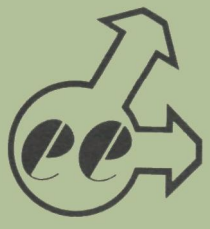
TEST NO. 73 - FRT - 10
C. F. CHEMICALS, INC.
RUN-OF-PILE TRIPLE SUPERPHOSPHATE
PLANT CITY, FLORIDA

SEPTEMBER 14 - 15, 1972



environmental engineering, inc.

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

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Test Conducted By:

Environmental Engineering, Inc.

Contract No. 68-02-0232

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I. INTRODUCTION

Under the direction of the Environmental Protection Agency, Environmental Engineering, Inc. conducted emission tests at the C. F. Chemical phosphate works in Plant City, Florida. On September 14 and 15, 1972, three two-hour test runs were performed on C. F. Chemical's Run-of-Pile Triple Superphosphate production facilities. The purpose of the test was to obtain data for the use of both the Industrial Studies Branch and the Performance Standards Branch of the EPA.

Measurements were made for soluble and insoluble fluorides at the off-gas outlet to the atmosphere. In addition, grab samples of the scrubbing liquids, the process reactants, and the process products were analyzed for fluoride and P_2O_5 content.

A schematic flow diagram of the process operation and the sample location is presented in Figure 1. Pertinent test results are listed in Table 1; complete results are given in Appendix A.

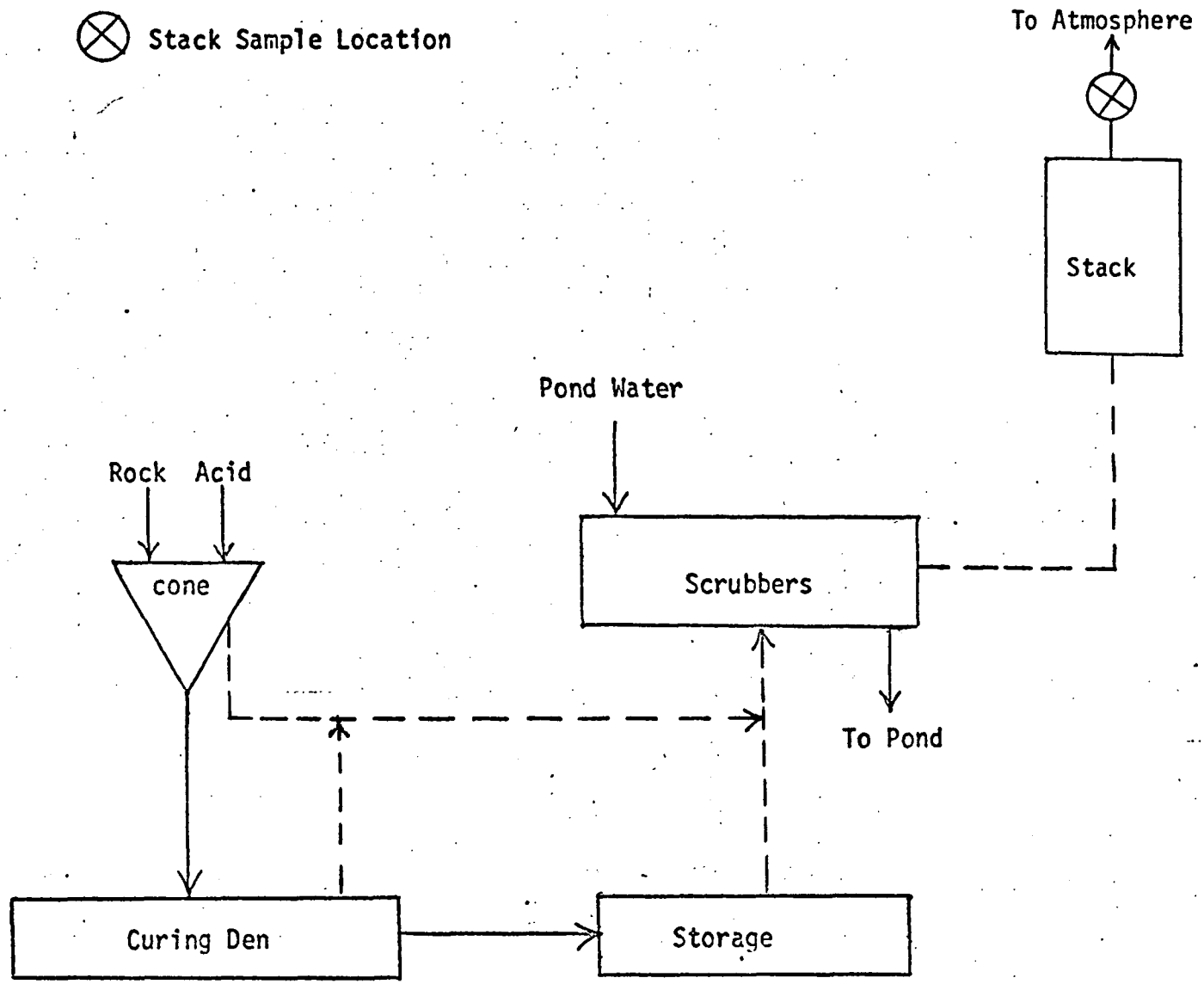


Figure 1
Schematic Flow Diagram
C. F. Chemicals

II. SUMMARY OF RESULTS

The plant was operating under normal process conditions during all of the test runs. During the first test run, the plant shut down for three hours due to a process malfunction. However, advance warning was given and the test was simply stopped prior to the shut down and then resumed when the plant once again achieved normal operation. No other test problems were encountered.

