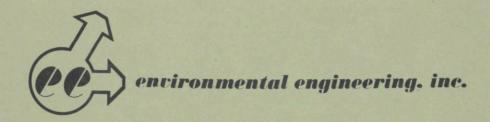
TEST NO. 71 - CI - 32 TEXAS GULF, INC. SUPER PHOSPHORIC ACID AURORA, NORTH CAROLINA

NOVEMBER 19, 1971



2324 S. W. 34th STREET / GAINESVILLE, FLORIDA 32601 / PHONE 904 / 372-3318

TEST NO. 71 - CI - 32 TEXAS GULF, INC. SUPER PHOSPHORIC ACID AURORA, NORTH CAROLINA

NOVEMBER 19, 1971

Test Conducted By:
Environmental Engineering, Inc.
Contract No. CPA 70 - 82

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### I. INTRODUCTION

Under the direction of the Environmental Protection Agency, Environmental Engineering, Inc. conducted emission tests at the Texas Gulf, Inc. phosphate complex in Aurora, North Carolina. On November 19, 1971, three two-hour test runs were conducted on TGI's super phosphoric acid production facilities. The purpose of the tests was to obtain data for the use of both the Industrial Studies Branch and the Performance Standards Branch of the EPA.

The outlet stack of the off-gas scrubber was measured for soluble and insoluble fluorides. Grab samples of the scrubbing liquid, the process reactant, and the process product were also analyzed for fluoride content. A schematic flow diagram indicating the sampling location is given in Figure 1.

Complete test restuls are listed in Appendix A.

Figure 1
VACUUM EVAPORATION SUPER PHOSPHORIC ACID

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### II. SUMMARY OF RESULTS

The plant was operating under normal process conditions during all of the test runs.

One deviation from standard testing procedure was necessary. The velocity of the exit gas was too low to be measured by Method 2 of the Federal Register even when using a micromanometer. At the time of the tests, no other method was available. The EPA project officer decided to conduct the tests, sampling at a constant rate determined by the ΔH@ of the meter box. Jerome Rom (EPA) returned to the test site at a later date to measure the stack gas velocity. At this time, he measured the gas velocity by igniting a colored smoke bomb in the stack and measuring the time required for the smoke to appear at the stack outlet. The length of the stack from the sample port to the exit was known; consequently, the stack gas velocity could be calculated. Several such tests were made, and the times from all the tests were averaged; this average time was used to calculate the gas velocity. Needless to say, this method will not give the exact velocity. However, because the process operation does not vary a great deal, this method should give: an approximate value for the gas velocity during the actual fluoride tests.

A complete summary of stack gas conditions and emission levels is given in Table 1.

TABLE 1 SUMMARY OF RESULTS FLUORIDES

# TEXAS GULF, INC. SUPER PHOSPHORIC ACID OUTLET

Run No.	] .	2	3
Date	11/19/71	11/19/71	11/19/71
Barometric pressure, inches Hg	30	30	30
Stack pressure, inches Hg	30	30	30
Stack gas moisture, % volume	1.9	1.9	1.8
Average stack gas temperature, °F	72	72	71
Stack gas flow rate @ S.T.P.*, SCFM	420	420	420
Volume of gas sampled @ S.T.P.*	90.315	88.809	90.157
Fluoride, water soluble, mg	13.148	35.910	11.781
Fluoride, total, mg	13.148	35.910	11.781
Fluoride, water soluble, gr/SCF	0.002	0.006	0.002
Fluoride, total, gr/SCF	0.002	0.006	0.002
Fluoride, water soluble, gr/CF stk. cond.	0.002	0.006	0.002
Fluoride, total, gr/CF stk. cond.	0.002	0.006	0.002
Fluoride, water soluble, lb/hour	0.008	0.022	0.007
Fluoride, total, lb/hour	0.008	0.022	0.007

<sup>\*</sup> Dry, 70°F., 29.92 inches Hg.

### III. PROCESS DESCRIPTION

Texas Gulf, Inc. operates two super phosphoric acid (SPA) production trains at their Lee Creek, North Carolina operations. Emissions were sampled from the south production train only. Texas Gulf, Inc. produces SPA by concentrating 54 percent  $P_2O_5$  phosphoric acid to 68-72 percent  $P_2O_5$ .

