Mosaic Phosphate Company

Hydrogen Fluoride Emission Rate Measurements

South Pierce Mosaic Facility

12 – 18 May 2005

ARCADIS

Infrastructure, environment, buildings

Final Report
Hydrogen Fluoride Emission Rate Measurements

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Executive Summary

ARCADIS has completed the data analysis of the measurement campaign conducted between 12 May and 18 May 2005 at the South Pierce Mosaic Facility. We sampled for hydrogen fluoride (HF) fluxes at four different, well-defined areas (two up on the gypsum stack and one at each of the extreme ends of the cooling pond). At each area we deployed a Vertical Radial Plume Mapping configuration on the predicted downwind boundary of the sampled area using an IMACC OP-FTIR instrument. On the upwind boundary we deployed an AIL RAM2000 OP-FTIR instrument to measure HF background concentrations over a single optical path.

After thorough verification of the input downwind concentration data, wind data, upwind/background data, and flux calculations, we arrived at an emission factor for each of the sampled areas in units of ton/year/acre. We then integrated these emission factors, incorporating the appropriate acreage of the various emitting surfaces to obtain an estimate of total mass emission for the whole year encompassing all of the emitting water surfaces. It should be noted that our calculations are based solely on a week's data collected during daytime only; and that diurnal, seasonal, and operational variations are not considered in this total yearly emission rate estimate.

The emission factors for the cooling pond are much higher than the emission factors at gypsum stack (4.8 and 2.0 ton/tear for the inlet and end areas of the cooling pond respectively; 1.0 and 0.25 ton/year for the inlet and the NE corner areas of the gypsum stack respectively). All of the calculations of the total emission rate are assuming that the high emission factors in the inlet areas (both at the cooling pond and gypsum stack) represent hotspots of emissions at the local small area around the inlet, and the rest of surface area emits according to the lower measured emission factor. For example, it was assumed for the cooling pond that 57 acres out the total 58 emitted at the lower rate of 2.0 ton/year/acre (57x2.0=114 ton/year). The total estimated emission rate is **at least** 160 ton/year assuming spring daytime emissions level all year long.

1. Methodology

1.1 Open Path FTIR and Vertical Radial Plume Mapping

The Open-Path Fourier Transform Infrared (OP-FTIR) Spectrometer combined with the Vertical Radial Plume Mapping (VRPM) method is designed for area and fugitive source emission characterization. In the OP-FTIR spectrometer, an infrared light beam, modulated by a Michelson interferometer, is transmitted from a single telescope to a retro-reflecting mirror target, which is usually set up at a range of 100 to 500 m. The returned light signal is received by the single telescope and directed to a detector. The light is absorbed by the molecules in the beam path as the light propagates to the retro-reflecting mirror and again as the light is reflected back to the analyzer. Thus, the round-trip path of the light doubles the chemical absorption signal. One advantage of OP-FTIR monitoring is that the concentrations of a multitude of infrared absorbing gaseous chemicals can be detected and measured simultaneously, with high temporal resolution. The chemical vapor, emitted from an emission source, forms a plume, which is carried by the wind across the multiple infrared beams. The OP-FTIR concentration measurements can be used with wind data to calculate the emission rate applying the VRPM method.

The VRPM method maps the concentrations in the vertical plane by scanning the OP-FTIR system in a vertical plane downwind from an area source. One can obtain the plane-integrated concentration from the reconstructed concentration maps. The flux is calculated by multiplying the plane-integrated concentration by the wind speed component perpendicular to the vertical plane. Thus, the VRPM method leads to a direct measurement-based determination of the upwind source emission rate (Hashmonay et al., 1998; Hashmonay and Yost, 1999, Hashmonay et al., 2001).

Figure 1-1 shows a schematic of the experimental setup used for vertical scanning. Several mirrors are placed in various locations on a vertical plane in-line with the scanning OP-FTIR (denoted as the PI-ORS instrument in the figure). A vertical platform (scissorjack) is used to place two of the mirrors at a pre-determined height above the surface. The location of the vertical plane is selected so that it intersects the mean wind direction as close to perpendicular as is practical. Wind data is collected, concurrent with the OP-FTIR measurements, at the base and top of the vertical platform.

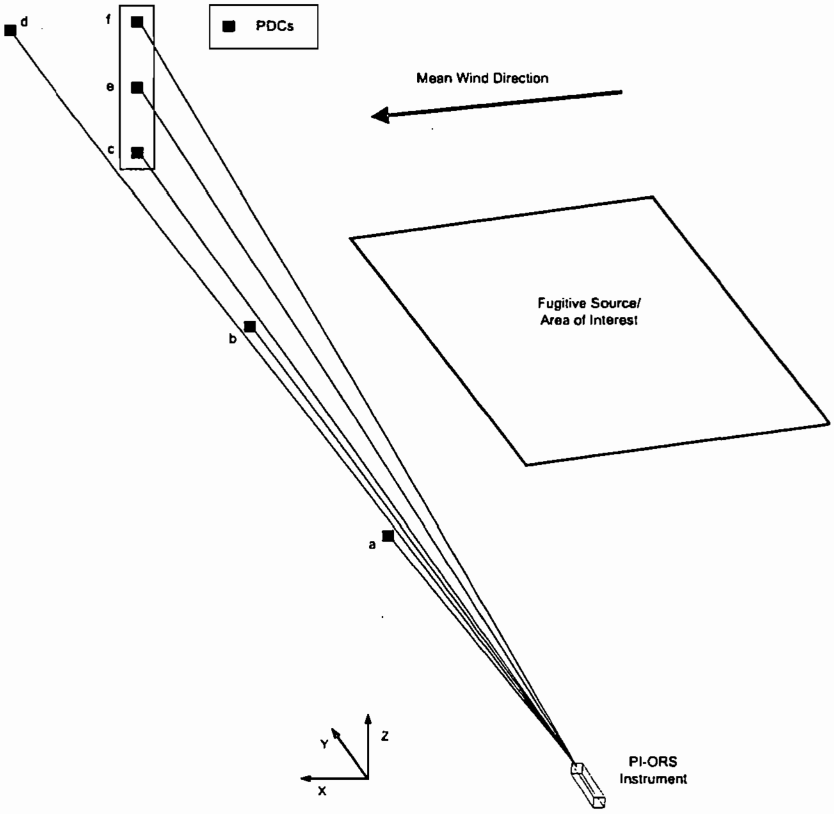

Figure 1-1. Example of a VRPM Configuration

ARCADIS uses the smooth basis function minimization (SBFM) reconstruction approach with up to two bivariate Gaussian functions in order to reconstruct the smoothed mass equivalent concentration map (one bivariate Gaussian function is used in cases where up to five mirrors are used in the configuration, and two bivariate Gaussian functions are used when six or more mirrors are used in the configuration). In the SBFM approach, a superposition of smooth basis functions is assumed to describe the distribution of concentrations, and the search is for the unknown parameters of the basis functions.

In each iterative step of the SBFM search procedure, the assumed path-integrated concentration (PIC) values, calculated from the new set of function parameters, are compared with the measured values. In order to compute the assumed PIC values, the superimposed basis functions are integrated along a matching beam path's direction and path-length. As mentioned earlier, ARCADIS' interest is in the plane-integrated concentration; therefore, we fit one (or two) bivariate Gaussian surface(s) to match the volume under the underlying true concentration distribution surface.

Once the parameters of the function are found for the matching run, we calculate the concentration values for every square elementary unit in a vertical domain. Then, we integrate these values, incorporating wind speed data at each height level to compute the flux. The wind speed and direction at each height level are determined through linear interpolation of the wind data collected with the two meteorological instruments (located at the base and top of the vertical structure). In this stage, we convert the concentration values from parts per million by volume to grams per cubic meter, considering the molecular weight of the target gas and ambient temperature. This enables us to directly calculate the flux in grams per second by multiplying by the wind speed component (in meters per second) normal to the vertical plane.

1.2 Specific Example Using Actual Data

The following section presents an example of data from an actual experiment conducted using the VRPM method. PIC and wind data were collected, and the downwind flux from an ethylene tracer gas release was calculated. The PIC data for each beam path of each measurement cycle is shown in Table 1-1. The average wind data for each cycle at four meters above the ground are also shown. Wind measurements were interpolated every 2 meters between the 2-m and 13-m heights. The wind direction from normal is measured clockwise. Positive values indicate that the wind is moving towards the diverging end of the beam paths and negative values indicate that the wind is moving towards the converging end of the beam paths emerging from the OP-FTIR.

The mass equivalent reconstructed plume along the measurement plane is shown in Figure 1-2. Data from the three ground-level mirrors provide spatial information on the plume along the crosswind axis (giving a plume center at approximately 80 m from the scanning OP-FTIR at the origin), and data from the two elevated mirrors provide information on the vertical concentration gradient of the plume.

Table 1-1. PIC and Wind Data for Ethylene Release for Flux Estimation

Beam Path no.	1	2	3	4	5	WS (m/s)	WD from Normal (deg)
Physical Beam Path Length (m)	52.1	90.1	137	141.5	141.9		
Cycle 1 (ppm-m)	0.0	13.4	17.8	0.0	0.0	2.7	5
Cycle 2 (ppm-m)	6.4	28.0	19.4	9.0	0.0	2.6	1
Cycle 3 (ppm-m)	4.5	23.4	23.1	8.8	0.0	2.1	0
Cycle 4 (ppm-m)	0.0	28.3	20.4	10.0	0.0	2.1	14
Cycle 5 (ppm-m)	5.0	19.8	34.7	0.0	0.0	2.9	13
Cycle 6 (ppm-m)	0.0	27.9	14.4	11.1	0.0	2.9	3
Cycle 7 (ppm-m)	10.8	14.1	28.5	0.0	0.0	3.0	-11
Cycle 8 (ppm-m)	0.0	10.3	49.1	4.9	0.0	2.3	18
Cycle 9 (ppm-m)	0.0	23.1	25.8	23.3	0.0	1.8	-2
Cycle 10 (ppm-m)	6.7	11.6	18.7	0.0	11.1	1.7	-15
Cycle 11 (ppm-m)	14.4	20.2	36.0	15.7	0.0	2.0	-8
Cycle 12 (ppm-m)	0.0	0.0	30.9	8.3	0.0	2.2	14
12-Cycle Average (ppm-m)	3.98	18.34	26.57	7.59	0.93	2.35	3

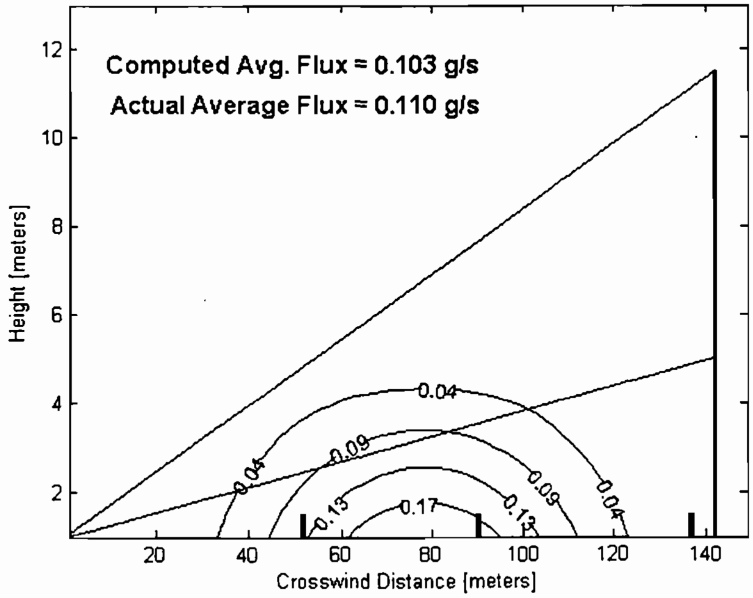

Figure 1-2. Example Reconstructed Plume Using the VRPM Method

2. Field Measurements and Results

2.1 Introduction

We have completed the data analysis of the measurement campaign conducted between 12 May and 18 May 2005 at the South Pierce Mosaic Facility. We sampled for hydrogen fluoride (HF) fluxes at four different, well-defined areas (two up on the gypsum stack and one at each of the extreme ends of the cooling pond). At each area we deployed a VRPM configuration on the predicted downwind boundary of the sampled area using the IMACC OP-FTIR instrument. On the upwind boundary we deployed an AIL RAM2000 OP-FTIR instrument to measure HF background concentrations over a single optical path.

After thorough verification of the input downwind concentration data, wind data, upwind/background data, and flux calculations, we arrived at an emission factor for each of the sampled areas in units of ton/year/acre. We then integrated these emission factors, incorporating the appropriate acreage of the various emitting surfaces to obtain an estimate of total mass emission for the whole year encompassing all of the emitting water surfaces. It should be noted that our calculations are based solely on a week's data collected during daytime only; and that diurnal, seasonal, and operational variations are not considered in this total yearly emission rate estimate.

2.2 Cooling Pond – Inlet Area

On 13 May 2005, the VRPM measurement configuration was set up at the western boundary of the cooling pond inlet area (see orange line in Figure 2-1). The upwind background measurement was at the eastern boundary of the sampled area (see blue line in Figure 2-1). The calculated emission data is for the time interval between 13:37 and 14:52. Although data was collected in this area for a longer period of time, this time interval was chosen for emission calculations because it includes the period that the prevailing winds were most consistent in representing emissions from the defined area of concern. The average ground level (about 2 m high) wind direction was 116° (clockwise from the north) as given in Table 2-1 and shown in Figure 2-1 by a light blue arrow. This configuration and wind direction define an estimated emitting water surface area of 1 acre. The measured input path average concentration data for the above mentioned time period are given in Table 2-2. Table 2-3 gives the 2σ error term of the classical least square (CLS) analysis, which essentially states the minimum detection limit for each measured concentration.

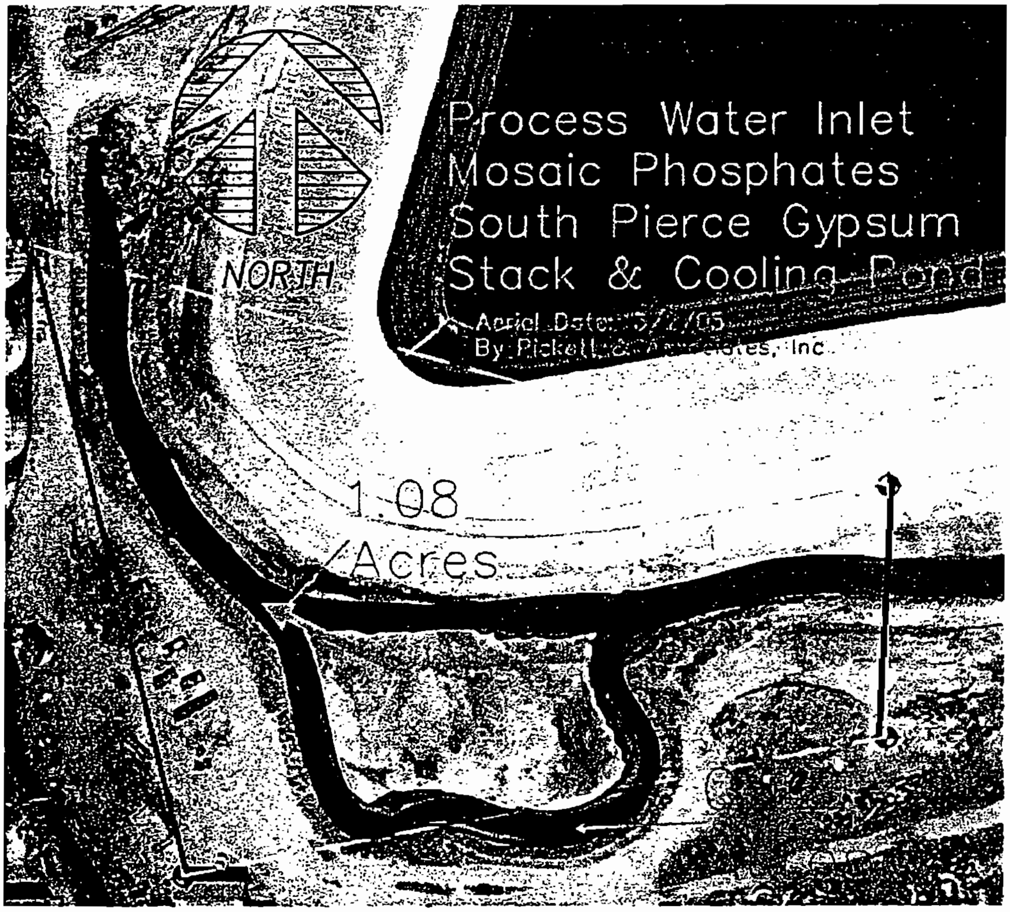

Figure 2-1. **Experimental Layout and Average Wind Direction at the Cooling Pond Inlet Area**

Table 2-1. Wind Speed and Direction at the Cooling Pond Inlet Area

Cycle#	Wind Speed [m/s]	Wind Direction [deg]
1	3.4	111
2	3.3	122
3	3.3	123
4	3.3	133
5	4.3	105
6	3.1	123
7	3.7	114
8	3.8	124
9	3.9	115
10	4.2	118
11	3.3	110
12	4.3	99
13	4.7	107
Average	**3.7**	**116**
Std. Dev.	**0.508**	**9.34**

Table 2-2. Input Average Concentration Data for the Cooling Pond Inlet Area

Cycle #	Concentration [ppb] Path 1	Concentration [ppb] Path 2	Concentration [ppb] Path 3	Concentration [ppb] Path 4	Concentration [ppb] Path 5
1	65	47	31	30	16
2	29	48	33	35	23
3	11	42	39	37	11
4	17	47	27	32	12
5	23	51	44	29	19
6	27	53	37	41	10
7	14	36	32	39	16
8	19	50	37	34	12
9	6	43	33	24	14
10	33	45	38	32	21
11	21	45	44	33	21
12	27	48	39	36	23
13	24	47	38	34	24
Average	**24**	**46**	**36**	**33**	**17**
Std. Dev.	**14**	**4.3**	**4.9**	**4.4**	**5.0**

Table 2-3. Error in Input Average Concentration Data for the Cooling Pond Inlet Area

Cycle #	Error [ppb] Path 1	Error [ppb] Path 2	Error [ppb] Path 3	Error [ppb] Path 4	Error [ppb] Path 5
1	26	8.9	7.5	7.2	6.2
2	15	8.6	7.8	7.5	6.6
3	14	8.7	7.5	7.1	6.2
4	15	8.5	7.5	6.8	6.1
5	14	8.8	7.2	7.3	6.2
6	15	8.7	7.5	6.9	6.3
7	15	8.3	7.4	7.3	6.3
8	15	8.5	7.3	7.1	6.3
9	15	8.7	7.3	7.5	6.3
10	15	8.4	7.5	7.0	6.3
11	14	8.4	7.4	7.3	6.2
12	14	8.6	7.6	6.9	6.2
13	14	8.7	7.7	7.3	6.0
Average	**15**	**8.6**	**7.5**	**7.2**	**6.2**
Std. Dev.	**3.21**	**0.173**	**0.164**	**0.225**	**0.139**

The averaged plume map and the respective measured flux for this time interval are given in Figure 2-2 along with the dimensions of the vertical plane defined by the VRPM beam configuration. The measured flux for this 1.08-acre area was 0.14 g/s, applying the measured background concentration of 20 ppb. Multiplying by 31,536,000 seconds in a year and divide by 908,000 grams in a ton will convert the units of the flux from g/s to ton/year for the all sampled emitting area. Dividing by the emitting area acreage provides the emission factor for sampled area. The calculated emission factor for the cooling pond inlet area is 4.5 ton/year/acre.

