

Air



Urea Manufacture

Emission Test Report C. F. Industries Donaldsonville, Louisiana

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REPORT ON "B" GRANULATOR AND
SYNTHESIS TOWER EMISSIONS
AT THE CF INDUSTRIES, INC.,
UREA FERTILIZER PLANT
IN DONALDSONVILLE, LOUISIANA



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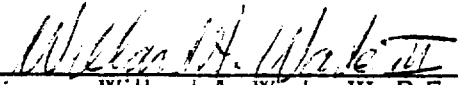
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March 1, 1980

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PREFACE

The work reported herein was conducted by personnel from TRC - Environmental Consultants, Inc. (TRC), the GCA/Technology Division (GCA), CF Industries, Inc. (CFI), Donaldsonville, Louisiana, and the U.S. Environmental Protection Agency (EPA).

The scope of work issued under EPA Contract No. 68-02-2820, Work Assignment No. 10 was under the supervision of the TRC Project Manager, Mr. Willard A. Wade, III. Mr. Reed W. Cass of TRC served as Project Engineer and was responsible for summarizing the test and analytical data in this report. Analysis of the samples was performed at the CFI Donaldsonville, Louisiana plant under the direction of Ms. Margaret Fox of TRC and at the TRC labs in Wethersfield, Connecticut under the direction of Ms. Joanne J. Marchese.

Mr. Stephen A. Capone and Mr. Timothy L. Curtin of GCA were responsible for monitoring the process operations during the testing program. GCA personnel were also responsible for writing the Process Description and Operation section along with Appendix I of this report.

Members of CF Industries, Inc., Donaldsonville, Louisiana whose assistance and guidance contributed greatly to the success of the test program include Mr. Thomas Carville, Senior Environmental Engineer, Mr. Stephen Thompson, Area Supervisor, and Mr. David Campo, Laboratory Supervisor.

Mr. Eric A. Noble, Office of Air Quality Planning and Standards, Industrial Studies Branch, EPA, served as Test Process Project Engineer and was responsible for coordinating the process operations monitoring.

Mr. Clyde E. Riley, Office of Air Quality Planning and Standards, Emission Measurement Branch, EPA, served as Technical Manager and was responsible for coordinating the emission test program.

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SECTION 1
INTRODUCTION

Section III of the Clean Air Act of 1970 charges the Administrator of the U.S. Environmental Protection Agency (EPA) with the responsibility of establishing Federal standards of performance for new stationary sources which may significantly contribute to air pollution. When promulgated, these standards of performance for new stationary sources (SPNSS) are to reflect the degree of emission limitation achievable through application of the best demonstrated emission control technology. To assemble this background information, EPA utilizes emission data obtained from controlled sources in the particular industry under consideration.

EPA's Office of Air Quality Planning and Standards (OAQPS) selected the CF Industries, Inc. urea manufacturing plant at Donaldsonville, Louisiana as a site for an emission test program. The test program was designed to provide a portion of the emission data base required for SPNSS for the processes associated with the production of urea.

The CF Industries, Inc. urea manufacturing plant at Donaldsonville, Louisiana produces granulated urea for fertilizer use. The urea is made by seven Spherodizers* (granulators) which operate continuously, 24 hours a day and 7 days a week, as production demands.

Each granulator has its own impingement type water scrubber. The granulator exhaust is ducted through the scrubber fan and is discharged from a stack. Flow through the granulator to the constant flow scrubber is controlled with a dilution damper which varies the ratio of dilution air to exhaust gas. A schematic of the granulator and control system is shown in Figure 1-1.

Emissions sampling was conducted on the "B" granulator of the three older units while its urea production rate was approximately 333 ton/day. Emission sampling was also conducted on the main exhaust vent atop the urea synthesis tower (see Figure 1-1). This vent combines the various solution forming process gases into one common stack before exhausting them to the atmosphere.

EPA engaged TRC to measure urea, ammonia and formaldehyde concentrations and mass flow rates; particle size distributions; and plume opacities. All measurements made at this facility were performed during times of normal operation of the urea production process as described in Section 3, "Process Description and Operation."

The measurement program was conducted at the CF Industries, Inc. urea manufacturing facility in Donaldsonville, Louisiana during the week of January 15 through January 19, 1979 and consisted of the following:

"B" Granulator Scrubber Measurements

1. Urea, Formaldehyde, and Ammonia in Gas Stream

Three runs of concurrent inlet and outlet tests were performed. The tests were conducted in accordance with the prescribed EPA methods for urea, ammonia, and formaldehyde and provided velocity, moisture, ammonia, formaldehyde and urea particulate mass flow rate data.

2. Particle Size Distributions in Gas Streams

Three runs of an inlet test were conducted. The tests were performed using the procedures provided by the cascade impactor manufacturer.

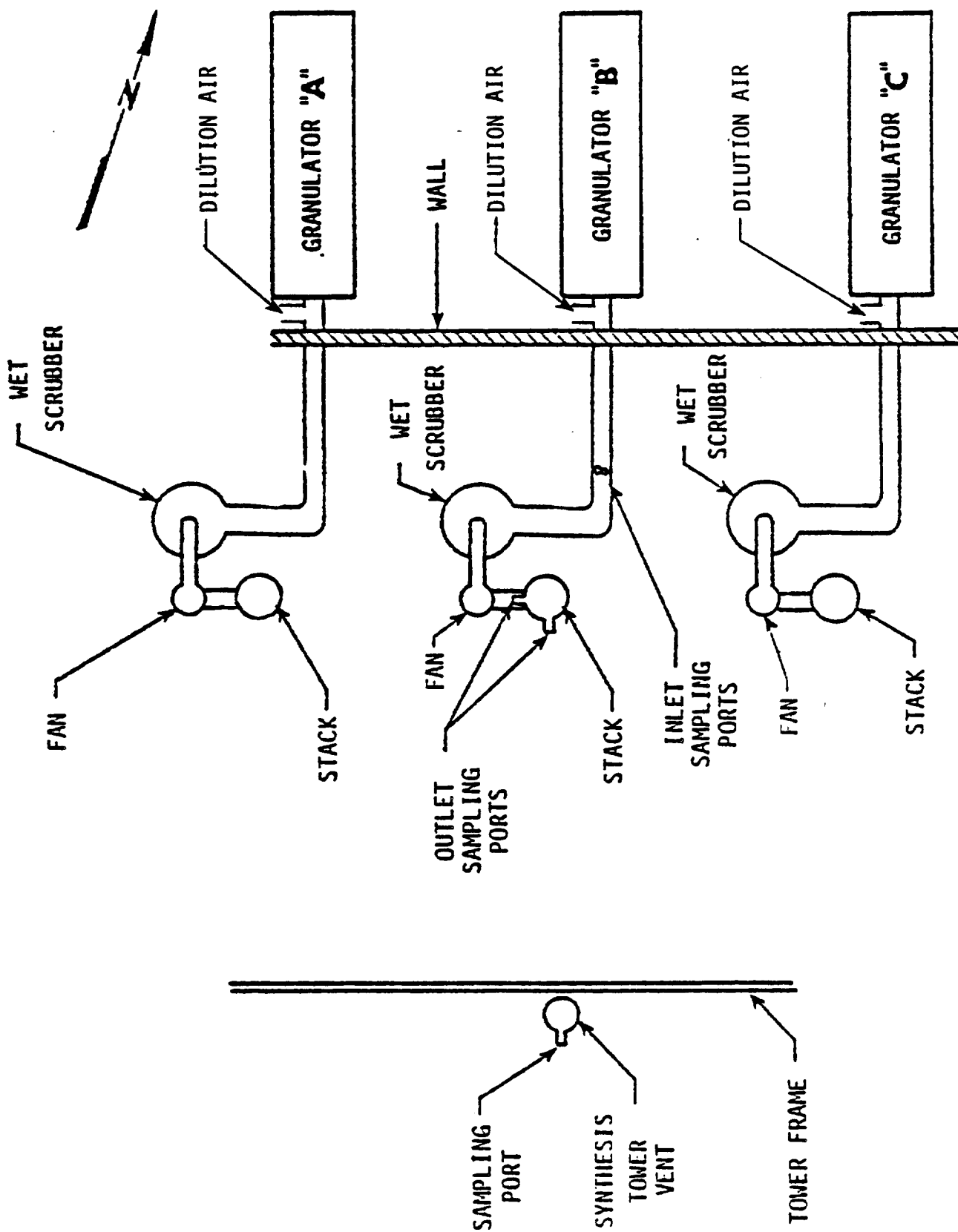


FIGURE 1-1: OVERHEAD VIEW SHOWING LOCATIONS OF SYNTHESIS TOWER VENT & GRANULATORS A, B & C EXHAUST DUCTING, SCRUBBERS & SAMPLING POINTS AT CF INDUSTRIES, INC., DONALDSONVILLE, LOUISIANA

3. Visible Emissions from Scrubber Stack

Approximately six hours of visible emissions observations were recorded on the scrubber stack discharge. Observations were performed in accordance with EPA Method 9 guidelines.

4. Gas Pressure Drop Across Scrubber

Pressure drop measurements were recorded approximately every five minutes during the testing periods.

5. Ambient Air Temperature, Pressure and Relative Humidity

Wet and dry bulb ambient air temperatures and the barometric pressure were recorded at frequent intervals during the test days.

6. Scrubber Liquid Sampling

Samples of inlet and outlet scrubber water were taken during the urea, ammonia and formaldehyde testing. The pH and temperature of each sample were recorded. The samples were composited into three inlet and three outlet samples which were analyzed for urea, ammonia, and formaldehyde, and percent solids.

7. Urea, Ammonia, and Formaldehyde Contents of Product

A single grab sample of the urea melt was taken and analyzed for urea, ammonia, and formaldehyde. In addition, three grab samples of the unscreened granulator product were analyzed for sieve, bulk density, and percent moisture. These samples were then combined for a urea and formaldehyde determination. The percent moisture determinations were performed by CFI personnel.

Urea Synthesis Tower Vent Measurements

Urea and Ammonia in Gas Stream

Three test runs were performed according to procedures prescribed in EPA methods for velocity, urea, and ammonia and for sampling gas streams with high moisture content.

TRC personnel were responsible for collecting and measuring the above emission parameters. Concurrently, GCA was responsible for monitoring and recording necessary process parameters. A copy of TRC's Work Assignment and Technical Directives is included in Appendix N.

The sequence of events for this sampling program is shown in Tables 1-1 through 1-3 (Daily Summary Logs).

Several of the test runs were discontinuous due to the excessive particulate loading at the granulator scrubber inlet sampling location. These interruptions, which also delayed the concurrent outlet sampling, occurred throughout the test program.

The following sections of this report cover the summary of results (Section 2), process description and operation (Section 3), location of sampling points (Section 4), and sampling and analysis methodologies (Section 5). In addition, Appendix L contains the results of the cleanup evaluations performed on the sample collectors used during the test program. Detailed descriptions of methods and procedures, field and laboratory data, and calculations are presented in various appendices, as noted in the Table of Contents.

