TEST NO. 72 - CI - 1
W. R. GRACE AND COMPANY
WET PROCESS PHOSPHORIC ACID
BARTOW, FLORIDA

JANUARY 4 - 5, 1972

environmental engineering, inc.

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Test Conducted By:

Environmental Engineering, Inc.

Contract # CPA - 70 - 82

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INTRODUCTION

Under the direction of the Environmental Protection Agency, Environmental Engineering, Inc., conducted emission tests at the W. R. Grace and Company wet process phosphoric acid plant located in Bartow, Florida. Three test runs were conducted on January 4 - 5, 1972. The purpose of the test was to obtain data to be used by both the Industrial Studies Branch and the Performance Standards Branch of the EPA.

Measurements for soluble and insoluble fluorides were made in the inlet ducts and outlet stack of the crossflow scrubber. The inlet leading from the attack tank and the inlet leading from the filtration system were sampled. Numerous grab samples of the process reactants and products were taken and analyzed for fluoride and P_2O_5 content. A schematic diagram of the process flow and all sampling locations is given in Figure 1.

Pertinent results of the test are listed in Tables 1 - 3; complete test results are given in Appendix A.

SUMMARY OF RESULTS

Although no major problems were encountered with process operation, some of the filter pans were not dumping dry. This condition had existed for some time before the tests were begun. Other operating conditions were typical of normal operation. There were no apparent irregularities in the actual sampling or analytical procedures.

For runs one, two, and three, respectively, the scrubber inlet from the

attack tank showed fluoride emissions of 0.033, 0.182, and 0.187 lb/ton P_2O_5 fed. Emissions at the inlet from the filtration system were 0.031, 0.025, and 0.024 lb/ton P_2O_5 fed, while at the scrubber outlet to the atmosphere, total fluoride emissions were 0.004, 0.008, and 0.004 lb/ton P_2O_5 fed. From this data, scrubber efficiency was calculated to be 93.3%, 96.0%, and 97.7% for runs one, two, and three, respectively.

Tables 1 - 3 give a complete summary of stack gas conditions, sample volumes collected, and fluoride emissions.

PROCESS DESCRIPTION

The first step in the basic process involves mixing phosphate rock, sulfuric acid, and water to form a reaction slurry.

The basic reaction is the acidulation of tricalcium phosphate in the rock with sulfuric acid and water to produce phosphoric acid and calcium sulfate dihydrate (gypsum). The reaction is:

$$Ca_3(PO_4)_2 + 3H_2SO_4 + 6H_2O \rightarrow 2H_3PO_4 + 3CaSO_4 \cdot 2H_2O$$

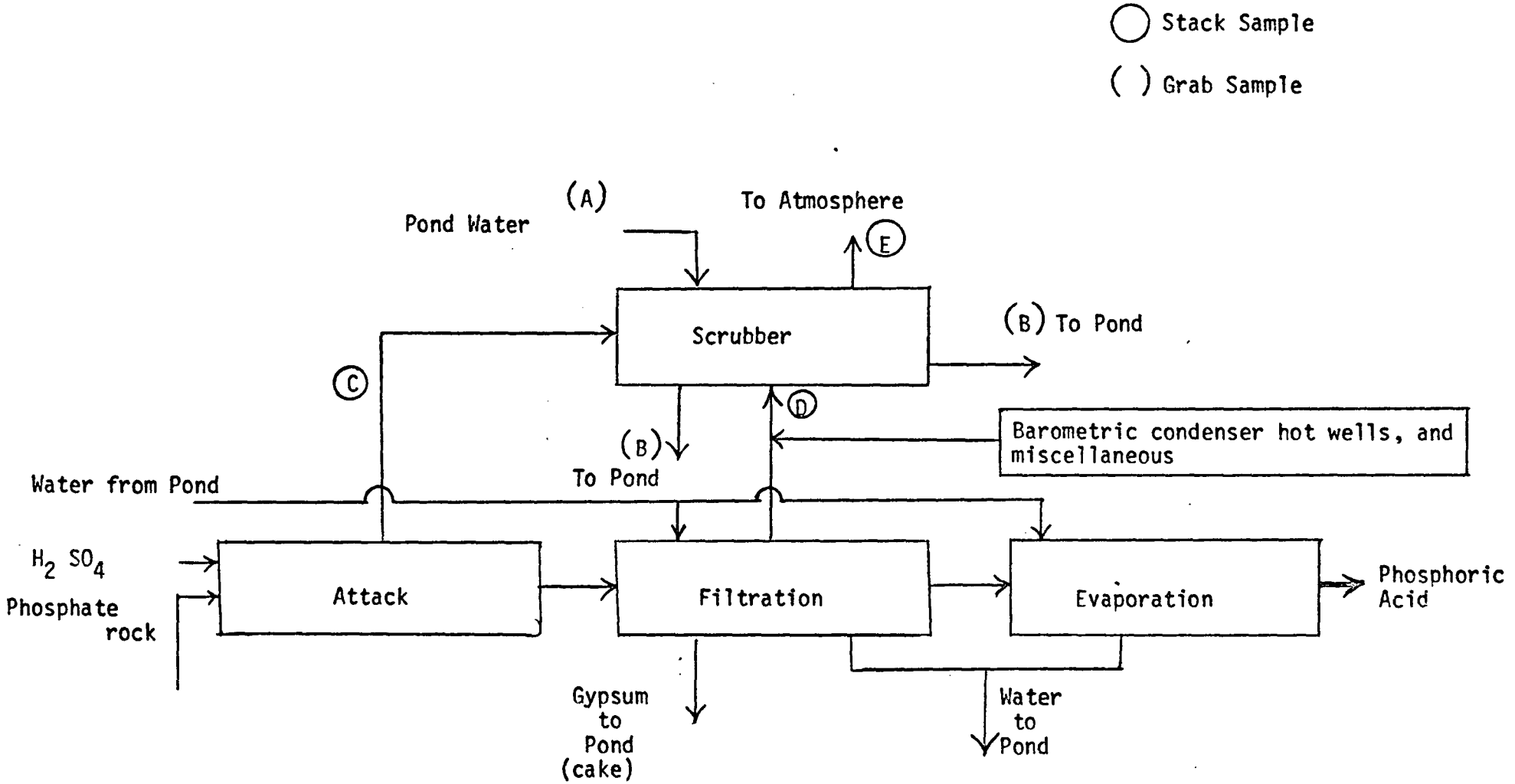
Hydrogen fluoride gas (HF) is produced by a side reaction between the fluorine in the rock and sulfuric acid. HF subsequently reacts with the silicates in the digesting slurry to form fluosilicic acid as follows:

$$CaF_2 + H_2SO_4 + 2H_2O \rightarrow CaSO_4 \cdot 2H_2O + 2HF$$

$$6\ HF + SiO_2 \rightarrow H_2SiF_6 + 2H_2O$$

The fluosilicic acid in turn can decompose:

$$H_2SiF_6 + \text{heat and/or Acid} \rightarrow SiF_4 + 2\ HF$$



Schematic Flow Diagram with Locations of Sampling Stations

W. R. GRACE

Wet Phosphoric Acid

Figure 1

TABLE 1

SUMMARY OF RESULTS
FLUORIDES

STATION C
Scrubber Inlet from Attack Tank

Run No.	1	2	3
Date	1-4-72	1-5-72	1-5-72
Barometric pressure, inches Hg	29.92	29.92	29.92
Stack pressure, inches Hg	29.92	29.92	29.92
Stack gas moisture, % volume	10.4	28.2	27.2
Average stack gas temperature, °F.	150	151	154
Stack gas flow rate @ S.T.P., SCFM	5813	4867	5010
Vol. dry gas @ S.T.P.*, SCF	78.951	97.143	83.965
Fluoride, water soluble, mg	106.95	837	603
Fluoride, total, mg	106.95	837	737
Fluoride, water soluble, gr/SCF	0.020	0.132	0.108
Fluoride, total, gr/SCF	0.020	0.132	0.132
Fluoride, water soluble, gr/CF stk. cond.	0.016	0.082	0.067
Fluoride, total, gr/CF stk. cond.	0.016	0.082	0.082
Fluoride, water soluble, lb/hour	1.01	5.51	4.62
Fluoride, total, lb/hour	1.01	5.51	5.65
Fluoride, water soluble, lb/ton P_2O_5 Fed.	0.033	0.182	0.153
Fluoride, total, lb/ton P_2O_5 Fed.	0.033	0.182	0.187
Scrubber efficiency, %			

* Dry, 70°F., 29.92 inches Hg.

TABLE 2

SUMMARY OF RESULTS
FLUORIDES

STATION D
Scrubber Inlet from Filtration System

Run No.	1	2	3
Date	1-4-72	1-5-72	1-5-72
Barometric pressure, inches Hg	30	30	30
Stack pressure, inches Hg	29.68	29.68	29.68
Stack gas moisture, % volume	6.6	6.2	6.9
Average stack gas temperature, °F.	103	103	105
Stack gas flow rate @ S.T.P., SCFM	11686	11231	11407
Vol. dry gas @ S.T.P.*, SCF	77.449	77.631	78.343
Fluoride, water soluble, mg	47.7	39.165	38.1
Fluoride, total, mg	47.7	39.755	38.1
Fluoride, water soluble, gr/SCF	0.009	0.008	0.007
Fluoride, total, gr/SCF	0.009	0.008	0.007
Fluoride, water soluble, gr/CF stk. cond.	0.008	0.007	0.006
Fluoride, total, gr/CF stk. cond.	0.008	0.007	0.006
Fluoride, water soluble, lb/hour	0.92	0.71	0.71
Fluoride, total, lb/hour	0.92	0.75	0.71
Fluoride, water soluble, lb/ton P_2O_5 Fed.	0.031	0.025	0.024
Fluoride, total, lb/ton P_2O_5 Fed.	0.031	0.025	0.024
Scrubber efficiency, %			

* Dry, 70°F., 29.92 inches Hg.

TABLE 3

SUMMARY OF RESULTS
FLUORIDES

STATION E
Scrubber Outlet

Run No.	1	2	3
Date	1-4-72	1-5-72	1-5-72
Barometric pressure, inches Hg	30	30	30
Stack pressure, inches Hg	30	30	30
Stack gas moisture, % volume	4.9	5.2	5.2
Average stack gas temperature, °F.	93	94	96
Stack gas flow rate @ S.T.P., SCFM	15981	16832	16825
Vol. dry gas @ S.T.P.*, SCF	55.057	63.498	63.282
Fluoride, water soluble, mg	3.6	7.2	3.535
Fluoride, total, mg	3.6	7.2	3.609
Fluoride, water soluble, gr/SCF	0.001	0.002	0.001
Fluoride, total, gr/SCF	0.001	0.002	0.001
Fluoride, water soluble, gr/CF stk. cond.	0.001	0.002	0.001
Fluoride, total, gr/CF stk. cond.	0.001	0.002	0.001
Fluoride, water soluble, lb/hour	0.13	0.25	0.12
Fluoride, total, lb/hour	0.13	0.25	0.12
Fluoride, water soluble, lb/ton P_2O_5 Fed.	0.004	0.008	0.004
Fluoride, total, lb/ton P_2O_5 Fed.	0.004	0.008	0.004
Scrubber efficiency, %	93.3	96.0	97.7

* Dry, 70°F., 29.92 inches Hg.

W.R. GRACE & COMPANY

FUME SCRUBBER - WET PROCESS PHOSPHORIC ACID

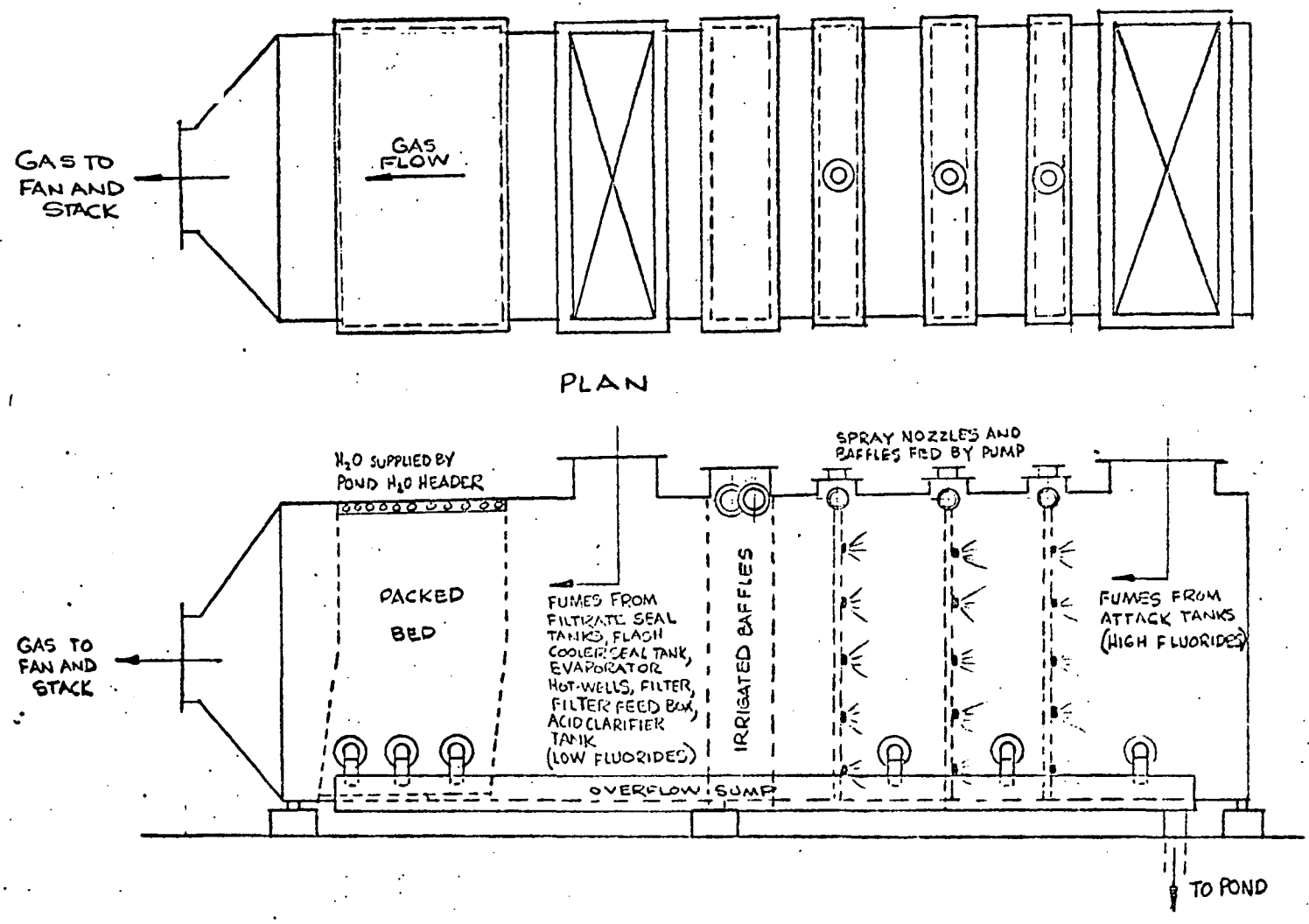


Figure 2

The reactor consists of a series of tanks with the slurry alternately overflowing and underflowing from one compartment to the next. The multi-compartment design allows temperature and agitation to vary throughout the reaction sequence as slurry recirculates through the tank arrangement. The acidulation or digestion step is a highly exothermic reaction requiring considerable apparatus for cooling. A vacuum flash cooler maintains temperature in the reactor and degasifies the recirculated slurry of dissolved air, carbon dioxide, and fluorides.