In concentrating the acid, 54 percent  $P_2O_5$  phosphoric acid is continuously fed to the vacuum evaporator (Figure 1). The overheads, containing fluorides and water vapor, are condensed in a barometric condenser. The barometric condenser water flows to the barometric condenser hotwell after which it is sewered to the gypsum pond. The product acid (68-72 percent  $P_2O_5$ ) is continuously tapped from the evaporator and pumped to the product acid cooling tank where it is cooled before being pumped to storage.

Fluoride emissions from the barometric condenser hotwell and the product acid cooling tank are controlled by a scrubber.

4

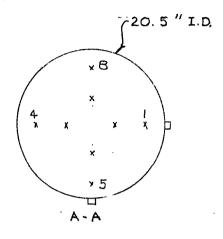
### IV. PROCESS OPERATION

All three test runs were conducted on November 19, 1971. The process operated normally throughout the collection of all samples.

# V. LOCATION OF SAMPLING POINTS

The sampling sites and number of traverse points were selected as per "Method I - Sample and Velocity Traverses for Stationary Sources, Part 60, Subchapter C, Chapter 1, Title 40," Federal Register, No. 247-Pt. II-1.

Figure 2 is a schematic diagram of the stack configuration near the sampling location, and the sampling points traversed during the emission tests.



SAMPLE	DISTANCE FROM INSIDE WALL (INCH)
1, 5	1.25
2, 6	5.00
3, 7	15.00
4, 8	18.75

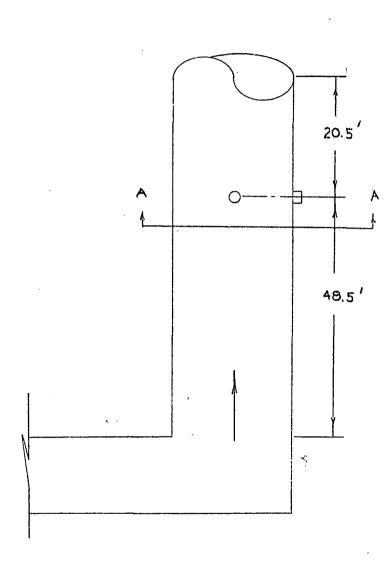


Figure 2
SAMPLE PORT LOCATION

### VI. SAMPLING AND ANALYTICAL PROCEDURES

### A. Preliminary Moisture Determination

The preliminary moisture content of the stack gas was found by using the wet bulb-dry bulb method as referred to in the <u>Federal</u> Register (Vol. 36, No. 247, Part II, December 23, 1971).

## B. Preliminary Velocity Determination

Because of such a low flow rate, the velocity could not be measured as per Method 2 of the above referenced Federal Register. A micromanometer was used with an S-type pitot tube in an attempt to measure the flow; however, the flow rate was so low that this method did not work. The EPA project officer decided to perform the tests, sampling at a constant rate determined by running the sample train at the  $\Delta H \Theta$  of the meter box. The stack velocity was determined by Jerome Rom (EPA) at a later date. The method used consisted of igniting a smoke flare in the stack at the sample port and measuring the time required for the smoke to travel the known distance to the stack outlet. Several such tests were made, and the average time of the tests was used to calculate the velocity of the stack gas.

# C. <u>Sampling for Fluoride Emissions</u>

The sampling procedure used for determining fluoride emissions was similar to Method 5 of the <u>Federal Register</u>. Other than the velocity measurement, the major difference between the two methods

was the configuration of the sampling train. The sampling train described in the <u>Federal Register</u> has a heated box containing the filter holder directly following the glass probe. The sampling train used in these tests contained no heated box and the filter holder was placed between the third and fourth impingers (between dry impinger and silica gel impinger) to prevent sample carry over. Figure 3 is a schematic diagram of the sampling train used.

After the selection of the sampling site and the minimum number of sampling points per Method 2 of the above referenced <u>Federal Register</u>, three separate test runs were performed. For each run, the required stack and sampling parameters were recorded on field data sheets. They are included in Appendix B. Readings were taken at each traverse point. As already mentioned, because of the low flow rate, it was decided to sample at the  $\Delta H0$  of the meter box (1.62 inches  $H_20$ ). The traverse points were selected to maintain at least one inch from the inner stack wall.