TABLE I-1

DAILY SUMMARY LOG FOR "B" GRANULATOR SAMPLING ON JANUARY 17, 1979
AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

Clock Time	Production Rate tons/hour		"B" Granulator		Stack Visible Emissions	Scrubber Liquid				Ambient Conditions		Process Sampling
	Granulator	Syn. Tower	Inlet	Outlet		pH	Temp (°F)	pH	Temp (°F)	Temp (°F)	R.H. (%)	Pressure (inHg)
1200	Confidential Per CFI Request		Start Run 1	Start Run 1	Start	9.50	135	8.15	126	21.8	76	30.40
1201			Pitot Plugged									
1202												
1207												
1210												
1212												
1215												
1217												
1222												
1223												
1226												
1227												
1230												
1232												
1237												
1242												
1245												
1247												
1252												
1255												
1257												
1301												
1306												
1310												
1316												
1318												
1320												
1321												
1326												
1327												
1331												
1333												
1336												
1341												
1343												
1346												
1351												
1356												

Unscreened
Product

TABLE I-1 (Continued)

DAILY SUMMARY LOG FOR "B" GRANULATOR SAMPLING ON JANUARY 17, 1979
AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

Clock Time	Production Rate tons/hour Granulator	"B" Granulator		Stack Visible Emissions	Scrubber		Scrubber Liquid		Pressure Drop ("H ₂ O)	Ambient Conditions		Process Sampling
		Inlet	Outlet		Inlet	Outlet	Inlet	Outlet		Temp (°F)	R.H. (%)	
1401	Confidential Per CFI Request								20.7			
1406									20.5			
1411									20.5			
1416									20.5			
1417												
1422												
1423												
1448												
1523												
1548												
1549												
1550												
1554												
1559												
1604												
1609												
1614												
1619												
1620												
1624												
1629												
1634												
1635												
1637												
1639												
1644												
1649												
1653												
1655												
1657												
1658												

TABLE 1-1 (Continued)

DAILY SUMMARY LOG FOR "B" GRANULATOR SAMPLING ON JANUARY 17, 1979
AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

Clock Time	Production Rate tons/hour	"B" Granulator Scrubber		Stack Visible Emissions	Scrubber Liquid			Pressure Drop (¹¹ H ₂ O)	Ambient Conditions		Process Sampling	
		Urea Particulate			Inlet Temp (°F)	pH	Outlet Temp (°F)		Temp (°F)	R.H. (%)		Pressure (¹¹ Hg)
		Inlet	Outlet									
1702	Confidential Per CFI Request	Run Continued	Complete Run 2	Complete Run 2	9.0	172	8.0	129	66	85	30.38	
1703												
1708												
1713												
1715												
1718												
1723												
1725												
1728												
1730												
1733	Complete Run 2	Complete Run 2	Complete Run 2	9.0	172	8.0	129	66	85	30.38		
1738												
1743												
1748												
1753												
1756												
1800												
1815												

Unscreened Product

TABLE 1-2

DAILY SUMMARY LOG FOR "B" GRANULATOR SAMPLING ON JANUARY 18, 1979
AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

Clock Time	Production Rate tons/hour		"B" Granulator Urea Particulate		Stack Visible Emissions	Scrubber Liquid				Pressure Drop ("H ₂ O)	Ambient Conditions		Process Sampling	Synthesis Tower Sampling																																													
	Granulator	Syn. Tower	Inlet	Outlet		pH	Temp (°F)	Inlet Temp (°F)	Outlet Temp (°F)		Temp (°F)	R.H. (%)			Pressure ("Hg)																																												
0945	Confidential Per CFI Request		Start Run 3	Start Run 3																																																							
0946															Complete 1st Traverse	21.6	30.39																																										
0947																		9.2	180	8.3	128	21.2																																					
0951																								62	94	30.39																																	
0955																											64	90	30.39																														
0956																														20.7																													
1001																																20.5																											
1005																																		20.7																									
1006																																				20.7																							
1011																																						20.7																					
1016																																								20.6																			
1017																																										20.7																	
1020																																												64.5	87	30.38													
1021																																															66	82	30.38										
1026																																																		67.5	85	30.36							
1030																																																					20.5						
1031																																																											
1036																																																											
1040																																																											
1041																																																											
1045																																																											
1046																																																											
1047																																																											
1055																																																											
1102																																																											
1105																																																											
1107																																																											
1108																																																											
1112																																																											
1115																																																											
1117																																																											
1122																																																											

TABLE 1-2 (Continued)

DAILY SUMMARY LOG FOR "B" GRANULATOR SAMPLING ON JANUARY 18, 1979

AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

Clock Time	Production Rate tons/hour Granulator	"B" Granulator		Stack Visible Emissions	Scrubber Liquid				Pressure Drop ("H ₂ O)	Ambient Conditions		Process Sampling	Synthesis Tower Sampling
		Inlet	Outlet		pH	Temp (°F)	pH	Temp (°F)		Temp (°F)	R.H. (%)		
1127	Confidential Per CFJ Request								20.5				
1132									20.4				
1137									20.6				
1140										67	83	30.36	
1142													
1147													
1152													
1157										68	85	30.36	
1200													
1202									19.8				
1207									20.3				
1210									20.3				
1212									20.2				
1215									20.1				
1217													
1244									19.7				
1344									19.5				
1435										69	85	30.28	
1500										71	77	30.28	
1525										73	73	30.27	
1528													
1540										71	77	30.27	Start Run 1 (Test 4)
1543													
1555													Complete Run 1 (Test 4)
1615													
1649													

TABLE 1-3

DAILY SUMMARY LOG FOR "B" GRANULATOR SAMPLING ON JANUARY 19, 1979
AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

Clock Time	Production Rate tons/hour Granulator	Syn. Tower Request	"B" Granulator Scrubber		Stack Visible Emissions	Scrubber Liquid				Pressure Drop ("H ₂ O)	Ambient Conditions		Process Sampling	Synthesis Tower Sampling
			Inlet	Outlet		Inlet	Temp (°F)	pH	Outlet Temp (°F)		Temp (°F)	R.H. (%)		
0953	Confidential Per CFI Request	Start Cascade Run 2 Complete Cascade Run 2	Start	Stop	Start	Start	68	90	30.17	30.17	68	90	30.17	Start Run 2 (Test 5)
1000														
1015							68							
1020							70							
1030							70							
1040							70							
1046							70							
1100							70							
1120							70							
1125							70							
1135							75							
1140							77							
1150							73							
1153							77							
1200							73							
1230							77							

SECTION 2

SUMMARY OF RESULTS

INTRODUCTION

This section presents the results of a testing program conducted during the week of January 15 through 19, 1979 at the CF Industries, Inc., urea manufacturing facility in Donaldsonville, Louisiana. Testing was performed on gas and water streams entering and exiting the "B" Granulator Scrubber and on the gas stream venting from the Urea Synthesis Tower.

The inlet gas sampling location for the "B" Granulator Scrubber (designated TP-1) was in a 23-foot straight section of horizontal duct located upstream of the scrubber. The integrated gas samples for the urea, ammonia and formaldehyde tests were collected isokinetically from 36 traverse points located in accordance with EPA Reference Method.¹ The gas samples for the particle sizing tests were collected from a single point located at the centroid of the duct's cross sectional area.

The outlet gas sampling locations for the "B" Granulator Scrubber (designated TP-2) was in the 80-foot vertical stack located downstream of the scrubber. The integrated gas samples for the urea, ammonia and formaldehyde tests were

¹Standards of Performance for New Stationary Sources, Appendix A. Federal Register, Vol. 42, No. 160-Thursdays, August 18, 1977, pp. 41756-41758.

collected isokinetically from 12 traverse points located in accordance with EPA Reference Method I.

The gas sampling location for the Synthesis Tower Vent (designated TP-1) was in the 46-foot straight section of vertical duct. The samples were extracted from a single point located at the centroid of the duct's cross sectional area.

UREA, FORMALDEHYDE AND AMMONIA TESTS ON "B" GRANULATOR SCRUBBER

The overall summaries of the urea, formaldehyde and ammonia results at the "B" Granulator inlet (TP-1) and outlet (TP-2) are contained in Tables 2-1(a) and 2-1(b) in English and SI metric units, respectively. The calculated urea, formaldehyde and ammonia removal efficiencies of the "B" Granulator Scrubber averaged 99.8%, 50.2% and 20.5%, respectively.

Tables 2-2 and 2-3 present separately the inlet and outlet data, respectively. In addition, these two tables show the "corrected" urea data (corrected for possible urea loss during the Kjeldahl analysis method), and the "corrected" and "uncorrected" ammonia data resulting from the distillation-and-Nesslerization analysis method. The ammonia correction is for potential conversion of urea to ammonia during distillation. This second ammonia analysis method was added because of the potential susceptibility of the direct Nesslerization method to interferences.² Details of the analysis methods are contained in Section 5 and Appendix K. Because of the uncertainties involved in correcting the urea and ammonia data for urea conversion during analysis, the data in Table 2-1

²Standard Methods for the Analysis of Water and Wastewater, 14th Edition, APHA, AWWA, WPCF, 1975.

TABLE 2-1(a) ENGLISH

SUMMARY OF RESULTS OF UREA, AMMONIA AND FORMALDEHYDE TESTS ON GASES ENTERING AND LEAVING THE
"B" GRANULATOR SCRUBBER ON JANUARY 17 AND 18, 1979 AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

Run Number Date Location	Run 1 01-17-79		Run 2 01-17-79		Run 3 01-18-79		Average	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Volume of Gas Sampled - DSCF (a)	63.18	102.5	65.97	102.7	66.45	102.4	65.20	102.5
Percent Moisture by Volume (b)	2.257	5.522	2.253	5.733	2.257	5.608	2.256	5.621
Average Gas Temperature (°F)	192.1	100.5	190.2	100.7	183.1	102.5	188.5	101.2
Stack Volumetric Flowrate - DSCFM (c)	40180	46260	41410	45590	41760	45760	41117	45870
Scrubber Liquid pH	9.25	8.08	9.32	8.16	9.26	8.24	9.28	8.16
Average Percent Opacity		7.7		8.7		5.5		7.3
Production Rate* (tons/hr)	104.5	100.7	105.0	102.4	102.9	101.6	104.1	101.6
Percent Isokinetic								
UREA DATA								
Kjeldahl Analysis Method								
Total Sample Weight (mg)	23920	70.6	20690	73.1	26258	62.3	25423	68.7
gr/DSCF	5.830	0.0106	6.090	0.0110	6.085	0.0094	6.005	0.0103
lb/hr	2007	4.205	2161	4.283	2178	3.674	2116	4.058
lb/ton	111.5	0.232	120.1	0.238	125.2	0.211	118.9	0.228
collection efficiency (%)	99.8		99.8		99.8		99.8	
AMMONIA DATA								
Direct Nesslerization Analysis Method								
Total Sample Weight (mg)	410	450	441	694	514	387	455	510
gr/DSCF	0.0999	0.0676	0.1029	0.1041	0.1191	0.0582	0.1073	0.0766
lb/hr	34.40	26.80	36.52	40.17	42.62	22.82	37.85	30.10
lb/ton	1.90	1.48	2.03	2.26	2.45	1.31	2.13	1.69
collection efficiency (%)	22.1		<0		46.5		20.5	
FORMALDEHYDE DATA								
Chromotropic Acid Analysis Method								
Total Weight of Sample (mg)	2.476	2.523	1.545	1.172	3.087	1.296	2.369	1.664
gr/DSCF	0.000604	0.000379	0.000361	0.000176	0.000715	0.000195	0.000560	0.000250
lb/hr	0.2078	0.1503	0.1280	0.0687	0.2560	0.0764	0.1972	0.0983
lb/ton	0.0115	0.0083	0.0071	0.0038	0.0147	0.0044	0.0111	0.0055
collection efficiency (%)	27.7		46.3		70.2		50.2	

(a) Dry standard cubic feet @ 68°F, 29.92 inches Hg

(b) Inlet values based on separate moisture run

(c) Dry standard cubic feet per minute

(d) Uncorrected for urea loss during distillation

(e) Grains per dry standard cubic foot

* Production rate information is confidential per CFI request

TABLE 2-1(b) METRIC

SUMMARY OF RESULTS OF UREA, AMMONIA AND FORMALDEHYDE TESTS ON GASES ENTERING AND LEAVING THE "B" GRANULATOR SCRUBBER
ON JANUARY 17 AND 18, 1979 AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