The acid slurry from the last attack compartment of the reactor is pumped to a rotating-tilting-pan filter where the phosphoric acid is filtered from the gypsum. The byproduct gypsum is repulped and pumped to a nearby pond. The product acid is pumped to a storage vessel and then to vacuum evaporators in which the acid can be concentrated from 30 to 54% P_2O_5.

The crossflow scrubber design has a primary scrubbing section consisting of countercurrent sprays of gypsum pond water. The gases then pass through a section of irrigated baffles before flowing through the packing in the secondary scrubbing section.

PROCESS OPERATION

Run 1 was conducted from 2:15 p.m. to 4:15 p.m. on January 4, 1972, on WRG's "V" train phosphoric acid plant. This test involved three sampling points (2 inlets and one outlet). Process operation was normal except that some filter pans were dumping wet. This condition had existed for several days.

Less than optimum filtration continued throughout Run II, which was begun at 9:00 a.m. on January 5. Other process conditions were essentially normal. No. 1 and 2 evaporators were brought on stream at the beginning of the run, thereby doubling the production rate of 54% acid. With all four evaporators running, the production of fluosilicic acid was also increased.

Scrubber conditions changed slightly during Run II. The booster water pump discharge pressure decreased from 42 psig during Run I to 40 psig just prior to Run II. This was caused by reduced pond water header pressure. Pressure drop through the scrubber remained constant at 5" H_2O during Run II.

The third run got underway at 12:00 p.m. the same day. Process conditions remained basically the same as for Run II except that evaporation rates were increased slightly.

LOCATION OF SAMPLING POINTS

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

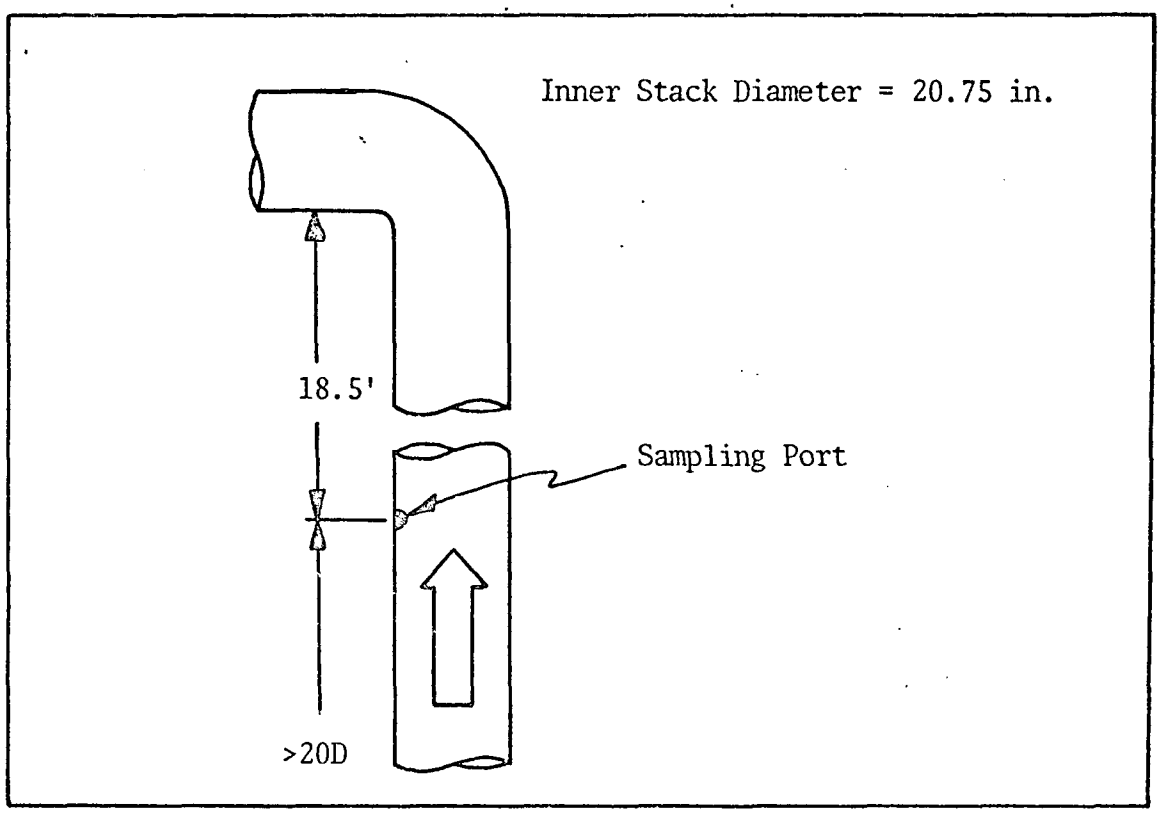
The above method suggests using two perpendicular diameters of traverse points per sampling station; however, on-site conditions necessitated the use of only one traverse diameter. The suggested number of traverse points per diameter was used where possible without sampling within one inch of the inner wall.

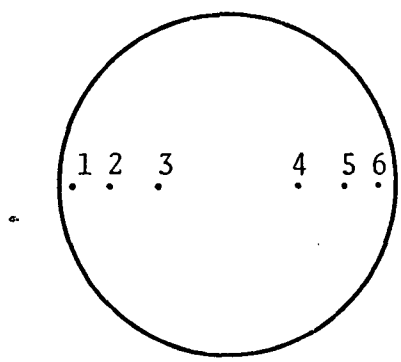
Figures 3 through 5 are schematic diagrams of the stack configuration near the sampling location, and the sampling points traversed during the emission tests.

TABLE 4

DESCRIPTION OF SAMPLING POINTS

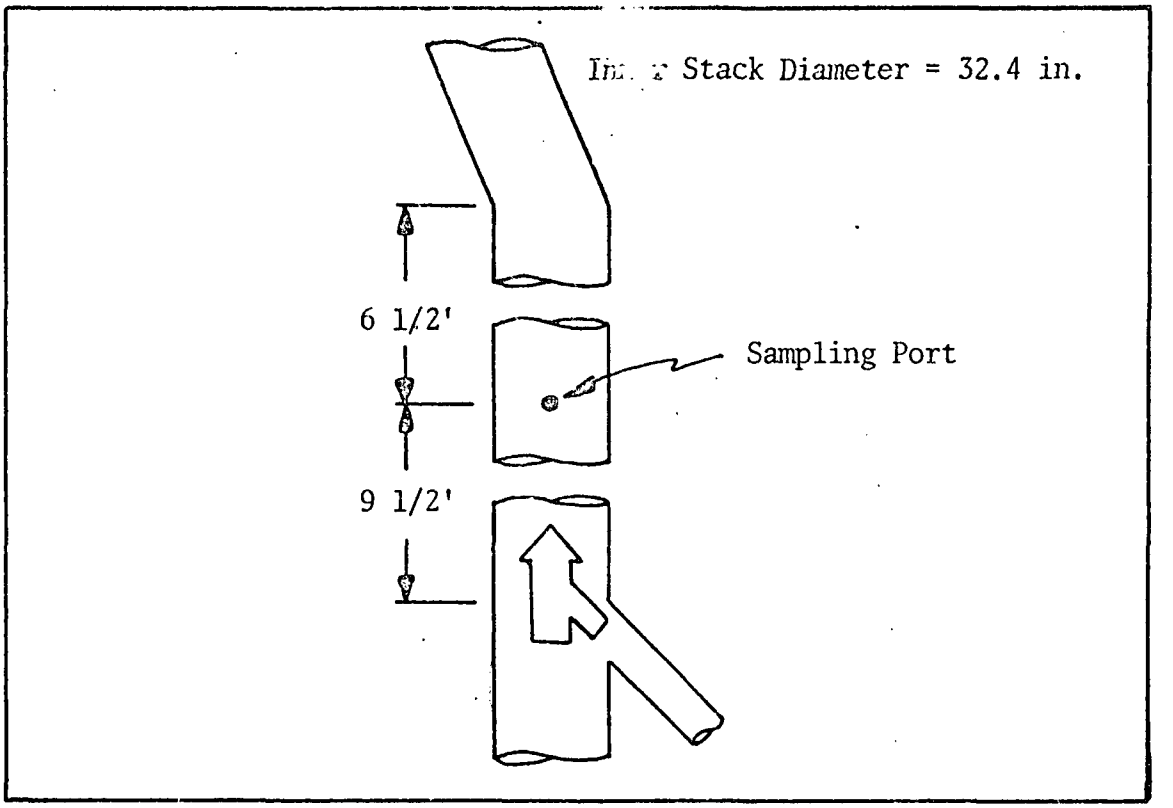
Sampling Point Description	Sampling Point Identification	Number of Traverse Points	Stack Diameter (Ft.)
Attack tank effluent	C	6	1.72
Filtration effluent	D	20	2.70
Scrubber outlet	E	6	3.9

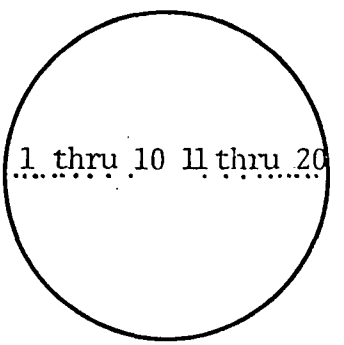




Sampling Point	Distance From Inner Wall (in.)
1	1
2	3
3	6
4	14 5/8
5	17 3/4
6	19 3/4

FIGURE 3
LOCATION OF PORT AND SAMPLING POINTS
STATION C, W.R. GRACE

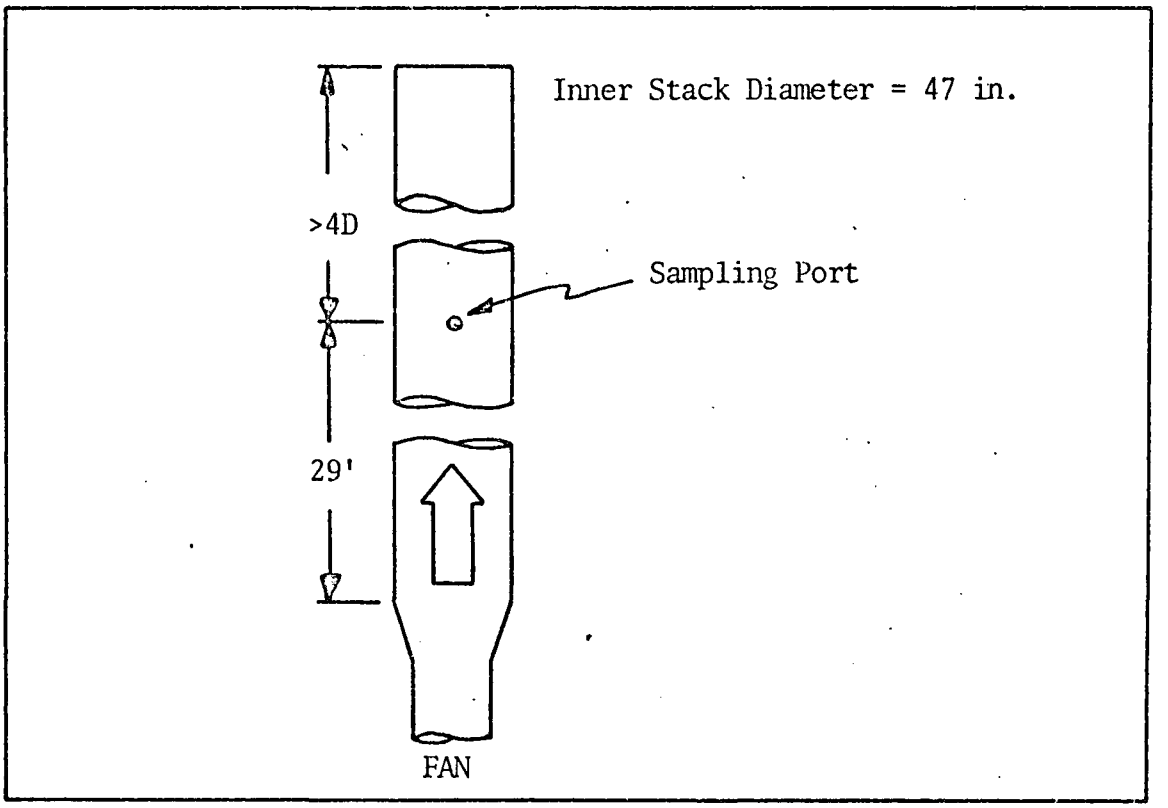


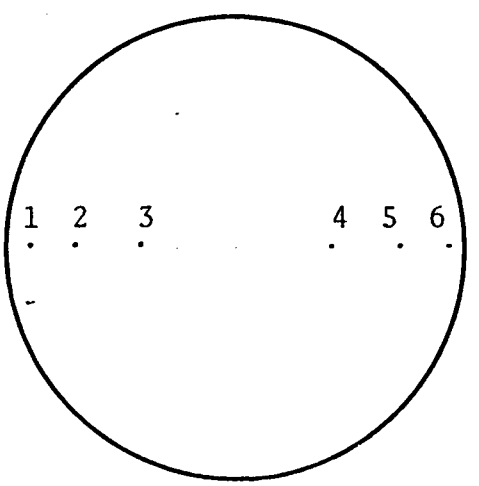


Sampling Point	Distance from Inner Wall (in.)
1	1 1/8
2	1 15/16
3	2 13/16
4	3 3/4
5	4 23/32
6	5 27/32
7	7 1/16
8	8 15/32
9	10 7/32
10	12 3/4
11	19 11/16
12	22 3/16
13	23 15/16
14	25 11/32
15	26 9/16
16	27 11/16
17	28 21/32
18	29 19/32
19	30 15/32
20	31 1/8

FIGURE 4

LOCATION OF PORT AND SAMPLING POINTS
STATION D, W.R. GRACE





Sampling Point	Distance From Inner Wall (in.)
1	2 1/16
2	6 7/8
3	13 7/8
4	33 1/8
5	40 3/32
6	44 15/16

FIGURE 5

LOCATION OF PORT AND SAMPLING POINTS

STATION E, W.R. GRACE

SAMPLING AND ANALYTICAL PROCEDURES

A. Preliminary Moisture Determination

The preliminary moisture content of the stack gases at each sampling site was determined by Method 4 of the Federal Register (Volume 36, Number 247, Part II, December 23, 1971).

