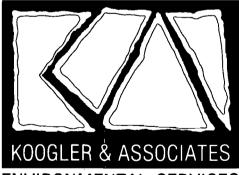
FLUORIDE EMISSION MEASUREMENTS
MULTIFOS MIXED-FEED BUILDING

IMC-AGRICO NEW WALES, POLK COUNTY, FLORIDA

APRIL 5-7, 2000

KOOGLER & ASSOCIATES ENVIRONMENTAL SERVICES 4014 N.W. 13TH STREET GAINESVILLE, FLORIDA 352-377-5822



ENVIRONMENTAL SERVICES

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FLUORIDE EMISSION MEASUREMENTS **MULTIFOS MIXED-FEED BUILDING**

IMC-AGRICO
NEW WALES, POLK COUNTY, FLORIDA RECEIVED

JUN 12 2000 BUREAU OF AIR REGULATION

APRÎL 5-7, 2000

KOOGLER & ASSOCIATES ENVIRONMENTAL SERVICES 4014 N.W. 13TH STREET GAINESVILLE, FLORIDA 352-377-5822



To the best of my knowledge, all applicable field and 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.

State of Florida Registration No. 12925

Date



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1.0 INTRODUCTION

IMC-Agrico operates a phosphate fertilizer complex at New Wales, Polk County, Florida. The fertilizer complex includes manufacturing facilities for phosphate based animal feed ingredients. During the period April 5-7, 2000, Koogler & Associates Environmental Services of Gainesville, Florida, conducted fluoride emission measurements from the multifos mixed-feed storage building.

The multifos mixed-feed (mixed-feed) storage building is 90 feet long (east-west), 67.5 feet wide (north-south) and 55 feet high. The building is a modified A-frame building with north and south sidewalls approximately 14 feet high. The building is ventilated only by the natural ventilation. The openings in the building include a large door at the west end for front-end loader ingress and egress. A man door at the west end of the building and a conveyor opening at the east end of the building, both approximately 25 feet below the ridge line, man doors at the east end of the building and the south side of the building both near the southeast corner of the building and an equipment opening at ground level near the southeast corner of the building. Additionally, there is a ridge vent which runs the entire length (90 feet) of the building. During the period of testing, the man doors were closed. The openings that remained were the loader door at the west end of the building, the east end of the building, the equipment opening at the east



end of the building and the roof vent. A sketch of the building and openings is included in the Appendix of this report.

The sampling procedure used to measure fluoride emissions from the mixed-feed building was based upon a protocol previously approved by FDEP for measuring fluoride emissions from GTSP storage buildings. The method includes the measurement of the areas of all openings in the building, periodic measurements of airflow through these openings (either into or out of the building) and the measure of the fluoride concentration of the gases entering or leaving the building at each opening. For the April 5-7, 2000, test period, the roof vent was divided into five discrete openings, the conveyor opening and the equipment opening at the east end of the building were considered as discrete openings and the loader door at the west end of the building was divided into two discrete openings.

During the period of testing, the multifos production rate and hence, the multifos transfer rate into the mixed-feed building, averaged 79.7 tons per hour. During the three day test period, the building ranged between 10-80 percent filled with product. During all three days of the test, material was transferred into the building and loaded out of the building.

The emission measurements consisted of three separate determinations with each determination being made over a 6-7 hour period. The measured fluoride emission



rate from the building ranged from 0.0050 to 0.0210 pounds per hour and averaged 0.0108 pounds per hour. During the test period, the building ventilation rate averaged approximately 20,000 cubic feet per minute.



2.0 BUILDING DESCRIPTION

Multifos is a phosphate based animal feed product consisting of phosphoric acid, soda ash and phosphate rock. During the period of testing, the multifos production rate averaged 79.7 tons per hour. This was also the rate at which multifos was transferred into the mixed-feed storage building by overhead conveyor. The conveyor traverses the storage building from east-west approximately 25 feet below the ridge line of the building and approximately 30 feet above the floor.

The mixed-feed building is 90 feet long, 67.5 feet wide and 55 feet high. During the time of testing, the openings in the building included the ridge vent, a conveyor opening and an equipment opening at the east end of the building, and a loader door at the west end of the building. Three man doors exist in the building but were closed during the three day test period. The ridge vent has an effective width of 15 inches and a length of 90 feet resulting in a total open area of 112.5 feet. This vent was divided into five discrete openings, each with an area of 22.5 square feet. The conveyor opening at the east end of the building has an opening of 8 feet by 10 feet or 80 square feet and the equipment opening at the east end of the building has an opening 8 feet by 10.5 feet or 84 square feet. The loader door at the west end of the building is 16.5 feet high and 16 feet wide; an area of 256 square feet.



Each of the openings was represented with a single sampler. Two samplers were located in the opening of the loader door (representing the two discrete openings) and one sampler was placed upwind of the building during each of the three sampling periods to provide background fluoride measurements. The roof Samplers were numbered 1 through 5, the conveyor Sampler at the east end of the building was numbered 6, the background Sampler was numbered 7, the equipment opening Sampler at the east end of the building was numbered 8 and the loader door Samplers were numbered 9 and 10.

During the test period, multifos was fed to the building at daily rates that ranged from 75.3 to 84.0 tons per hour with an average of 79.7 tons per hour. The discharge of multifos from the overhead transfer conveyor to the storage building was generally near the center of the building; under Samplers 3 and 4. During all three sampling periods, material was being removed from the storage building by front-end loader transfer to a hopper feeding a discharge conveyor at the east end of the building. The three test periods represented normal operation of the mixed-feed building.



3.0 SAMPLING POINT LOCATIONS

As previously described, the four building openings were divided into nine discrete sampling areas. The roof vent was subdivided into five areas each with an open area of 22.5 square feet, the conveyor opening and equipment opening at the east end of the building were treated as single areas with openings of 80 and 84 square feet, respectively, and the loader door at the west end of the building was subdivided into two areas each with an area of 132 square feet. Fluoride concentrations were measured near the centroid of each of these areas and airflow and direction (in or out of the building) was measured at each open area nominally once per hour. At the beginning and end of each test period, the flow rate of each sampler (which was controlled with a critical orifice) was measured with a mass flow meter.



4.0 SAMPLING PROCEDURE

The fluoride emissions from the mixed-feed building were determined generally in accordance with a protocol previously approved by FDEP for measuring fluoride emissions from GTSP storage buildings. A copy of this protocol is included in the Appendix of this report.

The test method involves measuring the airflow into and out of the building at several locations and simultaneously measuring the fluoride concentration of the air at each of the locations. From these measurements, the mass flow of fluoride at each sampling point is calculated and the masses are algebraically summed to determine the net fluoride emissions from the building. Prior to conducting any fluoride measurements, smoke tracers were used to determine the general airflow patterns within the building. The smoke tracers demonstrated that the airflow through all openings, except the loader door, was quite well defined. This can be observed in Tables 1, 2 and 3; summaries of data representing each of the three test runs. During all three test periods, the airflow was out of the building at the five openings representing the roof vent (Samplers 1-5) and the opening on the east end of the building were the conveyor entered the building (Sampler No. 6).

At Sampler No. 8 (the equipment opening on the east end of the building), the airflow fluctuated between in and out during Run 1 and was consistently into the



building during Runs 2 and 3. The airflow at the loader opening (Samplers 9 and 10) was variable during each run. A review of the field data sheets demonstrates that the flow fluctuated from in to out and at some times the flow was into the building at the bottom of the opening and out of the building at the top (or vice versa).

To account for the fluctuating airflow at openings 9 and 10, the following procedure was used:

- A total airflow volume was calculated for each area for each time step increment
 of each of the three test runs. These airflow volumes were calculated by
 multiplying the area of the opening by the air velocity measured with a thermistor
 type airflow meter (ft/min) by the duration of the time increment.
- The incremental volumes for each opening were totaled for each test run to determine a total raw airflow volume for each opening (see Tables 1-3). The flows for all openings were summed to provide a total raw airflow balance for the building. For all three test runs, the measured airflow into the building exceed the airflow out of the building (resulting in a negative raw volume balance) because of the uncertainty caused by the fluctuating flow at Areas 9 and 10.



• As the only questionable airflow volumes were those measured at Areas 9 and 10, a total adjusted airflow volume was calculated by adjusting the volumetric flows for Areas 9 and 10 such that the total adjusted airflow volume balance for the building would be zero; that is, the airflow out of the building would equal the airflow into the building.

To determine the mass flow of fluoride at each opening, the fluoride concentration measured by the sampler at the opening was multiplied by the adjusted airflow volume for the area to give a total mass flow of fluoride (either into or out of the building) at each opening for the total duration of each test run. These calculations are summarized in Tables 1-3. The hourly fluoride emission rates for each test run were determined by dividing the total fluoride emissions for each test run by the duration of each test run. These data are summarized in Table 4.

The background fluoride concentration measured during each test run with Sampler 7 was not used to correct the mass fluoride entering or leaving the mixed-feed storage building at each sampler location. It was determined that actual measurements of the fluoride in the air entering the building (at points where the airflow was into the building) would provide a self-correcting mechanism for background fluoride concentrations. It is interesting to note, however, that for Runs 1 and 2, the fluoride concentrations measured by the background sampler approached the fluoride levels in the distilled water blanks and for Run 3 (with the



airflow across the chemical plant), the fluoride level measured by the background sampler was approximately 3.5 times of that of the distilled water blank. It should be noted that all samples were corrected for the distilled water blanks.



5.0 TEST CONDITIONS AND SUMMARY OF RESULTS

During the test period of April 5-7, 2000, the multifos plant operated normally and the transfer of product to the mixed-feed storage building and the removal of the product from the building for shipment was normal. During the test period, the multifos production rate ranged from 75.3 to 84.0 tons per hour and averaged 79.7 tons per hour.

Following are general comments and/or conditions related to each of the test runs.