Run Number Date Location	Run 1 01-18-79		Run 2 01-18-79		Run 3 01-19-79		Average	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Volume of Gas Sampled-Nm ³ (a)	1.789	2.903	1.868	2.908	1.882	2.899	1.846	2.903
Percent Moisture by Volume (b)	2.257	5.522	2.253	5.733	2.257	5.608	2.256	5.621
Average Gas Temperature (°C)	88.9	38.0	87.8	38.2	83.9	39.2	86.9	38.4
Stack Volumetric Flowrate-Nm ³ /M (c)	1137	1310	1173	1291	1187	1296	1164	1299
Scrubber Liquid pH	9.25	8.08	9.32	8.16	9.26	8.24	9.28	8.16
Average Percent Opacity		7.7		8.7		5.5		7.3
Production Rate* (Mg/Hour)								
Percent Isokinetic	104.5	100.7	105.0	102.4	102.9	101.6	104.1	101.6
UREA DATA								
Kjeldahl Analysis Method (d)								
Total Sample Weight (mg)	23920	70.6	26090	73.1	26258	62.3	25423	68.7
g/Nm ³	13.34	0.0242	13.93	0.0252	13.92	0.0215	13.74	0.0236
Kg/hour	910.4	1.907	980.23	0.0049	987.9	1.66	959.8	1.841
Kg/Mg	55.75	0.116	60.05	0.119	62.6	0.105	59.4	0.114
Collection Efficiency (%)		99.8		99.8		99.8		99.8
AMMONIA DATA								
Direct Nesslerization Analysis Method								
Total Sample Weight (milligrams)	410	450	441	694	514	387	344	510
g/Nm ³	0.2286	0.155	0.235	0.238	0.272	0.133	0.2455	0.1753
Kg/hour	15.60	12.16	16.56	18.45	19.33	10.35	17.17	13.65
Kg/Mg	0.95	0.74	1.01	1.13	1.22	0.65	1.06	0.85
Collection Efficiency (%)		22.1		<0		46.5		20.5
FORMALDEHYDE DATA								
Chromotropic Acid Analysis Method								
Total Weight of Sample (milligrams)	2.476	2.523	1.545	1.172	3.087	1.296	2.369	1.664
g/Nm ³	0.00138	0.0022	0.000826	0.000402	0.00164	0.00045	0.0013	0.000372
Kg/hour	0.0942	0.068	0.058	0.0311	0.116	0.035	0.0894	0.0446
Kg/Mg	0.00575	0.0041	0.00355	0.0019	0.00735	0.0022	0.0055	0.0027
Collection Efficiency (%)		27.8		46.3		70.1		50.3

(a) Normal Cubic Meters @ 20°C, 760 mm Hg.

(b) Inlet values based on separate moisture run.

(c) Normal Cubic Meters per minute.

(d) Uncorrected for Urea loss during distillation.

(e) Grains per Normal Cubic Meter.

* Confidential Per CFI Request

TABLE 2-2

SUMMARY OF RESULTS OF UREA, AMMONIA AND FORMALDEHYDE TESTS ON GASES ENTERING THE
"B" GRANULATOR SCRUBBER ON JANUARY 17 AND 18, 1979 AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

Run Number	Run 1 01-17-79	Run 2 01-17-79	Run 3 01-18-79	Average
Volume of Gas Sampled-DSCF (a)	63.18	65.97	66.45	65.2
Percent Moisture by Volume (b)	2.257	2.253	2.257	2.256
Average Gas Temperature (°F)	192.1	190.2	183.1	188.5
Stack Volumetric (c)	40180	41410	41760	41117
Flowrate-DSCFM	9.25	9.32	9.26	9.28
Scrubber Liquid pH				
Production Rate* (Tons/hr)	104.5	105.0	102.9	104.1
Percent Isokinetic	111	110	111	111
Net Sampling Time (Minutes)				
UREA DATA				
Kjeldahl Analysis Method:				
Total Sample Weight (mg)	23920	26090	26258	25423
gr/DSCF	5.830	6.090	6.085	6.005
lb/hr	2007	2161	2178	2116
lb/ton	111.5	120.1	125.2	118.9
AMMONIA DATA				
Nesslerization Analysis Method:				
Total Sample Weight (mg)	410	441	514	455
gr/DSCF	0.0999	0.1029	0.1191	0.1073
lb/hr	34.40	36.52	42.62	37.85
lb/ton	1.90	2.03	2.45	2.13
FORMALDEHYDE DATA				
Chromotropic Acid Analysis Method:				
Total Sample Weight (mg)	2.476	1.545	3.087	2.369
gr/DSCF	0.000604	0.000361	0.000715	0.000560
lb/hr	0.2078	0.1280	0.2560	0.1972
lb/ton	0.0115	0.0071	0.0147	0.0111

(a) Dry Standard Cubic Feet @ 68°F, 29.92 inches Hg.

(b) based on separate moisture run

(c) Dry Standard Cubic Feet per minute

(d) Grains per DSCF.

(e) Uncorrected for urea conversion during distillation.

(f) Corrected for urea conversion during distillation. Corrected = uncorrected x 1.07.

(g) Direct Nesslerization.

(h) Distillation and Nesslerization, uncorrected for conversion of urea to ammonia.

(i) Distillation and Nesslerization, corrected for conversion of urea to ammonia. Corrected = uncorrected -0.07 x corrected urea/1.765

* Confidential

SUMMARY OF RESULTS OF UREA, AMMONIA AND FORMALDEHYDE TESTS ON GASES LEAVING THE "B" GRANULATOR SCRUBBER ON JANUARY 17 and 18, 1979 AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

(a) Dry Standard Cubic Feet @ 68°F, 29.92 inches Hg
 (b) Dry Standard Cubic Feet per minute
 (c) grains per DSCF
 (d) Uncorrected for urea conversion during distillation
 (e) Corrected for urea conversion during distillation. Corrected = uncorrected x 1.07
 (f) Direct Nesslerization
 (g) Distillation and Nesslerization, uncorrected for conversion of urea to ammonia
 (h) Distillation and Nesslerization, corrected for conversion of urea to ammonia (corrected = uncorrected - 0.07 x corrected urea/l.765)
 * Confidential Per CFI Request

(uncorrected urea and direct Nesslerization ammonia) should be viewed as the most accurate data, with the following qualification.

The direct Nesslerization outlet ammonia mass flow rate appears to be very high in Run 2 compared to those of Runs 1 and 3 (Table 2-3). The following observations clarify this point:

1. Runs 1 and 3 each have mass flow rates which are nearly equal for both the direct-Nesslerization and the distillation-and-Nesslerization analysis.
2. Run 2 has a mass flow rate (by distillation-and-Nesslerization) consistent with the mass flow rates of Runs 1 and 3.
3. Run 2 has a mass flow rate (by direct-Nesslerization) more than 50% greater than those of Runs 1 and 3.

Thus, the Run 2 direct Nesslerization ammonia outlet result is suspect.

SAMPLING AND ANALYSIS PROBLEMS WITH UREA, FORMALDEHYDE AND AMMONIA TESTS

As noted in Section 1, some test runs were discontinuous because the high concentration and large size of urea particles at the scrubber inlet caused plugging of the pitot tubes and the probe and nozzle. In order to quickly remove these plugs from the nozzle, water was squirted into the probe each time a plug occurred. Since this additional water contributed to the total amount of water collected during each run, a separate run for moisture alone was performed at the scrubber inlet. Details of these problems are contained in Section 5.

At the B granulator scrubber inlet, the large difference between the results of the two ammonia analysis methods is believed to be due to the conversion of the

urea to ammonia during distillation. The analysis by direct Nesslerization was performed at CF Industries in Donaldsonville, and the distillation/Nesslerization analysis was performed at the TRC laboratory in Wethersfield, CT. The distillation/Nesslerization method was done in addition to the direct Nesslerization because of interferences due to turbidity in the direct Nesslerization method. However, it is documented in the literature that approximately 7 percent of the urea is converted to ammonia during the distillation procedure. This conversion factor is not precise, and when the concentration of urea greatly exceeds that of ammonia, even a small deviation from the 7 percent conversion factor would mean a considerable amount of ammonia when compared to amount of ammonia actually measured. Details of this urea conversion are presented in Section 5. The negative ammonia concentrations shown in Table 2-2 serve to illustrate the magnitude of error that can result with the distilled Nesslerization method. Therefore, the ammonia results determined by the direct Nesslerization procedure are considered to be the more accurate values.

The average ammonia results at the scrubber outlet (Table 2-3) are contrary to those expected because conversion of urea to ammonia during distillation should yield a greater ammonia mass flow rate for the distillation/Nesslerization method than for the direct Nesslerization method. The probability that the direct Nesslerization results for Run 2 may be anomalously high, as discussed above, may partially explain this situation.

VISIBLE EMISSIONS FROM "B" GRANULATOR SCRUBBER STACK

The opacity of the plume from the "B" Granulator Scrubber stack ranged from 5 to 10 percent. The six-minute arithmetic averages are presented

graphically in Figure 2-1 and are summarized in Table 2-4. The observations were made from the synthesis tower with the local terrain as background. The detailed information on the visible emission measurements can be found in Appendix C.

PARTICLE SIZE TESTS ON "B" GRANULATOR SCRUBBER INLET (TP-1)

Particle size distribution tests were conducted on the "B" Granulator Scrubber Inlet (TP-1). Summaries of these test results are presented in Table 2-5. The size of the particulates entering the "B" Granulator Scrubber was 100% >5.7 μm . That is, all the particulate was collected in the cyclone precollector. Therefore, it was not possible to plot cumulative size distribution curves.

PRESSURE DROP MEASUREMENTS ACROSS "B" GRANULATOR SCRUBBER

The pressure drop measurements across the "B" Granulator Scrubber were made with a vertical U-tube water manometer which was connected to pressure taps at the scrubber inlet and outlet. The pressure drop across the scrubber was recorded at approximately 5 to 15 minute intervals during the tests for urea, ammonia and formaldehyde.

The pressure drops across the "B" Granulator Scrubber ranged from 19.5 to 21.8 inches of vertical water column and are presented in Table 2-6.