After each run, the liquid volume in the first three impingers was measured volumetrically and the silica gel was reweighed. The impinger liquid, the filter, plus the water washings of the probe and other sampling train components up to the silica gel were placed into polyethylene containers. During some runs the different sample fractions were placed in separate containers, while during others all the recovered sample was placed into one container. Field data sheets are included in Appendix B.

## D. <u>Liquid and Product Grab Samples</u>

Periodically, during each test run, grab samples of the raw materials, finished product, and scrubber liquid were taken, and the temperature and pH were determined at the site.

# E. Laboratory Analysis Procedures

Water soluble fluorides were done by a sulfuric acid distillation followed by the SPADNS-ZIRCONIUM LAKE METHOD. Water insoluble fluorides were first fused with NaOH followed by a sulfuric acid distillation then by the SPADNS-ZIRCONIUM LAKE METHOD.

For more details of exact method used, see Appendix C.

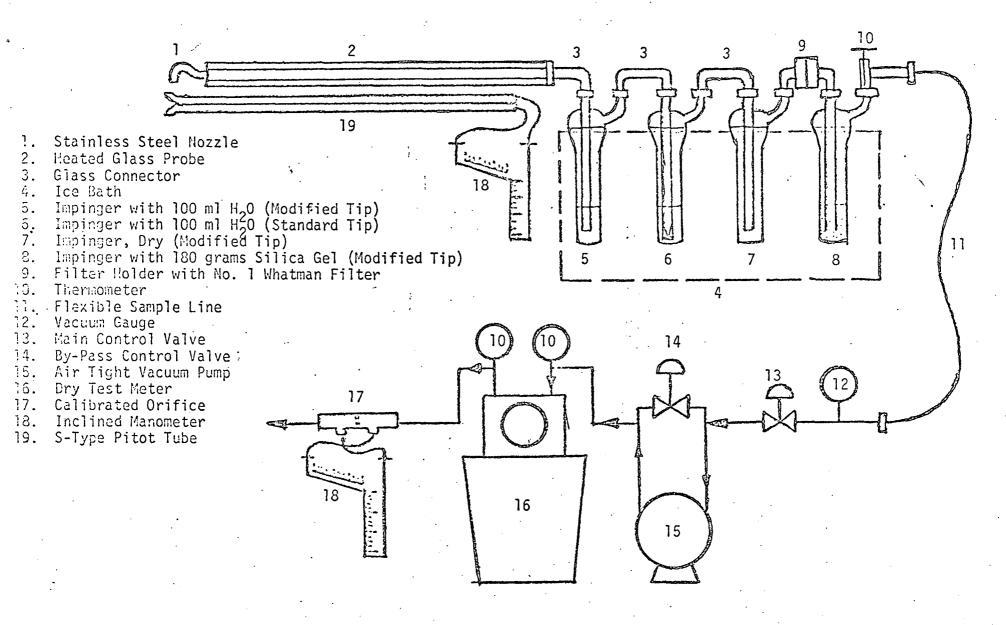


Figure 3

FLUORIDE SAMPLING TRAIN

APPENDICES

APPENDIX A Emission Calculations and Results

#### E.E.I. 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 ze

TS - Stack temperature, °R TM - Meter temperature, °R

H - Average square root of velocity head, √inches H<sub>2</sub>O

ΔH - 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 impringers, milliliters

Po - Pressure at the dry test meter orifice,  $\overline{PB} + \Delta \overline{H}$  inches Hg

STP - Standard conditions, dry, 70°F, 29.92 inches Hg

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, %

 $\sqrt{\text{HxIS}}$  - 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)

QSTPD - Stack gas flow rate, cubic feet per minute (STP)

PISO - Percent isokinetic volume sampled (method described in Federal Register)

Time - Total sample time, minutes

## EQUATIONS FOR CALCULATING FLUORIDE EMISSIONS

$$VWV = (0.0474) \times (VC)$$

$$VSTPD = (17.71 \times (VM) \times (PB + \frac{H}{13.6}) \div TM$$

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

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

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

$$FNOIST = Assumed moisture fraction$$

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

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

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

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

$$U = (174) \times (CP) \times (H) \times \sqrt{(TS \times 29.92) \div (GS \times PS)}$$