2.3 Cooling Pond – End Area

On 18 May 2005, the VRPM measurement configuration was set up at the western boundary of the cooling pond end area (see orange line in Figure 2-3). The upwind background measurement was at the eastern boundary of the sampled area (see blue line in Figure 2-3). The calculated emission data is for the time interval between 13:47 and 14:09. Although data was collected in this area for a longer period of time, this time interval was chosen for emission calculations because it includes the period that the prevailing winds were most consistent in representing emissions from the defined area of concern. The average ground level (about 2 m high) wind direction was 70° (clockwise from the north) as given in Table 2-4 and shown in Figure 2-3 by a light blue arrow. This configuration and wind direction define an estimated emitting water surface area of 4.3 acres. The measured input path average concentration data for the above mentioned time period are given in Table 2-5. Table 2-6 gives the 2σ error term of the classical least square (CLS) analysis, which essentially states the minimum detection limit for each measured concentration.

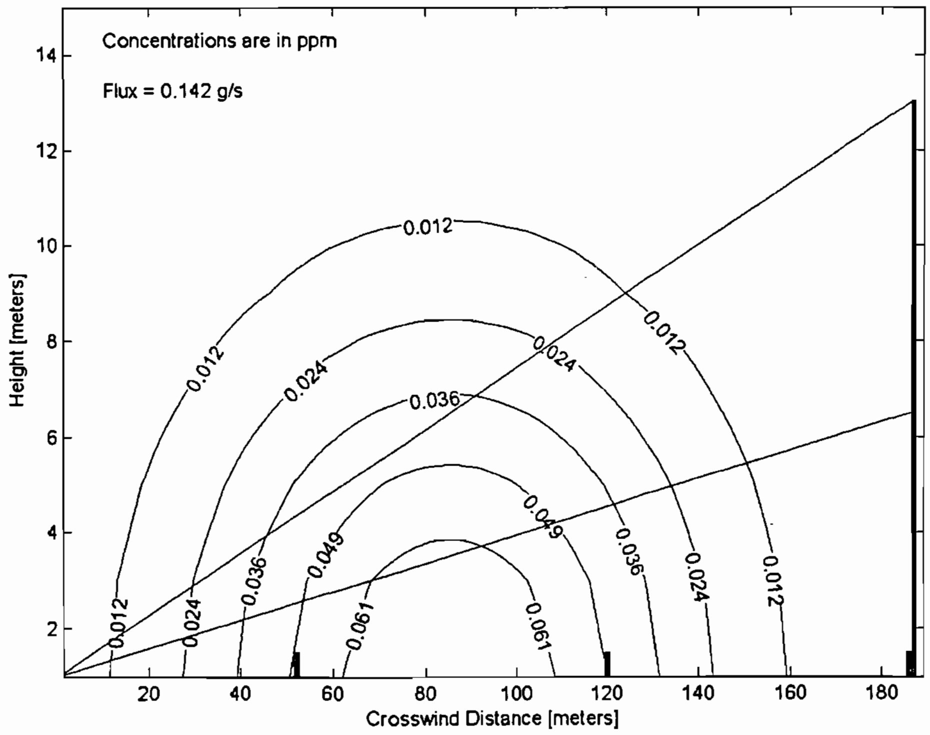

Figure 2-2. **The Averaged Plume Map and the Respective Measured Flux for the Cooling Pond Inlet Area**

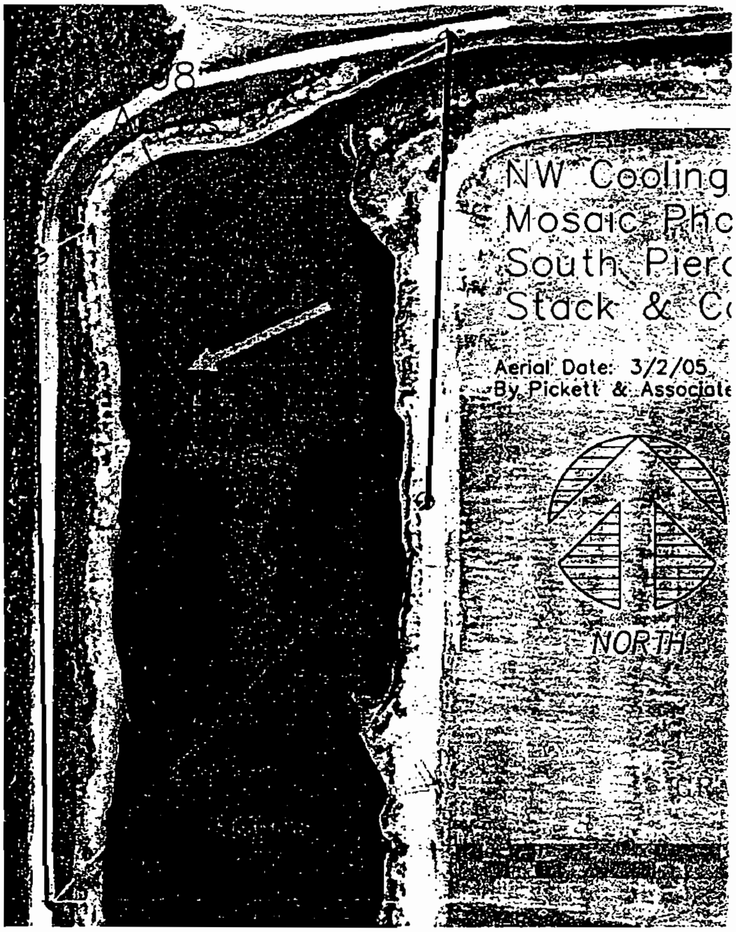

Figure 2-3. **Experimental Layout and Average Wind Direction at the Cooling Pond End Area**

Table 2-4. **Wind Speed and Direction at the Cooling Pond End Area**

Cycle#	Wind Speed [m/s]	Wind Direction [deg]
1	4.6	70
2	4.9	65
3	4.4	74
Average	**4.6**	**70**
Std. Dev.	**0.25**	**4.5**

Table 2-5. Input Average Concentration Data for the Cooling Pond End Area

Cycle #	Concentration [ppb] Path 1	Concentration [ppb] Path 2	Concentration [ppb] Path 3	Concentration [ppb] Path 4	Concentration [ppb] Path 5	Concentration [ppb] Path 6
1	96	92	64	56	44	52
2	84	92	59	64	45	37
3	70	72	74	65	46	39
Average	**83**	**85**	**65**	**61**	**45**	**43**
Std. Dev.	**13.1**	**11.5**	**7.64**	**4.93**	**1.00**	**8.14**

Table 2-6. Error in Input Average Concentration Data for the Cooling Pond End Area

Cycle #	Error [ppb] Path 1	Error [ppb] Path 2	Error [ppb] Path 3	Error [ppb] Path 4	Error [ppb] Path 5	Error [ppb] Path 6
1	15	11	9	8	7	8
2	14	11	10	8	7	8
3	14	11	10	8	7	8
Average	**14**	**11**	**9**	**8**	**7**	**8**
Std. Dev.	**0.58**	**0**	**0.58**	**0**	**0**	**0**

The averaged plume map and the respective measured flux for this time interval are given in Figure 2-4 along with the dimensions of the vertical plane defined by the VRPM beam configuration. The measured flux for this 4.3 acres area was 0.25 g/s, applying the measured background concentration of 10 ppb. The calculated emission factor for the cooling pond end area is 2.0 ton/year/acre.

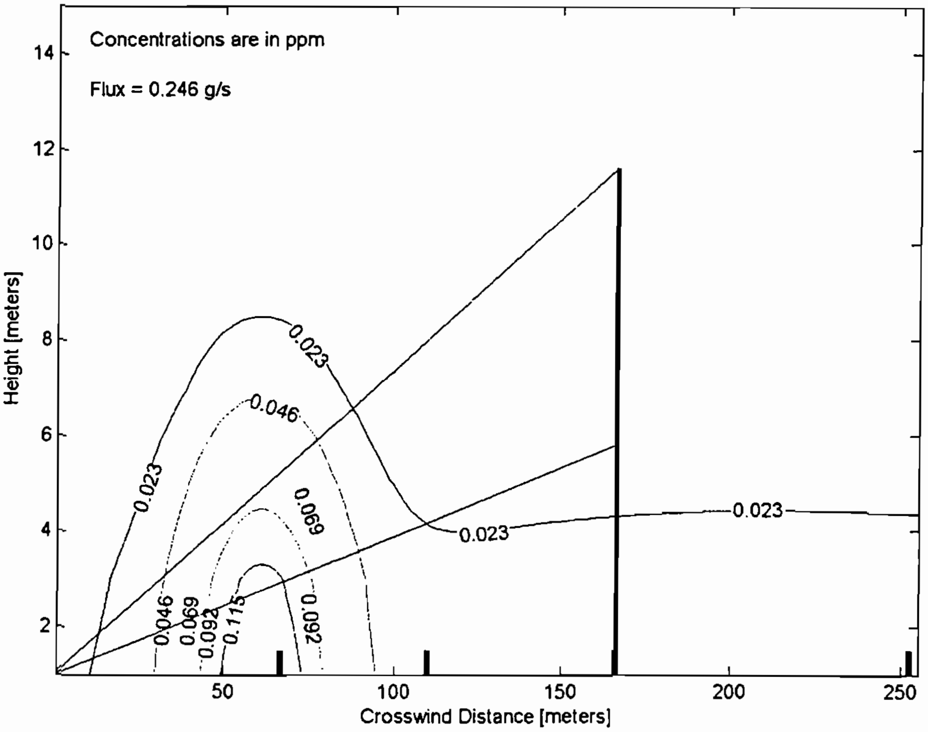

Figure 2-4. **The Averaged Plume Map and the Respective Measured Flux for the Cooling Pond End Area**

2.4 Gypsum Stack – Inlet Area

On 17 May 2005, the VRPM measurement configuration was set up at the southwestern boundary of the gypsum stack inlet area (see orange line in Figure 2-5). The upwind background measurement was at the southeastern boundary of the sampled area (see blue line in Figure 2-5). The calculated emission data is for the time interval between 12:27 and 12:50. Although data was collected in this area for a longer period of time, this time interval was chosen for emission calculations because it includes the period that the prevailing winds were most consistent in representing emissions from the defined area of concern. The average ground level (about 2 m high) wind direction was 85° (clockwise from the north) as given in Table 2-7 and shown in Figure 2-5 by a light blue arrow. This configuration and wind direction define an estimated emitting water surface area of 5.7 acres. The measured input path average concentration data for the above mentioned time period are given in Table 2-8. Table 2-9 gives the 2σ error term of the classical least square (CLS) analysis, which essentially states the minimum detection limit for each measured concentration.

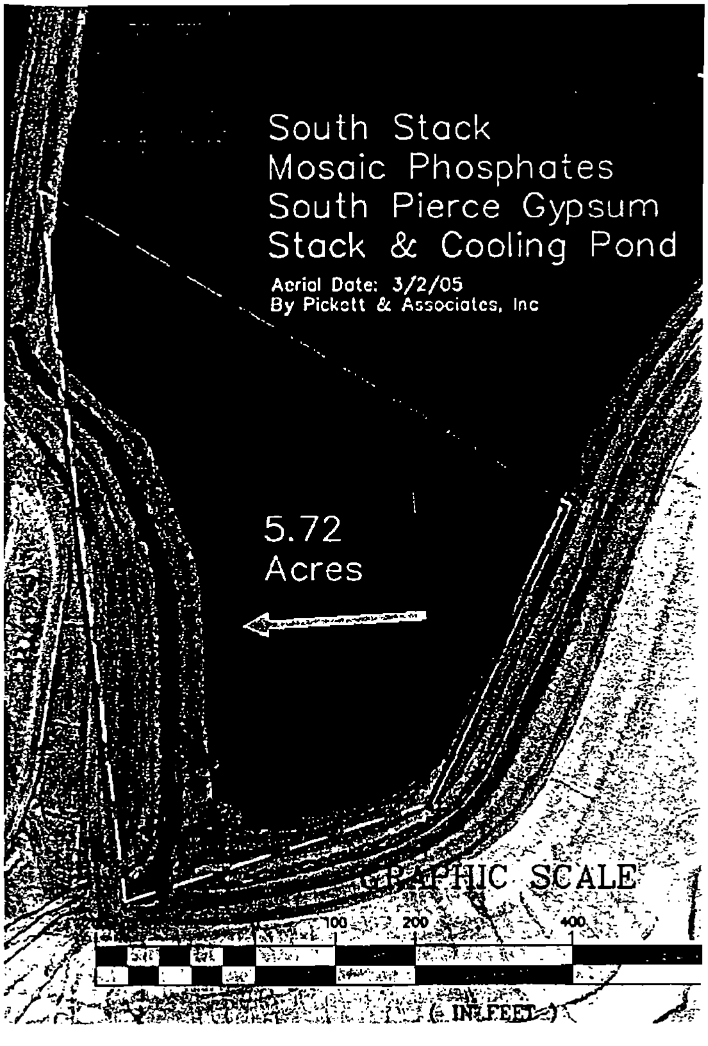

Figure 2-5. Experimental Layout and Average Wind Direction at the Gypsum Stack Inlet Area

Table 2-7. Wind Speed and Direction at the Gypsum Stack Inlet Area

Cycle#	Wind Speed [m/s]	Wind Direction [deg]
1	2.6	92.2
2	1.8	80.0
3	1.4	83.9
Average	**1.9**	**85.3**
Std. Dev.	**0.611**	**6.23**

Table 2-8. Input Average Concentration Data for the Gypsum Stack Inlet Area

Cycle #	Concentration [ppb] Path 1	Concentration [ppb] Path 2	Concentration [ppb] Path 3	Concentration [ppb] Path 4	Concentration [ppb] Path 5	Concentration [ppb] Path 6
1	92	133	83	88	63	39
2	71	106	81	129	23	12
3	58	107	74	101	58	37
Average	**73**	**115**	**79**	**106**	**48**	**29**
Std. Dev.	**17.2**	**15.3**	**4.73**	**20.9**	**21.8**	**15.1**

Table 2-9. Error in Input Average Concentration Data for the Gypsum Stack Inlet Area

Cycle #	Error [ppb] Path 1	Error [ppb] Path 2	Error [ppb] Path 3	Error [ppb] Path 4	Error [ppb] Path 5	Error [ppb] Path 6
1	11	9	8	7	7	6
2	10	8	8	9	6	6
3	10	8	8	8	6	7
Average	**10**	**9**	**8**	**8**	**7**	**6**
Std. Dev.	**0.58**	**0.58**	**0**	**1.0**	**0.58**	**0.58**

The averaged plume map and the respective measured flux for this time interval are given in Figure 2-6 along with the dimensions of the vertical plane defined by the VRPM beam configuration. The measured flux for this 5.7 acres area was 0.17 g/s, applying the measured background concentration of 20 ppb. The calculated emission factor for the gypsum stack inlet area is 1.0 ton/year/acre.

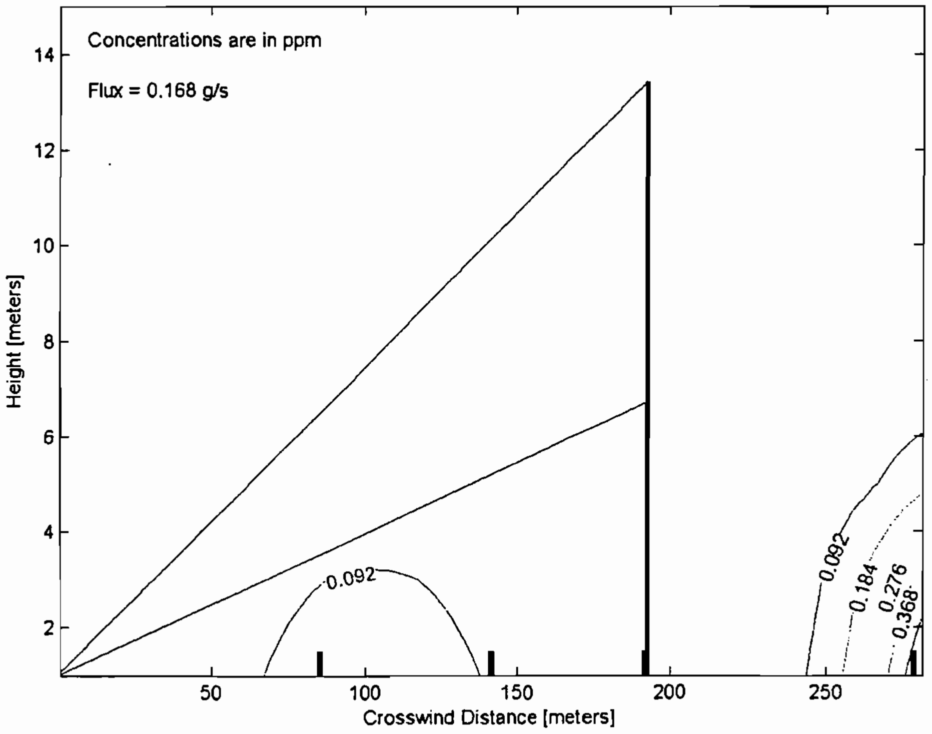

Figure 2-6. **The Averaged Plume Map and the Respective Measured Flux for the Gypsum Stack Inlet Area**

2.5 Gypsum Stack – NE Corner

On 16 May 2005, the VRPM measurement configuration was set up at the north boundary of the gypsum stack NE corner (see orange line in Figure 2-7). The upwind background measurement was at the southern boundary of the sampled area (see blue line in Figure 2-7). The calculated emission data is for the time interval between 15:47 and 15:55. Although data was collected in this area for a longer period of time, this time interval was chosen for emission calculations because it includes the period that the prevailing winds were most consistent in representing emissions from the defined area of concern. The average ground level (about 2 m high) wind direction was 203° (clockwise from the north) as given in Table 2-10 and shown in Figure 2-7 by a light blue arrow. This configuration and wind direction define an estimated emitting water surface area of 7.5 acres. The measured input path average concentration data for the above mentioned time period are given in Table 2-11. Table 2-12 gives the 2σ error term of the classical least square (CLS) analysis, which essentially states the minimum detection limit for each measured concentration.

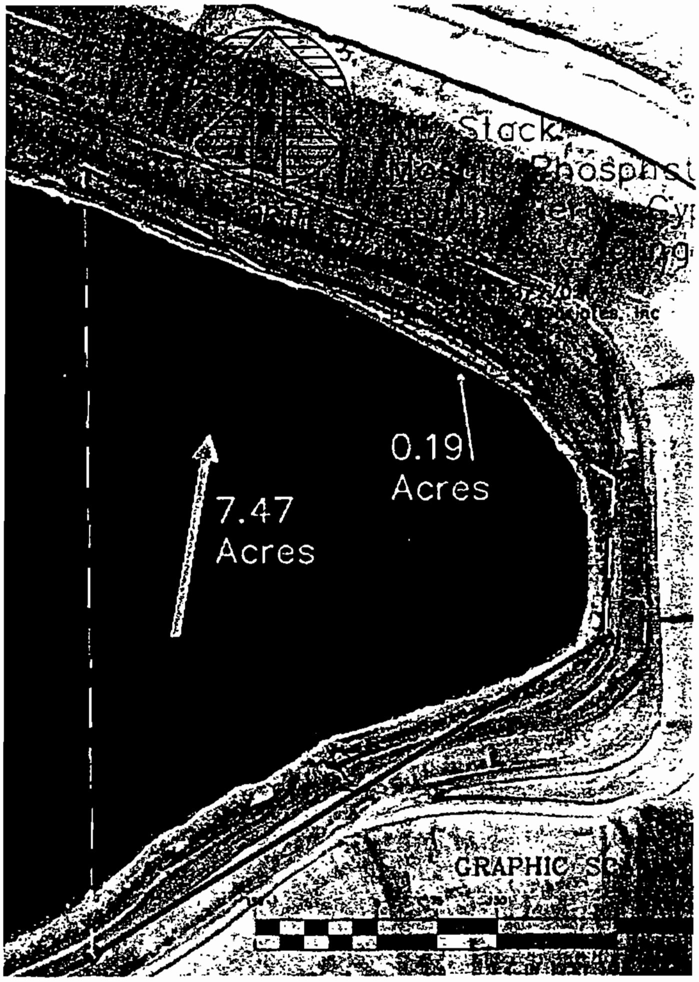

Figure 2-7. Experimental Layout and Average Wind Direction at the Gypsum Stack NE Corner

Table 2-10. Wind Speed and Direction at the Gypsum Stack NE Corner

Cycle#	Wind Speed [m/s]	Wind Direction [deg]
1	0.7	239.5
2	0.3	167.9
3	1.5	200.0
Average	**0.8**	**202.5**
Std. Dev.	**0.61**	**35.9**

Table 2-11. Input Average Concentration Data for the Cooling Pond Inlet Area

Cycle #	Concentration [ppb] Path 1	Concentration [ppb] Path 2	Concentration [ppb] Path 3	Concentration [ppb] Path 4	Concentration [ppb] Path 5	Concentration [ppb] Path 6
1	279	229	66	117	75	67
2	267	211	125	146	146	156
3	430	292	147	145	149	116
Average	**325**	**244**	**113**	**136**	**124**	**113**
Std. Dev.	**90.8**	**42.5**	**41.9**	**16.5**	**41.9**	**44.6**

Table 2-12. Error in Input Average Concentration Data for the Cooling Pond Inlet Area

Cycle #	**Error [ppb] Path 1**	**Error [ppb] Path 2**	**Error [ppb] Path 3**	**Error [ppb] Path 4**	**Error [ppb] Path 5**	**Error [ppb] Path 6**
1	39	12	7	8	11	7
2	23	12	9	9	12	10
3	36	15	9	10	11	7
Average	**33**	**13**	**8**	**9**	**11**	**8**
Std. Dev.	**8.5**	**1.7**	**1.2**	**1.0**	**0.58**	**1.7**

The averaged plume map and the respective measured flux for this time interval are given in Figure 2-8 along with the dimensions of the vertical plane defined by the

VRPM beam configuration. The measured flux for this 7.5 acres area was 0.054 g/s, applying the measured background concentration of 60 ppb. The calculated emission factor for the NE corner of the gypsum stack is 0.25 ton/year/acre.