UREA, AMMONIA AND FORMALDEHYDE IN SCRUBBING LIQUID ENTERING AND EXITING "B" GRANULATOR SCRUBBER

Half-liter samples of the scrubbing liquid streams entering and leaving the "B" Granulator Scrubber were collected at approximately 30-minute intervals during the test runs. The solution temperature was measured immediately after

FIGURE 2-1: SIX MINUTE ARITHMETIC AVERAGES OF JANUARY 17, 18,
AND 19, 1979 OPACITY READINGS ON "B" GRANULATOR
SCRUBBER STACK AT CF INDUSTRIES, INC.,
DONALDSONVILLE, LOUISIANA

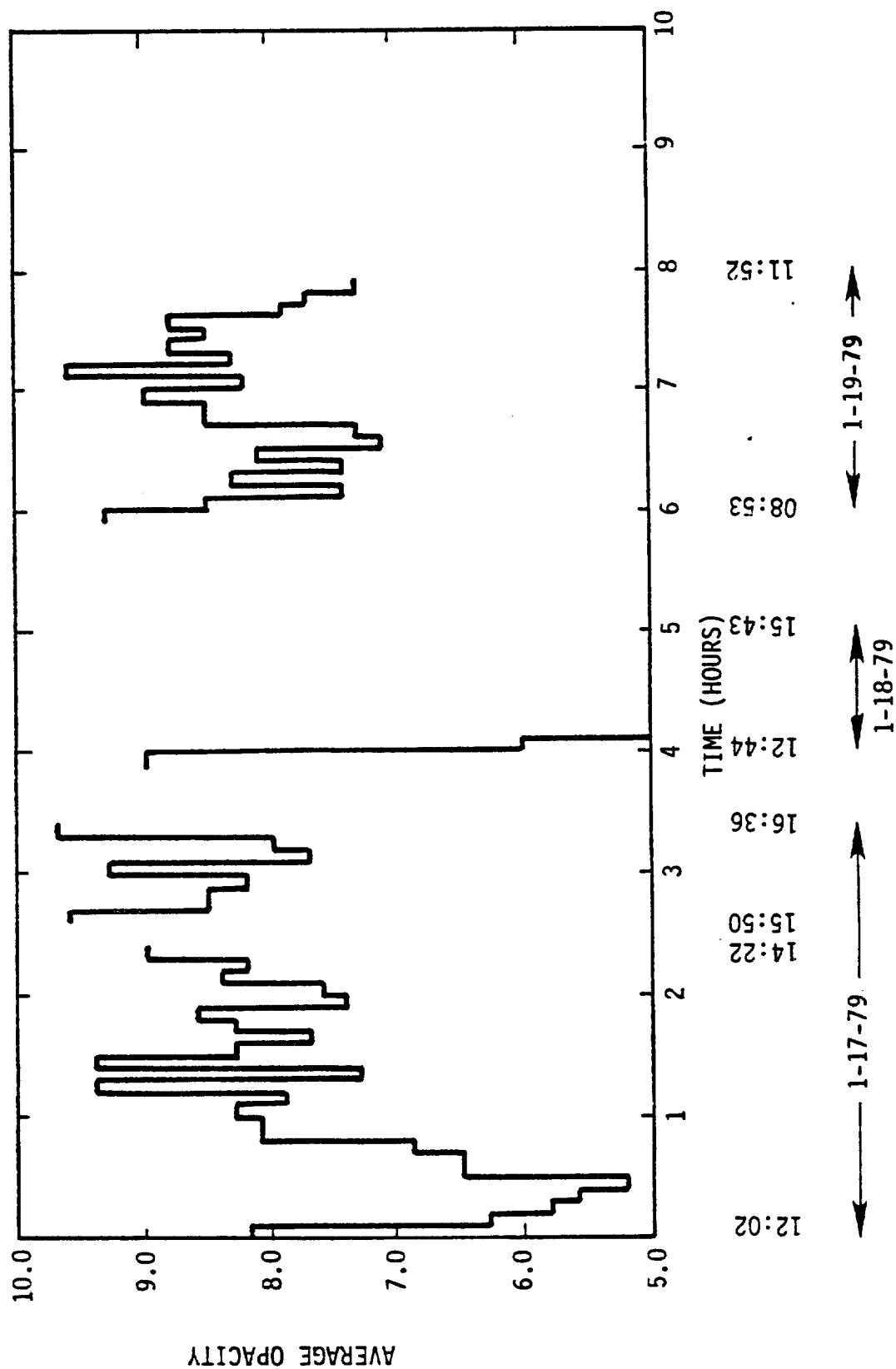


TABLE 2-4

SIX MINUTE ARITHMETIC AVERAGE OPACITY READINGS
ON "B" GRANULATOR SCRUBBER STACK
ON JANUARY 17, 1979, AT CF INDUSTRIES, INC.,
DONALDSONVILLE, LOUISIANA

TIME	AVG. OPACITY FOR 6 MIN.	DATE
12:02 - 12:07	8.2	1-17-79
12:08 - 12:13	6.3	
12:14 - 12:17	5.8	
12:20 - 12:25	5.6	
12:26 - 12:31	5.2	
12:32 - 12:37	6.5	
12:38 - 12:43	6.5	
12:44 - 12:49	6.9	
12:50 - 12:55	8.1	
12:56 - 13:01	8.1	
13:02 - 13:07	8.3	
13:08 - 13:13	7.9	
13:14 - 13:19	9.4	
13:20 - 13:25	7.3	
13:26 - 13:31	9.4	
13:32 - 13:37	8.3	
13:38 - 13:43	7.7	
13:44 - 13:49	8.3	
13:50 - 14:55	8.6	
13:56 - 14:01	7.4	
14:02 - 14:07	7.6	
14:08 - 14:13	3.4	
14:14 - 14:19	8.8	
14:20 - 14:22*	9.0	
15:50 - 15:55	9.6	
15:56 - 16:01	8.5	
16:02 - 16:07	8.5	
16:08 - 16:13	8.2	
16:14 - 16:19	9.3	
16:20 - 16:25	7.7	
16:26 - 16:31	8.0	
16:32 - 16:36*	9.7	

* Less than 6 min average

TABLE 2-4 (Continued)

SIX MINUTE ARITHMETIC AVERAGE OPACITY READINGS
ON "B" GRANULATOR SCRUBBER STACK
ON JANUARY 18, 1979, AT CF INDUSTRIES, INC.,
DONALDSONVILLE, LOUISIANA

TIME	AVG. OPACITY FOR 6 MIN.	DATE
12:44 - 12:49	9.0	1-18-79 ↓
12:50 - 12:55	6.0	
12:56 - 13:01	5.0	
13:02 - 13:07	5.0	
13:08 - 13:13	5.0	
13:14 - 13:19	5.0	
13:20 - 13:25	5.0	
13:26 - 13:31	5.0	
13:32 - 13:37	5.0	
13:38 - 13:43	5.0	

TABLE 2-4 (Continued)

SIX MINUTE ARITHMETIC AVERAGE OPACITY READINGS
ON "B" GRANULATOR SCRUBBER STACK
ON JANUARY 19, 1979, AT CF INDUSTRIES, INC.,
DONALDSONVILLE, LOUISIANA

TIME	AVG. OPACITY FOR 6 MIN.	DATE
09:53 - 09:58	9.3	1-19-79
09:59 - 10:04	8.5	
10:05 - 10:10	7.4	
10:11 - 10:16	8.3	
10:17 - 10:22	7.4	
10:23 - 10:28	8.1	
10:29 - 10:34	7.1	
10:35 - 10:40	7.3	
10:41 - 10:46	8.5	
10:47 - 10:52	8.5	
10:53 - 10:58	9.0	
10:59 - 11:04	8.2	
11:05 - 11:10	9.6	
11:11 - 11:16	8.3	
11:17 - 11:22	8.3	
11:23 - 11:28	8.5	
11:29 - 11:34	8.8	
11:35 - 11:40	7.9	
11:41 - 11:46	7.7	
11:47 - 11:52	7.3	

TABLE 2-5

SUMMARY OF INLET PARTICLE SIZE TEST RESULTS ON "B" GRANULATOR
 SCRUBBER ON JANUARY 18, 1979, AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

TRC Test No.	Sampling Location	Test Date	Test Time	Particulate Concentration, grains/dscf	Aerodynamic Size Range, μ m	Mass In Size Range, %
CI-1	Scrubber Inlet	1/18/79	15:28-15:43	4.512	> 6.0	99+
CI-2	Scrubber Inlet	1/19/79	10:00-10:15	3.717	> 5.7	99+
CI-3	Scrubber Inlet	1/19/79	11:25-11:40	0.935	> 5.8	99+

TABLE 2-6

SUMMARY OF JANUARY 17 AND 18, 1979 "B" GRANULATOR
SCRUBBER PRESSURE DROP MEASUREMENTS AT CF INDUSTRIES, INC.,
DONALDSONVILLE, LOUISIANA

<u>DATE</u>	<u>RUN</u>	<u>CLOCK TIME</u>	<u>ΔP, "H₂O</u>	<u>DATE</u>	<u>RUN</u>	<u>CLOCK TIME</u>	<u>ΔP "H₂O</u>
01/17/79	1	1207	21.8	01/17/79	2	1644	20.2
		1212	21.6			1658	20.4
		1217	21.5			1703	20.4
		1222	20.9			1708	20.4
		1227	20.7			1713	20.2
		1232	20.7			1718	20.3
		1237	20.7			1723	20.4
		1242	21.1			1728	20.6
		1247	21.1			1733	20.6
		1252	20.7			1738	20.6
		1257	20.5			1743	20.4
		1321	20.5			1748	20.5
		1326	20.7			1753	20.2
		1331	20.9				
01/17/79		1336	20.9	01/18/79	3	Average	20.5
		1341	20.7			0946	21.6
		1346	20.7			0951	21.2
		1351	20.7			0956	21.0
		1356	20.9			1001	21.1
		1401	20.7			1006	21.0
		1406	20.5			1011	21.2
		1411	20.5			1016	21.0
		1416	20.5			1021	20.7
		Average	20.9			1026	20.5
						1031	20.7
						1036	20.6
						1041	20.7
						1122	20.5
						1127	20.5
01/17/79	2	1549	20.2	01/18/79		1137	20.6
		1554	20.2			1142	20.3
		1559	20.2			1147	20.3
		1604	20.8			1152	20.2
		1609	20.9			1157	20.1
		1614	20.9			1202	19.8
		1619	20.7			1207	20.0
		1624	20.8			1212	19.7
		1629	20.9			1217	19.5
		1634	21.0				
		1639	21.0			Average	20.6

each sample was collected. The pH was measured once the sample had cooled to approximately 70° F.

For each test run, the individual samples were combined into two composite samples (one inlet and one outlet) which were analyzed for urea, ammonia and formaldehyde content. Table 2-7 presents a summary of the measured urea, ammonia and formaldehyde concentrations and the percent solids in the scrubber liquid composite samples. Table 2-8 contains the pH and temperature readings of each of the individual samples.

The results of the scrubber liquid analyses show that the average urea concentration of the outlet liquid is more than 5400 times greater than that of the inlet liquid. This would be expected because urea is being scrubbed out of the gas stream entering the scrubber. Analysis of the outlet liquid for ammonia by direct Nesslerization was unsuccessful because the sample solutions turned cloudy when the Nessler reagent was added. This is indicative of interference, due perhaps to the high urea concentration. The distillation-and-Nesslerization ammonia results are questionable, especially the outlet liquid results, because of the possible conversion of urea to ammonia during distillation. The uncertainty of the urea conversion is illustrated by the negative ammonia concentrations that result when the conventional correction is applied.

The average formaldehyde concentration of the inlet liquid is 400 times more than that of the outlet liquid, indicating that formaldehyde may be reacting with the urea in the outlet liquid. The lower temperature of the outlet liquid reflects the endothermic dissolution of urea into the scrubbing medium.

TABLE 2-7

SUMMARY OF UREA, AMMONIA AND FORMALDEHYDE MEASUREMENTS ON
THE SCRUBBING LIQUID ENTERING AND LEAVING THE
"B" GRANULATOR SCRUBBER AT CF INDUSTRIES INC.,
DONALDSONVILLE, LOUISIANA

Run Number	Run 1		Run 2		Run 3		Average	
	01-17-79		01-17-79		01-18-79			
Date Location	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Urea Concentration (mg/l) ^e								
Uncorrected ^a	114	516300	83	525100	94	538300	97	526600
Corrected ^b	122	552400	89	561900	101	576000	104	563400
Ammonia Concentration (mg/l)								
Direct Nesslerization	180	f	44	f	23	f	82	-
Distilled Uncorrected ^c	138 ⁱ	14100	52 ^j	12800	31	13100	74	13330
Distilled Corrected ^d	133	-7800	85	-9500	27	-9700	82	-
Formaldehyde Concentration (mg/l)	26	0.01	20	0.08	14	0.05	20	0.05
pH ^g	9.25	8.08	9.32	8.16	9.26	8.24	9.28	8.16
Temperature (°F) ^h	151	124	172	128	175	127	166	126
Percent Solids	1.2	3.9	0.9	4.6	3.6	4.8	1.9	4.4

^a Kjeldahl analysis results uncorrected for urea conversion during distillation.