The only significant difference between F.R. Method 4 and the method used was the configuration of the sampling train (see Figure 6). The sampling train used in these tests consisted of the first two midget impingers with 5 grams of distilled-deionized water followed by two dry midget impingers in place of a silica gel cartridge.

At sampling sites where liquid entrainment was a problem, the preliminary and final moisture contents were determined from wet and dry bulb thermometry. See Appendix B for the data used in determining the preliminary moisture content of the stack gases.

After completing the moisture run, the total impinger liquid plus water rinsings of the probe tip through the fourth impinger were placed in an 8 ounce polyethylene container. The samples were held by EPA personnel for further analyses.

Field data sheets are contained in Appendix B.

B. Preliminary Velocity Determination

Method 2 of the above mentioned Federal Register was used as a guide in determining the preliminary stack gas velocity for each source tested. The major difference was that only the maximum and minimum velocity heads

across each stack area were determined so that 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 determinations.

C. 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 7 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 polyethylene containers. During some runs the different sample fractions were placed in separate containers, while during others, all of the recovered sample was placed into one container.

D. 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. On some occasions, the samples were split with the plant personnel so that comparative analyses could be performed. Field data sheets are included in Appendix B.

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.

1. Heated Glass Probe With Pyrex Wool as Filter
2. Glass Connector
3. Ice Bath
4. Midget Impinger With 5 Grams H_2O
5. Midget Impinger With 5 Grams H_2O
6. Midget Impinger, Dry
7. Midget Impinger, Dry
8. Flexible Sample Line
9. Vacuum Gauge
10. Main Control Valve
11. By-Pass Control Valve
12. Air Tight Vacuum Pump
13. Dry Test Meter
14. Thermometer
15. Calibrated Orifice
16. Inclined Manometer
17. S-Type Pitot Tube

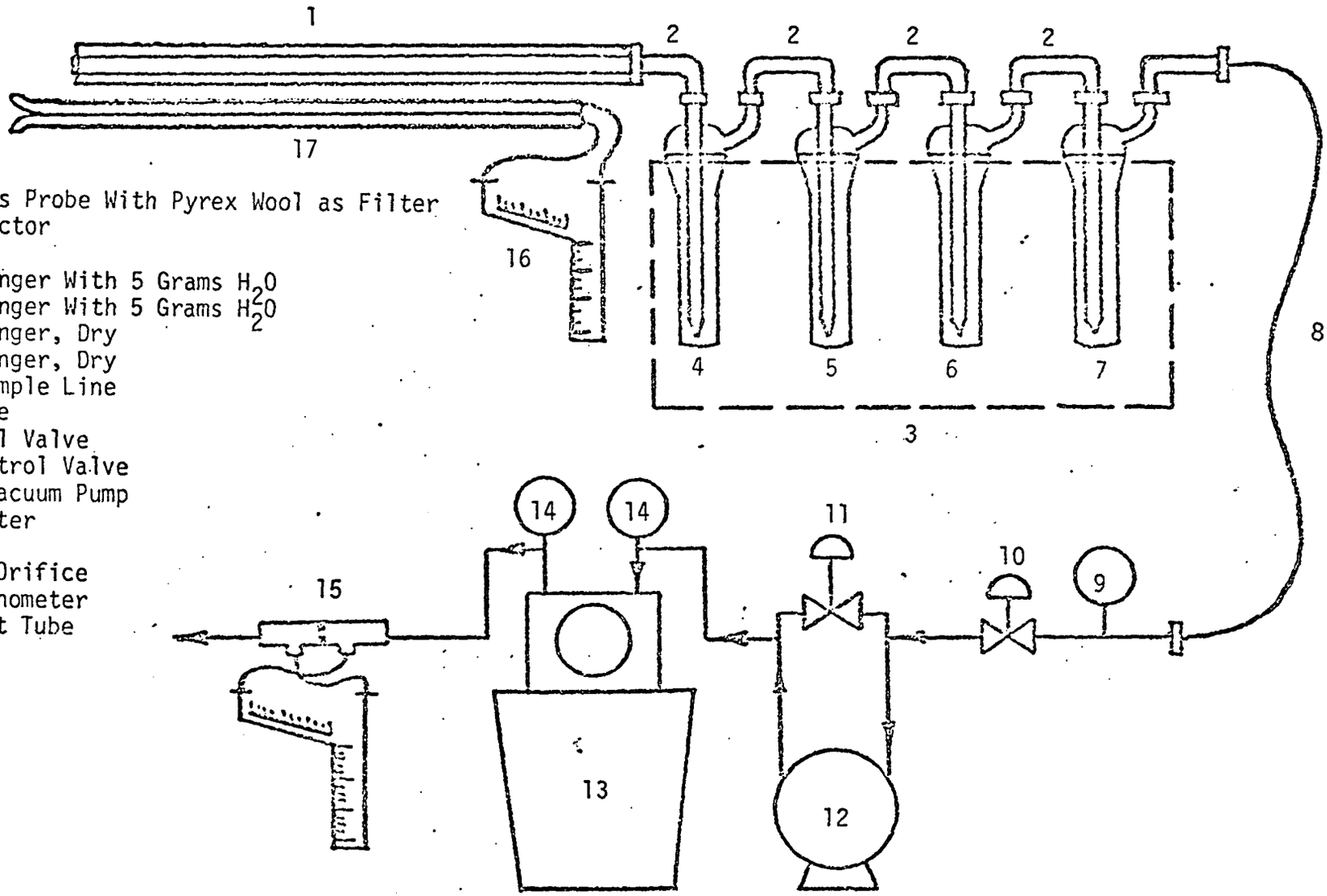


Figure 6

MOISTURE SAMPLING TRAIN

1. Stainless Steel Nozzle
2. Heated Glass Probe
3. Glass Connector
4. Ice Bath
5. Impinger with 100 ml H_2O (Modified Tip)
6. Impinger with 100 ml H_2O (Standard Tip)
7. Impinger, Dry (Modified Tip)
8. Impinger with 180 grams Silica Gel (Modified Tip)
9. Filter Holder with No. 1 Whatman Filter
10. Thermometer
11. Flexible Sample Line
12. Vacuum Gauge
13. Main Control Valve
14. By-Pass Control Valve
15. Air Tight Vacuum Pump
16. Dry Test Meter
17. Calibrated Orifice
18. Inclined Manometer
19. S-Type Pitot Tube

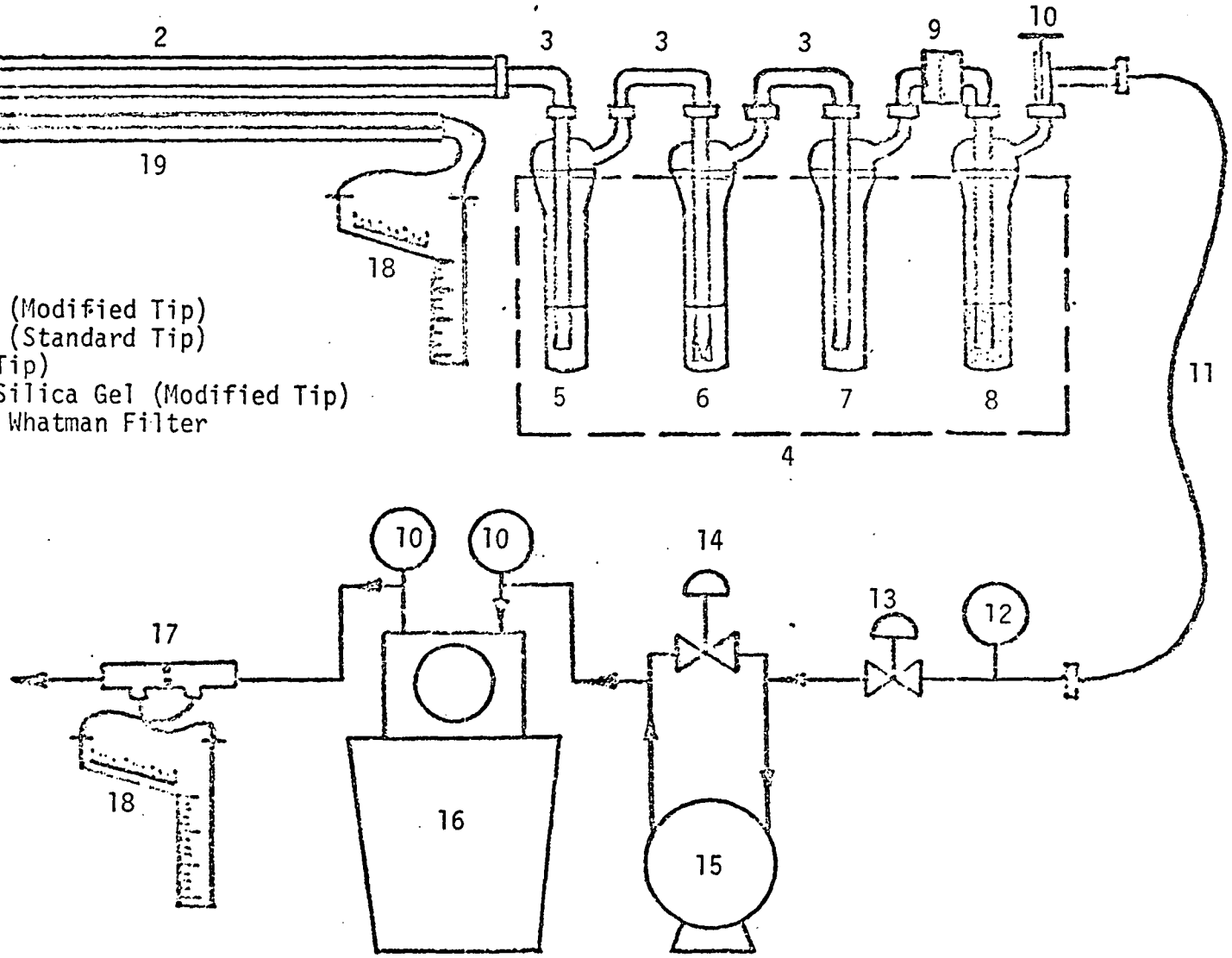


Figure 7

FLUORIDE SAMPLING TRAIN

APPENDIX

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, $\left[\overline{PB} + \frac{\overline{\Delta H}}{13.6}\right]$ 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, %
$\sqrt{\text{HxTS}}$ - 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

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

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

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

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

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

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

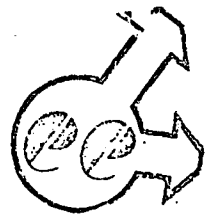
Grains/CF, Stack Cond. = (17.71) x (PS) x (FDA) x (Grains/SCF) ÷ (TS)

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)

FLUORIDE EMISSIONS



SOURCE TEST DATA

TEST NO. _____ NO. OF RUNS 3

NAME OF FIRM W. R. Grace

LOCATION OF PLANT Bartow, Florida

TYPE OF PLANT Wet Phosphoric Acid

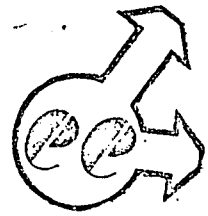
CONTROL EQUIPMENT _____

SAMPLING POINT LOCATION Station "C" -- Inlet to the Scrubber

POLLUTANTS SAMPLED Fluoride

	1	2	3
1) RUN NO.	1	2	3
2) DATE	1/4/72	1/5/72	1/5/72
3) TIME BEGAN	14:18	9:00	12:00
4) TIME END	15:58	11:^5	14:05
5) BAROMETRIC PRESSURE, "Hg ABSOLUTE	29.92	29.92	29.92
6) METER ORIFICE PRESSURE DROP, "H_2O	2.02	2.29	1.63
7) VOL DRY GAS @ METER CONDITIONS, ft^3	81.085	97.605	86.296
8) AVERAGE GAS METER TEMPERATURE, °F	87	75.5	86.9
9) VOL DRY GAS @ S.T.P.*, ft^3	78.951	97.143	83.965
10) TOTAL H_2O COLLECTED, ml	193.5	803.8	661.9
11) VOL WATER VAPOR COLL. @ S.T.P., ft^3	9.17	38.1	31.37
12) STACK GAS MOISTURE, %VOLUME	10.4	28.2	27.2
13) ASSUMED STACK GAS MOISTURE, %VOLUME	1850	1050	3000
14) % CO_2			
15) % O_2			
16) % CO			
17) % N_2			
18) % EXCESS AIR	0	0	0
19) MOLECULAR WT. OF STACK GAS, DRY	28.99	28.99	28.99
20) MOLECULAR WT. OF STACK GAS, STACK COND.	27.85	25.89	26
21) STACK GAS SP. GRAVITY, REF. TO AIR	0.96	0.89	0.9
22) AVG $\sqrt{\text{VEL. HEAD OF STACK GAS}}$, "$H_2O$	0.873	0.88	0.897
23) AVERAGE STACK GAS TEMPERATURE, °F	150.4	151.8	154
24) PITOT CORRECTION FACTOR	0.83	0.83	0.83
25) STACK PRESSURE, "Hg ABSOLUTE	29.9	29.9	29.9
26) STACK GAS VEL @ STACK COND., fpm	3178.9	3328	3392.1
27) STACK AREA, ft^2	2.35	2.35	2.35
28) STACK GAS FLOW RATE @ S.T.P., scfm	5813	4867	5010
29) NET TIME OF TEST, min.	100	120	120
30) SAMPLING NOZZLE DIAMETER, in.	0.25	0.25	0.25
31) PERCENT ISOKINETIC	93.7	114.7	96.3
32) FLUORIDE - WATER SOLUBLE, MG	106.95	837	603
33) FLUORIDE - TOTAL, MG	106.95	837	737
34) FLUORIDE - WATER SOLUBLE, GR/SCF	0.0203	0.1321	0.1076
35) FLUORIDE - TOTAL, GR/SCF	0.0203	0.1321	0.1315
36) FLOURIDE - WATER SOL. GR/CF STK COND.	0.0158	0.082	0.0675
37) FLOURIDE - TOTAL GR/CF STK COND.	0.0158	0.082	0.0825
38) FLUORIDE - WATER SOLUBLE, LB/HOUR	1.0119	5.5083	4.6203
39) FLUORIDE - TOTAL, LB/HOUR	1.0119	5.5083	5.647
41) FLOURIDE - WATER SOL. LB/TON P2O5 FED.	0.0335	0.1824	0.153
42) FLUORIDE - TOTAL, LBS/TON P2O5 FED.	0.0335	0.1824	0.187