A complete summary of the stack gas conditions and the fluoride emissions for each test run is given in Table 1.

TABLE 1

SUMMARY OF RESULTS
FLUORIDES
C.F. CHEMICALS
R.O.P. OUTLET

Run No.	1	2	3
Date	9-14-72	9-15-72	9-15-72
Barometric pressure, inches Hg	30	30	30
Stack pressure, inches Hg	30	30	30
Stack gas moisture, % volume	4.8	5.1	5.3
Average stack gas temperature, °F.	101	101	103
Stack gas flow rate @ S.T.P.*, SCFM	60485	59578	58156
Volume of gas sampled @ S.T.P.*	88.185	86.482	83.932
Fluoride, water soluble, mg	51	53	54
Fluoride, total, mg	51	53	54
Fluoride, water soluble, gr/SCF	0.009	0.009	0.01
Fluoride, total, gr/SCF	0.009	0.009	0.01
Fluoride, water soluble, gr/CF stk. cond.	0.008	0.009	0.009
Fluoride, total, gr/CF stk. cond.	0.008	0.009	0.009
Fluoride, water soluble, lb/hour	4.6	4.8	4.9
Fluoride, total, lb/hour	4.6	4.8	4.9
Fluoride, water soluble, lb/ton P_2O_5 Fed.	0.1	0.1	0.1
Fluoride, total, lb/ton P_2O_5 Fed.	0.1	0.1	0.1

* Dry, 70°F., 29.92 inches Hg.

III. PROCESS DESCRIPTION

R.O.P. triple superphosphate is made by reacting 32 - 34 % P_2O_5 rock with phosphoric acid in a TVA cone-type continuous mixer to yield a product containing 46% P_2O_5 . The principal reaction is as follows:

$$Ca_3(PO_4)_2 + 4H_3PO_4 + 3H_2O \rightarrow 3CaH_4(PO_4)_2\ H_2O$$

The cone discharges to a slowly moving belt called the "den" on which the reactions continue until the slurry solidifies and is discharged to the storage pile. The reactions go to near completion in the pile where, after sufficient curing, the product is ready for shipment.

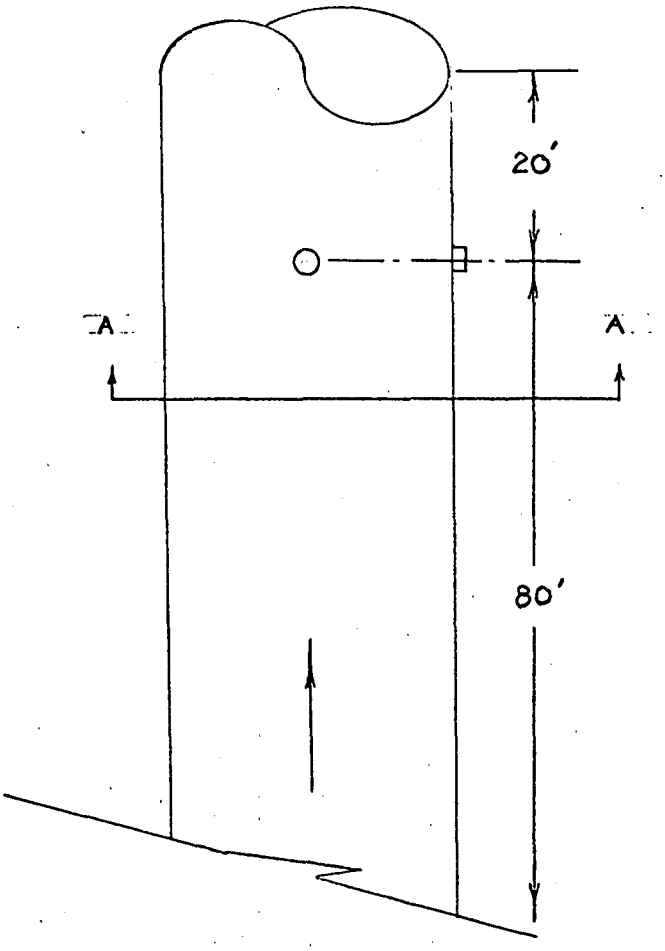
IV. PROCESS OPERATION

Three test runs were conducted; one on Thursday, September 14, from 2:30 pm to 4:30 pm and two on Friday, September 15, from 8:15 am to 10:15 am and from 12:00 pm to 2:00 pm. Plant operation was normal for all three runs.

