relief from EPC's 20% Rule

Time \_\_\_\_\_

Permit No. \_\_\_\_\_

M Fred Mullins

Department \_\_\_\_\_

Representing AMAX

Telephone No. 687-2561

Telephoned Me       Was Called       Scheduled Meeting       Unscheduled Meeting

Other Individuals Involved in Conversation/Meeting \_\_\_\_\_

Summary of Conversation/Meeting

Told Fred that he needs to submit a letter summarizing the information presented at the meeting this AM. Once this is accomplished we will sit down with the Commission & discuss the mechanism which would allow EPC the right to relieve AMAX from the 20% rule. I made it clear we would not recommend such relief until the EPC is satisfied that AMAX has done everything possible to reduce opacity. AMAX is going to request a BACT or LAER determination from CAPS. Discussed all this with Bill Thomas this PM and he is in agreement.

(continue on another sheet, if necessary)

Signature Jeremy Campbell  
Title \_\_\_\_\_

To: Files

FROM: Jerry Campbell

DATE: Mar 2, 1974

SUBJECT: FBRs #1 and #2 + Paragon Kiln / AMAX's  
Request for Relief from City's 20% Capacity Std

The following items were discussed at a meeting attended by the people listed on the attached sheet on this date:

1. The only other FBR in Florida is at Occidental Chemical with a crossflow scrubber for controls.
2. AMAX's 2 ambient stations have been set up for a 15u cut + have not recorded any #s approaching the proposed 24 hour and annual standards over 3 years.
3. Fred has discussed this with his lawyers who have contacted Sara + they feel our rule allows for a modification of the City 20%.
4. AMAX is withdrawing their request for a variance, instead they will attempt to accomplish the same end through the lawyers.
5. A compliance schedule was discussed to keep AMAX out of enforcement.
6. AMAX offered to request a BACT or LAER determination from CAPS.

462

07503

**AMAX** Chemical Corporation

A SUBSIDIARY OF AMAX INC.

402 SOUTH KENTUCKY AVENUE • LAKELAND, FLORIDA 33801 • (813) 687-2561

April 3, 1984

752-1161

Mr. Anthony Jones  
Air Engineering Department  
Hillsborough County  
Environmental Protection Commission  
1900 Ninth Avenue  
Tampa, FL 33605

Dear Mr. Jones:

In accordance with the requirements of operating permit No. A029-66058, a compliance test was conducted at the No. 2 Feed Preparation wet scrubber, located at the Plant City Facility, on March 27, 1984. We are transmitting the results on the attached report.

Should you have any questions, please do not hesitate to contact me.

Sincerely,

*Robert L. Harrison J.*

Robert L. Harrison  
Central Monitoring Supervisor

RH/ds  
Attachment

- cc: Bob Garrett, DER
- N. W. Lowrey
- F. G. Mullins
- G. Townsend
- J. J. Lewis (w/o attachment)

**RECEIVED**  
APR 9 1984  
H.C.E.P.A.

4h2 (AMAX)

COUNTY



OF HILLSBOROUGH

TO

MEMORANDUM

JUL 11 1983

E.C.L.P.A.

Date July 6, 1983

To ~~Bill Thomas~~

From Jerry Campbell JC

Subject: NEDS Number for Amax R&D FBR Scrubber

The construction permit AC29-65451 issued for the above source lists the NEDS number as 231. Other sources at this same Plant City facility have a NEDS number of 75. Please explain why this difference exists or if this was simply an oversight just advise us of the correct number.

Thank you for your cooperation.

ck

When this permit was issued, it was to be operated by a separate agency - other than AMAX - hence it was given a separate plant no. to differentiate it from the AFI plant complex at Plant City.

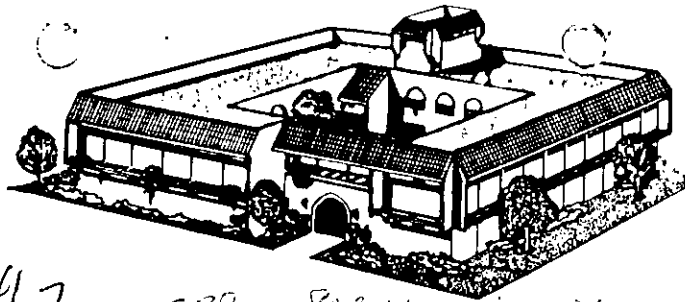
Bob.

JUL 21 1983

COMMUNITY DISTRICT  
PLANT CITY

HILLSBOROUGH COUNTY  
ENVIRONMENTAL PROTECTION  
COMMISSION

E. L. BING  
RODNEY COLSON  
MATT JETTON  
JOHN R. PAULK  
JAN KAMINIS PLATT



ROGER P. STEWART  
DIRECTOR

1900 - 8th AVE  
TAMPA, FLORIDA 33605

TELEPHONE (813) 272-5960

fhz FBR - PARAGON KILLS

September 16, 1983

Mr. Dan Williams  
Air Engineer  
Florida Department of Environmental Regulation  
Southwest District  
7601 Highway 301 North  
Tampa, Florida 33610

Re: AMAX Chemical Corporation

Dear Mr. Williams:

This is in response to AMAX Chemical Corporation's request to have Florida Department of Environmental Regulation establish a 40% opacity limit for the two fluid bed reactors at their Coronet Junction defluorination plant. While Chapter 17-2.610, FAC, provides the mechanism by which the Department may establish such a standard, doing so in this case would create a permit condition which conflicts with the Rules of the Hillsborough County Environmental Protection Commission.

Our rules are specific with respect to visible emissions. Please refer to Chapter 1-3.031. This section states that no person shall emit pollutants in excess of 20% opacity. The general particulate emission limiting standard varies proportionally with the amount of material processed. Nothing in the VE rule can be construed as allowing the particulate mass emission rate limit to supercede the opacity limit. The two are measurements of different emission phenomena. Particulate limits are gravimetric efficiency measurements based on process weight rate. The VE is a volumetric estimation of the small particle content of the gas stream. Due to the relatively low mass of these small particles, large increases in their concentration may not substantially increase total particulate mass. However, it is these small particles that have the greater impact on public health, and visibility.

Granted, relationships between particulate concentrations and visible emissions can be established, but few conclusions can be made based on results from a single Method 5 test. More needs to be known about size distribution over a variety of operating conditions and product matrices.

Mr. Dan Williams  
September 16, 1983  
Page 2

The character and composition of the particles need to be determined. Many process and control equipment parameters must be measured under varied conditions too. Even then AMAX must show that compliance with our rule creates some insurmountable hardship. Under these circumstances the Director may request that the Commission establish an alternative emission plan for AMAX. Only then would an increase in the visible emission limit for this source be possible.

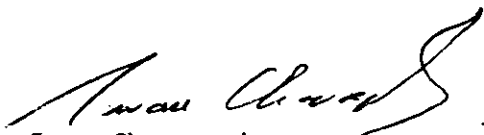
Perhaps it is important to note that AMAX recently requested, and was granted, an increase in the process rates and allowable particulate emissions for these two units. Until this increase became effective AMAX apparently had no difficulty in meeting the opacity limit. In any case no such problem was reported to our office. It appears, on the surface, that what we have is a control device that is unable to handle the increased load from the reactors. If this is the problem, AMAX's approach should be to upgrade the control equipment not to raise the emission limit. Maybe to comply with our VE rule AMAX may have to return to the lesser process rate.

We cannot support AMAX's request to circumvent our VE rule. In Hillsborough County the VE is not a surrogate standard. It is a separate, independently enforceable regulation. It is equally as important as the particulate and other emission limits, and ranks first among the prohibited acts listed in our rules. Since ours is a more stringent regulation than 17-2.610, the Department is compelled by the provisions of Chapter 403.186(6), F.S., to enforce this stricter rule.

We recently issued two Notices of Alleged Violation to AMAX requiring that they cease and desist the excess visible emissions. The Notices will be rescinded but Hillsborough County Environmental Protection Commission will initiate administrative enforcement should they continue to exceed the limits of Chapter 1-3.03.

Hopefully, this letter clarifies our position on this and any similar request that your office may receive concerning other sources located in Hillsborough County. If you have any questions or require further comments, please contact Anthony Jones of the Air Compliance Section.

Sincerely,



Iwan Choronenko  
Air Pollution Control Programs Manager  
Hillsborough County Environmental  
Protection Commission

IC/lw

cc: Fred G. Mullins, AMAX Chemical Corp.

# LEGAL BRIEFS

by H. Newcomb Morse, J.D., LL.M., F.A.A.F.S.

**VISUAL OPACITY STANDARDS:** The Chemithon Corporation appealed a fine for violating a regional air pollution regulation.

The Puget Sound Air Pollution Control Agency (PSAPCA) is a regional air pollution control agency which adopted procedural and substantive rules, under the heading Regulation I, to implement its authority. Among the Regulation I rules is § 9.03, which pertains to visual opacity standards and states in part:

(b) After July 1, 1975, it shall be unlawful for any person to use or allow the emission of any air contaminant . . . which is:

(1) Darker in shade than that designated as No. 1 (20% density) . . .

(e) This section shall not apply when the presence of uncombined water is the only reason for failure of the emission to meet the requirements of this section.

Section 9.09, which pertains to particulate emission standards, provides in relevant part:

It shall be unlawful for any person to cause or allow the emission of particulate matter if the emission is in violation of Section 9.03 or if the particulate matter discharged into the atmosphere from any single source exceeds the following weights at the point of discharge: . . .

(Emphasis added.) The penalties for violating any of the standards are set forth in § 3.29 of Regulation I:

In addition to or as an alternate to or any other penalty provided for herein or by law, any person who violates any of the provisions of this regulation shall incur a penalty in the form of a fine in an amount not to exceed two hundred fifty dollars per day for each violation.

(Emphasis added.)

On September 17, 1976, a PSAPCA enforcement officer made a *visual opacity* observation of a plume rising from a stack of a Chemithon plant and discovered the plume had a density of 50 to 80 percent. As a result of this observation, a \$250 penalty was imposed on Chemithon for violating § 9.03. Chemithon appealed the penalty to the Pollution Control Hearings Board (Board), presenting evidence that the stack emission at the time of the alleged violation was within the *particulate* weight standards established by PSAPCA. The Board found that the particulate emissions were within the weight standards established by the PSAPCA, but affirmed the penalty notwithstanding such findings, reasoning that Chemithon violated a separate visual opacity standard. On review, the Superior Court of King County, Washington, rendered judgment holding the findings and conclusions of the Board were not clearly erroneous, nor arbitrary or capricious, and upheld the penalty.

Chemithon argued that it should not be fined for violation of the opacity standard, § 9.03, when the requirements as established by the particulate standard, § 9.09, are not also violated. According to Chemithon, the visual opacity test is a less reliable measure of pollution in a stack emission than actual particulate analysis of the emission. From this premise, Chemithon reasoned that the visual opacity test creates only a presumption of illegal pollution and is subject to rebuttal proof that actual emissions are within particulate standards; thus, superior particulate evidence should rebut opacity observations. Chemithon stated that Regulation I did not intend that a plant emitting dry (particulate) pollutants up to the amount allowed in § 9.09 complies with the Regulation, but a plant emitting the same pollutants mixed with water, a nonpollutant, is subject to a civil penalty because of the § 9.03 opacity standard, especially since § 9.03 is not a "cosmetic" regulation. Since PSAPCA requires Chemithon to demonstrate that all particulate emissions have been removed in order to be exempt from the visual opacity standards, Chemithon asserted it is being fined merely because it mixes harmless water vapor with an allowable amount of particulate pollution.

Chemithon's contention was rejected by the Court of Appeals of Washington, which affirmed the judgement of the Superior Court.

The Court of Appeals declared: "We are unpersuaded by Chemithon's argument and hold a visual opacity civil penalty legal even when the particulate standards are not violated. The language of § 3.29 makes it clear that a violation of any of the provisions of Regulation I will subject the violator to a civil penalty. Furthermore, the Pollution Control Hearings Board provided that a person shall violate § 9.09 if he violates the opacity standards in § 9.03, or if he exceeds the particulate standards in § 9.09. We believe that violations of § 9.03 may be determined independently of the particulate standard of § 9.09. It does not matter that water vapor might be the primary cause of the observed visual opacity. For the uncombined water exception of § 9.03 to apply, the effluent must be free of all particulate contaminants . . . We do not speculate here why the Board decided that lawful particulate effluents when mixed with nonpolluting water vapor create unlawful pollution. It is enough to say that there are possible rational bases for the Board's decision; for example, particulates uncombined with water may dissipate resulting in no harm, but when combined with water the particulates may quickly fall to earth and have a harmful effect."

Citation: *Chemithon Corporation v. Puget Sound Air Pollution Control Agency*, 31 Wash. App. 276, 640 P.2d 1085 (1982).



VISIBLE EMISSION OBSERVATION FORM

SOURCE NAME <b>AMAX CHEMICAL</b>			OBSERVATION DATE <b>5-3-84</b>				START TIME <b>11:33</b>		STOP TIME <b>11:45</b>	
ADDRESS			M		S		M		S	
CITY <b>PLANT CITY</b>			STATE		ZIP		1		2	
PHONE			SOURCE ID NUMBER <b>0075 24</b>		3		4		5	
PROCESS EQUIPMENT <b>FBR #2</b>			OPERATING MODE		6		7		8	
CONTROL EQUIPMENT <b>SCRUBBER + IWS</b>			OPERATING MODE		9		10		11	
DESCRIBE EMISSION POINT <b>STACK FOR FBR #2</b>			HEIGHT ABOVE GROUND LEVEL <b>155'</b>		HEIGHT RELATIVE TO OBSERVER <b>150'</b>		12		13	
DISTANCE FROM OBSERVER <b>150'</b>			DIRECTION FROM OBSERVER <b>W</b>		14		15		16	
DESCRIBE EMISSIONS			EMISSION COLOR <b>WHITE</b>		PLUME TYPE: CONTINUOUS <input checked="" type="checkbox"/>		17		18	
WATER DROPLETS PRESENT <b>NO</b>			IS WATER DROPLET PLUME ATTACHED <input type="checkbox"/> DETACHED <input type="checkbox"/>		19		20		21	
AT WHAT POINT IN THE PLUME WAS OPACITY DETERMINED <b>2 STACK DIA. DOWNWIND</b>			BACKGROUND <b>SKY</b>		BACKGROUND COLOR <b>BLUE</b>		SKY CONDITIONS <b>30% OVERCAST</b>		22	
WIND SPEED <b>5-10 mph</b>			WIND DIRECTION <b>SSW</b>		23		24		25	
AMBIENT TEMPERATURE <b>85°F</b>			RELATIVE HUMIDITY		26		27		28	
SOURCE LAYOUT SKETCH			DRAW NORTH ARROW		29		30		31	
<p>The sketch shows a vertical line representing the emission point. A horizontal line below it represents the sun shadow line, with angles of 70° and 20° marked. A circle with an arrow pointing right represents the observer's position.</p>			AVERAGE OPACITY FOR HIGHEST PERIOD <b>31%</b>		NUMBER OF READINGS ABOVE <b>20 % WERE 47</b>		32		33	
			RANGE OF OPACITY READINGS <b>20% MINIMUM 35% MAXIMUM</b>		OBSERVER'S NAME (PRINT) <b>JERRY CAMPBELL</b>		OBSERVER'S SIGNATURE <i>Jerry Campbell</i>		DATE <b>5/3/84</b>	
COMMENTS <b>WE TAKEN DURING SECOND RUN OF COMPLIANCE TEST / OPERATING RATE UNKNOWN</b>			ORGANIZATION <b>HCEPC</b>		CERTIFIED BY <b>ETA</b>		DATE <b>3/84</b>		35	
I HAVE RECEIVED A COPY OF THESE OPACITY OBSERVATIONS			VERIFIED BY		DATE		36		37	
SIGNATURE			DATE		38		39		40	
TITLE			DATE		41		42		43	
			DATE		44		45		46	
			DATE		47		48		49	
			DATE		50		51		52	
			DATE		53		54		55	
			DATE		56		57		58	
			DATE		59		60		61	
			DATE		62		63		64	
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			DATE		68		69		70	
			DATE		71		72		73	
			DATE		74		75		76	
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			DATE		95		96		97	
			DATE		98		99		100	

Type III

HILLSBOROUGH COUNTY ENVIRONMENTAL PROTECTION COMMISSION

INSPECTION REPORT EXECUTIVE SUMMARY

PLANT NAME Amox Phosphate NEDS 075 DATE/TIME 3 May 84 11:00 AM

PLANT LOCATION Plant City, FL # OF NEDS POINTS 25

PROCESS DESCRIPTION Fluidized bed reactor #2, a packed bed scrubber and an electrostatic precipitator type scrubber, and a spray tower are used to defluorinate phosphate-based animal feed.

COMPLIANCE VERIFICATION ENFORCEMENT (X) PERMIT REVIEW ( ) OTHER ( )

PERSONS CONTACTED-TITLE George Townsend - Plant Engineer

NEDS POINTS CHECKED 24 NEDS POINTS IN COMPLIANCE NEDS POINTS IN VIOLATION 24

SUMMARY OF FINDINGS A Visible Emissions test (EPA Method 9) was performed to measure the opacity of the plume arising from the process stack. This stack is the common emission point for Fluidized Bed Reactor #2 and Paragon Defluorinating Kiln #2; however, only Fluidized Bed Reactor #2 was on-line at the time of the inspection. The visible emission (opacity) was approximately 35%. The process controls and operations logs for the reactor and precipitator were inspected. Stack testing was in progress and was monitored briefly. Technique and equipment appeared to be in order. Operating rate at the time of inspection was not available. George Townsend was directed to perform a VE concurrent with the stack test.

INSPECTION COMMENTS FOR APIS (LIMIT 50 SPACES) A 12 min. VE showed average 31% opacity

INSPECTOR'S SIGNATURE Steve Sprog

AIR POLLUTION EMISSIONS  
COMPLIANCE TEST

BIG FOUR MINE DRYER

AMAX CHEMICAL CORPORATION

FEBRUARY 1984

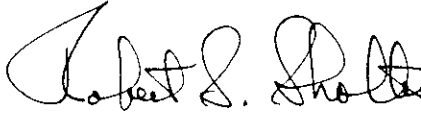
SHOLTES & KOOGLER  
ENVIRONMENTAL CONSULTANTS, INC.  
1213 N.W. 6TH STREET  
GAINESVILLE, FLORIDA 32601  
(904) 377-5822

TABLE OF CONTENTS

	<u>PAGE</u>
1.0 EXECUTIVE SUMMARY.....	1
2.0 INTRODUCTION.....	2
3.0 SAMPLING PROCEDURES.....	3
4.0 RESULTS.....	10

APPENDICES

To the best of my knowledge, all applicable field and analytical procedures comply with Florida Department of Environmental Regulation requirements and all test data and plant operating data are true and correct.



ROBERT S. SHOLTES, PH.D., P.E.

3-9-84

DATE

SEAL

## 1.0 EXECUTIVE SUMMARY

On February 16 and 17, 1984, Sholtes & Koogler, Environmental Consultants, Inc. (SKEC) of Gainesville, Florida performed emission tests on the Big Four Mine Dryer for the pollutants sulfur dioxide, nitrogen oxides, particulate matter and visible emissions. The measured emissions proved to be in compliance with all limitations specified in Permit AC29-65834 for this installation. The specific limits and measured emissions are summarized on Page 11 of this report.

## 2.0 INTRODUCTION

AMAX Chemical Corporation operates a 300 tph fluid-bed phosphate rock dryer and associated dry rock storage and handling equipment at their Big Four Mine located near the intersection of State Road 674 and Bethlehem Road, Fort Lonesome, Hillsborough County, Florida.

The company has recently been permitted (AC29-65834) for a coal-oil-water mix fuel (COWM) containing up to 2.5 percent sulfur by weight.

The moisture content of 300 tph wet phosphate rock from the beneficiation plant is reduced from approximately 14 percent to 2.5 percent in a Heyl Patterson fluid bed dryer. The pollution from the dryer is controlled by a Peabody emission control system consisting of a twin cyclone and a wet impingement scrubber with a demisting section. Sulfur dioxide, produced from burning fuel containing sulfur, is absorbed in the scrubber and the phosphate rock product.

Specific Condition 17 of the above referenced permit requires emission testing for particulate matter, sulfur dioxide, nitrogen oxides and opacity while firing with the two alternate fuels, No. 6 fuel oil and COWM. This report constitutes an evaluation of these emissions in fulfillment of this specific permit condition.

### 3.0 SAMPLING PROCEDURES

#### A. Sampling Location

Sampling for particulates required traversing of the dryer discharge in accordance with EPA Reference Methods 1 and 5. Figure 1 illustrates the geometry of this sampling location and specifies the specific traverse points used in these testing efforts. The other emission monitoring methods do not require traversing, however, sampling for these methods with the exception of opacity, were taken at one of the described sampling ports and at a location approximately one-third to one-half of the stack diameter away from the inner wall.

#### B. Particulate Matter

Particulate matter samples were collected by the particulate matter emission measurement method adopted by the FDER in October 1980. A schematic diagram of the sampling train used is shown in Figure 2. All particulate matter captured from the nozzle to, and including, the filter was included in the calculation of the emission rate of particulate matter.

#### Preparation of Equipment

1. Filters - Gelman type "A" filters were placed in a drying oven for two hours at 105 degrees C, removed and placed in a standard glass desiccator containing indicating silica gel, allowed to cool for two hours, and weighed to the nearest 0.1 mg.



2. Nozzle, Filter Holder and Sampling Probe - The nozzle, filter holder and sampling probe were washed vigorously with soapy water and brushes, rinsed with acetone and distilled water and dried prior to the test program. All openings on the sampling equipment were sealed while in transit to the test site.
3. Impingers - The Greenburg-Smith Impingers were cleaned with a warm soapy water solution and brushes, rinsed with distilled water and acetone and dried. The impingers were sealed tightly during transit.

#### Test Procedure

Prior to performing the actual particulate matter sample runs, certain stack and stack gas parameters were measured. These preliminary measurements included the average gas temperature, the stack gas velocity head, the stack gas moisture content, and the stack dimensions at the point where the tests were being performed. The stack gas temperature was determined by using an iron-constantan thermocouple with a digital readout. Velocity head measurements were made with calibrated type "S" pitot tube and an inclined manometer.

The sampling traverse points were selected so that a representative sample could be extracted from the gas stream. The traverse points were located in the center of equal areas, which were dependent upon the distance upstream and downstream from flow disturbances.

Each particulate matter test run consisted of sampling for a specific time at each traverse point. The type "S" pitot tube was connected to the sampling probe so that an instantaneous velocity head measurement could be made at each traverse point while making the test run. The stack gas temperature was also measured at each traverse point. Nomographs were used to calculate the isokinetic sampling rate at each traverse point during each test run.

The gases sampled passed through the following components: a stainless steel nozzle and probe; a glass fiber filter; two impingers with 100 ml of distilled water; one impinger dry; one impinger with 180 grams of silica gel; a flexible sample line; an air-tight pump; a dry test meter; and a calibrated orifice. The second impinger had a standard tip, while the first, third and fourth impingers had modified tips with a 0.5 inch I.D. opening.

Sample recovery was accomplished by the following procedures:

1. The pretared filter was removed from its holder and placed in Container 1 and sealed.
2. All sample-exposed surfaces prior to the filter were washed with acetone and placed in Container 2.
3. The volume of water from the first three impingers was measured for the purpose of calculating the moisture in the stack gas and then discarded.
4. The used silica gel from the fourth impinger was transferred to the original tared container and sealed.

### Laboratory Analysis

The three sample containers from each sample run were analyzed according to the following procedures:

1. The contents of Container 1 were transferred to a tared glass weighing dish, dried, and desiccated to a constant weight.
2. The acetone from Container 2 was transferred to a tared beaker and evaporated to dryness at 212 degrees F. It was then desiccated to a constant weight.
3. The used silica gel in its tared container was weighed to the nearest gram.

The total sample weight included the weight of material collected on the filter plus the weight of material collected in the nozzle, sampling probe, and front half of the filter holder.

### Data

The field data sheets, calculation sheets, and nomenclature definitions are included in the Appendix of this report.

C. Sulfur Dioxide Testing

As specified in the FDER permit, testing for sulfur dioxide was carried out in accordance with EPA Reference Method 6. The procedures used in this testing were those outlined in the Method, a copy of which is appended hereto, along with field data and computations.

D. Nitrogen Oxides Testing

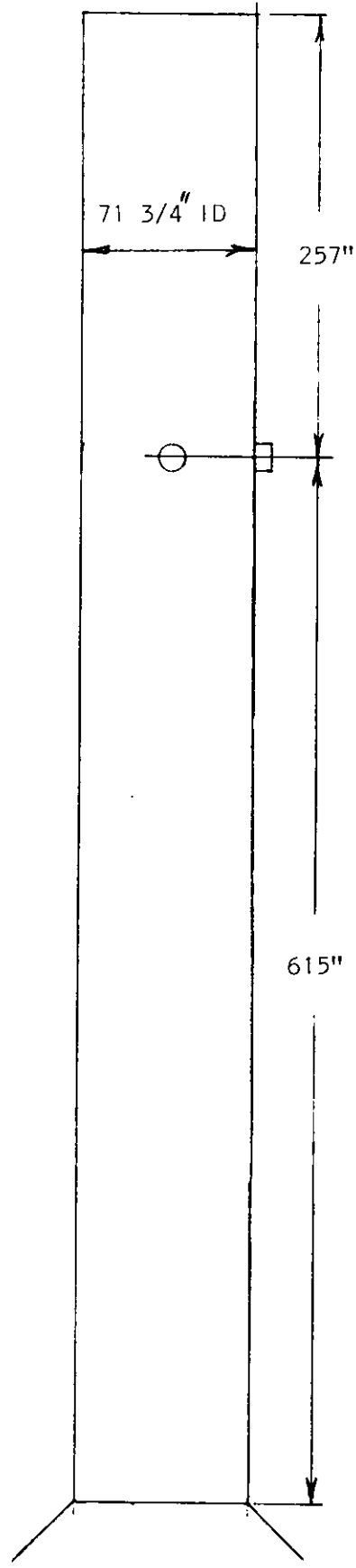
As specified in the FDER permit testing for nitrogen oxides was carried out in accordance with EPA Reference Method 7. The procedures used in this testing were those outlined in the Method, a copy of which is appended hereto, along with field data and computations.