Run No. 1

- Date April 5, 2000
- Wind generally from the north at 10-15 miles per hour
- Ambient temperature 51°F at 0800
- Material transfer to building began at 0928
- Sampling began nominally at 0922 and ended at 1621
- Building was approximately 10 percent full of product at the beginning of the run
- The average transfer rate of product to the mixed-feed building was 75.3 tons
 per hour for the test period.
- The material drop point (product discharge point into the mixed-feed storage building) was below Sampler 4 (near the center of the building)
- Material was being removed from the building for shipment by front-end loader during the test period.



Run No. 2

- Date April 6, 2000
- Wind generally from the east at 3-5 miles per hour
- Material transfer to building began at 0800
- Sampling began nominally at 0805 and ended at 1446. The samplers did not operate for approximately 33 minutes between 1217 and 1250 because of a power outage.
- Building was approximately 80 percent full of product at the beginning of the run
- The average transfer rate of product to the mixed-feed building was 84.0 tons per hour for the test period.
- The material drop point (product discharge point into the mixed-feed storage building) was below Sampler 4 (near the center of the building)
- Material was being removed from the building for shipment by front-end loader during the test period.

Run No. 3

- Date April 7, 2000
- Wind generally from the southeast
- Material transfer to building began at 0800
- Sampling began nominally at 0804 and ended at 1718. Samplers were off between 1012 and 1218 due to plant shutdown.
- Building was approximately 65 percent full of product at the beginning of the run
- The average transfer rate of product to the mixed-feed building was 79.7 tons per hour for the test period.
- The material drop point (product discharge point into the mixed-feed storage building) was between Samplers 3 and 4 (near the center of the building but closer to the west end).



 Material was being removed from the building for shipment by front-end loader during the test period.

The fluoride emission calculations are summarized in Tables 1-3 and the test results are summarized in Table 4. The fluoride emission rate from the building averaged 0.0108 pounds per hour. This emission rate is based on three separate measurements ranging in duration from 6.13-7.13 hours. The rate at which multifos product was being transferred into the building during the test period averaged 79.7 tons per hour and the building ventilation rate (natural ventilation) averaged 20,103 cubic feet per minute.

All field and analytical data sheets are included in the Appendix of this report as is a copy of the general test protocol.



FLUORIDE EMISSION TEST - RUN NO. 1 MULTIFOS MIXED FEED BUILDING IMC-AGRICO NEW WALES CHEMICAL COMPLEX APRIL 5, 2000

Sample					•									Total
	Area		(1)										l	Raw
•	Opening	Velocity	Volume	Velocity	Volume	Velocity	Volume	Velocity	Volume	Velocity	Volume	Velocity	Volume	Volume
	(ft ^ 2)	(ft/min)	(ft ^ 3)	(ft/min)	(ft^3)	(ft/min)	(ft ^ 3)	(ft/min)	(ft ^ 3)	(ft/min)	(ft ^ 3)	(ft/min)	(ft ^ 3)	(ft ^ 3)
Roof									· · ·					
1	22.5	60	74385	50	47138	20	27945	_ 30	- 53528	60	90720	40	101520	395235
2	22.5	60	74385	90	84848	150	209588	150	267638	230	347760	190	482220	1466438
3	22.5	30	37193	70	65993	30	41918	20	35685	.100	151200	90	228420	560408
4	22.5	20	24795	20	18855	90	125753	. 150	267638	75	113400	75	190350	740790
5	22.5	120	148770	20	18855	30	41918	170	303323	130	196560	130	329940	1039365
Ground								l .	•					
6	80	120	528960	75	251400	30	149040	110	697840	80	430080	175	1579200	3636520
8	84	100	462840	-195	-686322	-190	-991116	-70	-466284	85	479808	60	568512	-632562
9	132	-95	-690954	75	414810	-200	-1639440	-172	-1800427	-102	-904781	-157	-2337667	-6958459
10	132	-95	-690954	75	414810	-200	-1639440	-172	-1800427	-102	-904781	-157	-2337667	-6958459
Volume	Balance (ft	^3)	-30581		630386		-3673836		-2441488.4		-33.6		-1195172	-6710725(2
Time Ste	ep (min)		55.1		41.9		62.1		79.3		67.2		112.8	418.4

Sample			Initial	Final	Average	Start	End	Total	Sampled	Sample	Fluoride	Total	Total
	Sampled E	missions	Flow	Flow	Flow	time	time	time	Volume	Volume	Sample	Adjusted	Emissions
	mg F	lb F	(I/min)	(I/min)	(I/min)			(min)	(I)	(ft ^ 3)	(lb/ft^3)	Volume	(lb)
	(3)							(4)				(ft^3)	(5)
Roof													
1	0.095	2.09E-07	0.95	0.99	0.97	9:49	16:21	392.00	380.24	13.43	1.56E-08	395235	0.00616
2	0.097	2.15E-07	0.93	0.92	0.93	9:40	16:21	401.00	370.93	13.10	1.64E-08	1466438	0.02404
3	0.490	1.08E-06	0.93	0.93	0.93 ⁻	9:28	16:21	413.00	384.09	13.56	7.96E-08	560408	0.04463
4	0.122	2.68E-07	0.96	0.89	0.93	9:13	16:21	428.00	395.90	13.98	1.92E-08	740790	0.01421
5	0.174	3.84E-07	0.94	0.93	0.94	9:13	16:21	428.00	400.18	14.13	2.72E-08	1039365	0.02823
Ground			ı										
6	0.069	1.52E-07	0.90	0.93	0.92	9:19	16:21	422.00	386.13	13.64	1.12E-08	3636520	0.04065
8	0.019	4.25E-08	0.98	1.00	0.99	9:13	16:21	428.00	423.72	14.96	2.84E-09	-632562	-0.00180
9	0.009	2.03E-08	0.90	0.89	0.90	9:14	16:21	427.00	382.17	13.50	1.50E-09	-3603096.5	-0.00541
10	0.009	1.97E-08	0.99	1.03	1.01	9:14	16:21	427.00	431.27	15.23	1.29E-09	-3603096.5	-0.00465
Total						n, t		418.44				0	0.1461

- Area x Velocity x Time Step; (+) Volume is flow out of building and (-) Volume is flow into building.
 Imbalance into building is result of fluctuating flow direction of Areas 9 and 10.
 DI water blank corrected.
 Time may be adjusted for sampler downtime
 Total fluoride emissions for entire test period.

FLUORIDE EMISSION TEST - RUN NO. 2 MULTIFOS MIXED FEED BUILDING IMC-AGRICO NEW WALES CHEMICAL COMPLEX **APRIL 6, 2000**

Sample									,				_	Total
	Area		(1)						7		•			Raw
	Opening	Velocity	Volume	Velocity	Volume	Velocity	Volume	Velocity	Volume	Velocity	Volume	Velocity	Volume	Volume
	(ft ^ 2)	(ft/min)	(ft ^ 3)	(ft/min)	(ft ^ 3)	(ft/min)	(ft ^ 3)	(ft/min)	(ft ^ 3)	(ft/min)	(ft ^ 3)	(ft/min)	(ft ^ 3)	(ft ^ 3)
Roof			_										–	
1	22.5	90	92138	30	43538	40	63000	100	163125	. 20	13950	20	38025	413775
2	22.5	30	30713	50	72563	20	31500	75	122344	50	34875	30	57038	349031
3	22.5	60	61425	50	72563	40	63000	30	48938	60	41850	20	38025	325800
4	22.5	25	25594	40	58050	0	0	50	81563	20	13950	20	38025	217181
5	22.5	110	112613	120	174150	40	63000	70	114188	30	20925	20	38025	522900
Ground														
6	80	90	327600	18	92880	40	224000	26	150800	35	86800	15	101400	983480
8	84	-70	-267540	-125	-677250	-250	-1470000	-273	-1662570	-340	-885360	-245	-1739010	-6701730
9	132	-35	-210210	-45	-383130	-70	-646800	60	574200	35	143220	70	780780	258060
10	132	, 35	-210210	-45	-383130	-70	-646800	60	574200	35	143220	70	780780	258060
Volume	Balance (f	t^3)	-37879		-929768		-2319100		166786.25		-386570		133088	-3373443(2)
Time Ste	ep (min.)		45.5		64.5		70		72.5		31		84.5	368

Sample			Initial	Final	Average	Start	End	Total	Sampled	Sample	Fluoride	Total	Total
	Sampled	Emissions	Flow	Flow	Flow	time	time	time	Volume	Volume	Sample	Adjusted	Emissions
	· mg F	lb F	(I/min)	(I/min)	(l/min)			(min)	(I)	(ft ^ 3)	(lb/ft^3)	Volume	(lb)
	(3)						,	(4)				(ft ^ 3)	(5)
Roof													
1	0.104	2.29E-07	0.99	0.97	0.98	8:05	14:46	368.00	360.64	12.74	1.80E-08	413775	0.00745
2	0.066	1.45E-07	0.92	0.99	0.96	8:05	14:46	368.00	351.44	12.41	1.16E-08	349031	0.00407
3	0.087	1.91E-07	0.95	0.92	0,94	8:05	14:46	368.00	344.08	12.15	1.57E-08	325800	0.00511
4	0.147	3.24E-07	0.86	0.85	0.86	8:05	14:46	368.00	314.64	11.11	2.91E-08	217181	0.00633
5	0.178	3.92E-07	0.95	0.91	0.93	8:05	14:46	368.00	342.24	12.09	3.25E-08	522900	0.01698
Ground												l	
6	0.037	8.16E-08	0.90	0.92	0.91	8:05	14:46	368.00	334.88	11.83	6.90E-09	983480	0.00679
8	0.013	2.92E-08	0.97	0.99	0.98	8:05	14:46	368.00	360.64	12.74	2.30E-09	-6701730	-0.01538
9	0.009	1.98E-08	0.91	0.90	0.91	8:05	14:46	368.00	333.04	11.76	1.69E-09	1944781.25	0.00328
10	0.012	2.66E-08	1.00	1.04	1.02	8:05	14:46	368.00	375.36	13.26	2.01E-09	1944781.25	0.00391
Total								368.00				0	0.0385

Area x Velocity x Time Step; (+) Volume is flow out of building and (-) Volume is flow into building.
 Imbalance into building is result of fluctuating flow direction of Areas 9 and 10.
 DI water blank corrected.