*DRY 70 °F 29.92 in Hg



SOURCE TEST DATA

TEST NO. _____ NO. OF RUNS 3

NAME OF FIRM W. R. Grace

LOCATION OF PLANT Bartow, Florida

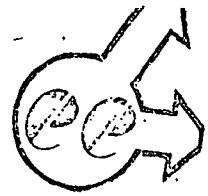
TYPE OF PLANT Wet Phosphoric Acid

CONTROL EQUIPMENT _____

SAMPLING POINT LOCATION Station "D" -- Inlet to the Scrubber

POLLUTANTS SAMPLED Fluoride

	1	2	3
1) RUN NO.	1	2	3
2) DATE	1/4/72	1/5/72	1/5/72
3) TIME BEGAN	14:20	9:05	12:00
4) TIME END	16:20	11:05	14:00
5) BAROMETRIC PRESSURE, "Hg ABSOLUTE	30	30	30
6) METER ORIFICE PRESSURE DROP, "H_2O	1.38	1.33	1.38
7) VOL DRY GAS @ METER CONDITIONS, ft^3	79.552	78.054	80.261
8) AVERAGE GAS METER TEMPERATURE, °F	87.7	76.1	86.3
9) VOL DRY GAS @ S.T.P.*, ft^3	77.449	77.631	78.343
10) TOTAL H_2O COLLECTED, ml	116.3	109	121.9
11) VOL WATER VAPOR COLL. @ S.T.P., ft^3	5.51	5.17	5.78
12) STACK GAS MOISTURE, %VOLUME	6.6	6.2	6.9
13) ASSUMED STACK GAS MOISTURE, %VOLUME	9	670	670
14) % CO_2	0.2	0.2	0.4
15) % O_2	20.5	20.5	20.4
16) % CO	0	0	0
17) % N_2	79.3	79.3	79.2
18) % EXCESS AIR	3452	3452	3058
19) MOLECULAR WT. OF STACK GAS, DRY	28.85	28.85	28.88
20) MOLECULAR WT. OF STACK GAS, STACK COND.	28.13	28.17	28.13
21) STACK GAS SP. GRAVITY, REF. TO AIR	0.97	0.97	0.97
22) AVG $\sqrt{\text{VEL. HEAD OF STACK GAS}}$, "$H_2O$	0.665	0.637	0.652
23) AVERAGE STACK GAS TEMPERATURE, °F	103.1	103.4	105
24) PITOT CORRECTION FACTOR	0.83	0.83	0.83
25) STACK PRESSURE, "Hg ABSOLUTE	29.68	29.68	29.68
26) STACK GAS VEL @ STACK COND., fpm	2322.9	2223.7	2280.5
27) STACK AREA, ft^2	5.73	5.73	5.73
28) STACK GAS FLOW RATE @ S.T.P., scfm	11686	11231	11407
29) NET TIME OF TEST, min.	120	120	120
30) SAMPLING NOZZLE DIAMETER, in.	0.25	0.25	0.25
31) PERCENT ISOKINETIC	93.5	97.5	96.9
32) FLUORIDE - WATER SOLUBLE, MG	47.7	39.165	38.1
33) FLUORIDE - TOTAL, MG	47.7	39.755	38.1
34) FLUORIDE - WATER SOLUBLE, GR/SCF	0.0092	0.0077	0.0073
35) FLUORIDE - TOTAL, GR/SCF	0.0092	0.0078	0.0073
36) FLOURIDE - WATER SOL. GR/CF STK COND.	0.008	0.0068	0.0063
37) FLOURIDE - TOTAL GR/CF STK COND.	0.008	0.0069	0.0063
38) FLUORIDE - WATER SOLUBLE, LB/HOUR	0.9248	0.7437	0.7146
39) FLUORIDE - TOTAL, LB/HOUR	0.9248	0.7549	0.7146
41) FLOURIDE - WATER SOL. LB/TON P2O5 FED.	0.0306	0.0246	0.0237
42) FLUORIDE - TOTAL, LBS/TON P2O5 FED.	0.0306	0.025	0.0237



SOURCE TEST DATA

TEST NO. _____ NO. OF RUNS 3

NAME OF FIRM W. R. Grace

LOCATION OF PLANT Bartow, Florida

TYPE OF PLANT Wet Phosphoric Acid

CONTROL EQUIPMENT Fume Scrubber

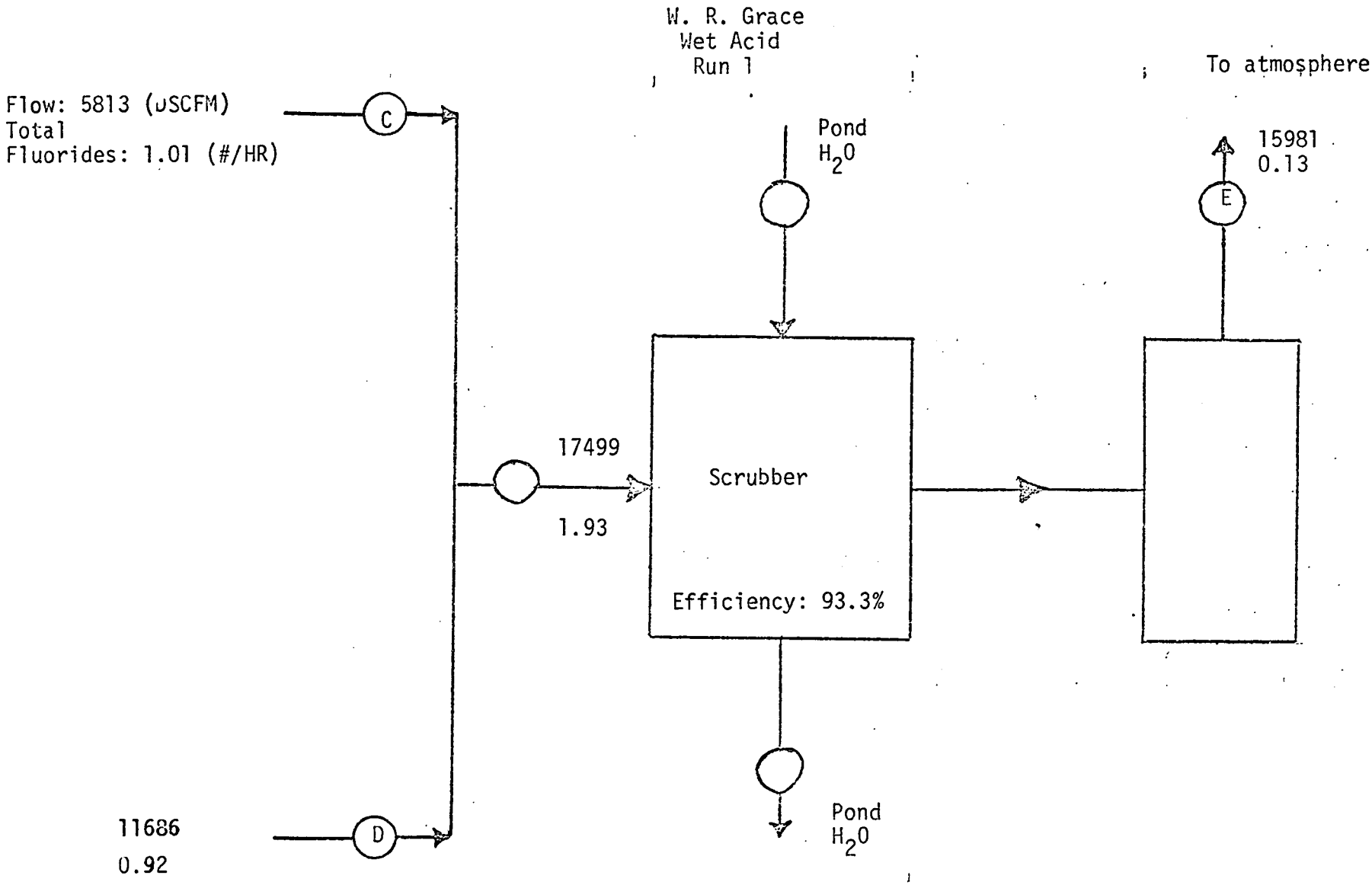
SAMPLING POINT LOCATION Station "E" -- Outlet from the Scrubber

POLLUTANTS SAMPLED Fluoride

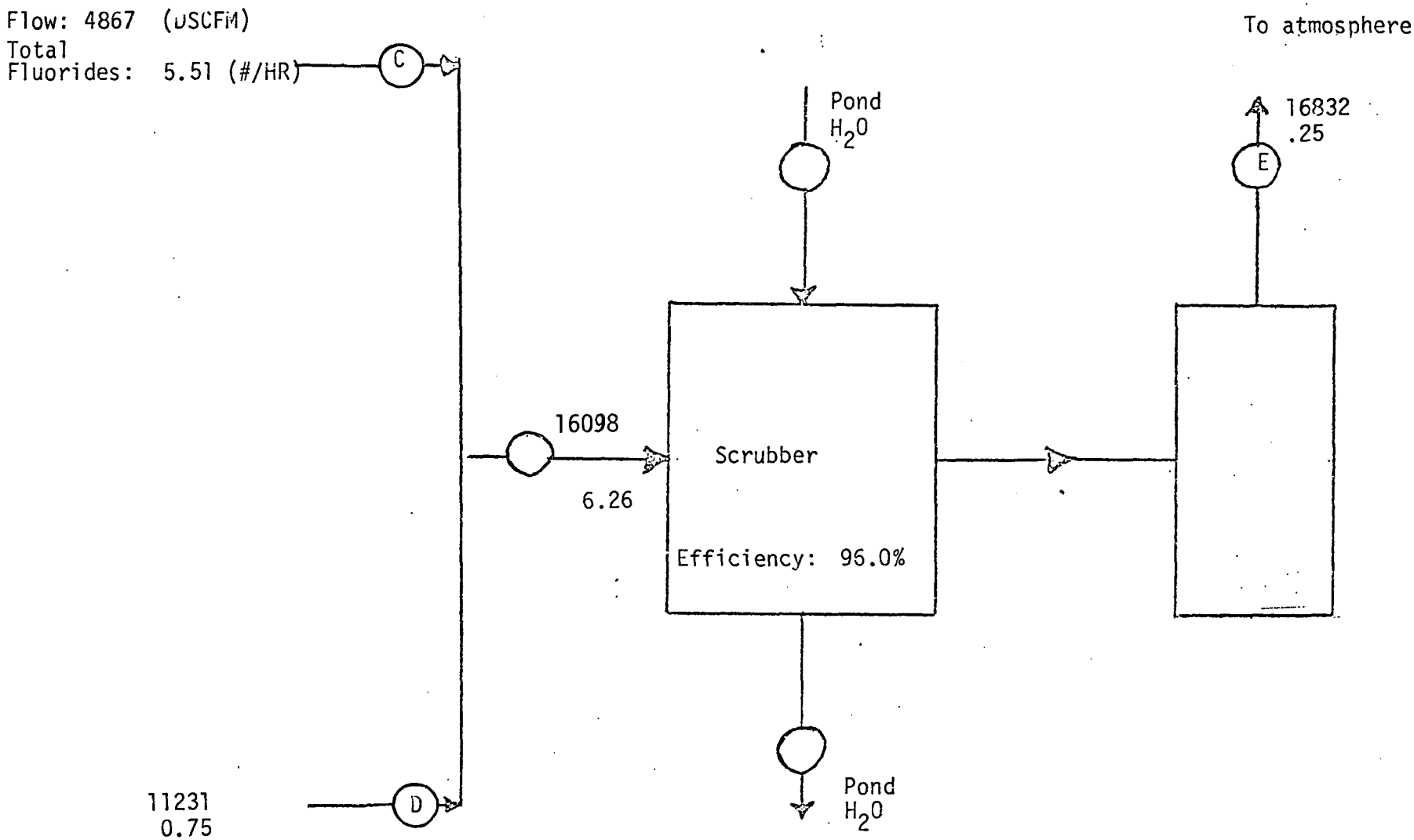
	1	2	3
1) RUN NO.	1	2	3
2) DATE	1/4/72	1/5/72	1/5/72
3) TIME BEGAN	14:15	9:00	12:00
4) TIME END	16:15	11:00	14:00
5) BAROMETRIC PRESSURE, "Hg ABSOLUTE	30	30	30
6) METER ORIFICE PRESSURE DROP, "H_2O	0.58	0.76	0.76
7) VOL DRY GAS @ METER CONDITIONS, ft^3	56.293	63.593	64.278
8) AVERAGE GAS METER TEMPERATURE, °F	84.1	73.2	80.8
9) VOL DRY GAS @ S.T.P.*, ft^3	55.057	63.498	63.282
10) TOTAL H_2O COLLECTED, ml	60.1	74.2	73.8
11) VOL WATER VAPOR COLL. @ S.T.P., ft^3	2.85	3.52	3.5
12) STACK GAS MOISTURE, %VOLUME	4.9	5.2	5.2
13) ASSUMED STACK GAS MOISTURE, %VOLUME	1200	500	50
14) % CO_2			
15) % O_2			
16) % CO			
17) % N_2			
18) % EXCESS AIR	0	0	0
19) MOLECULAR WT. OF STACK GAS, DRY	28.99	28.99	28.99
20) MOLECULAR WT. OF STACK GAS, STACK COND.	28.45	28.41	28.41
21) STACK GAS SP. GRAVITY, REF. TO AIR	0.98	0.98	0.98
22) AVG $\sqrt{\text{VEL. HEAD OF STACK GAS}}$, "$H_2O$	0.425	0.45	0.45
23) AVERAGE STACK GAS TEMPERATURE, °F	93.8	94.2	96.4
24) PITOT CORRECTION FACTOR	0.83	0.83	0.83
25) STACK PRESSURE, "Hg ABSOLUTE	30	30	30
26) STACK GAS VEL @ STACK COND., fpm	1457.8	1541.7	1547.2
27) STACK AREA, ft^2	12.05	12.05	12.05
28) STACK GAS FLOW RATE @ S.T.P., scfm	15981	16832	16825
29) NET TIME OF TEST, min.	120	120	120
30) SAMPLING NOZZLE DIAMETER, in.	0.25	0.25	0.25
31) PERCENT ISOKINETIC	101.2	110.8	110.5
32) FLUORIDE - WATER SOLUBLE, MG	3.6	7.2	3.535
33) FLUORIDE - TOTAL, MG	3.6	7.2	3.609
34) FLUORIDE - WATER SOLUBLE, GR/SCF	0.001	0.0017	0.0008
35) FLUORIDE - TOTAL, GR/SCF	0.001	0.0017	0.0009
36) FLOURIDE - WATER SOL. GR/CF STK COND.	0.0009	0.0016	0.0008
37) FLUORIDE - TOTAL GR/CF STK COND.	0.0009	0.0016	0.0008
38) FLUORIDE - WATER SOLUBLE, LB/HOUR	0.1349	0.2515	0.1221
39) FLUORIDE - TOTAL, LB/HOUR	0.1349	0.2515	0.1247
41) FLOURIDE - WATER SOL. LB/TON P2O5 FED.	0.0045	0.0083	0.004
42) FLUORIDE - TOTAL, LBS/TON P2O5 FED.	0.0045	0.0083	0.0041