E. Opacity Determinations

Opacity observations were made on the discharge while burning oil and COWM for periods of one-half hour in accordance with the provisions of Chapter 17-2, Florida Administrative Code. The observation period was selected on the basis that this source constituted a minor source with respect to particulate emissions.

FIGURE 1

AMAX CHEMICAL CORPORATION  
BIG FOUR MINE DRYER  
STACK GEOMETRY



<u>Traverse Point</u>	<u>Distance From Inside Wall (inches)</u>
1	3.2
2	10.5
3	21.2
4	50.6
5	61.2
6	68.6

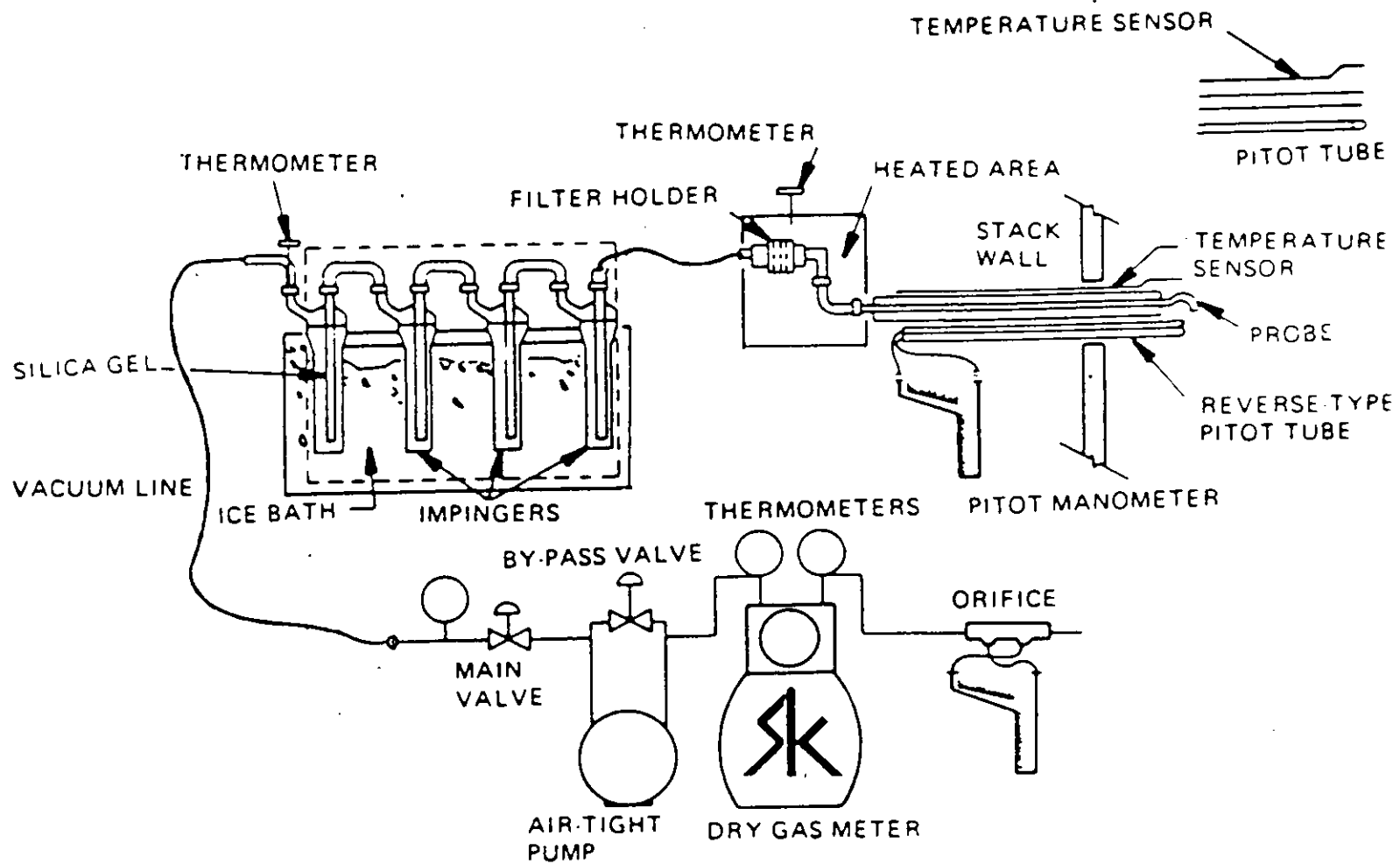


FIGURE 2

FDER PARTICULATE MATTER SAMPLING TRAIN

#### 4.0 RESULTS

Table I summarizes the results of these test series. It will be observed that for both fuel regimes, all specified emission limits were attained during the testing period.

From the point of view of testing, there are no known limitations upon these data and it is purported that they represent a fair and representative evaluation of the emissions involved.

TABLE I

EMISSIONS TESTS  
BIG FOUR MINE DRYER

AMAX CHEMICAL CORPORATION

Date	Test No.	Feed Rate (tph)	Fuel / Rate (gal/hr)	Heat Input * (10 <sup>6</sup> btu/hr)	Opacity (%)	Particulate Matter (lb/hr)	Particulate Matter (lb/ton)	Sulfur Dioxide (lb/hr)	Sulfur Dioxide (lb/10 <sup>6</sup> btu)	Nitric Oxides (lb/hr)	
02-16-84	1	270	COWM	600	81.7	0	11.6	0.04	3.76	0.046	
02-16-84	2	270	COWM	600	81.7		13.5	0.05	19.95	0.244	19.5
02-16-84	3	270	COWM	600	81.7		<u>13.4</u>	<u>0.05</u>	<u>38.39</u>	<u>0.470</u>	
Average							12.6	0.043	20.7	0.253	
02-16-84	1	277	Oil	600	77.7		12.5	0.05	64.02	0.824	
02-17-84	2	300	Oil	625	77.7	5	10.2	0.03	23.38	0.301	28.1
02-17-84	3	270	Oil	640	77.7		<u>10.4</u>	<u>0.04</u>	<u>63.69</u>	<u>0.820</u>	
Average							11.0	0.040	50.36	0.648	

Allowable Emissions:

Particulate Matter 0.06 lb/ton  
 Sulfur Dioxide 1.1 lb/10<sup>6</sup> btu  
 Nitrogen Oxide 35.5 lb/hr  
 Opacity 10 Percent

\*Heat Input Based on Duration of Method 6 (SO<sub>2</sub>) Tests.

?



## APPENDICES

- Appendix A - Field Data Sheets and Computations
  - A1 - Particulate Sampling/Method 5
  - A2 - Sulfur Dioxide Sampling/Method 6
  - A3 - Nitrogen Oxide Sampling/Method 7
  - A4 - Opacity/Method 9
- Appendix B - Plant Operating Data
- Appendix C - Calibrations and Project Participants

APPENDIX A1

Field Data Sheets and Computations  
Particulate Sampling/Method 5

EQUATIONS FOR CALCULATING PARTICULATE EMISSIONS

$$VWV = 0.0000893(TSTD)(VC)$$

$$VSTPD = (VM)(PB + \frac{\Delta H}{13.6}) \left( \frac{TSTD}{TM} \right) \left( \frac{1}{29.92} \right)$$

$$VT = (VWV) + (VSTPD)$$

$$W = (VWV) \div (VT)$$

$$FDA = (1.0) - (W)$$

FMOIST = Assumed moisture fraction

$$MD = (0.44 \times \% CO_2) + (0.32 \times \% O_2) + (0.28 \times \% N_2) + (0.28 \times \% CO)$$

$$MS = (MD \times FDA) + (18 \times W)$$

$$GS = (MS) \div (28.99)$$

$$EA = \left[ (100) \times (\% O_2 - \frac{\% CO}{2}) \right] \div \left[ (0.266 \times \% N_2) - (\% O_2 - \frac{\% CO}{2}) \right]$$

$$U = 4,006(CP) \sqrt{H} \sqrt{\left( \frac{TS}{TSTD} \right) \left( \frac{29.92}{PS} \right) \left( \frac{1}{GS} \right)}$$

$$QS = (U) \times (AS)$$

$$QD = (QS) \times (FDA)$$

$$QSTPD = TSTD(QD)(PS) \div [TS(29.92)]$$

$$PISO = \left[ (0.00267 \times VC \times TS) + (P_0 \times TS \times VM \div TM) \right] \div \left[ (Time \times U \times PS \times AN) \right]$$

$$ESTP = \frac{\left( \frac{15.43 \text{ grains}}{\text{gram}} \right) (y)}{VSTPD}$$

$$E_{12} = \frac{(ESTP) (12)}{(CO_2 \%)}$$

$$E_{50} = \frac{(ESTP) (100 + EA)}{150}$$

$$EM = (ESTP) (QSTPD) \left( 60 \frac{\text{min}}{\text{hr}} \right) \left( \frac{1 \text{ lb}}{7000 \text{ grains}} \right)$$

## SOURCE SAMPLING NOMENCLATURE SHEET

- PB - Barometric pressure, inches Hg  
 PS - Stack pressure, inches Hg  
 AS - Stack area, sq. ft.  
 AS' - Effective area of positive stack gas flow, sq. ft.  
 NPTS - Number of traverse points where the pitot velocity head was greater than zero  
 TSTD - Standard temperature, °R  
 TS - Stack temperature, °R  
 TM - Meter temperature, °R  
 H - Average square root of velocity head,  $\sqrt{\text{inches H}_2\text{O}}$   
 $\bar{H}$  - Average meter orifice pressure differential, inches H<sub>2</sub>O  
 AN - Sampling nozzle area, square feet  
 CP - S-type pitot tube correction factor  
 VM - Recorded meter volume sample, cubic feet (meter conditions)  
 VC - Condensate and silica gel increase in impingers, milliliters  
 Po - Pressure at the dry test meter orifice,  $\left[ \text{PB} + \frac{\Delta H}{13.6} \right]$  inches Hg  
 STP - Standard conditions
- 
- VWV - Conversion of condensate in milliliters to water vapor in cubic feet (STP)  
 VSTPD - Volume sampled, cubic feet (STP)  
 VT - Total water vapor volume and dry gas volume sampled, cubic feet (STP)  
 W - Moisture fraction of stack gas  
 FDA - Dry gas fraction  
 MD - Molecular weight of stack gas, lbs/lb-mole (dry conditions)  
 MS - Molecular weight of stack gas, lbs/lb-mole (stack conditions)  
 GS - Specific gravity of stack gas, referred to air  
 EA - Excess air, %  
 $\sqrt{H \times TS}$  - Average square root of velocity head times stack temperature  
 U - Stack gas velocity, feet per minute  
 QS - Stack gas flow rate, cubic feet per minute (stack conditions)  
 QD - Stack gas flow rate, cubic feet per minute (dry conditions)  
 QSTD - Stack gas flow rate, cubic feet per minute (STP)  
 PISO - Percent isokinetic volume sampled (method described in Federal Register)  
 ESTP - Particulate concentration at standard and dry conditions, grains/scf  
 E<sub>12</sub> - ESTP corrected to 12% CO<sub>2</sub>, grains/scf  
 E<sub>50</sub> - ESTP corrected to 50% excess Air, grains/scf  
 EM - Mass Emission Rate, lbs/hr  
 \* - Stack Gas Saturated

Table 1

SUMMARY OF PARTICULATE MATTER EMISSIONS

AMAX  
BIG 4 DRYER COAL/OIL  
2/16/84

Run No.	Process Weight Rate (Tons/Hr)	Stack Gas Flow Rate (SCFMD)	Stack Gas Temperature (Deg F)	Stack Gas Moisture (%)	Particulate Matter	
					Conc. (gr/SCF)	Emission Rate (Lbs/Hr)
1	270.0	52612	131.5	14.1	.0258	11.64
2	270.0	52099	134.2	14.3	.0302	13.50
3	270.0	51949	131.2	14.0	.0300	13.40
Avg	270.0	52220	132.3	14.1	.0287	12.85

Allowable Particulate Matter Emission Rate =  
(Chap. 17-2, Florida Administrative Code)

Lbs/Hr

SHOLTES AND KOOGLER ENVIRONMENTAL CONSULTANTS

Source Sampling Calculations

Plant: AMAX  
 Stack: BIG 4 DRYER COAL/OIL  
 Weather: PARTLY CLOUDY

Date: 2/16/84  
 Run 1 From 1106 - 1210  
 Total Time: 60 Min

Stack Area	28.08 Sq Ft	Nozzle Area	.000539 Sq Ft
Stack Temp	131.5 Deg F	Meter Temp	87.3 Deg F
Stack Pressure	30.12 "Hg	Baro. Pressure	30.12 "Hg
Stack Vel Head	.666 "H2O	Meter Press Diff	3.45 "H2O
		Meter Volume	58.372 cf
Pitot Tube Factor	.84	Condensate Volume	198.7 ml

- |   |             |
|---|-------------|
| 1. Volume Water Vapor                               | 9.359 SCF   |
| 2. Gas Volume Sampled - STPD                        | 57.167 SCFD |
| 3. Total Volume                                     | 66.526 SCF  |
| 4. Moisture in Stack Gas - Volume Fraction          | .141        |
| 5. Dry Stack Gas - Volume Fraction                  | .859        |
| 6. Molecular Weight of Stack Gas - Dry Basis        | 29.00       |
| 7. Molecular Weight of Stack Gas - Stack Conditions | 27.45       |
| 8. Specific Gravity of Stack Gas Relative to Air    | .95         |
| 9. Excess Air - Percent                             |             |
| 10. Average Stack Velocity                          | 2426.4 FPM  |
| 11. Average Stack Gas Flow Rate                     | 68133 ACFM  |
| 12. Actual Stack Gas Flow Rate Dry                  | 58548 CFMD  |
| 13. Stack Gas Flow Rate STPD                        | 52612 SCFMD |
| 14. Percent Isokinetic                              | 94.3 %      |

Probe Wash:	8.70 Mg	.0023 Gr/SCF	1.06 Lbs/Hr
Filter:	86.90 Mg	.0234 Gr/SCF	10.58 Lbs/Hr
===Totals===	95.60 Mg	.0258 Gr/SCF	11.64 Lbs/Hr

SHOLTES AND KOOGLER ENVIRONMENTAL CONSULTANTS

Source Sampling Calculations

Plant: AMAX  
 Stack: BIG 4 DRYER COAL/OIL  
 Weather: PARTLY CLOUDY

Date: 2/16/84  
 Run 2 From 1237 - 1342  
 Total Time: 60 Min

Stack Area	28.08 Sq Ft	Nozzle Area	.000539 Sq Ft
Stack Temp	134.2 Deg F	Meter Temp	94.2 Deg F
Stack Pressure	30.12 "Hg	Baro. Pressure	30.12 "Hg
Stack Vel Head	.663 "H2O	Meter Press Diff	3.48 "H2O
		Meter Volume	59.900 cf
Pitot Tube Factor	.84	Condensate Volume	204.6 ml

- |   |             |
|---|-------------|
| 1. Volume Water Vapor                               | 9.637 SCF   |
| 2. Gas Volume Sampled - STPD                        | 57.937 SCFD |
| 3. Total Volume                                     | 67.574 SCF  |
| 4. Moisture in Stack Gas - Volume Fraction          | .143        |
| 5. Dry Stack Gas - Volume Fraction                  | .857        |
| 6. Molecular Weight of Stack Gas - Dry Basis        | 29.00       |
| 7. Molecular Weight of Stack Gas - Stack Conditions | 27.43       |
| 8. Specific Gravity of Stack Gas Relative to Air    | .95         |
| 9. Excess Air - Percent                             |             |
| 10. Average Stack Velocity                          | 2419.1 FPM  |
| 11. Average Stack Gas Flow Rate                     | 67929 ACFM  |
| 12. Actual Stack Gas Flow Rate Dry                  | 58241 CFMD  |
| 13. Stack Gas Flow Rate STPD                        | 52099 SCFMD |
| 14. Percent Isokinetic                              | 96.5 %      |

Probe Wash:	9.60 Mg	.0026 Gr/SCF	1.14 Lbs/Hr
Filter:	103.90 Mg	.0276 Gr/SCF	12.36 Lbs/Hr
===Totals===	113.50 Mg	.0302 Gr/SCF	13.50 Lbs/Hr

SHOLTES AND KOOGLER ENVIRONMENTAL CONSULTANTS

Source Sampling Calculations

Plant: AMAX  
 Stack: BIG 4 DRYER COAL/OIL  
 Weather: PARTLY CLOUDY

Date: 2/16/84  
 Run 3 From 1405 - 1508  
 Total Time: 60 Min

Stack Area	28.08 Sq Ft	Nozzle Area	.000539 Sq Ft
Stack Temp	131.2 Deg F	Meter Temp	92.9 Deg F
Stack Pressure	30.12 "Hg	Baro. Pressure	30.12 "Hg
Stack Vel Head	.657 "H2O	Meter Press Diff	3.43 "H2O
		Meter Volume	59.988 cf
Pitot Tube Factor	.84	Condensate Volume	200.8 ml

- |   |             |
|---|-------------|
| 1. Volume Water Vapor                               | 9.458 SCF   |
| 2. Gas Volume Sampled - STPD                        | 58.152 SCFD |
| 3. Total Volume                                     | 67.610 SCF  |
| 4. Moisture in Stack Gas - Volume Fraction          | .140        |
| 5. Dry Stack Gas - Volume Fraction                  | .860        |
| 6. Molecular Weight of Stack Gas - Dry Basis        | 29.00       |
| 7. Molecular Weight of Stack Gas - Stack Conditions | 27.46       |
| 8. Specific Gravity of Stack Gas Relative to Air    | -.95        |
| 9. Excess Air - Percent                             |             |
| 10. Average Stack Velocity                          | 2392.4 FPM  |
| 11. Average Stack Gas Flow Rate                     | 67178 ACFM  |
| 12. Actual Stack Gas Flow Rate Dry                  | 57781 CFMD  |
| 13. Stack Gas Flow Rate STPD                        | 51949 SCFMD |
| 14. Percent Isokinetic                              | 97.2 %      |

Probe Wash:	5.60 Mg	.0015 Gr/SCF	.66 Lbs/Hr
Filter:	107.80 Mg	.0285 Gr/SCF	12.74 Lbs/Hr
===Totals===	113.40 Mg	.0300 Gr/SCF	13.40 Lbs/Hr



Source Sampling Input Data

09:33 24-Feb-84

Plant Name: AMAX  
 Stack Location: BIG 4 DRYER COAL/OIL  
 Weather: PARTLY CLOUDY  
 Run Date: 2/16/84  
 Run #: 1  
 Time Start: 1106  
 Time End: 1210  
 Total Min: 60  
 Baro. Press: 30.12  
 Stack Press: 30.12  
 Nozzle Diam: .3143  
 Stack Area: 28.08  
 Final Meter: 332.872  
 Initial Meter: 274.5  
 Cond. Volume: 198.7  
 Stack Velocity: .444  
 Meter Diff: 3.45  
 Stack Gas Temp: 131.5  
 Meter Temp: 87.3

H2SO4 --- Vt:  
 Vtb:  
 N:  
 Vsoln:  
 Va:  
 SO2 --- Vt:  
 Vtb:  
 N:  
 Vsoln:  
 Va:  
 Prod Rate(TPD):  
 % O2:

Pollutant: 3  
 H2SO4=1  
 Part=2  
 Scrubber=3

Source Type: 2  
 Combustion=1  
 Non-Comb.=2  
 ORSAT=3

%CO2:  
 ORSAT - % CO:  
 % O2:

Weights for -  
 Probe Wash: 8.70  
 Filter: 86.9  
 :  
 :  
 :

Source Samplings Input Data

09:36 24-Feb-84

Plant Name: AMAX  
 Stack Location: BIG 4 DRYER COAL/OIL  
 Weather: PARTLY CLOUDY  
 Run Date: 2/16/84  
 Run #: 2  
 Time Start: 1237  
 Time End: 1342  
 Total Min: 60  
 Baro. Press: 30.12  
 Stack Press: 30.12  
 Nozzle Diam: .3143  
 Stack Area: 28.08  
 Final Meter: 393.2  
 Initial Meter: 333.3  
 Cond. Volume: 204.6  
 Stack Velocity: .439  
 Meter Diff: 3.476  
 Stack Gas Temp: 134.2  
 Meter Temp: 94.2

H2SO4 --- Vt:  
 Vtb:  
 N:  
 Vsoln:  
 Va:  
 SO2 --- Vt:  
 Vtb:  
 N:  
 Vsoln:  
 Va:  
 Prod Rate(TPD):  
 % O2:

Pollutant: 3  
 H2SO4=1  
 Part=2  
 Scrubber=3

Source Type: 2  
 Combustion=1  
 Non-Comb.=2  
 QRSAT=3

%CO2:  
 QRSAT - % CO:  
 % O2:

Weights for -  
 Probe Wash: 9.60  
 Filter: 103.9  
 :  
 :  
 :

Source Sampling Input Data

09:39 24-Feb-84

Plant Name: AMAX  
 Stack Location: BIG 4 DRYER COAL/OIL  
 Weather: PARTLY CLOUDY  
 Run Date: 2/16/84  
 Run #: 3  
 Time Start: 1405  
 Time End: 1508  
 Total Min: 60  
 Baro. Press: 30.12  
 Stack Press: 30.12  
 Nozzle Diam: .3143  
 Stack Area: 28.08  
 Final Meter: 453.888  
 Initial Meter: 393.9  
 Cond. Volume: 200.8  
 Stack Velocity: .432  
 Meter Diff: 3.43  
 Stack Gas Temp: 131.2  
 Meter Temp: 92.9

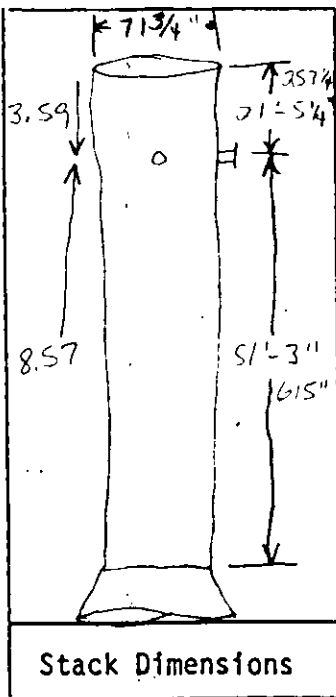
H2SO4 --- Vt:  
 Vtb:  
 N:  
 Vsoln:  
 Va:  
 SO2 --- Vt:  
 Vtb:  
 N:  
 Vsoln:  
 Va:  
 Prod Rate(TPD):  
 % O2:

Pollutant: 3  
 H2SO4=1  
 Part=2  
 Scrubber=3  
  
 Source Type: 2  
 Combustion=1  
 Non-Comb.=2  
 ORSAT=3  
  
 %CO2:  
 ORSAT - % CO:  
 % O2:  
  
 Weights for -  
 Probe Wash: 5.60  
 Filter: 107.8  
 :  
 :  
 :



SOURCE SAMPLING FIELD DATA SHEET

Plant Amax  
 Sampling Location Big 4  
 Type of Control Scrubber  
 Type of Samples Part  
 Date 2-16-84 Run No. 1 Cool off  
 Time Start 1106 Time End 1210  
 Sample Time 5 min/pt 60 Total min  
 DB      °F, WB      °F, VP @ DP      "Hg  
 Bar. Press. 30.12 "Hg, Stack Press. 30.12 "Hg  
 Moisture 15 %, FDA     , Gas Density Factor       
 Temp. 74 °F, W/D Vari., W/S 3-5  
 Weather Ptly Cloudy Thermocouple Readout Sk-1  
 Sample Box No. Sk-1 meter Box No. Sk-1  
 Meter ΔH@ 1.83 Pitot Corr. Factor 0.84  
 Nozzle Dia. 5/16 in., Probe Length Sk-91 ft  
 Probe Heater Setting 4 Nomograph C<sub>f</sub> 7.76  
 Stack Dimensions 71.75 in  
 Stack Area 28.08 ft<sup>2</sup>  
 Effective Stack Area 28.08 ft<sup>2</sup>  
 Stack Height      ft



Stack Dimensions

Umbilical Cord 100' 200'

Mat'l Processing Rate       
 Final Gas Meter Reading 332,872 ft<sup>3</sup>  
 Initial Gas Meter Reading 274,500 ft<sup>3</sup> 58,372  
 Condensate Increase in Impingers 188 ml 198.7  
 Moisture in Silica Gel 10.7 gm  
 Silica Gel Container No. 27 Filter No. 4-045

Orsat: %CO <sub>2</sub>				
%O <sub>2</sub>				
%CO				
%N <sub>2</sub>				

Test Conducted By: R Paul  
D Patel

Stack Test Observers:     

Leak Check Meter Box Initial 0.00 cfm @ 15 In H<sub>2</sub>  
 Final 0.015 cfm @ 15 In Hg

Pitot Tubes Impact 3 In H<sub>2</sub>O for 15 sec Stable Leak  
 Static 3 In H<sub>2</sub>O for 15 sec Stable Leak

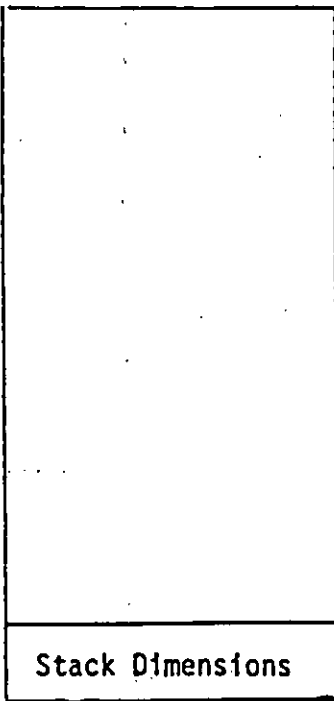
Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual					
1	3.2		74.5	.43	3.34	3.34	143	81	248	57	8
2	10.5		79.2	.49	3.8	3.8	142	81	254	46	10
3	21.2		84.3	.46	3.57	3.57	137	82	262	48	10
4	50.6		89.5	.40	3.11	3.11	131	84	260	48	9
5	61.2		94.1	.2541	3.98	3.98	126	85	266	48	9
6	68.6		98.5	.36	2.79	2.79	122	88	239	49	8
1			02.8	.47	3.65	3.65	134	89	257	54	10
2			7.8	.47	3.65	3.65	132	90	250	50	10

Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual					
3			12.8	.45	3.49	3.49	128	90	247	49	10
4			17.8	.45	3.49	3.49	128	92	243	49	10
5			22.8	.47	3.65	3.65	127	92	242	50	11
6			27.9	.48	3.72	3.72	128	93	242	50	12



SOURCE SAMPLING FIELD DATA SHEET

Plant Amat  
 Sampling Location Blg 4 Dryer  
 Type of Control Scrubber  
 Type of Samples Part  
 Date 2-16-84 Run No. 2 Coal Oil  
 Time Start 1237 Time End 1342  
 Sample Time 5 min/pt 100 Total min  
 DB      °F, WB      °F, VP @ DP      "Hg  
 Bar. Press. 30.12 "Hg, Stack Press. 30.12 "Hg  
 Moisture 15 %, FDA     , Gas Density Factor       
 Temp. 77 °F, W/D Vari., W/S 3-5  
 Weather Pbly Cloudy Thermocouple Readout Ab-1  
 Sample Box No. Ab-1 meter Box No. Ab-1  
 Meter ΔH@ 1.83 Pitot Corr. Factor 0.84  
 Nozzle Dia. 3/16 in., Probe Length Ab-81 ft  
 Probe Heater Setting 4 Nomograph C<sub>f</sub> 7.905  
 Stack Dimensions 76.75 " in  
 Stack Area 28.08 ft<sup>2</sup>  
 Effective Stack Area 28.08 ft<sup>2</sup>  
 Stack Height      ft



Stack Dimensions

Umbilical Cord 100' 200'

Mat'l Processing Rate       
 Final Gas Meter Reading 393.200 ft<sup>3</sup>  
 Initial Gas Meter Reading 333.300 ft<sup>3</sup>  
 Condensate Increase in Impingers 196 ml 204.6  
 Moisture in Silica Gel 8.6 gm  
 Silica Gel Container No. 31 Filter No. 5-046  
 Orsat: %CO<sub>2</sub>                      
 %O<sub>2</sub>                      
 %CO                      
 %N<sub>2</sub>                    

Test Conducted By: R Paul  
B. J. J. J.