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

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

$$QSTPD = (530) \times (QD) \div (TS) \times (PS) \div (29.92)$$

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

### Fluoride Emissions:

MG = Milligrams of fluoride from lab analysis

Grains/SCF = (0.01543) x (MG)  $\div$  VSTPD

Grains/CF, Stack Cond. = (17.71) x (PS) x (FDA) x (Grains/SCF)  $\div$  (TS)

Lbs/hour = (Grains/SCF) x (0.00857) x (QSTPD)

P<sub>2</sub>0<sub>5</sub> Fed = Tons/hour, determined from plant data

Lbs/ton P<sub>2</sub>0<sub>5</sub> Fed = (1bs/hour)  $\div$  (Tons/hour P<sub>2</sub>0<sub>5</sub> Fed)

# SOURCE TEST DATA

TEST NO. PLANT - TEXAS GULF SULFUR, AURORA, NORTH CAROLINA
SOURCE - SUPERPHOSPHORIC ACID (S. Stack)
TYPE OF PLANT - SUPER PHOSPHORIC ACID
CONTROL EQUIPMENT POLLUTANTS SAMPLED - FLUORIDES

1)RUN NUMBER		12	l3
1 2) DATE $1$	<u> 11/19/71</u>	L11/19/71	l11/19/71
3)TIME BEGAN	10:45	l13:30	L_17:05
4) TIME END	12:50	<u> </u>	19:05
5)BAROMETRIC PRESSURE, IN HG	30	130	l30
6) METER ORIFICE PRESSURE DROP, IN H20	1.62	1.62	1_1.62
7) VOL DRY GAS, METER COND, CUBIC FEET	90.243	89,238	90.254
8) AVERAGE GAS METER TEMPERATURE, DEG F	73	76	1_74
9) VOL DRY GAS, S.T.P. CUBIC FEET	90.315	88.809	90.157
10) TOTAL H2O COLLECTED, ML	37.3	36.2	35.4
11) VOL H20 VAPOR COLLECTED, S.T.P., CU FT	1.77	1.72	1.68
12)STACK GAS MOISTURE, PERCENT VOLUME	1.9	1.9	1_1.8
14) PERCENT CO2	0		
15) PERCENT 02	19.2		
16) PERCEUT CO	0		[
17) PERCENT N2	80.8		
19)MOLECULAR WEIGHT OF STACK GAS, DRY	28.77	28.77	l   28.77
20) MOLECULAR WEIGHT OF STACK GAS, STK COUD	28.54	28.54	28.54
21)STACK GAS SPECIFIC GRAVITY	0.98	0.98	0.98
23)AVERAGE STACK CAS TEMPERATURE, DEG F $\frac{1}{1}$	72	72	1
<u></u>			l
26)STACK PRESSURE, IN HG, ABSOLUTE 1	30	30	<u> 30</u>
27) STACK GAS VEL, STACK COND, F.P.M.	197	197	197
28) STACK AREA, SQ FEET $\overline{1}$	2.18	2.18	2.18
29) EFFECTIVE STACK AREA, SQUARE FEET	2.18	2.18	2.18
30) STACK GAS FLOW RATE, S.T.P., SCFMD $\widehat{oxed{1}}$	420	420	420
31) NET TIME OF TEST, MINUTES $\overline{1}$	120	120	1.20
32) $SAMPLING NOZZLE DIAMETER, INCHES \overline{1}$	0.25	0.25	0.25
34)FLUORIDE - WATER SOLUBLE, MG	13.148	35.910	l  11.781
35) FLUORIDE - TOTAL, MG	13.148	35.910	11.781
36) FLUORIDE - WATER SOLUBLE, GR/SCF	0.002246	0.006239	0.002016
37) FLUORIDE - TOTAL, GR/SCF	0.002246	0.006239	0.002016
38) FLUORIDE - WATER SOL., GR/CF, STK CND. $\overline{1}$	0.002194	0.00606	0.001965
39) FLUORIDE - TOTAL, GR/CF, STK CND. $\overline{1}$	0.002194	0.00606	0.001965
40) FLUORIDE - WATER SOLUBLE, LB/HOUR	0.00808	0.022457	0.007256
41) FLUORIDE - TOTAL, LB/HOUR	0.00808	0.022457	0.007256
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APPENDIX B Field Data