^b Kjeldahl analysis results corrected for urea conversion during distillation (corrected = uncorrected x 1.07).

^c Distillation/Nessleration (D/N) analysis results uncorrected for urea conversion during distillation.

^d D/N analysis results corrected for urea conversion (corrected = uncorrected - 0.07*corrected urea/1.765).

^e Milligrams per liter.

^f Average spectrophotometric measurement not possible due to turbidity.

^g Average of several individual liquid samples taken during each run.

^h Average of individual sample temperatures taken immediately after collection.

ⁱ Average slight turbidity may have given high instrument readings. Visual estimate for this sample is 60 mg/l.

^j Average slight turbidity may have given high instrument readings. Visual estimate for this sample is 30 mg/l.

TABLE 2-8

SUMMARY OF pH AND TEMPERATURE
MEASUREMENTS ON INDIVIDUAL SAMPLES OF SCRUBBING LIQUID
ENTERING AND EXITING "B" GRANULATOR SCRUBBER AT
CF INDUSTRIES, INC., DONALDSONVILLE, LOUISIANA

Run No.	Date	Sampling Time	Scrubber Inlet		Scrubber Outlet	
			pH	°F	pH	°F
1	01/17-79	1215	9.50	135	8.15	126
		1255	9.25	154	8.10	124
		1331	9.15	164	8.10	124
		1416	9.10	152	7.95	120
		Average	9.25	151	8.08	124
2	01/17/79	1550	9.70	166	8.30	129
		1620	9.50	183	8.20	128
		1655	9.20	174	8.20	129
		1725	9.20	167	8.10	128
		1800	9.00	172	8.00	129
		Average	9.32	172	8.16	129
3	01/18/79	0955	9.20	180	8.30	128
		1030	9.30	172	8.25	126
		1105	9.30	177	8.25	129
		1140	9.30	173	8.20	127
		1215	9.20	173	8.20	124
		Average	9.26	175	8.24	127

The higher percent solids evident in the outlet scrubber liquid compared to the inlet may be attributed to organic and inorganic materials insoluble at room temperature (e.g., biuret and pipescale).

"B" GRANULATOR PROCESS SAMPLES UREA, AMMONIA, AND FORMALDEHYDE COMPOSITION

Process samples were taken of the urea melt and the urea product before screening. These samples were analyzed for urea, ammonia and formaldehyde and the results of these analyses are summarized in Table 2-9. CF Industries performed periodic checks on the moisture content of the granulated product. The average values for the testing days is included in Table 2-9. Bulk density and sieve analyses were performed on the granulated product samples. These results are presented in Table 2-10.

AMBIENT TEMPERATURES, BAROMETRIC PRESSURE AND RELATIVE HUMIDITIES AT CF INDUSTRIES, INC., DONALDSONVILLE, LOUISIANA

Table 2-11 summarizes the periodic meteorological measurements taken at CF Industries, Inc., on January 17 through 19, 1979.

UREA AND AMMONIA TESTS ON SYNTHESIS TOWER MAIN VENT

A summary of the urea and ammonia test results on the Synthesis Tower Main Vent (TP-1) are presented in Table 2-12. The average urea and ammonia mass flow rates were 1.70 and 431 pounds per hour, respectively.

Appendix F contains the pertinent sampling data for the synthesis tower plus all calculations. Special calculations were required because of the high ammonia content in the gas stream.

TABLE 2-9

SUMMARY OF UREA, AMMONIA AND FORMALDEHYDE MEASUREMENTS*
ON THE "B" GRANULATOR MELT AND PRODUCT-BEFORE-SCREENING
ON JANUARY 17 AND 18, 1979
AT CF INDUSTRIES INC., DONALDSONVILLE LOUISIANA

	Percent By Weight	
	Urea Melt	Urea Product Before Screening ^a
Urea Composition		
Uncorrected ^b	90.5	96.1
Corrected ^c	96.8	102.8
Ammonia Composition		
Direct Nesslerization	g	g
Distilled Uncorrected ^d	1.7	2.1
Distilled Corrected ^e	-2.1	-2.0
Formaldehyde Composition	0.4	0.6
Moisture ^f	-	0.2

^aComposite sample

^bKjeldahl analysis results uncorrected for urea conversion during distillation.

^cKjeldahl analysis results corrected for urea conversion. (corrected = uncorrected x 1.07)

^dDistillation/Nesslerization (D/N) analysis results uncorrected for urea conversion during distillation.

^eD/N analysis results corrected for urea conversion. (corrected = uncorrected - 0.07 * corrected urea / 1.765.

^fAverage of measurements by CF Industries Inc.

^gTurbidity prevented sample evaluation.

*The data in this table are considered questionable, based on other analyses performed by CFI and the fact that product samples contain many other compounds not analyzed for here.

TABLE 2-10

SUMMARY ON SIEVE AND BULK DENSITY MEASUREMENTS
ON THE "B" GRANULATOR PRODUCT BEFORE SCREENING ON JANUARY 17 and 18, 1979
AT CF INDUSTRIES, INC., DONALDSONVILLE, LOUISIANA

	Granulator Unscreened Product Sample #1 Collected @1343 on 01/17/79			Granulator Unscreened Product Sample #2 Collected @1730 on 01/17/79			Granulator Unscreened Product Sample #3 Collected @1045 on 01/18/79		
	Mass, gm	Percent of Total Mass	Cumula- tive Percent of Total Mass	Mass gm	Percent of Total Mass	Cumula- tive Percent of Total Mass	Mass, gm	Percent of Total Mass	Cumula- tive Percent of Total Mass
Total Sample To Sieves	237.36			245.26			224.37		
Sieve No. 6	28.25	11.90	100	24.44	10.03	100	16.55	7.39	100
Sieve No. 8	44.50	18.75	88.10	46.77	19.20	89.97	42.92	19.12	92.63
Sieve No. 10	58.42	24.62	69.34	60.74	24.93	70.77	56.15	25.01	73.51
Sieve No. 12	45.56	19.20	44.73	48.87	20.06	45.84	46.20	20.58	48.49
Sieve No. 14	39.91	16.82	25.52	40.43	16.59	25.78	40.58	18.08	27.91
Sieve No. 16	13.75	5.79	8.71	14.74	6.05	9.19	14.37	6.40	9.83
Bottom	6.92	2.92	2.92	7.64	3.14	3.14	7.70	3.43	3.43
Sum of Mass on Sieves	237.31			243.63			224.47		
Bulk Density, Grams Per 250 ml	213.11			214.64			213.53		

TABLE 2-11

SUMMARY OF JANUARY 17, 18 and 19, 1979 AMBIENT TEMPERATURE
RELATIVE HUMIDITY AND BAROMETRIC PRESSURE MEASUREMENTS
FOR "B" GRANULATOR AT CF INDUSTRIES, INC., DONALDSONVILLE, LOUISIANA

DATE	RUN	SAMPLING TIME	AMBIENT TEMPERATURE °F	RELATIVE HUMIDITY %	BAROMETRIC PRESSURE INCHES Hg	
01/17/79	1	1230	69	76		
		1245	69	76		
		1306	69	76	30.40	
		1320	69	76	30.40	
		1356	69	76	30.39	
		1448	70	74	30.39	
		Average	69	76	30.40	
	2	1523	69	79	30.39	
		1635	67	85	30.39	
		1715	68	80	30.39	
		1815	66	85	30.38	
		Average	68	82	30.39	
	01/18/79	3	0945	62	94	30.39
			1005	62	94	30.39
1020			64	90	30.39	
1040			64	87	30.38	
1055			66	82	30.38	
1115			67	85	30.36	
1140			67	83	30.36	
1200			68	85	30.36	
1435			69	85	30.28	
1500			71	77	30.28	
1525			73	73	30.27	
1540			71	77	30.27	
Average			67	84	30.34	
01/19/79			1000	68	90	30.17
		1020	68	90	30.17	
		1040	70	86	30.17	
		1100	70	86	30.17	
		1120	70	86	30.14	
		1140	75	70	30.12	
		1200	77	66	30.10	
		1230	73	75	30.10	
	Average	71	81	30.14		

SUMMARY OF RESULTS OF UREA AND AMMONIA TESTS ON GASES EXITING THE UREA SYNTHESIS TOWER VENT ON JANUARY 18 and 19, 1979 AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

(a) Dry standard cubic feet @ 68°F, 29.92 inches Hg
 (b) Dry standard cubic feet per minute
 (c) Grains per DSCF
 (d) Uncorrected for urea conversion during distillation
 (e) Corrected for urea conversion during distillation (corrected = uncorrected x 1.07)
 (f) Direct Nesslerization
 (g) Distillation and Nesslerization - uncorrected for urea conversion during distillation
 (h) Distillation and Nesslerization - corrected for urea conversion during distillation (corrected = uncorrected - 0.07 x corrected urea/1.765)
 * All production rate information is confidential, per CFI request

SECTION 3

PROCESS DESCRIPTION AND OPERATION

PROCESS EQUIPMENT

Urea is produced by reacting liquid ammonia (NH_3) with carbon dioxide (CO_2) at an elevated temperature and pressure. The reaction is exothermic and spontaneous and results in formation of liquid ammonium carbamate ($\text{NH}_2 \text{CO}_2 \text{NH}_4$). The liquid ammonium carbamate is subsequently decomposed to urea ($\text{CO}(\text{NH}_2)_2$) and water. The resulting solution of urea in water is concentrated to 89+ percent urea when it is finally solidified.

The Stamicarbon CO_2 Stripping Process is the urea synthesis method employed at this facility. This particular process incorporates emission reduction and energy recovery techniques in its standard design. The exhaust from the steam jet ejector vacuum system on the evaporators usually consists of inerts, ammonia, and traces of urea.

Liquid (70 percent urea) leaving the solution production area goes to a holding tank prior to being concentrated to 99.5 percent in a two-stage vacuum evaporator.

The concentrated molten urea, referred to as melt, leaves the solution synthesis process and is pumped to the solids formation equipment. This facility employs rotary drum granulators, designed by C&I Girdler, as the solids forming devices. A typical diagram displaying the process components is presented in Figure 3-1.