Stack Test Observers:     

Leak Check Meter Box Initial 0.00 cfm @ 15 In H<sub>2</sub>  
 Final 0.00 cfm @ 13 In Hg

Pitot Tubes Impact 3 In H<sub>2</sub>O for 15 sec. Stable Leak  
 Static 3 In H<sub>2</sub>O for 15 sec. Stable Leak

Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual					
1			33.3	.40	3.16	3.16	139	93	232	63	9
2			38.1	.47	3.72	3.72	134	93	241	46	11
3			43.1	.47	3.72	3.72	135	93	257	46	11
4			48.3	.47	3.72	3.72	133	93	259	47	11
5			53.6	.47	3.72	3.72	131	94	250	45	11
6			58.6	.42	3.3	3.3	129	94	247	45	11
1			63.6	.42	3.3	3.3	142	95	263	47	11
2			68.5	.49	3.87	3.87	135	95	242	47	13

Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual					
					3						
4			78.8	.44	3.48	3.48	133	95	264	49	13
5			83.9	.39	3.08	3.08	132	95	265	48	12
6			88.8	.37	2.92	2.92	131	95	260	48	12

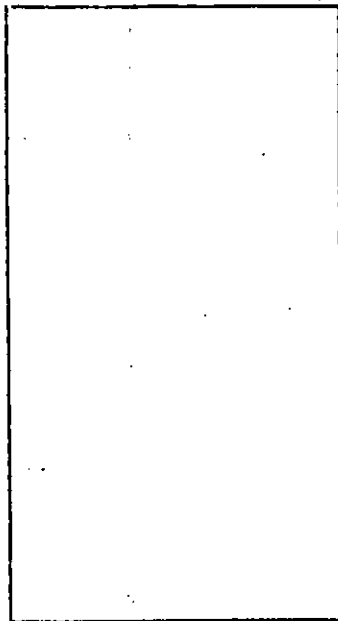
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SK



SOURCE SAMPLING FIELD DATA SHEET

Plant Broad  
 Sampling Location Big 4 Dryer  
 Type of Control Scrubber  
 Type of Samples Part  
 Date 2-16-84 Run No. 3 Coalat  
 Time Start 1405 Time End 1508  
 Sample Time 5 min/pt 100 Total min  
 DB        °F, WB        °F, VP @ DP        "Hg  
 Bar. Press. 30.12 "Hg, Stack Press. 30.12 "Hg  
 Moisture 15 %, FDA       , Gas Density Factor         
 Temp. 75 °F, W/D Var, W/S 3-8  
 Weather Ptly Cloudy Thermocouple Readout Ab-t  
 Sample Box No. Ab-1 meter Box No. Ab-1  
 Meter ΔH<sub>0</sub> 1.83 Pitot Corr. Factor 0.84  
 Nozzle Dia. 5/16 in., Probe Length Ab-81 ft  
 Probe Heater Setting 4 Nomograph C<sub>p</sub> 7.905  
 Stack Dimensions 71.75 in  
 Stack Area 28.08 ft<sup>2</sup>  
 Effective Stack Area 28.08 ft<sup>2</sup>  
 Stack Height        ft



Stack Dimensions

Umbilical Cord 100' (200')

Mat'l Processing Rate         
 Final Gas Meter Reading 453.888 ft<sup>3</sup>  
 Initial Gas Meter Reading 393.900 ft<sup>3</sup>  
 Condensate Increase in Impingers 192 ml 200.8  
 Moisture in Silica Gel 8.8 gm  
 Silica Gel Container No. 3 Filter No. 6-047

Orsat: %CO <sub>2</sub>				
%O <sub>2</sub>				
%CO				
%N <sub>2</sub>				

Test Conducted By: R Paul  
A Habel

Stack Test Observers:       

Leak Check Meter Box Initial 0.00 cfm @ 15 In H<sub>2</sub>  
 Final 0.006 cfm @ 11 In Hg

Pitot Tubes Impact 3 In H<sub>2</sub>O for 15 sec. Stable, Leak  
 Static 3 In H<sub>2</sub>O for 15 sec. Stable, Leak

Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual					
1			93.9	.42	3.82	3.32	143	92	260	60	10
2			99.1	.47	3.72	3.72	137	92	264	54	11
3			4.0	.47	3.72	3.72	132	91	259	55	11
4			9.2	.42	3.32	3.32	123	91	260	55	11
5			14.3	.39	3.08	3.08	122	92	267	56	10
6			19.0	.36	2.85	2.85	124	93	254	56	10
1			23.5	.41	3.24	3.24	134	94	250	61	11
2			28.5	.41	3.24	3.24	133	94	256	52	9



Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual					
3			33.3	.47	3.72	3.72	132	94	242	51	10
4			38.5	.47	3.72	3.72	132	94	243	50	11
5			43.6	.47	3.72	3.72	130	94	260	49	11
6			49.0	.44	3.48	3.48	132	94	264	48	11



SAMPLING RATE  
CALCULATIONS

S&K NOMOGRAPH

CO. NAME: Amal  
DATE: 2-10-84  
STACK: Reg 4  
LOCATION: \_\_\_\_\_

- $\Delta H$  = ORIFICE READING (INCHES H<sub>2</sub>O)
- $D_n$  = NOZZLE DIA. (INCHES)
- $\Delta H_0$  = METER BOX CONSTANT
- $B_w$  = MOISTURE FRACTION
- $T_m$  = METER TEMP (°F)
- $T_s$  = STACK TEMP (°F)
- $M_s$  = WET MOLECULAR WEIGHT OF STACK GAS (FROM TABLE)
- $\Delta P$  = PITOT READING (INCHES H<sub>2</sub>O)

$$\left[ \frac{T_m + 460}{M_s(T_s + 460)} (1 - B_w)^2 \Delta H_0 (D_n)^4 17741 \right] \Delta P = \Delta H$$

130  
.45-.30

MOISTURE FRACTION	$M_s$
0.0	29.0
0.05	28.5
0.10	27.9
0.15	27.4
0.20	26.8
0.25	26.2
0.30	25.7
0.35	25.2
0.40	24.6

548  
27.4 (590) 16164  
558

	<u>Run 1</u>	<u>Run 2</u>	<u>Run 3</u>
$\frac{T_m + 460}{M_s(T_s + 460)} =$	<u>0.033898</u>	<u>0.03452</u>	_____
x $(1 - B_w)^2 =$	<u>0.7225</u>	_____	_____
x $\Delta H_0 =$	<u>183</u>	_____	_____
x $(D_n)^4 =$	<u>0.009763</u>	_____	_____
x 17741 =	<u>17741</u>	<u>17741</u>	<u>17741</u>
x $\Delta P =$	<u>7.76</u>	<u>7.904</u>	_____

METER ORIFICE CHECK

Meter Box No. Ab-1

$\Delta$  Ha of Box 1.83

Date 2-16-84

Time for 10 ft<sup>3</sup> = 13 min. 24 sec.  
264.1

10  $\div$  13.4 total minutes = 0.7463  $\Delta$  Ha<sub>2</sub>

$\frac{0.75}{\Delta \text{Ha}_2} = 1.005 \times 100 = \underline{100.5\%}$

RC Paul  
Signature

Procedure: Set flow rate of meter box at  $\Delta$ Ha of meter box--measure the amount of time required to sample 10.00 ft<sup>3</sup>.

Note: If answer is within 5 percent meter is o.k. If not, recalibrate meter.

NOZZLE CALIBRATION

Nozzle 5/14

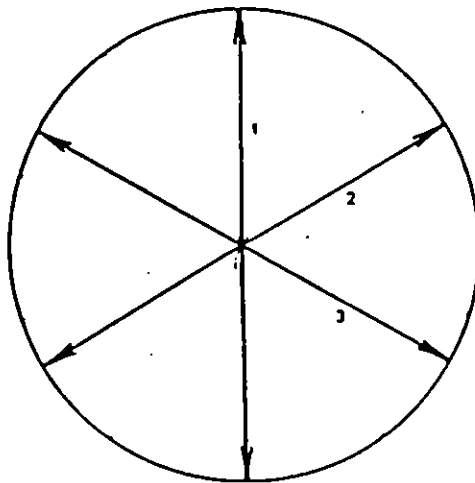
Date 2-16-84

<u>Measurement No.</u>	<u>Inside Diameter (inches)</u>
1	<u>.314</u>
2	<u>.314</u>
3	<u>.315</u>

Average .3143

Area of Nozzle \_\_\_\_\_ Ft<sup>2</sup>

Calibrated by: Rodney Paul



Nozzle X-section

SAMPLE CHAIN OF CUSTODY

PLANT NAME: Ames  
SOURCE NAME: Big 4 Dryer cool oil  
DATE: 2-16-84 TYPE OF SAMPLE: Part

SAMPLE RECOVERY

<u>CONTAINER NO.</u>	<u>DESCRIPTION</u>
<u>1-ACC-P</u>	<u>Probe Wash Run 1</u>
<u>2-ACC-P</u>	<u>} } } 2</u>
<u>3-ACC-P</u>	<u>} } } 3</u>
<u>4</u>	<u>Filter Holder Run 1</u>
<u>5</u>	<u>} } } 2</u>
<u>6</u>	<u>} } } 3</u>
<u>27</u>	<u>Silica Gel Run 1</u>
<u>31</u>	<u>} } } 2</u>
<u>3</u>	<u>} } } 3</u>

Sample Recovered By: RA & RR  
Particulate Analysis By: R. Paul

SHOLTES & KOOGLER, ENVIRONMENTAL CONSULTANTS, INC.  
PARTICULATE LAB DATA SHEET

Project No. AMAX-COAL/OIL  
2-16-84

	<u>Run 1</u>	<u>Run 2</u>	<u>Run 3</u>	<u>Blank</u>
Container No.	<u>16-29</u>	<u>16-47</u>	<u>16-31</u>	<u>16-3</u>
Total Volume (ml)	<u>253</u>	<u>247</u>	<u>236</u>	<u>100</u>
Aliquot Evaporated (ml)	<u>253</u>	<u>247</u>	<u>236</u>	<u>100</u>
Final Weight (g)	<u>108.8817</u>	<u>97.3080</u>	<u>88.0466</u>	<u>90.4763</u>
Tare Weight (g)	<u>-108.8730</u>	<u>-97.2984</u>	<u>-88.0410</u>	<u>-90.4763</u>
Gross Weight Gained (g)	<u>0.0087</u>	<u>0.0096</u>	<u>0.0056</u>	<u>0.0000</u>
Average Blank (g)	<u>-</u>	<u>-</u>	<u>-</u>	<u>-</u>
Net Weight (g)	<u>0.0087</u>	<u>0.0096</u>	<u>0.0056</u>	<u>-</u>
Aliquot Factor	<u>x 1.0</u>	<u>x 1.0</u>	<u>x 1.0</u>	<u>x 1.0</u>
Total Net Weight (mg)	<u>8.7</u>	<u>9.6</u>	<u>5.6</u>	<u>-</u>

Container No.	<u>1F</u>	<u>2F</u>	<u>3F</u>	<u>-</u>
Filter No.	<u>045</u>	<u>046</u>	<u>047</u>	<u>-</u>
Final Weight (g)	<u>0.5444</u>	<u>0.5609</u>	<u>0.5638</u>	<u>-</u>
Tare Weight (g)	<u>0.4575</u>	<u>0.4570</u>	<u>0.4560</u>	<u>-</u>
Gross Weight Gained (g)	<u>0.0869</u>	<u>0.1039</u>	<u>0.1078</u>	<u>-</u>
Average Blank	<u>-</u>	<u>-</u>	<u>-</u>	<u>-</u>
Total Net Weight (mg)	<u>86.9</u>	<u>103.9</u>	<u>107.8</u>	<u>-</u>

Tare Balance Check

0 0.0 50g 50.0000  
 10g 10.0000 100g 100.0000  
 0.5g 5.000

By RC Paul  
 Date 2-22-84

Final Balance Check

0 0.0000 50g 50.0000  
 10g 10.0000 100g 100.0000  
 0.5g 0.5000

By R Paul  
 Date 2-23-84

Table 1

SUMMARY OF PARTICULATE MATTER EMISSIONS

AMAX  
BIG 4 DRYER  
2/17/84

Run No.	Process Weight Rate (Tons/Hr)	Stack Gas Flow Rate (SCFMD)	Stack Gas Temperature (Deg F)	Stack Gas Moisture (%)	Particulate Matter	
					Conc. (gr/SCF)	Emission Rate (Lbs/Hr)
1	276.7	54051	130.7	14.2	.0268	12.45
2	270.0	53472	141.9	14.4	.0223	10.24
3	270.0	53324	144.8	14.2	.0228	10.43
Ave	272.2	53616	139.1	14.2	.0240	11.04

Allowable Particulate Matter Emission Rate =  
(Chap. 17-2, Florida Administrative Code)

Lbs/Hr

SHOLTES AND KOOGLER ENVIRONMENTAL CONSULTANTS

Source Sampling Calculations

Plant: AMAX  
 Stack: BIG 4 DRYER - OIL  
 Weather: PARTLY CLOUDY

Date: 2/16/84  
 Run 1 From 1617 - 1719  
 Total Time: 60 Min

Stack Area	28.08 Sq Ft	Nozzle Area	.000539 Sq Ft
Stack Temp	130.7 Deg F	Meter Temp	91.3 Deg F
Stack Pressure	30.12 "Hg	Baro. Pressure	30.12 "Hg
Stack Vel Head	.685 "H2O	Meter Press Diff	3.72 "H2O
		Meter Volume	61.972 cf
Pitot Tube Factor	.84	Condensate Volume	211.5 ml

- |   |             |
|---|-------------|
| 1. Volume Water Vapor                               | 9.962 SCF   |
| 2. Gas Volume Sampled - STPD                        | 60.292 SCFD |
| 3. Total Volume                                     | 70.253 SCF  |
| 4. Moisture in Stack Gas - Volume Fraction          | .142        |
| 5. Dry Stack Gas - Volume Fraction                  | .858        |
| 6. Molecular Weight of Stack Gas - Dry Basis        | 29.00       |
| 7. Molecular Weight of Stack Gas - Stack Conditions | 27.44       |
| 8. Specific Gravity of Stack Gas Relative to Air    | .95         |
| 9. Excess Air - Percent                             |             |
| 10. Average Stack Velocity                          | 2492.6 FPM  |
| 11. Average Stack Gas Flow Rate                     | 69993 ACFM  |
| 12. Actual Stack Gas Flow Rate Dry                  | 60068 CFMD  |
| 13. Stack Gas Flow Rate STPD                        | 54051 SCFMD |
| 14. Percent Isokinetic                              | 96.8 %      |

Probe Wash:	13.00 Mg	.0033 Gr/SCF	1.54 Lbs/Hr
Filter:	92.00 Mg	.0235 Gr/SCF	10.91 Lbs/Hr
===Totals===	105.00 Mg	.0268 Gr/SCF	12.45 Lbs/Hr



SHOLTES AND KOOGLER ENVIRONMENTAL CONSULTANTS

Source Sampling Calculations

Plant: AMAX  
 Stack: BIG 4 DRYER - OIL  
 Weather: CLEAR

Date: 2/17/84  
 Run 2 From 0841 - 0943  
 Total Time: 60 Min

Stack Area	28.08 Sq Ft	Nozzle Area	.000539 Sq Ft
Stack Temp	141.9 Deg F	Meter Temp	75.8 Deg F
Stack Pressure	30.14 "Hg	Baro. Pressure	30.14 "Hg
Stack Vel Head	.685 "H2O	Meter Press Diff	3.51 "H2O
		Meter Volume	59.830 cf
Pitot Tube Factor	.84	Condensate Volume	213.2 ml

- |   |             |
|---|-------------|
| 1. Volume Water Vapor                               | 10.042 SCF  |
| 2. Gas Volume Sampled - STPD                        | 59.902 SCFD |
| 3. Total Volume                                     | 69.943 SCF  |
| 4. Moisture in Stack Gas - Volume Fraction          | .144        |
| 5. Dry Stack Gas - Volume Fraction                  | .856        |
| 6. Molecular Weight of Stack Gas - Dry Basis        | 29.00       |
| 7. Molecular Weight of Stack Gas - Stack Conditions | 27.42       |
| 8. Specific Gravity of Stack Gas Relative to Air    | -.95        |
| 9. Excess Air - Percent                             |             |
| 10. Average Stack Velocity                          | 2516.2 FPM  |
| 11. Average Stack Gas Flow Rate                     | 70655 ACFM  |
| 12. Actual Stack Gas Flow Rate Dry                  | 60511 CFMD  |
| 13. Stack Gas Flow Rate STPD                        | 53472 SCFMD |
| 14. Percent Isokinetic                              | 97.2 %      |

Probe Wash:	15.00 Mg	.0039 Gr/SCF	1.77 Lbs/Hr
Filter:	71.70 Mg	.0184 Gr/SCF	8.47 Lbs/Hr
===Totals===	86.70 Mg	.0223 Gr/SCF	10.24 Lbs/Hr

SHOLTES AND KOOGLER ENVIRONMENTAL CONSULTANTS

Source Sampling Calculations

Plant: AMAX  
 Stack: BIG 4 DRYER - OIL  
 Weather: CLEAR

Date: 2/17/84  
 Run 3 From 1003 - 1107  
 Total Time: 60 Min

Stack Area	28.08 Sq Ft	Nozzle Area	.000539 Sq Ft
Stack Temp	144.8 Deg F	Meter Temp	90 Deg F
Stack Pressure	30.14 "Hg	Baro. Pressure	30.14 "Hg
Stack Vel Head	.683 "H2O	Meter Press Diff	3.65 "H2O
		Meter Volume	61.300 cf
Pitot Tube Factor	.84	Condensate Volume	209.7 ml

- |   |             |
|---|-------------|
| 1. Volume Water Vapor                               | 9.877 SCF   |
| 2. Gas Volume Sampled - STPD                        | 59.808 SCFD |
| 3. Total Volume                                     | 69.685 SCF  |
| 4. Moisture in Stack Gas - Volume Fraction          | .142        |
| 5. Dry Stack Gas - Volume Fraction                  | .858        |
| 6. Molecular Weight of Stack Gas - Dry Basis        | 29.00       |
| 7. Molecular Weight of Stack Gas - Stack Conditions | 27.44       |
| 8. Specific Gravity of Stack Gas Relative to Air    | .95         |
| 9. Excess Air - Percent                             |             |
| 10. Average Stack Velocity                          | 2515.9 FPM  |
| 11. Average Stack Gas Flow Rate                     | 70648 ACFM  |
| 12. Actual Stack Gas Flow Rate Dry                  | 60635 CFMD  |
| 13. Stack Gas Flow Rate STPD                        | 53324 SCFMD |
| 14. Percent Isokinetic                              | 97.3 %      |

Probe Wash:	9.60 Mg	.0025 Gr/SCF	1.13 Lbs/Hr
Filter:	78.80 Mg	.0203 Gr/SCF	9.29 Lbs/Hr
===Totals===	88.40 Mg	.0228 Gr/SCF	10.43 Lbs/Hr

Source Sampling Input Data

09:08 24-Feb-84

Plant Name: AMAX  
 Stack Location: BIG 4 DRYER - OIL  
 Weather: PARTLY CLOUDY  
 Run Date: 2/16/84  
 Run #: 1  
 Time Start: 1617  
 Time End: 1719  
 Total Min: 60  
 Baro. Press: 30.12  
 Stack Press: 30.12  
 Nozzle Diam: .3143  
 Stack Area: 28.08  
 Final Meter: 516.172  
 Initial Meter: 454.2  
 Cond. Volume: 211.5  
 Stack Velocity: .469  
 Meter Diff: 3.716  
 Stack Gas Temp: 130.7  
 Meter Temp: 91.3

H2SO4 --- Vt:  
 Vtb:  
 N:  
 Vsoln:  
 Va:  
 SO2 --- Vt:  
 Vtb:  
 N:  
 Vsoln:  
 Va:  
 Prod Rate(TPD):  
 % O2:

Pollutant: 3  
 H2SO4=1  
 Part=2  
 Scrubber=3

Source Type: 2  
 Combustion=1  
 Non-Comb.=2  
 ORSAT=3

%CO2:  
 ORSAT - % CO:  
 % O2:

Weights for -  
 Probe Wash: 13.0  
 Filter: 92.0  
 :  
 :  
 :

Source Sampling Input Data

09:13 24-Feb-84

Plant Name: AMAX  
 Stack Location: BIG 4 DRYER - OIL  
 Weather: CLEAR  
 Run Date: 2/17/84  
 Run #: 2  
 Time Start: 0841  
 Time End: 0943  
 Total Min: 60  
 Baro. Press: 30.14  
 Stack Press: 30.14  
 Nozzle Diam: .3143  
 Stack Area: 28.08  
 Final Meter: 576.630  
 Initial Meter: 516.8  
 Cond. Volume: 213.2  
 Stack Velocity: .469  
 Meter Diff: 3.513  
 Stack Gas Temp: 141.9  
 Meter Temp: 75.8

H2SO4 --- Vt:  
 Vtb:  
 N:  
 Vsoln:  
 Va:  
 SO2 --- Vt:  
 Vtb:  
 N:  
 Vsoln:  
 Va:  
 Prod Rate(TPD):  
 % O2:

Pollutant: 3  
 H2SO4=1  
 Part=2  
 Scrubber=3

Source Type: 2  
 Combustion=1  
 Non-Comb.=2  
 ORSAT=3

%CO2:  
 ORSAT - % CO:  
 % O2:

Weights for -  
 Probe Wash: 15.0  
 Filter: 71.7  
 :  
 :  
 :

Source Sampling Input Data

09:16 24-Feb-84

Plant Name: AMAX  
Stack Location: BIG 4 DRYER - OIL  
Weather: CLEAR  
Run Date: 2/17/84  
Run #: 3  
Time Start: 1003  
Time End: 1107  
Total Min: 60  
Baro. Press: 30.14  
Stack Press: 30.14  
Nozzle Diam: .3143  
Stack Area: 28.08  
Final Meter: 648.6  
Initial Meter: 587.3  
Cond. Volume: 209.7  
Stack Velocity: .467  
Meter Diff: 3.649  
Stack Gas Temp: 144.8  
Meter Temp: 90

H2SO4 --- Vt:  
Vtb:  
N:  
Vsoln:  
Va:  
SO2 --- Vt:  
Vtb:  
N:  
Vsoln:  
Va:  
Prod Rate(TPD):  
% O2:

Pollutant: 3  
H2SO4=1  
Part=2  
Scrubber=3

Source Type: 2  
Combustion=1  
Non-Comb.=2  
ORSAT=3

%CO2:  
ORSAT - % CO:  
% O2:

Weights for -  
Probe Wash: 9.60  
Filter: 78.8  
:  
:  
:



SOURCE SAMPLING FIELD DATA SHEET

Plant A. mad  
 Sampling Location Big 4 Dryer  
 Type of Control scrubber  
 Type of Samples Part  
 Date 2-16-84 Run No. 1 ~~1011~~  
 Time Start 1617 Time End 1719  
 Sample Time 5 min/pt 60 Total min  
 DB        °F, WB        °F, VP @ DP        "Hg  
 Bar. Press. 30.12 "Hg, Stack Press. 30.12 "Hg  
 Moisture 15 %, FDA       , Gas Density Factor         
 Temp. 72 °F, W/D Var., W/S 3-8  
 Weather Partly Cloudy Thermocouple Readout Abt  
 Sample Box No. 16-1 meter Box No. 16-1  
 Meter ΔH 1.83 Pitot Corr. Factor 0.84  
 Nozzle Dia. 5/16 in., Probe Length 16-81 ft  
 Probe Heater Setting 4 Nomograph C<sub>f</sub> 7.905  
 Stack Dimensions 71.75 in  
 Stack Area 28.08 ft<sup>2</sup>  
 Effective Stack Area 28.08 ft<sup>2</sup>  
 Stack Height        ft

0.12

Stack Dimensions

Umbilical Cord 100' (200)

Mat'l Processing Rate         
 Final Gas Meter Reading 516.172 ft<sup>3</sup>  
 Initial Gas Meter Reading 454.200 ft<sup>3</sup>  
 Condensate Increase in Impingers 200 ml 211.5  
 Moisture in Silica Gel 11.5 gm  
 Silica Gel Container No. 10 Filter No. 1-1252  
 Orsat: %CO<sub>2</sub>                              
 %O<sub>2</sub>                              
 %CO                              
 %N<sub>2</sub>                            

Test Conducted By: R Paul  
H Haber

Stack Test Observers:       

Leak Check  
 Meter Box Initial 0.00 cfm @ 15 In H<sub>2</sub>  
 Final 0.00 cfm @ 12 In Hg

Pitot Tubes  
 Impact 3 In H<sub>2</sub>O for 15 sec. Stable, Leak  
 Static 3 In H<sub>2</sub>O for 15 sec. Stable, Leak

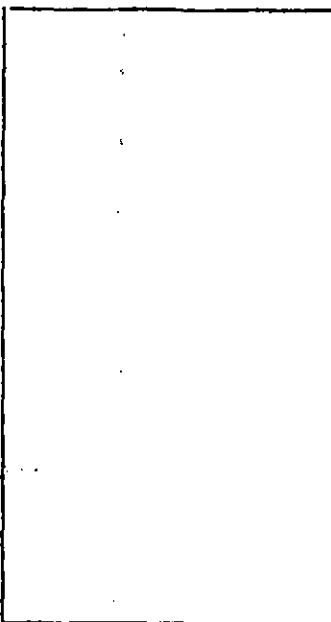
Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)	
					Calc.	Actual						
1			54.2	.47	3.72	3.72	133	260	90	260	49	10
2			59.4	.52	4.11	4.11	137	90	251	48	11	
3			64.7	.52	4.11	4.11	139	90	263	51	11	
4			70.2	.47	3.72	3.72	128	90	270	52	11	
5			75.4	.49	3.87	3.87	129	91	261	51	11	
6			80.7	.50	3.95	3.95	131	91	266	51	12	
1			86.0	.45	3.55	3.55	138	91	269	52	11	
2			90.9	.47	3.72	3.72	131	92	256	52	12	

Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual					
3			96.0	.44	3.87	3.87	133	92	262	53	12
4			501.5	.45	3.56	3.56	126	92	262	53	12
5			505.9	.45	3.56	3.56	123	93	255	53	12
6			11.5	.36	2.85	2.85	120	93	253	54	10



SOURCE SAMPLING FIELD DATA SHEET

Plant A-mat  
 Sampling Location Big 4 Dryer  
 Type of Control scrubber  
 Type of Samples Past  
 Date 2-17-84 Run No. 2 oil  
 Time Start 0841 Time End 0943  
 Sample Time 5 min/pt 100 Total min  
 DB        °F, WB        °F, VP @ DP        "Hg  
 Bar. Press. 30.14 "Hg, Stack Press. 30.14 "Hg  
 Moisture 15 %, FDA       , Gas Density Factor         
 Temp. 55 °F, W/D Var, W/S 2-3  
 Weather Clear Thermocouple Readout Ab-1  
 Sample Box No. Ab-1 Meter Box No. Ab-1  
 Meter ΔH<sub>0</sub> 1.83 Pitot Corr. Factor 0.84  
 Nozzle Dia. 5/16 in., Probe Length Ab-81 ft  
 Probe Heater Setting        Nomograph C<sub>f</sub> 7.512  
 Stack Dimensions 71.75 in  
 Stack Area 28.08 ft<sup>2</sup>  
 Effective Stack Area 28.08 ft<sup>2</sup>  
 Stack Height        ft



Stack Dimensions

Umbilical Cord 100' 200'

Mat'l Processing Rate         
 Final Gas Meter Reading 576.630 ft<sup>3</sup>  
 Initial Gas Meter Reading 516.800 ft<sup>3</sup>  
 Condensate Increase in Impingers 203 ml 213.2  
 Moisture in Silica Gel 10.2 gm  
 Silica Gel Container No. 22 Filter No. 2-1253  
 Orsat: %CO<sub>2</sub>                              
 %O<sub>2</sub>                              
 %CO                              
 %N<sub>2</sub>                            

Test Conducted By: R Paul  
H. F. F.