# SOURCE SAMPLING FLELD DATA SHEET

Plant / 6-	S Supera	cid					· 1	Mat'1 Proces	sing R	ate			
•	ocation			Sket	ch of Stacl	k		Final Gas Me	ter Re	ading_	755	.e70	ft <sup>3</sup>
Date <u>[] - /</u>	-71	Run No.				,		Initial Gas	Meter	Reading	8 / 6 41.	827	ft <sup>3</sup>
	Ti					·	1	Total Conden			,	_	m1
	me/Point_15		,				]	Moisture in	Silica	Gel			gm
DB <u>72</u> °F,	WB°F,	VF @ DP_	''Hg					Silica Gel C	ontain	er No.	7.06 F	ilter No.	4
Mbisture	%,FDA,Ga	s Density	Factor		1	11		Orsat: CO <sub>2</sub>	0		<u> </u>		11
Barometric	Press 3 c 'Hg	, Stack F	ress3c;"Hg		10 20	)		02	_	,			
			1	<b>!</b>	7:		1			1			
	°F, W/D				, , , , , ,	a managan		$N_2$	• •				]
Sample Box	NoM	leter Box	No.	٠				Excess					
Meter Ali@ /	-67 Pitot	Corr. Fac	tor.83		/			Air	-	·			
Nozzle Dia	Vy_in., Pr	obe Lengt	h 4 ft			•		Test Conduct	ed By:	2 . )	12004		
•	er Setting 😓		1								· ,		
Stack Dimer	nsions: Insid	e Diamete	er 20 in -										
	Insid Heigh	le Area itな 100	ft2 ft	/	5 13	,-		Remarks:				·	
	**********					•				·		······································	
							•	· · · · · · · · · · · · · · · · · · ·		····· 4- + 4	<del></del>	<del></del>	
Fort and	Distance	Clock	Gas Meter		Stack	Meter		Stack Gas	Gas S	Sample	Sample	Last	Vacuum
Traverse	from End of Port	Time	Reading		Velocity	Orifi		Temp.	Temp.	@ Dry	Box	Impinger	on
Point No.	(in)		(ft <sup>3</sup> )		Head (''H <sub>2</sub> 0)	Press		(°F)	Gas N		Temp.	Temp.	Sample   Train
<del></del>	17	-			2	<del>!</del>	Actual	<del></del>	In	Out			("Hg)
· <u>(-/</u>	1/4		664.82	1		1.62	1.63	72	71	72.	160	72	3.0
·		10:50	668.6			Si .			7/_	1/1		64	3~)_
1 /-7-	5	110:53	672.26 675.97			2	į	700	71	71	160	66	3.5
/ <del>-{</del>	J	11: 05	777 64				7	<b>,</b>	-71	71 -	1800	67	3.8
·	·	11: 10	63344					The state of the s	7/	71		67	4.0
1-3	15	11:15	687.19			4.,		\$\$	72_	12	160	68	4.0
·		11:20	690 94			V.	Vin	- 6/	72	72		68	4.1
	l	11:25	694 69	····		1063-	1.82	1 72.	172	172	<u> </u>	68	4,3