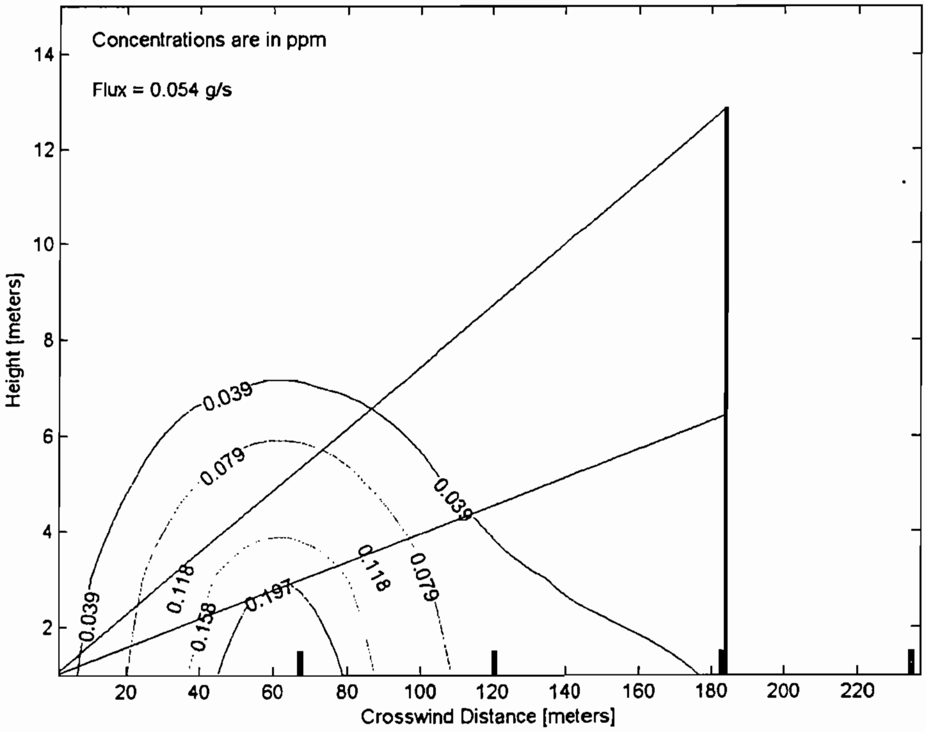

Figure 2-8. **The Averaged Plume Map and the Respective Measured Flux for the Gypsum Stack NE Corner**

3. QA/QC

3.1 Validation of Concentration Data Collected with the OP-FTIR

During the analysis of the OP-FTIR data, a validation procedure was performed to aid in identifying the presence of hydrogen fluoride in the dataset. This validation procedure involves visually comparing an example of the measured spectra to a laboratory-measured reference spectrum.

Figure 3-1 shows an example of a validation done using a spectrum collected at the South Stack area on 17 May 2005. Hydrogen fluoride was detected in this particular spectrum. The hydrogen fluoride features can be seen in the measured field spectrum (red trace). Classical Least Squares (CLS) analysis performed on this spectrum resulted in determinations of 155.1 ± 7.0 ppb of hydrogen fluoride. The uncertainty value is equal to three times the standard error in the regression fit of the measured spectrum to a calibrated reference spectrum, propagated to the concentration determination.

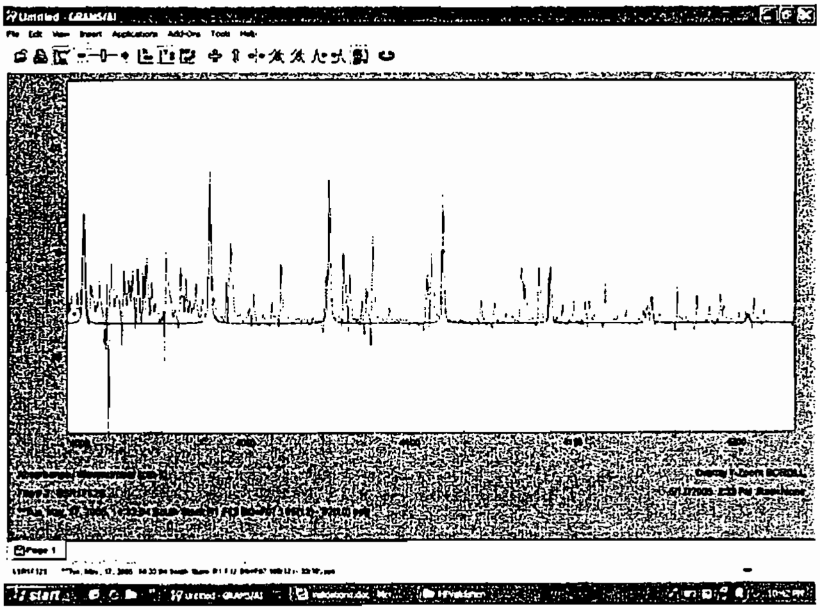

Figure 3-1. **Comparison of a Spectrum Measured at the Site (red trace) to the Reference Spectrum of Hydrogen Fluoride (blue trace)**

Figure 3-2 shows the same comparison as Figure 3-1, after water vapor spectrum was subtracted from the field spectrum to better show the six hydrogen fluoride absorption lines.

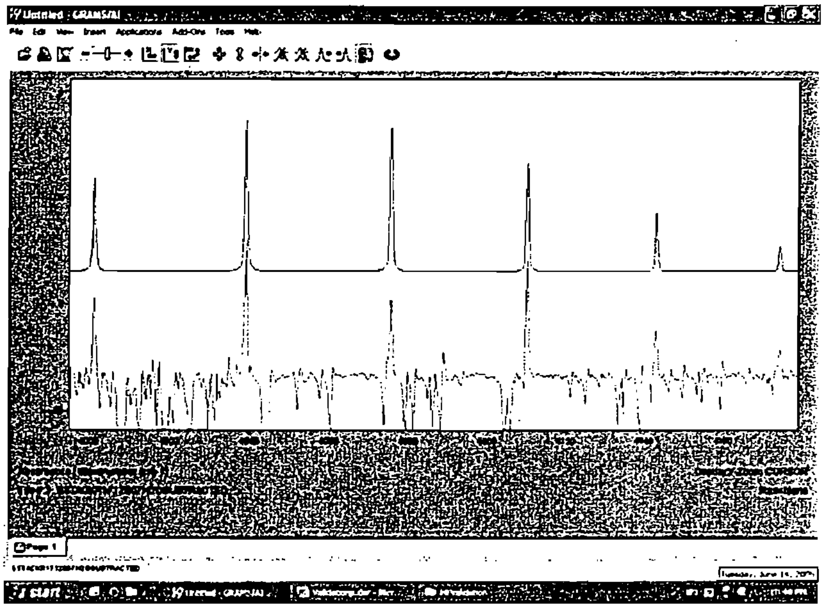

Figure 3-2. **Comparison of a Spectrum Measured at the Site (red trace) to the Reference Spectrum of Hydrogen Fluoride (blue trace) after Water Vapor Subtraction from the Field Spectrum**

3.2 Data Quality Indicator (DQI) Check for Analyte PIC Measurement

The precision and accuracy of the OP-FTIR PIC measurements was assessed by analyzing the measured methane and nitrous oxide concentrations in the atmosphere. A typical background atmospheric concentration for methane is approximately 1.7 ppm, and nitrous oxide is approximately 315 ppb. This value may fluctuate slightly due to seasonal variations or elevation of the site. Tables 3-1 and 3-2 present a sample of the methane and nitrous oxide concentrations, respectively, measured along the longest beam path used in each of the four survey areas.

Table 3-1. Sample of Sequential 1-minute Average Methane Concentrations (in ppm) Measured at the Site

Cycle	Cooling Pond-Inlet Area	Cooling Pond-End Area	Gypsum Stack-Inlet Area	Gypsum Stack-NE Corner
1	1.81	1.73	1.73	1.73
2	1.82	1.73	1.72	1.73
3	1.81	1.73	1.74	1.76
4	1.83	1.74	1.72	1.76
5	1.83	1.73	1.75	1.74
6	1.83	1.73	1.71	1.78
7	1.84	1.73	1.72	1.74
8	1.84	1.73	1.71	1.78
9	1.85	1.73	1.71	1.77
10	1.84	1.72	1.76	1.81
Avg.	**1.83**	**1.73**	**1.73**	**1.76**
Std. Dev.	**0.0133**	**0.0047**	**0.0177**	**0.0258**

Table 3-2. Sample of Sequential 1-minute Average Nitrous Oxide Concentrations (in ppb) Measured at the Site

Cycle	Cooling Pond-Inlet Area	Cooling Pond-End Area	Gypsum Stack-Inlet Area	Gypsum Stack-NE Corner
1	326	328	324	312
2	328	325	320	314
3	325	323	321	315
4	325	325	319	314
5	326	323	325	314
6	325	321	320	317
7	326	318	315	318
8	328	324	312	316
9	328	324	320	314
10	327	321	320	320
Avg.	**326**	**323**	**320**	**315**
Std. Dev.	**1.265**	**2.741**	**3.806**	**2.366**

A review of the methane and nitrous oxide concentrations measured during the project indicates that greater than 99 percent of the concentration values measured met the acceptance criteria for precision (±5%) and accuracy (± 10% of atmospheric background levels).

3.3 VRPM Validation

To perform a check for reasonableness in the results of the calculated flux by the VRPM software (Figure 3-3), it is necessary to compare an estimated flux value with the flux value calculated using the VRPM configuration. This check is performed using the plume map generated by the VRPM configuration. The following steps detail how to perform this check as specified in the EPA's RPM draft protocol:

1. Construct horizontal paths across the map, at heights of 1 m, 3 m, 5 m, and so on until the next horizontal line will be above the lowest concentration contour of the plume map.
2. Divide each path into ten equidistant segments.
3. Mark the center point of each segment to create ten points along the path.
4. Estimate the concentration at each of the ten points by interpolating between the concentration contours.
5. Sum the concentration values (in ppm) at each of the ten points along the path. Multiply this value by 1/10 of the actual distance (in meters) of the path. The resulting value, which is the reconstructed PIC, is in ppm-m.
6. Repeat this procedure for each of the horizontal paths. Sum the seven values, and multiply this sum by 2. The resulting value, which is the plane-integrated concentration, is in ppm-m^2.
7. Multiply the plane-integrated concentration by the cosine of the observed surface wind direction, obtained directly from the data.
8. Multiply by the observed surface wind speed (in meters/second).
9. Take the resulting value of steps 1 to 8, and plug into the following equation (the value is denoted by RESULT in the equation):

$$\left[\left(\frac{MolecularWeightofCompound}{0.0245}\right)(RESULT)\right](.000001)$$

10. The resulting value is the manually estimated flux, in grams per second. Compare this value to the calculated flux by the VRPM software (depicted at the top of the plume map). The estimated flux must be within 30 percent of the calculated flux. If this is not the case, repeat this procedure to confirm the results. If the results are confirmed, the parameters input into the algorithm are verified for accuracy. Figure 3-3 shows a plume map generated from data collected at the site, and the interpolated values used to perform the VRPM check.

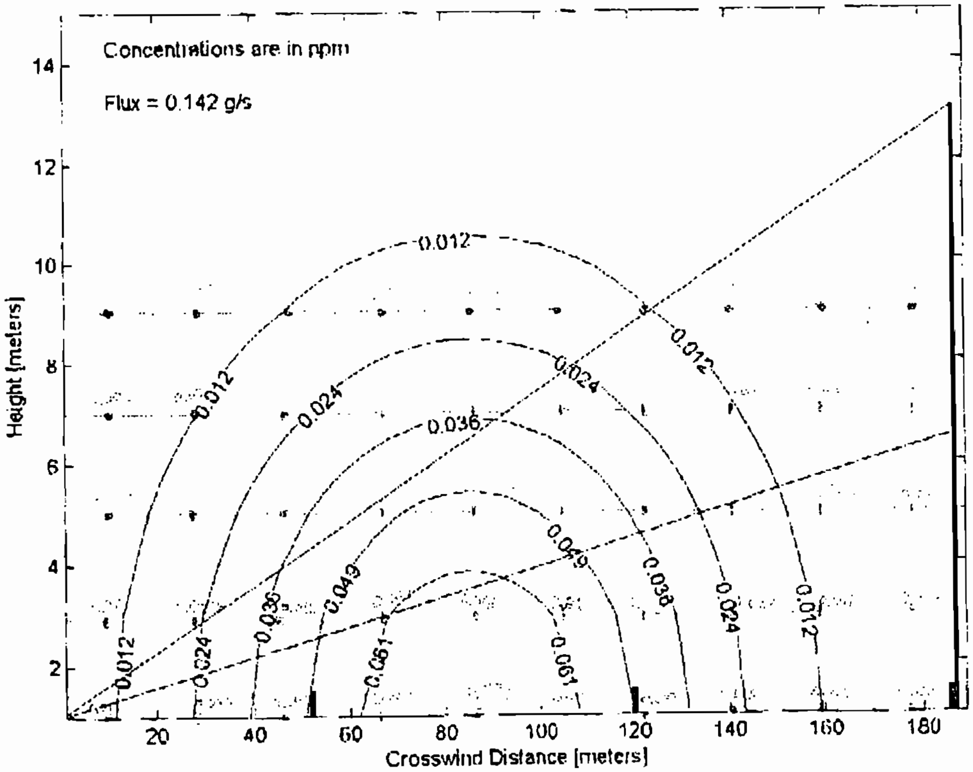

Figure 3-3. **The Plume Map from the Cooling Pond Inlet Area with Manual VRPM Validation Procedure Performed**

Performing this manual calculation of the flux provided a value of 0.12, which is within the ±30 percent criterion (-15%). There is another simple check that may verify the accuracy of the plume map. We compared the ground level averaged measured PIC with the manually calculated PIC from the map. From Table 2-2 the average concentration at the ground level (path 3) is 36 ppb. Multiplying by the pathlength (186 m) and divide by 1000 (conversion to ppm) gives 6.7 ppm-m as the measured PIC. The manually calculated PIC in our VRPM validation procedure was 6.9 ppm-m.

4. Summary

4.1 Total Yearly Estimated HF Emission Rate for South Pierce Facility

The results are summarized in Table 4-1 below:

Table 4-1. Summary of the Emission Results for the Mosaic South Pierce Facility

	Area [acres]	Measured Flux [g/s]	Emission Factor [ton/yr/acre]	Total Area [acres]	Total Emission Rate [ton/year]
Cooling Pond Inlet Area	1.08	0.14	4.5	58	119
Cooling Pond End (NW of the stack)	4.3	0.25	2.0		
Gypsum Stack Inlet (south location)	5.7	0.17	1.0	155	43
Gypsum Stack NE Corner	7.5	0.054	0.25		

The emission factors for the cooling pond are much higher than the emission factors at gypsum stack. It should be noted that although the measured flux (0.14 g/s) at inlet area to the cooling pond is small, the fact that it represents an area of only 1 acre results in highest emission factor. The plume map from Figure 3-3 demonstrates the actual low concentrations that generated this high emission factor.

All of the calculations of the total emission rate are assuming that the high emission factors in the inlet areas (both at the cooling pond and gypsum stack) represent hotspots of emissions at the local small area around the inlet, and the rest of surface area emits according to the lower measured emission factor. For example, it was assumed that 57 acres out the total 58 emitted at the lower rate of 2.0 ton/year/acre (57x2.0=114 ton/year). This drives the results of the total emission rate to minimal estimates and should be viewed as such.

The total estimated emission rate is **at least** 160 ton/year for the meteorological conditions tested.

5. References

Hashmonay, R.A., D.F. Natschke, K.Wagoner, D.B. Harris, E.L.Thompson, and M.G. Yost, “Field evaluation of a method for estimating gaseous fluxes from area sources using open-path Fourier transform infrared,” *Environ. Sci. Technol.*, 35, 2309-2313, 2001.

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Hashmonay, R.A., M.G. Yost, D.B. Harris, and E.L. Thompson, “Simulation study for gaseous fluxes from an area source using computed tomography and optical remote sensing,” presented at SPIE Conference on Environmental Monitoring and Remediation Technologies, Boston, MA, Nov. 1998, in SPIE, Vol. 3534, 405-410.

URS

Extractive FTIR Testing by EPA Method 320 of the 3rd Train, AFI, MultiPhos C, A/B, and Clarifier Stack Emissions

June 2002

Submitted to:

IMC Phosphates MP, Inc.
Mulberry, Florida

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APPENDIX A – Spiking/Validation Procedures

1.0 Executive Summary and Introduction

Total HF emissions from the process stacks (3^{rd} Train, AFI, MultiPhos C, A/B, and the Clarifier) are <5 ton/year, assuming a measured average concentration of 161 ppbv at the 3^{rd} Train stack, 1.507 ppmv at the AFI stack, 109 ppbv at the MultiPhos C stack, 307 ppbv at the MultiPhos A/B stack and 81 pbbv at the Clarification stack. It is important to note that the stack chemistry also prohibits sample filtering at elevated temperatures, for biasing effects due to particulate outgassing lead to the inaccurate reporting of HF vapor emissions. A sampling probe and filter assembly maintained just above stack temperatures was used to prevent condensation losses and aerosol dissociation, so that accurate reporting of HF vapor was ensured. The extractive FTIR method was validated by Method 301 spiking for HF monitoring at CF Industries' B-PAP stack just prior to arrival and by EPA Method 320 spiking at all IMC stacks.

This document presents the results of a series of emission stack gas measurement tests performed by URS for IMC Phosphates MP Inc. at the 3rd Train, AFI, MultiPhos C, A/B and Clarifier stacks. The measurements were made in a continuous and real-time fashion with an extractive Fourier Transform Infrared (FTIR) spectroscopic system. The system also included an aerosol/particulate filter assembly, with accommodations for spiking (diluting) the sample stream with certified gas standards per EPA Method 301 and 320.

The objective of this testing was to measure the gaseous HF emissions at each stack over a continuous one hour period during normal process conditions. The accuracy of the analytical monitoring method was validated by Method 301 to ensure that all the data were representative of actual stack emissions.

Testing took place between 14 May and 21 May 2002. The test schedule, sampling locations, and test conditions are summarized in Table 1-1. The process and stack conditions are summarized in Table 1-2.