⁽⁴⁾ Time may be adjusted for sampler downtime(5) Total fluoride emissions for entire test period.

FLUORIDE EMISSION TEST - RUN NO. 3 MULTIFOS MIXED FEED BUILDING IMC-AGRICO NEW WALES CHEMICAL COMPLEX **APRIL 7, 2000**

Sample		•		_										Total
	Area		(1)								İ			Raw
	Openin	Velocity	Volume	Volume										
	(ft ^ 2)	(ft/min)	(ft ^ 3)	(ft/min)	(ft ^ 3)	(ft/min)	(ft^3)	(ft/min)	(ft ^ 3)	(ft/min)	(ft ^ 3)	(ft/min)	(ft ^ 3)	(ft ^ 3)
Roof														
1	22.5	30	29363	30	57038	80	90000	60	96525	¨ 120	198450	20	47250	518625
2	22.5	30	29363	30	57038	60	67500	70	112613	150	248063	50	118125	632700
3	22.5	30	29363	20	38025	75	84375	60	96525	20	33075	50	118125	399488
4	22.5	30	29363	20	38025	50	56250	30	48263	90	148838	80	189000	509738
5	22.5	80	78300	20	38025	50	56250	120	193050	100	165375	150	354375	885375
Ground]		1								
6	80	30	104400	10	67600	10	40000	30	171600	25	147000	60	504000	1034600
8	84	-280	-1023120	-260	-1845480	-300	-1260000	-230	-1381380	-270	-1666980	-320	-2822400	-9999360
9	132	-30	-172260	-40	-446160	-240	-1584000	50	471900	45	436590	33	457380	-836550
10	132	-30	-172260	-40	-446160	-240	-1584000	50	471900	45	436590	33	457380	-836550
Volume	Balance (ft ^ 3)	-1067490		-2442050		-4033625		280995		147000		-576765	-7691935(2
Time Ste	ep (min.)		43.5		84.5		50.0		71.5		73.5		105.0	428.0

Sample			Initial	Final	Average	Start	End	Total	Sampled	Sample	Fluoride	Total ·	Total
	Sampled	Emissions	Flow	Flow	Flow	time	time	time	Volume	Volume	Sample	Adjusted	Emissions
	mg F	lb F	(I/min)	(I/min)	(I/min)			(min)	(1)	(ft ^ 3)	(lb/ft^3)	Volume	(ib)
	(3)							(4)				(ft ^ 3)	(5)
Roof													
1	0.084	1.86E-07	0.99	0.98	0.99	8:04	17:18	428.00	421.58	14.89	1.25E-08	518625	0.00647
2	0.113	2.49E-07	0.92	0.88	0.90	8:04	17:18	428.00	385.20	13.60	1.83E-08	632700	0.01157
3	0.115	2.54E-07	0.92	0.90	0.91	8:04	17:18	428.00	389.48	13.75	1.85E-08	399488	0.00739
4	0.086	1.90E-07	0.94	0.95	0.95	8:04	17:18	428.00	404.46	14.28	1.33E-08	509738	0.00678
5	0.079	1.75E-07	0.89	0.88	0.89	8:04	17:18	428.00	378.78	13.38	1.31E-08	885375	0.01158
Ground	ļ								,	ļ			
6	0.050	1.10E-07	0.98	0.91	0.95	8:04	17:18	428.00	404.46	14.28	7.69E-09	1034600	0.00795
8	0.024	5.26E-08	0.98	0.96		8:04.	17:18	428.00	415.16	14.66	3.59E-09	-9999360	-0.03588
9	0.020	4.43E-08	0.89	0.88	0.89	8:04	17:18	428.00	378.78	13.38	3.31E-09	3009418	0.00996
10	0.022	4.91E-08	1.00	0.98	0.99	8:04	17:18	428.00	423.72	14.96	3.28E-09	3009418	0.00987
Total								428.00				0	0.0357

Area x Velocity x Time Step; (+) Volume is flow out of building and (-) Volume is flow into building.
 Imbalance into building is result of fluctuating flow direction of Areas 9 and 10.
 DI water blank corrected.

 ⁽⁴⁾ Time may be adjusted for sampler downtime
 (5) Total fluoride emissions for entire test period.

TABLE 4

FLUORIDE EMISSION SUMMARY IMC-AGRICO MULTIFOS MULTI-FEED STORAGE BUILDING NEW WALES CHEMICAL COMPLEX APRIL 5-7, 2000

Run		Sample	Period		Product Transfer Rate to Bldg		oride sions	Avg. Bldg Ventilation Rate
	Start	End	Time (min)	Time (hr)	(tph)	(lb/test)	(lb/hr)	(cfm)
1	0922	1621	418	6.97	75.4	0.1461	0.0210	18,735
2	0805	1446	368	6.13	84.0	0.0385	0.0063	18,211
3	0804	1718	428	7.13	79.7	0.0357	0.0050	23,363
Avg				6.74	79.7		0.0108	20,103

APPENDIX

GENERAL TEST PROTOCOL

SAMPLER LOCATIONS

FIELD AND ANALYTICAL DATA SHEETS

PLANT OPERATING DATA

PROJECT PARTICIPANTS

GENERAL TEST PROTOCOL

METHOD FOR DETERMINATION OF GASEOUS AND WATER SOLUBLE FLUORIDE EMISSIONS FROM A GTSP STORAGE BUILDING

1. Principle and Applicability

- 1.1 Principle. Gaseous and water soluble fluorides are withdrawn from various predetermined sample points in the roof monitor and leeward eave vents using several modified EPA Method 13B sampling trains. The concentration of fluoride captured in the sample line, impinger water and filter of each sample train is then determined, using a specific ion electrode.
- 1.2 Applicability. This method may be used, subject to Department approval and possible site-specific modifications, for determining gaseous and water soluble fluoride emissions from GTSP storage buildings and similar structures.

2. Apparatus

- 2.1 Sampling Train. Sampling equipment shall meet the specifications listed for Method 13B of 40CFR60, Appendix A with several exceptions as follows. See Figure 1 for sampling train schematic.
- 2.1.1 Sample Inlet. The standard sampling nozzle and probe of the 13B sample train shall be replaced with a sample inlet constructed of a material inert to fluoride. The inlet shall consist of an approximate 65 mm diameter funnel fitted into the free end of the sample line. The funnel shall be inverted (facing downward) to sample the area of maximum flow out of the building at the sampling site.
- 2.1.2 Sampling Line. The sampling line connecting the sample inlet to the impinger assembly shall be leak free and of a material inert to fluoride.
- 2.1.3 Impinger Assembly. The impinger train shall consist of three (3) midget impingers followed by a dry trap. All three impingers will be of the standard design with standard tips. Each of the three (3) impingers will be charged with 15 ml of distilled-deionized water. The dry trap shall be empty.

Alternatively, the impinger assembly can consist of two polypropylene bubblers followed by a dry trap. Each of the two bubblers will be filled with 50 ml of deionized-distilled water. The polypropylene tube shall be 32 mm in diameter and 164 mm; long. The cap of the absorber must be polypropylene cap with two ports. A glass impinger

stem, 6 mm in diameter and 158 mm long, is inserted into one port of the absorber cap. The tip of the stem is tapered, as is the tip of the standard midget impinger. Clearance from the bottom of the absorber to the tip of the stem must be 6 \pm 2 mm.

After the first run, specific ion electrode readings will be taken on the three impingers from the sampling train at the site that would reasonably be expected to have the highest fluoride concentrations. If over five percent of the captured fluoride is in the third impinger (or second bubbler), the volume of water in all the sampling equipment will be increased by an amount specified by the Department representative before the second run begins.

- 2.1.4 Filter. A Whatman No. 1 or comparable filter will be located behind the impinger assembly.
- 2.1.5 Metering System. The metering system as described in EPA Method 13B can be replaced with a critical flow device and a vacuum pump equipped with a vacuum gage that would allow a constant sampling rate of 1.0 liters per minute. All other necessary equipment will be as described in EPA Method 13B.
- 2.1.6 Hot-Wire Anemometers. Hot-wire anemometers will be used to measure air velocities in the building. The anemometers shall be calibrated in a manner acceptable to the Department prior to the test. The calibration range shall include the expected velocities within the building, i.e., 0.2-10.0 fps.
- 2.1.7 Flow Direction Indicator. Since the hot-wire anemometers that will be used to measure velocities cannot measure the direction of those velocities, a device must be used to indicate flow directions at all of the designated velocity measurement points. The type of device used will be at the discretion of the company, subject to approval by the Department.
- 2.1.8 Wind Speed and Direction Indicator. A wind speed and direction indicator will be located in the vicinity of the GTSP storage building during the sampling effort.
- 2.2 Sampling Recovery.
- 2.2.1 Probe Brushes and Extensions. Probe brushes and extensions will be of a material inert to fluoride.
- 2.2.2 Sample Containers. All containers used to recover wash and impinger solutions will be of a polyethylene material inert to fluoride. Containers will be washed with HCl prior to use.
- 2.3 Analysis. All analytical equipment will be as described in Method 13B with the exception that all apparatus associated with he fusion and distillation steps will be eliminated.

3. Reagents

Reagents for sampling and analysis will be the same as those described in EPA Method 13B with the exception that all reagents associated with the fusion and distillation steps will be eliminated.