*DRY, 70 °F, 29.92 in. Hg

SCRUBBER EFFICIENCY

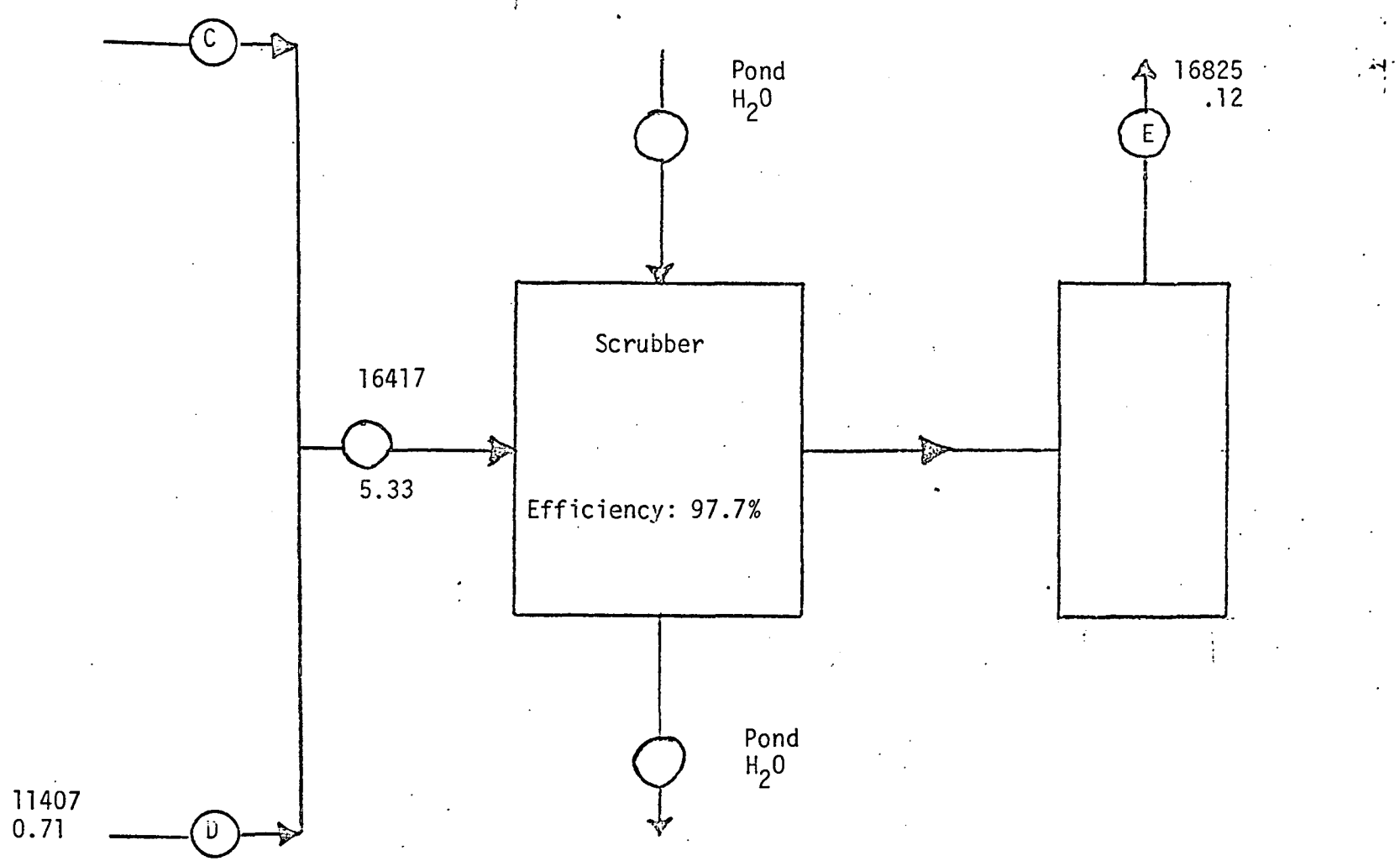

W. R. Grace
Wet Acid
Run 1
Flow: 5813 (uSCFM)
Total
Fluorides: 1.01 (#/HR)
C
Pond
H_2O
To atmosphere
15981
0.13
E
17499
1.93
Scrubber
Efficiency: 93.3%
11686
0.92
D
Pond
H_2O

W. R. Grace
Wet Acid
Run 2



W. R. Grace
Wet Acid
Run 3

Flow: 5010 (DSCFM)
Total 4.62 (#/HR)
Fluorides:



To atmosphere

APPENDIX B

Field Data

PRELIMINARY MOISTURE DETERMINATION

PRELIMINARY CHECK FOR STACK GAS

MOISTURE CONTENT AND SPECIFIC GRAVITY

Plant W. R. GRACE Stack Wet Phos. Acid - Station "C"

Date 1-4-72 Sample Time 1000 Barometric Pressure 30 "Hg

Moisture Content -- Method 1

Final Dry Test Meter Reading	213.725	Ft^3
Initial Dry Test Meter Reading	212.725	Ft^3
Dry Test Meter Volume Sampled	1.000	Ft^3
Average Meter Temperature	71	°F
Average Meter Vacuum	—	"Hg
Average Meter Orifice Δ H	0.05	"H_2O
Sampling Rate	2.8	LPM
Barometric Pressure @ Meter Orifice	30	"Hg
Dry Gas Volume Sampled @ 70 °F, 29.92 "Hg	1.00	Ft^3
Condensate Volume	5	ml
Water Vapor Volume @ 70 °F, 29,92 "Hg	0.24	Ft^3

Moisture Fraction, H_2O 0.19 Fraction Of Dry Air, FDA 0.81

Moisture Content -- Method 2

Dry Bulb Temp. 153 °F Wet Bulb Temp. 149 °F Dew Point Temp. 128 °F

Vapor Pressure Of H_2O @ DP 2.18 "Hg Stack Pressure 29.9 "Hg

Moisture Fraction, H_2O 0.24 Fraction Of Dry Air, FDA 0.76

Specific Gravity

Dry Molecular Weight, $M_d = [0.44(\%CO_2)] + [0.32(\%O_2)] + [0.28(\%N_2 + CO)] =$ _____

Molecular Weight @ Stack Conditions, $M_s = [(M_d)x(FDA)] + [(18)x(H_2O)] =$ _____

Specific Gravity (Referred to air), $G_s = (M_s) \div (28.99) =$ _____

PRELIMINARY CHECK FOR STACK GAS

MOISTURE CONTENT AND SPECIFIC GRAVITY

Plant W. R. GRACE Stack Wet Phos. Acid - Station "D"

Date 1-4-72 Sample Time 1000-1015 Barometric Pressure 30 "Hg

Moisture Content -- Method 1

Final Dry Test Meter Reading	908.700	Ft^3
Initial Dry Test Meter Reading	906.700	Ft^3
Dry Test Meter Volume Sampled	2.000	Ft^3
Average Meter Temperature	71	°F
Average Meter Vacuum	—	"Hg
Average Meter Orifice Δ H	0.05	"H_2O
Sampling Rate	3.8	LPM
Barometric Pressure @ Meter Orifice	30	"Hg
Dry Gas Volume Sampled @ 70 °F, 29.92 "Hg	2.00	Ft^3
Condensate Volume	9	ml
Water Vapor Volume @ 70 °F, 29,92 "Hg	0.43	Ft^3

Moisture Fraction, H_2O 0.18 Fraction Of Dry Air, FDA 0.82

Moisture Content -- Method 2

Dry Bulb Temp. 101 °F Wet Bulb Temp. 91 °F Dew Point Temp. 88 °F

Vapor Pressure Of H_2O @ DP 1.3 "Hg Stack Pressure 29.68 "Hg

Moisture Fraction, H_2O 0.04 Fraction Of Dry Air, FDA 0.96

Specific Gravity

Dry Molecular Weight, $M_d = [0.44(\%CO_2)] + [0.32(\%O_2)] + [0.28(\%N_2 + CO)] =$ _____

Molecular Weight @ Stack Conditions, $M_s = [(M_d)x(FDA)] + [(18)x(H_2O)] =$ _____

Specific Gravity (Referred to air), $G_s = (M_s) \div (28.99) =$ _____

PRELIMINARY CHECK FOR STACK GAS

MOISTURE CONTENT AND SPECIFIC GRAVITY

Plant W. R. Grace Stack Wet Phos. Acid - Station "E"

Date 1-4-72 Sample Time 1000 Barometric Pressure 30 "Hg

Moisture Content -- Method 1

Final Dry Test Meter Reading	702.589	Ft^3
Initial Dry Test Meter Reading	700.607	Ft^3
Dry Test Meter Volume Sampled	1.972	Ft^3
Average Meter Temperature	74	°F
Average Meter Vacuum	—	"Hg
Average Meter Orifice Δ H	0.06	"H_2O
Sampling Rate	2.8	LPM
Barometric Pressure @ Meter Orifice	30	"Hg
Dry Gas Volume Sampled @ 70 °F, 29.92 "Hg	1.96	Ft^3
Condensate Volume	16	ml
Water Vapor Volume @ 70 °F, 29,92 "Hg	0.76	Ft^3

Moisture Fraction, H_2O 0.28 Fraction Of Dry Air, FDA 0.72

Moisture Content -- Method 2

Dry Bulb Temp. 100 °F Wet Bulb Temp. 95 °F Dew Point Temp. 95 °F

Vapor Pressure Of H_2O @ DP 1.66 "Hg Stack Pressure 30 "Hg

Moisture Fraction, H_2O 0.06 Fraction Of Dry Air, FDA 0.94

Specific Gravity

Dry Molecular Weight, $M_d = [0.44(\%CO_2)] + [0.32(\%O_2)] + [0.28(\%N_2 + CO)] =$ _____

Molecular Weight @ Stack Conditions, $M_s = [(M_d)x(FDA)] + [(18)x(H_2O)] =$ _____

Specific Gravity (Referred to air), $G_s = (M_s) \div (28.99) =$ _____

FLUORIDE EMISSIONS

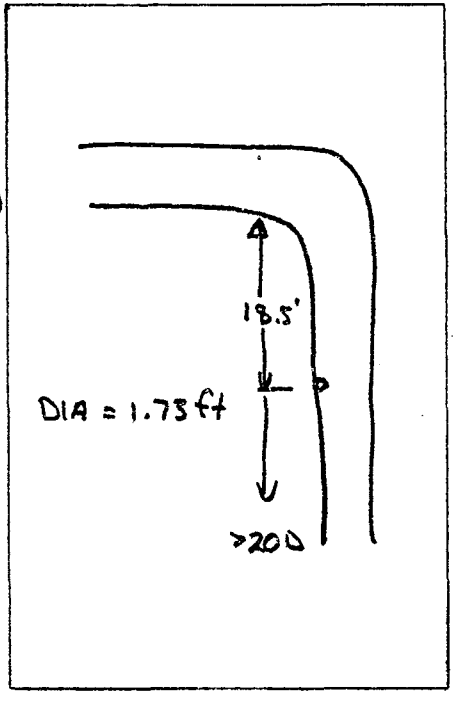
19
200
184

ENVIRONMENTAL ENGINEERING, INC.
Gainesville, Florida

Wt. of Silica Gel Start 240.5 gm

SOURCE SAMPLING FIELD DATA SHEET

Plant W.R. GRACE
Sampling Location Wet Phos. Acid, Stat "C"
Date Tues Jan 4 Run No. 1
Time Start 14:18 Time End 15:58
Sampling Time/Point (100 min) (5 @ 20 min)
DB 153 °F, WB 149 °F, VP @ DP 7.18 "Hg
Moisture 18.5 %, FDA .815, Gas Density Factor
Barometric Press 29.92 "Hg, Stack Press 29.90 "Hg
Weather Over Cast
Temp. 80 °F, W/D , W/S
Sample Box No. 4 Meter Box No. 4
Meter ΔH@ 1.62 Pitot Corr. Factor .83
Nozzle Dia. .25 in., Probe Length 4 ft
Probe Heater Setting 35 %
Stack Dimensions: Inside Diameter 20.76 in
Inside Area 2.35 ft2
Height ft



Mat'l Processing Rate
Final Gas Meter Reading 295.085 ft3
Initial Gas Meter Reading 214.000 ft3
Total Condensate in Impingers 81.5 ml
Moisture in Silica Gel 9.5 gm
Silica Gel Container No. 305 Filter No. 72037
Orsat: CO_2
O_2
CO
N_2
Excess Air
Test Conducted by: A.L. Wilson
BILL DEMERY

Remarks: 1st POINT IS TOO CLOSE TO STACK WALL.