Stack Test Observers:         
      

Leak Check Meter Box Initial 0.00 cfm @ 15 In H<sub>2</sub>  
 Final 0.00 cfm @ 13 In Hg

Pitot Tubes Impact 3 In H<sub>2</sub>O for 15 sec. Stable Leak  
 Static 3 In H<sub>2</sub>O for 15 sec. Stable Leak

Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual					
1			16.8	.45	3.38	3.38	143	66	266	62	10
2			21.6	.45	3.38	3.38	146	66	268	55	10
3			26.5	.50	3.76	3.76	146	69	270	53	11
4			31.7	.48	3.61	3.61	145	71	267	51	11
5			37.2	.50	3.76	3.76	146	74	261	51	12
6			41.7	.47	3.53	3.53	145	76	257	51	11
1			46.7	.44	3.31	3.31	152	78	259	53	11
2			51.7	.51	3.83	3.83	148	80	266	51	13



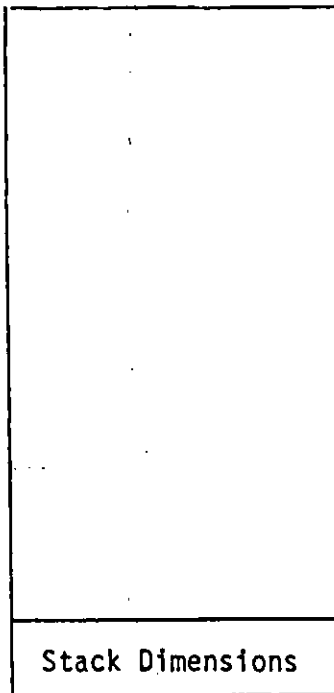
Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual					
3			56.9	.49	3.68	3.68	144	81	251	51	13
4			61.9	.47	3.53	3.53	134	82	252	51	13
5			66.9	.44	3.31	3.31	127	83	250	51	12
6			72.0	.41	3.08	3.08	127	84	248	51	12

RK



SOURCE SAMPLING FIELD DATA SHEET

Plant Amad  
 Sampling Location Big 4 Dyes  
 Type of Control Scrubber  
 Type of Samples Part  
 Date 2-17-84 Run No. 3 oil  
 Time Start 1003 Time End 1107  
 Sample Time 5 min/pt 60 Total min  
 DB      °F, WB      °F, VP @ DP      "Hg  
 Bar. Press. 30.14 "Hg, Stack Press. 30.14 "Hg  
 Moisture 15 %, FDA     , Gas Density Factor       
 Temp. 103 °F, W/D Var, W/S 3-5  
 Weather Clear Thermocouple Readout skt  
 Sample Box No. skt meter Box No. skt  
 Meter ΔH@ 1.83 Pitot Corr. Factor 0.84  
 Nozzle Dia. 3/16 in., Probe Length skt-81 ft  
 Probe Heater Setting 4 Nomograph C<sub>p</sub> 1.808  
 Stack Dimensions 71.75 in  
 Stack Area 28.08 ft<sup>2</sup>  
 Effective Stack Area 28.08 ft<sup>2</sup>  
 Stack Height      ft



Stack Dimensions

Umbilical Cord 100' (200)

Mat'l Processing Rate       
 Final Gas Meter Reading 648.600 ft<sup>3</sup>  
 Initial Gas Meter Reading 587.300 ft<sup>3</sup>  
 Condensate Increase in Impingers 198 ml 209.7  
 Moisture in Silica Gel 11.7 gm  
 Silica Gel Container No. 37 Filter No. 3-1254

Orsat: %CO <sub>2</sub>				
%O <sub>2</sub>				
%CO				
%N <sub>2</sub>				

Test Conducted By: R Paul  
D. Hubel

Stack Test Observers:     

Leak Check Meter Box Initial 0.00 cfm @ 15 In H<sub>2</sub>  
 Final 0.00 cfm @ 12 In Hg

Pitot Tubes Impact 3 In H<sub>2</sub>O for 15 sec. Stable, Leak  
 Static 3 In H<sub>2</sub>O for 15 sec. Stable, Leak

Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual					
1			87.3	.47	3.61	3.67	156	87	235	61	9
2			92.4	.52	4.06	4.06	154	87	241	53	10
3			97.7	.46	3.59	3.59	153	88	259	55	10
4			28	.46	3.59	3.59	151	89	257	54	10
5			7.8	.41	3.2	3.2	145	90	251	55	9
6			12.7	.41	3.2	3.2	145	91	260	54	9
1			17.3	.45	3.51	3.51	146	91	266	57	10
2			22.5	.50	3.9	3.9	139	91	247	54	12

Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual					
3			27.7	.48	3.75	3.75	139	91	257	55	12
4			33.0	.48	3.75	3.75	133	91	239	54	12
5			38.2	.51	3.98	3.98	133	92	245	55	12
6			63.7	.46	3.59	3.59	143	92	256	56	12

RK

# SAMPLING RATE

## CALCULATIONS

### S&K NOMOGRAPH

CO. NAME: \_\_\_\_\_

DATE: \_\_\_\_\_

STACK: \_\_\_\_\_

LOCATION: \_\_\_\_\_

$\Delta H$  = ORIFICE READING (INCHES  $H_2O$ )

$D_n$  = NOZZLE DIA. (INCHES)

$\Delta H_0$  = METER BOX CONSTANT

$B_w$  = MOISTURE FRACTION

$T_m$  = METER TEMP ( $^{\circ}F$ )

$T_s$  = STACK TEMP ( $^{\circ}F$ )

$M_s$  = WET MOLECULAR WEIGHT OF STACK GAS (FROM TABLE)

$\Delta P$  = PITOT READING (INCHES  $H_2O$ )

$$\left[ \frac{T_m + 460}{M_s(T_s + 460)} (1 - B_w)^2 \Delta H_0 (D_n)^4 17741 \right] \Delta P = \Delta H$$

MOISTURE FRACTION	$M_s$
0.0	29.0
0.05	28.5
0.10	27.9
0.15	27.4
0.20	26.8
0.25	26.2
0.30	25.7
0.35	25.2
0.40	24.6

$$\frac{533}{27.4} (593) (6248)$$

	RUN 1	RUN 2	RUN 3
$\frac{T_m + 460}{M_s(T_s + 460)} =$	0.032803	0.034094	
x $(1 - B_w)^2 =$	0.7225		
x $\Delta H_0 =$	1.83		
x $(D_n)^4 =$	0.009763		
x 17741 =	17741	17741	17741
x $\Delta P =$	7.512		

METER ORIFICE CHECK

Meter Box No. \_\_\_\_\_

Δ Ha of Box \_\_\_\_\_

Date \_\_\_\_\_

Time for 10 ft<sup>3</sup> = 13 min. 20 sec.

576.900  
10 : 13.33 total minutes = 0.75 Δ Ha<sub>2</sub>

$$\frac{0.75}{\Delta Ha_2} = 1.00 \times 100 = \underline{100\%}$$

RC Paul  
Signature

Procedure: Set flow rate of meter box at ΔHa of meter box--measure the amount of time required to sample 10.00 ft<sup>3</sup>.

Note: If answer is within 5 percent meter is o.k. If not, recalibrate meter.

NOZZLE CALIBRATION

Nozzle 5/14

Date 2-16+17-84

<u>Measurement No.</u>	<u>Inside Diameter (inches)</u>
<u>1</u>	<u>.314</u>
<u>2</u>	<u>.310</u>
<u>3</u>	<u>.315</u>

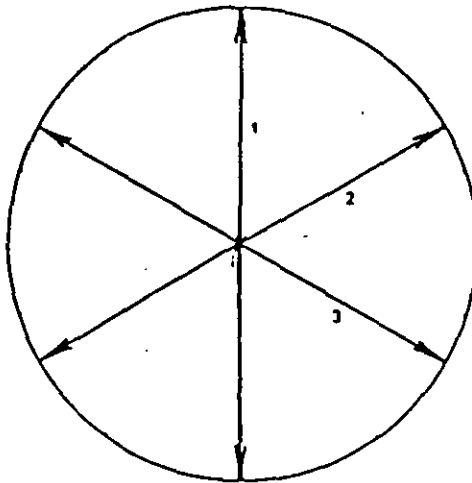
Average

.3143

Area of Nozzle

                     Ft<sup>2</sup>

Calibrated by: RC Paul



Nozzle X-section

SAMPLE CHAIN OF CUSTODY

PLANT NAME: Amax Chemical Corp.  
 SOURCE NAME: Big Bend Dryer  
 DATE: 2-17-84 TYPE OF SAMPLE: Particulate

SAMPLE RECOVERY

CONTAINER NO.	DESCRIPTION
<u>SK-41</u>	<u>Run 1 Oil Probe Wash</u>
<u>SIC-33</u>	<u>Run 2 " " "</u>
<u>SIC-301</u>	<u>Run 3 " " "</u>
<u>SK-45</u>	<u>Acetone Blank Oil</u>
<u>15</u>	<u>Run 1 Filter Oil</u>
<u>25</u>	<u>Run 2 " "</u>
<u>35</u>	<u>Run 3 " "</u>

Sample Recovered By: R. Paul - R.S. Sholtes  
 Particulate Analysis By: R. Paul

SHOLTES & KOOGLER, ENVIRONMENTAL CONSULTANTS, INC.  
PARTICULATE LAB DATA SHEET

Project No. AMAX - 01L  
2-17-84

	<u>Run 1</u>	<u>Run 2</u>	<u>Run 3</u>	<u>Blank</u>
Container No.	<u>16-41</u>	<u>16-33</u>	<u>16-301</u>	<u>16-45</u>
Total Volume (ml)	<u>203</u>	<u>183</u>	<u>226</u>	<u>100</u>
Aliquot Evaporated (ml)	<u>203</u>	<u>183</u>	<u>226</u>	<u>100</u>
Final Weight (g)	<u>93.8620</u>	<u>91.8932</u>	<u>101.1009</u>	<u>99.2636</u>
Tare Weight (g)	<u>-93.8490</u>	<u>-91.8782</u>	<u>-101.0913</u>	<u>-99.2634</u>
Gross Weight Gained (g)	<u>0.0130</u>	<u>0.0150</u>	<u>0.0096</u>	<u>0.0002</u>
Average Blank (g)	<u>-</u>	<u>-</u>	<u>-</u>	<u>-</u>
Net Weight (g)	<u>0.0130</u>	<u>0.0150</u>	<u>0.0096</u>	<u>0.0002</u>
Aliquot Factor	<u>x 10</u>	<u>x 10</u>	<u>x 1.0</u>	<u>x 1.0</u>
Total Net Weight (mg)	<u>13.0</u>	<u>15.0</u>	<u>9.6</u>	<u>0.2</u>

Container No.	<u>15</u>	<u>25</u>	<u>35</u>	<u>    </u>
Filter No.	<u>1252</u>	<u>1253</u>	<u>1254</u>	<u>    </u>
Final Weight (g)	<u>0.5455</u>	<u>0.5235</u>	<u>0.5312</u>	<u>    </u>
Tare Weight (g)	<u>-0.4535</u>	<u>-0.4518</u>	<u>-0.4524</u>	<u>-</u>
Gross Weight Gained (g)	<u>0.0920</u>	<u>0.0717</u>	<u>0.0788</u>	<u>    </u>
Average Blank	<u>-</u>	<u>-</u>	<u>-</u>	<u>-</u>
Total Net Weight (mg)	<u>92.0</u>	<u>71.7</u>	<u>78.8</u>	<u>    </u>

Tare Balance Check

0 0.0000 50g 50.0000  
 10g 10.0000 100g 100.0000  
 0.5g 0.5000

By R Paul  
 Date 2-22-84

Final Balance Check

0 0.0000 50g 50.0000  
 10g 10.0000 100g 100.0000  
 0.5g 0.5000

By R Paul  
 Date 2-23-84



APPENDIX A2

Field Data Sheets and Computations  
Sulfur Dioxide Sampling/Method 6

Run 1 2-16-84  
1045 Lm.

(Coal/Oil)

From Run 1 of Method 5  
1106-1210 Lm.  
Flow = 52612 SCFM

$$V_{m(\text{std})} = 17.4 \frac{0.700 \times 30.12}{(86+460)} = 0.673 \text{ ft}^3$$

$$C_{\text{SO}_2} = \frac{0.3989 \times 10^{-5} (0.5 - 0.3)}{0.673} = 0.119 \times 10^{-5} \text{ lb/dscf}$$

$$\text{Mass Emission} = 0.119 \times 10^{-5} \times 52,612 \times 60 = 3.76 \text{ lb/hr.}$$

Run 2 1140 Lm.

$$V_{m(\text{std})} = 17.4 \frac{0.849 \times 30.12}{(460+104)} = 0.789 \text{ ft}^3$$

$$C_{\text{SO}_2} = \frac{0.3989 \times 10^{-5} (1.55 - 0.3)}{0.789} = 0.632 \times 10^{-5} \text{ lb/dscf}$$

$$\text{Mass Emission} = 0.632 \times 10^{-5} \times 52,612 \times 60 = 19.95 \text{ lb/hr.}$$

Run 3 1230 Lm.

$$V_{m(\text{std})} = 17.4 \frac{1.065 \times 30.12}{(460+107)} = 0.984 \text{ ft}^3$$

$$C_{\text{SO}_2} = \frac{0.3989 \times 10^{-5} (3.3 - 0.3)}{0.984} = 1.216 \times 10^{-5} \text{ lb/dscf}$$

$$\text{Mass Emission} = 1.216 \times 10^{-5} \times 52,612 \times 60 = 38.39 \text{ lb/hr.}$$

$$\text{Avg Mass Emission} = 20.7 \text{ lb/hr SO}_2$$

From Run 1 of Method 5  
1617 - 1719  
Flow = 54051 SCFMD

2-16-84 Oil

Run 1 1610 hr

$$V_m(\text{std}) = 17.4 \frac{0.705 \times 30.12}{(460 + 94)} = 0.667 \text{ ft}^3$$

$$C_{\text{SO}_2} = \frac{0.3989 \times 10^{-5} (3.6 - .3)}{0.667} = 1.974 \times 10^{-5} \text{ lb/SCFD}$$

$$\text{Mass Emission} = 1.974 \times 10^{-5} \times 54051 \times 60 = 64.02 \text{ lb/hr SO}_2$$

Run 2 1645 hr

$$V_m(\text{std}) = 17.4 \frac{0.777 \times 30.12}{(460 + 106)} = 0.719 \text{ ft}^3$$

$$C_{\text{SO}_2} = \frac{0.3989 \times 10^{-5} (1.6 - .3)}{0.719} = 0.721 \times 10^{-5} \text{ lb/SCFD}$$

$$\text{Mass Emission} = 0.721 \times 10^{-5} \times 54,051 \times 60 = 23.38 \text{ lb/hr SO}_2$$

Run 3

~1700 hr.

$$V_m(\text{std}) = 17.4 \frac{0.935 \times 30.12}{(460 + 108)} = 0.863 \text{ ft}^3$$

$$C_{\text{SO}_2} = \frac{0.3989 \times 10^{-5} (4.55 - .3)}{0.863} = 1.964 \times 10^{-5} \text{ lb/SCFD}$$

$$\text{Mass Emission} = 1.964 \times 10^{-5} \times 54,051 \times 60 = 63.69 \text{ lb/hr SO}_2$$

$$\text{Average Mass Emission} = 50.36 \text{ lb/hr SO}_2$$

## Method 6 Computations

SO<sub>2</sub> in Stack Gases

From Method 6

$$C_{SO_2} = K_2 \frac{(V_t - V_{tb}) N \left( \frac{V_{std}}{V_a} \right)}{V_{m(std)}}$$

where  $K_2 = 7.061 \times 10^{-5}$  for English Units

$$V_a = 20 \text{ ml}$$

$$V_{std} = 100 \text{ ml.}$$

$$V_{tb} = 0.3$$

$$N = 10113 \quad \text{all constant}$$

$$C_{SO_2} = \frac{7.061 \times 10^{-5} (V_t - 0.3) (10113) \left( \frac{100}{20} \right)}{V_{m(std)}}$$

$$C_{SO_2} = \frac{0.3989 \times 10^{-5} (V_t - 0.3)}{V_{m(std)}} \quad \text{in } \frac{\text{lb}}{\text{dscf}}$$

$$V_{m(std)} = K_1 Y \frac{V_m P_{bar}}{T_m}$$

where  $K_1 = 17.64$  for English Units.

$$Y = 1,000$$

$$V_{m(std)} = 17.64 \frac{V_m P_{bar}}{T_m} \quad \text{in std ft}^3$$

# Fuel Consumption / Heat Release

Coal/Oil Mix

14,660 Btu/lb heat value

9.29 lb/gal @ 72F      8.61 lb/gal @ 122F

~~assume 8.5 lb/gal @ metered temp.~~ <sup>not metered</sup>

Given 600 gal/hr. @ 72F

Heat input =  $600 \times \frac{9.3}{9.29} \times 14,660 = 81.7 \times 10^6 \text{ Btu/hr.}$

#6 Oil

Heat Value given to be

~~18,357 Btu/lb.~~

148,000 Btu/gal

~~8.28 lb/gal~~

Oil Meter @ 1600 = 1391160

@ 1700 = 1391760

2/16/84

600 gal/hr.

@ 1700 = 1391760

@ 1800 = 1392210

450 gal/hr.

SO<sub>2</sub> Tests  
started 1610 &  
Rn thru 1730

Avg = 525 gal/hr.

Heat Release =  $77.7 \times 10^6 \text{ Btu/hr.}$



SOURCE SAMPLING FIELD DATA SHEET

Plant AMAX  
 Sampling Location Fluor Bed Dryer  
 Type of Control Scrubber  
 Type of Samples SO2  
 Date 2-16 Run No. 1 Com  
 Time Start 1045 Time End \_\_\_\_\_  
 Sample Time \_\_\_\_\_ min/pt \_\_\_\_\_ Total min  
 DB \_\_\_\_\_ °F, WB \_\_\_\_\_ °F, VP @ DP \_\_\_\_\_ "Hg  
 Bar. Press. 30.12 "Hg, Stack Press. \_\_\_\_\_ "Hg  
 Moisture \_\_\_\_\_ %, FDA \_\_\_\_\_, Gas Density Factor \_\_\_\_\_  
 Temp. \_\_\_\_\_ °F, W/D \_\_\_\_\_, W/S \_\_\_\_\_  
 Weather \_\_\_\_\_ Thermocouple Readout \_\_\_\_\_  
 Sample Box No. \_\_\_\_\_ meter Box No. \_\_\_\_\_  
 Meter ΔH0 \_\_\_\_\_ Pitot Corr. Factor \_\_\_\_\_  
 Nozzle Dia. \_\_\_\_\_ in., Probe Length \_\_\_\_\_ ft  
 Probe Heater Setting \_\_\_\_\_ Nomograph C<sub>f</sub> \_\_\_\_\_  
 Stack Dimensions \_\_\_\_\_ in  
 Stack Area \_\_\_\_\_ ft<sup>2</sup>  
 Effective Stack Area \_\_\_\_\_ ft<sup>2</sup>  
 Stack Height \_\_\_\_\_ ft

Method 6  
 Begin Purge  
 ED 1137

Stack Dimensions

Umbilical Cord 100' 200'

Mat'l Processing Rate \_\_\_\_\_  
 Final Gas Meter Reading 723.292 ft<sup>3</sup>  
 Initial Gas Meter Reading 722.592 ft<sup>3</sup> 0.7  
 Condensate Increase in Impingers \_\_\_\_\_ ml  
 Moisture in Silica Gel \_\_\_\_\_ gm  
 Silica Gel Container No. \_\_\_\_\_ Filter No. \_\_\_\_\_  
 Orsat: %CO<sub>2</sub> \_\_\_\_\_  
 %O<sub>2</sub> \_\_\_\_\_  
 %CO \_\_\_\_\_  
 %N<sub>2</sub> \_\_\_\_\_

Test Conducted By: R. Shalters

Stack Test Observers: \_\_\_\_\_

Leak Check Meter Box Initial 0.00 cfm @ NA In H<sub>2</sub>  
 Final 0.00 cfm @ 7 In Hg

Pitot Tubes Impact 3 In H<sub>2</sub>O for 15 sec. Stable, Leak  
 Static 3 In H<sub>2</sub>O for 15 sec. Stable, Leak

Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual					
		1045	722.592				130	86			1.8
		1050	22.695				130	80			1.8
		1055	22.782				130	82			2.2
		1100	22.880					23			2.0
		1105	22.992				130	87			2.2
		1110	23.078					90			2.2
		1115	23.182					92			2.2
		1120	23.292					94			2.2









SOURCE SAMPLING FIELD DATA SHEET

Plant AMAX  
 Sampling Location Fluid Bed Scrubber  
 Type of Control Scrubber  
 Type of Samples SO<sub>2</sub>  
 Date Run No. 1 Oil  
 Time Start 1610 Time End \_\_\_\_\_  
 Sample Time \_\_\_\_\_ min/pt Total min \_\_\_\_\_  
 DB \_\_\_\_\_ °F, WB \_\_\_\_\_ °F, VR @ DP \_\_\_\_\_ "Hg  
 Bar. Press. \_\_\_\_\_ "Hg, Stack Press. \_\_\_\_\_ "Hg  
 Moisture \_\_\_\_\_ %, FDA \_\_\_\_\_, Gas Density Factor \_\_\_\_\_  
 Temp. \_\_\_\_\_ °F, W/D \_\_\_\_\_, W/S \_\_\_\_\_  
 Weather \_\_\_\_\_ Thermocouple Readout \_\_\_\_\_  
 Sample Box No. \_\_\_\_\_ meter Box No. \_\_\_\_\_  
 Meter ΔH<sub>0</sub> \_\_\_\_\_ Pitot Corr. Factor \_\_\_\_\_  
 Nozzle Dia. \_\_\_\_\_ in., Probe Length \_\_\_\_\_ ft  
 Probe Heater Setting \_\_\_\_\_ Nomograph C<sub>f</sub> \_\_\_\_\_  
 Stack Dimensions \_\_\_\_\_ in  
 Stack Area \_\_\_\_\_ ft<sup>2</sup>  
 Effective Stack Area \_\_\_\_\_ ft<sup>2</sup>  
 Stack Height \_\_\_\_\_ ft

Method 6  
 Oil  
 St. @ 1631

Stack Dimensions  
 Umbilical Cord 100' 200'

Mat'l Processing Rate \_\_\_\_\_  
 Final Gas Meter Reading 727.915 ft<sup>3</sup>  
 Initial Gas Meter Reading 727.210 ft<sup>3</sup> 0.705  
 Condensate Increase in Impingers \_\_\_\_\_ ml  
 Moisture in Silica Gel \_\_\_\_\_ gm  
 Silica Gel Container No. \_\_\_\_\_ Filter No. \_\_\_\_\_  
 Orsat: %CO<sub>2</sub> \_\_\_\_\_  
 %O<sub>2</sub> \_\_\_\_\_  
 %CO \_\_\_\_\_  
 %N<sub>2</sub> \_\_\_\_\_

Test Conducted By: R. Shults

Stack Test Observers: \_\_\_\_\_

Leak Check Meter Box Initial 0.0 cfm @ 10 In H<sub>2</sub>  
 Final 0.0 cfm @ 10 In Hg

Pitot Tubes Impact 3 In H<sub>2</sub>O for 15 sec. Stable, Leak  
 Static 3 In H<sub>2</sub>O for 15 sec. Stable, Leak

Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> O)	Meter Orifice Press. Diff. ("H <sub>2</sub> O)		Stack Gas Temp. (°F)	Meter Temp. (°F)	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual					
		1610	27.210				94				
		1615	27.310				92				2.7
		1620	27.470				93				2.8
		1625	27.730				94				4.5
		1630	27.915				95				4.0
							96				4.0





SAMPLE CHAIN OF CUSTODY

PLANT NAME: Amex Chemical Corp.