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Port and Traverse	Distance from End	Clock Time	Gas Meter Reading	Stack Velocity	Mete	er fice	Stack Gas Temp.	Gas S	Sample .0 Dry	Sample Box	Last Impinger	Vacuum on
Point No.	of Port	Line	(ft <sup>3</sup> )	Head	Pres	ss.Diff.	(°F)	Gas N	leter	Temp:	Temp:	Sample
	(in)		ĺ	(,,H <sup>5</sup> 0)	1	H <sub>2</sub> 0)		(°1		(°F)	(°F)	Train ("Hg)
		-	1	ļ	Cal.			In	Out	[		
1-4	18/4	11.30	698.41		1.62	1.60	72.	72	172	152	68	166
		11:35	702.13					7.3	7.2.		68	5.1
		11:40	705,59		<u>                                     </u>		- 3 Y	2.3	73		68	5,22
			<u> </u>	<del> </del>				<u> </u>				
Era 1/2		11:45	709.666	<u> </u>			<u> </u>	73	73	175	68_	5,2
			-	<u> </u>			*	ļ	<u> </u>		· · · · · · · · · · · · · · · · · · ·	ļ
2-1		11:50	709.67	<u> </u>	/ \		4	73	73	175	68	5.2-
	<u></u>	11:55	713.6	<u> </u>	<u>'   ' -</u>			1.74	73		68	5.5
		12.00	717,2		1	***************************************		24	73		68	5.5
2-2		12:05	721.1		1/			214	7.3		11	55
		10:10	704.8		<u>                                     </u>	the state of the s		74	.73		71	5.6
		18:15	728.7			. Solita		.74	74		70-	56
2-3		12:20	732.3		<u>                                     </u>		, ,	24	74	170	70	5,6
	, ,	12:25	736.1		<u>                                     </u>		<b>1</b>	75	74		67	5.6
	·	12:30	739,9				1000	75	74		67	5.5
2- 4		12:35	143,7			: Ct		7.5	24	172	66	5.5
1		12:40	747.42			Case of the Case o	- AME	75	74:		67	5.7
		12:45	751.19		1	· voice	35.726	7.5	75		67	5.7
					V		V					
End		12:50	755.070		1.62	1.42	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	74	75		67	5.7
			664.827		13	J.	odu.	Ð:				
			90743		16	ŧ .	72	7	3			
			/ <u> </u>									
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# SOURCE SAMELING FILLD LAIR SHEET

Plant T 65 Super Acid			Mat'l Process	sing Rate			
Sampling Location	Sketch of Stac	k	Final Gas Met	ter Reading	844.	308	ft <sup>3</sup>
Date 11/19/71 Run No. 7			Initial Gas N	_			ft <sup>3</sup>
Time Start 13.30 Time End			Total Condens				m1
Sampling Time/Point 15			Moisture in S	Silica Gel			gm
DB 77 °F, WB °F, VF @ DP 'H	- I		Silica Gel Co	ontainer No.	200 F	ilter No.	5
Moisture %, FDA , Gas Density Factor			Orsat: CO2				
Barometric Press 3 c 'Hg, Stack Press 3 "Hg		igwedge	02				
Weather			co				
Temp. 70 °F, W/D , W/S	-		N <sub>2</sub>				·
Sample Box No. Meter Box No. 4	ì		Excess				
Meter AHS 1/2 Pitot Corr. Factors 83			Air		<del></del>		<del></del>
Nozzle Dia. 1/2/ in., Probe Length / / ft			Test Conducte	ed By: (r.	Allen		
Probe Heater Setting					Johnso		
Stack Dimensions: Inside Diameter 20 in	1	<u></u>	_}		Falgout		
Inside Area ft	2	•	Remarks:				
Height 100 f							
					···· - 7		
Port and Distance Clock Gas Mr Traverse from End Time Read	1.	Meter Orifice	Stack Gas Temp.	Gas Sample Temp.@ Dry		Last Impinger	Vacuu

Port and Traverse Point No.	Distance from End of Port (in)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> 0)	Meter Orifi Press ("H <sub>2</sub> Calc,	ce Diff.	Stack Gas Temp. (°F)	Gas S Temp. Gas M (°F	@ Dry cter	Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train (''Hg)
1-1-1		130	155.070	·	1.62	1.62	8 4 6 6 6 6 6 6 6 6 6 6 6 6 6 6 6 6 6 6	75	76	665	72	3.3
	N.	1:35	758.9		1/1	8	5	75.5	1		12	3.3
		1:40	112.6			C381	Ä	7.5			72-	3.3
1-2		1:45	766.5			. 14	200	76		172	7.2-	3.3
	Ŋ.	1:50	770.0			<b>3</b>	. c1/an	76			74	3.3
	5	1:55	773.7			, can	**	76	1/-		12	3.2-
_1-3	,	2:00	717.4		1	C		76	11/	160	73	3.2-
		2:05	781.		1.1	1	1 9	76	V		13	3.2
		2:10	184.7		1.62	\$ C.S.	29 %.	76	76	148	74	3.3