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 sample location, and the sampling points traversed during the emission tests.



TRAVERSE POINT NO.	DISTANCE FROM INSIDE WALL (FT)
1, 7	0.26
2, 8	0.88
3, 9	1.77
4, 10	4.23
5, 11	5.12
6, 12	5.74

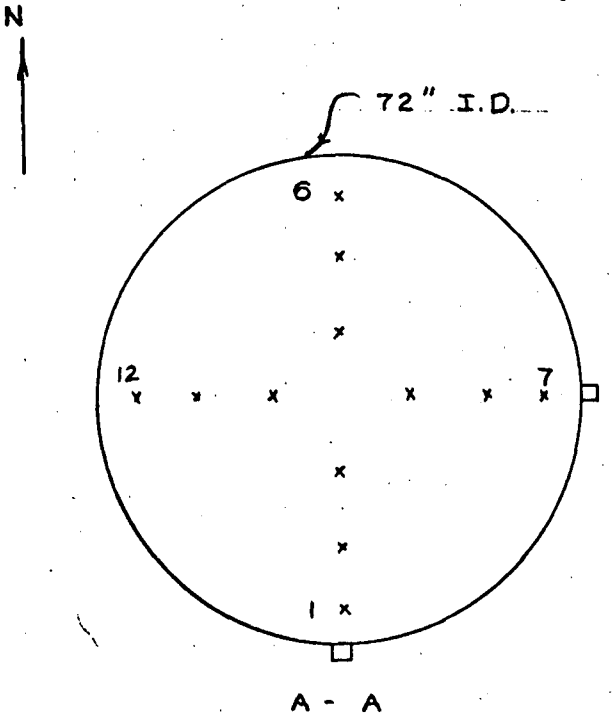


Figure 2

SAMPLE PORT DESCRIPTION

VI. SAMPLING AND ANALYTICAL PROCEDURES

Preliminary Moisture Determination

The preliminary moisture content of the stack gas was determined by wet bulb-dry bulb thermometry as referred to in the Federal Register (Volume 36, Number 247, Part II, December 23, 1971).

Preliminary Velocity Determination

Method 2 of the above referenced Federal Register was used as a guide in determining the preliminary stack gas velocity. The major difference was that only the maximum and minimum velocity heads across the stack area were determined so a proper nozzle size could be selected. During each of the three fluoride emission tests, velocity head readings were taken at points selected by using Method 1 of the Federal Register.

Stack pressure and temperature measurements were also made during the preliminary velocity determination.

Sampling for Fluoride Emissions

The sampling procedure used for determining fluoride emissions was similar to Method 5 of the Federal Register. The major difference between the two methods was the configuration of the sampling train. The sampling train described in the Federal Register 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 carryover. 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 mentioned Federal Register, 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 at least every five minutes, and when significant changes in stack parameters necessitated additional adjustments to maintain an isokinetic flow rate. Nomographs were used to aid in the rapid adjustment of the sampling rate. 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 a single polyethylene container.

Field data sheets are included in Appendix B.