4. Procedure

4.1 Pretest Preparation

- 4.1.1 Plant. Prior to and during all test runs, the GTSP plant will be operated within 10 percent of its permitted capacity. The storage building will be filled to at least 10 percent capacity, of which 20 percent shall be freshly manufactured GTSP (produced no more than five days prior to the test).
- 4.1.2 Storage Building. Prior to and during the test, all openings, with the exception of the roof monitor, eave vent, end wall vents and other designed openings in the building, will be sealed.
- 4.1.3 Outloading. During one sampling run (8 hours), normal outloading of the GTSP product shall occur from the GTSP storage building.
- 4.1.4 Product Drop point. For the duration of the test periods, the drop point of GTSP into the storage building will be as close as possible to where the product enters the building.
- 4.1.5 Sampling Train. Clean all impingers as described in EPA Method 13B. Charge all impingers with the appropriate amounts of distilled-deionized water. Perform all necessary calibrations as described in Method 13B. If a critical flow device is used to control the flow through the sampling train, it shall be calibrated prior to the test with a standard dry gas meter or mass flow meter.

4.2 Preliminary Determinations

- 4.2.1 Weather Conditions. Assess what the probable weather conditions will be during the test effort. If they are less than ideal, the test may be postponed at the option of the Department or the Company.
- 4.2.2 Sampling Locations. A minimum of one sampler for each 100 feet of building length will be located in the roof monitor. Additionally, one sampler shall be as close as physically practical to the product drop point during the test. The samplers shall be located beginning 50 feet from each end wall and at approximate 100 foot intervals in between. The extra sampler should be located over the GTSP drop point (see Figure 2).

A minimum of three (3) samplers shall be located along the sidewall building vent to obtain a sample of the air leaving the leeward side of the building. The sampling system inlet shall be at the same elevation as the vent opening and between 1 and 2 feet inside the building. The locations of the samplers along the sidewall building vent shall be determined just prior to each test run.

At the option of the Company, a single sampler may be used to measure an upwind or background fluoride concentration. The sampling point shall be no closer than 100 feet to any part of the storage building, including the railcar loading shelter.

4.2.3 Leak Checks. The sampling train shall be checked for leaks before and after each run as per EPA Method 13B.

4.3 Sampling

4.3.1 Velocity Determinations. For the determination of air flow in the roof monitor vent, velocity measurements shall be made in line with each sampling point along the roof monitor vent and 1 to 1.5 feet below the level of the walkway. An average velocity shall be determined, either by taking four readings across the vent each time or by characterizing the flow pattern across the vent at each sampling point and choosing a point of average velocity. The flow pattern should be defined prior to the start of each run and verified at the end. If the Company wishes to use the single, average point option, it shall conduct a one or two day study prior to the actual test to demonstrate that a single point can be used to indicate an average velocity during the entire run.

The velocity measurements at the eave vents shall be made in centroid of the vent opening. Velocity measurements shall be made at least at each sampling point, but no more than 100 feet apart. The velocity reading shall be made for at least 30 seconds at each point and visually averaged by the operator. A velocity measurement shall be made at each point immediately prior to the start of a test run and approximately every hour thereafter until the end of the run. In addition, the flow direction shall be determined when and where each velocity measurement is made.

4.3.2 Sampling Data. Prior to the start of the test, at 60-minute intervals during each test run and at the conclusion of each test run, the velocity, flow direction, DGM reading, temperature and all other pertinent data for each sampling point will be recorded on field data sheets. If a critical flow device is used in lieu of a DGM, the pressure differential across the device shall be recorded at the 60-minute intervals and at the end of the test period. A final flow check shall be made on with a standard dry gas meter or a mass flow meter. The flow rate through the critical flow device test period shall be the average of the pre-test and post-test flow rates.

- 4.3.3 Test Duration. A test shall consist of three runs. Each run shall be a minimum of eight hours. One of the three 8-hour test runs shall coincide with the shift during which GTSP is being loaded into the railcars. The Company shall arrange to load out at a maximum rate of the 8-hour period.
- 4.3.4 Weather Data. Record the ambient wind speed and direction at hourly intervals during the testing period. If unfavorable weather conditions arise, the test may be halted and/or postponed at the option of the Department representative or the Company.
- 4.3.5 Plant Stoppages. If the supply of GTSP to the building is reduced or halted during the testing for approximately 15 minutes or more, the testing should be stopped. Sampling should be restarted 15 minutes after the GTSP supply has reached its previous rate to allow the emissions to maximize. The time for that run must be extended by the length of the sampling train downtime.

5. <u>Sample Recovery</u>

- 5.1 Sample Inlet and Sample Line. At the conclusion of each sampling run, and before the sample pump is turned off, the sample line shall be elevated above the impingers in such a way that particulate matter collected in the sample inlet and sample line cannot be lost. The interior surfaces of each sample inlet and sample line shall then be brushed and rinsed at least three times with distilled-deionized water at per EPA Method 13B, Section 7.2.1. The washings shall be added to a clean polyethylene container.
- 5.2 Impingers. The solutions from all three impingers will be added to the sample inlet and sample line washings with the exception of the sample train expected to contain the highest fluoride concentration. The impingers and connecting glassware shall be rinsed three (3) times and the washings added to the existing sample container. The filter following the impingers shall be recovered and added to the existing sample container.
- 5.2.1 Highest Fluoride Sample Train. At the conclusion of each sample run the sample inlet and sample line washings, first and second impinger solutions and washings, and the third impinger solution and washings shall be placed in three (3) separate containers. The filter following the impingers shall be combined with the sample from the third impinger. After fluoride analysis of the impinger solutions, the washings may be combined into one (1) container.
- 5.3 Prior to analysis, all washings must be measured volumetrically.

6. Analysis

Analysis of all fluoride samples will be as described in EPA Method 13B with the following exceptions:

- 1. The fusion and distillation steps will be eliminated.
- 2. The impinger solution may be divided into two or three portions if requested by the Department: one for Company analysis, one for Department analysis and possibly one sealed as a reference sample. The Company's sample will contain the filter.

7. Calculations

For calculating the mass emission rate, each sampling point shall be considered to represent the emissions from a specific area and should be centered in that area. The mass rate from each area will be the measured concentration times the measured flow rate (area represented by monitor times average measured velocity for area). Then the total mass rate for the building will be the sum of all the individual mass rates, as follows:

e = $C_X Q_X$; where e = average emission rate from one area, lbs/hr C_X = average concentration from one area, lbs/dscf Q_X = average volumetric flow rate from one area, dscf/hr

then:

$$E = e_1 + e_2 + ... + e_n$$

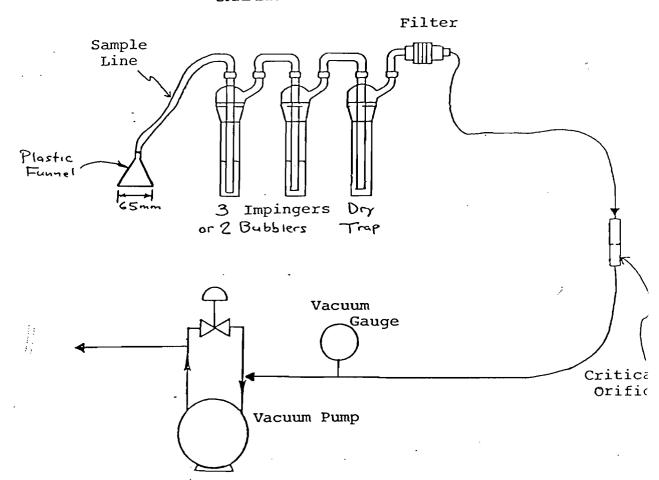
where E = total mass rate from the building, lb/hr, for the run. The test results will be the average emissions, lb/hr, for the three runs.

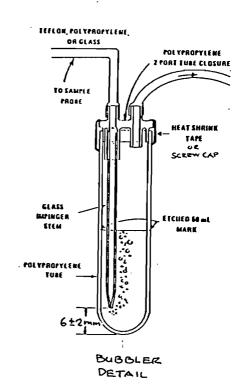
8. Test Report

The test report shall include all applicable sections described in Subsection 17-2.700(7), Florida Administrative Code (FAC) and all other pertinent data collected during the test.