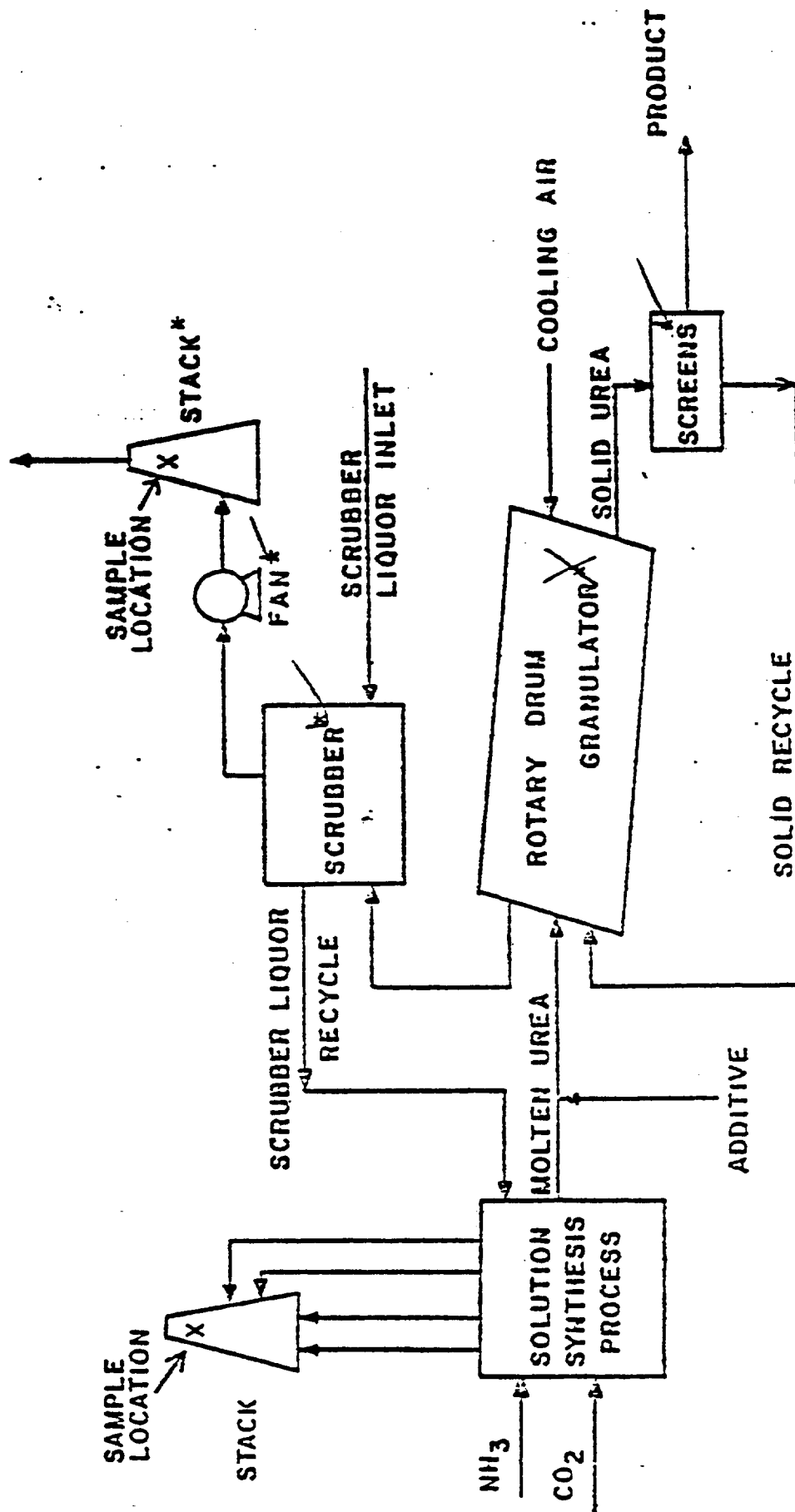


FIGURE 3-1 TYPICAL UREA MANUFACTURING PROCESS AS DESIGNED BY C & I GIRDLER

The molten urea is sprayed onto a bed of solid urea "seed" particles at the higher end of the inclined granulator. Lifting flights arrayed inside the granulator cause the solid urea "seed" particles to continually fall through the molten sprays and a counter-current flow of cooling air. The molten urea solidifies on these "seed" particles, increasing their size. As the particles grow in size, they eventually spill over a retaining dam into the cooling section of the granulator.

Cooled granules leaving the rotary drum granulator are screened. Oversize granules are crushed, combined with undersize granules, and returned in solid form to the bed of material at the spray end of the granulator as make-up "seed." Product size granules are loaded and/or conveyed to a bulk storage warehouse.

This facility does not coat the product urea granules. Instead, a formaldehyde-based additive is added to the molten urea before it is sprayed in the granulator.

Emissions points are the various vents in the solution synthesis process and the granulator cooling air exhaust. The solution synthesis process vents are combined into one stack before exhausting to the atmosphere. The granulator cooling air passes through a scrubber and fan before being exhausted out a stack. Scrubber liquid is returned to the solution synthesis process for urea recovery.

There are two separate urea production lines at this facility. Each line has its own urea solution production plant and rotary drum granulator units for granular urea formation. The production lines are identical except for size and age.

Production line No. 1 was put into service in October 1974, and is rated at 998 Mg/day (1100 tons/day) of solid urea granules. Solid urea is formed in three parallel rotary drum granulators.

Production line No. 2 was put into service in June of 1977 and is rated at 907 Mg/day (1000 tons/day) of solid urea granules. Solid urea is formed in four parallel rotary drum granulators. The urea solution process feeding line No. 2 is sized to allow production of 500 TPD of solution in addition to that required for solids formation.

EMISSION CONTROL EQUIPMENT

Emission points from each urea solution production line include the high pressure scrubber vent, the low pressure scrubber vent, and the steam jet ejector exhaust from the vacuum evaporator concentrators. The high pressure scrubber is utilized to recover ammonia from various process equipment overhead streams. The vent is necessary to remove inerts from the overall process.

^aNote 3 The scrubber liquor is carbamate recycle. The low pressure vent on the total recycle part of the plant vents ammonia, water, and inerts. The steam jet ejectors that maintain a vacuum on the vacuum evaporators also emit ammonia, water, and inerts. There are no controls on these three emission points other than standard process stream controls on a total recycle plant.

The exhaust air from each granulator is ducted to separate
^bNote 4 scrubbers ^cNote 5 On line No. 1, each scrubber exhausts through its own stack.

^aNote 3 - See Item 3, Confidential Addendum, Contact Eric Noble, EPA (919) 541-5213.

^bNote 4 - See Item 4, Confidential Addendum, Contact Eric Noble, EPA (919) 541-5213.

^cNote 5 - See Item 5, Confidential Addendum, Contact Eric Noble, EPA (919) 541-5213.

PROCESS OPERATION

In order to determine whether production line 1 was operating at representative steady-state conditions during testing, various process and control equipment operating parameters were monitored.

PRODUCTION RATES

Production rates were calculated using the flow rate as measured by magnetic volumetric flow meters. These meters measured the urea melt to the rotary drum granulators in conjunction with the insitu density of the urea melt (1.2 gm/cc).¹ As a check, totalizer readings on the product conveyor weigh belt were also monitored. Average product rates for the B granulator during emissions tests of its scrubber are presented in Table 3-1. Average product rates of the solution production equipment during emissions testing of this equipment are presented in Table 3-2. These were calculated using the totals of the feed rates to all three granulators.

PRODUCTION AND CONTROL EQUIPMENT OPERATION

In addition to the process rate parameters mentioned above, many other parameters were monitored to insure representative steady-state process operation. During testing of B granulator emissions, inlet and outlet temperatures of the urea and air streams through the B granulator were monitored, as was the inhibitor addition rate. The ammonia feed rate for production line 1 was also monitored. For the B granulator scrubber, liquor level, temperature, outlet concentration, and inlet and outlet flow rates were monitored.

TABLE 3-1 GRANULATOR B FEED RATE DURING GRANULATION EMISSIONS TESTING

Test	Granulator B Feed Rate
Scrubber Inlet and Outlet Test No. 1	...
Scrubber Inlet and Outlet Test No. 2	...
Scrubber Inlet and Outlet Test No. 3	...
Scrubber Inlet Particle Size Test No. 1	...
Scrubber Inlet Particle Size Test No. 2	...
Scrubber Inlet Particle Size Test No. 3	...

See
Note 6^a

^aNote 6 - See Item 6, Confidential Addendum, Contact Eric Noble, EPA (919) 541-5213.

TABLE 3-2 SOLUTION FORMATION RATE FOR PRODUCTION LINE 1 DURING SOLUTION FORMATION EMISSIONS TESTING

Test	Output as 100 Percent
1	...
2	...
3	...

See
Note 7^a

^aNote 7 - See Item 7, Confidential Addendum, Contact Eric Noble, EPA (919) 541-5213.

During emissions testing of the solution production equipment, CO₂ compressor rate and vent scrubber overheads pressure (T105) were monitored in addition to the above parameters.

Relative average values and relative standard deviations of all the above parameters monitored during the emissions tests are shown in Table 3-3. A value of 100 percent for a parameter represents the exact arithmetic average of all values of that parameter monitored during all tests. Granulator and scrubber parameters were monitored during the particle size and solution tower tests, and these values were incorporated into the relative averages and standard deviations shown for the granulator scrubber inlet and outlet tests. Standard deviations are not presented for tests where the number of readings was three or less. Actual values of monitored parameters are presented in Appendix I.

"B" GRANULATOR AND SCRUBBER OPERATION

Average temperatures of the air inlet and outlet streams and the urea outlet stream to the granulator show considerable variation between test periods. The difference between average temperatures of a given stream for two test periods often exceeds the standard deviations of the temperatures during the two tests. The effect of these variations on the particulate concentration entering the 'B' granulator scrubber cannot be predicted. Inhibitor addition rate was gradually reduced between January 17, when the first scrubber air inlet and outlet tests were made, and January 18, when the last tests were made. It was maintained at a constant value during particle size characterization tests, although this rate was somewhat lower than the rates used during scrubber inlet and outlet tests.

TABLE 3-3 RELATIVE AVERAGE VALUES OF OPERATING PARAMETERS DURING TESTING

PARAMETER	Granulator Scrubber Inlet and Outlet Tests				Particle Size Tests				Solution Tower Tests			
	1/17, 1200	1/17, 1545	1/17, 1545-1800	1/18, 0945-1217	1545-1800	1/18, 1618-1630	1/18, 1630-1645	1/18, 1645-1655	1/18, 1655-1700	1/18, 1700-1715	1/18, 1715-1730	1/18, 1730-1745
	Avg.	Std. Dev.	Avg.	Std. Dev.	Avg.	Std. Dev.	Avg.	Std. Dev.	Avg.	Std. Dev.	Avg.	Std. Dev.
A Production Rate Parameters												
Urea Melt Feed to Granulator A	100	0.9	83	0.6	111	0.7	113	100	111	100	100	100
Urea Melt Feed to Granulator B	101	0.8	100	0.0	97	0.7	98	100	98	100	100	100
Urea Melt Feed to Granulator C	105	0.5	90	0.0	108	0.5	105	94	102	94	94	94
Total Solids from A, B, and C	100	-	97	-	104	-	119	98	80	97	101	102
B Granulator Operating Parameters												
Air Inlet Temperature	101	1.6	100	1.7	92	1.5	92	100	111	100	100	100
Air Outlet Temperature	100	0.3	103	0.6	98	0.5	98	100	98	100	100	100
Urea Inlet Temperature	100	0.7	100	0.6	100	0.3	100	94	102	94	94	94
Solids Outlet Temperature	101	1.7	99	0.7	94	0.7	94	98	80	97	101	102
Formaldehyde Addition Rate	106	4.7	104	0.0	100	3.3	100	98	97	101	101	101
Scrubber Operating Parameters												
Liquor Level	101	1.0	101	1.0	101	1.0	101	98	98	99	99	99
Liquor Temperature	99	0.5	104	0.5	99	0.0	99	99	102	99	99	99
Liquor Feed Rate	105	0.0	107	5.6	111	9.2	111	99	101	99	99	98
Liquor Blowdown Rate	70	68.	120	63.	136	136.	136	98	97	101	101	101
Blowdown Urea Content	101	0.8	98	2.0	100	4.2	100	98	97	101	101	101
Solution Production Operating Parameters												
Ammonia Feed Pump Rate (RPM)	100	1.1	102	1.0	100	2.1	100	99	98	99	99	99
CO ₂ Compressor Rate (SCFH)									102	99	99	99
T105 Overheads Pressure									101	94	94	98
Solution Feed to Evaporator									97	101	101	101

Scrubber liquor level was maintained constant during tests of scrubber air outlet. There was some variation of average liquor feed and blow-down rates, temperature, and concentration between the three tests, as can be seen in Table 3-4. The effect of variations in these last three parameters on emissions cannot be predicted.