2.0 Principles of FTIR Monitoring

Almost every chemical compound absorbs infrared (IR) light to some degree in a particular region of the mid-infrared spectrum. These absorption properties can be used to identify and quantify chemical compounds in a complex mixture of gases. As stated by Beer's Law, the magnitude of a compound's IR absorbance is directly proportional to the product of its concentration in the mixture and the sample cell optical path length. This is otherwise known as the compound's *optical depth.* The extractive FTIR instrument used by URS is able to achieve parts-per-billion (ppb) detection levels because the optical path length within the measurement cell is magnified many times by reflecting the IR beam between a series of mirrors before it reaches the detector. The mirrors provide a fixed optical path length best suited to the gas mixture being sampled. In this case, an optical path length of 20.1 meters was utilized.

2.1 The Spectrum Analysis Method

An infrared spectrum analysis is performed by matching the features of an observed spectrum to those of reference standards. If more than one feature is present in the same region, then a linear combination of references is used to match the compound feature. The standards are scaled to match the observed band intensities in the sample. This scaling also matches the unknown concentrations. An infrared spectrum can be collected and analyzed in approximately one second, but spectra are normally averaged over a one- or two-minute integration period to produce adequate signal-to-noise limits and ppb detection levels.

The scaled references are added together to produce a composite which represents the best match with the sample. A classical least squares mathematical function is used to match the standards' absorption profiles with those of the observed spectrum in specified spectral analysis regions. The compounds of interest together with compounds expected to cause spectral interference are included in the analysis region.

2.1.1 Creating the Spectrum Analysis Method

The spectrum analysis method used for the stack tests at IMC Phosphates was developed by selecting the spectral regions and sub-regions that are least affected by primary IR absorbers (H_2O and CO_2, in this case) while also producing the best detection limit possible for the target compounds (HF, SiF_4 and NH_3). Typically, an analysis method will be iteratively refined by using it to analyze a representative set of infrared spectra while varying the method. The optimum method is indicated when both the 95% confidence levels and the bias on the individual compounds are minimized.

3.0 Stack Sampling Considerations

The stack chemistry at each location dictates how the sampling system must be configured to deliver representative gas streams to the FTIR sample cell. Regardless of sampling location, the sample probe and filter assembly must be maintained at a relatively low temperature (60-65°C), but still above stack temperatures, to preclude any condensation losses and the dissociation of HF-containing particulates (aerosols) in the filters. It was the placement of the spiking tee that required special consideration at each stack because of the chemistry involved. The tee must be located at a point within the sampling system as close as possible to where stack gas is being initially extracted. Therefore, the spiking tee was located immediately downstream of the filters, but upstream of the extraction line, when sampling the 3rd Train stack.

However, it was realized that HF recoveries would still not be sufficient at this spiking tee location for the other stacks. The potential presence of various particulates at AFI and MultiFos C, A/B requires the introduction of the HF standard into a hot (above 100°C) gas stream to prevent the nucleation of gaseous HF (leading to incomplete recoveries). As a result, the spiking tee was placed further downstream within the sampling system, downstream of the extraction line but upstream of the sample cell. This location ensured mixing of the HF standard with stack gas under hot conditions.

It was also required to sample stack gas under isokinetic conditions. This meant matching the exhaust gas velocity within the stack to the sample probe extraction flow rate at each traverse point. Following EPA Method 1, the number of points was determined along two right angle traverses at each of the stacks. The number of traverse points was determined to be: 3rd Train - 6 points/traverse (12 points total); AFI - 10 points/traverse (20 points total); MultiFos C - 12 points/traverse (24 points total); MultiFos A/B - 6 points/traverse (12 points total); and the Clarifier - 4 points/traverse (8 points total). Table 3-1 summarizes the stack flow rate at each location and traverse point (as measured by IMC Phosphates personnel via EPA Method 2 during each compliance test) and the corresponding FTIR extraction flow rate.

5.0 Stack Sampling Test Results

The results for each emissions test are reported in this section. They are categorized according to sampling location. Each compliance test consisted of a set of pre-test HF spikes (per Method 320), a continuous one-hour sampling run, with the sampling time at each traverse point equally divided, and a set of post-test HF spikes (per Method 320). See Sections and 4 for details of the sampling system and method.

5.1 Third Train Emissions Test

Extractive FTIR monitoring of HF at 3rd Train was conducted on 14 May 2002. The emission profile is plotted in Figure 5-1. Noted on the graph is when stack sampling began and ended, along with the times when the probe was out of the stack and ambient air was sampled. Upon averaging the concentrations reported in Figure 5-1, and factoring in the average stack flow rate reported in Table 3-1, an average HF mass emission rate of 0.010 lb/hr can be calculated. Following a convention often recommended by the EPA, a value of ½ the FTIR method MDL (Minimum Detection Limit) was assigned to each data point that was initially reported below the MDL for purposes of averaging. Accordingly, a value of 28 ppbv was substituted for non-detects (the MDL for hydrogen fluoride was conservatively estimated to be 56 ppbv - see Section 2.1.1.)

HF spiking, per Method 320, was conducted prior to and immediately after this emissions test. The results are summarized in Section 5.1.1.

5.1.1 Third Train Validation Spikes

Spiking with an HF certified gas standard was performed as stack gas was continuously extracted and analyzed at a central traverse point within the 3rd Train stack. A gas standard containing HF (at 3.5 ppmv) was injected at constant flows (regulated by a mass flow controller) of 0.6 and 0.8 liters per minute over the sample gas. A spectroscopic tracer, SF_6 (at 2.12 ppmv), was also in the gas standard blend to provide a precise means by which to calculate dilution ratios. Appendix A describes in detail how the tracer is used. Tables 5-1 and 5-2 summarize the results, which show good recovery of the expected HF concentration under stack dilution, thereby validating the FTIR analysis method for this compound.

5.2.1 AFI Validation Spikes

Spiking with an HF certified gas standard was performed as stack gas was continuously extracted and analyzed at a central traverse point within the AFI stack. Section 3 explains the spiking procedure in more detail. A gas standard containing HF (at 3.5 ppmv) was injected at constant flows (regulated by a mass flow controller) of 1.0 and 0.4 liters per minute over the sample gas for the pre-spike and 1.0 and 0.6 liters per minute for the post-spike. A spectroscopic tracer, SF_6 (at 2.12 ppmv), was also in the gas standard blend to provide a precise means by which to calculate dilution ratios. Appendix A describes in detail how the tracer is used. Tables 5-3 and 5-4 summarize the results, which show good recovery of the expected HF concentration under stack dilution, thereby validating the FTIR analysis method for this compound.

Table 5-3. HF Spiking Parameters and Results at AFI – Pre-test

SF_6 concentration in gas cylinder (ppm), C_{undil}:	2.12
SF_6 concentration returned by method after analyte injection (ppm), C_{dil}:	1st spike: 1.05 2nd spike: 0.46
HF certified concentration in gas cylinder (ppm), T_{cert}:	3.5
Average Native HF concentration in stack (ppm), T_{native}:	1.41
Expected HF concentration after analyte injection (ppm), T_{exp}:	1st spike: 2.42 2nd spike: 1.83
Measured HF concentration (ppm):	1st spike: 2.03 2nd spike: 1.76
% Recovery	1st spike: 84% 2nd spike: 96%

Table 5-4. HF Spiking Parameters and Results at AFI – Post-test

SF_6 concentration in gas cylinder (ppm), C_{undil}:	2.12
SF_6 concentration returned by method after analyte injection (ppm), C_{dil}:	1st spike: 1.18 2nd spike: 0.69
HF certified concentration in gas cylinder (ppm), T_{cert}:	3.5
Average Native HF concentration in stack (ppm), T_{native}:	1.48
Expected HF concentration after analyte injection (ppm), T_{exp}:	1st spike: 2.58 2nd spike: 2.11
Measured HF concentration (ppm):	1st spike: 2.73 2nd spike: 2.77
% Recovery	1st spike: 106% 2nd spike: 131%

Table 5-5. HF Spiking Parameters and Results at MultiPhos – Pre-test

SF_6 concentration in gas cylinder (ppm), C_{undil}:	2.12
SF_6 concentration returned by method after analyte injection (ppm), C_{dil}:	1st spike: 0.73 2nd spike: 0.56
HF certified concentration in gas cylinder (ppm), T_{cert}:	3.5
Average Native HF concentration in stack (ppm), T_{native}:	0.109
Expected HF concentration after analyte injection (ppm), T_{exp}:	1st spike: 1.24 2nd spike: 0.98
Measured HF concentration (ppm):	1st spike: 1.00 2nd spike: 0.36
% Recovery	1st spike: 81% 2nd spike: 37%

Table 5-6. HF Spiking Parameters and Results at MultiPhos – Post-test

SF_6 concentration in gas cylinder (ppm), C_{undil}:	2.12
SF_6 concentration returned by method after analyte injection (ppm), C_{dil}:	1st spike: 1.86 2nd spike: 1.63
HF certified concentration in gas cylinder (ppm), T_{cert}:	3.5
Average Native HF concentration in stack (ppm), T_{native}:	0.358
Expected HF concentration after analyte injection (ppm), T_{exp}:	1st spike: 3.07 2nd spike: 2.74
Measured HF concentration (ppm):	1st spike: 3.08 2nd spike: 2.78
% Recovery	1st spike: 100% 2nd spike: 102%

5.4 Clarifier Emissions Test

Extractive FTIR monitoring of HF at the Clarifier was conducted on 21 May 2002. The emission profile is plotted in Figure 5-5. Noted on the graphs are when stack sampling began and ended, along with the times when the probe was out of the stack. Upon averaging the concentrations reported in Figure 5-5, and factoring in the average stack flow rate reported in Table 3-1, an average HF mass emission rate of only 0.002 lb/hr can be calculated.

HF spiking, per Method 320, was conducted prior to and immediately after this emissions test. The results are summarized in the following section.

6.0 Method 301 Validation at CF Industries' B-PAP Stack

The analyte spiking procedure outlined in the EPA document *Method 301-Field Validation of Pollutant Measurement Methods from Various Waste Media* was carried out at CF Industries' B-PAP on 18 April, 2002. Method 301 requires 24 test runs, 12 spiked and 12 unspiked, so this validation was completed upon the 12th paired HF spiking run when it was observed that the sampling trial met the necessary statistical allowances. The EPA method also calls for spiking as close as possible to the point of stack gas extraction, so the spiking "tee" was located in the primary position, as discussed in Section 2 and shown in Figure 2-1. A gas standard containing HF (at 3.5 ppmv) was injected at constant flows (regulated by a mass flow controller) on the order of 0.6 to 0.8 liters per minute over the sample gas. A spectroscopic tracer, SF_6 (at 2.12 ppmv), was also in the gas standard blend to provide a precise means by which to calculate dilution ratios. Appendix A describes in detail how the tracer is used.

Measurements consisted of comparing complete runs of unspiked and spiked analysis samples. The first run consisted of continuously monitoring the unspiked sample stream for a several-minute period, followed by a second run to continuously monitor the spiked sample stream for several minutes. The volume of spiked gas was limited to approximately 20-30% of the total sample gas volume.

The mean and standard deviation of the spiked members of the 12 pairs are dependent on the means and standard deviations of the analyte native to the stack exhaust and of the analyte spike itself. Since only one measurement system was used for this test, the variability in the concentrations within the stack background was combined with any variability from the instrument and cannot be separated. Table 6-1 shows the FTIR validation results for HF at B-PAP. Fortunately, the combined variances were not considered statistically significant. The validation statistics met all the allowable criteria for precision and accuracy without the need of a correction factor. A small negative bias was observed (possibly due to residual HF line effects after repeated spikes in a short time period), but was found to be statistically insignificant according to the t-statistic. Therefore, the FTIR system was deemed to be an acceptable field analysis tool in reporting HF emissions. Table 6-2 shows the raw data collected during the Method 301 validation, in comparison with the expected HF spiking concentrations based on the tracer.

Appendix A
Spiking/Validation Procedures

Accuracy tests for critical target compounds (like HF) were performed against the FTIR analysis method to assure the validity of the test data. This was accomplished by injecting measured volumes of certified gas into the extraction line as the FTIR system was drawing stack gas through its cell. A chemically inert compound known to possess a broad spectroscopic absorption pattern over a large range of concentrations (SF_6) was included in the gas mixture to calibrate dilution ratios. The linear behavior of the SF_6 concentrations returned by the analysis method provides a precise measure of the dilution factor associated with each analyte injection. The procedure used in calculating the concentrations expected during analyte spiking was as follows:

- The gas standard was introduced directly into the heated sample cell while bypassing the extraction line assembly. After the cell was sufficiently purged with the standard containing SF_6, the analysis method would return a value (called C_{undil}) which represents the concentration of SF_6 in the gas cylinder, as measured by the FTIR.
- The gas standard would then be injected at the sampling point of the extraction line as stack gas is drawn through the heated lines and sample cell. The analyte injection flow would be maintained at a low, constant rate with the aid of a mass flow controller. After the cell was sufficiently purged with the gas standard/stack gas mix, the analysis method would return a value (called C_{dil}) which represents the concentration of SF_6 diluted by stack gas, as measured by the FTIR.
- The expected concentration of the target compound, T_{exp} (which was also diluted by stack gas under the same proportions as SF_6), during analyte injection is thus:

$$T_{\exp} = \frac{C_{dil}}{C_{undil}} \times T_{cert} + \left(1 - \frac{C_{dil}}{C_{undil}}\right) \times T_{native}$$

Where:

T_{cert} is the certified concentration of the target compound in the gas cylinder; and

T_{native} is the average concentration of target compound present in the stack.

Extractive FTIR Testing
of
Hydrogen Fluoride Emissions

No. 1 DAP Plant

IMC PHOSPHATES MP, INC.
New Wales, Florida

Test Dates: April 10, 2002
Report Date: May 16, 2002

Koogler & Associates Environmental Services
4014 N.W. 13th Street
Gainesville, Florida 32609
352-377-5822

124-02-01

Revised 07/24/02

KOOGLER & ASSOCIATES
ENVIRONMENTAL SERVICES

I certify that this document and all attachments were prepared under my direction or supervision according to a system designed to assure that qualified personnel properly gather and evaluate the information submitted. To the best of my knowledge, all applicable analytical procedures comply with the Florida Department of Environmental Protection requirements and all test data and plant operating data are true and correct.

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

5/16/02 , Rev 7/24/02

Date

State of Florida Registration No. 12925

Max Lee, Ph.D., P.E.

5/16/02 Rev 7/24/02

Date

State of Florida Registration No. 58091

KOOGLER & ASSOCIATES
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1.0 INTRODUCTION

IMC Phosphates MP, Inc. (IMC) operates a phosphate fertilizer chemical complex located at 3095 Highway 640, Mulberry, Polk County, Florida. The facility operates under Title V Air Operating Permit No. 1050059-014-AV. At this facility, phosphate rock is processed into various fertilizer and animal feed supplements. The fertilizer complex includes the No. 1 Di-ammonium Phosphate (DAP) fertilizer plant (Emission Unit 009).

Koogler & Associates conducted hydrogen fluoride (HF) emission measurements on the No. 1 DAP plant on April 10, 2002 in accordance with EPA Method 320 as described in 40 CFR 63, Appendix A. The purpose of testing was to determine HF emissions from the DAP Plant No. 1 (EU-009) to assist IMC in the determination of facility HAP emission status required by Maximum Achievable Control Technology (MACT) standards (40 CFR 63, subparts AA and BB).

Measurements of HF emissions were made using an extractive sampling system with a Fourier Transform Infrared (FTIR) analyzer. Dynamic spiking in accordance with EPA Method 320 was performed following each run of the three test runs to ensure valid emissions data.

It was observed during testing of the No. 1 DAP Plant that the temperature of the extractive sampling system and free ammonia affected the measured HF concentrations in the stack gas and HF spikes. A similar observation was made during independent HF testing by EPA Method 320 at similar fertilizer facilities* and in the K&A Laboratory. As a result, dynamic spiking could not be performed within acceptable limits (± 30 percent) as will be discussed later in the report.

Table 1 provides a chronology of testing at the facility on April 10, 2002, and Table 2 summarizes plant conditions during testing.

Table 1. Testing Schedule

Date	Testing Activity
04/10/2002	- Setup/calibration of FTIR system at DAP Plant No. 1 - Sample on No. 1 Dap Plant - sample at 180°F (unacceptable spike recovery)

Table 2. Plant Operation Conditions

DAP Plant - No. 1 (EU-009)	
Date:	April 10, 2002
Stack Flow (DSCFM)	127,818
Stack Temperature (°F)	171
Production Rate (P_2O_5)	131 tons per hour
Fuel	Natural Gas
Dryer Venturi Scrubber ΔP (in. H_20)	20.0
Dryer Venturi Scrubber Flow Rate (GPM)	1513
RGC Scrubber ΔP (in. H_20)	24.0
RGC Scrubber Flow Rate (GPM)	1839
Cyclonic Scrubber ΔP (in. H_20)	11.2
Cyclonic Scrubber Flow Rate (GPM)	955
Equipment Scrubber ΔP (in. H_20)	21.7
Equipment Scrubber Flow Rate (GPM)	581

- *Koogler & Assoc. "Extractive FTIR Testing of Hydrogen Fluoride Emissions at DAP and Phosphoric Acid Production Units," Farmland-Hydro, LLP, April, 2002*
- *URS Corporation, Extractive FTIR Testing of the X-DAP, Y-MAP and B-PAP Stack Emissions," CF Industries, Inc., Plant City, Florida, January, 2002*

2.0 PROCESS DESCRIPTION

DAP Plant No. 1 (EU-009) produces a maximum input rate of 105 tons per hour of ammonium phosphates (DAP or granular MAP). Emissions from the reactor/granulator and dryer are controlled by a prescrubber, two (2) Venturi scrubbers, one (1) tailgas scrubber, and two (2) cyclonic wet scrubbers in series. Emissions from the product cooler are controlled by a baghouse. The dryer can be fired with natural gas, No. 6 fuel oil, or better grade fuel oil at a maximum heat input rate of 27.7 mmBTU per hour. During the test period, the dryer was fired with natural gas.

3.0 SAMPLING POINT LOCATIONS

Two sample ports are located in the 84-inch diameter stack of the reactor/granulator and dryer system. The two ports are at 90 degrees to one another and are located 7.0 diameters above (downstream) the point where the gases enter the stack and 3.18 diameters below the top of the stack. Flow measurements were performed in accordance with EPA Methods 1 and 2 (40 CFR 60, Appendix A). Figure 1 provides an elevation view of the DAP Plant stack.

4.0 EXTRACTIVE FTIR TESTING (EPA Method 320)

Chemical absorption of infrared light (IR) can be used as an analytical method to determine the concentration of IR-absorbing gases. Accordingly, EPA Method 320 (40 CFR 63, Appendix A), promulgated by EPA (June 14, 1999), is referred to as the Fourier Transform Infrared Radiation (FTIR) Method. A computerized analytical program is used to analyze IR absorption spectrum and determine the concentrations of IR-absorbing gases. The method is self-validating through a quality assurance (QA) spike procedure.

QA Spiking Procedures

QA spiking involves injecting a known concentration of an analyte (HF for these tests) into the extracted stack gas sample at approximately a 1:10 volumetric ratio. Based on the average amount of analyte measured in the stack gas alone, and the fraction of analyte spike injected into the sampled stack gas, an expected concentration of analyte in the QA-spiked stack gas stream can be calculated. A comparison of the calculated (i.e. expected) and the observed analyte concentration in the QA spiked stack gas is used to evaluate sampling analyte recovery and ensure data quality. The QA spiking is valid of the comparison of expected and observed concentration do not vary by more than ± 30 percent.

QA spiking was performed following each of three runs within a test. The spike recovery during the DAP Plant test did not meet Method criterium as will be discussed later.