Port and Traverse Point No.	Distance from End of Port (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 Test (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual		In	Out			
1	.08	TOO	CLOSE TO	WALL								
1		1418	214.0									
2	.25	1423	217.4	0.4	1.05	1.05	152	82	87	—	83	13
2	.25	1428	220.5	0.39	1.0	1.0	152	87	87	—	83	11
2	.25	1433	223.6	0.4	1.05	1.05	152	87	87	—	83	11

Port and Traverse Point No.	Distance from End of Port (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
					Calc.	Actual		In	Out			
2		1438	226.6	0.4	1.05	1.05	151.0	87	87	—	88	11
3	.51	1443	230.5	0.7	1.8	1.8	150.0	86	87	—	88	9
3	.51	1448	234.3	0.7	1.8	1.8	150.0	86	87	—	83	2
3	.51	1453	238.3	0.7	1.8	1.8	150.0	87	87	—	83	2
3	.51	1458	242.2	0.7	1.8	1.8	150.0	87	87	—	83	3
4	1.22	1503	246.5	0.85	2.2	2.2	150.6	87	87	—	83	3
4	1.22	1508	251.0	0.95	2.45	2.45	150.0	88	87	—	81	3
4	1.22	1513	255.3	0.90	2.4	2.4	150.0	88	87	—	80	3
4	1.22	1518	259.7	0.90	2.4	2.4	150.0	88	87	—	80	3
5	1.48	1523	264.2	.95	2.45	2.45	150.0	88	87	—	80	3
5	1.48	1528	267.3	.95	2.45	2.45	150.0	87	87	—	81	3
5	1.48	1533	273.0	.95	2.45	2.45	150.0	87	87	—	81	3
5	1.48	1538	277.5	.95	2.45	2.45	150.0	87	87	—	81	3
6	1.65	1543	281.8	0.95	2.45	2.45	150.0	88	87	—	81	3
6	1.65	1548	286.2	0.95	2.45	2.45	150.0	88	87	—	81	3
6	1.65	1553	290.6	0.95	2.45	2.45	150.0	88	87	—	81	3
6	1.65	1558	295.085	0.95	2.45	2.45	150.0	88	87	—	81	3

ENVIRONMENTAL ENGINEERING, INC. Initial wt of 1235.0 g
Gainesville, Florida
18.8

SOURCE SAMPLING FIELD DATA SHEET

Plant W.R. GRACE
Sampling Location Wet Phos Acid, Stat "C"
Date ~~July~~ Jun 3 (Wed) Run No. 2
Time Start 9:00 Time End 11:05
Sampling Time/Point 120 min (6 at 20 min)
DB ____ °F, WB ____ °F, VF @ DP ____ "Hg
Moisture 0.5 %, FDA ____, Gas Density Factor ____
Barometric Press ____ "Hg, Stack Press ____ "Hg
Weather Over Cast
Temp. ____ °F, W/D ____, W/S ____
Sample Box No. ____ Meter Box No. 4
Meter ΔH@ 1.62 Pitot Corr. Factor .83
Nozzle Dia. .25 in., Probe Length 4 ft
Probe Heater Setting 35%
Stack Dimensions: Inside Diameter 20.76 in
Inside Area 2.35 ft²
Height ____ ft

4 × 785 = 3140
.05 × 785 = 39.25
49 / 785 = .3

Mat'l Processing Rate ____
Final Gas Meter Reading 392.605 ft³
Initial Gas Meter Reading 295.600 ft³
985 - 200 = 785
Total Condensate in Impingers 785 ml
Moisture in Silica Gel 18.8 gm
Silica Gel Container No. 305 Filter No. 72041
Orsat: CO_2 ____
O_2 ____
CO ____
N_2 ____
Excess Air ____
Test Conducted by: A. L. WILSON
W. P. DEMERY

Remarks: [illegible]
Points No. 1 & 6 were ≈ .085 ft from wall.

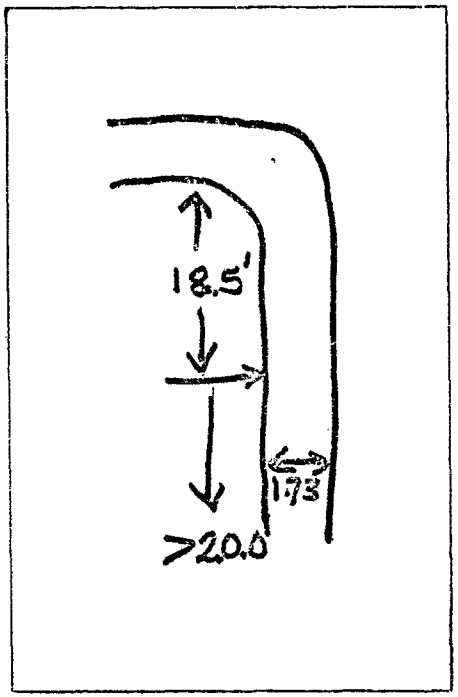
Port and Traverse Point No.	Distance from End of Port ~~(in.)~~ (Ft.)	Clock Time	Gas Meter Reading (ft^3)	Stack Velocity Head ("H_2O)	Meter Orifice Press. Diff. ("H_2O)		Stack Gas Temp. (°F)	Gas Sample Temp. @ Dry Gas Meter (°F)		Sample Box Temp. (°F)	Last Impirger Test (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual		In	Out			
		0900	295.0									
1	.08+	0905	298.4	.4	1.18	1.18	150	73	74	—	70	6
1	.08+	0910	301.5	.4	1.18	1.18	150	73	74	—	70	6
1	.08+	0915	304.9	.4	1.18	1.18	150	73	74	—	70	6
1	.08+	0920	307.3	.4	1.18	1.18	150	73	74	—	70	6
2	.25	0925	311.0	.6	1.75	1.75	150	73	74	—	70	9
2	.25	0930	314.7	.65	1.9	1.9	150	73	74	—	70	10
2	.25	0935	318.6	.65	1.9	1.9	150	73	74	—	70	10

Port and Traverse Point No.	Distance from End of Port (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
					Calc.	Actual		In	Out			
2		0940	322.3	.65	1.9	1.9	150	73	74	—	70	10
3	.51	0945	326.7	.9	2.6	2.6	150	73	74	—	70	14
3		0950	331.1	.9	2.6	2.6	150	73	74	—	70	14
3		0955	335.5	.9	2.6	2.6	150	74	74	—	72	14
3		1000	339.9	.9	2.6	2.6	150	75	74	—	74	14
4	1.22	1010	349.7	1.1	3.15	3.15	152	75	75	—	75	18
4		1015	349.4	1.1	3.15	3.15	152	75	75	—	75	18
4		1020	354.3	1.1	3.15	3.15	152	76	75	—	88	19
4		1025	358.8	.9	2.6	2.6	152	77	75	—	85	16
5	1.48	1030	363.1	.9	2.6	2.6	151	78	76	—	82	15
5		1035	367.4	.9	2.6	2.6	154	78	76	—	83	16
5		1040	371.7	.9	2.6	2.6	154	78	76	—	85	16
5		1045	375.9	.9	2.6	2.6	154	78	76	—	85	16
6	1.65	1050	379.8	.8	2.3	2.3	154	78	76	—	85	15
6		1055	384.2	.85	2.5	2.5	154	80	77	—	85	19
6		1100	388.4	.9	2.6	2.6	154	84	79	—	85	19
6		1105	392.605	.9	2.6	2.6	154	85	80	—	85	20

EN...NMENTAL ENGINEERING, INC. Final Si Gel 241.1
Gainesville, Florida

SOURCE SAMPLING FIELD DATA SHEET

Plant W.R. GRACE
Sampling Location Wet Phos. Acid, STAT "C"
Date JAN 5 Run No. 3
Time Start 1200 Time End 1405
Sampling Time/Point 120mm (6 at 20min)
DB °F, WB °F, VF @ DP "Hg
Assumed Moisture 30%, FDA , Gas Density Factor
Barometric Press 29.91 "Hg, Stack Press 29.90 "Hg
Weather Over Cast
Temp. 85 °F, W/D , W/S
Sample Box No. Meter Box No. 4
Meter ΔH@ 1.62 Pitot Corr. Factor .83
Nozzle Dia. .25 in., Probe Length 4 ft
Probe Heater Setting 35%
Stack Dimensions: Inside Diameter 20.76 in
Inside Area 2.35 ft²
Height ft



Mat'l Processing Rate
Final Gas Meter Reading 478.901 ft³
Initial Gas Meter Reading 392.605 ft³
843 - 200 = 643
Total Condensate in Impingers 643 ml
Moisture in Silica Gel 18.9 gm
Silica Gel Container No 3/0 Filter No. 72044
Orsat: CO_2
O_2
CO
N_2
Excess Air
Test Conducted by: A.L. WILSON
W.P. EMERY

Remarks: Orsat CO_2 reading 7.21%

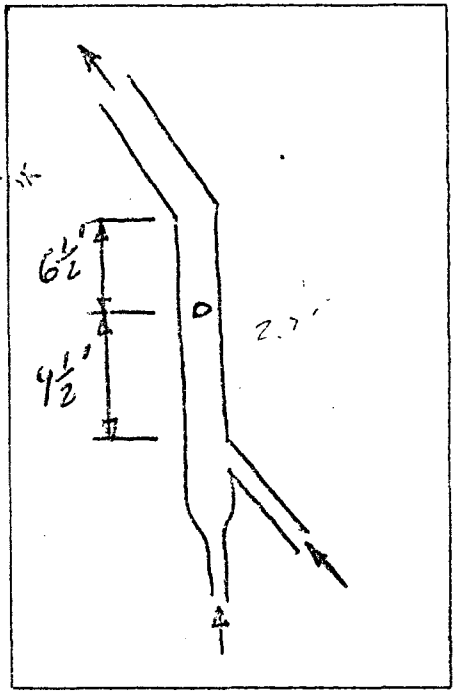
Port and Traverse Point No.	Distance from End of Port (in.)	Clock Time	Gas Meter Reading (ft^3)	Stack Velocity Head ("H_2O)	Meter Orifice Press. Diff. ("H_2O)		Stack Gas Temp. (°F)	Gas Sample Temp. @ Dry Gas Meter (°F)		Sample Box Temp. (°F)	Last Impinger Test (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual		In	Out			
		1200	392.605									
1	.08+	1205	394.9	.3	.58	.58	154	85	85	—	90	4
1	.08+	1210	397.4	.3	.58	.58	154	82	85	—	90	4
1	.08+	1215	399.9	.4	.79	.79	154	81	86	—	95	5
1	.08+	1220	402.3	.3	.58	.58	154	81	84	—	95	4
2	.25	1225	405.0	.45	.89	.89	154	83	84	—	90	
2	.25	1230	407.9	.45	.89	.89	154	84	84	—	90	
2	.25	1235	411.0	.45	.89	.89	154	85	85	—	90	

Port and Traverse Point No.	Distance from End of Port (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
					Calc.	Actual		In	Out			
2	.25	1240	414.4	1.2	2.3	2.3	154	86	86		90	14
3	.51	1245	419.5	2.0	3.9	3.1	154	87	87		90	20
3		1250	424.2	1.3	2.5	2.5	154	88	86		90	10
3		1255	428.3	1.1	2.1	2.1	154	88	87		90	8
3		1300	432.3	.97	1.85	1.85	154	88	87		90	7
4	1.22	1310	436.1	.83	1.65	1.65	154	89	87		90	6
4		1315	439.5	.70	1.35	1.35	154	87	87		87	6
4		1320	442.9	.70	1.35	1.35	154	86	87		87	6
4		1325	446.3	.70	1.35	1.35	154	86	87		90	6
5	1.48	1330	451.0	1.2	2.3	2.3	154	86	87		95	10
5		1335	456.0	1.7	3.3	3.3	154	90	87		95	17
5		1340	460.3	1.2	2.3	2.3	154	93	89		92	11
5		1345	464.2	.92	1.8	1.8	154	90	90		90	9
6	1.65	1350	468.1	.92	1.8	1.8	154	90	90		93	9
6		1355	471.8	.87	1.7	1.7	154	90	90		93	9
6		1400	475.4	.87	1.7	1.7	154	90	90		95	9
6		1405	478.9	.75	1.45	1.45	154	90	90		95	8

ENVIRONMENTAL ENGINEERING, INC.
Gainesville, Florida

SOURCE SAMPLING FIELD DATA SHEET

Plant W.R. GRACE
Sampling Location STATION "D"
Date 1/4/72 Run No. 1
Time Start 14:20 Time End 16:20
Sampling Time/Point 6 min @ 20 pts = 120 min
DB ____ °F, WB ____ °F, VF @ DP ____ "Hg
Moisture 0.09 %, FDA 0.91, Gas Density Factor ____
Barometric Press 30 "Hg, Stack Press 2268 "Hg
Weather CLEAR
Temp. 25 °F, W/D ____, W/S ____
Sample Box No. ____ Meter Box No. 4
Meter ΔH@ 1.74 Pitot Corr. Factor 0.83
Nozzle Dia. .025 in., Probe Length 6 ft
Probe Heater Setting ____
Stack Dimensions: Inside Diameter 32.4 in
Inside Area 5.72 ft2
Height Horizontal ft



Mat'l Processing Rate ____
Final Gas Meter Reading 988.257 ft3
Initial Gas Meter Reading 902.705 ft3
Total Condensate in Impingers 101 ml
Moisture in Silica Gel 249.0 - 233.7 = 15.3 gm
Silica Gel Container No. 306 Filter No. 72-038
Orsat: CO_2 0.2
O_2 20.5
CO 0
N_2 79.3
Excess Air ____
Test Conducted by: J. Dollar
A. Arroyo
R. Wickers

Remarks: Optimum sampling pts. = 45
Only one port available, thus 22 pts.