SOLUTION PRODUCTION EQUIPMENT OPERATION

Average values of the ammonia feed pump RPM, CO₂ compressor rate, and urea solution feed to the evaporators were fairly uniform for the various test periods. Vent scrubber overheads pressure also showed little variation.

TABLE 3-4 SCRUBBER OPERATING PARAMETERS DURING SCRUBBER
AIR INLET AND OUTLET PARTICULATE LOADING TESTS

Test Number	See Note 8 ^a
1	
2	See Note 8 ^a
3	

^aNote 8 - See Item 8, Confidential Addendum, Contact Eric Noble,
EPA (919) 541-5213.

On the morning of January 18, the plant was having trouble maintaining the correct ammonia to CO₂ feed ratio. No testing of solution formation equipment was conducted that morning, and the feed ratio problem is not expected to have affected emissions from solids formation.

REFERENCES

1. Letter to T. Curtin, GCA/Technology Division, from M. Dial, Vice President, Manufacturing, Louisiana Region, CF Industries, Inc., Donaldsonville, Louisiana, May 28, 1979.

SECTION 4

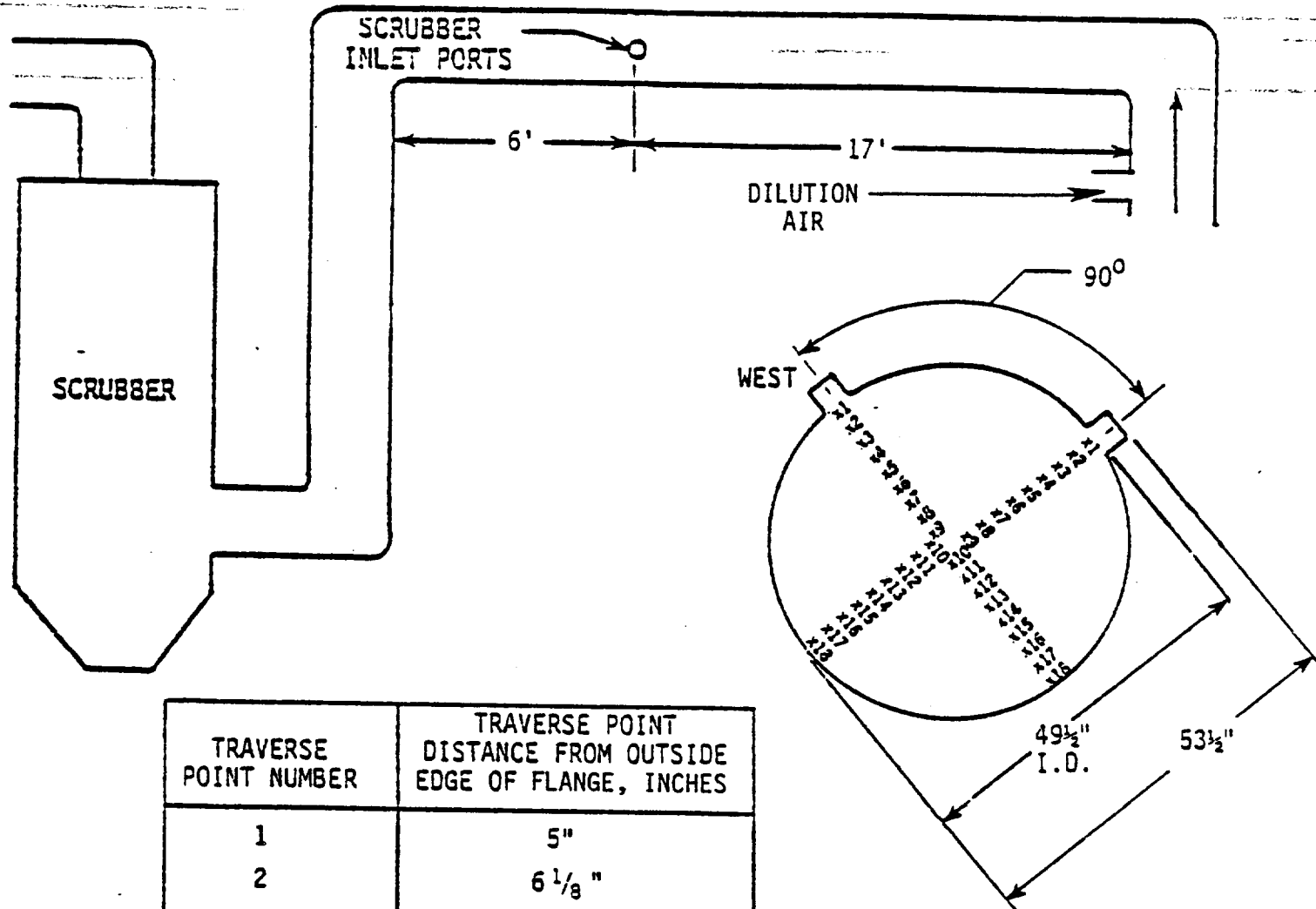
LOCATION OF SAMPLING POINTS

Testing was conducted on the "B" Granulator Scrubber and the Synthesis Tower Vent at the CF Industries, Inc., urea plant in Donaldsonville, Louisiana. This section presents the detailed descriptions of the sampling locations for the urea, ammonia, formaldehyde, particle size and the opacity measurements.

SCRUBBER INLET, TP-1

The scrubber inlet sampling site was located in a 49 1/2 inch I.D. horizontal section of steel duct. A schematic of the sampling site including the traverse point sampling locations and duct dimensions is presented in Figure 4-1. Two 4-inch pipe-flange sampling ports positioned 90° apart were located 6 feet (1.45 duct diameters) upstream of a short radius right-angle bend. The distance from the ports to the nearest upstream disturbance (another right-angle bend) was 17 feet (4.12 duct diameters).

This inlet sampling location did not meet the "eight and two diameter" criterion as outlined in EPA Method 1; consequently 18 sampling points were chosen for each axis traverse for a total of 36 sampling points as specified in the method. Figure 4-1 shows a cross sectional view of the duct at the sampling location and lists the exact distance of each traverse point from the outside flange edge.



TRAVERSE POINT NUMBER	TRAVERSE POINT DISTANCE FROM OUTSIDE EDGE OF FLANGE, INCHES
1	5"
2	6 1/8 "
3	7 3/4 "
4	9 3/8 "
5	11 1/4 "
6	13 1/4 "
7	15 5/8 "
8	18 5/8 "
9	22 7/8 "
10	34 5/8 "
11	38 7/8 "
12	41 7/8 "
13	44 1/4 "
14	46 1/4 "
15	48 1/8 "
16	49 3/4 "
17	51 3/8 "
18	52 1/2 "

FIGURE 4-1:

LOCATIONS OF "B" GRANULATOR SCRUBBER INLET
TEST PORTS AND POINTS AT CF INDUSTRIES INC.,
DONALDSONVILLE, LOUISIANA

SCRUBBER OUTLET, TP-2

The cleaned gases exiting the scrubber unit are ducted to an induced draft fan adjacent to the scrubber system. The fan discharge is directed vertically through a steel stack to the atmosphere.

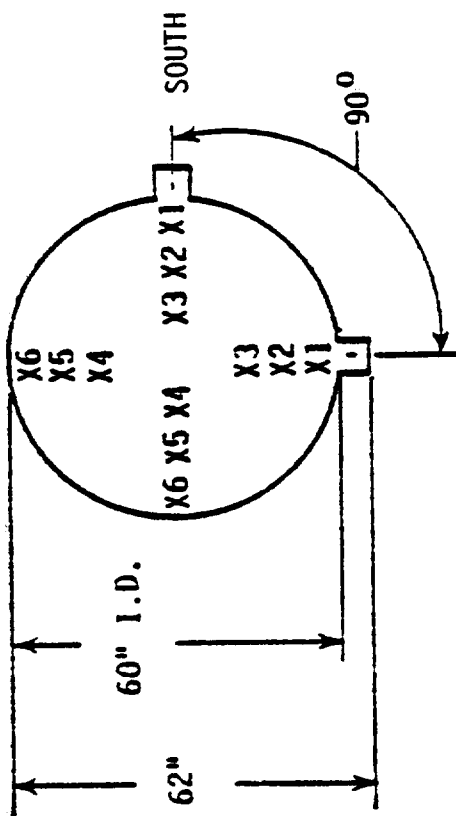
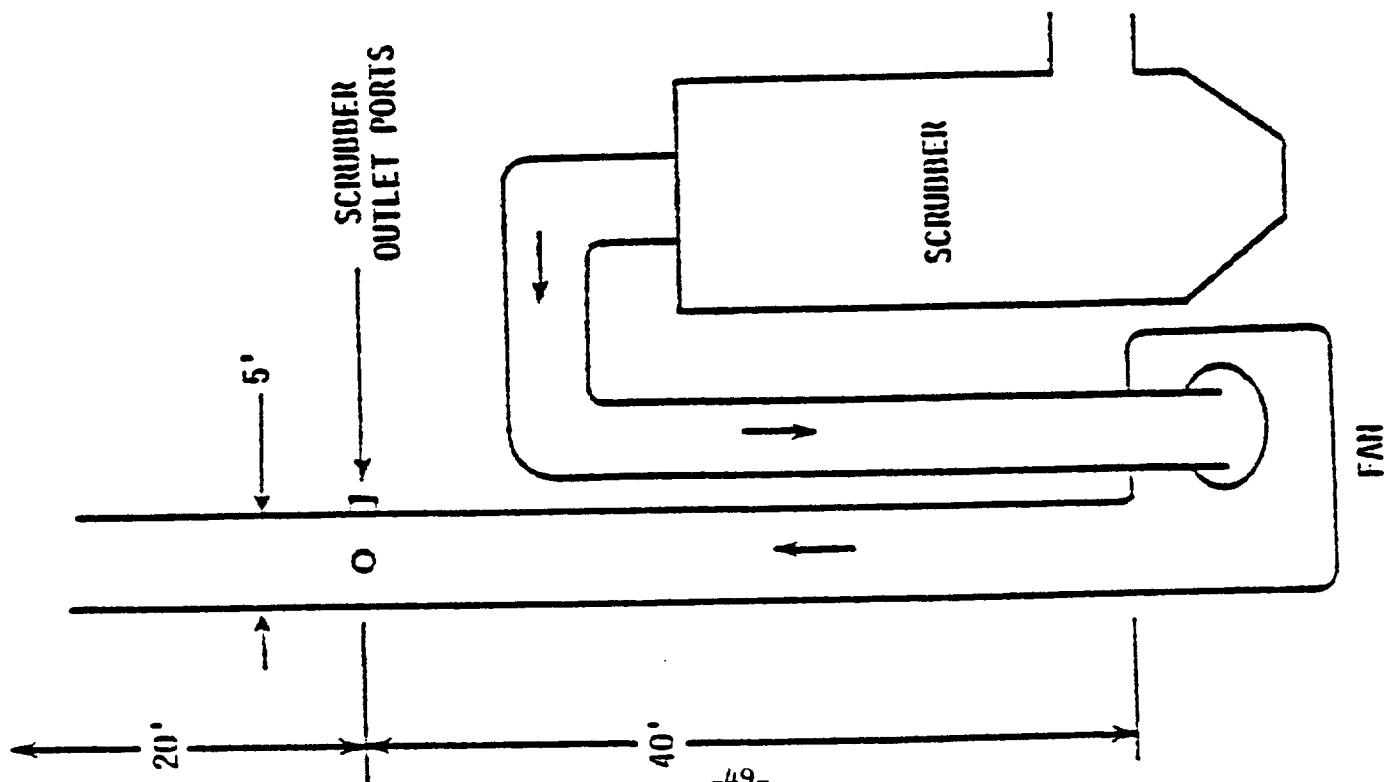
The "B" scrubber 60 inch I.D. outlet stack was fitted with two 3-inch pipe flange sampling ports positioned 90° apart in a horizontal plane. The two ports were located 40 feet (8 stack diameters) downstream from the fan discharge, and 20 feet (4 stack diameters) upstream of the top of the stack. See Figure 4-2. The port locations met the "eight and two diameter" criterion as delineated in EPA Method 1; consequently 6 sampling points were used for each axis traverse for a total of 12 sampling points as specified in the method.