The amount of spike gas was determined by mass flow measurements. The spike gas and stack sample gas were extracted through the FTIR and midget impingers (to remove gas moisture) followed by a critical orifice and vacuum pump. The critical orifice controlled the flow at 2.4 standard liters per minute. Figure 2 shows the extractive sampling system. Direct FTIR calibration of HF was performed before and after all testing by injecting a known concentration of HF directly into the FTIR analyzer. The average HF concentration from calibrations and the dilution of spike gas was used to determine the concentration of HF spike gas during QA spiking.

Testing Sequence

The following sequence of spectra collection describe the general procedure of FTIR spectra/gas measurements used to complete a Method 320 test. See the following sections for description of terminology:

PRE TEST

1. Background
2. Baseline (cylinder N2) (i.e. zero check)
3. Direct measure of cylinder HF
4. CTS (ethylene) (i.e. span check)

TEST

5. Spectra of stack gas collected
6. QA spike

RUN 2

7. Spectra of stack gas collected
8. QA spike

Run 3

9. Spectra of stack gas collected
10. QA spike

POST TEST

11. CTS
12. Direct measure of cylinder HF

FTIR Analyzer and Sampling System Setup and Checks

A Gasmet DX4000 (TEMET Instruments Oy, Helsinki, Finland) FTIR analyzer with Calcmet (TEMET Instruments Oy) analytical software was used to determine gas concentrations. FTIR instrument specifications are attached (Attachment 1). The FTIR analyzer includes a fixed 9.8-meter pathlength goldplated cell maintained at 200°C with IR detection provided by an MCT detector. Cell pressure and temperature are continually monitored at the cell and compensated by the Calcmet analytical software. The wavenumber resolution was set to 8 cm^{-1}. In comparison to FTIR analysis performed at higher resolutions (e.g. 0.5 cm^{-1}), "low" resolution spectra generally provide higher Signal-to-Noise Ratios (SNR) and greater signal stability which is more suitable for testing at the stack platform.

Due to the extremely reactive nature of HF, the sampling system was designed to minimize gas–surface contact prior to FTIR analysis. The entire sampling system and FTIR analyzer were placed on the sampling platform with gas contact limited to the eight-foot-PFA-lined probe, a QA spiking manifold, a 47-mm diameter 1-µm pore filter, and a 3-foot sampling line prior to entering the FTIR cell. All gas-wetted lines are made of PFA-teflon. Figure 2 provides a diagram of the sampling setup.

The probe and sampling system are typically maintained at a constant temperature of 350°F. However, as stated previously and described in the Summary of Results, it was found during field measurements and subsequent laboratory measurements that sampling at a temperature above 150°F-200°F resulted in levels of HF that were near the level of total fluorides measured by EPA Method 13B. Therefore, the FTIR gas sampling system was maintained approximately 20°F above the stack gas temperature; just enough temperature differential to prevent moisture condensation.

Following the FTIR, two chilled impingers were placed in-line to remove moisture, followed by a critical orifice and a vacuum pump. The critical orifice flow was 2.4 standard liters per minute. Orifice flow was checked before and after testing. Total sample flow (wet) was determined by combining the measured volumetric dry sample gas flow and the volumetric moisture fraction measured by the FTIR.

Leak Checks

A system leak check was performed before and after testing. After capping the probe inlet, a vacuum of more than 15 in. Hg was pulled on the system for two minutes and monitored by a pressure gauge. A change of less than 1.0 in. Hg over two minutes is required to ensure the system is not leaking.

Calibration Transfer Standard (CTS)

In addition to QA spiking to evaluate data quality, a Calibration Transfer Standard (CTS) gas was measured to ensure the accurate response of the FTIR. The CTS is a nonreactive gas that indicates whether the FTIR hardware has been damaged or misaligned during transportation and testing. Ethylene was chosen as the CTS gas and was measured at the beginning and end of testing. The initial and final readings of the CTS gas must indicate less than ± 5 percent variation from the mean value. HF emissions testing at the IMC facility was validated according to this criterium.

5.0 ANALYTICAL PROCEDURES

An analysis of an FTIR spectrum is performed by comparing reference spectra to the sampled gas spectrum. The reference spectra comprise a "library" of spectra that accurately characterize the gases within the sample gas. The computer program (Calcmet) linearly scales the library references using Classical Least Squares (CLS) fitting to determine the concentration of gases in the sample gas. It is critical that the library include all potential gases expected to be present in the sample gas. In essence, the analysis is only as good as the library. If gases are present in the sample that are not included in the library, analysis can provide false positive concentrations (i.e. erroneous data). The library errors can be detected but it may not be possible to account for the unknown chemicals in the field. Therefore, it is critical that a general knowledge of the sample gas chemical composition be known prior to testing.

Regarding the DAP Plant stack, the only gases expected to be present in the IR regions analyzed for HF are HF and H_20. To produce adequate detection limits, one-minute averaged spectra of sample gas were collected. The analytical areas were chosen by an interactive process based on minimizing interference by water and minimizing the residual noise (i.e. confidence interval). The following table provides the analytical parameters for the pollutant (HF) and interferants (H_20):

Compound	Optical Depth Range of References (ppm x m)**
H_2O	49,000 - 3,626,000
HF	31.4 - 245

**reference gas concentration (ppm) x cell path length (9.8 meters)

Signal-to-Noise-Ratio (SNR) Tests

The SNR test provides a measure of the FTIR analyzer noise (i.e. minimum detection limit, MDL) in the spectral regions analyzed. The MDL is determined by comparison of two spectra of a non-IR absorbing gas (e.g. ultra high purity nitrogen (UHP-N_2). The first spectra is called the "background" and the comparison of the background to a second spectra UHP-N_2 will provide a "baseline" spectra (i.e. a zero check). The baseline indicates the spectral noise within the IR regions analyzed. Based on the noise and the analyte reference spectrum, the lowest possible or minimum detection limit (MDL) that the FTIR hardware will allow can be determined. The results of SNR tests are provided below:

Stack	Analytical Region	RMS Noise (%)	SNR	HF (MDL)	DL 3 x MDL
DAP Plant	2550-2650	0.0038	26315		
	4020-4220	0.0128	7813	0.13 ppm	0.39 ppm

When interferences are taken into account, the lowest detection limit is expected to increase. The amount of DL increase, based on testing experience, is typically between 2-4 times the MDL. However, the analytical region to detect

HF can be set to minimize the interference of water. Therefore, for this testing of HF, the DL is conservatively set at three times the MDL.

6.0 RESULTS AND SUMMARY

Initial test data at a sample system temperature of 300°F indicated a concentration of HF greater than the expected concentration based on concurrent EPA Method 13B tests. Noting that HF testing at similar DAP plants* had indicated the artifact of high HF concentrations when extracting gas samples at temperatures significantly above stack temperature, the sample system temperature was reduced to a temperature of about 20°F above the stack gas temperature.

The data collected at these temperatures showed HF concentrations below the detection limit, however the spike recovery for method validation was not successful. Attempts to perform QA spiking and demonstrate HF spike recovery within the acceptable range (± 30 recovery) yielded no detectable spike recovery. As acceptable HF spike recoveries were demonstrated on phosphoric acid plant stacks and as the FTIR successfully calibrated with the direct injection of HF calibration gas before and after the DAP plant tests, it was suspected that the HF spike (and HF that may have been in the stack gas) reacted with excess ammonia in the gas stream. This phenomenon (the reaction of HF and NH_3) was subsequently demonstrated under laboratory conditions at the Koogler & Associates offices.

- *Koogler & Assoc. "Extractive FTIR Testing of Hydrogen Fluoride Emissions at DAP and Phosphoric Acid Production Units," Farmland-Hydro, LLP, April, 2002*
- *URS Corporation, Extractive FTIR Testing of the X-DAP, Y-MAP and B-PAP Stack Emissions," CF Industries, Inc., Plant City, Florida, January, 2002*

Laboratory testing demonstrated that under both wet gas (up to 20 percent moisture tested) and dry gas conditions, HF and NH_3 maintained their unique identities and could accurately be measured by the FTIR at sample system temperatures above 150-180°F. As the temperature of the sample system was decreased below 150-180°F (with excess NH_3), however, the HF concentration was observed to approach zero; indicating a temperature dependent reaction between NH_3 and HF.

It was further observed that as the sample system temperature was lowered to about 100°F and then increased to the 150-180°F range, there were HF and NH_3 spikes of greater concentrations than the concentrations of the gases original input to the sample system; indicating a breakdown of the HF/NH_3 compound(s) and the desorption of the compounds from the sample system.

Although the results of the testing on the DAP Plant stack have not been validated by QA spiking, it can be stated with confidence (based on the laboratory tests and field observations) that the HF concentration in the DAP plant stack gas was less than the MDL (0.13 ppm). This statement is based on the performance of the FTIR sampling system on phosphoric acid plants, the response of the FTIR to a direct HF spike and the above described laboratory tests indicating interaction(s) between HF and NH_3 (including the HF QA spike).

Summary

Measurements of HF at the No. 1 DAP stack were below the minimum detection limit of 0.13 ppm, which corresponds to a mass emission rate of 0.063 lb/hr or 0.28 tons per year (based on 8760 hours of operation per year). The measurements on the DAP plant were not validated according to EPA Method 320, however it has been demonstrated that HF (both in the stack gas and in the calibration gas spike) react with ammonia in the stack gas. This phenomenon (the reaction of HF and NH_3) has been demonstrated under laboratory conditions at the Koogler & Associates offices and observed in the field, and indicates that under conditions in the DAP plant stack, HF should not be present.

ATTACHMENT 1

FTIR MANUFACTURER SPECIFICATIONS

Temet Instruments Oy

GASMET Production Check List

Customer: AQA
GASMET SN: 02340
Model: DX4000

DSP Board SN: 45/ 01 168

☒ DSP program Checksum : 0047C660
☒ Altera program
☒ Resolution: 8
☒ Communication port speed: 57600 bps
☒ Default parameters
☒ Jumper
☒ Pressure sensor calibration 1023 mbar

Power Board SN: 45/01 168

☒ Operating voltages
Speed settings:
☒ 2 Hz
☒ 5 Hz
Control outputs:
☒ Pump ON/OFF OK
☒ Valve ON/OFF OK

Detector

☒ Type MCT SNR 1774 Case 108 version

Detector preamplifier

☒ Detector temperature -35.00 °C
☒ Det. heat exchanger temp 25.00 °C
☒ Gain: High

Enclosure

Groundings:
☒ Sample cell
☒ Electronics
Other:
☒ Fans
☒ Delay adjustment
☒ IR source voltage: 9.20 V
☒ Line voltage: 115V (fuse 4AT)
☒ Electronics temperature: 42.0 °C
☒ Gain setting: 4

Extractive FTIR Testing
of
Phosphoric Acid Train No. 3

IMC PHOSPHATES MP, INC.
New Wales, Florida

Test Dates: April 8-9, 2002
Report Date: May 16, 2002

Koogler & Associates Environmental Services
4014 N.W. 13th Street
Gainesville, Florida 32609
352-377-5822

124-02-01

Revised 07/24/02

KOOGLER & ASSOCIATES
ENVIRONMENTAL SERVICES

I certify that this document and all attachments were prepared under my direction or supervision according to a system designed to assure that qualified personnel properly gather and evaluate the information submitted. To the best of my knowledge, all applicable analytical procedures comply with the Florida Department of Environmental Protection requirements and all test data and plant operating data are true and correct.

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

5/16/02, Rev 7/24/02

Date

State of Florida Registration No. 12925

Max Lee, Ph.D., P.E.

5/16/02, Rev 7/24/02

Date

State of Florida Registration No. 58091

Table of Contents

1.0 INTRODUCTION

IMC Phosphates MP, Inc. operates a phosphate fertilizer chemical complex located at 3095 Highway 640, Mulberry, Polk County, Florida. The facility operates under Title V Air Operating Permit No. 1050059-014-AV. At this facility, phosphate rock is processed into various fertilizer and animal feed supplements. The fertilizer complex includes Phosphoric Acid Plant–No. 3 Train (Emission Unit 039).

Koogler & Associates conducted hydrogen fluoride (HF) emission measurements on the No. 3 Phosphoric Acid Plant on April 8-9, 2002 in accordance with EPA Method 320 as described in 40 CFR 63, Appendix A. The purpose of testing was to determine HF emissions from the No. 3 Phosphoric Acid Plant (EU-039) to assist IMC in the determination of facility HAP emission status required by Maximum Achievable Control Technology (MACT) standards (40 CFR 63, subparts AA and BB).

Measurements of HF emissions were made using an extractive sampling system with a Fourier Transform Infrared (FTIR) analyzer. Dynamic spiking in accordance with EPA Method 320 was performed following each run of the three test runs to ensure valid emissions data.

It was observed during testing that the temperature of the extractive sampling system affected the measured HF concentrations in the stack and the spikes. A similar observation was made during independent HF testing by EPA Method 320 at similar fertilizer facilities.* Therefore, extractive sampling was performed at temperatures slightly above stack temperatures. Dynamic spiking was performed within acceptable limits (± 30 percent). Table 1 provides a chronology of testing at the facility during April 8-9, 2002. Table 2 summarizes plant conditions during testing.

Table 1. Testing Schedule

Date	Testing Activity

04/08/2002	- Setup/calibration of FTIR system at Phosphoric Acid 3rd Train Stack
	- Test Phosphoric Acid 3rd Train
	  - sample at 250°F (excess HF)
	  - plant downtime

04/09/2002	- Test Phosphoric Acid 3rd Train - Three-run test on 3rd Train - sample at 110°F (acceptable spike recovery on three runs)

Table 2. Plant Operation Conditions

Phosphoric Acid - No. 3 Train (EU-039)	
Date:	April 9, 2002
Stack Flow (DSCFM)	17,626
Stack Temperature (°F)	102
Production Rate (P_2O_5)	2028 tons per day
Crossflow Scrubber Flow Rate (GPM)	863
Crossflow Scrubber ΔP (in. $H_2$0)	0.36

- *Koogler & Assoc. "Extractive FTIR Testing of Hydrogen Fluoride Emissions at DAP and Phosphoric Acid Production Units," Farmland-Hydro, LLP, April, 2002*
- *URS Corporation, Extractive FTIR Testing of the X-DAP, Y-MAP and B-PAP Stack Emissions," CF Industries, Inc., Plant City, Florida, January, 2002*

2.0 PROCESS DESCRIPTION

Phosphoric Acid Train No. 3 (EU-039) is designed to produce 2,400 tons per day of phosphoric acid. Fluoride emissions are controlled by a 25,000 ACFM crossflow packed scrubber (using pond water) followed by a cyclonic demister. Gases exhausted from the scrubbing system are vented to the atmosphere through a 52-inch diameter stack 110 feet high.

The plant consists of a digester where phosphate rock and sulfuric acid react to form a mixture of phosphoric acid and gypsum. This mixture is then filtered to separate the two products. Phosphoric acid is an intermediate product used in the production of various phosphate fertilizer products and/or animal feed supplements. Gypsum is a byproduct hydraulically transported to the phosphogypsum stack for storage.

3.0 SAMPLING POINT LOCATIONS

Two sample ports are located in the 52-inch diameter exhausting the scrubbing system of the phosphoric acid plant. The two ports are at 90 degrees to one another and are located 18.29 diameters above (downstream) the point where the gases enter the stack and 5.33 diameters below the top of the stack. Flow measurements were performed in accordance with EPA Methods 1 and 2 (40 CFR 60, Appendix A). Figure 1 provides an elevation view of the 3rd train stack.

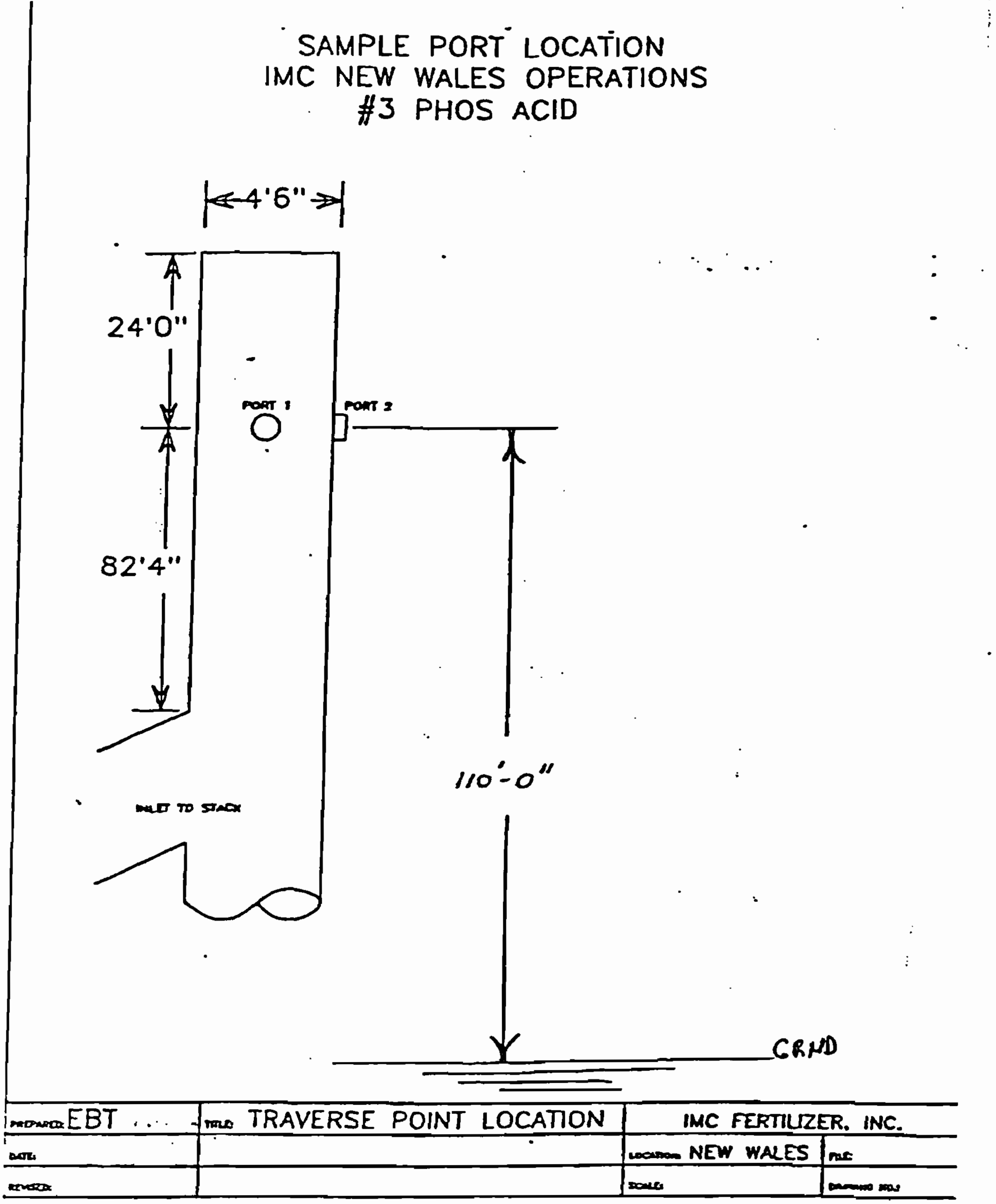

Figure 1. IMC, New Wales - Phosphoric Acid - Train No.3 stack

4.0 EXTRACTIVE FTIR TESTING (EPA Method 320)

Chemical absorption of infrared light (IR) can be used as an analytical method to determine the concentration of IR-absorbing gases. Accordingly, EPA Method 320 (40 CFR 63, Appendix A), promulgated by EPA (June 14, 1999), is referred to as the Fourier Transform Infrared Radiation (FTIR) Method. A computerized analytical program is used to analyze IR absorption spectrum and determine the concentrations of IR-absorbing gases. The method is self-validating through a quality assurance (QA) spike procedure.