Liquid and Product Grab Samples

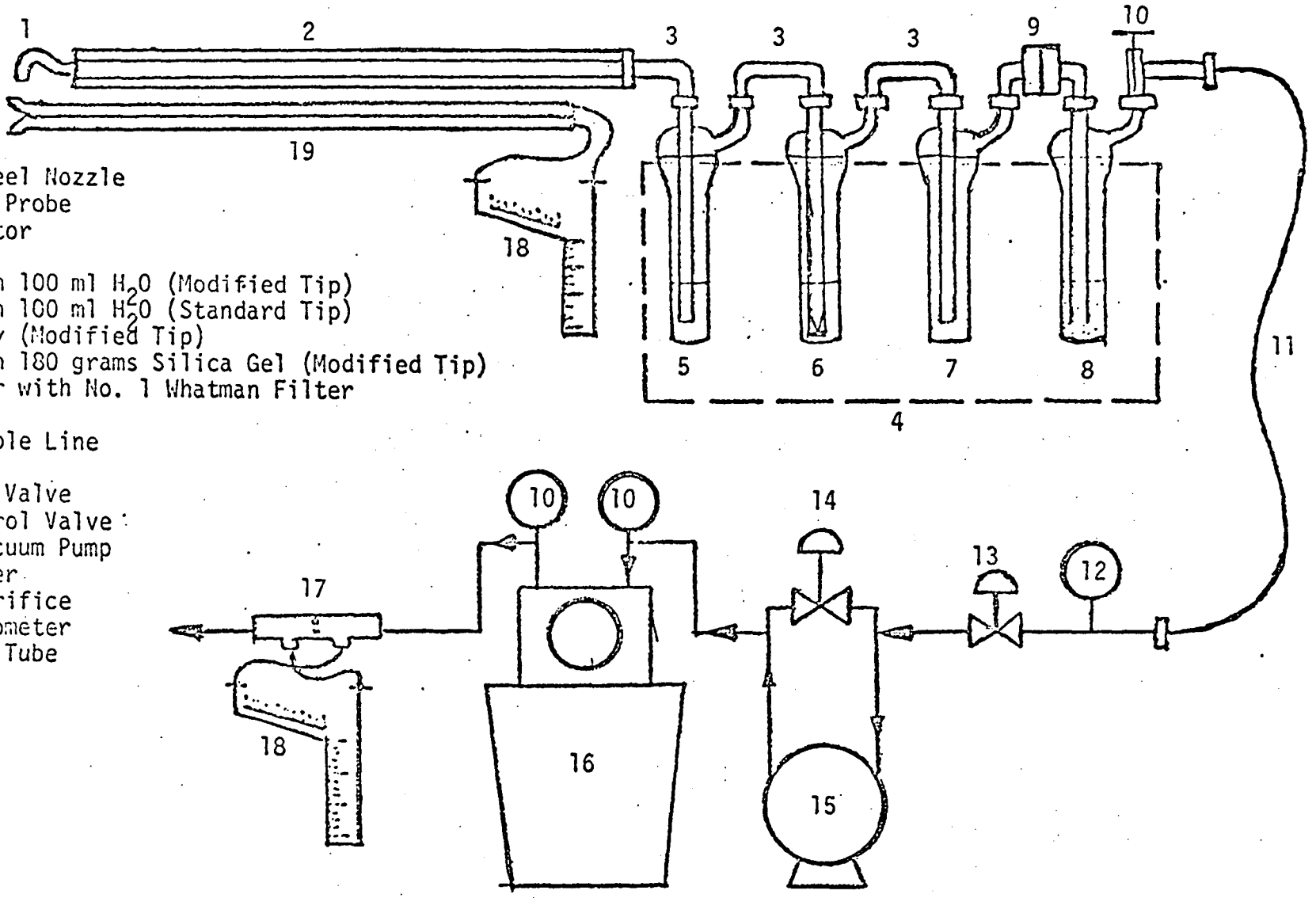
Periodically, during each test run, grab samples of the raw materials, finished product, and scrubber liquid were taken, and the temperature and pH were determined at the site. The samples were split with the plant personnel so that comparative analyses could be performed.

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.

P_2O_5 analysis of the stack effluent was done by EPA personnel. All other P_2O_5 analyses were done by plant personnel.

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



. Stainless Steel Nozzle
. Heated Glass Probe
. Glass Connector
. Ice Bath
. Impinger with 100 ml H_2O (Modified Tip)
. Impinger with 100 ml H_2O (Standard Tip)
. Impinger, Dry (Modified Tip)
. Impinger with 180 grams Silica Gel (Modified Tip)
. Filter Holder with No. 1 Whatman Filter
. Thermometer
. Flexible Sample Line
. Vacuum Gauge
. Main Control Valve
. By-Pass Control Valve
. Air Tight Vacuum Pump
. Dry Test Meter
. Calibrated Orifice
. Inclined Manometer
. S-Type Pitot Tube

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 zer
TS - Stack temperature, °R
TM - Meter temperature, °R
$\overline{H}$ - Average square root of velocity head, $\sqrt{\text{inches } H_2O}$
$\overline{\Delta H}$ - Average meter orifice pressure differential, inches H_2O
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, $\left[PB + \frac{\Delta H}{13.6}\right]$ 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, %
$\sqrt{H \times TS}$ - Average square root of velocity head times stack temperature
$\overline{U}$ - 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)

EQUATIONS FOR CALCULATING FLUORIDE EMISSIONS

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

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

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

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

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

FMOIST = Assumed moisture fraction

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

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

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

$$EA = \left[(100) \times (\% O_2 - \frac{\% CO}{2})\right] \div \left[(o.266 \times \% N_2) - (\% O_2 - \frac{\% CO}{2})\right]$$

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

$$QS = (\underline{U}) \times (AS)$$

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

$$QSTPD = (QD) \times (\frac{530}{29.92}) \times (\frac{PS}{TS})$$

$$PISO = (o.oo267 \times VC \times TS) + (P_o \times TS \times VM \div TM) \div (Time \times \underline{U} \times PS \times AN)$$

Fluoride Emissions:

MG = Milligrams of fluoride from lab analysis

Grains/SCF = (0.01543) x (MG) ÷ VSTPD

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

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

P_2O_5 Fed = Tons/hour, determined from plant data

Lbs/ton P_2O_5 Fed = (lbs/hour) ÷ (Tons/hour P_2O_5 Fed)

SOURCE TEST DATA

TEST NO - NO OF RUNS - 3
PLANT - C.F. CHEMICALS PLANT CITY
SOURCE - R.O.P. OUTLET
TYPE OF PLANT - R.O.P.
CONTROL EQUIPMENT -
POLLUTANTS SAMPLED - FLUORIDE