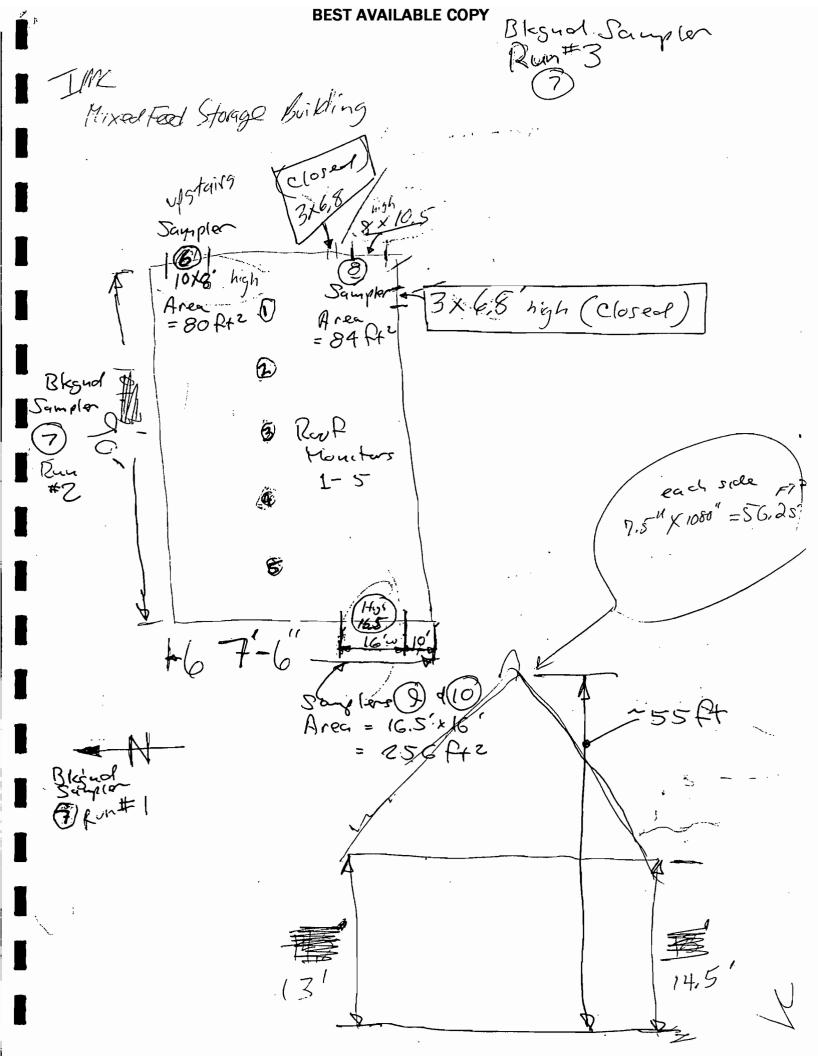
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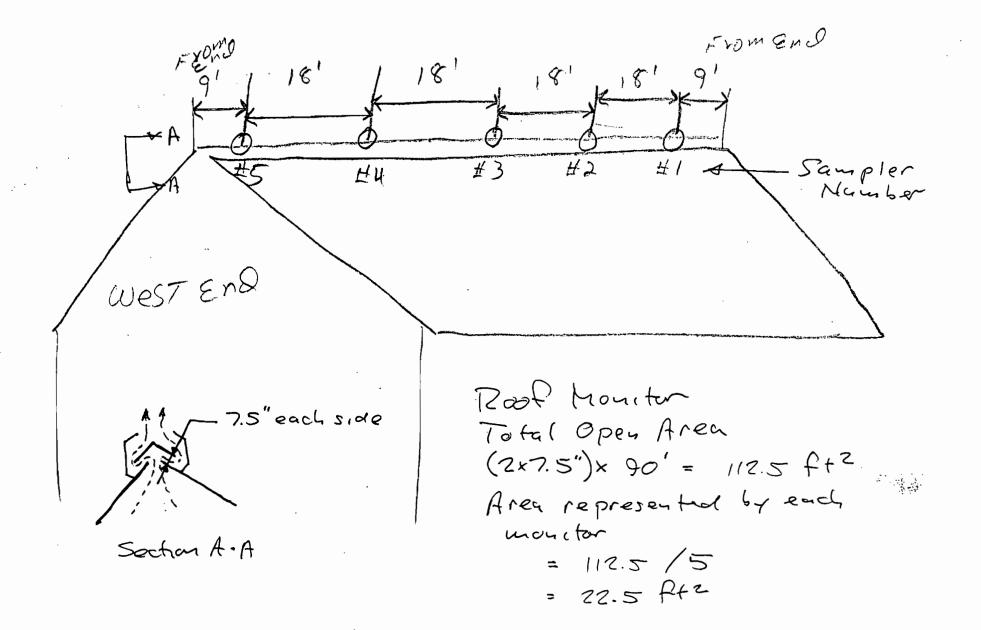
Figure 1
SAMPLING TRAIN





SAMPLER LOCATIONS





FIELD AND ANALYTIAL DATA SHEETS

RUN NO. 1

COMPANY: IMC-Agrico
LOCATION: NEW WALES
PLANT: MIXED FEED
DATE: 04/05/00

RUN: ONE

FLUORIDE ANALYSIS

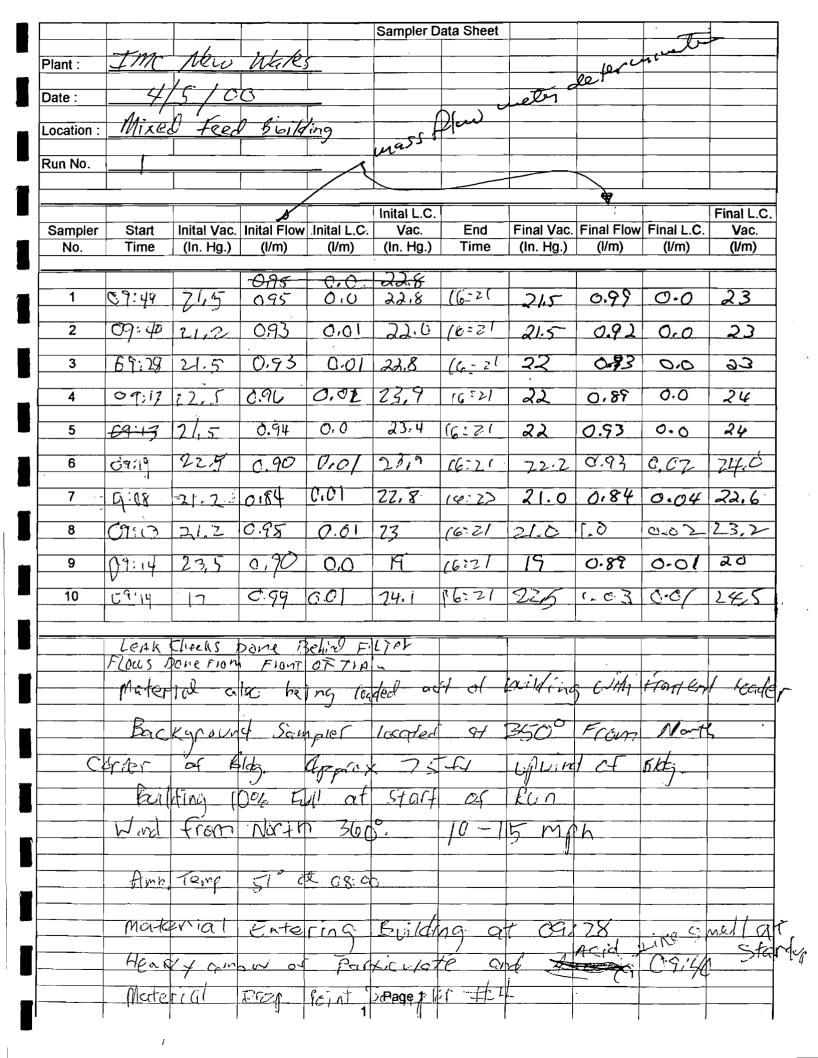
PERFORMED BY: DAVID CARROLL

SAMPLE	TOTAL VOLUME	РРМ	TOTAL MG. F
1	189	0.54	0.102
2	194	0.54	0.105
3	199	2.5	0.498
4.A.	56	1.9	0.106)
4.B.	141	0.12	0.017 g total
4.C.	55	0.076	0.004
5	181	1	0.181
6	191	0.4	0.076
7	168	0.041	0.007
8	158	0.16	0.025
9	184	0.088	0.016
10	182	0.087	0.016
DI BLANK		0.038	

Total mg. values not corrected for Blank.

Analysis performed in accordance with method 13B.

Equipment: Orion Expandable ionanalyzer EA 920 with fluoride electrode.



		SAMPLER DATA SHEET					
			le Volu				
PLANT:			15 410				
DATE:			7				
LOCATION:							
RUN NO:	1						
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3		09:31	30	OUT		1
4		09:19	20	OUT		
5		9:04	120	OUT		8088 vènt
6	_	9:40	120	OUT		
7	GKG.	10:73	700	30° f	m Morth	
8		9:45	001	OUT		
9	79:48	9:45	95	IN	; ·	
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			AIR VELOCITY DATA SHEET				
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RUN NO							
SAMPLER		TIME			APPROACH ANGLE		
1		10:44	(FT/MIN)	(IN OR OUT	(DEGREES)		
2		10:48	90	CUT			
3		10.53	70	Cost	5625		
4		16:56	70	007			
5		(1:01	20-	0 07			
6		10:58	75	0121	6060		
7	DK-6	4 - 0	(_)				
8		10:53	195	IN	16380		
9	-	10.37		DIV			
10	7	10:51	75	CUT	19,800		
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Run 1

Shoke Candle #1 -11:15

In from reader dear.
Fising vertically to roof monitor and upstairs

Gening no visible smoke heading to wards hopper gening.

#Z 13:25

SMIKE CANDRE (it near front end looder door Smoke wa Bushed in bottom section of looder door and came back out the top section of the opaning.

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PLANT							
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6		17:00	(10.	GUT	8800
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TN OUT 130-220 (x 6 27,720 308525

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2		10:48	21,7		11-50	215:
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6		10:28	Z35		16:52	235
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.7		10:22	2115		11:32	21,5
			,			
8		10:26	21.5		11:49	21.5
		·				<u> </u>
9		10.24	[7,8]		11:34	17.7
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		SAMPLER			
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PLANT:					,
DATE:					
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1	(2:4)	24.5	-		
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5	1303	Z2.5			
6	(2:58	72.5			
7	12:42	21.5			
8	12:50	21.5			
9	12:44	17.5			
10	12:45	22,5			
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Project Number	
Project Name	IMC New Wales
Sample Location Milbe	erry New Water, FL
	Ambient Sampling Feed Builling
Sample Identification	. Remarks
Cont. 1	Samples # 1 Imc
1 2 :	2
3	3
4 A+B+C	4 3 containers
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	[6]
7 .	7
. 8.	8
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16	V. 10
Sampled By: (Signature)	Blanks Lane Date: 4/5/00 Time: See DATA SHEETS
Relinquished By: (Sign)	Oate:Time:
	Culsel_Date: 4/5/00_Time:
Relinquished By: (Sign)	Date:Time:
Received By: (Sign)	Oate:Time:
Relinquished By: (Sign)	Oate:Time:
Received By Lab: (Sign)	Oate:Time:
	Fed Express Bus
Snipping Bill Number:	

CHAIN OF CUSTODY RECORD

RUN NO. 2

1

COMPANY:

IMC-Agrico

LOCATION:

NEW WALES

PLANT:

MIXED FEED

DATE:

04/06/00

RUN:

TWO

FLUORIDE ANALYSIS

PERFORMED BY: DAVID CARROLL

	TOTAL		TOTAL
SAMPLE	VOLUME	PPM	MG. F
1	183	0.59	0.108
2	169	0.41	0.069
3 - <i>i</i>	181	0.49	0.089
4.A.	102	1.3	0.133
4.B.	141	0.13	0.018
4.C.	81	0.038	0.003
5	182	1	0.182
6	178	0.23	0.041
7	183	0.03	0.005
8	170	. 0.1	0.017
9	180	0.072	0.013
. 10	198	0.083	0.016
DI BLANK		0.022	

Total mg. values not corrected for Blank.