QA Spiking Procedures

QA spiking involves injecting a known concentration of an analyte (HF for these tests) into the extracted stack gas sample at approximately a 1:10 volumetric ratio. Based on the average amount of analyte measured in the stack gas alone, and the fraction of analyte spike injected into the sampled stack gas, an expected concentration of analyte in the QA-spiked stack gas stream can be calculated. A comparison of the calculated (i.e. expected) and the observed analyte concentration in the QA spiked stack gas is used to evaluate sampling analyte recovery and ensure data quality. The QA spiking is valid of the comparison of expected and observed concentration do not vary by more than ± 30 percent.

QA spiking was performed following each of three runs within a test. HF emissions testing at the phosphoric acid plant stack was validated according to this criterium.

The amount of spike gas was determined by mass flow measurements. The spike gas and stack sample gas were extracted through the FTIR and midget impingers (to remove gas moisture) followed by a critical orifice and vacuum pump. The critical orifice controlled the flow at 2.4 standard liters per minute. Figure 2 shows the extractive sampling system. Direct FTIR calibration of HF was performed before and after all testing by injecting a known concentration of HF directly into the FTIR analyzer. The average HF concentration from calibrations and the dilution of spike gas was used to determine the concentration of HF spike gas during QA spiking.

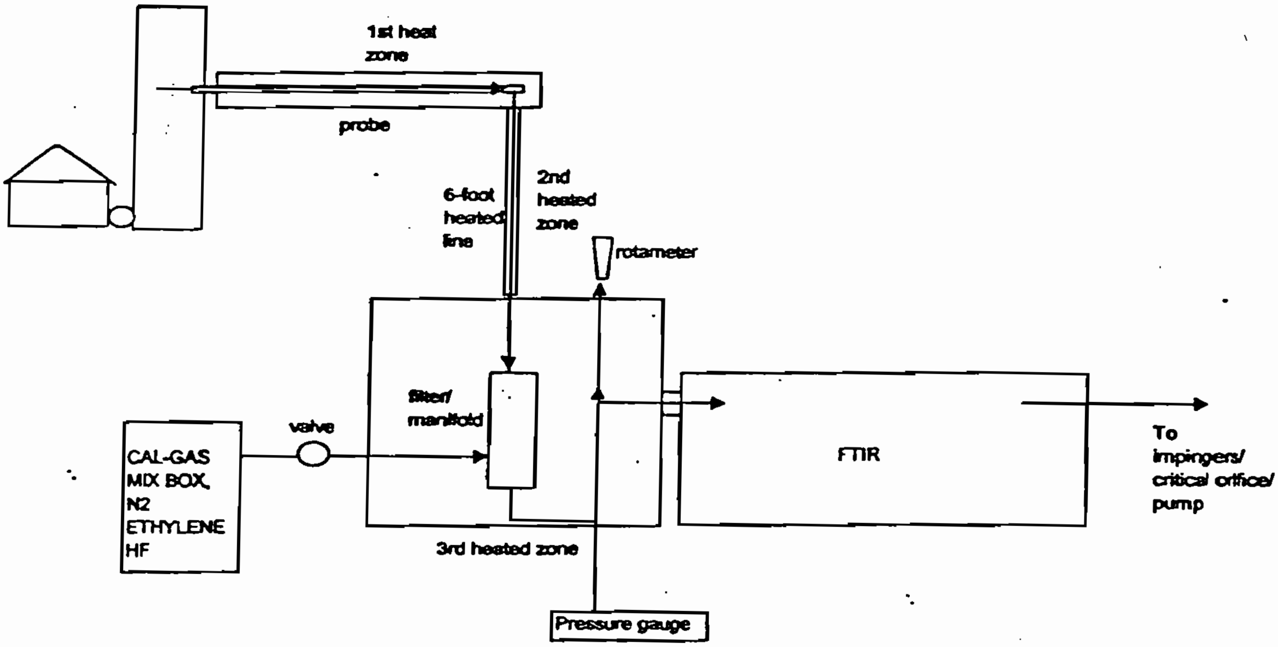

Figure 2. EPA Method 320 Sampling System

Testing Sequence

The following sequence of spectra collection describe the general procedure of FTIR spectra/gas measurements used to complete a Method 320 test. See the following sections for description of terminology:

PRE TEST

1. Background
2. Baseline (cylinder N2) (i.e. zero check)
3. Direct measure of cylinder HF
4. CTS (ethylene) (i.e. span check)

TEST

5. Spectra of stack gas collected
6. QA spike

RUN 2

7. Spectra of stack gas collected
8. QA spike

Run 3

9.. Spectra of stack gas collected
10. QA spike

POST TEST

11. CTS
12. Direct measure of cylinder HF

FTIR Analyzer and Sampling System Setup and Checks

A Gasmet DX4000 (TEMET Instruments OY, Helsinki, Finland) FTIR analyzer with Calcmet (TEMET Instruments OY) analytical software was used to determine gas concentrations. FTIR instrument specifications are attached (Attachment 1). The FTIR analyzer includes a fixed 9.8-meter pathlength goldplated cell maintained at 200°C with IR detection provided by an MCT detector. Cell pressure and temperature are continually monitored at the cell and compensated by the Calcmet analytical software. The wavenumber resolution was set to 8 cm^{-1}. In comparison to FTIR analysis performed at higher resolutions (e.g. 0.5 cm^{-1}), "low" resolution spectra generally provide higher Signal-to-Noise Ratios (SNR) and greater signal stability which is more suitable for testing at the stack platform.

Due to the extremely reactive nature of HF, the sampling system was designed to minimize gas–surface contact prior to FTIR analysis. The entire sampling system and FTIR analyzer were placed on the sampling platform with gas contact limited to the eight-foot-PFA-lined probe, a QA spiking manifold, a 47-mm diameter 1-μm pore filter, and a 3-foot sampling line prior to entering the FTIR cell. All gas-wetted lines are made of PFA-teflon. Figure 2 provides a diagram of the sampling setup.

The probe and sampling system are typically maintained at a constant temperature of 350°F. However, as stated previously and described in the Summary of Results, it was found during field measurements and subsequent laboratory measurements that sampling at a temperature above about 150°F resulted in high levels of HF that were near the level of total fluorides measured by EPA Method 13B. Therefore, an attempt was made to maintain the FTIR gas sampling system approximately 20°F above the stack gas temperature; just enough temperature differential to prevent moisture condensation. QA spiking with HF demonstrate that scrubbing of HF by condensed moisture in the sampling line at temperatures slightly above stack temperature did not occur.

Following the FTIR, two chilled impingers were placed in-line to remove moisture, followed by a critical orifice and a vacuum pump. The critical orifice flow was 2.4 standard liters per minute. Orifice flow was checked before and after testing. Total sample flow (wet) was determined by combining the measured volumetric dry sample gas flow and the volumetric moisture fraction measured by the FTIR.

Leak Checks

A system leak check was performed before and after testing. After capping the probe inlet, a vacuum of more than 15 in. Hg was pulled on the system for two minutes and monitored by a pressure gauge. A change of less than 1.0 in. Hg over two minutes is required to ensure the system is not leaking.

Calibration Transfer Standard (CTS)

In addition to QA spiking to evaluate data quality, a Calibration Transfer Standard (CTS) gas was measured to ensure the accurate response of the FTIR. The CTS is a nonreactive gas that indicates whether the FTIR hardware has been damaged or misaligned during transportation and testing. Ethylene was chosen as the CTS gas and was measured at the beginning and end of testing. The initial and final readings of the CTS gas must indicate less than ± 5 percent variation from the mean value. HF emissions testing at the IMC facility was validated according to this criterium.

5.0 ANALYTICAL PROCEDURES

An analysis of an FTIR spectrum is performed by comparing reference spectra to the sampled gas spectrum. The reference spectra comprise a "library" of spectra that accurately characterize the gases within the sample gas. The computer program (Calcmet) linearly scales the library references using Classical Least Squares (CLS) fitting to determine the concentration of gases in the sample gas. It is critical that the library include all potential gases expected to be present in the sample gas. In essence, the analysis is only as good as the library. If gases are present in the sample that are not included in the library, analysis can provide false positive concentrations (i.e. erroneous data). The library errors can be detected but it may not be possible to account for the unknown chemicals in the field. Therefore, it is critical that a general knowledge of the sample gas chemical composition be known prior to testing.

Regarding the phosphoric acid plant stack, the only gases expected to be present in the IR regions analyzed for HF are HF and H_20. To produce adequate detection limits, one-minute averaged spectra of sample gas were collected. The analytical areas were chosen by an interactive process based on minimizing interference by water and minimizing the residual noise (i.e. confidence interval). The following table provides the analytical parameters for the pollutant (HF) and interferants (H_20):

Compound	Optical Depth Range of References (ppm x m)**
H_20	49,000 - 3,626,000
HF	31.4 - 245

**reference gas concentration (ppm) x cell path length (9.8 meters)

Signal-to-Noise-Ratio (SNR) Tests

The SNR test provides a measure of the FTIR analyzer noise (i.e. minimum detection limit, MDL) in the spectral regions analyzed. The MDL is determined by comparison of two spectra of a non-IR absorbing gas (e.g. ultra high purity nitrogen (UHP-N_2). The first spectra is called the "background" and the comparison of the background to a second spectra UHP-N_2 will provide a "baseline" spectra (i.e. a zero check). The baseline indicates the spectral noise within the IR regions analyzed. Based on the noise and the analyte reference spectrum, the lowest possible or minimum detection limit (MDL) that the FTIR hardware will allow can be determined. The results of SNR tests are provided below:

Stack	Analytical Region	RMS Noise (%)	SNR	HF (MDL)	DL 3 x MDL
Phos Acid	2550-2650	0.0038	26315		
	4020-4220	0.0144	0.0144	0.14 ppm	0.42 ppm

When interferences are taken into account, the lowest detection limit is expected to increase. The amount of DL increase, based on testing experience, is typically between 2-4 times the MDL. However, the analytical region to detect

HF can be set to minimize the interference of water. Therefore, for this testing of HF, the DL is conservatively set at 3 times the MDL.

6.0 RESULTS AND SUMMARY

HF concentrations measured in the Phosphoric Acid Plant No. 3 Train stack gas on April 9, 2002 were above the detection limit of 0.42 ppm but are questionable because of fluoride compound sensitivity to sample system temperature and fluoride compound desorption from the sample system. Sampling conducted on April 8, 2002 was conducted at the Method recommended sample system temperature of 350°F. At this system temperature, the apparent HF concentration was higher than expected based on total fluoride concentrations measured by EPA Method 13B.

Subsequent laboratory testing with the FTIR and sampling system, and a mix of HF calibration gas and water vapor (up to 18 percent, v/v), demonstrated that HF is stable at temperatures from 350°F down to less than 150°F. However, during the tests on the No. 3 Phosphoric Acid Train, it was observed that as the sample system temperature was decreased (from 350°F to 150°F), the apparent HF concentration decreased significantly. This indicated that the HF observed at the higher sample system temperatures (350°F) is most likely from the decomposition of some fluoride compound present in the stack gas at stack gas temperature; yielding HF as an artifact.

In addition to the question of the temperature stability of fluoride compounds, fluoride sorption was encountered in the sample system making it difficult to differentiate between HF in the stack gas and HF that may have subsequently bled off the sampling system. It is suspected that the sorbed compounds were primarily artifacts (including HF) created at temperatures between 150°F and 350°F.

The QA spike recovery was successful for all three runs, averaging 89 percent of the expected recovery; however this recovery could be influenced by the bleed-off of sorbed HF as just discussed.

Summary

HF concentration in the Phosphoric Acid Plant–No. 3 Train stack gas were measured by FTIR to be 2.66 ppm (v/v, wet gas). This concentration results in a mass emission rate of 0.67 lb/hr. This concentration and emission rate are questionable, however, because of the observed fluoride compound decomposition (as a function of sample system temperature) and fluoride compound sorption and bleed-off in the sample system as discussed in the preceding section.

Table 3. Summary of Emissions – Phosphoric Acid Plant Train No. 3

Test Date: 4/9/2002

Test Run	Stack Gas Flow		Hydrogen Fluoride Emissions		
			Stack Gas Conc.	Emission Rate	Emission Rate**
	scfm, wet	dscfm*	(ppm)	(lb/hr)	(ton/yr)
1	18455	17626	2.06	0.119	0.52
2	18493	17626	2.85	0.165	0.72
3	18402	17626	3.06	0.175	0.77
Avg.	18450	17626	2.66	0.153	0.67

** Flow (scfmd) based on average of flow measurements made during total Fluoride testing, see K&A report. Individual flow values = 18,186 and 17,066 dscfm.*

*** based on 8760 hour/yr operation*

Emission Rate (lb/hr):

HF = (stack gas flow scfm, wet) (60 min/hr) (10-6) (conc. - ppm) (20/385 lb/ft^3)

Emission Rate (ton/yr)= Emission Rate (lb/hr) (8760 hr/yr) / (2000 ton/lb)

QA Spike • Run	Observed Average Spike	Expected Average Spike	Recovery Fraction
	ppm	ppm	percent
1	2.40	2.80	85.5
2	3.19	3.52	90.7
3	3.37	3.69	91.5

** Bias of Concentration must be + or - 30% of 100% recovery*

ATTACHMENT 1

FTIR MANUFACTURER SPECIFICATIONS

Sample Cell

☒ Type: 1L
☒ Path length: 9,8m
☒ Window material: BaF2
☒ Heating element power: 200 W (115/230VAC)
100 W (12 VDC)
☒ Temperature: 200 °C

Interferometer

☒ Beamsplitter material: ZnSe
☒ Window material: ZnSe
☒ Laser signal amplitude: 14,40 Vpp
☒ Interferogram height: 13,0 V
☒ Interferogram center pos. 2387

☒ Parameter values: A52 O63 L41 F3 T200 S5 G4 X134 K40 S/N340 N980 Y1 !8 %2Huom! Laserputken poikkeava läpimitta 24,2mm

Vibration test

Date: 31.01.2002 Tested by : _____

Interferometer temperature : 40,1°C
Source Intensity : 51,21
Duration : 10 min

	Initial	Final
Gas cell temperature (°C)	199,2	199,50
Interferogram height (V)	3,61	3,60
Interferogram center	2380	2364
Maximum tilt at 4000 cm^{-1} (A.U.)	0	0,0023
Maximum offset (A.U.)	0	0,0009

Temperature stability test

Date: 31.01.2002 Tested by : _____

Duration: 1455 – 1655 – 1900 – 0900

	Initial	Phase 1	Phase 2	Final
Ambient temperature (°C)	19,2	19,4		17,90
Climate chamber temperature (°C)	20,8	39,1		20,10
Interferometer temperature (°C)	41,0	62,1	42,00	41,80
Interferogram center	2367	2155		2336
Interferogram height (V)	3,53	3,42		3,57
Source Intensity	51,21	51,24		3,57
Maximum tilt at 4200 cm^{-1} (A.U.)	0	0,0145	0,0036	-0,0048
Baseline @ 2500 cm^{-1}	0	0,0063	0,0009	-0,0013
Baseline @ 1200cm^{-1}	0	0,0148	0,0129	-0,0020

Maximum offset (A.U.)	0	0,0387	0,0050	-0,0139

Calibration data

Software version: 2,6

Sample meas. time (s)	rms-noise (A.U.)			
	900-1100 cm^{-1}	2000-2200 cm^{-1}	2900-3100 cm^{-1}	4000-4200 cm^{-1}
1	0,0017466000	0,0003117000	0,0004064000	0,0013220000
5	0,0007471000	0,0001195000	0,0001597000	0,0006593000
20	0,0003162000	0,0000732000	0,0000544000	0,0003313000
60	0,0003510000	0,0000445000	0,0000467000	0,0002191000
180	0,0001893000	0,0000441000	0,0000404000	0,0001097000

Ambient pressure : 1017mbar
100 ppm N_2O peak absorbance at 1304 cm^{-1} : 0,1119A.U.

Hardware status:

	Value
Source Intensity	51,00
Interferogram Height (V)	3,48
Interferogram center	2339
Interferogram Temperature °C	42,00
Sample Cell Temperature °C	199,00

☒ Bkg measured & saved
☒ 100% line measured & saved

Notes

Test spectra have been measured.

Date: 2002-02-01 ***Checked by:*** TT

SEVERN TRENT STL

ANALYTICAL REPORT

Job Number: 660-1947.1

Job Description: SP West Cooling Pond

For:

Mosaic Phosphates
P.O. Box 2000
Mulberry, FL 33860

Attention: Mr. Dave Turley

Tina Fritz
Project Manager II
tfritz@stl-inc.com
06/07/2005

Methods: FDEP, DOH Certification #: E84282 These test results meet all the requirements of NELAC. All questions regarding this test report should be directed to the STL Project Manager who signed this test report. The estimated uncertainty associated with these reported results is available upon request.

Severn Trent Laboratories, Inc.
STL Tampa 6712 Benjamin Road, Suite 100, Tampa, FL 33634
Tel 813-8857427 Fax 813-8857049 www.stl-inc.com

METHOD SUMMARY

Client: Mosaic Phosphates

Job Number: 660-1947.1

Description	Method	Preparation Method
Matrix: Water		
ICP Metals by 200.7 CWA	40CFR136A 200.7 Appx C	
Sample Filtration		FILTRATION
ICP Metals by 200.7	EPA 200.7 Rev 4.4	
Total Recoverable Metals Digestion for 200.7		40CFR136A 200.7 Appx C
Field Sampling	EPA Field Sampling	
Chloride (Colorimetric, Automated Ferricyanide, AAI	MCAWW 325.2	
Nitrogen (Ammonia, Colorimetric, Automated Phenate)	MCAWW 350.1	
Phosphorus, All Forms, Colorimetric, Single Reagent	EPA 365.2	
Sample Digestion for Total Phosphorous		MCAWW 365.2/365.3
Sulfate (Turbidimetric)	MCAWW 375.4	
Fluoride (Ion-selective Electrode)	SM18 4500-F_C	

REFERENCES

40CFR136A - "Methods for Organic Chemical Analysis of Municipal Industrial Wastewater", 40CFR, Part 136, Appendix A, October 26, 1984 and subsequent revisions.

EPA - US Environmental Protection Agency

MCAWW - "Methods For Chemical Analysis Of Water And Wastes", EPA-600/4-79-020, March 1983 And Subsequent Revisions.

SM18 - "Standard Methods For The Examination Of Water And Wastewater", 18th Edition, 1992.