	1	2	3
1)RUN NUMBER	1	2	3
2)DATE	9/14/72	9/15/72	9/15/72
3)TIME BEGAN	11:30	8:25	11:50
4)TIME END	16:23	10:40	14:00
5)BAROMETRIC PRESSURE, IN HG	30	30	30
6)METER ORIFICE PRESSURE DROP, IN HG	1.7	1.67	1.61
7)VOL DRY GAS, METER COND, CUBIC FEET	91.35	89	88.845
8)AVERAGE GAS METER TEMPERATURE, DEG F	92.8	89.1	104.8
9)VOL DRY GAS, S.T.P. , CUBIC FEET	88.185	86.482	83.932
10)TOTAL H2O COLLECTED, ML	92.9	97.2	98.6
11)VOL H2O VAPOR COLLECTED, S.T.P. , CU FT	4.4	4.61	4.67
12)STACK GAS MOISTURE, PERCENT VOLUME	4.8	5.1	5.3
13)ASSUMED STACK GAS MOISTURE, PCT VOL	5	4	4
14)PERCENT CO2			
15)PERCENT O2			
16)PERCENT CO			
17)PERCENT N2			
18)PERCENT EXCESS AIR	0	0	0
19)MOLECULAR WEIGHT OF STACK GAS, DRY	28.85	28.85	28.85
20)MOLECULAR WEIGHT OF STACK GAS, STK COND	28.33	28.3	28.28
21)STACK GAS SPECIFIC GRAVITY	0.98	0.98	0.98
22)AVG SQUARE ROOT (VEL HEAD), IN H2O	0.686	0.677	0.664
23)AVERAGE STACK GAS TEMPERATURE, DEG F	100.8	100.5	103.3
24)AVG SQUARE ROOT (STK TEMP×VEL HEAD)	16.251	16.039	15.765
25)PITOT CORRECTION FACTOR	0.83	0.83	0.83
26)STACK PRESSURE, IN HG, ABSOLUTE	30	30	30
27)STACK GAS VEL, STACK COND, F.P.M.	2370.7	2341.2	2302.1
28)STACK AREA, SQ FEET	28.27	28.27	28.27
29)EFFECTIVE STACK AREA, SQUARE FEET	28.27	28.27	28.27
30)STACK GAS FLOW RATE, S.T.P. , SCFMD	60485	59578	58156
31)NET TIME OF TEST, MINUTES	120	120	120
32)SAMPLING NOZZLE DIAMETER, INCHES	0.25	0.25	0.25
33)PERCENT ISOKINETIC	100.7	100.3	99.7
34)FLUORIDE - WATER SOLUBLE, MG	51	53	54
35)FLUORIDE - TOTAL, MG	51	53	54
36)FLUORIDE - WATER SOLUBLE, GR/SCF	0.0089	0.0094	0.0099
37)FLUORIDE - TOTAL, GR/SCF	0.0089	0.0094	0.0099
38)FLUORIDE - WATER SOL., GR/CF, STK CND.	0.008	0.0085	0.0088
39)FLUORIDE - TOTAL, GR/CF, STK CND.	0.008	0.0085	0.0088
40)FLUORIDE - WATER SOLUBLE, LB/HOUR	4.6167	4.8188	4.9382
41)FLUORIDE - TOTAL, LB/HOUR	4.6167	4.8188	4.9382
42)P2O5 FED - TONS/HOUR			
43)FLUORIDE - WATER SOL., LB/TON P2O5 FED	0.1247	0.1255	0.125
44)FLUORIDE - TOTAL, LB/TON P2O5 FED	0.1247	0.1255	0.125

S.T.P.↔DRY, 70 DEGREES F, 29.92 INCHES MERCURY

APPENDIX B

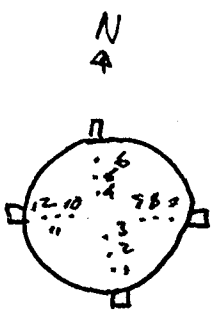
Field Data

[redacted] RON [redacted] TAL [redacted] INE [redacted] G,
Gainesville, Florida

SOURCE SAMPLING FIELD DATA SHEET

Plant CF CHEMICALS PLANT CITY
Sampling Location ROP OUTLET
Date 9-14-72 Run No. 1
Time Start 1110 Time End 16:23
Sampling Time/Point 10 min/pt (12 pts) = 120 min
DB 102 °F, WB ____ °F, VP @ DP ____ "Hg
Bar. Press. 30 "Hg, Stack Press. 30 "Hg
Moisture 5 %, FDA 95, Gas Density Factor 1
Weather SUNNY
Temp. 90 °F, W/D S, W/S 0-10
Sample Box No. S Meter Box No. 1
Meter ΔH@ 1.68 Pitot Corr. Factor 0.83
Nozzle Dia. 0.25 in., Probe Length 8 ft
Probe Heater Setting 420 Nomograph C_f ____
Stack Dimensions 72 in
Stack Area 28.27 ft^2
Stack Height 100 ft