Port and Traverse Point No.	Distance from End of Port (in)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head (''H <sub>2</sub> 0)	Mete Orif Pres (''h	ice s.Diff.	Stack Gas Temp, (°F)	Temp	Sample @ Dry Meter F)	Sample Box Temp. (°F)	Last Impinger Temp: (°F)	Vacuum on Sample Train (''Hg)
1-4		2.15	788,4		162	242	71	76	76		10	3,2
		शरु०	792.1			ĮĮ.	· · · · · · · · · · · · · · · · · · ·	1	76		69	3,2_
	;	Diaz-	Pump b	coke		73, F plants						
		3.27	Restart		•	E. CO. JA		78	178	155	69	3,3
		3:30	795.8		,	N. Carrier		76	18		64	3.5
						40.09						
:		3:35	199.728					78	78		64	3.6
				:				<u> </u>				
2-1		3140	799,728			100		78	7.5	163	68	3.8
		3:45	803.4					7 €	Zê		68	<b>3</b> -3
	,	3:50	807.2			*****		77	78	11.0	68	3.7
22.		3:55	8/0.9		ļ <b>.</b>	200		77	72	166	67	3.9
		4:00	814.6		<b> </b>	100	- , ; 	76.	75		67	4.0
		4:05	818,3		1	T. Taken	',	27	7.8		67	4.2
2-3		4:10	882.2			:	-,	76_	28	178	66	4.2_
		4:15	885,9		7 7		7 / //	76	78		66	4.6
		4:20	829.9			a (		76	177		66	4.9
2-4		4:25	233.1			1.57	- 1	76	72.	153	67	5,8
		4:30	836,9		V	<b>N</b>	1 1/	76	77		67	6.1
		4:35	840.5		1.62	, -	72	26	177		67	6.5
-		· · · · · · · · · · · · · · · · · · ·	S 11 11 0 = 0		g1-325		PåU.	8-0	P			· ·
End	·	4.40	844.308 955.070_		1.6	A.	72	7 4	<del> </del>			
	<u> </u>	u-	452.070_						<del> </del>	<del> </del>		<del> </del>
			89.7.38		<del> </del>				<del> </del>			-
		1						l	<u> </u>	<u></u>		<u> </u>
•												

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## SOURCE SAMPLING FIELD DATA SHEET

Sketch of Stack

Mat'l Processing Rate

Final Gas Meter Reading

934.562

Plant Super Acid

Sampling Location

Date //-/	9071	Run No	.3			1	nitial Gas	Meter 1	keadin,	g <u>844</u>	1. 302	tt~
Time Start_	5.e5_Ti	me End				Т	otal Conden	sate in	n Impi	ngers	14	m1
	me/Point					M	bisture in	Silica	Gel			gmi
	NB °F,				•		Silica Gel C					6
Mbisture_	, Ga, Ga	ıs Density	Factor_				rsat: CO		<u> </u>			
Barometric	Press 'Hg	g, Štack	Press "Hg				02					
Weather	DaRK	. 60	1.0				CO				:	
	°F, W/D		1				$N_2$					
	No. N			· .			Excess					
	1,62 Pitot		,	•			Air			····		
Nozzle Dia.	// in., Pr	obe Leng	th 4 ft	•		r	Cest Conduct	ed By:	F	3/000	+.	
D 1 11 1	0		1					-	<u>بر</u> بر	<u> Man</u> ohns		<del></del>
Stack Dimer	nsions: Insid	le Diamet	er Zo in ft2					-		<u>orny</u>	υ <b>γ</b> ι	
	Insid Heigh	le Area	ft2 Jà ft			·	Remarks:					
	re igi.	··	)			_						
										<del> </del>		
Port and	Distance	Clock	Gas Meter	Stack	Meter	<u> </u>	Stack Gas	Gas S	ample	Sample	Last	Vacuum
Traverse	from End	Time	Reading	Velocity	Orifi	Lce	Temp.	Temp.	@ Dry	Box	Impinger	on
Point No.	of Port		(ft <sup>3</sup> )	Head	Press	Diff.	(°F)	Gas M		Temp.	Temp.	Sample Train
	(in)			(''H <sub>2</sub> 0)		Actual		In	Out	( F)	(°F)	Train ("Hg)
1-1	(4)	5:05	344.3		1.62	1.62	72	76	76		66	2.1
	5	5:10	849.6	-	1.62			25	27	178	67	2.1
	- 12	575	851.8		1.62			2.5	2.7		67	2.1
1-2		5:20	855.5	.	1.62	-	<del>                                     </del>	75	26		65	2.2
	<u></u>	5.25	857.3		1.62	<del>                                     </del>	<del>  </del>	74.5	76		63	2,2
<del></del>	2	5.30	863.		1.62	11	1	74	76	172	62	2.2