SAMPLE SUMMARY

Client: Mosaic Phosphates

Job Number: 660-1947.1

Lab Sample ID	Client Sample ID	Client Matrix	Date/Time Sampled	Date/Time Received
660-1947-1	WEST POND	Water	05/16/2005 1440	05/17/2005 0801

Mr. Dave Turley
Mosaic Phosphates
P.O. Box 2000
Mulberry, FL 33860

Job Number: 660-1947.1
Lab Sample Id: 660-1947-1
Date Sampled: 05/16/2005 1440
Date Received: 05/17/2005 0801

Client Sample ID: WEST POND

	Result/Qualifier	Unit	RL	Method	Date Prepared	Date Analyzed	Dilution
METALS							
SiO2, Silica	6100000	ug/L	50000	200.7 Appx C-	05/20/2005 1102	05/23/2005 1503	500
Aluminum	140	mg/L	0.70	200.7 Rev 4.4	05/20/2005 1634	05/23/2005 1554	10
Calcium	1800	mg/L	0.85	200.7 Rev 4.4	05/20/2005 1634	05/23/2005 1554	10
Iron	180	mg/L	0.22	200.7 Rev 4.4	05/20/2005 1634	05/23/2005 1554	10
Potassium	230	mg/L	1.9	200.7 Rev 4.4	05/20/2005 1634	05/23/2005 1554	10
Magnesium	260	mg/L	1.1	200.7 Rev 4.4	05/20/2005 1634	05/23/2005 1554	10
Sodium	1900	mg/L	3.1	200.7 Rev 4.4	05/20/2005 1634	05/23/2005 1554	10
GENERAL CHEMISTRY							
Chloride	110	mg/L	0.90	325.2		05/31/2005 2100	1.0
Ammonia	35	mg/L	0.040	350.1		05/18/2005 1303	1.0
Phosphorus, Total	8900	mg/L	170	365.2	05/27/2005 1105	05/27/2005 1105	10,000
Sulfate	3100	mg/L	170	375.4		05/23/2005 1100	100
Fluoride	18000	mg/L	440	4500-F_C		05/18/2005 1100	10,000
FIELD SERVICE / MOBILE LAB							
Field pH	1.76D	SU		Field Sampling		05/16/2005 1440	1.0
Field Temperature	33.6D	Degrees		Field Sampling		05/16/2005 1440	1.0
Specific Conductance	35400D	umhos/cm		Field Sampling		05/16/2005 1440	1.0
Oxygen, Dissolved	1.91D	mg/L		Field Sampling		05/16/2005 1440	1.0
Oxidation Reduction Potential	351D	millivolts		Field Sampling		05/16/2005 1440	1.0
Turbidity	27.9D	NTU		Field Sampling		05/16/2005 1440	1.0

Serial Number 25606

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

SEVERN TRENT STL

STL Tampa
6712 Benjamin Road, Suite 100
Tampa, FL 33634

Website: www.stl-inc.com
Phone: (813) 885-7427
Fax: (813) 885-7049

○ Alternate Laboratory Name/Location
Phone:
Fax:

PROJECT REFERENCE Pond Sampling / South Pierce	PROJECT NO.	PROJECT LOCATION (STATE) Fl
SAMPLER'S SIGNATURE Barbara S Hartley	P.O. NUMBER	CONTRACT NO.
CLIENT (SITE) PM	CLIENT PHONE	CLIENT FAX
CLIENT NAME Mosaic	CLIENT E-MAIL	
CLIENT ADDRESS		
COMPANY CONTRACTING THIS WORK (if applicable)		

PAGE 1 OF 1

STANDARD REPORT DELIVERY ○
DATE DUE ______

EXPEDITED REPORT DELIVERY (SURCHARGE) ○
DATE DUE ______

NUMBER OF COOLERS SUBMITTED PER SHIPMENT:

SAMPLE DATE	SAMPLE TIME	SAMPLE IDENTIFICATION	COMPOSITE (C) OR GRAB (G) INDICATE	AQUEOUS (WATER)	SOLID OR SEMISOLID	AIR	NONAQUEOUS LIQUID (OIL, SOLVENT,...)	F, SO4, Cl	T.P., NH3	Silica (Gravimetric)	Al, Ca, Fe, K, Mg, Na							REMARKS
		PRESERVATIVE						Cool	H_2SO_4	Cool	HNO_3							
		NUMBER OF CONTAINERS SUBMITTED																
05/16/05	1440	West Pond	G	✓				1	1	1	1							pH 1.76 Temp 33.6
																		Cnd. 35400
																		Turb. 27.9
																		Redox 351
																		D.O. 1.91

RELINQUISHED BY: (SIGNATURE)	DATE 5/12/05	TIME 1410	RELINQUISHED BY: (SIGNATURE) Barbara S Hartley	DATE 05/16/05	TIME 1504	RELINQUISHED BY: (SIGNATURE)	DATE 5/17/05	TIME 0645
RECEIVED BY: (SIGNATURE) Barbara S Hartley	DATE 05/12/05	TIME 1630	RECEIVED BY: (SIGNATURE)	DATE 5/16/05	TIME 1504	RECEIVED BY: (SIGNATURE)	DATE	TIME

LABORATORY USE ONLY

RECEIVED FOR LABORATORY BY: (SIGNATURE)	DATE	TIME	CUSTODY INTACT YES ○ NO ○	CUSTODY SEAL NO.	STL TAMPA LOG NO.	LABORATORY REMARKS

STL8240-660 (12/02)

SEVERN TRENT STL

ANALYTICAL REPORT

Job Number: 660-1919.1

Job Description: SP WS3 Cooling Pond

For:

Mosaic Phosphates
P.O. Box 2000
Mulberry, FL 33860

Attention: Mr. Dave Turley

Tina Fritz
Project Manager II
tfritz@stl-inc.com
06/02/2005

Methods: FDEP, DOH Certification #: E84282 These test results meet all the requirements of NELAC. All questions regarding this test report should be directed to the STL Project Manager who signed this test report. The estimated uncertainty associated with these reported results is available upon request.

Severn Trent Laboratories, Inc.
STL Tampa 6712 Benjamin Road, Suite 100, Tampa, FL 33634
Tel 813-8857427 Fax 813-8857049 www.stl-inc.com

METHOD SUMMARY

Client: Mosaic Phosphates

Job Number: 660-1919.1

Description	Method	Preparation Method
Matrix: Water		
ICP Metals by 200.7 CWA	40CFR136A 200.7 Appx C	
Sample Filtration		FILTRATION
Inductively Coupled Plasma - Atomic Emission Spectrometry	SW846 6010B	
Acid Digestion of Waters for Total Recoverable or Dissolved Metals		SW846 3005A
Chloride (Colorimetric, Automated Ferricyanide, AAI	MCAWW 325.2	
Nitrogen (Ammonia, Colorimetric, Automated Phenate)	MCAWW 350.1	
Phosphorus, All Forms, Colorimetric, Single Reagent	EPA 365.2	
Sample Digestion for Total Phosphorous		MCAWW 365.2/365.3
Sulfate (Turbidimetric)	MCAWW 375.4	
Fluoride (Ion-selective Electrode)	SM18 4500-F_C	

REFERENCES

40CFR136A - "Methods for Organic Chemical Analysis of Municipal Industrial Wastewater", 40CFR, Part 136, Appendix A, October 26, 1984 and subsequent revisions.

EPA - US Environmental Protection Agency

MCAWW - "Methods For Chemical Analysis Of Water And Wastes", EPA-600/4-79-020, March 1983 And Subsequent Revisions.

SM18 - "Standard Methods For The Examination Of Water And Wastewater", 18th Edition, 1992.

SW846 - "Test Methods For Evaluating Solid Waste, Physical/Chemical Methods", Third Edition, November 1986 And Its Updates.

SAMPLE SUMMARY

Client: Mosaic Phosphates

Job Number: 660-1919.1

Lab Sample ID	Client Sample ID	Client Matrix	Date/Time Sampled	Date/Time Received
660-1919-1	WS3	Water	05/13/2005 1324	05/13/2005 1400

Mr. Dave Turley
Mosaic Phosphates
P.O. Box 2000
Mulberry, FL 33860

Job Number: 660-1919.1
Lab Sample Id: 660-1919-1
Date Sampled: 05/13/2005 1324
Date Received: 05/13/2005 1400

Client Sample ID: WS3

	Result/Qualifier	Unit	RL	Method	Date Prepared	Date Analyzed	Dilution
METALS							
SiO2, Silica	5800000	ug/L	50000	200.7 Appx C-	05/20/2005 1102	05/23/2005 1450	500
Aluminum	120	mg/L	0.70	6010B-Total	05/19/2005 1150	05/23/2005 1219	10
Calcium	1800	mg/L	0.85	6010B-Total	05/19/2005 1150	05/23/2005 1219	10
Iron	180	mg/L	0.22	6010B-Total	05/19/2005 1150	05/23/2005 1219	10
Potassium	300	mg/L	1.9	6010B-Total	05/19/2005 1150	05/23/2005 1219	10
Magnesium	250	mg/L	1.1	6010B-Total	05/19/2005 1150	05/23/2005 1219	10
Sodium	2000	mg/L	3.1	6010B-Total	05/19/2005 1150	05/23/2005 1219	10
GENERAL CHEMISTRY							
Chloride	0.90 U	mg/L	0.90	325.2		05/17/2005 1900	1.0
Ammonia	31	mg/L	0.040	350.1		05/16/2005 2045	1.0
Phosphorus, Total	160000	mg/L	3400	365.2	06/01/2005 1200	06/01/2005 1200	10,000
Sulfate	5800	mg/L	340	375.4		05/16/2005 0900	200
Fluoride	9800	mg/L	440	4500-F_C		05/17/2005 0830	10,000

DATA REPORTING QUALIFIERS

Client: Mosaic Phosphates

Job Number: 660-1919.1

Lab Section	Qualifier	Description
General Chemistry		
	U	*Indicates that the compound was analyzed for but not detected.*

STL Tampa Water Sampling Log

[illegible] Mosaic	SITE LOCATION: N South Pierce
[illegible] Cooling Pond	SAMPLE ID: WS3 | DATE: 05/13/05

Calibration Record

[illegible]	Date	Time	Standard Value	Response	% Deviation	Calibrated/ Checked
[illegible] (units)						
[illegible]						
[illegible] (µmhos/cm)						
[illegible] (NTU)						
[illegible] (mV)						
D.O. [illegible] (% saturation)						

Meter Type - QED MP20 Flow Cell (SN -) -Hach 2100P Turbidimeter (SN -) - Other: ______ (SN-)

Other: ______ (SN -) - Other: ______ (SN -) - Other: ______ (SN-)

[illegible] - see page: 99 for calibration information if none listed

Initial - ______ Continuing - ✓ End of Project Check - ______

PURGING DATA

WELL DIAMETER (inches): NA	TUBING DIAMETER (inches): NA	WELL SCREEN INTERVAL DEPTH: feet to NA feet	STATIC DEPTH TO WATER (feet): NA	PURGE PUMP TYPE OR BAILER: NA

WELL VOLUME PURGE: 1 WELL VOLUME = (TOTAL WELL DEPTH – STATIC DEPTH TO WATER) X WELL CAPACITY
(only fill out if applicable) NA = (feet – feet) X gallons/foot = gallons

EQUIPMENT VOLUME PURGE: 1 EQUIPMENT VOL. = PUMP VOLUME + (TUBING CAPACITY X TUBING LENGTH) + FLOW CELL VOLUME
(only fill out if applicable) NA = gallons + (gallons/foot X feet) + gallons = gallons

INITIAL PUMP OR TUBING DEPTH IN WELL (feet): NA	FINAL PUMP OR TUBING DEPTH IN WELL (feet): NA	PURGING INITIATED AT:	PURGING ENDED AT:	TOTAL VOLUME PURGED (gallons):

TIME	VOLUME PURGED (gallons)	CUMUL. VOLUME PURGED (gallons)	PURGE ~~RATE~~ (gpm)	DEPTH TO ~~WATER~~ (feet)	pH (standard units)	TEMP. (°C)	COND. (µmhos/cm)	DISS. OXYGEN (mg/L)	TURBIDITY (NTU)	Redox	COLOR	ODOR (Y/N)
1324	(WS3)				1.61	47.5	32.9	4.28	113	367	brown	Yes
1311	W1				1.59	35.3						
1315	W2				1.61	46.9						
1344	W4				1.63	46.8						
1351	W5				1.66	46.6						

no samples collected from these locations, only Temp and ph

WELL CAPACITY (Gallons Per Foot): 0.75" = 0.02; 1" = 0.04; 1.25" = 0.06; 2" = 0.16; 3" = 0.37; 4" = 0.65; 5" = 1.02; 6" = 1.47; 12" = 5.88
TUBING INSIDE DIA. CAPACITY (Gal./Ft.): 1/8" = 0.0006; 3/16" = 0.0014; ¼" = 0.0026; 5/16" = 0.004; 3/8" = 0.006; 1/2" = 0.010; 5/8" = 0.016

SAMPLING DATA

SAMPLED BY (PRINT) / AFFILIATION: STL-Tampa	SAMPLER(S) SIGNATURES: Barbara [illegible]	SAMPLING INITIATED AT:	SAMPLING ENDED AT:
PUMP OR TUBING DEPTH IN WELL (feet): NA	SAMPLE PUMP FLOW RATE (mL per minute): NA	SAMPLING EQUIPMENT / TUBING MATERIAL CODE: PE Longarm Sampler	
FIELD DECONTAMINATION: Y [✓ N]	FIELD-FILTERED: Y [✓ N] FILTER SIZE: ____ µm Filtration Equipment Type:	DUPLICATE: Y [✓ N]	

REMARKS:
Samples taken from cooling pond

PH CHECK PERFORMED ON PRESERVED SAMPLE Y / [✓ N]

PRESERVATIVE USED - ______ TOTAL VOLUME ADDED IN FIELD - ______ FINAL PH - ______

SAMPLING/PURGING EQUIPMENT CODES: APP = After Peristaltic Pump; B = Bailer; BP = Bladder Pump; ESP = Electric Submersible Pump; PP = Peristaltic Pump; RFPP = Reverse Flow Peristaltic Pump; SM = Straw Method (Tubing Gravity Drain); VT = Vacuum Trap; O = Other (Specify)

NOTES: 1. The above do not constitute all of the information required by Chapter 62-160, F.A.C.
2. STABILIZATION CRITERIA FOR RANGE OF VARIATION OF LAST THREE CONSECUTIVE READINGS (SEE FS 2212, SECTION 3)
pH: ± 0.2 units Temperature: ± 0.2 °C Specific Conductance: ± 5% Dissolved Oxygen: all readings ≤ 20% saturation (see Table FS 2200-2); optionally, ± 0.2 mg/L or ± 10% (whichever is greater) Turbidity: all readings ≤ 20 NTU; optionally ± 5 NTU or ± 10% (whichever is greater)

Serial Number 24097

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

SEVERN TRENT STL

☒ **STL Tampa**
6712 Benjamin Road, Suite 100
Tampa, FL 33634

Website: www.stl-inc.com
Phone: (813) 885-7427
Fax: (813) 885-7049

660-1919

○ Alternate Laboratory Name/Location
Phone:
Fax:

PROJECT REFERENCE Cooling Pond / South Pierce	PROJECT NO.	PROJECT LOCATION (STATE) FL	PAGE 1 OF 1
SAMPLER'S SIGNATURE Barbara S Hartley	P.O. NUMBER	CONTRACT NO.	STANDARD REPORT DELIVERY ○ DATE DUE ______
CLIENT (SITE) PM FL	CLIENT PHONE	CLIENT FAX	
CLIENT NAME Mosaic	CLIENT E-MAIL		EXPEDITED REPORT DELIVERY (SURCHARGE) ○ DATE DUE ______
CLIENT ADDRESS			
COMPANY CONTRACTING THIS WORK (if applicable)			NUMBER OF COOLERS SUBMITTED PER SHIPMENT:

MATRIX TYPE — REQUIRED ANALYSIS — PRESERVATIVE — NUMBER OF CONTAINERS SUBMITTED

SAMPLE DATE	SAMPLE TIME	SAMPLE IDENTIFICATION	COMPOSITE (C) OR GRAB (G) INDICATE	AQUEOUS (WATER)	SOLID OR SEMISOLID	AIR	NONAQUEOUS LIQUID (OIL, SOLVENT,...)	F, SO4, Cl cool	TP, NH3 H2SO4	Silica (Sm) cool	Al, Ca, Fe, K, Mg, Na HNO3							REMARKS
05/13/05	1324	WS3	G	✓				1	1	1	1							

RELINQUISHED BY: (SIGNATURE) EMPTY CONTAINERS	DATE	TIME	RELINQUISHED BY: (SIGNATURE) Barbara S Hartley	DATE 05/13/05	TIME	RELINQUISHED BY: (SIGNATURE)	DATE	TIME
RECEIVED BY: (SIGNATURE) EMPTY CONTAINERS Barbara S Hartley	DATE 05/13/05	TIME 1238	RECEIVED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME

LABORATORY USE ONLY 4

RECEIVED FOR LABORATORY BY (SIGNATURE)	DATE 5-13-05	TIME 1400	CUSTODY INTACT YES ☒ NO ○	CUSTODY SEAL NO. N/S	STL TAMPA LOG NO. 660-1919	LABORATORY REMARKS

STL8240-660 (12/02)

Mar-05			tons F	tpy F	
			41975	160	
acre-ft	cf	lb total	lb F	lb F/day	delta/day
1723	74950500	4676911200	83950556	877	0.0010%
			% F		
			1.8		
			1.3		
			3.1		
			0.98		
			average		
			1.795		

SEVERN TRENT STL

ANALYTICAL REPORT

Job Number: 660-1966.1

Job Description: SP East Cooling Pond

For:

Mosaic Phosphates
P.O. Box 2000
Mulberry, FL 33860

Attention: Mr. Dave Turley

Tina Fritz

Tina Fritz
Project Manager II
tfritz@stl-inc.com
06/14/2005

Methods: FDEP, DOH Certification #: E84282 These test results meet all the requirements of NELAC. All questions regarding this test report should be directed to the STL Project Manager who signed this test report. The estimated uncertainty associated with these reported results is available upon request.

METHOD SUMMARY

Client: Mosaic Phosphates

Job Number: 660-1966.1

Description	Method	Preparation Method
Matrix: Water		
ICP Metals by 200.7 CWA	40CFR136A 200.7 Appx C	
Sample Filtration		FILTRATION
ICP Metals by 200.7	EPA 200.7 Rev 4.4	
Total Recoverable Metals Digestion for 200.7		40CFR136A 200.7 Appx C
Field Sampling	EPA Field Sampling	
Chloride (Colorimetric, Automated Ferricyanide, AAI	MCAWW 325.2	
Nitrogen (Ammonia, Colorimetric, Automated Phenate)	MCAWW 350.1	
Phosphorus, All Forms, Colorimetric, Single Reagent	EPA 365.2	
Sample Digestion for Total Phosphorous		MCAWW 365.2/365.3
Sulfate (Turbidimetric)	MCAWW 375.4	
Fluoride (Ion-selective Electrode)	SM18 4500-F_C	

REFERENCES

40CFR136A - "Methods for Organic Chemical Analysis of Municipal Industrial Wastewater", 40CFR, Part 136, Appendix A, October 26, 1984 and subsequent revisions.

EPA - US Environmental Protection Agency

MCAWW - "Methods For Chemical Analysis Of Water And Wastes", EPA-600/4-79-020, March 1983 And Subsequent Revisions.

SM18 - "Standard Methods For The Examination Of Water And Wastewater", 18th Edition, 1992.

SAMPLE SUMMARY

Client: Mosaic Phosphates

Job Number: 660-1966.1

Lab Sample ID	Client Sample ID	Client Matrix	Date/Time Sampled	Date/Time Received
660-1966-1	EAST POND	Water	05/17/2005 1444	05/18/2005 0812

Mr. Dave Turley
Mosaic Phosphates
P.O. Box 2000
Mulberry, FL 33860

Job Number: 660-1966.1
Lab Sample Id: 660-1966-1
Date Sampled: 05/17/2005 1444
Date Received: 05/18/2005 0812

Client Sample ID: EAST POND

	Result/Qualifier		Unit	RL	Method	Date Prepared	Date Analyzed	Dilution
METALS								
SiO2, Silica	5600000		ug/L	50000	200.7 Appx	05/20/2005 1102	05/23/2005 1505	500
Aluminum	140		mg/L	0.70	200.7 Rev 4.4	05/20/2005 1634	05/23/2005 1600	10
Calcium	1900		mg/L	0.85	200.7 Rev 4.4	05/20/2005 1634	05/23/2005 1600	10
Iron	190		mg/L	0.22	200.7 Rev 4.4	05/20/2005 1634	05/23/2005 1600	10
Potassium	200		mg/L	1.9	200.7 Rev 4.4	05/20/2005 1634	05/23/2005 1600	10
Magnesium	270		mg/L	1.1	200.7 Rev 4.4	05/20/2005 1634	05/23/2005 1600	10
Sodium	1800		mg/L	3.1	200.7 Rev 4.4	05/20/2005 1634	05/23/2005 1600	10
GENERAL CHEMISTRY								
Chloride	62		mg/L	0.90	325.2		06/01/2005 1830	1.0
Ammonia	34		mg/L	0.040	350.1		05/20/2005 2128	1.0
Phosphorus, Total	0.017	U	mg/L	0.017	365.2	05/26/2005 2300	05/26/2005 2300	1.0
Sulfate	4200		mg/L	340	375.4		05/24/2005 1100	200
Fluoride	31000	V	mg/L	1100	4500-F_C		05/23/2005 1030	25,000
FIELD SERVICE / MOBILE LAB								
Field pH	1.88D		SU		Field Sampling		05/17/2005 1444	1.0
Field Temperature	49.2D		Degrees		Field Sampling		05/17/2005 1444	1.0
Temperature, Air	38.3D		Degrees		Field Sampling		05/17/2005 1444	1.0

DATA REPORTING QUALIFIERS

Client: Mosaic Phosphates

Job Number: 660-1966.1

Lab Section	Qualifier	Description
General Chemistry		
	U	Indicates that the compound was analyzed for but not detected.
	V	Indicates the analyte was detected in both the sample and the associated method blank.