Sketch of Stack



Mat'l Processing Rate ____
Final Gas Meter Reading 762.010 ft^3
Initial Gas Meter Reading 670.660 ft^3
Condensate Increase in Impingers 80 ml
Moisture in Silica Gel ≈ 12.9 gm
Silica Gel Container No. 73 Filter No. 72-11-16

Orsat:				
CO_2				
O_2				
CO				
N_2				
Excess Air				

Test Conducted by: WILSON
NECK

Remarks: * PROCESS WENT DOWN FOR 3 HOURS

Port and Traverse Point No.	Distance From Inside Stack Wall (~~in.~~) (ft)	Clock Time	Gas Meter Reading (ft^3)	Stack Velocity Head ("H_2O)	Meter Orifice Press. Diff. ("H_2O)		Stack Gas Temp. (°F)	Gas Sample Temp.@Dry Gas Meter (°F)		Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual		In	Out			
		1110	670.660									
1	0.26	1120	677.170	0.35	1.28	1.28	102	87	86		88	7
2	0.88	1130	684.410	0.45	1.63	1.63	102	87	86		80	9
*3	1.77	1420 1430 1440	691.925	0.47	1.70	1.70	102	94	94		78	9
4	4.23	1445	699.120	0.42	1.52	1.52	100	94	94		1	9
5	5.12	1455	706.600	0.42	1.52	1.52	100	94	94			9
6	5.79	1505	714.900	0.56	2.00	2.00	100	95	94		82	9½

Port and Traverse Point No.	Distance From Inside Stack Wall (in.)	Clock Time	Gas Meter Reading (ft^3)	Stack Velocity Head ("H_2O)	Meter Orifice Press. Diff. ("H_2O)		Stack Gas Temp. (°F)	Gas Sample Temp.@ Dry Gas Meter (°F)		Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual		In	Out			
		1520										
7	0.26	1530	722.63	.47	1.70	1.70	100	94	94		82	9
8	0.88	1540	730.96	.50	1.80	1.80	100	94	94			9
9	1.77	1550	738.48	.53	1.90	1.90	100	94	94			9 ½
		1553										
10	4.23	1603	746.44	.50	1.80	1.80	102	94	94			9
11	5.12	1613	754.28	.50	1.80	1.80	102	94	94			9
12	5.74	1623	762.01	.50	1.80	1.80	102	94	94			9

[redacted] ENVIRONMENTAL [redacted] ENGINEERING, [redacted] INC.
Gainesville, Florida

SOURCE SAMPLING FIELD DATA SHEET

Plant C F CHEMICAL
Sampling Location PLANT CITY (ROP OUTLET)
Date 9-15-72 Run No. 2
Time Start 0825 Time End 1040
Sampling Time/Point 12 @ 10 min ea. = 120 min
DB 100 °F, WB ____ °F, VP @ DP ____ "Hg
Bar. Press. 30 "Hg, Stack Press. 30 "Hg
Moisture 4%, FDA 96, Gas Density Factor 1
Weather SUNNY - CLEAR
Temp. 80 °F, W/D SE, W/S 0-3 mph
Sample Box No. ____ Meter Box No. 5
Meter ΔH@ 1.68 Pitot Corr. Factor .83
Nozzle Dia. 1/4 in., Probe Length 8 ft
Probe Heater Setting 30 Nomograph C_f .92
Stack Dimensions 72" ϕ in
Stack Area ____ ft²
Stack Height ____ ft

Sketch of Stack

Mat'l Processing Rate ____
Final Gas Meter Reading 851.9 ft³
Initial Gas Meter Reading 762.9 ft³
Condensate Increase in Impingers 83 ml
Moisture in Silica Gel 14.2 gm
Silica Gel Container No. 66 Filter No. 72-16-72

Orsat:					
CO_2					
O_2					
CO					
N_2					
Excess Air					

Test Conducted by: A.L. WILSON
S. NECK

Remarks: ____

Port and Traverse Point No.	Distance From Inside Stack Wall (in.) (Ft.)	Clock Time	Gas Meter Reading (ft^3)	Stack Velocity Head ("H_2O)	Meter Orifice Press. Diff. ("H_2O)		Stack Gas Temp. (°F)	Gas Sample Temp. @ Dry Gas Meter (°F)		Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual		In	Out			
7	.126	0835	770.30	.47	1.70	1.70	100	79	78		80	9"
8	.88	0845	778.325	.56	2.0	2.0	100	81	79		76	13
9	1.77	0858	786.425	.56	2.0	2.0	100	83	80		76	14
10	4.23	0910	793.72	.43	1.57	1.57	100	86	81		76	13
11	5.12	0920	800.70	.43	1.57	1.57	100	89	83		80	13
12	5.74	0930	807.72	.43	1.57	1.57	100	91	85		82	13