SOURCE NAME: Big Bend Dryer.

DATE: 2-17-84 TYPE OF SAMPLE: SO<sub>2</sub> on Oil

SAMPLE RECOVERY

CONTAINER NO.

DESCRIPTION

<u>CONTAINER NO.</u>	<u>DESCRIPTION</u>
<u>Run 1</u>	<u>Method 6 on Oil</u>
<u>Run 2</u>	<u>" " "</u>
<u>Run 3</u>	<u>" " "</u>

Sample Recovered By: R. Sholtz

<sup>SO<sub>2</sub></sup> Particulate Analysis By: George Gabel

SO<sub>2</sub>  
LAB DATA

Plant Name AMAX OIL Date Analyzed 2-17-84  
 Analyzed By Geo. Seibel

Stack	Sample No.	V.T.	V.T.B.	N.	V.Soln.	V.A.
DRYER	Run 1	3.6	0.3	.0113	100	20
OIL	"	3.6				
	Run 2	1.6				
	"	1.6				
	Run 3	4.5				
		4.6	∇	∇		

- V.T.   ▪ Volume of Barium perchlorate titrant used for sample (ml)
- V.T.B. ▪ Volume of Barium perchlorate titrant used for blank (ml)
- N.     ▪ Normality of Barium perchlorate
- V.Soln. ▪ Total solution volume
- V.A.   ▪ Volume of sample aliquot titrated (ml)

SAMPLE CHAIN OF CUSTODY

PLANT NAME: Amax Chemical Corp.  
SOURCE NAME: Big Bend Dryer.  
DATE: 2-16-84 TYPE OF SAMPLE: SO<sub>2</sub> on Coal/Oil

SAMPLE RECOVERY

<u>CONTAINER NO.</u>	<u>DESCRIPTION</u>
<u>Run 1</u>	<u>Method 6 on coal/oil</u>
<u>Run 2</u>	<u>" " "</u>
<u>Run 3</u>	<u>" " "</u>
<u> </u>	<u> </u>
<u> </u>	<u> </u>
<u> </u>	<u> </u>
<u> </u>	<u> </u>
<u> </u>	<u> </u>
<u> </u>	<u> </u>
<u> </u>	<u> </u>
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Sample Recovered By: R. Sholtz  
<sup>SO<sub>2</sub></sup> Particulate Analysis By: George Gabel

SO<sub>2</sub>  
LAB DATA

Plant Name AMAX COAL/OIL Date Analyzed 2-17-84  
 Analyzed By Geo. Label

Stack	Sample No.	V.T.	V.T.B.	N.	V.Soln.	V.A.
DRYER	Run 1	0.5	0.3	.0113	100	20
COAL/OIL	"	0.5				
	Run 2	1.6				
	"	1.5				
✓	Run 3	3.3				
		3.3				
			↓	↓	↓	↓

- V.T. = Volume of Barium perchlorate titrant used for sample (ml)
- V.T.B. = Volume of Barium perchlorate titrant used for blank (ml)
- N. = Normality of Barium perchlorate.
- V.Soln. = Total solution volume
- V.A. = Volume of sample aliquot titrated (ml)

$K_1 = 0.00133 \text{ m}^3/\text{ml}$  for metric units  
 $K_1 = 0.04707 \text{ ft}^3/\text{gal}$  for English units

Note.—Equation 5-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the post-test leak check or leak checks conducted prior to component changes) exceeds  $L_c$ . If  $L_c$  or  $L_e$  exceeds  $L_c$ , Equation 5-1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace  $V_m$  in Equation 5-1 with the expression:

$$V_m - (L_p - L_e)\theta$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace  $V_m$  in Equation 5-1 by the expression:

$$V_m - (L_1 - L_e)\theta_1 - \sum_{i=2}^n (L_i - L_e)\theta_i - (L_p - L_e)\theta_p$$

and substitute only for those leakage rates ( $L_i$  or  $L_p$ ) which exceed  $L_c$ .

6.4 Volume of water vapor.

$$V_{w(sat)} = V_{1s} \left( \frac{P_w}{P_{atm}} \right) \left( \frac{RT_{sat}}{P_{atm}} \right) = K_2 V_{1s} \quad \text{Equation 5-2}$$

where:  
 $K_2 = 0.00133 \text{ m}^3/\text{ml}$  for metric units  
 $K_2 = 0.04707 \text{ ft}^3/\text{gal}$  for English units.

6.5 Moisture Content.

$$B_{w,s} = \frac{V_{w(sat)}}{V_m(sat) + V_{w(sat)}}$$

Equation 5-3

$$I = \frac{100 T_s [K_3 V_{1s} + (V_m/T_m) (P_{atm} + \Delta H/13.6)]}{60 \theta v_s P_s A_s} \quad \text{Equation 5-7}$$

where:  
 $K_3 = 0.00354 \text{ mm Hg} \cdot \text{m}^3/\text{ml} \cdot \text{K}$  for metric units.  
 $K_3 = 0.002660 \text{ in. Hg} \cdot \text{ft}^3/\text{gal} \cdot \text{K}$  for English units.

6.11.2 Calculation From Intermediate Values.

$$I = \frac{T_s V_m(sat) P_{sat} 100}{T_{sat} v_s \theta A_s P_s 60 (1 - B_{w,s})}$$

$$= K_4 \frac{T_s V_m(sat)}{P_s V_s A_s \theta (1 - B_{w,s})}$$

Equation 5-8

where:  
 $K_4 = 4.320$  for metric units  
 $K_4 = 0.04450$  for English units.

6.12 Acceptable Results. If 80 percent  $\leq I \leq 110$  percent, the results are acceptable. If the results are low in comparison to the standard and  $I$  is beyond the acceptable range, or if  $I$  is less than 80 percent, the Administrator may opt to accept the results. Use Citation 4 to make judgments. Otherwise, reject the results and repeat the test.

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Note.—In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (Equation 5-3), and a second from the assumption of saturated conditions. The lower of the two values of  $B_{w,s}$  shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the Note of Section 1.2 of Method 4. For the purposes of this method, the average stack gas temperature from Figure 5-2 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is  $\pm 1^\circ \text{C}$  ( $2^\circ \text{F}$ ).

6.6 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a \rho_a}$$

Equation 5-4

6.7 Acetone Wash Blank.

$$W_a = C_a V_w \rho_w$$

Equation 5-5

6.8 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from containers 1 and 2 less the acetone blank (see Figure 5-2). Note.—Refer to Section 4.1.5 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

6.9 Particulate Concentration.

$$c_p = (0.001 \text{ g/mg}) (m_p/V_m(sat))$$

Equation 5-6

6.10 Conversion Factors:

From	To	Multiply by
cc/ft <sup>3</sup>	m <sup>3</sup>	0.02832
g/ft <sup>3</sup>	g/ft <sup>3</sup>	15.43
g/ft <sup>3</sup>	lb/ft <sup>3</sup>	2.205 × 10 <sup>-3</sup>
g/ft <sup>3</sup>	g/m <sup>3</sup>	25.31

6.11 Isokinetic Variation.

6.11.1 Calculation From Raw Data.

8. Vellaro, R. F. A Survey of Commercially Available Instrumentation For the Measurement of Low-Range Gas Velocities. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, N.C. November, 1974 (unpublished paper).
9. Annual Book of ASTM Standards, Part 26, Gaseous Fuels; Coal and Coke; Atmospheric Analysis. American Society for Testing and Materials, Philadelphia, Pa. 1974, pp. 617-622.

#### METHOD 6—DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

##### 1. Principles and Applicability

1.1 Principle. A gas sample is extracted from the sampling point in the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated. The sulfur dioxide fraction is measured by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfur dioxide emissions from stationary sources. The minimum detectable limit of the method has been determined to be 3.4 milligrams (mg) of SO<sub>2</sub>/m<sup>3</sup> (2.12 × 10<sup>-4</sup> lb/ft<sup>3</sup>). Although no upper limit has been established, tests have shown that concentrations as high as 80,000 mg/m<sup>3</sup> of SO<sub>2</sub> can be collected efficiently in two midjet impingers, each containing 15 milliliters of 3 percent hydrogen peroxide, at a rate of 1.0 lpm for 20 minutes. Based on theoretical calculations, the upper concentration limit in a 20-liter sample is about 93,300 mg/ft<sup>3</sup>.

Possible interferences are free ammonia, water-soluble cations, and fluorides. The cations and fluorides are removed by glass wool filters and an isopropanol bubbler, and hexos do not affect the SO<sub>2</sub> analysis. When samples are being taken from a gas stream with high concentrations of very fine metallic fumes (such as in inlets to control devices), a high-efficiency glass fiber filter must be used in place of the glass wool plug (i.e., the one in the probe) to remove the cation interferences.

Free ammonia interferences by reacting with SO<sub>2</sub> to form particulate sulfate and by reacting with the indicator. If free ammonia is present (this can be determined by knowledge of the process and noticing white particulate matter in the probe and isopropanol bubbler), alternative methods, subject to the approval of the Administrator, U.S. Environmental Protection Agency, are required.

##### 2. Apparatus



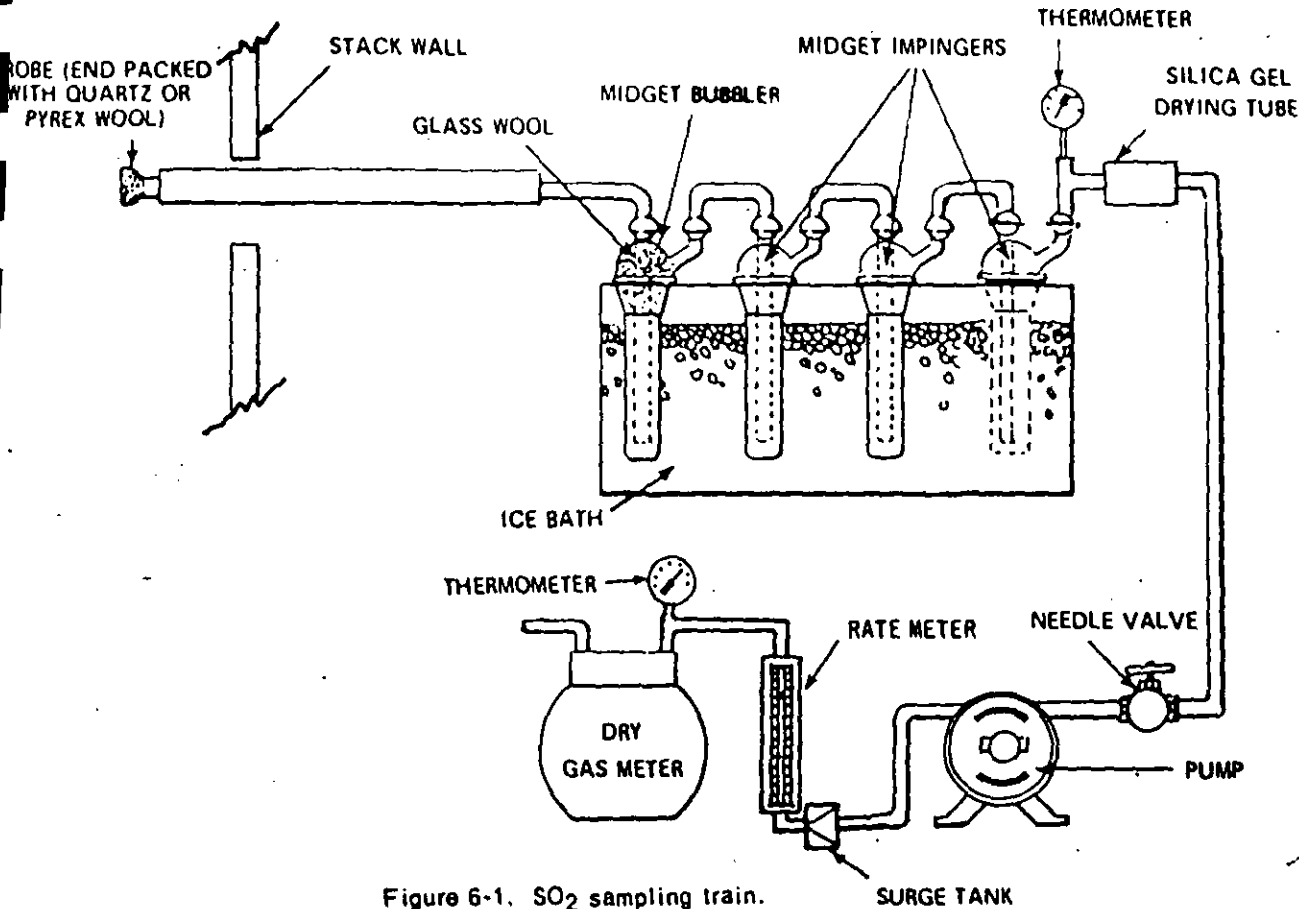


Figure 6-1. SO<sub>2</sub> sampling train.

**2.1 Sampling.** The sampling train is shown in Figure 6-1, and component parts are discussed below. The tester has the option of substituting sampling equipment described in Method 5 in place of the midget impinger equipment of Method 6. However, the Method 6 train must be modified to include a heated filter between the probe and isopropanol impinger, and the operation of the sampling train and sample analysis must be at the flow rates and solution volumes defined in Method 6. The tester also has the option of determining SO<sub>2</sub> simultaneously with particulate matter and moisture determinations by (1) replacing the water in a Method 5 impinger system with 3 percent peroxide solution, or (2) by replacing the Method 5 water impinger system with a Method 5 isopropanol-filter-peroxide system. The analysis for SO<sub>2</sub> must be consistent with the procedure in Method 6.

**2.1.1 Probe.** Borosilicate glass, or stainless steel (other materials of construction may be used, subject to the approval of the Administrator), approximately 6-mm inside diameter, with a heating system to prevent water condensation and a filter (either in-stack or heated outside) to remove particulate matter, including sulfuric acid mist. A plug of glass wool is a satisfactory filter.

**2.1.2 Bubbler and Impingers.** One midget bubbler, with medium-coarse glass frit and borosilicate or quartz glass wool packed in top (see Figure 6-1) to prevent sulfuric acid mist carryover, and three 80-ml midget impingers. The bubbler and midget impingers must be connected in series with leak-free glass connectors. Silicone grease may be used, if necessary, to prevent leakage. At the option of the tester, a midget impinger may be used in place of the midget bubbler.

Other collection absorbers and flow rates may be used, but are subject to the approval of the Administrator. Also, collection efficiency must be shown to be at least 90 percent for each test run and must be documented in the report. If the efficiency is found to be acceptable after a series of three tests, further documentation is not required. To conduct the efficiency test, an extra absorber must be added and analyzed separately. This extra absorber must not contain more than 1 percent of the total SO<sub>2</sub>.

**2.1.3 Glass Wool.** Borosilicate or quartz.

**2.1.4 Stopcock Grease.** Acetone-insoluble, heat-stable silicone grease may be used, if necessary.

**2.1.5 Temperature Gauge.** Dial thermometer, or equivalent, to measure temperature of gas leaving impinger train to within 1° C (2° F.).

**2.1.6 Drying Tube.** Tube packed with 4- to 14-mesh indicating type silica gel, or equivalent, to dry the gas

sample and to protect the meter and pump. If the silica gel has been used previously, dry at 176° C (350° F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to approval of the Administrator.

**2.1.7 Valve.** Needle valve, to regulate sample gas flow rate.

**2.1.8 Pump.** Leak-free diaphragm pump, or equivalent, to pull gas through the train. Install a small tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

**2.1.9 Rate Meter.** Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of about 1000 cc/min.

**2.1.10 Volume Meter.** Dry gas meter, sufficiently accurate to measure the sample volume within 2 percent, calibrated at the selected flow rate and conditions actually encountered during sampling, and equipped with a temperature gauge (dial thermometer, or equivalent) capable of measuring temperature to within 2° C (3.6° F.).

**2.1.11 Barometer.** Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 80 m (100 ft) elevation increase or vice versa for elevation decrease.

**2.1.12 Vacuum Gauge.** At least 760 mm Hg (30 in. Hg) gauge, to be used for leak check of the sampling train.

**2.2 Sample Recovery.**

**2.2.1 Wash bottles.** Polyethylene or glass, 500 ml, two.

**2.2.2 Storage Bottles.** Polyethylene, 100 ml, to store impinger samples (one per sample).

**2.3 Analysis.**

**2.3.1 Pipettes.** Volumetric type, 5-ml, 20-ml (one per sample), and 25-ml sizes.

**2.3.2 Volumetric Flasks.** 100-ml size (one per sample) and 100-ml size.

**2.3.3 Burettes.** 5- and 50-ml sizes.

**2.3.4 Erlenmeyer Flasks.** 350-ml size (one for each sample, blank, and standard).

**2.3.5 Dropping Bottle.** 125-ml size, to add indicator.

**2.3.6 Graduated Cylinder.** 100-ml size.

**2.3.7 Spectrophotometer.** To measure absorbance at 352 nanometers.

**3. Reagents**

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

**3.1 Sampling.**

**3.1.1 Water.** Deionized, distilled to conform to ASTM specification D1193-74, Type 2. At the option of the analyst, the KMnO<sub>4</sub> test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

**3.1.2 Isopropanol, 90 Percent.** Mix 80 ml of isopropanol with 20 ml of deionized, distilled water. Check each lot of isopropanol for peroxide impurities as follows: shake 10 ml of isopropanol with 10 ml of freshly prepared 10 percent potassium iodide solution. Prepare a blank by similarly treating 10 ml of distilled water. After 1 minute, read the absorbance at 352 nanometers on a spectrophotometer. If absorbance exceeds 0.1, reject alcohol for use.

Peroxides may be removed from isopropanol by redistilling or by passage through a column of activated alumina; however, reagent grade isopropanol with suitably low peroxide levels may be obtained from commercial sources. Rejection of contaminated lots may, therefore, be a more efficient procedure.

**3.1.3 Hydrogen Peroxide, 3 Percent.** Dilute 30 percent hydrogen peroxide 1:9 (v/v) with deionized, distilled water (80 ml is needed per sample). Prepare fresh daily.

**3.1.4 Potassium Iodide Solution, 10 Percent.** Dissolve 10.0 grams KI in deionized, distilled water and dilute to 100 ml. Prepare when needed.

**3.2 Sample Recovery.**

**3.2.1 Water.** Deionized, distilled, as in 3.1.1.

**3.2.2 Isopropanol, 90 Percent.** Mix 80 ml of isopropanol with 20 ml of deionized, distilled water.

**3.3 Analysis.**

**3.3.1 Water.** Deionized, distilled, as in 3.1.1.

**3.3.2 Isopropanol, 100 Percent.**

**3.3.3 Thorin Indicator.** 1-(6-aminophenylazo)-3-naphthol-3,6-disulfonic acid, diacidum salt, or equivalent. Dissolve 0.20 g in 100 ml of deionized, distilled water.

**3.3.4 Barium Perchlorate Solution, 0.0100 N.** Dissolve 1.95 g of barium perchlorate trihydrate [Ba(ClO<sub>4</sub>)<sub>2</sub>·3H<sub>2</sub>O] in 200 ml distilled water and dilute to 1 liter with isopropanol. Alternatively, 1.22 g of [BaCl<sub>2</sub>·2H<sub>2</sub>O] may be used instead of the perchlorate. Standardize as in Section 5.5.

1.5 Sulfuric Acid Standard, 0.0100 N. Purchase or standardize to  $\pm 0.0002$  N against 0.0100 N NaOH which has previously been standardized against potassium acid phthalate (primary standard grade).

#### 4. Procedure.

##### 4.1 Sampling.

4.1.1 Preparation of collection train. Measure 15 ml of 50 percent isopropanol into the midjet bubbler and 15 ml of 5 percent hydrogen peroxide into each of the first two midjet impingers. Leave the final midjet impinger dry. Assemble the train as shown in Figure 6-1. Adjust probe heater to a temperature sufficient to prevent water condensation. Place crushed ice and water around the impingers.

4.1.2 Leak-check procedure. A leak check prior to the sampling run is optional; however, a leak check after the sampling run is mandatory. The leak-check procedure is as follows:

With the probe disconnected, place a vacuum gauge at the inlet to the bubbler and pull a vacuum of 150 mm (10 in.) Hg; plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum shall remain stable for at least 30 seconds. Carefully release the vacuum gauge before releasing the flow meter and to prevent back flow of the impinger fluid.

Other leak-check procedures may be used, subject to the approval of the Administrator, U.S. Environmental Protection Agency. The procedure used in Method 5 is not suitable for diaphragm pumps.

4.1.3 Sample collection. Record the initial dry gas meter reading and barometric pressure. To begin sampling, position the tip of the probe at the sampling point, connect the probe to the bubbler, and start the pump. Adjust the sample flow to a constant rate of approximately 1.0 liter/min as indicated by the rotameter. Maintain this constant rate ( $\pm 10$  percent) during the entire sampling run. Take readings (dry gas meter, temperatures at dry gas meter and at impinger outlet and rate meter) at least every 5 minutes. Add more ice during the run to keep the temperature of the gases leaving the last impinger at  $20^\circ\text{C}$  ( $68^\circ\text{F}$ ) or less. At the conclusion of each run, turn off the pump, remove probe from the stack, and record the final readings. Conduct a leak check as in Section 4.1.2. (This leak check is mandatory.) If a leak is found, void the test run. Drain the ice bath, and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes at the sampling rate.

Clean ambient air can be provided by passing air through a charcoal filter or through an extra midjet impinger with 15 ml of 5 percent  $\text{H}_2\text{O}_2$ . The latter may opt to simply use ambient air, without purification.

4.1.4 Sample Recovery. Disconnect the impingers after purging. Discard the contents of the midjet bubbler. Pour the contents of the midjet impingers into a leak-free polyethylene bottle for shipment. Rinse the three midjet impingers and the connecting tubes with deionized, distilled water, and add the washings to the same storage container. Mark the fluid level, seal and identify the sample container.

4.1.5 Sample Analysis. Note level of liquid in container, and confirm whether any sample was lost during shipment; note this on analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

Transfer the contents of the storage container to a 100-ml volumetric flask and dilute to exactly 100 ml with deionized, distilled water. Pipette a 20-ml aliquot of this solution into a 250-ml Erlenmeyer flask, add 90 ml of 100 percent isopropanol and two to four drops of thion indicator, and titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat and average the titration volumes. Run a blank with each series of samples. Replicate titrations must agree within 1 percent or 0.5 ml, whichever is larger.

(NOTE.—Protect the 0.0100 N barium perchlorate solution from evaporation at all times.)

#### 4. Calibration

##### 4.1 Metering System.

4.1.1 Initial Calibration. Before its initial use in the field, first leak check the metering system (drying tube, paddle valve, pump, rotameter, and dry gas meter) as

follows: place a vacuum gauge at the inlet to the drying tube and pull a vacuum of 150 mm (10 in.) Hg; plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum shall remain stable for at least 30 seconds. Carefully release the vacuum gauge before releasing the flow meter end.

Next, calibrate the metering system (at the sampling flow rate specified by the method) as follows: connect an appropriately sized wet test meter (e.g., 1 liter per revolution) to the inlet of the drying tube. Make three independent calibration runs, using at least five revolutions of the dry gas meter per run. Calculate the calibration factor,  $Y$  (wet test meter calibration volume divided by the dry gas meter volume, both volumes adjusted to the same reference temperature and pressure), for each run, and average the results. If any  $Y$  value deviates by more than 2 percent from the average, the metering system is unacceptable for use. Otherwise, use the average as the calibration factor for subsequent test runs.

4.1.2 Post-Test Calibration Check. After each field test series, conduct a calibration check as in Section 4.1.1 above, except for the following variations: (a) the leak check is not to be conducted, (b) three, or more revolutions of the dry gas meter may be used, and (c) only two independent runs need be made. If the calibration factor does not deviate by more than 5 percent from the initial calibration factor (determined in Section 4.1.1), then the dry gas meter volumes obtained during the test series are acceptable. If the calibration factor deviates by more than 5 percent, recalibrate the metering system as in Section 4.1.1, and for the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

4.2 Thermometers. Calibrate against mercury-in-glass thermometers.

4.3 Rotameter. The rotameter need not be calibrated but should be cleaned and maintained according to the manufacturer's instruction.

4.4 Barometer. Calibrate against a mercury barometer.

4.5 Barium Perchlorate Solution. Standardize the barium perchlorate solution against 25 ml of standard sulfuric acid to which 100 ml of 100 percent isopropanol has been added.

#### 6. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the required data. Round off figures after final calculation.

##### 6.1 Nomenclature.

$C_s$ —Concentration of sulfur dioxide, dry basis corrected to standard conditions, mg/dscm (lb/dscf).

$N$ —Normality of barium perchlorate titrant, milliequivalents/ml.

$P_{bar}$ —Barometric pressure at the exit orifice of the dry gas meter, mm Hg (in. Hg).

$P_{std}$ —Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

$T_m$ —Average dry gas meter absolute temperature,  $^\circ\text{K}$  ( $^\circ\text{C}$ ).

$T_{std}$ —Standard absolute temperature,  $293^\circ\text{K}$  ( $20^\circ\text{C}$ ).