STL Tampa Water Sampling Log

CLIENT NAME: Mosaic
SITE LOCATION: South Pierce
PROJECT NAME: Gyp Pond Sampling Cooling Ponds
SAMPLE ID: East Pond
DATE: 05/17/05

Calibration Record

Calibration	Date	Time	Standard Value	Response	% Deviation	Calibrated/ Checked
7.00/10.00 (units)						
N.A. (°C)						
(umhos/cm)						
(NTU)						
(mV)						
D.O. (% saturation)						

Meter Type - QED MP20 Flow Cell (SN -) -Hach 2100P Turbidimeter (SN -) - Other________ (SN-)
Other ________ (SN -) - Other: ________ (SN -) - Other________ (SN-)
see page: 105 for calibration Information if none listed
Initial ________ Continuing - ✓ End of Project Check - ________

PURGING DATA

WELL DIAMETER (inches): NA
TUBING DIAMETER (inches): NA
WELL SCREEN INTERVAL DEPTH: feet to feet NA
STATIC DEPTH TO WATER (feet): NA
PURGE PUMP TYPE OR BAILER:

WELL VOLUME PURGE: 1 WELL VOLUME = (TOTAL WELL DEPTH − STATIC DEPTH TO WATER) X WELL CAPACITY
only fill out if applicable) NA = (feet − feet) X gallons/foot = gallons

EQUIPMENT VOLUME PURGE: 1 EQUIPMENT VOL = PUMP VOLUME + (TUBING CAPACITY X TUBING LENGTH) + FLOW CELL VOLUME
(only fill out if applicable) NA = gallons + (gallons/foot X feet) + gallons = gallons

INITIAL PUMP OR TUBING DEPTH IN WELL (feet): NA
FINAL PUMP OR TUBING DEPTH IN WELL (feet): NA
PURGING INITIATED AT: NA
PURGING ENDED AT: NA
TOTAL VOLUME PURGED (gallons): NA

TIME	VOLUME PURGED (gallons)	CUMUL. VOLUME PURGED (gallons)	PURGE RATE (gpm)	DEPTH TO WATER (feet)	pH (standard units)	TEMP. (°C)	COND. (µmhos/cm)	DISS. OXYGEN (mg/L)	TURBIDITY (NTU)	Redox (mV)	COLOR	ODOR (Y/N)
1444					1.78	33.9	35500	3.29	44.1	343	Tan	Yes
		W 1			1.76	39.0						
		W 2			1.88	49.2						

WELL CAPACITY (Gallons Per Foot): 0.75" = 0.02; 1" = 0.04; 1.25" = 0.06; 2" = 0.16; 3" = 0.37; 4" = 0.65; 5" = 1.02; 6" = 1.47; 12" = 5.88
TUBING INSIDE DIA. CAPACITY (Gal./Ft.): 1/8" = 0.0006; 3/16" = 0.0014; ¼" = 0.0026; 5/16" = 0.004; 3/8" = 0.006; 1/2" = 0.010; 5/8" = 0.016

SAMPLING DATA

SAMPLED BY (PRINT) / AFFILIATION: STL Tampa
SAMPLER(S) SIGNATURES: Barbara S Hartley / Joe Miller
SAMPLING INITIATED AT: 1325
SAMPLING ENDED AT: 1645
PUMP OR TUBING DEPTH IN WELL (feet): NA
SAMPLE PUMP FLOW RATE (mL per minute): NA
SAMPLING EQUIPMENT / TUBING MATERIAL CODE:
FIELD DECONTAMINATION: Y [✓ N]
FIELD-FILTERED: Y [✓ N] FILTER SIZE: ____ µm
Filtration Equipment Type: ________
DUPLICATE: Y [✓ N]
REMARKS: 38.3 = Air Temp Sample collected from East Pond Only
PH CHECK PERFORMED ON PRESERVED SAMPLE Y [✓ N]
PRESERVATIVE USED: TOTAL VOLUME ADDED IN FIELD - ________ FINAL PH - ________

SAMPLING/PURGING EQUIPMENT CODES: APP = After Peristaltic Pump; B = Bailer; BP = Bladder Pump; ESP = Electric Submersible Pump; PP = Peristaltic Pump; RFPP = Reverse Flow Peristaltic Pump; SM = Straw Method (Tubing Gravity Drain); VT = Vacuum Trap; O = Other (Specify)

NOTES: 1. The above do not constitute all of the information required by Chapter 62-160, F.A.C.
2. STABILIZATION CRITERIA FOR RANGE OF VARIATION OF LAST THREE CONSECUTIVE READINGS (SEE FS 2212, SECTION 3)
pH: ± 0.2 Units Temperature: ± 0.2 °C Specific Conductance: ± 5% Dissolved Oxygen: all readings ≤ 20% saturation (see Table FS 2200-2);

Serial Number 24093

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

SEVERN TRENT **STL**

STL Tampa
6712 Benjamin Road, Suite 100
Tampa, FL 33634

Website: www.stl-inc.com
Phone: (813) 885-7427 660-1966
Fax: (813) 885-7049

○ Alternate Laboratory Name/Location
Phone:
Fax:

OJECT REFERENCE uth Pierce/Gyp Pnd/Cooling	PROJECT NO.	PROJECT LOCATION (STATE) FL
MPLER'S SIGNATURE Barbara S Hartley	P.O. NUMBER	CONTRACT NO.
IENT (SITE) PM TF	CLIENT PHONE	CLIENT FAX
IENT NAME Mosaic	CLIENT E-MAIL	
ENT ADDRESS		
MPANY CONTRACTING THIS WORK (if applicable)		

PAGE 1 OF 1

MATRIX TYPE: COMPOSITE (C) OR GRAB (G) INDICATE; AQUEOUS (WATER); SOLID OR SEMISOLID; AIR; NONAQUEOUS LIQUID (OIL, SOLVENT,...)

REQUIRED ANALYSIS:
- F, SO4, Cl — cool
- TP, NH3 — H2SO4
- Silica (Sunnerell) — cool
- Al, Ca, Fe, K, Mg, Na — HNO3

STANDARD REPORT DELIVERY ○
DATE DUE ____________

EXPEDITED REPORT DELIVERY (SURCHARGE) ○
DATE DUE ____________

NUMBER OF COOLERS SUBMITTED PER SHIPMENT:

SAMPLE DATE	SAMPLE TIME	SAMPLE IDENTIFICATION	COMPOSITE (C) OR GRAB (G)	AQUEOUS (WATER)	SOLID OR SEMISOLID	AIR	NONAQUEOUS LIQUID	F, SO4, Cl	TP, NH3	Silica	Al, Ca, Fe, K, Mg, Na	REMARKS
5/17/05	1444	East Pond						1	1	1	1	ph 1.78

NUMBER OF CONTAINERS SUBMITTED

LINQUISHED BY: (SIGNATURE)	DATE	TIME	RELINQUISHED BY: (SIGNATURE) Barbara S Hartley	DATE 05/18/05	TIME 0812	RELINQUISHED BY: (SIGNATURE)	DATE	TIME
CEIVED BY: (SIGNATURE) Barbara S Hartley	DATE 05/09/05	TIME 1630	RECEIVED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME

LABORATORY USE ONLY 4

CEIVED FOR LABORATORY BY: NATURE) Charles Cloby	DATE 5-18-05	TIME 0812	CUSTODY INTACT ● YES ○ NO	CUSTODY SEAL NO. N/S	STL TAMPA LOG NO. 660-1966	LABORATORY REMARKS

SEVERN TRENT STL

ANALYTICAL REPORT

Job Number: 660-1995.1

Job Description: SP NW Cooling Pond

For:

Mosaic Phosphates
P.O. Box 2000
Mulberry, FL 33860

Attention: Mr. Dave Turley

Tina Fritz
Project Manager II
tfritz@stl-inc.com
06/14/2005

Methods: FDEP, DOH Certification #: E84282 These test results meet all the requirements of NELAC. All questions regarding this test report should be directed to the STL Project Manager who signed this test report. The estimated uncertainty associated with these reported results is available upon request.

Severn Trent Laboratories, Inc.
STL Tampa 6712 Benjamin Road, Suite 100, Tampa, FL 33634
Tel 813-8857427 Fax 813-8857049 www.stl-inc.com

METHOD SUMMARY

Client: Mosaic Phosphates

Job Number: 660-1995.1

Description	Method	Preparation Method
Matrix: Water		
ICP Metals by 200.7 CWA	40CFR136A 200.7 Appx C	
Sample Filtration		FILTRATION
ICP Metals by 200.7	EPA 200.7 Rev 4.4	
Total Recoverable Metals Digestion for 200.7		40CFR136A 200.7 Appx C
Field Sampling	EPA Field Sampling	
Chloride (Colorimetric, Automated Ferricyanide, AAI	MCAWW 325.2	
Nitrogen (Ammonia, Colorimetric, Automated Phenate)	MCAWW 350.1	
Phosphorus, All Forms, Colorimetric, Single Reagent	EPA 365.2	
Sample Digestion for Total Phosphorous		MCAWW 365.2/365.3
Sulfate (Turbidimetric)	MCAWW 375.4	
Fluoride (Ion-selective Electrode)	SM18 4500-F_C	

REFERENCES

40CFR136A - "Methods for Organic Chemical Analysis of Municipal Industrial Wastewater", 40CFR, Part 136, Appendix A, October 26, 1984 and subsequent revisions.

EPA - US Environmental Protection Agency

MCAWW - "Methods For Chemical Analysis Of Water And Wastes", EPA-600/4-79-020, March 1983 And Subsequent Revisions.

SM18 - "Standard Methods For The Examination Of Water And Wastewater", 18th Edition, 1992.

SAMPLE SUMMARY

Client: Mosaic Phosphates

Job Number: 660-1995.1

Lab Sample ID	Client Sample ID	Client Matrix	Date/Time Sampled	Date/Time Received
660-1995-1	Northwest Cooling Pond	Water	05/18/2005 1339	05/18/2005 1525

Mr. Dave Turley
Mosaic Phosphates
P.O. Box 2000
Mulberry, FL 33860

Job Number:	660-1995.1
Lab Sample Id:	660-1995-1
Date Sampled:	05/18/2005 1339
Date Received:	05/18/2005 1525

Client Sample ID: Northwest Cooling Pond

	Result/Qualifier		Unit	RL	Method	Date Prepared	Date Analyzed	Dilution
METALS								
SiO2, Silica	6800000		ug/L	50000	200.7 Appx	05/20/2005 1102	05/23/2005 1507	500
Aluminum	130		mg/L	0.70	200.7 Rev 4.4	05/21/2005 1319	05/25/2005 1425	10
Calcium	2000	V	mg/L	0.85	200.7 Rev 4.4	05/21/2005 1319	05/25/2005 1425	10
Iron	210		mg/L	0.22	200.7 Rev 4.4	05/21/2005 1319	05/25/2005 1425	10
Potassium	330		mg/L	1.9	200.7 Rev 4.4	05/21/2005 1319	05/25/2005 1425	10
Magnesium	280		mg/L	1.1	200.7 Rev 4.4	05/21/2005 1319	05/25/2005 1425	10
Sodium	2300		mg/L	3.1	200.7 Rev 4.4	05/21/2005 1319	05/25/2005 1425	10
GENERAL CHEMISTRY								
Chloride	0.90	U	mg/L	0.90	325.2		05/20/2005 2230	1.0
Ammonia	35		mg/L	0.040	350.1		05/20/2005 2135	1.0
Phosphorus, Total	8400		mg/L	170	365.2	05/27/2005 1105	05/27/2005 1105	10,000
Sulfate	5500		mg/L	340	375.4		05/24/2005 1300	200
Fluoride	13000	V	mg/L	440	4500-F_C		05/23/2005 1030	10,000
FIELD SERVICE / MOBILE LAB								
Field pH	1.83D		SU		Field Sampling		05/18/2005 1339	1.0
Field Temperature	4.79D		Degrees		Field Sampling		05/18/2005 1339	1.0
Temperature, Air	33.4D		Degrees		Field Sampling		05/18/2005 1339	1.0

DATA REPORTING QUALIFIERS

Client: Mosaic Phosphates

Job Number: 660-1995.1

Lab Section	Qualifier	Description
Metals		
	V	Indicates the analyte was detected in both the sample and the associated method blank.
General Chemistry		
	U	Indicates that the compound was analyzed for but not detected.
	V	Indicates the analyte was detected in both the sample and the associated method blank.

STL Tampa Water Sampling Log

CLIENT NAME: Mosaic		SITE LOCATION: South Pierce	
PROJECT NAME: Gyp Stack Sampling Ind	SAMPLE ID: Northwest Cooling Pond		DATE: 05/18/05

Calibration Record

Calibration	Date	Time	Standard Value	Response	% Deviation	Calibrated/ Checked
7.00/10.01 (pH)						
[illegible]						
[illegible] (umhos/cm)						
[illegible] (NTU)						
[illegible] (mg/L)						
D.O. (% saturation)						

Meter Type: QED MP20 Flow Cell (SN -) -Hach 2100P Turbidimeter:(SN -) - Other:____ (SN-)
Other: ____ (SN -) - Other: ____ (SN -) - Other:____ (SN-)
[✓ see page: 108] for calibration information if none listed
Initial - ____ Continuing - ____ End of Project Check - ____

PURGING DATA

WELL DIAMETER (inches): NA	TUBING DIAMETER (inches): NA	WELL SCREEN INTERVAL DEPTH: feet to NA feet	STATIC DEPTH TO WATER (feet): NA	PURGE PUMP TYPE OR BAILER: NA

WELL VOLUME PURGE: 1 WELL VOLUME = (TOTAL WELL DEPTH – STATIC DEPTH TO WATER) X WELL CAPACITY
(only fill out if applicable) NA = (feet – feet) X gallons/foot = gallons

EQUIPMENT VOLUME PURGE: 1 EQUIPMENT VOL = PUMP VOLUME + (TUBING CAPACITY X TUBING LENGTH) + FLOW CELL VOLUME
(only fill out if applicable) NA = gallons + (gallons/foot X feet) + gallons = gallons

INITIAL PUMP OR TUBING DEPTH IN WELL (feet): NA	FINAL PUMP OR TUBING DEPTH IN WELL (feet): NA	PURGING INITIATED AT: NA	PURGING ENDED AT: NA	TOTAL VOLUME PURGED (gallons): NA

TIME	VOLUME PURGED (gallons)	CUMUL. VOLUME PURGED (gallons)	PURGE RATE (gpm)	DEPTH TO WATER (feet)	pH (standard units)	TEMP. (°C)	COND. (μmhos/cm)	DISS. OXYGEN (mg/L)	TURBIDITY (NTU)		COLOR	ODOR (Y/N)
1339					1.83	42.4						
		W1			1.80	39.1						
		W2			1.83	47.9						

WELL CAPACITY (Gallons Per Foot): 0.75" = 0.02; 1" = 0.04; 1.25" = 0.06; 2" = 0.16; 3" = 0.37; 4" = 0.65; 5" = 1.02; 6" = 1.47; 12" = 5.88
TUBING INSIDE DIA. CAPACITY (Gal./Ft.): 1/8" = 0.0006; 3/16" = 0.0014; ¼" = 0.0026; 5/16" = 0.004; 3/8" = 0.006; 1/2" = 0.010; 5/8" = 0.016

SAMPLING DATA

SAMPLED BY (PRINT) / AFFILIATION: STL Tampa	SAMPLER(S) SIGNATURES: Barbara S Hartley	SAMPLING INITIATED AT: 1242	SAMPLING ENDED AT: 1402
PUMP OR TUBING DEPTH IN WELL (feet): NA	SAMPLE PUMP FLOW RATE (mL per minute): NA	SAMPLING EQUIPMENT / TUBING MATERIAL CODE: NA	
FIELD DECONTAMINATION: Y [✓ N]	FIELD-FILTERED: Y [✓ N] Filtration Equipment Type:	FILTER SIZE: ___ μm	DUPLICATE: Y [✓ N]

REMARKS: 33.4°C = Air Temp

PH CHECK PERFORMED ON PRESERVED SAMPLE Y / N
PRESERVATIVE USED - ____ TOTAL VOLUME ADDED IN FIELD - ____ FINAL PH - ____

SAMPLING/PURGING EQUIPMENT CODES: APP = After Peristaltic Pump; B = Bailer; BP = Bladder Pump; ESP = Electric Submersible Pump; PP = Peristaltic Pump; RFPP = Reverse Flow Peristaltic Pump; SM = Straw Method (Tubing Gravity Drain); VT = Vacuum Trap; O = Other (Specify)

NOTES: 1. The above do not constitute all of the information required by Chapter 62-160, F.A.C.
2. STABILIZATION CRITERIA FOR RANGE OF VARIATION OF LAST THREE CONSECUTIVE READINGS (SEE FS 2212, SECTION 3)
pH: ± 0.2 units Temperature: ± 0.2 °C Specific Conductance: ± 5% Dissolved Oxygen: all readings ≤ 20% saturation (see Table FS 2200-2); optionally, ± 0.2 mg/L or ± 10% (whichever is greater) Turbidity: all readings ≤ 20 NTU; optionally + 5 NTU or + 10% (whichever is greater)

24094

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

SEVERN TRENT STL

STL Tampa
6712 Benjamin Road, Suite 100
Tampa, FL 33634

Website: www.stl-inc.com
Phone: (813) 885-7427
Fax: (813) 885-7049
660-1995

○ Alternate Laboratory Name/Location
Phone:
Fax:

ROJECT REFERENCE Gyp Pond Sampling South Pierce	PROJECT NO.	PROJECT LOCATION (STATE) Fl
AMPLER'S SIGNATURE Barbara S Hartley	P.O. NUMBER	CONTRACT NO.
CLIENT (SITE) PM	CLIENT PHONE	CLIENT FAX
CLIENT NAME Mosaic	CLIENT E-MAIL	
CLIENT ADDRESS		
COMPANY CONTRACTING THIS WORK (if applicable)		

MATRIX TYPE: COMPOSITE (C) OR GRAB (G) INDICATE / AQUEOUS (WATER) / SOLID OR SEMISOLID / AIR / NONAQUEOUS LIQUID (OIL, SOLVENT,...)

REQUIRED ANALYSIS / NUMBER OF CONTAINERS SUBMITTED

SAMPLE DATE	SAMPLE TIME	SAMPLE IDENTIFICATION	C/G	AQUEOUS (WATER)	SOLID OR SEMISOLID	AIR	NONAQUEOUS LIQUID	F, SO4, Cl Cool	TP, NH3 H2SO4	Silica ([illegible]) Cool	Al, Ca, Fe, K, Mg, Na HNO3	REMARKS
05/18/05	1339	Northwest Cooling Pond	G	✓				1	1	1	1	Ph = 1.83

PAGE OF

STANDARD REPORT DELIVERY ○
DATE DUE

EXPEDITED REPORT DELIVERY (SURCHARGE) ○
DATE DUE

NUMBER OF COOLERS SUBMITTED PER SHIPMENT:

RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RELINQUISHED BY: (SIGNATURE) Barbara S Hartley	DATE 05/18/05	TIME 1525	RELINQUISHED BY: (SIGNATURE)	DATE	TIME
RECEIVED BY: (SIGNATURE) Barbara S Hartley	DATE 05/09/05	TIME 1630	RECEIVED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME

LABORATORY USE ONLY 4

RECEIVED FOR LABORATORY BY: (SIGNATURE)	DATE 5-18-05	TIME 1525	CUSTODY INTACT YES ☒ NO ○	CUSTODY SEAL NO. N/S	STL TAMPA LOG NO. 660-1995	LABORATORY REMARKS

STL8240-660 (12/02)