Port and Traverse Point No.	Distance From Inside Stack Wall ~~(in.)~~ (Ft)	Clock Time 0940	Gas Meter Reading (ft^3)	Stack Velocity Head ("H_2O)	Meter Orifice Press. Diff. ("H-O)		Stack Gas Temp. (°F)	Gas Sample Temp.@ Dry Gas Meter (°F)		Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual		In	Out			
1	.26	0950	814.76	.36	1.30	1.30	100	93	88		85	9
2	.88	1000	822.70	.56	2.0	2.0	100	96	89		"	13
3	1.77	1010	830.90	.56	2.0	2.0	100	98	91		"	13
4	4.23	1020	837.40	.33	1.2	1.2	102	100	93		"	9
5	5.12	1030	844.26	.38	1.37	1.37	102	102	94		"	10
6	5.74	1040	851.90	.48	1.73	1.73	102	104	96		"	11

E[redacted]ONM[redacted]AL E[redacted]NEE[redacted], I[redacted]
Gainesville, Florida

SOURCE SAMPLING FIELD DATA SHEET

Plant C.F. CHEMICAL - PLANT CITY
Sampling Location ROP OUTLET
Date 9-15-72 Run No. 3
Time Start 1150 Time End 1400
Sampling Time/Point 12@10 min ea. = 120 min.
DB 100 °F, WB ____ °F, VP @ DP ____ "Hg
Bar. Press. 30 "Hg, Stack Press. 30 "Hg
Moisture 4 %, FDA 96, Gas Density Factor 1
Weather SUNNY - HOT
Temp. 90 °F, W/D NW, W/S NW 0-3
Sample Box No. ____ Meter Box No. 5
Meter ΔH@ 1.68 Pitot Corr. Factor .83
Nozzle Dia. 1/4 in., Probe Length 8 ft
Probe Heater Setting 40 Nomograph C_f .92
Stack Dimensions 72" ϕ in
Stack Area ____ ft^2
Stack Height ____ ft

Sketch of Stack

Mat'l Processing Rate ____
Final Gas Meter Reading 940.900 ft^3
Initial Gas Meter Reading 852.055 ft^3
Condensate Increase in Impingers 84 ml
Moisture in Silica Gel 19.6 gm
Silica Gel Container No. 71 Filter No. 72-16-20

Orsat:					
CO_2					
O_2					
CO					
N_2					
Excess Air					

Test Conducted by: A.L. WILSON
S. NECK

Remarks: ____

Port and Traverse Point No.	Distance From Inside Stack Wall ~~(in.)~~ (FT)	Clock Time	Gas Meter Reading (ft^3)	Stack Velocity Head ("H_2O)	Meter Orifice Press. Diff. ("H_2O)		Stack Gas Temp. (°F)	Gas Sample Temp. @Dry Gas Meter (°F)		Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual		In	Out			
1	.26	1200	859.13	.40	1.45	1.45	103	104	103	—	85	12
2	.88	1210	867.00	.53	1.90	1.90	103	106	102			13
3	1.77	1220	875.20	.56	2.0	2.0	103	107	102	-		15
4	4.23	1230	881.64	.30	1.1	1.1	102	106	103			11
5	5.12	[illegible]	887.28	.35	1.28	1.28	102	106	103			12
6	5.74	1250	895.665	.44	1.60	1.60	103	106	103			12

Port and Traverse Point No.	Distance From Inside Stack Wall ~~(in.)~~ (FT)	Clock Time 1300	Gas Meter Reading (ft^3)	Stack Velocity Head ("H_2O)	Meter Orifice Press. Diff. ("H-O)		Stack Gas Temp. (°F)	Gas Sample Temp. @ Dry Gas Meter (°F)		Sample Box Temp. (°F)	Last Impinger Temp. (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual		In	Out			
7	.26	1310	903.08	.42	1.53	1.53	104	107	106			12
8	.88	1320	911.33	.56	2.0	2.0	104	107	105			13
9	1.77	1330	919.85	.60	2.15	2.15	104	106	105			14
10	4.23	1340	927.05	.42	1.53	1.53	104	105	105			12
11	5.12	1350	934.22	.42	1.53	1.53	104	105	105			12
12	5.74	1400	940.900	.35	1.28	1.28	104	103	104			11

GRAB SAMPLE DATA SHEET

Plant C F Ind

EPA Sample No. 72-000-	614			
Run No.	1	1	1	
Date	9/14/72			
Time	11:30 am	2:45 pm	3:45 pm	
Sampling Point	Pond Water Feed	→	→	Composite
Temperature, °F	93	97	91	95
pH	1.5	1.5	1.5	1.5
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant CF Ind

EPA Sample No. 72-000-	615			
Run No.	1	1	1	
Date 9/14/72				
Time 11:30	11:30	2:45	3:45	
Sampling Point Process	Primary Scrubber	→	→	Composite
Temperature, °F	90	92	92	92
pH	1.5	1.5	1.5	1.5
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant C-F Ind.