$V_s$ —Volume of sample aliquot titrated, ml.

$V_m$ —Dry gas volume as measured by the dry gas meter, dcm (dcf).

$V_{m(std)}$ —Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dcf).

$V_{sol}$ —Total volume of solution in which the sulfur dioxide sample is contained, 100 ml.

$V_t$ —Volume of barium perchlorate titrant used for the sample, ml (average of replicate titrations).

$V_{bl}$ —Volume of barium perchlorate titrant used for the blank, ml.

$Y$ —Dry gas meter calibration factor.

32.03—Equivalent weight of sulfur dioxide.

6.2 Dry sample gas volume, corrected to standard conditions.

$$V_{m(std)} = V_m Y \left( \frac{T_{std}}{T_m} \right) \left( \frac{P_{bar}}{P_{std}} \right) = K_1 Y \frac{V_m P_{bar}}{T_m}$$

Equation 6-1

where:

$K_1 = 0.2838^\circ\text{K}/\text{mm Hg}$  for metric units.

$= 17.64^\circ\text{R}/\text{in. Hg}$  for English units.

6.3 Sulfur dioxide concentration.

$$C_{SO_2} = K_2 \frac{(V_t - V_{bl}) N \left( \frac{V_{sol}}{V_{std}} \right)}{V_{m(std)}}$$

Equation 6-2

where:

$K_2 = 32.03$  mg/meq. for metric units.

$= 17.681 \times 10^{-4}$  lb/meq. for English units.

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#### METHOD 7—DETERMINATION OF NITROGEN OXIDE EMISSIONS FROM STATIONARY SOURCES

##### 1. Principle and Applicability

1.1 Principle. A grab sample is collected in an evacuated flask containing a dilute sulfuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except nitrous oxide, are measured colorimetrically using the phenoldisulfonic acid (PDS) procedure.

1.2 Applicability. This method is applicable to the measurement of nitrogen oxides emitted from stationary sources. The range of the method has been determined to be 2 to 400 milligrams  $\text{NO}_x$  (as  $\text{NO}_2$ ) per dry standard cubic meter, without having to dilute the sample.

##### 2. Apparatus

2.1 Sampling (see Figure 7-1). Other grab sampling systems or equipment, capable of measuring sample volume to within  $\pm 2.0$  percent and collecting a sufficient sample volume to allow analytical reproducibility to within  $\pm 5$  percent, will be considered acceptable alternatives, subject to approval of the Administrator, U.S. Environmental Protection Agency. The following equipment is used in sampling:

2.1.1 Probe. Borosilicate glass tubing, sufficiently heated to prevent water condensation and equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Stainless steel or Teflon tubing may also be used for the probe. Heating is not necessary if the probe remains dry during the purging period.

\* Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

APPENDIX A3

Field Data Sheets and Computations  
Nitrogen Oxide Sampling/Method 7

## NO<sub>x</sub> Mass Emissions

Coal/Oil Sample time 1315 thru 1600 on 2/16/84

$$\text{Average Concentration} = 8.968 \times 10^{-6} \text{ lb/scf.}$$

Particulate Runs 1, 2 & 3 span this time period.

$$\text{Avg Flow} = 52220 \text{ SCFMD.}$$

$$\begin{aligned} \text{NO}_x \text{ Mass Emission} &= 8.968 \times 10^{-6} \times 52,220 \times 60 \\ &= 28.1 \text{ lb/hr.} \end{aligned}$$

Oil

Sample time 0830 thru 1115 on 2/17/84

$$\text{Average Concentration} = 6.080 \times 10^{-6} \text{ lb/scf.}$$

Particulate Runs 2 & 3 span this time period

$$\text{Average Flow} = 53398 \text{ SCFMD}$$

$$\begin{aligned} \text{NO}_x \text{ Mass Emission} &= 6.080 \times 10^{-6} \times 53398 \times 60 \\ &= 19.5 \text{ lb/hr.} \end{aligned}$$

PLANTS: 925 N. Lane Avenue Jacksonville, Florida 32201 904-786-0750  
Stephens Drive Nicholasville, Kentucky 40356 606-885-6011

Coal/oil

$$8.968 \times 10^{-6} \text{ lb/scf} \quad 75.4 \text{ ppm} \\ = 28.1 \text{ lb/hr}$$

oil

$$6.080 \times 10^{-6} \text{ lb/scf} \quad 51.1 \\ = 19.5 \text{ lb/hr}$$

as  $\text{NO}_2$

$$\text{lb/scf} \times \frac{\text{scf}}{\text{hr}}$$

James G. Nolan  
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P. O. Box 20283  
Louisville, Ky. 40220



## TABLE

NO<sub>x</sub>

## EMISSION DATA

PLANT AMAX Fluid Bed Dryer  
 STACK Coal/Oil

Bar 30.12

Run No.	1	2	3	4
Date	2-16-84	2-16	2-16	2-16
Time	1315	1330	1345	1400
Flask No.	28	38	39	44
V <sub>f</sub> = Flask + Valve Volume, ml	2090	2026	2039	2017
V <sub>a</sub> = Absorbing Soln. Volume, vl	25	25	25	25
T <sub>i</sub> = Initial Flask Temp., °F	78	77	79	79
T <sub>f</sub> = Final Flask Temp., °F	63	63	63	63
P <sub>i</sub> = Initial Flask Vacuum, "Hg >	28.3"	28.3	28.2	28.0
P <sub>f</sub> = Final Flask Vacuum, "Hg	.2	0	0	0
V <sub>stpd</sub> = Gas Sample Volume, ml*	1939.5	1892.7	1898.8	1865.0
m = Mass of NO <sub>2</sub> in Gas Sample, µg	285.0	285.0	349.1	270.8
NO <sub>2</sub> Concentration, lbs/scf	9.174 E-6	9.401 E-6	11.478 E-6	9.065 E-6
NO <sub>2</sub> Concentration, ppm	77.12	79.02	96.48	76.20

\*Dry, 70°F, 29.92"Hg

$$V_{stpd} = 17.64 \times (V_f - V_a) \times \left[ \frac{P_{bf} - P_f}{T_f} - \frac{P_{bi} - P_i}{T_i} \right], \text{ when } P_{bf} \text{ \& } P_{bi} =$$

the final and initial barometric pressures.

$$\text{NO}_2 \text{ (lbs/scf)} = 6.243 \times \left( \frac{m}{V_{stpd}} \right) \times 10^{-5}$$

$$\text{NO}_2 \text{ (ppm)} = 8.406 \times 10^6 \times \frac{\text{lbs}}{\text{ft}^3} \text{ NO}_2$$

# SK

TABLE

NO<sub>x</sub>

EMISSION DATA

PLANT AMAX

STACK \_\_\_\_\_

~~Barometer 30.18~~ 30.12

Run No.	9	10	11	12
Date	2/16/84	2/16/84	2/16/84	2/16/84
Time	1515	1530	1545	1600
Flask No.	32	31	40	36
V <sub>f</sub> = Flask + Valve Volume, ml	2079	2080	2017	2117
V <sub>a</sub> = Absorbing Soln. Volume, vl	25	25	25	25
T <sub>i</sub> = Initial Flask Temp., °F	80	84	89	83
T <sub>f</sub> = Final Flask Temp., °F	62	62	62	62
P <sub>i</sub> = Initial Flask Vacuum, "Hg	28.2	27.0	28.1	27.7
P <sub>f</sub> = Final Flask Vacuum, "Hg	-0.1	-0.4	-0.7	-0.2
V <sub>stpd</sub> = Gas Sample Volume, ml*	1933.8	1834.8	1830.6	1929.2
m = Mass of NO <sub>2</sub> in Gas Sample, µg	256.5	277.9	257.9	327.8
NO <sub>2</sub> Concentration, lbs/scf	8.281 E-6	9.456 E-6	8.795 E-6	10.607 E-6
NO <sub>2</sub> Concentration, ppm	69.61	79.48	73.93	89.17

\*Dry, 70°F, 29.92"Hg

FINAL-PB-29.80

$$V_{stpd} = 17.64 \times (V_f - V_a) \times \left[ \frac{P_{bf} - P_f}{T_f} - \frac{P_{bi} - P_i}{T_i} \right], \text{ when } P_{bf} \text{ \& } P_{bi} =$$

the final and initial barometric pressures.

$$NO_2 \text{ (lbs/scf)} = 6.243 \times \left( \frac{m}{V_{stpd}} \right) \times 10^{-5}$$

$$NO_2 \text{ (ppm)} = 8.406 \times 10^6 \times \left( \frac{\text{lbs}}{\text{ft}^3} \right) NO_2$$



## TABLE

NO<sub>x</sub>  
EMISSION DATABarometric 2/17/84  
30.12PLANT AMAX  
STACK Scrubber Oil  
2/17/84 Bar = 30.10

Run No.	1	2	3	4
Date	2/17/84	2/17	2/17	2/17
Time	0830	0845	0900	0915
Flask No.	10	5	21	24
V <sub>f</sub> = Flask + Valve Volume, ml	2047	2024	2078	2083
V <sub>a</sub> = Absorbing Soln. Volume, vl	25	25	25	25
T <sub>i</sub> = Initial Flask Temp., °F	65	61	60	60
T <sub>f</sub> = Final Flask Temp., °F	63	63	63	63
P <sub>i</sub> = Initial Flask Vacuum, "Hg	28.5	27.8	28.0	28.5
P <sub>f</sub> = Final Flask Vacuum, "Hg	-1	0	-12	0
V <sub>stpd</sub> = Gas Sample Volume, ml*	1917.9	1854.6	1904.5	1957.9
m = Mass of NO <sub>2</sub> in Gas Sample, µg	185.3	175.3	153.9	213.8
NO <sub>2</sub> Concentration, lbs/scf	6.032 E-6	5.901 E-6	5.045 E-6	6.817 E-6
NO <sub>2</sub> Concentration, ppm	50.70	49.60	42.41	57.31

\*Dry, 70°F, 29.92"Hg

$$V_{stpd} = 17.64 \times (V_f - V_a) \times \left[ \frac{P_{bf} - P_f}{T_f} - \frac{P_{bi} - P_i}{T_i} \right], \text{ when } P_{bf} \text{ \& } P_{bi} =$$

the final and initial barometric pressures.

$$\text{NO}_2 \text{ (lbs/scf)} = 6.243 \times \left( \frac{m}{V_{stpd}} \right) \times 10^{-5}$$

$$\text{NO}_2 \text{ (ppm)} = 8.406 \times 10^6 \times \frac{\text{lbs}}{\text{ft}^3} \text{ NO}_2$$





## TABLE

NO<sub>x</sub>

## EMISSION DATA

PLANT AMAX  
 STACK Scrubber 0:1

Bar 30.10

Run No.	5	6	7	8
Date	2/17/67	2/17	2/17	2/17
Time	0930	0945	1000	1015
Flask No.	43	42	25	27
V <sub>f</sub> = Flask + Valve Volume, ml	2051	2026	2076	2107
V <sub>a</sub> = Absorbing Soln. Volume, vl	25	25	25	25
T <sub>i</sub> = Initial Flask Temp., °F >	67	69	73	74
T <sub>f</sub> = Final Flask Temp., °F	63	64	64	64
P <sub>i</sub> = Initial Flask Vacuum, "Hg >	28.3	28.4	28.3	28.3
P <sub>f</sub> = Final Flask Vacuum, "Hg	- .5	- 1.1	- 1.1	- .4
V <sub>stpd</sub> = Gas Sample Volume, ml*	1881.2	1820.9	1860.5	1937.9
m = Mass of NO <sub>2</sub> in Gas Sample, μg	188.1	186.7	216.6	183.8
NO <sub>2</sub> Concentration, lbs/scf	6.242 E-6	6.401 E-6	7.268 E-6	5.921 E-6
NO <sub>2</sub> Concentration, ppm	52.47	53.81	61.10	49.77

\*Dry, 70°F, 29.92"Hg

$V_{stpd} = 17.64 \times (V_f - V_a) \times \left[ \frac{P_{bf} - P_f}{T_f} - \frac{P_{bi} - P_i}{T_i} \right]$ , when P<sub>bf</sub> & P<sub>bi</sub> =  
 the final and initial barometric pressures.

$$NO_2 \text{ (lbs/scf)} = 6.243 \times \left( \frac{m}{V_{stpd}} \right) \times 10^{-5}$$

$$NO_2 \text{ (ppm)} = 8.406 \times 10^6 \times \frac{\text{lbs}}{\text{ft}^3} NO_2$$



TABLE  
NO<sub>x</sub>  
EMISSION DATA

PLANT AMAX  
STACK Scrubber Oil

Bar 30.10

Run No.	9	10	11	12
Date	2/17/84	2/17	2/17	2/17
Time	1030	1045	1100	1115
Flask No.	3	13	4	15
V <sub>f</sub> = Flask + Valve Volume, ml	2013	2076	2051	2075
V <sub>a</sub> = Absorbing Soln. Volume, vl	25	25	25	25
T <sub>i</sub> = Initial Flask Temp., °F	69	76	76	76
T <sub>f</sub> = Final Flask Temp., °F	64	64	63	62
P <sub>i</sub> = Initial Flask Vacuum, "Hg	28.2	28.4	28.2	28.2
P <sub>f</sub> = Final Flask Vacuum, "Hg	-0.4	-0.7	-0.6	-0.5
V <sub>stpd</sub> = Gas Sample Volume, ml*	1842.7	1895.5	1869.7	1902.7
m = Mass of NO <sub>2</sub> in Gas Sample, µg	142.5	185.3	175.3	199.5
NO <sub>2</sub> Concentration, lbs/scf	4.828 E-6	6.103 E-6	5.853 E-6	6.546 E-6
NO <sub>2</sub> Concentration, ppm	40.58	51.30	49.30	55.03

\*Dry, 70°F, 29.92"Hg

$$V_{stpd} = 17.64 \times (V_f - V_a) \times \left[ \frac{P_{bf} - P_f}{T_f} - \frac{P_{bi} - P_i}{T_i} \right], \text{ when } P_{bf} \text{ \& } P_{bi} =$$

the final and initial barometric pressures.

$$\text{NO}_2 \text{ (lbs/scf)} = 6.243 \times \left( \frac{m}{V_{stpd}} \right) \times 10^{-5}$$

$$\text{NO}_2 \text{ (ppm)} = 8.406 \times 10^6 \times \frac{\text{lbs NO}_2}{\text{ft}^3}$$

NO<sub>x</sub> LABORATORY DATA

LAB. NO. 5

DATE 2-22-84

SAMPLE SOURCE \_\_\_\_\_

SAMPLE NUMBER	ABSORBANCE 410 mu	VOLUME ml	ALIQOT - DILUTION ml ml	DILUTION FACTOR	TOTAL ug
3	0.100	100	50 - -	2	142.5
4	0.123				175.3
5	0.123				175.3
10	0.130				185.3
15	0.140				199.5
21	0.108				153.9
24	0.150				213.8
25	0.152				216.6
27	0.129				183.8
28	0.200				285.0
30	0.172				245.1
32	0.180				256.5
31	0.195				277.9
33	0.155				220.9
36	0.230				327.8
38	0.200				285.0
39	0.245				349.1
40	0.181				257.9
41	0.170				242.3
STANDARDS					
1	.135	$K_C = \frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2} (100)$ $K_C = \frac{712.5}{}$			100
2	.275				200
3	.420				300
4	.565				400

ANALYST M. Pisman

NO<sub>x</sub> LABORATORY DATA

LAB. NO. \_\_\_\_\_

DATE 2-22-84

SAMPLE SOURCE \_\_\_\_\_

SAMPLE NUMBER	ABSORBANCE 410 mu	VOLUME ml	ALIQOT - DILUTION		DILUTION FACTOR	TOTAL ug
			ml	ml		
35	0.165	100	50	-	2	235.1
42	0.131					186.7
43	0.132					188.1
44	0.190					270.8
13	0.130					185.3
STANDARDS						
1	.135	$K_C = \frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2} (100)$ $K_C = \underline{712.5}$				100
2	.275					200
3	.420					300
4	.565					400

ANALYST M. Eisman

NO. <sub>x</sub> FLASK CALIBRATION

<u>FLASK NO.</u>	<u>VOLUME (ml)</u>	<u>FLASK NO.</u>	<u>VOLUME (ml)</u>
1	2016	29	2070
2	2063	30	2055
3	2013	31	2080
4	2051	32	2079
5	2024	33	2075
6	2045	34	2104
7	2064	35	2067
8	2069	36	2117
9	2065	37	2058
10	2047	38	2026
11	2071	39	2039
12	2055	40	2017
13	2076	41	2050
14	2043	42	2026
15	2075	43	2051
16	2010	44	2017
17	2099	45	2047
18	2086	46	2063
19	2096		
20	2086		
21	2078		
22	2011		
23	2061		
24	2083		
25	2076		
26	2085		
27	2107		
28	2090		

CALIBRATED BY: Scott M. Lee  
 9/23/83

SAMPLE CHAIN OF CUSTODY

PLANT NAME: Amex Chemical Corp.  
 SOURCE NAME: Big Bend Dryer.  
 DATE: 2-16/17/84 TYPE OF SAMPLE: NOx

SAMPLE RECOVERY

<u>CONTAINER NO.</u>	<u>DESCRIPTION</u>		
	<u>NOx</u>	<u>Flask</u>	<u>Coal/Oil</u>
<u>28</u>			<u>1315</u>
<u>38</u>	"	"	<u>1330</u>
<u>39</u>	"	"	<u>1345</u>
<u>44</u>	"	"	<u>1400</u>
<u>33</u>	"	"	<u>1415</u>
<u>35</u>	"	"	<u>1430</u>
<u>41</u>	"	"	<u>1445</u>
<u>30</u>	"	"	<u>1500</u>
<u>32</u>	"	"	<u>1515</u>
<u>31</u>	"	"	<u>1530</u>
<u>40</u>	"	"	<u>1545</u>
<u>36</u>	"	"	<u>1600</u>

Sample Recovered By: M. Eisman  
~~Particulate~~ <sup>NOx</sup> Analysis By: M. Eisman  
 Sample Collection By: R. Skeltes

SAMPLE CHAIN OF CUSTODY

PLANT NAME: Amex Chemical Corp.  
 SOURCE NAME: Big Bend Dryer  
 DATE: 2-17-84 TYPE OF SAMPLE: NOx

SAMPLE RECOVERY

CONTAINER NO.	DESCRIPTION		
	NOx Flask	Oil Firing	
<u>10</u>			<u>0830</u>
<u>5</u>	"	"	<u>0845</u>
<u>21</u>	"	"	<u>0900</u>
<u>24</u>	"	"	<u>0915</u>
<u>43</u>	"	"	<u>0930</u>
<u>42</u>	"	"	<u>0945</u>
<u>25</u>	"	"	<u>1000</u>
<u>27</u>	"	"	<u>1015</u>
<u>3</u>	"	"	<u>1030</u>
<u>13</u>	"	"	<u>1045</u>
<u>4</u>	"	"	<u>1100</u>
<u>15</u>	"	"	<u>1115</u>

Sample Recovered By: M. Eisman

<sup>NOx</sup> Particulate Analysis By: M. Eisman

Sample Collection By: R. Sholtes

#### 4. Procedure

##### 4.1 Sampling.

4.1.1 Preparation of Collection Train. Preparation of the sample train is the same as described in Method 6A, Section 4.1.4, with the addition of the following:

The sampling train is assembled as shown in Figure 6A-1, except the isopropanol bubbler is not included. The probe must be heated to a temperature sufficient to prevent water condensation and must include a filter (either in-stack, out-of-stack, or both) to prevent particulate entrainment in the peroxide impingers. The electric supply for the probe heat should be continuous and separate from the timed operation of the sample pump.

Adjust the timer-switch to operate in the "on" position from 2 to 4 minutes on a 2-hour repeating cycle or other cycle specified in the applicable regulation. Other timer sequences may be used with the restriction that the total sample volume collected is between 25 and 60 liters for the amounts of sampling reagents prescribed in this method.

Add cold water to the tank until the impingers and bubblers are covered at least two-thirds of their length. The impingers and bubbler tank must be covered and protected from intense heat and direct sunlight. If freezing conditions exist, the impinger solution and the water bath must be protected.

**Notes.**—Sampling may be conducted continuously if a low flow-rate sample pump (20 to 40 ml/min for the reagent volumes described in this method) is used. Then the timer-switch is not necessary. In addition, if the sample pump is designed for constant rate sampling, the rate meter may be deleted. The total gas volume collected should be between 25 and 60 liters for the amounts of sampling reagents prescribed in this method.

4.1.2 Leak-Check Procedure. The leak-check procedure is the same as described in Method 6, Section 4.1.2.

4.1.3 Sample Collection. Record the initial dry gas meter reading. To begin sampling, position the tip of the probe at the sampling point, connect the probe to the first impinger (or filter), and start the timer and the sample pump. Adjust the sample flow to a constant rate of approximately 1.0 liter/min as indicated by the rotameter. Assure that the timer is operating as intended, i.e., in the "on" position for the desired period and the cycle repeats as required.

During the 24-hour sampling period, record the dry gas meter temperature one time

between 9:00 a.m. and 11:00 a.m., and the barometric pressure.

At the conclusion of the run, turn off the timer and the sample pump, remove the probe from the stack, and record the final gas meter volume reading. Conduct a leak check as described in Section 4.1.2. If a leak is found, void the test run or use procedures acceptable to the Administrator to adjust the sample volume for leakage. Repeat the steps in this section (4.1.3) for successive runs.

4.2 Sample Recovery. The procedures for sample recovery (moisture measurement, peroxide solution, and ascarite bubbler) are the same as in Method 6A, Section 4.2.

4.3 Sample Analysis. Analysis of the peroxide impinger solutions is the same as in Method 6, Section 4.3.

#### 5. Calibration

##### 5.1 Metering System.

5.1.1 Initial Calibration. The initial calibration for the volume metering system is the same as for Method 6, Section 5.1.1.

5.1.2 Periodic Calibration Check. After 30 days of operation of the test train, conduct a calibration check as in Section 5.1.1 above, except for the following variations: (1) The leak check is not to be conducted. (2) Three or more revolutions of the dry gas meter must be used, and (3) only two independent runs need be made. If the calibration factor does not deviate by more than 5 percent from the initial calibration factor determined in Section 5.1.1, then the dry gas meter volumes obtained during the test series are acceptable and use of the train can continue. If the calibration factor deviates by more than 5 percent, recalibrate the metering system as in Section 5.1.1; and for the calculations for the preceding 30 days of data, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run. Use the latest calibration factor for succeeding tests.

5.2 Thermometers. Calibrate against mercury-in-glass thermometers initially and at 30-day intervals.

5.3 Rotameter. The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instruction.

5.4 Barometer. Calibrate against a mercury barometer initially and at 30-day intervals.

5.5 Barium Perchlorate Solution. Standardize the barium perchlorate solution against 25 ml of standard sulfuric acid to which 100 ml of 100 percent isopropanol has been added.

#### 6. Calculations

The nomenclature and calculation procedures are the same as in Method 6A with the following exceptions:

$P_{bar}$  = Initial barometric pressure for the test period, mm Hg.

$T_m$  = Absolute meter temperature for the test period, °K.

#### 7. Emission Rate Procedure

The emission rate procedure is the same as described in Method 6A, section 7, except that the timer is needed and is operated as described in this method.

#### 8. Bibliography

The bibliography is the same as described in Method 6A, Section 8.

#### METHOD 7—DETERMINATION OF NITROGEN OXIDE EMISSIONS FROM STATIONARY SOURCES

##### 1. Principle and Applicability

1.1 Principle. A grab sample is collected in an evacuated flask containing a dilute sulfuric acid-hydrogen peroxide absorbing solution, and the nitrogen oxides, except nitrous oxide, are measured colorimetrically using the phenoldisulfonic acid (PDS) procedure.

1.2 Applicability. This method is applicable to the measurement of nitrogen oxides emitted from stationary sources. The range of the method has been determined to be 2 to 400 milligrams NO<sub>x</sub> (as NO<sub>2</sub>) per dry standard cubic meter, without having to dilute the sample.

##### 2. Apparatus

2.1 Sampling (see Figure 7-1). Other grab sampling systems or equipment, capable of measuring sample volume to within ±2.0 percent and collecting a sufficient sample volume to allow analytical reproducibility to within ±5 percent, will be considered acceptable alternatives, subject to approval of the Administrator, U.S. Environmental Protection Agency. The following equipment is used in sampling:

2.1.1 Probe. Borosilicate glass tubing, sufficiently heated to prevent water condensation and equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Stainless steel or Teflon<sup>1</sup> tubing may also be used for the probe. Heating is not necessary if the probe remains dry during the purging period.

<sup>1</sup>Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.