Analysis performed in accordance with method 13B.

Equipment: Expandable ionanalyzer EA 920 with fluoride electrode.

_					BEST A	VAILABL	E COPY				
						Sampler D	ata Sheet				
	Diant :	1 TAK	Nen	Wate	<u></u>		· -				
_	Plant :		1 1		/					00	
	Date :	4/6	12000						70	11	
-		M;	Lat Es	al Ria				700			-
	Location :	741	XU4 PE	E4 5 K	E Ja			Jal J			
	D - No		<u> </u>								
	Run No.		<u> </u>		<u></u>						
					1		_				
						Inital L.C.					Final L.C.
_	Sampler	Start	Inital Vac.	Inital Flow		Vac.	End		Final Flow		Vac.
	No.	Time	(In. Hg.)	(l/m)	(l/m)	(In. Hg.)	Time	(In. Hg.)	(l/m)	(l/m)	(l/m)
		T_	1	<u> </u>						0.0	23
	1	F. 05	21.5	0.99	0.0	23.0	14:46	21.5	0,97	0.0	23.0
							1 12 192		_		
	2		21.5	0.97	0.0	240			0.89	0,0	23,0
	3	 	21.5	0.95	0.0	23,0	 	224	0.92	0,6	23.0
			מוני)	0.13	0.0	2310	 	22.0	. O . IX_		× 3_0
_	4		22.0	0.86	0.0	24.0		22.5	0.85	0,0	24.0
			0.6.3	***		33.2			AC /	<u> </u>	
_	5	 	22,0	0.75	0,0	23.5	_	22.0	0.91	0.0	23.5
	6		220	0.90	0.0	24.0		22,5	0.97	0.00	24,5
					,	24	V				
_	7	98-07	21.5	0.82	0.0	23,0	14:51	21,7	051	0,01)	23.3
	8	9:05	21.0	098	0.0	23.0	14:40	71,8	0.99	0.01	23,1
		1	2110		, ,		1		. ,		
	9		18.0	0.91	0.0	20.5	<u> </u>	18.8	0.90	0,01	20.4
	10		21.5	1.00	0.0	24.5		22,5	1011	0001	748
ŀ		A	~ 1'0	1.00	0,0	070	•	245	1,04	0707	24.0
									· ·		
		(6)		• • •	1 -1 -1	i		<u> </u>		7	
	X CX	60, 12	Kg Sar	mer	located		014	(α) Γ		V	<i>5/019.</i>
	Juckor Com		<u> </u>	1			,				
787	50×°	7	Of 8	Cint	Sample	· 77	-4				
			J		7	-1	7				
		Materi	i	100 1	. •	/= / /			3 1/6		
_ }		raier,	al a	150 p	ing	Caded	cut	<u>o+</u> /	Sicilding	with	FICAT
	4	ad loc	der	16					·		
-				2/	Loa	ter w	ories	TNI	7.1		
					-	1-1-		27. 10	701	15 U	ses .
						oca-ra	7 36,	(14: 00)	;		
ı		D 511	_	4-		_		_		-	
		Builtin	: X0		-ull con	Star	t of	Run.			
		, , , , , , , , , , , , , , , , , , , ,		-							
		win d	979	E 95+	3.	5 mph	. [
	<i>:</i>	Δ		`		,					
-	X	San A	y cour d	Sa	upler	SHU	t of	(e)	- 3 m	in So	Thay
	<u>, , , , , , , , , , , , , , , , , , , </u>	1000	, ,		<u>'</u>	Page 1		·			
	1	it our	16 6	$\sim m$	'' (h.an r	I Ho	· (is all	of not	u loca	ted 2	00
	•	200	"A FE	• > 10	V	171144	-1	~/ · Z	tost at	15/16	$\mathcal{L}(C, X)$

				DATA SHE		
DI 4417	- Voil 1		· / /	1e Vol4	mes	
PLANT DATE				1941d)		
LOCATION	1 11 7 7	Fied	solding			
RUN NO	: 2	I GCM	30100 reg			
		FINAL				
SAMPLER	MO	ISTURE &		<u>TO</u>	TAL VOLU	<u>ME</u>
1		104	Rinse 79		183	
2		106	63		169	
3		165	76		18	
4 🖟		104	37		[4]	-
5		105	77		182.	
6		104	74		178	
7		101	82		-	[8 3
8		[00	70		170	70
9		102	28		180	, , , , , , , , , , , , , , , , , , ,
10		101	9/		198	
HA			102		[02	
40			80		81	
		<u> </u>				
				4		

.

		AIR VELO	CITY DATA	SHEET
PLANT: INC			·	
DATE: 4//6/	90 - 10	(
OCATION: MIXED	Feel f	kG		
RUN NO: 2		1_/	-	
AMDI ED		1/7/ 00/70	DIDECTION	1 400004041411045
SAMPLER	TIME			APPROACH ANGLE
VAC	20:15	(FT/MIN)	(IN OR OUT	(DEGREES)
<u> </u>	08:10	70	CUT	
21,5	20 (2	3 4	<u> </u>	
2	08:13	30	and .	<u> </u>
3		/ 0	C(.7	
3	08:17	60	47/	27175
22	10.10	~ /	A	8212.5
	98:19	75	OUT	<)
22.5 5	60-21	1763	001	
2 3	08:24	(10)	W1	
•	a Cila C	1 12		4500
<u>6</u> 7ス,5	081 /L.	60	001	677
7			`	
212	10 AC	0 7 (1)		40 :
8 8	08.09	200		
	CX:14	65	INTO	J 5460
21.2	((0,0)1)	\$ 4		<u>. </u>
9 (%)	CS-12	80	1 1/ / dil	64 126
		180	LN109	21,126
10 /		200	7	
27,5			f	·
	_		6	
	·	Temp	5/0	
$\rightarrow i M D$	(cn1		-)6 F	
SMoka oxida	f	onton d	10 × / +1	7-4.
-Moke Candle	97 1	oader a	cor/1	n of bottom
of abor 9	1 dixxx	ut at	100	
- \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	prived			1 · · · · · · · · · · · · · · · · · · ·
Drop foint	1 10	Some	2r # 5	1 08:40

(8:16)

CUT 13672.5

			AIR VELO	CITY DATA	SHEET
PLAN	r: Inc				
DATE	<u> </u>	_			
LOCATIO	1/ 4/00	Feed k	1/6		
RUN NO		1 CEG 1	195		
			_		
SAMPLER		TIME	VELOCITY	DIRECTION	APPROACH ANGLE
VPC INF	6		(FT/MIN)	(IN OR OUT	(DEGREES)
1		09:34	30	001	
ンジブ		7			
2		09:31	50	00+	
ス ² 3					6575
		09:28	50	CUT	
22					
4		9-24	40	OUT	
75. 5		,			·
5		09:20	120	cut	
23					
		09:24	18	0117	1446
22,8	_		•		
7.	BKG	09:15	200		90°E
21.5					
8		09.23	250	INOU	1 /6,800
2112		.,		(
'9					:
18.5					
10	Ş	09-20	80	OUT	100
21.8			450	EN	18,480
			200	FN	1.
		09.9	0 1.60	0V1	
		. 7			
1					
	-				
					<u> </u> :
		(9:25)		
					1

TOF

			AIR VELC	CITY DATA	SHEET
DI ANT	- T 11.				
PLANT					
DATE	161				
CATION		Feed 1	ildci,		
RUN NO:	~~		<i>C'</i>		
MPLER		TIME	VELOCITY	DIRECTION	APPROACH ANGLE
		111415	(FT/MIN)	(IN OR OUT	
AC UH		(C)	40		(DEGNEES)
2211		10:50	40	OUT	,
2		16:25	70	DUT	
		10.25	120	(;c)	
7.2		10:26	40	001	> 3150 00+
22		10-25	40	001	/
4	· · · · · · · · · · · · · · · · · · ·	10.01	6	2	
		10:234			27 M = 26
5		10=26	40	cut	3200 ast
22		10-50	40	C 01	
6		(0=35	40	E30	
2.5		(0.52			
7		10.5	225		30°
21.8		10,0	000		
8		1(2: 31	250	+1	21,00 ia
21.2		10.51	12	7 /	21,00
9		15.27		^	
		11.61	10	INOT	(8480
8,5 10				TO F	() () ()
					<u> </u>
		sind	SEI	5000	11:21
	V	V - 1 - 2 - 1			
		·.			
					:
	/.		• ,		
	(10):25)	VI _	80 2	700
			1011		U071
			774	80	フィン