EPA Sample No. 72-000-	616			
Run No.	1	1	1	
Date	9/14/72			
Time	11:30 am	2:45 pm	3:45	
Sampling Point	Process Secondary Scrubber	→	→	Composite
Temperature, °F	87	90	90	90
pH	1.5	1.5	1.5	1.5
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant C-F Ind

EPA Sample No. 72-000-	617			
Run No.	1	1	1	
Date	9/14/72			
Time	11:30	2:45	3:45	
Sampling Point	Storage Scrubber	→	→	Composite
Temperature, °F	90	98	98	98
pH	1.5	1.5	1.5	1.5
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant CF Ind

EPA Sample No. 72-000-	619			
Run No. Fluoride	1	1	1	1
Date 9/14/72				
Time 2 pm.				
Sampling Point	Acid Feed	Rock Feed	Product	
Temperature, °F				
pH				
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant C-F Ind.

EPA Sample No. 72-000-	622			
Run No.	2			
Date	9/15/22			
Time	8:15 am →	10:30 am		
Sampling Point	Pond water Feed			
Temperature, °F	80	88	88	88
pH	1.5	1.45	1.8	1.5
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant C.F Ind.

EPA Sample No. 72-000-	623			
Run No.	2		1	
Date	9/15/72			
Time	8:15 → 10:30 am			
Sampling Point	Process Primary Scrubber			
Temperature, °F	90	90	92	90
pH	1.5	1.45	1.5	1.5
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant C.F Industries

EPA Sample No. 72000-	624			
Run No.	2			
Date	9/15/72			
Time	8:15 10:15 a.m.			
Sampling Point	Process Secondary Scrubber			
Temperature, °F	87	89	90	87
pH	1.5	1.45	1.5	1.5
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant C.F. Ind.

EPA Sample No. 72-000-	625			
Run No.	2			
Date	9/15/72			
Time	9:15 → 10:30 am			
Sampling Point	Storage Scrubber			
Temperature, °F	94	97	97	97
pH	1.5	1.45	1.5	1.5
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant CF Ind.

EPA Sample No. 72000-	626			
Run No.	2			
Date	9/15/72			
Time	9:15am			
Sampling Point	Product	Acid Feed	Rock Feed	
Temperature, °F				
pH				
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant _____

EPA Sample No. 72-000-	630			
Run No.	3			
Date	9/15/72			
Time	11:45 am →	2 pm		
Sampling Point	Pond Water Inlet			
Temperature, °F				
pH				
Fluorides				
P_2O_5				
Trace Metals				

Remarks _____

GRAB SAMPLE DATA SHEET

Plant C.F. Ind

EPA Sample No. 72-000-	631			
Run No.	3			
Date	9/15/72			
Time	11:45 am →	2 pm.		
Sampling Point	Process Primary Scrubber			
Temperature, °F	92			
pH	1.5			1.5
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant C.F. Ind

EPA Sample No. 72-000-	632			
Run No.	3			
Date	9/15/72			
Time	11AS am →	2 pm		
Sampling Point	Process Secondary Scrubber			
Temperature, °F				
pH				
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant CF Ind

EPA Sample No. 72-000-	633			
Run No.	3			
Date	9/15/72			
Time	11:45 a.m. → 2 p.m.			
Sampling Point	Storage Scrubber			
Temperature, °F				
pH				
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant CF Ind.

EPA Sample No. 72-000-	634	635	636	
Run No.	3		1	
Date	9/15/72			
Time	1 p.m.			
Sampling Point	Product	Feed Acid	Feed Rock	
Temperature, °F				
pH				
Fluorides				
P_2O_5				
Trace Metals				

Remarks

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_2So_4 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 slurry of CAO, and then fused with NAOH. The fusate is dissolved with distilled water, neutralized with dilute H_2So_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 Crist

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

cc: R. E. Lee

Phosphorous Pentoxide Determination

Colorimetric Molybdovanadophosphate Method

An aliquot of sample is hydrolyzed in the presence of HCl and HNO_3 acids by boiling almost to dryness.

The sample is cooled to room temperature, transferred to a 250 ml volumetric flask and diluted to volume with distilled water. A 20 ml aliquot is transferred to a 100 ml volumetric flask, 20 ml of molybdovanadate reagent is added and the flask is diluted to volume.

The absorbance of the yellow color is determined after ten minutes at 400 nm. The concentration of phosphorous pentoxide is determined from a calibration curve prepared with standard solutions.

APPENDIX D

Project Participants

PROJECT PARTICIPANTS

Environmental Engineering, Inc.

Name	Title
John Dollar, E.I.T., M.S.E.	Project Manager
George Allen	Environmental Specialist
Marvin Hamlin	Environmental Specialist
Steve Neck	Environmental Specialist
A. L. Wilson	Environmental Specialist

Environmental Protection Agency

Roy Neulicht

John Reynolds