.62

Port and Traverse Point No.	Distance from End of Port (in)	Clock Time	Gas Meter Reading (ft <sup>3</sup> )	Stack Velocity Head ("H <sub>2</sub> 0)	Meter Orifice Press.Diff. ("H <sub>2</sub> 0)	Stack Gas Temp, (°F)	Gas Sample Temp.@ Dry Gas Meter (°F)	Sample Box Temp: (°F)	Last Impinger Temp. (°F)	Vacuu on Sampl Train ("Hg)
					Cal. Actual		In Out			L
1-4		556	878.4		1.62 1.62	71	23 75		62	2.2
·		5:55	582.2			21	73 25		62	3.3
	·····	6:00	8 55.7	-		7.	74 75		62	3.3
2-1		6:05	889.2			7.1	74 75	172-	62_	2.2
	· · · · · · · · · · · · · · · · · · ·	6:10	89.4.0			71	77 24		62	0.2
		6:15	898.7		<del>   </del>	7/	73 74	148	62-	2.2
2-2	·		900.9		<del>                                     </del>	70	73 74		62	2,5
			904.5	· ·		70	73 74		67-	2.3
		(	908,3		<u>                                     </u>	20	7.3 74	<u> </u>	62	2.3
2-3			912.2			70	72 74	172_	63	2.3
·			915.9			70	72 74		63	2.3
			919.7		4	70	72 74		63	2.3
2-4/			923.4		11/15//	70	72 73	178	64	2.3
	· <del></del>		927.6			70	72 73	,	64_	2.3
	·	7:00	930.7		1.621.62	70	72 73		64	2.3
	النه ع	7-05	934.562		0 03.	5000	Do U.		ļ	ļ
		<u> </u>	894.300		1.18-	71	Field.			<u> </u>
		/	90.254/	<u> </u>						
		1 (	V /							<u> </u>
		1.	:							
		·								ļ
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APPENDIX C
Standard Analytical Procedures

### ENVIRONMENTAL PROTECTION AGENCY

Research Triangle Park, North Carolina 27711

Reply to Attn of:

Date: 12-21-72

Subject: Summary of Fluoride Analysis

To: R. Neulicht, EMB, IRL

This memorandum is in response to your request for a brief summary of our SPADNS-Zirconium Lake procedure for determination of fluoride in stack emission samples.

Samples received in our laboratory are filtered through fluoride free paper filters to yield water soluble and water insoluble portions. The water insoluble particulate collected on the filter is rinsed throughly to be sure that all water soluble fluoride is rinsed through. The water soluble fraction is distilled from sulfuric acid to a maximum temperature of 180°C. If chloride is suspected in the sample Ag<sub>2</sub>So<sub>4</sub> is added to the still. SPADNS solution is added to an aliquot of the distillate and the absorbance is read at 570 nm. The concentration of the sample is determined from a calibration curve prepared from standard fluoride solutions. It is very important that the temperature of the samples be the same as that of the standards when absorbances are recorded.

The water insoluble fraction of the sample is evaporated to dryness in the presence of a sturry or CAO, and then fused with NACH. The fusate is dissolved with distilled water, neutralized with dilute  $^{\rm H}_2{}^{\rm So}_4$ , distilled and analyzed as described for the soluble portion.

Paper filters containing particulate are cut into small pieces, suspended in a slurry of CAO, evaporated to dryness and ashed prior to the alkali fusion and distillation.

If you have any questions about this procedure, let me know.

Howard Erist

Howard L. Crist
Chief, Source Sample Analysis Section
SSFAB, QAEML

cc: R. E. Lee

APPENDIX D
Project Participants

### PROJECT PARTICIPANTS

# Environmental Engineering, Inc.

Name

<u>Title</u>

John Koogler, Ph.D., P.E.

Project Director

Dennis Falgout

Project Manager

George Allen

Environmental Specialist

Eric Johnson

Environmental Specialist

# Environmental Protection Agency

John Reynolds

Jerome Rom

Roy Neulicht