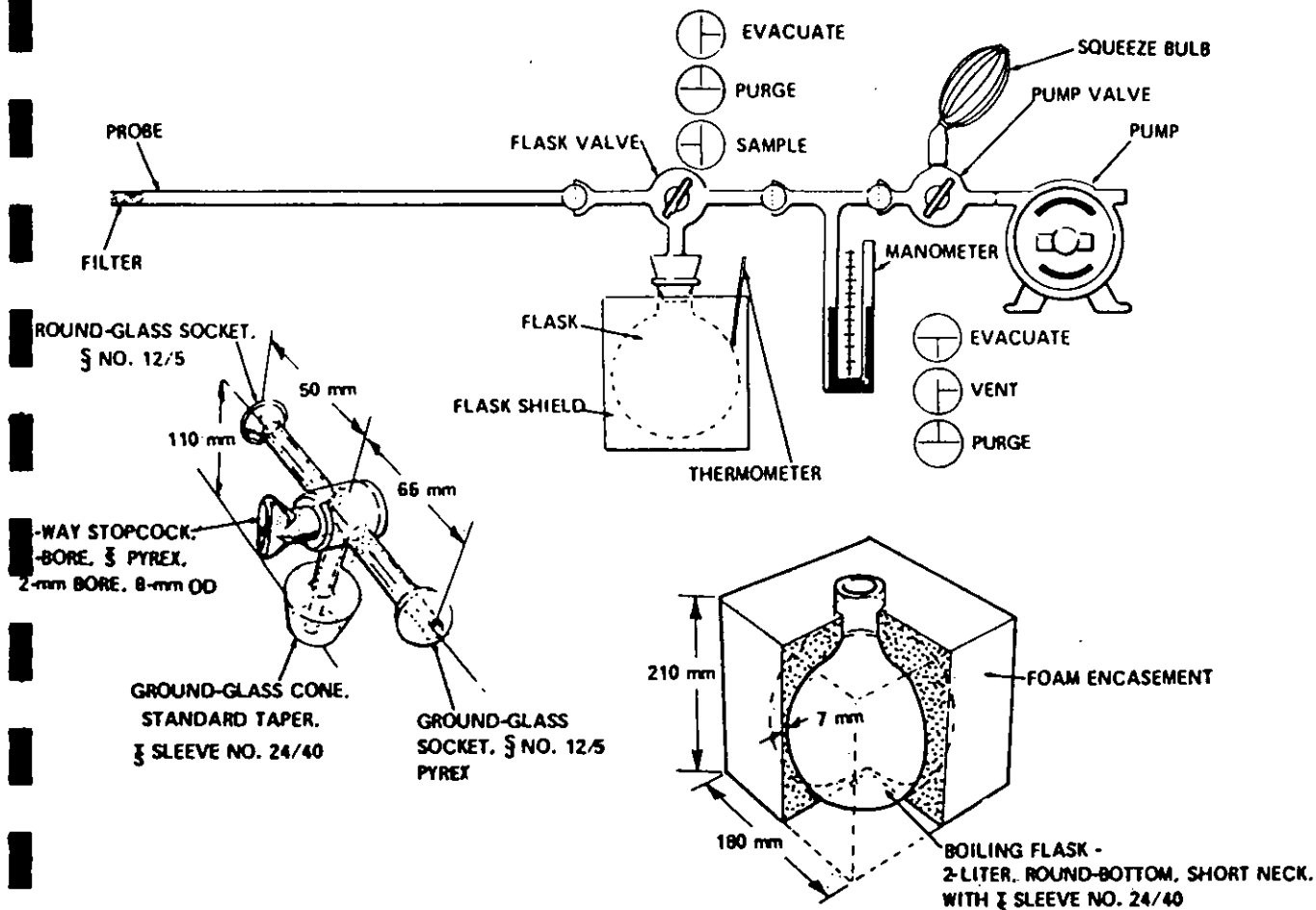


Figure 7-1. Sampling train, flask valve, and flask.

2.1.2 Collection Flask. Two-liter borosilicate, round bottom flask, with short neck and 24/40 standard taper opening, protected against implosion or breakage.

2.1.3 Flask Valve. T-bore stopcock connected to a 24/40 standard taper joint.

2.1.4 Temperature Gauge. Dial-type thermometer, or other temperature gauge, capable of measuring 1° C (2° F) intervals from -5 to 50° C (25 to 125° F).

2.1.5 Vacuum Line. Tubing capable of withstanding a vacuum of 75 mm Hg (3 in. Hg) absolute pressure, with "T" connection and T-bore stopcock.

2.1.6 Vacuum Gauge. U-tube manometer, 1 meter (36 in.), with 1-mm (0.1-in.) divisions, or other gauge capable of measuring pressure to within ± 2.5 mm Hg (0.10 in. Hg).

2.1.7 Pump. Capable of evacuating the collection flask to a pressure equal to or less than 75 mm Hg (3 in. Hg) absolute.

2.1.8 Squeeze Bulb. One-way.

2.1.9 Volumetric Pipette. 25 ml.

2.1.10 Stopcock and Ground Joint Grease. A high-vacuum, high-temperature chlorofluorocarbon grease is required. Halocarbon 25-5S has been found to be effective.

2.1.11 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase, or vice versa for elevation decrease.

2.2 Sample Recovery. The following equipment is required for sample recovery:

2.2.1 Graduated Cylinder. 50 ml with 1-ml divisions.

2.2.2 Storage Containers. Leak-free polyethylene bottles.

2.2.3 Wash Bottle. Polyethylene or glass.

2.2.4 Glass Stirring Rod.

2.2.5 Test Paper for Indicating pH. To cover the pH range of 7 to 14.

2.3 Analysis. For the analysis, the following equipment is needed:

2.3.1 Volumetric Pipettes. Two 1 ml, two 2 ml, one 3 ml, one 4 ml, two 10 ml, and one 25 ml for each sample and standard.

2.3.2 Porcelain Evaporating Dishes. 175- to 250-ml capacity with lip for pouring, one for each sample and each standard. The Coors No. 45006 (shallow-form, 195 ml) has been found to be satisfactory. Alternatively, polymethyl pentene beakers (Nalge No. 1203, 150 ml), or glass beakers (150 ml) may be used. When glass beakers are used, etching of the beakers may cause solid matter to

[Appendix A, Method 7]

be present in the analytical step; the solids should be removed by filtration (see Section 4.3).

2.3.3 Steam Bath. Low-temperature ovens or thermostatically controlled hot plates kept below 70° C (160° F) are acceptable alternatives.

2.3.4 Dropping Pipette or Dropper. Three required.

2.3.5 Polyethylene Pileman. One for each sample and each standard.

2.3.6 Graduated Cylinder. 100 ml with 1-ml divisions.

2.3.7 Volumetric Flasks. 50 ml (one for each sample and each standard), 100 ml (one for each sample and each standard, and one for the working standard KNO<sub>3</sub> solution), and 1000 ml (one).

2.3.8 Spectrophotometer. To measure absorbance at 410 nm.

2.3.9 Graduated Pipette. 10 ml with 0.1-ml divisions.

2.3.10 Test Paper for Indicating pH. To cover the pH range of 7 to 14.

2.3.11 Analytical Balance. To measure to within 0.1 mg.

### 3. Reagents

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

3.1 Sampling. To prepare the absorbing solution, cautiously add 2.8 ml concentrated H<sub>2</sub>SO<sub>4</sub> to 1 liter of deionized, distilled water. Mix well and add 6 ml of 3 percent hydrogen peroxide, freshly prepared from 30 percent hydrogen peroxide solution. The absorbing solution should be used within 1 week of its preparation. Do not expose to extreme heat or direct sunlight.

3.2 Sample Recovery. Two reagents are required for sample recovery:

3.2.1 Sodium Hydroxide (1N). Dissolve 40 g NaOH in deionized, distilled water and dilute to 1 liter.

3.2.2 Water. Deionized, distilled to conform to ASTM specification D1193-77 Type 3 (incorporated by reference—see § 60.17). At the option of the analyst, the KMnO<sub>4</sub> test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

[3.2.2 amended by 48 FR 3735, January 27, 1983]

3.3 Analysis. For the analysis, the following reagents are required:

3.3.1 Fuming Sulfuric Acid. 15 to 18 percent by weight free sulfur trioxide. **HANDLE WITH CAUTION.**

3.3.2 Phenol. White solid.

3.3.3 Sulfuric Acid. Concentrated, 95 percent minimum assay. **HANDLE WITH CAUTION.**

3.3.4 Potassium Nitrate. Dried at 105 to 110° C (220 to 230° F) for a minimum of 2

hours just prior to preparation of standard solution.

3.3.5 Standard KNO<sub>3</sub> Solution. Dissolve exactly 2.198 g of dried potassium nitrate (KNO<sub>3</sub>) in deionized, distilled water and dilute to 1 liter with deionized, distilled water in a 1,000-ml volumetric flask.

3.3.6 Working Standard KNO<sub>3</sub> Solution. Dilute 10 ml of the standard solution to 100 ml with deionized distilled water. One milliliter of the working standard solution is equivalent to 100 µg nitrogen dioxide (NO<sub>2</sub>).

3.3.7 Water. Deionized, distilled as in Section 3.2.2.

3.3.8 Phenoldisulfonic Acid Solution. Dissolve 25 g of pure white phenol in 150 ml concentrated sulfuric acid on a steam bath. Cool, add 75 ml fuming sulfuric acid, and heat at 100° C (212° F) for 2 hours. Store in a dark, stoppered bottle.

### 4. Procedures

#### 4.1 Sampling.

4.1.1 Pipette 25 ml of absorbing solution into a sample flask, retaining a sufficient quantity for use in preparing the calibration standards. Insert the flask valve stopper into the flask with the valve in the "purge" position. Assemble the sampling train as shown in Figure 7-1 and place the probe at the sampling point. Make sure that all fittings are tight and leak-free, and that all ground glass joints have been properly greased with a high-vacuum, high-temperature chlorofluorocarbon-based stopcock grease. Turn the flask valve and the pump valve to their "evacuate" positions. Evacuate the flask to 75 mm Hg (3 in. Hg) absolute pressure, or less. Evacuation to a pressure approaching the vapor pressure of water at the existing temperature is desirable. Turn the pump valve to its "vent" position and turn the off the pump. Check for leakage by observing the manometer for any pressure fluctuation. (Any variation greater than 10 mm Hg (0.4 in. Hg) over a period of 1 minute is not acceptable, and the flask is not to be used until the leakage problem is corrected. Pressure in the flask is not to exceed 75 mm Hg (3 in. Hg) absolute at the time sampling is commenced.) Record the volume of the flask and valve ( $V_f$ ), the flask temperature ( $T_f$ ), and the barometric pressure. Turn the flask valve counterclockwise to its "purge" position and do the same with the pump valve. Purge the probe and the vacuum tube using the squeeze bulb. If condensation occurs in the probe and the flask valve area, heat the probe and purge until the condensation disappears. Next, turn the pump valve to its "vent" position. Turn the flask valve clockwise to its "evacuate" position and record the difference in the mercury levels in the manometer. The absolute internal pressure in the flask ( $V_i$ ) is equal to the barometric pressure less the manometer reading. Immediately turn the flask valve to the "sample" position and permit the gas to enter the flask until pressures in the flask and sample line (i.e., duct, stack) are equal. This will usually require

about 15 seconds; a longer period indicates a "plug" in the probe, which must be corrected before sampling is continued. After collecting the sample, turn the flask valve to its "purge" position and disconnect the flask from the sampling train. Shake the flask for at least 5 minutes.

4.1.2 If the gas being sampled contains insufficient oxygen for the conversion of NO to NO<sub>2</sub> (e.g., an applicable subpart of the standard may require taking a sample of a calibration gas mixture of NO in N<sub>2</sub>), then oxygen shall be introduced into the flask to permit this conversion. Oxygen may be introduced into the flask by one of three methods; (1) Before evacuating the sampling flask, flush with pure cylinder oxygen, then evacuate flask to 75 mm Hg (3 in. Hg) absolute pressure or less; or (2) inject oxygen into the flask after sampling; or (3) terminate sampling with a minimum of 50 mm Hg (2 in. Hg) vacuum remaining in the flask, record this final pressure, and then vent the flask to the atmosphere until the flask pressure is almost equal to atmospheric pressure.

4.2 Sample Recovery. Let the flask set for a minimum of 16 hours and then shake the contents for 2 minutes. Connect the flask to a mercury filled U-tube manometer. Open the valve from the flask to the manometer and record the flask temperature ( $T_f$ ), the barometric pressure, and the difference between the mercury levels in the manometer. The absolute internal pressure in the flask ( $P_f$ ) is the barometric pressure less the manometer reading. Transfer the contents of the flask to a leak-free polyethylene bottle. Rinse the flask twice with 5-ml portions of deionized, distilled water and add the rinse water to the bottle. Adjust the pH to between 9 and 12 by adding sodium hydroxide (1 N), dropwise (about 25 to 35 drops). Check the pH by dipping a stirring rod into the solution and then touching the rod to the pH test paper. Remove as little material as possible during this step. Mark the height of the liquid level so that the container can be checked for leakage after transport. Label the container to clearly identify its contents. Seal the container for shipping.

4.3 Analysis. Note the level of the liquid in container and confirm whether or not any sample was lost during shipment; note this on the analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Immediately prior to analysis, transfer the contents of the shipping container to a 50-ml volumetric flask, and rinse the container twice with 5-ml portions of deionized, distilled water. Add the rinse water to the flask and dilute to the mark with deionized, distilled water; mix thoroughly. Pipette a 25-ml aliquot into the porcelain evaporating dish. Return any unused portion of the sample to the polyethylene storage bottle. Evaporate the 25-

[Appendix A, Method 7]

Aliquot to dryness on a steam bath and allow to cool. Add 2 ml phenoldisulfonic acid solution to the dried residue and triturate thoroughly with a polyethylene policeman. Make sure the solution contacts all the residue. Add 1 ml deionized, distilled water and four drops of concentrated sulfuric acid. Heat the solution on a steam bath for 3 minutes with occasional stirring. Allow the solution to cool, add 20 ml deionized, distilled water, mix well by stirring, and add concentrated ammonium hydroxide, dropwise, with constant stirring, until the pH is 10 (as determined by pH paper). If the sample contains solids, these must be removed by filtration (centrifugation is an acceptable alternative, subject to the approval of the Administrator), as follows: filter through Whatman No. 41 filter paper into a 100-ml volumetric flask; rinse the evaporating dish with three 5-ml portions of deionized, distilled water; filter these three rinses through the filter with at least three 15-ml portions of deionized, distilled water. Add the filter washings to the contents of the volumetric flask and dilute to the mark with deionized, distilled water. If solids are present, the solution can be transferred directly to the 100-ml volumetric flask and diluted to the mark with deionized, distilled water. Mix the contents of the flask thoroughly, and measure the absorbance at the optimum wavelength used for the standards (Section 5.2.1), using the blank solution as a zero reference. Dilute the sample and the blank with equal volumes of deionized, distilled water if the absorbance exceeds  $A_4$ , the absorbance of the 400  $\mu\text{g NO}_2$  standard (see Section 5.2.2).

5. Calibration

5.1 **Flask Volume.** The volume of the collection flask-flask valve combination must be known prior to sampling. Assemble the flask and flask valve and fill with water, to the stopcock. Measure the volume of water  $\pm 10$  ml. Record this volume on the flask.

5.2 **Spectrophotometer Calibration.**  
 5.2.1 **Optimum Wavelength Determination.** Calibrate the wavelength scale of the spectrophotometer every 6 months. The calibration may be accomplished by using a mercury source with an intense line emission such as a mercury lamp, or by using a series of glass filters spanning the measuring range of the spectrophotometer. Calibration materials are available commercially from the National Bureau of Standards. Specific details on the use of such materials should be supplied by the vendor; general information about calibration techniques can be obtained from general reference books on analytical chemistry. The wavelength scale of the spectrophotometer must read correctly within  $\pm 5$  nm at all calibration points; otherwise, the spectrophotometer shall be repaired and recalibrated. Once the wavelength scale of the spectrophotometer is in proper calibration, use 410 nm as the optimum wavelength for the mea-

surement of the absorbance of the standards and samples.

Alternatively, a scanning procedure may be employed to determine the proper measuring wavelength. If the instrument is a double-beam spectrophotometer, scan the spectrum between 400 and 415 nm using a 200  $\mu\text{g NO}_2$  standard solution in the sample cell and a blank solution in the reference cell. If a peak does not occur, the spectrophotometer is probably malfunctioning and should be repaired. When a peak is obtained within the 400 to 415 nm range, the wavelength at which this peak occurs shall be the optimum wavelength for the measurement of absorbance of both the standards and the samples. For a single-beam spectrophotometer, follow the scanning procedure described above, except that the blank and standard solutions shall be scanned separately. The optimum wavelength shall be the wavelength at which the maximum difference in absorbance between the standard and the blank occurs.

5.2.2 **Determination of Spectrophotometer Calibration Factor  $K_c$ .** Add 0.0 ml, 2 ml, 4 ml, 6 ml., and 8 ml of the  $\text{KNO}_3$  working standard solution (1 ml = 100  $\mu\text{g NO}_2$ ) to a series of five 50-ml volumetric flasks. To each flask, add 25 ml of absorbing solution, 10 ml deionized, distilled water, and sodium hydroxide (1 N) dropwise until the pH is between 9 and 12 (about 25 to 35 drops each). Dilute to the mark with deionized, distilled water. Mix thoroughly and pipette a 25-ml aliquot of each solution into a separate porcelain evaporating dish. Beginning with the evaporation step, follow the analysis procedure of Section 4.3 until the solution has been transferred to the 100 ml volumetric flask and diluted to the mark. Measure the absorbance of each solution, at the optimum wavelength, as determined in Section 5.2.1. This calibration procedure must be repeated on each day that samples are analyzed. Calculate the spectrophotometer calibration factor as follows:

$$K_c = 100 \frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2}$$

Equation 7-1

where:

- $K_c$  = Calibration factor
- $A_1$  = Absorbance of the 100- $\mu\text{g NO}_2$  standard
- $A_2$  = Absorbance of the 200- $\mu\text{g NO}_2$  standard
- $A_3$  = Absorbance of the 300- $\mu\text{g NO}_2$  standard
- $A_4$  = Absorbance of the 400- $\mu\text{g NO}_2$  standard

5.3 **Barometer.** Calibrate against a mercury barometer.

5.4 **Temperature Gauge.** Calibrate dial thermometers against mercury-in-glass thermometers.

5.5 **Vacuum Gauge.** Calibrate mechanical gauges, if used, against a mercury manometer such as that specified in 2.1.6.

5.6 **Analytical Balance.** Calibrate against standard weights.

6. Calculations

Carry out the calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculations.

- 6.1 **Nomenclature.**  
 $A$  = Absorbance of sample.  
 $C$  = Concentration of  $\text{NO}_2$ , dry basis, corrected to standard conditions, mg/dscm (lb/dscf).  
 $F$  = Dilution factor (i.e., 25/5, 25/10, etc., required only if sample dilution was needed to reduce the absorbance into the range of calibration).  
 $K_c$  = Spectrophotometer calibration factor.  
 $m$  = Mass of  $\text{NO}_2$ , as  $\text{NO}_2$ , in gas sample,  $\mu\text{g}$ .  
 $P_f$  = Final absolute pressure of flask, mm Hg (in. Hg).  
 $P_i$  = Initial absolute pressure of flask, mm Hg (in. Hg).  
 $P_{std}$  = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).  
 $T_f$  = Final absolute temperature of flask, °K (°R).  
 $T_i$  = Initial absolute temperature of flask, °K (°R).  
 $T_{std}$  = Standard absolute temperature 293° K (528° R).  
 $V_s$  = Sample volume at standard conditions (dry basis), ml.  
 $V_f$  = Volume of flask and valve, ml.  
 $V_a$  = Volume of absorbing solution, 25 ml.  
 2 = 50/25, the aliquot factor. (If other than a 25-ml aliquot was used for analysis, the corresponding factor must be substituted).  
 6.2 **Sample volume, dry basis, corrected to standard conditions.**

$$V_s = \frac{T_{std}}{P_{std}} (V_f - V_a) \left[ \frac{P_f - P_i}{T_f - T_i} \right] - K_1 (V_f - 25 \text{ ml}) \left[ \frac{P_f - P_i}{T_f - T_i} \right]$$

Equation 7-2

where:

$$K_1 = 0.3858 \frac{\text{°K}}{\text{mm Hg}} \text{ for metric units}$$

$$= 17.64 \frac{\text{°R}}{\text{in. Hg}} \text{ for English units}$$

6.3 **Total  $\mu\text{g NO}_2$  per sample.**

$$m = 2 K_c A F$$

Equation 7-3

NOTE.—If other than a 25-ml aliquot is used for analysis, the factor 2 must be replaced by a corresponding factor.

6.4 **Sample concentration, dry basis, corrected to standard conditions.**

$$C = K_2 \frac{m}{V_{sc}}$$

Equation 7-4

APPENDIX A4

Field Data Sheets and Computations  
Opacity/Method 9

Emissions ~ 5-10 <sup>10'</sup> hr. on oil

SHOLTS & KOGLER, ENVIRONMENTAL CONSULTANTS  
 VISIBLE EMISSIONS DATA SHEET



Plant AMAX

Date 2-16-84

Wind Direction and Speed ENE - 5/8

Stack D-101 - Coal Dust Mix

Time 0810

Observer R. Sholtz

sec. min.	0	15	30	45
0	0	0	0	0
1	0	0	0	0
2	0	0	0	0
3	0	0	0	0
4	0	0	0	0
5	0	0	0	0
6	0	0	0	0
7	0	0	0	0
8	0	0	0	0
9	0	0	0	0
10	0	0	0	0
11	0	0	0	0
12	0	0	0	0
13	0	0	0	0
14	0	0	0	0
15	0	0	0	0
16	0	0	0	0
17	0	0	0	0
18	0	0	0	0
19	0	0	0	0
20	0	0	0	0
21	0	0	0	0
22	0	0	0	0
23	0	0	0	0
24	0	0	0	0
25	0	0	0	0
26	0	0	0	0
27	0	0	0	0
28	0	0	0	0
29	0	0	0	0

sec. min.	0	15	30	45
30	0	0	0	0
31	0	0	0	0
32	0	0	0	0
33	0	0	0	0
34	0	0	0	0
35	0	0	0	0
36	0	0	0	0
37	0	0	0	0
38	0	0	0	0
39	0	0	0	0
40	0	0	0	0
41	0	0	0	0
42	0	0	0	0
43	0	0	0	0
44	0	0	0	0
45	0	0	0	0
46	0	0	0	0
47	0	0	0	0
48	0	0	0	0
49	0	0	0	0
50	0	0	0	0
51	0	0	0	0
52	0	0	0	0
53	0	0	0	0
54	0	0	0	0
55	0	0	0	0
56	0	0	0	0
57	0	0	0	0
58	0	0	0	0
59	0	0	0	0

Sum of # Recorded \_\_\_\_\_

Total # of Readings \_\_\_\_\_

Opacity =  $\frac{\text{Sum of # Recorded}}{\text{Total # of Readings}}$  = \_\_\_\_\_

Plume Description

coning  Detached \_\_\_\_\_  
 lofting \_\_\_\_\_ Color \_\_\_\_\_  
 fanning \_\_\_\_\_ looping \_\_\_\_\_  
 fumigating \_\_\_\_\_

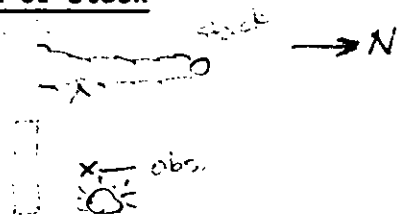
Point of Observation

Steam Plume Distance to base of Source 300'  
 Height of discharge above ground 100'

Back Ground Description

Plume obscured by background

Diagram of Stack



SHOLTES & KOGLER  
ENVIRONMENTAL CONSULTANTS, INC.

SUMMARY OF VISIBLE EMISSIONS

COMPANY NAME: AMAX Chemical Corporation      DATE: February 16, 1984  
SOURCE: Fluid Bed Scrubber      TIME: 0810 - 0839  
PERMIT NO.: AC29-65834

SUMMARY OF AVERAGE OPACITY

Set Number	Time Start - End	Opacity	
		Sum	Average
1	0810 - 0815	0	0.0
2	0816 - 0821	0	0.0
3	0822 - 0827	0	0.0
4	0828 - 0833	0	0.0
5	0834 - 0839	0	0.0

Readings ranged from 0 to 0 percent opacity. The source was in compliance with Chapter 17-2, Florida Administrative Code at the time evaluation was made.



THIS IS TO CERTIFY THAT

Robert S. Sholtes, has completed the STATE OF FLORIDA visible emissions evaluation training and is a qualified observer of visible emissions as specified by EPA reference method 9.  
This certificate expires on June 15, 1984

Judi Sears      Robert S. Sholtes  
Certification Officer      Bearer's Signature  
DER Form PERM 5-9 (Jun 79)

Signed: Robert S. Sholtes

Certificate Expires: June 15, 1984

SHOLTS & KOGLER, ENVIRONMENTAL CONSULTANTS  
 VISIBLE EMISSIONS DATA SHEET



Plant AMAX  
 Stack Fluid Bed Scrubber.

Date 2-17-84  
 Time 1120

Wind Direction and Speed Cal  
 Observer R. Sholts

min. \ sec.	0	15	30	45	min. \ sec.	0	15	30	45
0	5	5	5	5	30				
1	5	5	5	5	31				
2	5	5	5	5	32				
3	5	5	5	5	33				
4	5	5	5	5	34				
5	5	5	5	5	35				
6	5	5	5	5	36				
7	5	5	5	5	37				
8	5	5	5	5	38				
9	5	5	5	5	39				
10	5	5	5	5	40				
11	5	5	5	5	41				
12	5	5	5	5	42				
13	5	5	5	5	43				
14	5	5	5	5	44				
15	5	5	5	5	45				
16	5	5	5	5	46				
17	5	5	5	5	47				
18	5	5	5	5	48				
19	5	5	5	5	49				
20	5	5	5	5	50				
21	5	5	5	5	51				
22	5	5	5	5	52				
23	5	5	5	5	53				
24	5	5	5	5	54				
25	5	5	5	5	55				
26	5	5	5	5	56				
27	5	5	5	5	57				
28	5	5	5	5	58				
29	5	5	5	5	59				

Sum of # Recorded \_\_\_\_\_

Total # of Readings \_\_\_\_\_

Opacity =  $\frac{\text{Sum of # Recorded}}{\text{Total # of Readings}}$  = \_\_\_\_\_

Plume Description

coning \_\_\_\_\_ Detached \_\_\_\_\_  
 lofting \_\_\_\_\_ Color \_\_\_\_\_  
 fanning \_\_\_\_\_ looping \_\_\_\_\_  
 fumigating \_\_\_\_\_

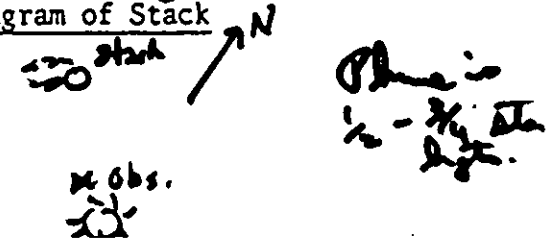
Point of Observation

Steam Plume Distance to base of Source 200'  
 Height of discharge above ground 100'

Back Ground Description

Blue Sky - No Clouds

Diagram of Stack



**SHOLTES & KOOGLER  
ENVIRONMENTAL CONSULTANTS, INC.**

**SUMMARY OF VISIBLE EMISSIONS**

COMPANY NAME: AMAX Chemical Corporation      DATE: February 17, 1984  
 SOURCE: Fluid Bed Scrubber      TIME: 1120 - 1149  
           (Fuel Is Oil)  
 PERMIT NO.: AC29-65834

**SUMMARY OF AVERAGE OPACITY**

Set Number	Time Start - End	Opacity	
		Sum	Average
1	1120 - 1125	120	5.0
2	1126 - 1131	120	5.0
3	1132 - 1137	120	5.0
4	1138 - 1143	120	5.0
5	1144 - 1149	120	5.0

Readings ranged from 5 to 5 percent opacity. The source was in compliance with Chapter 17-2, Florida Administrative Code at the time evaluation was made.