÷

			AIR VELO	CITY DATA S	SHEET
DI 411	.	-			
PLANT					
DATE			14		
LOCATION	7,11,702	Feed	Pld G		
RUN NO	· d		.,		
SAMPLER		TIME	VELOCITY	DIDECTION	APPROACH ANGLE
		TIME	(FT/MIN)	(IN OR OUT)	
VAC 149		66-57	(1 1/WIIN)	OUT	(DEGINEES)
27		(()	100	0(27	
		No sat	70	OUT	·
Gà		7	/~		·
3		11:41	36	2127	
<u> </u>		· · · · · · ·	13/		
4		4-36	50	COTT	
230					
23° 5		1(= 33	70	CUT	
22 4					
J· 6		11:43	260	DUI	2080
22,8		<u>'</u>		/	
7	٠	11:47	500	-	(50.0
8. C					·
8		[1:0f]	273	INE	22932 IN
7115 9				- 1	
9	\		(, , , ,	0.17	
18.5	/		(00 3M		2640 out
70	/		50 DC	YIN	2070
-65-4					
	1				
		_			
,		nbreht	10.	75°F	
		nuenz	TIEMP.	157	
			<u>. </u>		:
	ر استنسان استان استا ما ما م	(-)	IN	01	<u> </u>
	(11:4	``) \ \:	7927		- (

		_	AIR VELO	CITY DATA	SHEET
PLAN'					
DATE		<u> </u>			
LOCATIO		d Fred	Building		
RUN NC): 2				
CAMPLED		TIME	VELOCITY	DIDECTION	I ADDDOAGU ANGLE
SAMPLER	<u> </u>	TIME	VELOCITY	(IN OR OUT	APPROACH ANGLE (DEGREES)
1		(2:38	(FT/MIN)	GUT	(DEGREES)
<u>'</u>		2- 30	20	(, 0 1	
2		17-10	50	8.77	
		11-47	7 -		
3		12:48	(10)	047	
		70	W 5	\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\	7050
4		12.56	20 -	OUT	
		1			
5		12:57	30	007	
72' 6				~	
		(2:59.	35	COTTO	2808
22	·	· ·		1	
7		13:00	35-50		VAR,
21.8		75	- (>	<u>.</u>	
8		(2:510	340		28560
3(.5					·
185		17:50		OUTH	
10		(2.50	50		10 00
22.8			80	017/1	1 480 -
	D-	f	~ `	-01	
***	500	aker	111880	d some	tima between
	0 -			/ > - ///	
	1100	n an	9 (2)	30, al	50 mplers
	no [•.	<u> </u>	
	7707	YUV	MIMON	excep	107 (BKG)
					/
	Towa	(1es	fored	12:50	,
			M EL	1 . 7	
toyer	ott 6	13-0	restor		<u> </u>

(12:10)

25,330

			AIR VELO	CITY DATA	SHEET
PLANT:	Tarc		-		
DATE:		0			
LOCATION			Builda	10	
RUN NO:			- 01141/6	(1-)	
SAMPLER		TIME	VELOCITY	DIRECTION	APPROACH ANGLE
			(FT/MIN)	(IN OR OUT	(DEGREES)
1			20601	cut	
22					
2			50	OUT	
22					12475
3		13:54	70	00+) 2915
27					/
4		(3:51	20	OUT	
21°					
5		15:48	20	001	
27"					
6		J36 X	[5	CUTION	1200
23					
7			150-4	00	VAR EGST + SE
21.9					
8			245	BIN	20580
2/2					
9					
18,8		. , ,,,			
10		13:54	70	001/	100 SOM
			70	OVIA	1 (3, 7)V
		· · ·	70	007/	- /
			:		
		17: -7			
	\	13:53	j		

IN 2015 20580 ZZISS

CHAIN OF (70210D1	KELUKU
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Project Number	· ·
Project Name	IMC New Wales Facility
Sample Location	= Mulherry, FL
	Mixed Feed Building
Sample Identification	Remarks
<u> </u>	Sampler #1 Imp Catch Inc.
	42
3	#3
4A + 4B 4C	I14A, 4B+4C
5	tf 5
_6	+6
<u>7</u>	#7
. 8	# 8
9	# 9-
10	V . # 1/
Blank DI Hootfitter Sampled By: (Signature)	- 3/ Ad Date: C/6/60 Time: See DATHSHEETS
Relinquished By: (Sign)	Date:Time:
Received By: (Sign)	Oate:Time:
Relinquished By: (Sign)	Date:Time:
Received By: (Sign)	Date:Time:
Relinquished By: (Sign)	Date:Time:
	Oate:Time:
Sample Shipped VIA:UPS	_
Shipping Bill Number:	
	, WHITE SUSPENIES

RUN NO. 3

COMPANY: IMC-Agrico
LOCATION: NEW WALES
PLANT: MIXED FEED

DATE: 04/07/00 **RUN:** THREE

FLUORIDE ANALYSIS

PERFORMED BY: DAVID CARROLL

	TOTAL		TOTAL
SAMPLE	VOLUME	PPM	MG. F
1 ;	209	0.43	0.090
2	197	0.6	0.118
3 [/]	205	0.59	0.121
4.A.	199	0.46	0.092
4.B.			0.000
4.C.			0.000
5	197	0.43	0.085
6	176	0.31	0.055
7	176	0.094	0.017
8	194	0.15	0.029
9	195	0.13	0.025
10	197	0.14	0.028
DI BLANK		0.027	

Total mg. values not corrected for Blank.

Analysis performed in accordance with method 13B.

Equipment: Expandable ionanalyzer EA 920 with fluoride electrode.

					Sampler D	Data Sheet				
Plant :	IN	1C Nou	1 WALDS						_	
				· ·						
Date :	_4/	7/2000	1	<u> </u>						
Location :	· Ni Vo	I Faal	BURDA	<u></u>			<u> </u>			
Location .	1117 100		10000	<u> </u>		(2)				1.
Run No.	3	L				200	2	0,24		
								7		
)			7		Inital L.C.	Ţ	Т			Final L.C
Sampler	Start	Inital Vac.	Inital Flow	Inital I C	Vac.	End	Final Vac	Final Flow	Final I C	Vac.
No.	Time	(In. Hg.)	(l/m)	(l/m)	(In. Hg.)	Time	(In. Hg.)	(l/m)	(l/m)	(l/m)
	,	. 07			<u> </u>					1
	-0				23. U	17:18	01-	6.66		
1 (08:04	21.5	0.99	0.0	23.0	1	21,5	0.98	0.0	23.0
2	1	21.5	0.92	0.0	23.0		22,0	0.88	0,0	23.0
		<i>A</i> 113	0.10		20.0		adil.			<i>SC 7. C.</i>
3		22.0	0.9)	0.0	23.5		22.0	0.90	0.0	23,3
			. ~		5.7.			- M -	41 - 4	
4		2210	0.94	0.0	24.0		2215	0.75	0.0	24.0
5		210	065	a 6	24.0	 	22,0	0.88	9.0	7/1/0
	-17-	22.0	0.89	0.0	2410		Adio	~,,00	<u> </u>	24.0
6	#	225	0.89	0.0	24.0	V	22.5	0,91	0.02	24,4
)			/
7 (08:00	220	0.80	0.0	23.0	17:24	220	0.80	CLOZ	2=,1
		21.~	A 60			1-100	0 1 71	0.07	0.0	177 0
8	08:04	21.5	0.98	0.0	23.0	17:18	33.0	0.96	0.0	23,0
9		19.0	0.89	0.02	20.5		19,0	0.88	0.02	20.2
			- 01	0.00	~ ~ ~ ~ ~ ~					_
10	\bigvee	21.5	1,00	0.0	240		22.5	0.98	0.0	243
						•				
			T				_ ,	 		<u></u>
	3-251411	77-	Cuil	,						
	-	./		,						
Wind	from	SF	Q 174	- 0						
	r.	<u> </u>					·			
13ciCK 64	Con Hol	110 Course	356	ers	1 2 1	La lete				
Dood	Part	Scarald	~ + 1	ir e	> 0/68	2 C - F	£37.	.30		
7.9	10017		r # 4		i d			1		
1/200	Vy PC	rtico A	ate con	ď 🗯	The same	- Ferce	Smell	at f	aut Sta	7-19, C
	/	ĺ								
11(1-10-	riel p	eus 1	cr.dec c	007 A	y tren	teal.	londer			
		/			1					
17/40	7/	:e11-	Jania	0.54	10:0	4 5	AMIPL	ERS	Shirt	CFF
, <u>, , , , , , , , , , , , , , , , , , </u>		,				1	1 1/			
Cif	(0)	2					: :			
Rad	Control de	A	(:		40			· · · · · · · · · · · · · · · · · · ·		
1762	3 met	Scient	ling a	t 12	/.S					
			,,					,		
					Page 1					\\
				1						

Run # 3 Scaple Stat 8:04 Timo Stepa End 17:18 11:00 554.0 13:08 71.5 14:19.5 - 9 :9(60) = 73.7 15:33 105.0 917 428.0 --

SAMPLER DATA SHEET Sample Volune PLANT: IMC New 4/7/2000 Mixed DATE: LOCATION: **RUN NO:** MOISTURE GAIN TOTAL VOLUME SAMPLER Rinse

			AIR VELOCITY DATA SHEET				
PLAN			· ·				
DATE	Z-f-/	1/CC					
LOCATIO		Feed	Bldg.	_			
RUN NO): 3		_/				
0 4 1401 55		713.00	VEL 0017V	DIDECTION			
SAMPLER		TIME			APPROACH ANGLE		
			(FT/MIN)	(IN OR OUT) (DEGREES)		
22 2			30	(1)			
22		500.10	7.6	01:1			
32 -		CX-19	30.	CU7			
[2,5]		80. 1	2~	A 1 1-T			
		08:15	30·	CUT			
2/.5		O(2 1 '2	7~	611	17700		
72,5		C81Z	30	CUI			
5		CE:08	∇z	\$ out			
		~ ())	(A) (A)	D 001			
Z2-5		06.21.	7.	21/0	2400		
		00.61.	30	OUTIN	2400		
725		Der. 216	211	•	1350		
21.5	·	D8:44	270		(0 5		
8		68:18	7.60	TALE	97 to		
21.5		CV: 10/	-280)	LN	23520		
9		CB:15	1000	IN/CUT			
	<u> </u>	C (3.7)	100				
18.5 10			30	IN/OUT			
22.5				CITAN			
(4.)	Lorder		varies	as does	location & G		
	MARI	Doc	VUILEY	- S CICKS	iccation De		
					<u> </u>		
					<u> </u>		
		,					
				•			
	:				.,		
					:		
		8:13			· · · · · · · · · · · · · · · · · · ·		