Port and Traverse Point No.	Distance from End of Port (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 Test (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual		In	Out			
										Stop Watch		
1	23/64	14:20	908.705	—	—	—	—	—	—	0	—	—
2	1 1/8	14:26	913.100	.58	1.75	1.75	104	84	84	6	75	.5
3	1 15/16	14:32	916.050	.25	.73	.73	104	85	85	1	80	1.8
4	2 13/16	14:38	918.82	.23	.65	.65	103	86	85	3	83	1.8
5	3 3/4	14:44	921.65	.22	.63	.63	103	87	85	9	86	1.8
6	4 23/32	14:50	924.57	.25	.73	.73	103	8	1	15	83	2.1

Port and Traverse Point No.	Distance from End of Port (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) STOP WATCH	Last Impinger Temp. (°F)	Vacuum on Sample Train
					Calc.	Actual		In	Out			
7		0935	1008.60	0.29	0.91	0.91	102	75	75	15	72	3
8		0941	1012.0	0.29	0.91	0.91	102	75	75	6	72	3.1
9		0947	1015.32	0.28	0.86	0.86	102	75	75	12	72	3.0
10		0953	1018.52	0.27	0.83	0.83	102	75	75	3	72	3.0
11		0959	1022.0	0.27	0.83	0.83	102	75	75	9	72	3.2
12		1005	1026.1	.48	1.60	1.60	102	76	75	15	72	6.8
13		1011	1030.52	.52	1.7	1.7	105	76	75	6	72	7.1
14		1017	1034.9	0.54	1.75	1.75	105	76	75	12	77	7.5
15		1023	1039.4	0.53	1.72	1.72	105	77	76	3	79	7.8
16		1029	1043.9	0.54	1.75	1.75	105	78	76	9	80	8.0
17		1035	1048.4	0.55	1.78	1.78	105	78	77	15	80	8.2
18		1041	1052.9	0.56	1.80	1.80	105	78	79	6	81	8.3
19		1047	1057.6	0.55	1.78	1.78	105	79	77	12	81	8.3
20		1053	1062.0	0.54	1.75	1.75	105	80	78	3	81	8.3
21		1059	1066.254	0.51	1.62	1.62	105	80	78	9	81	8.2
22		1105								15		
										—		

E...RON...AL ...INEE...G, ...
Gainesville, Florida

SOURCE SAMPLING FIELD DATA SHEET

Plant W. R. GRACE
Sampling Location STATION "D"
Date 1/5/72 Run No. 2
Time Start 0905 Time End 11:05
Sampling Time/Point 6 min @ 20 pt 120 tot min
DB — °F, WB — °F, VF @ DP — "Hg
Moisture 6.7 %, FDA 93.3, Gas Density Factor .97
Barometric Press 30 "Hg, Stack Press 29.68 "Hg
Weather Overcast
Temp. 70 °F, W/D — , W/S —
Sample Box No. — Meter Box No. 1
Meter ΔH@ 1.74 Pitot Corr. Factor 0.83
Nozzle Dia 0.25 in., Probe Length 4 ft
Probe Heater Setting —
Stack Dimensions: Inside Diameter 32.4 in
Inside Area 5.72 ft²
Height ____ ft

See Run #1

Mat'l Processing Rate ____
Final Gas Meter Reading 1066.254 ft³
Initial Gas Meter Reading 988.200 ft³
Total Condensate in Impingers 96 ml (109)
Moisture in Silica Gel 250.8 - 237.8 = 13 gm
Silica Gel Container No. 304 Filter No. 72-039
Orsat: CO_2 .2
O_2 20.5
CO 0
N_2 79.3
Excess Air ____
Test Conducted by: J. Dollar
A. Arroyo
R. Wicker
Remarks: See Run #1

Port and Traverse Point No.	Distance from End of Port (in.)	Clock Time	Gas Meter Reading (ft³)	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)~~ STOP Watch	Last Impinger Test (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual		In	Out			
1			988.200									
2		0905	991.900	.29	.91	.91	102	75	75	15	75	2.9
3		0911	995.000	.34	1.05	1.05	102	75	75	6	75	3.5
4		0917	998.500	.35	1.1	1.1	102	75	75	12	75	3.0
5		0923	1001.9	.29	.91	.91	102	75	75	3	75	3.5
6		0929	1005.1	.30	0.95	0.95	102	75	75	9	75	3.0

Port and Traverse Point No.	Distance from End of Port (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) STOP WATCH	Last Impinger Temp. (°F)	Vacuum on Sample Train
					Calc.	Actual		In	Out			
7		0935	1008.60	0.29	0.91	0.91	102	75	75	15	72	3
8		0941	10012.0	0.29	0.91	0.91	102	75	75	6	72	3.1
9		0947	10 15.32	0.28	0.86	0.86	102	75	75	12	72	3.0
10		0953	1018.52	0.27	0.83	0.83	102	75	75	3	72	3.0
11		0959	1022.0	0.27	0.83	0.83	102	75	75	9	72	3.2
12		1005	1026.1	.48	1.50	1.60	102	76	75	15	72	6.8
13		1011	1030.52	.52	1.7	1.7	105	76	75	6	72	7.1
14		1017	1034.9	0.54	1.75	1.75	105	76	75	12	77	7.5
15		1023	1039.4	0.53	1.72	1.72	105	77	76	3	79	7.8
16		1029	1043.9	0.54	1.75	1.75	105	78	76	9	80	8.0
17		1035	1048.4	0.55	1.78	1.78	105	78	77	15	80	8.2
18		1041	1052.9	0.56	1.80	1.80	105	78	79	6	81	8.3
19		1047	1057.6	0.55	1.78	1.78	105	79	77	12	81	8.3
20		1053	1062.0	0.54	1.75	1.75	105	80	78	3	81	8.3
21		1059	1066.254	0.51	1.62	1.62	105	80	78	9	81	8.2
22		1105								15		
										—		

ENVIRONMENTAL ENGINEERING, INC.
Gainesville, Florida

SOURCE SAMPLING FIELD DATA SHEET

Plant W. R. GRACE
Sampling Location STATIONED "1"
Date 1/5/72 Run No. 3
Time Start 1200 Time End 1400
Sampling Time/Point 6min@20pts=120tot min
DB —°F, WB —°F, VF @ DP —"Hg
Moisture 6.7 %, FDA 93.3, Gas Density Factor
Barometric Press 30 "Hg, Stack Press 29.68 "Hg
Weather Overcast
Temp. 25 °F, W/D —, W/S —
Sample Box No. — Meter Box No. 1
Meter ΔH@ 1.74 Pitot Corr. Factor 0.83
Nozzle Dia. .25 in., Probe Length 4 ft
Probe Heater Setting
Stack Dimensions: Inside Diameter 32.4 in
Inside Area 5.72 ft2
Height ft

See Run #1

Mat'l Processing Rate
Final Gas Meter Reading 146.515 ft3
Initial Gas Meter Reading 66.254 ft3
Total Condensate in Impingers 108.5 ml
Moisture in Silica Gel 256 - 242.6 = 13.4 gm
Silica Gel Container No. 307 Filter No. 72-042
Orsat: CO_2 0.4
O_2 20.4
CO 0
N_2 79.2
Excess Air
Test Conducted by: J. Dollar
A. Arroyo
R. Wickker

Remarks: See Run #1

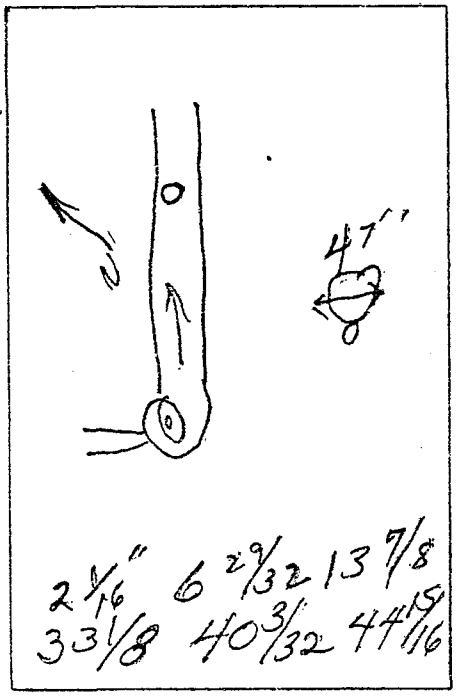
Port and Traverse Point No.	Distance from End of Port (in.)	Clock Time	Gas Meter Reading (ft^3)	Stack Velocity Head ("H_2O)	Meter Orifice Press. Diff. ("H_2O)		Stack Gas Temp. (°F)	Gas Sample Temp. @ Dry Gas Meter (°F)		Sample Box Temp. (°F)	Last Impinger Test (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual		In	Out	Stop watch		
	See Run #1											
1			66.254									
2		12.00	69.8	.35	1.12	1.12	105	82	82	1.5	87	2.5
3		12:06	74.54	.65	2.0	2.0	105	82	82	4	78	2.8
4	See Run #1	12.12	78.88	.55	1.75	1.75	105	83	82	12	78	5
5		12:18	82.05	.26	.77	.77	105	84	83	3	82	2.2
6		12.24	85.48	.30	.94	.94	105	86	84	9	81	2.8

Port and Traverse Point No.	Distance from End of Port (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) Stop Watch	Last Impinger Temp. (°F)	Vacuum on Sample Train
					Calc.	Actual		In	Out			
7		12:30	88.63	0.28	0.86	0.86	105	86	84	15	79	2.5
8		12:36	91.98	0.28	0.86	0.86	105	86	84	6	79	2.5
9		12:42	95.31	0.30	0.95	0.95	105	88	84	12	83	2.5
10		12:48	98.62	0.28	0.86	0.86	105	88	85	3	83	2.8
11		12:54	102.4	0.35	1.12	1.12	105	89	86	9	81	4
12	#	13:00	106.65	.48	1.6	1.6	105	89	86	15	76	6
13		13:06	111.2	.52	1.7	1.7	105	89	87	6	77	6.1
14		13:12	115.6	.52	1.7	1.7	105	89	87	12	78	6.3
15	Run	13:18	119.95	.54	1.75	1.75	105	89	87	3	77	7
16		13:24	124.4	.50	1.64	1.64	105	89	87	9	77	6.8
17		13:30	128.6	.52	1.7	1.7	105	89	87	15	78	6.9
18		13:36	133.8	.50	1.64	1.64	105	89	87	6	78	6.9
19		13:42	137.6	.48	1.6	1.6	105	89	87	12	79	6.9
20	Leak	13:48	142.3	.52	1.7	1.7	105	89	87	3	79	7
21		13:54	146.515	.48	1.6	1.6	105	89	87	9	80	6.8
22		12:00								15		

ENVIRONMENTAL ENGINEERING, INC.
Gainesville, Florida

SOURCE SAMPLING FIELD DATA SHEET

Plant W R. Grace (wet acid)
Sampling Location 5 Talton (E) (Outlet)
Date 1/4/72 Run No. 1
Time Start 2.15 14:15 Time End 4:15 16:15
Sampling Time/Point 20min (120 total)
DB 93 °F, WB ____ °F, VF @ DP ____ "Hg
Moisture 12 %, FDA 0.78, Gas Density Factor ____
Barometric Press 30 "Hg, Stack Press 30 "Hg
Weather clear
Temp. ____ °F, W/D ____, W/S ____
Sample Box No. ____ Meter Box No. ____
Meter ΔH@ ____ Pitot Corr. Factor .83
Nozzle Dia. .25 in., Probe Length 6 ft
Probe Heater Setting ____
Stack Dimensions: Inside Diameter 47 in
Inside Area 12.04 ft²
Height ____ ft



Mat'l Processing Rate ____
Final Gas Meter Reading 758.882 ft³
Initial Gas Meter Reading 702.589 ft³
Total Condensate in Impingers 48 ml
Moisture in Silica Gel (242.5)(254.6) = 12.1 gm
Silica Gel Container No. 303 Filter No. 72-036
Orsat: CO_2 0
O_2 20.9
CO 0
N_2 ____
Excess Air ____
Test Conducted by: C. Allen
E Johnson
Remarks: optimum # of Sampling Points is 12, only 1 port is Available So 6 pts.