THIS IS TO CERTIFY THAT

Robert S. Sholtes, has completed the STATE OF FLORIDA visible emissions evaluation training and is a qualified observer of visible emissions as specified by EPA reference method 9.  
 This certificate expires on June 15, 1984

Judi Sears      Robert S. Sholtes  
 Certification Officer      Bearer's Signature

DER Form PERM 5-9 (Jun 79)

Signed: Robert S. Sholtes

Certificate Expires: June 15, 1984



APPENDIX B  
Plant Operating Data

PART I  
SPECIFIC CONDITIONS

1. The phosphate rock dryer shall meet all applicable requirements of 40 CFR 60, Subpart NN - Standards of Performance for Phosphate Rock Plants.
2. Phosphate rock feed to the dryer shall not exceed 300 TPH.
3. Particulate emissions from the dryer shall not exceed 0.06 lbs/ton of wet phosphate rock feed or 18 pounds per hour, whichever is more restrictive. Visible emissions shall not exceed 10 percent opacity. Particulate emissions and opacity shall be determined in accordance with reference methods prescribed under 40 CFR §60.404.
4. Sulfur dioxide emissions shall not exceed 1.1 lbs per million BTU heat input. Sulfur dioxide emissions shall be determined by reference method 6, Appendix A, 40 CFR 60.
5. Nitrogen oxides emissions shall not exceed 35.5 lbs per hour. Nitrogen oxides emissions shall be determined by reference method 7, Appendix A, 40 CFR 60.
6. Heat input to the dryer shall not exceed 118 million BTU per hour.
7. Sulfur content of the fuels used in the dryer shall be of an appropriate value to ensure emissions of sulfur dioxide do not exceed the applicable limit (Specific Condition 4), but in no case shall the sulfur content exceed 2.5 percent by weight.
8. At a minimum, the owner/operator of the facility shall install, calibrate, and maintain devices meeting the requirements of 40 CFR §60.403(c) and (d), and maintain on site a daily log of scrubber operation which shall include:
  - a. Pressure drop of the gas in inches of water.
  - b. Flow rate of the scrubber water in GPM.
  - c. pH of the scrubber water.
  - d. Pressure of scrubber water feed.
  - e. Scrubber feed water make up rate.

These items shall be recorded at the time of performance tests and used in subsequent inspections as an indication of operating conditions necessary for compliance with emissions limits. Rock feed input rate, approximate product rock and rock feed moisture content, fuel sulfur content and heating value, scrubbing effluent particulate concentration, and scrubber water make up rate shall also be recorded during performance testing.



FUEL ANALYSIS SHEET

SAMPLE # 1033

DATE 02-13-84

CUSTOMER Mobil

COAL USED

Seam: Taggert / Imboden  
 Source: Mon Coal Comp.  
 BTU/Lb.: 12,485  
 Ash (%): 10.5  
 Sulfur (%): 0.8  
 - Moisture (%): 6.86  
 Hardness: 51.5  
 Fusion (Ash):  
 Volatiles (%):  
 Fixed Carbon (%):  
 - Percent Passing 200 Mesh: 50.65

OIL USED

Type: #6 Bunker C  
 Source: Union 76  
 BTU/Lb.: 18,357  
 Ash (%): 0.05  
 Sulfur (%): 2.42  
 B. S. & W.:  
 - Sp. Grav.: 0.9944  
 API: 10.8 @ 60°F  
 Lb/Gal.: 8.2814  
 - Viscosity (@ 122°F): 340 Cp  
 - Flash: 185°F

COM

- Coal (%): 50.06  
 - Oil (%): 39.66  
 - Water (%): 10.28  
 BTU/Lb.: 14,660  
 Sulfur: 1.69  
 Ash (%): 4.90  
 - Sp. Grav.: 1.115 @ 72°F  
 - Lb/Gal.: 9.29 @ 72°F ; 8.61 @ 122°F  
 - Flash:  
 - Viscosity (@ 122°F): 7650 Cp.

BROOKFIELD VISCOSITY (COM)

Temp. (F)	Centipoise	Temp. (F)	Centipoise
50		140	<u>5180</u>
60		150	<u>4200</u>
70		160	<u>3500</u>
80		170	<u>2850</u>
90		180	<u>2250</u>
100	<u>12,900</u>	190	
110	<u>10,100</u>	200	
120	<u>8080</u>	220	
130	<u>6300</u>	240	

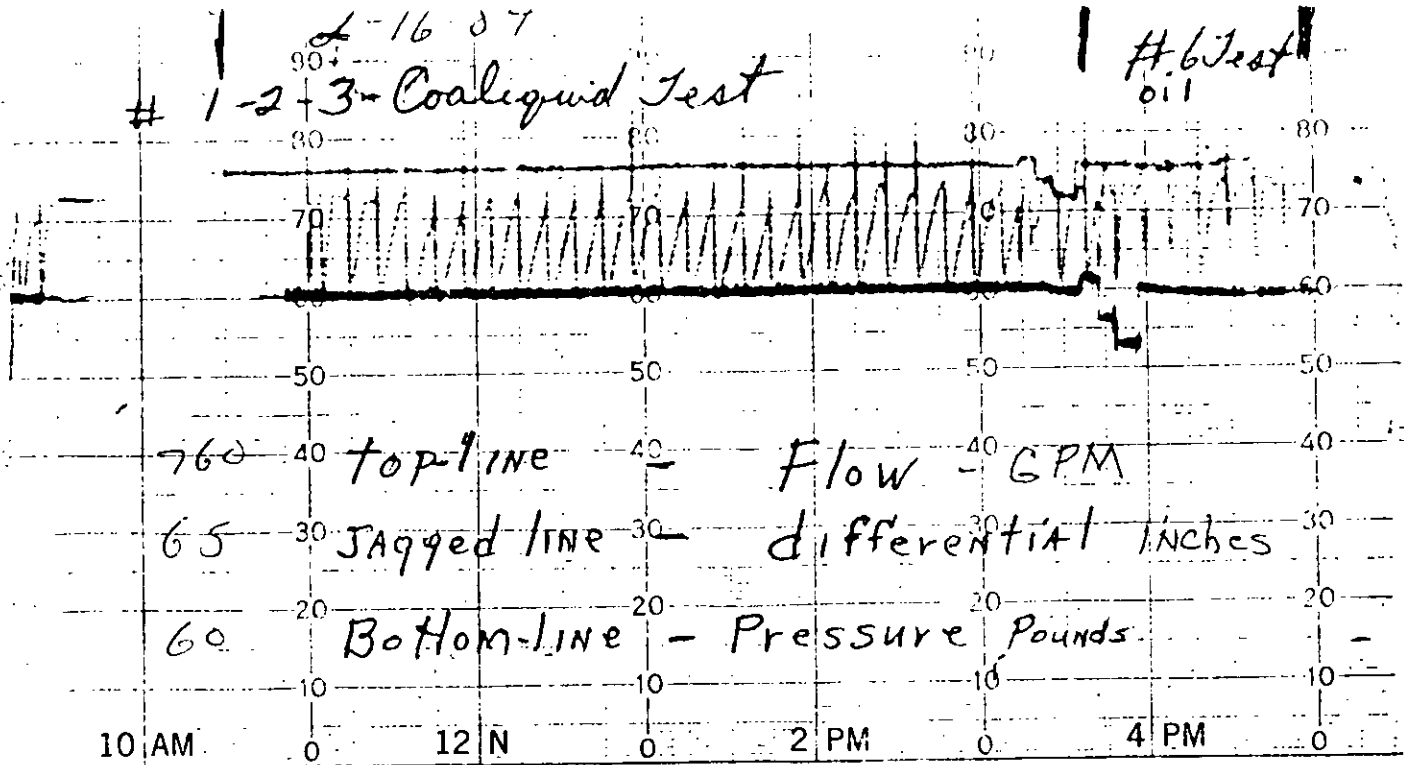
\* Percentages are by weight.

\* Typical Fuel

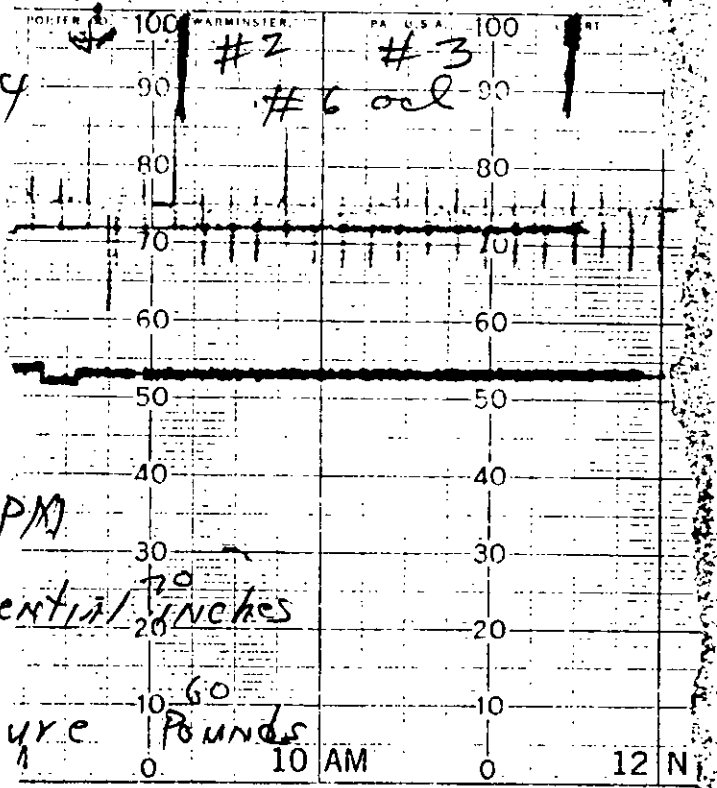
Name [Signature]  
 Position [Signature]

2-16-57  
# 1-2-3 Coalequid Test

#6 Test  
oil



2-17-84



Top line - Flow <sup>700</sup> GPM

Jagged line - differential <sup>70</sup> inches

Bottom line Pressure <sup>60</sup> Pounds

17 cross  
FON

DRYER LOG

OPERATOR: R. Guion

SHIFT: 1

DATE: 2-16-84

HOUR	SCALE READING	TONS PER HOUR	FUEL OIL READING	GALLONS PER HOUR	OIL PRESSURE-1	OIL PRESSURE-2	OIL TEMPERATURE	STEAM PRESS. BURNER	PLENIM TEMPERATURE	PLENIM PRESSURE	BED TEMPERATURE	BED PRESSURE	BED LEVEL	EXHAUST FAN INLET PRESS.	SCRUBBER INLET PRESS.	EXHAUST FAN AMPS	PRIMARY FAN AMPS	COMBUSTION FAN AMPS	PRODUCT TEMPERATURE	EXHAUST TEMPERATURE	TYPE ROCK	SILLO	REMARKS
6																							
7	150870		1396746	100	50	210			1600	33	210	7	9	5.5	0	59	51	20	245	185	A/D	B	change to
8	151090	220	1391050		70	230			1600	36	210	7	9	5.4	0	59	53	23	245	190	A/D	B	Block 8:00
9	151340	250			75	225			1700	35	215	7	9	5.2	0	58	52	20	245	185	B	B	Down 9:25
10	151520	110																			A/D	A	Start 10:10
11	151380	100			70	225			1750	37	210	7	9	5.4	0	58	52	20	245	185	B/D	A	
12	151820	270			70	225			1700	36	205	7	9	5.2	0	58	52	21	245	180	B/D	A	
1	152090	270			65	215			1700	36	205	7	9	4.8	0	58	53	21	245	180	B/D	A	
2	152340	270			68	180			1700	38	210	7	9	5.0	0	58	52	21	245	185	B/D	A	

Com = 132,525 btu/gal. 7.525/gal. 14,250 btu/lb.

SCALE STOP: 393120  
 SCALE START: 391370  
 TOTAL: 1750  
 8 68.70  
 12 2.09

A 6410  
 B 6550

1:30  
 A-51  
 B-11  
 C-E  
 D-E

OIL STOP: 1391050  
 OIL START: 1390320  
 TOTAL: 730

DRYER LOG

OPERATOR: M. Hawkins

SHIFT: 2nd

DATE: 2/16/54

HOUR	SCALE READING	TONS PER HOUR	FUEL OIL READING	GALLONS PER HOUR	OIL PRESSURE-1	OIL PRESSURE-2	OIL TEMPERATURE	STEAM PRESS. BURNER	PLENUM TEMPERATURE	PLENUM PRESSURE	BED TEMPERATURE	BED PRESSURE	BED LEVEL	EXHAUST FAN INLET PRESS.	SCRUBBER INLET PRESS.	EXHAUST FAN AMPS	PRIMARY FAN AMPS	COMBUSTION FAN AMPS	PRODUCT TEMPERATURE	EXHAUST TEMPERATURE	TYPE ROCK	SILLO	REMARKS
2	15230	270	Coal Liquid			68	180		1700	38	210	7	9	5.0	0	58	52	21	245	185	B/D	A	Change to #16
3	15160	270				65	180		1700	36	210	7	9	5.2	0	58	52	21	245	185	B/D	A	Oil 3:45
4	15290	270	1391160	110	100	25	220		1600	39	200	8	9	5.5	0	59	55	20	240	175	B/D	A	
5	153170	270	1391760	100	100	30	220		1700	36	210	8	8	5.2	0	59	53	20	245	185	B/D	A	changed to D
6	15350 15340	290	1392210	450	100	29	230		1600	34	190	8	8	5.6	0	62	55	20	230	165	B/D A	A/D	silos 5:30
7	153760	300	1392790	580	100	25	230		1500	33	175	7	8	5.5	0	60	54	20	220	165	D	D	
8	15400	300	1393340	550	100	22	230		1500	33	175	7	8	5.2	0	61	54	20	220	165	D	D	
9	154360	300	1393880	540	100	21	230		1400	33	170	8	8	5.4	0	61	55	20	215	160	D	D	
10	154700	300	1394420	540	100	21	250		1450	34	170	8	8	5.4	0	62	55	20	215	160	D	D	

1:30 A-51 9:05 A-39  
 B-11 B-F  
 C-E C-E  
 D-E D-45

SCALE STOP: 395390  
 SCALE START: 393120  
 TOTAL: 2270

OIL STOP: 1394426  
 OIL START: 1391050  
 TOTAL: 3370

2:00 - 2.4  
 4:00 - 2.57 68.00  
 6:00 - 1.51 72.80  
 8:00 - 72.20 1.87

#601 148,000 6000/gal.

DRYER LOG

OPERATOR: R. Quinn SHIFT: 1 DATE: 2-17-84

HOUR	SCALE READING	TONS PER HOUR	FUEL OIL READING	GALLONS PER HOUR	OIL PRESSURE-1	OIL PRESSURE-2	OIL TEMPERATURE	STEAM PRESS. BURNER	PLENUM TEMPERATURE	PLENUM PRESSURE	BED TEMPERATURE	BED PRESSURE	BED LEVEL	EXHAUST FAN INLET PRESS.	SCRUBBER INLET PRESS.	EXHAUST FAN AMPS	PRIMARY FAN AMPS	COMBUSTION FAN AMPS	PRODUCT TEMPERATURE	EXHAUST TEMPERATURE	TYPE ROCK	SILLO	REMARKS
6	156970	300	1398420	560	100	26	225		1400	34	170	8	8	5.3	0	61	56	20	215	170	D	C	Chm. p. test.
7	157210	300	1398960	560	100	26	225		1400	35	165	8	8	5.4	0	61	57	20	210	155	D	C	air test only
8	157510	300	1399510	750	100	28	225		1500	34	175	8	8	5.4	0	61	54	20	220	160	D	C	8:20
9	157640 157750	270	1400130	620	100	30	225		1550	36	205	8	8	5	0	61	55	19	240	180	D	C	
10	158000	270	14005760	630	100	30	225		1600	34	210	8	8	5	0	61	53	19	240	180	D	A	
11	158300	270	1401300	640	100	30	225		1600	34	210	8	8	5	0	61	54	19	245	180	D	A	
12	158590	270	1401940	640	100	30	220		1600	34	205	8	8	4.8	0	61	57	19	240	180	D	A	
1																							
2																							

A-39  
B-F  
C-E  
D-F

SCALE STOP: \_\_\_\_\_  
SCALE START: 397640  
TOTAL: \_\_\_\_\_

OIL STOP: \_\_\_\_\_  
OIL START: 1398420  
TOTAL: \_\_\_\_\_

6:00 17150  
10: 65.90 7.86

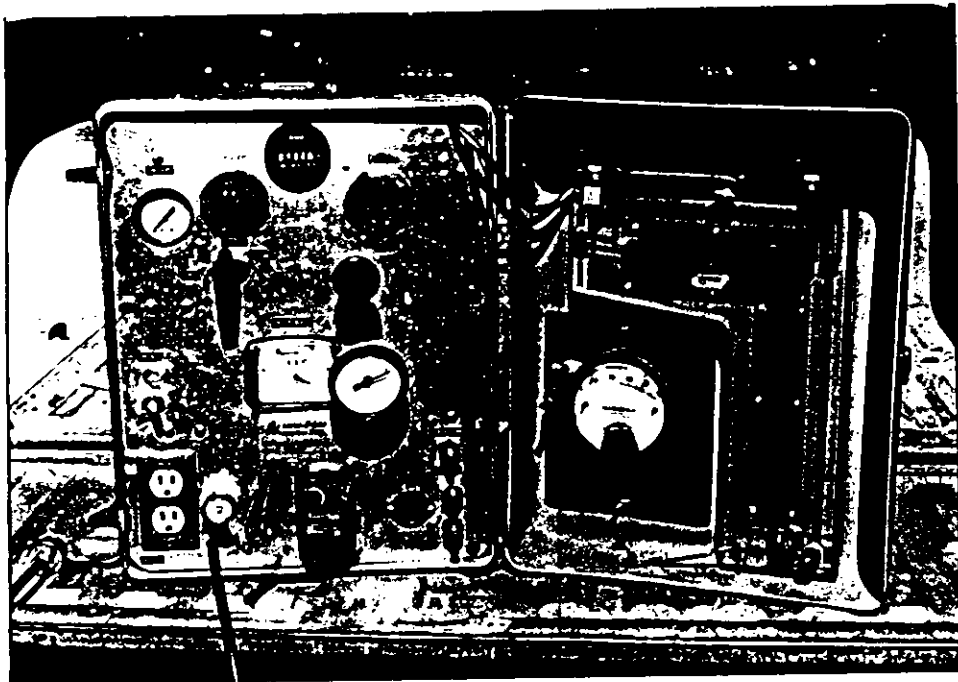


APPENDIX C

Calibrations and Project Participants

SHOLTES & KOOGLER  
ENVIRONMENTAL CONSULTANTS, INC.

SOURCE SAMPLING EQUIPMENT



S.K.E.C. Meter Box

Equipment used in Source Sampling is either manufactured by or assembled by SKEC. The guidelines followed are A.P.T.D. 0581, Details of Isokinetic Source Sampling Equipment, and A.P.T.D. 0576, Maintenance, Calibration and Operation of Isokinetic Source Sampling Equipment.

SK-81

PITOT TUBE CALIBRATION MEASUREMENTS

DATE CALIBRATED 5-11-82

Pitot tube assembly level? X yes \_\_\_\_\_ no

Pitot tube openings damaged? \_\_\_\_\_ yes (explain below) X no

$\alpha_1 = \underline{1.5}^\circ (<10^\circ)$ ,  $\alpha_2 = \underline{0.5}^\circ (<10^\circ)$ ,  $\beta_1 = \underline{1.5}^\circ (<5^\circ)$ ,

$\beta_2 = \underline{1.5}^\circ (<5^\circ)$

$\gamma = \underline{0.5}^\circ$ ,  $\theta = \underline{3.5}^\circ$ ,  $A = \underline{.960}$  in. = (Pa + Pb)

$z = A \sin \gamma = \underline{0.008}$  in. ;  $<0.32$   $<1/8$  in.

$w = A \sin \theta = \underline{0.059}$  in. ;  $<0.08$   $<1/32$  in.

$P_A \underline{.386}$  in.  $P_b \underline{.574}$  in.

$D_t = \underline{.375}$

Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Calibration required? \_\_\_\_\_ yes X no

Calibrated by: George F. Habel

CALIBRATION FORM

Date 2-8-84

Barometric Pressure 30.36 inches Hg

Box No. SK-1

ΔHT	ΔHD	Teledyne Mass Flow Meter		Gas Volume, Dry Gas Meter			Temp. Teledyne	Temp. of Dry Meter	Time Minutes
		Desired liters/min	Actual liters/min	Initial	Final	Actual ft. <sup>3</sup>			
0	0.97	15	15	7.0	9.724	2.724	68	79	5
0	1.75	20	20	45.0	48.654	3.654	68	87	5
0	2.60	25	25	1.0	5.489	4.489	68	80	5
0	3.85	30	30	39.0	44.352	5.352	68	86	5
0	5.00	35	35	16.0	22.169	6.169	68	83	5

GAS METER THERMOMETER CALIBRATION

N.B.S. °F	METER BOX
33	34
51	50
72	72
93	94
115	114

	1.368	1.800	2.232	2.664	3.096	--MEAN--
DELTA-H :	1.368	1.800	2.232	2.664	3.096	1.825
Y :	0.990	0.997	0.999	1.014	1.018	1.003
SCFM :	0.537	0.716	0.895	1.075	1.254	
GF :	4.150	5.615	6.800	8.321	9.457	

Signature George F. Gabel

SHOLTES & KOOGLER,  
ENVIRONMENTAL CONSULTANTS, INC.

THERMOCOUPLE CALIBRATIONS

Read Out No. SK-1 Umbilical Cord 200' Date 10-14-83

Probe Number	Ice Bath		Ambient		150°		400°	
	N.B.S.* °F	PROBE °F	N.B.S.* °F	PROBE °F	N.B.S.* °F	PROBE °F	N.B.S.* °F	PROBE °F
SK-21	32	32	65	66	171	168	391	395
SK-41	32	31	66	67	173	175	396	396
SK-61	32	31	66	66	168	168	420	422
SK-62	32	32	67	65	163	164	428	427
SK-71	33	34	68	68	158	155	419	421
SK-81	32	32	68	67	140	138	421	423
SK-91	33	35	68	69	151	152	398	400
SK-101	33	34	68	68	144	145	404	408
SK-111	32	33	68	70	162	160	378	377
SK-51-T	33	33	68	70	168	170	385	383
SK-101-T	34	32	67	68	170	170	391	392
HEATED BOX NO. 1	33	34	64	63	188	185	370	372
HEATED BOX NO. 2	33	35	64	64	186	188	374	374
LAST IMPINGER PROBE NO. 1	34	34	64	65	182	184	381	380
LAST IMPINGER PROBE NO. 2	34	33	64	65	178	176	386	387

\* National Bureau of Standards Calibrated Mercury Thermometer

*George F. Habel*  
Signature

CALIBRATION FORM

Date 7-18-83

Box No. 16-3

Barometric Pressure 30.05 inches Hg

ΔHT	ΔHD	Teledyne Mass Flow Meter		Gas Volume, Dry Gas Meter			Temp. Teledyne	Temp. of Dry Meter	Time Minutes
		Desired liters/min	Actual liters/min	Initial	Final	Actual ft. <sup>3</sup>			
0		15, <del>16</del>		94.2	97.115	2.915	68	95	5
0		<del>28</del> 100		97.5	99.359	1.859	68	100	5
0		25					68		
0		30					68		
0		35					68		

GAS METER THERMOMETER CALIBRATION

N.B.S. °F	METER BOX

Signature APaul

--MEAN--

DELTA-H :	0.000	0.000	0.000
Y :	0.998	1.007	1.000
SCFM :	0.553	0.355	
OF :	0.000	0.000	

PROJECT PARTICIPANTS

Robert S. Sholtes, Ph.D, P.E.

Project Advisor

Rodney C. Paul

Field Test Crew

George F. Gabel

Field Test Crew



Mail To: Gary Rust  
 BACT/LAER Clearinghouse  
 U.S. EPA (MD-15)  
 RTP, NC 27711

BACT/LAER DETERMINATION REPORT

9.16  
Source Category  
 5/10/84  
Date of This Report

Source Type/Size: Fluidized bed phosphate rock dryer 300 TPH

Company Name/Site Location: AMAX Phosphate, Inc. Bradley, Florida

Determination is: BACT/LAER For New/Modified Source:  
 (circle appropriate definition)  
 Permit No.: PSD-FL-094

Date of Permit Issuance: October 7, 1983

Date of Estimated Start-up: March 1, 1984

Determination Made By: Fl. Dept of Environmental Reg.  
 (Agency)

Willard Hanks  
 (Person Directly Knowledgeable About Permit) (904)488-1344  
 (Phone)

Permit Parameters: (List all processes subject to this permit)	Throughput capacity, weight rate, Btu input, etc.	Pollutant(s) emitted (SO <sub>2</sub> particulate, etc.)	BACT/LAER emission limit(s) and basis for the limits* (units of emissions/units of input)	Control equipment or process modification description**	Eff. %
Rock Dryer	118 x 10 <sup>6</sup> Btu/hr heat input	Particulate	.06 lb/ton wet feed	Cyclone Separator & Impingement Scrubber	97%
		SO <sub>2</sub>	1.1 lb/mmBtu		
		NO <sub>x</sub>	.3 lb/mmBtu		
		VE	Max 10% opacity		

Notes\*\*\*: The dryer was modified to fire a coal-oil-water mixture (COWM) containing max 2.5% Sulfur.

\*Indicate basis of emissions limit, i.e., BACT determined simply by technology and economics, NSPS, LAER, or determined by MAAQS or PSD increment constraints. (Example: 0.05 pounds particulate per million Btu input limit needed to protect Class I increment.) To promote consistency, please use NSPS emission limits where possible.

\*\*To the extent possible in the space available, describe basic control or process equipment design details. Indicate unique or innovative features.

\*\*\*Notes are optional and can address special items, unusual circumstances, or other clarifying information such as SIC codes.