Stent 8:04
8:13
9:22

196

(3:38

(33) 15:01 (64)

16:05

17:18 END

			AIR VELOCITY DATA SHEET				
PLAN			_		_		
DATE	1/0/00				-		
LOCATIO		d Feed	Bldg.				
RUN NO	: -3						
CAMPLED		TIBAT	VELOCITY	DIDECTION	ADDDOACH ANGLE		
SAMPLER		TIME	VELOCITY	(IN OR OUT	APPROACH ANGLE		
1		09:15	(FT/MIN)		(DEGREES)		
		C (* /_)	_><>	CUT			
<u>ZZ</u>		29:18	30	007			
		U 1. 0		,	2700		
27		023	70	CTIT			
				V 1 1			
22	,	09-25	7Ĉ	CUT			
22. 5 /		0 7. 28	20	CUT			
225							
6		0928	10	OVI	8-60		
23					δ.		
7		109:32	575		1/AR 135-175°		
22.8				,			
8		09:25	-260	IN	21840		
2115							
9 ′							
23,5							
10		19:23	300	OUTITA	(<u> </u>		
8.68			40	CUTTIN	9288		
		4	-40)	OUT/IN			
		*			i i		
_							
_					• •		
		•			,		
					:		

(9:22)

		AIR VELOCITY DATA SHEET				
PLANT: 1011		-				
DATE: 4/7/0	d					
OCATION: Mire	- Feed 1	Butteling				
RUN NO: 3	<u> </u>	-57 (3 17)				
AMPLER	TIME	VELOCITY	DIRECTIO	N APPROACE	ANGLE	
`			(IN OR OL			
1	(7) = CR		CUT			
715	1 6	2.30				
72	(2,4)	(()	OUT			
202	(~~	(s) (s)	0,1			
3	(2237	75	OUT	1900	2,5	
<u> </u>	1 (2. 2)	7~ ."	401			
4	17:20		GAT			
*	12:34	50	CCT			
5	15 0 5	<u> </u>	S			
3 3 ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~ ~	12:30	5°C	CUT			
6	1. (5) 1		S CIT	H		
6	12:13	f C .	OUT	Se 0	<u> </u>	
				—		
7		50		East		
2118						
8	12:40	-300	IN	326	80	
21.5	``]	· · · · · · · · · · · · · · · · · · ·		:		
9	C=24	30	グリブ			
(4,4	, 1	30	BUT			
10		40 -	OUT	880	O.	
7215		240)		,	1	
	(2:25	450	IN			
	ſ	225	IN.			
	1	150	IN			
		7			•	
	Airs	Wirlin	0.00	cail las	ecler do	
	P P	∞ 17	7 71:0	17/2 10	ecret se	
<u> </u>		<u> </u>				
				<u>.</u>		

(2:38)

			AIR VELOCITY DATA SHEET				
PLANT: -	IMC.						
DATE:	4/7/0						
LOCATION:	Plixe	=c Fee	I Elefo				
RUN NO:		3	J				
SAMPLER		TIME			APPROACH ANGLE		
				(IN OR OUT	(DEGREES)		
1		(30,32	al	OUT			
20.5							
2		13:35	70	CUT			
3			-				
3		15739	60	out	77650		
Ci_							
4		(3-42	30	CUT	2400		
5							
		() : 42	_l2c	CUT			
6		4: 16					
6		12-44	30	OUT	2460		
228							
		13:40	475		South		
218				TN			
8		13:40	- 230)	EUF	09320		
21,5		i * 5 5	11 -				
9		t z=3]	40	CUI	No. 7 a 4		
18.8			-50)		13200		
10			(c)				
22 4							
		-	<u> </u>				
			.				
					:		

13:38 GUT IN 23250 19320

	, , , , , , , , , , , , , , , , , , ,	AIR VELOCITY DATA SHEET				
-			ANT VELO		J. 1201	
PLANT	:IMC			 		
DATE		1)	_	 		
LOCATION		<i>!</i>	POLL	-	-	
		led Feed	1749.			
RUN NO	3		'			
0.4401.50		~:	VEL 0017/	DIDECTION	I ADDDO AOU ANOLE	
SAMPLER		TIME			APPROACH ANGLE	
		. 201	(FT/MIN)	(IN OR OUT	(DEGREES)	
1		1508	120	OU		
21.5						
2		5:04	it 150	an		
224						
3		15=00	20	Cit	10,800	
29				,]	/	
3 2 4		14-57	90	CUT		
22.15	-					
5		14:54	(CB.	GUT		
22.5			V = 4			
6		15-05	25	CUT	2000	
72		-J				
22 7		15:08	500		SE	
21.5						
8		15:03	27/2	TA	27680	
21.5				V		
9			(20/	OUT		
19)	14:59	(2/1)		11,800	
10		1-	7	1/	11/20	
22.5			13			
2017			+ 43			
-						
		_				
					<u>'</u>	
		11-		•		

24600

In 22680

		AIR VELOCITY DATA SHEET				
				_		
PLANT	: IMC					
DATE		0				
LOCATION	1: Mixey	Feet Bl	16.			
RUN NO			.)		_	
SAMPLER		TIME	VELOCITY	DIRECTION	APPROACH ANGLE	
			(FT/MIN)	(IN OR OUT,	(DEGREES)	
1		15:57	20	OUT		
265						
2 ′		TG:01	50	00+		
22		····				
22 3 22		16.05	50	001	7470	
22					/ '	
4		SE 258	20	017		
225						
5		16:11	15c	001		
6		12 6 12				
6		16-11	00	OUT	4800	
25.6		-		·		
7	\	16-51	500-700		South	
21,5		- 6/ 6				
8		16-00	-320)		IN 26880	
21.5		· .	<u> </u>	0.77	·	
9'		11-12	XQ	CUT	Och	
	_/	16-04	40			
10	-	·]				
62,7			+33)			
				-		
				++	-	
						
				:		
					:	
		- 6	j		<u> </u>	

260H 21870

CHAIN OF CUSTODY RECORD						
Project Number Project Name Sample Location	INC New Welles Mylberry FL Miced Ferd Bldg.					
Sample Identification 5 # [MC	Sample of # 1 Inc New holls					
3 4	3 4					
7	6					
9	9					
Sampled By: (Signature)	Date: 4/7/CC Time: See DATING HELTS					
Relinquished By: (Sign) Received By: (Sign)						
Relinquished By: (Sign)	Date:Time:					
Received By: (Sign)	Oate:Time:					
Relinquished By: (Sign)	Date:Time:					
Received By Lab: (Sign)	Oate:Time:					
Sample Shipped VIA:UPS	•					

PLANT OPERATING DATA

		Mixed feed operations during test				
		gal acid	tons soda ash	ton rock		
	4/5/00					_
	16:10	215380	12785	111682		
	9:15	199301	12757	111298		
	totals			384		
•	hours	1.63	specific gra	vity	tons	ton/hr
	6.916667	109.1595	28	384	521.1595	75.34837
	4/6/00					
	14:30	248594	12856	112464		
	10:00	236851	12831	112189		
	9:00	43.01	5.49	60.02	recorded a	s gpm and
	8:00	43	5.5		tph for 2 ho	
	totals	16903.6	35.99	395.02		
	hours	1.63	specific gra	vity	tons	ton/hr
	6.5	114.7577	35.99	395.02	545.7677	83.96426
	4/7/00		1		:	
	17:12	265077	12893	112857		
	12:10	start again				
	9:45	down				
	8:00	248594	12857	112464		
ĺ	totals	16483		393		
	hours	1.63	specific gra	vity	tons	ton/hr
Į	6.783333	111.9023	36	393	540.9023	79.73989
	the specific	gravity of t	he acid was	1.63-1.645	for the day	/S

:

Mixed Data Sheet for Building Test						
Date: 4/5/60 Operator: J. Nをいたのの						
·	7	otalizer Reading	s			
Time	Acid gpm	Soda Ash TPH	Rock TPH			
0915	199301	12757	111294			
1000	201417	12760	111350			
1100	202905	12763	111385			
1200	205528	12767	ンフャン			
1300	207420	12770	111491			
1400	210111	12776	111554			
1500	212074	12780	111604			
1600	214716	12784	111667			
1610	215380	12785	111682			

.

Mixed Data Sheet for Building Test					
Date:	4-6-00	Operator:	LUCAS		
		otalizer Reading	S		
Time	Acid gen	Soda Ash	Rock T.PH		
0800 -	43.00	5.50	60,00		
0900	43.04	5,49	60.02		
10:00	236851	12831	112189		
11:00	240270	12839	112269		
12:00	242331	12843	119318		
13:00	244867	12848	112376		
14:00	247343	12854	112435		
14:30	248594	12856	112464		

Did -

Mixe	ed Data Sheet	t for Building	Test	
Date:	4-7-200	O Operator:	B. W.7500	
	T	otalizer Reading		
Time	Acid g pm	Soda Ash	Rock #PH	
08:00	248594	12857	112464	·
09:10	251559	12864	112536	·
10:03:45	Down	1		
11:00	D	000 2 -		
TC:06	- 00	WP		·
12:10	252697	17866	112566	·
100013:20	255975	12872	112642	- Jour 7 mm
14:08	2572300	12876	112676	emy of the
15:00	259447	12881	112725	
16:00	262016	12887	112786	
17:12	265077	12893	112857	FINISH

PROJECT PARTICIPANTS

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KOOGLER & ASSOCIATES

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Project Advisor

Stephen S. Bell

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Glen A. Haven

Field Test Crew

IMC-AGRICO

C. D. Turley