Port and Traverse Point No.	Distance from End of Port (in.)	Clock Time	Gas Meter Reading (ft^3) 702.589	Stack Velocity Head ("H_2O)	Meter Orifice Press. Diff. ("H_2O) Calc.	Meter Orifice Press. Diff. ("H_2O) Actual	Stack Gas Temp. (°F)	Gas Sample Temp. @ Dry Gas Meter (°F) In	Gas Sample Temp. @ Dry Gas Meter (°F) Out	Sample Box Temp. (°F) Stop Watch	Last Impinger Test (°F)	Vacuum on Sample Train ("Hg)
1) 1	2 1/16	2.15	706.7	0.14	0.45	0.45	93	79	87	0		3.0
		2.25	710.9	0.14	0.45	0.45	93	81	84	10		3.6
2	6 29/33	2.35	715.5	0.18	0.58	0.58	92	84	83	5		3.0
		2.45	720.3	0.19	0.61	0.61	92	85	82	15		3.1
3	13 7/8	2.55	725.1	0.185	0.59	0.59	92	85	82	10		3.0
		3.65	729.8	0.185	0.59	0.59	92	86	83	5		3.0
4	33 1/8	3.15	734.4	0.18	0.58	0.58	96	86	82	15	85	3.0

Port and Traverse Point No.	Distance from End of Port (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
					Calc.	Actual		In	Out			
		3.25	739.2	0.18	0.58	0.58	96	86	83	10	90	3.0
5	40 3/32	3.35	744.1	0.21	0.665	0.665	96	86	83	5	82	3.2
		3.95	749.3	0.21	0.665	0.665	94	87	83	15	86	3.2
6	44 15/16	3.55	654.0	0.19	0.61	0.61	96	88	83	10	82	3.1
		4.05	758.82	0.19	0.61	0.61	94	88	83	5	89	3.1
		4.15	End.							15		

ENVIRONMENTAL ENGINEERING, INC.
Gainesville, Florida

SOURCE SAMPLING FIELD DATA SHEET

Plant W.R. Grace Wet phos Acid
Sampling Location Station E outlet
Date 1/5/72 Run No. 2
Time Start 9.00 Time End 11.00
Sampling Time/Point 20 min 6 pts (120 total)
DB ____ °F, WB ____ °F, VF @ DP ____ "Hg
Moisture 5 %, FDA 95, Gas Density Factor ____
Barometric Press 30 "Hg, Stack Press 30 "Hg
Weather Cloudy
Temp. 70 °F, W/D ____, W/S ____
Sample Box No. ____ Meter Box No. 5
Meter ΔH@ 1.66 Pitot Corr. Factor 0.83
Nozzle Dia. 1/4 in., Probe Length 6 ft
Probe Heater Setting ____
Stack Dimensions: Inside Diameter 47 in
Inside Area ____ ft²
Height ____ ft

Mat'l Processing Rate ____
Final Gas Meter Reading 822.485 ft³
Initial Gas Meter Reading 758.892 ft³
Total Condensate in Impingers 58 ml
Moisture in Silica Gel 248.1 231.9 [16.2 gm]
Silica Gel Container No. 301 Filter No. 72-040
Orsat: CO_2 ____
O_2 ____
CO ____
N_2 ____
Excess Air ____
Test Conducted by: C. Allen
E. Johnson
Remarks: ____

Port and Traverse Point No.	Distance from End of Port (in.)	Clock Time	Gas Meter Reading (ft^3) 758.892	Stack Velocity Head ("H_2O)	Meter Orifice Press. Diff. ("H_2O) Calc.	Meter Orifice Press. Diff. ("H_2O) Actual	Stack Gas Temp. (°F)	Gas Sample Temp. @ Dry Gas Meter (°F) In	Gas Sample Temp. @ Dry Gas Meter (°F) Out	Sample Box Temp. (°F) Stop Watch	Last Impinger Test (°F)	Vacuum on Sample Train ("Hg)
1		9.00	763.4	0.15	0.58	0.58	93	72	73	0	–	3.1
	8"	9.10	768.0	0.15	0.58	0.58	93	72	72	10	80	3.0
2	8"	9.20	773.0	0.18	0.68	0.68	93	72	72	5	83	3.1
	8"	9.30	778.0	0.18	0.68	0.68	93	72	72	15	84	3.2
3	8"	9.40	783.4	0.21	0.78	0.78	94	73	72	10	–	3.9
		9.50	788.9	0.22	0.815	0.815	95	74	72	5	–	4.0
4		10.00	794.7	0.24	0.90	0.90	95	74	72	15	75	4.7

Port and Traverse Point No.	Distance from End of Port (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
					Calc.	Actual		In	Out			
		1010	800.4	0.24	0.90	0.90	96	75	72	10	80	4.9
5		1020	806.5	0.24	0.90	0.90	94	75	73	5	81	5.0
		1030	812.0	0.23	0.86	0.86	94	75	73	15	82	5.0
6		1040	817.2	0.20	0.75	0.75	95	76	74	10	80	4.5
		1050	822.485	0.20	0.75	0.75	95	76	74	5	85	4.8
		11.00	end							15		

ENVIRONMENTAL ENGINEERING, INC.
Gainesville, Florida

SOURCE SAMPLING FIELD DATA SHEET

Plant W. R. Grace Wet phosAcid.
Sampling Location Station E outlet
Date 1/5/71 Run No. 3
Time Start 12.00 Time End 2.00 14.00
Sampling Time/Point 20 min 6 pts 120 tot.
DB ____ °F, WB ____ °F, VF @ DP ____ "Hg
Moisture 5 %, FDA 0.95, Gas Density Factor ____
Barometric Press 30 "Hg, Stack Press 30 "Hg
Weather Clear Hot
Temp. 80 °F, W/D ____, W/S ____
Sample Box No. ____ Meter Box No. 5
Meter ΔH@ 1.68 Pitot Corr. Factor 0.83
Nozzle Dia. 1/4 in., Probe Length 6 ft
Probe Heater Setting ____
Stack Dimensions: Inside Diameter 47 in
Inside Area ____ ft2
Height ____ ft

Mat'l Processing Rate ____
Final Gas Meter Reading 886.763 ft3
Initial Gas Meter Reading 822.485 ft3
Total Condensate in Impingers 60 ml
Moisture in Silica Gel 249.6 235.8 13.8 gm
Silica Gel Container No. 500 Filter No. 72-043
Orsat: CO_2 8.0
O_2 ____
CO ____
N_2 ____
Excess Air ____
Test Conducted by: C. Allen
E Johnson
Remarks: ____

Port and Traverse Point No.	Distance from End of Port (in.)	Clock Time	Gas Meter Reading (ft^3) 822.485	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) Stop Watch	Last Impinger Test (°F)	Vacuum on Sample Train ("Hg)
					Calc.	Actual		In	Out			
1	1	12.00	827.3	0.17	0.65	0.65	95	78	79	0	—	3.1
	8	12.10	832.1	0.16	0.605	0.603	96	79	78	10	—	3.0
2	Same as Run #1	12.20	832.3	0.19	0.715	0.715	96	79	78	5	—	3.6
		12.30	842.6	0.20	0.75	0.75	97	80	78	15	—	3.8
3		12.40	848.2	0.23	0.86	0.86	97	81	79	10	—	4.2
		12.50	854.1	0.23	0.86	0.86	97	82	80	5	—	4.3
4		1.00	859.5	0.23	0.86	0.86	96	83	80	15	82	4.4

Port and Traverse Point No.	Distance from End of Port (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
					Calc.	Actual		In	Out			
		110	865.8	0.23	0.86	0.86	97	83	87	10	85	4.6
5		120	870.7	0.20	0.75	0.75	97	83	81	5	82	4.0
		130	876.0	0.20	0.75	0.75	97	83	81	15	89	4.0
6		140	881.8	0.20	0.75	0.75	96	84	82	10	85	4.1
		150	886.763	0.20	0.75	0.75	96	84	83	5	83	4.1
		2.00	end							15		

GRAB SAMPLE COLLECTION

GRAB SAMPLE DATA SHEET

Plant W.R. GRACE, BARTOW, FLA

EPA Sample No.	351PD	351 PD	351 PD	351PD
Run No.	1	1	1	1-COMPOSITE
Date	1-4-72	1-4-72	1-4-71	1-4-72
Time	2:30	3:20	4:00	4:10
Sampling Point	A	A	A	A
Temperature, °F	76	77	77	75
pH	1.8	1.80	1.85	1.95
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant W.R. Grace Bartow, Fla

EPA Sample No.	352 PD	352 PD	352 PD	352 PD
Run No.	1	1	1	1-Composite
Date	1-4-72	1-4-71	1-4-71	1-4-71
Time	2:30	3:20	5:00	4:15
Sampling Point	B,	B,	B,	B,
Temperature, °F	90	90	90	81
pH	1.85	1.70	1.95	1.95
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant W R Grace' Barlow, Florida

EPA Sample No.	353 PD	353 PD	353 PD	353 PD
Run No.	1	1	1	1 - Composite
Date	1-4-72	1-4-72	1-4-72	1-4-72
Time	2:30	3:20	4:00	4:20
Sampling Point	B_2	B_2	B_2	B_2
Temperature, °F	90	88	91	83
pH	1.65	1.65	2.0	1.95
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant W R Grace Barlow Fla.

EPA Sample No.	354PD	354PD	354PD	354PD
Run No.	1	1	1	1
Date	1-4-72	1-4-72	1-4-71	
Time	2:30	2:20	4:00	
Sampling Point	Calc			
Temperature, °F				
pH				
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant W R Grace · Bartow

EPA Sample No.	366 PD	366 PD	366 PD	366 PD
Run No.	1	1	1	1
Date	1-4-72	1-4-72	1-4-72	1-4-72
Time	2:40	3:40	4:25	4:50
Sampling Point	F	F	F	F-Comp
Temperature, °F	95	94	94	86
pH	1.25	1.7	2.1	2.0
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant _____

EPA Sample No.	367PD	367PD	367PD	367PD
Run No.	1	1	1	1
Date	1-4-72	1-4-72	1-4-72	1-4-72
Time	2:40	3:40	4:20	4:35
Sampling Point	G	G	G	G-Pump
Temperature, °F	122	138	121	197
pH	1.9	1.95	2.15	2.05
Fluorides				
P_2O_5				
Trace Metals				

Remarks _____

GRAB SAMPLE DATA SHEET

Plant W R Grace Bartow Fla.

EPA Sample No.	371PD	371PD	371PD	371PD
Run No.	2	2	2	2 - Comp
Date	1-5-72	1-5-72	1-5-72	1-5-72
Time	9:00	10:00	10:50	11:00
Sampling Point	A	A	A	A
Temperature, °F	74	74	74	74
pH	2.1	2.0	2.0	2.0
Fluorides				
P_2O_5				
Trace Metals				

Remarks _____

GRAB SAMPLE DATA SHEET

Plant W. R. Grace Bartow Fla.

EPA Sample No.	372PD	372PD	373PD	373PD
Run No.	2	2	2	2-Comp.
Date	1-5-72	1-5-72	1-5-72	1-5-72
Time	9:10	10:00	10:50	11:00
Sampling Point	B,	B,	B,	B.-Comp.
Temperature, °F	89	90	89	83
pH	2.1	2.0	2.0	2.0
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant W R Grace Bartow Fla

EPA Sample No.	373PD	373PD	373PD	373PD
Run No.	2	2	2	2 Comp
Date	1-5-72	1-5-72	1-5-72	1-5-72
Time	9:10	10:00	10:50	11:00
Sampling Point	B_2	B_2	B_2	B_2 - Comp
Temperature, °F	87	90	90	83
pH	2.1	2.1	2.0	2.0
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant W R Grace Bartow Fla

EPA Sample No.	374PD	374PD	374PD	374PB
Run No.	2	2	2	2-Comp
Date	1-5-72	1-5-72	1-5-72	1-5-72
Time	9:25	10:35	10:55	11:10
Sampling Point	F	F	F	F-Comp
Temperature, °F	90	90	86	83
pH	2.1	2.1	2.0	2.0
Fluorides				
P_2O_5				
Trace Metals				

Remarks: Sample taken from each Hydrowell and mix for Sample

GRAB SAMPLE DATA SHEET

Plant (1) W R Grace - Bartow Fla

EPA Sample No.	375 PD	375 PD	375 PD	375 PD
Run No.	2	2	2	2-Comp
Date	1-5-72	1-5-72	1-5-72	1-5-72
Time	9:25	10:35	10:55	11:15
Sampling Point	G	G	G	G-Comp
Temperature, °F	125	121	110	95
pH	2.1	2.1	2.1	2.1
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant W R Grace Bartow Fla

EPA Sample No.	376PD	376PD	376PD	376PD
Run No.	2	2	2	2
Date	1-5-72	1-5-72	1-5-72	1-5-72
Time	9:00	10:00	10:50	10:50
Sampling Point				
Temperature, °F				
pH				
Fluorides				
P_2O_5				
Trace Metals				

Remarks Cake

GRAB SAMPLE DATA SHEET

Plant U.I.R. Fraco Bartow Fla.

EPA Sample No.	390 PD	390 PD	390 PD	390 PD
Run No.	3	3	3	3 - Comp
Date	1-5-72	1-5-72	1-5-72	1-5-72
Time	12:15	1:15	1:30	2:00
Sampling Point	A	A	A	A
Temperature, °F	75	75	75	75
pH	1.9	2.0	2.0	1.95
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant W R Grace, Bartow Fla

EPA Sample No.	391PD	391PD	391PD	391DD
Run No.	3	3	3	3-Comp
Date	1-5-72	1-5-72	1-5-72	1-5-72
Time	12:15	1:15	1:50	2:05
Sampling Point	B,	B.	B.	B.
Temperature, °F	88	89	87	84
pH	1.9	1.8	1.9	1.9
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant W R Grace Baytown Fla

EPA Sample No.	392PD	392PD	392PD	392PD
Run No.	3	3	3	3-Comp
Date	1-5-72	1-5-72	1-5-72	1-5-72
Time	12:15	1:15	1:50	2:10
Sampling Point	B.	B.	B.	B. - Comp
Temperature, °F	91	93	92	85
pH	1.8	1.7	1.9	1.9
Fluorides				
P_2O_5				
Trace Metals				

Remarks

GRAB SAMPLE DATA SHEET

Plant IU P Grace

EPA Sample No.	395 PD	395 PD	395 PD	395 PD
Run No.	3	3	3	3
Date	1-5-72	1-5-72	1-5-72	1-5-72
Time	12:15	1:15	1:50	1:50
Sampling Point				
Temperature, °F				
pH				
Fluorides				
P_2O_5				
Trace Metals				

Remarks Cake

GRAB SAMPLE DATA SHEET

Plant W. R. Grace Barlow Fla

EPA Sample No.	393 PD	393 PD	393 PD	393 PD
Run No.	3	3	3	3-Comp
Date	1-5-72	1-5-72	1-5-72	1-5-72
Time	12:20	1:20	1:55	2:20
Sampling Point	F	F	F	F-Comp
Temperature, °F	90	91	91	87
pH	1.9	1.9	1.9	1.9
Fluorides				
P_2O_5				
Trace Metals				

Remarks Sample taken from two Hot wells and combined

GRAB SAMPLE DATA SHEET

Plant W. R Grace Baltimore Plo

EPA Sample No.	394/P1	394/P1	394/P1	[illegible]
Run No.	3	3	3	3 - Comp
Date	1-5-72	1-5-72	1-5-72	1-5-72
Time	12:20	1:20	*2:30	2:35
Sampling Point	G	G	G	G
Temperature, °F	113	118	107	100
pH	1.9	1.9	1.9	1.9
Fluorides				
P_2O_5				
Trace Metals				

Remarks * Had to take one extra Sample one taken at 1:55 also spill

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 or 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 piece suspended in a slurry of CAO, evaporated to dryness and ashed prio 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 E

Project Participants

PROJECT PARTICIPANTS AND TITLES

John Dollar	Project Manager
Bill Demery	Environmental Specialist
A.L. Wilson	Environmental Specialist
George Allen	Environmental Specialist
Bob Durgan	Environmental Specialist
Eric Johnson	Environmental Specialist
Russ Wicker	Environmental Specialist
Tony Arroyo	Computer Analyst
Jerome Rom	EPA
John Reynolds	EPA
Roy Neulicht	EPA