INLET PARTICLE SIZE SAMPLING LOCATION, TP-1

Particle sizing test runs were performed in the "B" granulator scrubber inlet gas stream. An in-stack cascade impactor was positioned in the duct through the test ports used for the emissions tests. The impactor nozzle was located at the geometric center of the duct for each impactor run. Only this one central sampling point was used in order to maintain isokinetic sampling, per standard procedures established by the cascade impactor manufacturer.

VISIBLE EMISSIONS OBSERVATION LOCATIONS

The white scrubber stack plume was observed from a position 90 feet above ground level and about 10 feet above the top of the stack. Observations were made looking north to the stack, 150 feet away. The plume was observed against a background consisting of a railroad bed and the ground nearby. This position was



TRAVERSE POINT NO.	TRAVERSE POINT DISTANCE FROM OUTSIDE EDGE OF NIPPLE (INCHES)
1	4 ⁵ / ₈ "
2	10 ³ / ₄ "
3	19 ³ / ₄ "
4	44 ¹ / ₄ "
5	53 ¹ / ₄ "
6	59 ³ / ₈ "

FIGURE 4-2:
LOCATIONS OF "B" GRANULATOR SCRUBBER OUTLET
TEST PORTS AND POINTS AT CF INDUSTRIES, INC.,
DONALDSONVILLE, LOUISIANA

selected to conform to the EPA Method 9 requirements. The location of the smoke observer is shown in Figure 4-3.

SCRUBBER PRESSURE DROP MEASUREMENT LOCATIONS

Pressure drop across the "B" granulator scrubber was measured with a vertical U-tube water manometer which was connected to pressure taps at the scrubber inlet and outlet. The inlet pressure tap was located in the inlet duct 10 feet upstream of the scrubber. The outlet pressure tap consisted of a hole drilled through the transition ducting between the scrubber outlet and the inlet of the fan.

SCRUBBER LIQUID COLLECTION LOCATIONS

Scrubber solution samples were collected from the streams entering and leaving the "B" granulator scrubber. See Figure 4-4. The inlet sample was tapped from the solution line just before it entered the scrubber. The outlet sample was tapped from the pump discharge.

PROCESS SAMPLE COLLECTION LOCATIONS

Throughout the testing program, process samples were collected directly from their applicable process units/operations. These consisted of samples of urea melt and the granulator unscreened product.

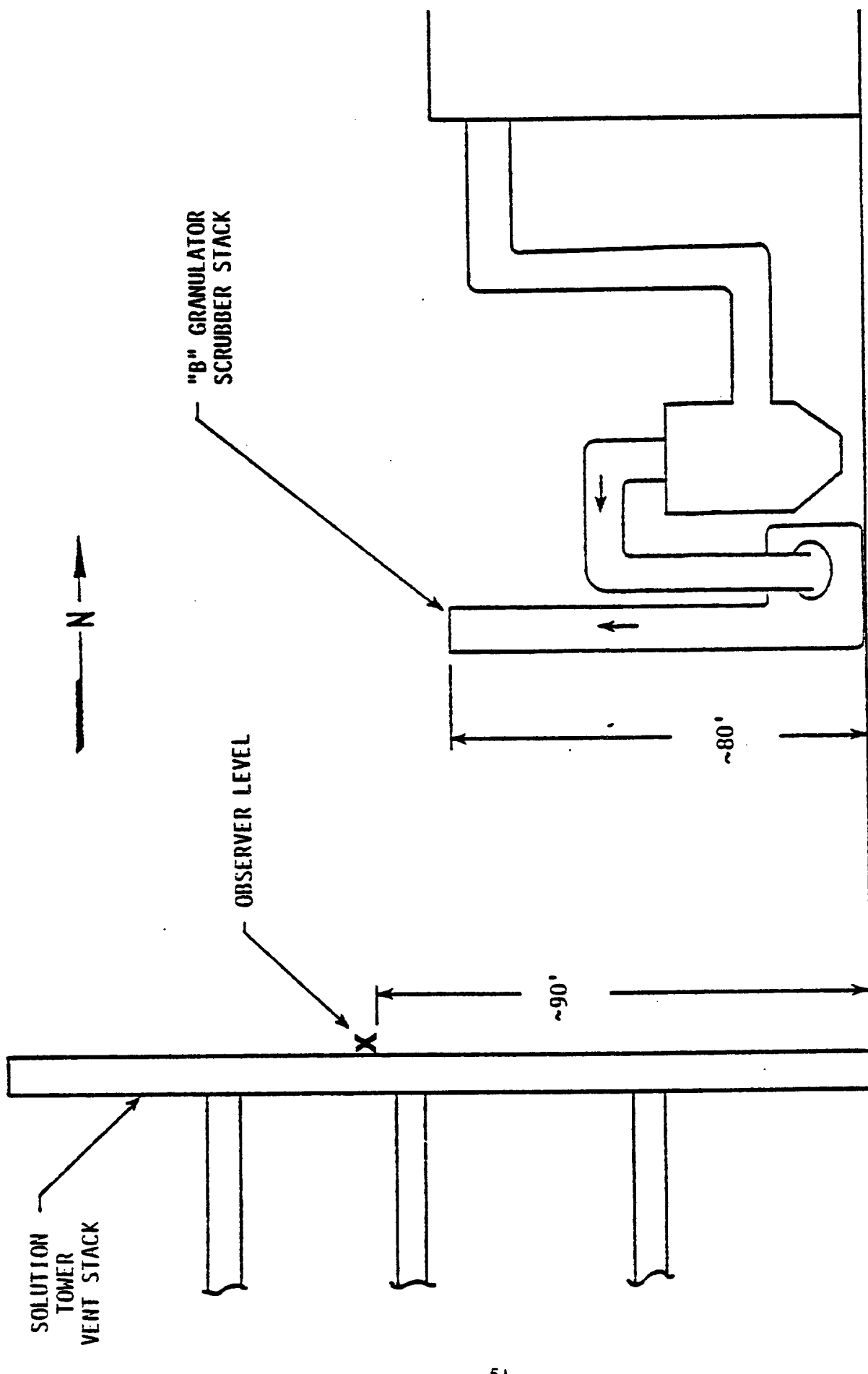


FIGURE 4-3:
LOCATION OF SMOKE OBSERVER FOR JANUARY 17 - 19,
1979 OPACITY READINGS ON "B" GRANULATOR SCRUBBER
STACK AT CF INDUSTRIES INC.,
DONALDSONVILLE, LOUISIANA

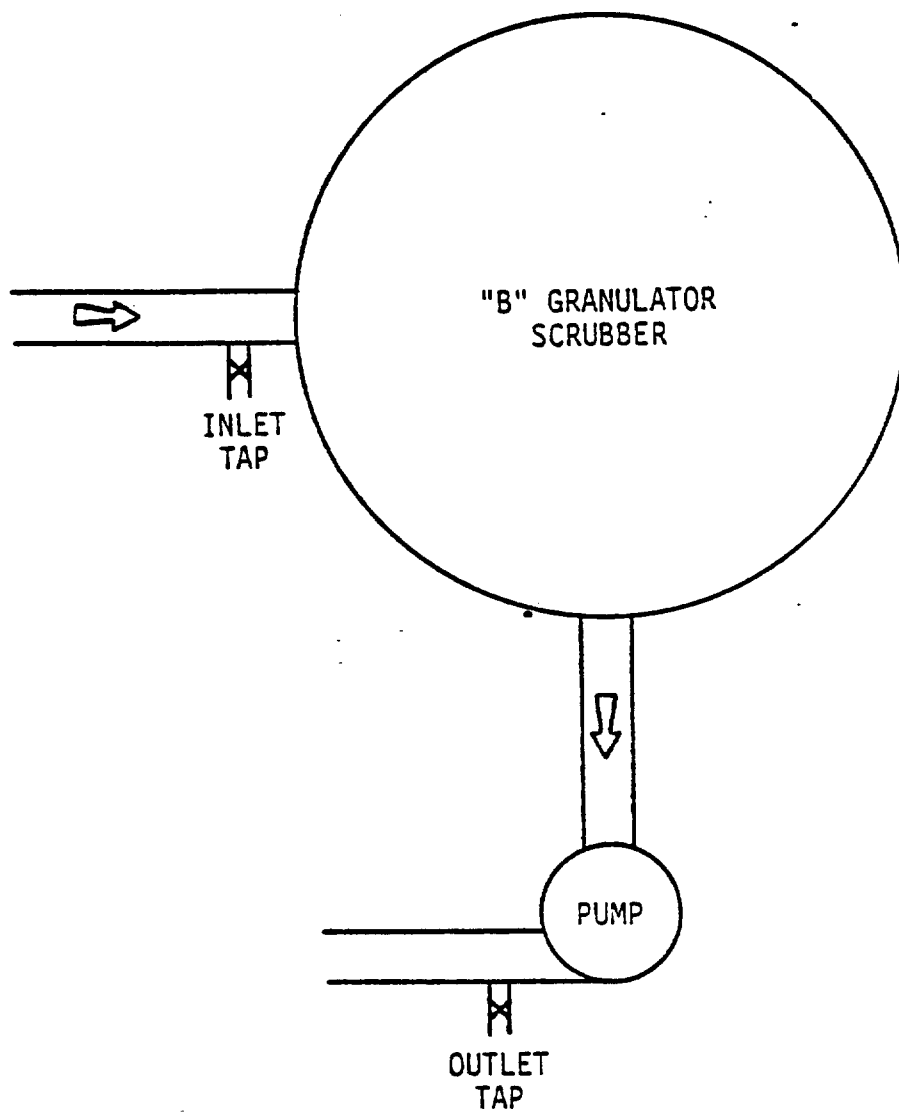


FIGURE 4-4:

LOCATIONS OF SCRUBBER LIQUID COLLECTION
TAPS FOR JANUARY 17 - 19, 1979 TESTS ON
"B" GRANULATOR AT CF INDUSTRIES INC.,
DONALDSONVILLE, LOUISIANA

UREA SYNTHESIS TOWER VENT LOCATION

The urea synthesis tower sampling site was located in a 14 inch I.D. vertical section of heavy gauge stainless steel pipe. A schematic of the sampling site is presented in Figure 4-5. One 6 inch I.D. pipe flange sampling port was positioned approximately 16 feet (13.7 stack diameters) upstream of a connecting exhaust vent. The distance between the sampling point and the stack vertex was approximately 30 feet (25.7 stack diameters). The port location met the EPA Method 1 criterion. The use of an in-stack orifice in the 14 inch I.D. stack restricted the sampling to a single point. The nozzle was positioned at its centroid of the stack for the three runs.

AMBIENT AIR TEMPERATURE AND RELATIVE HUMIDITY MEASUREMENT LOCATION

The ambient air temperature and relative humidity measurements were made on the ground next to the CFI chemical laboratory, within 300 yards of the "B" Granulator.

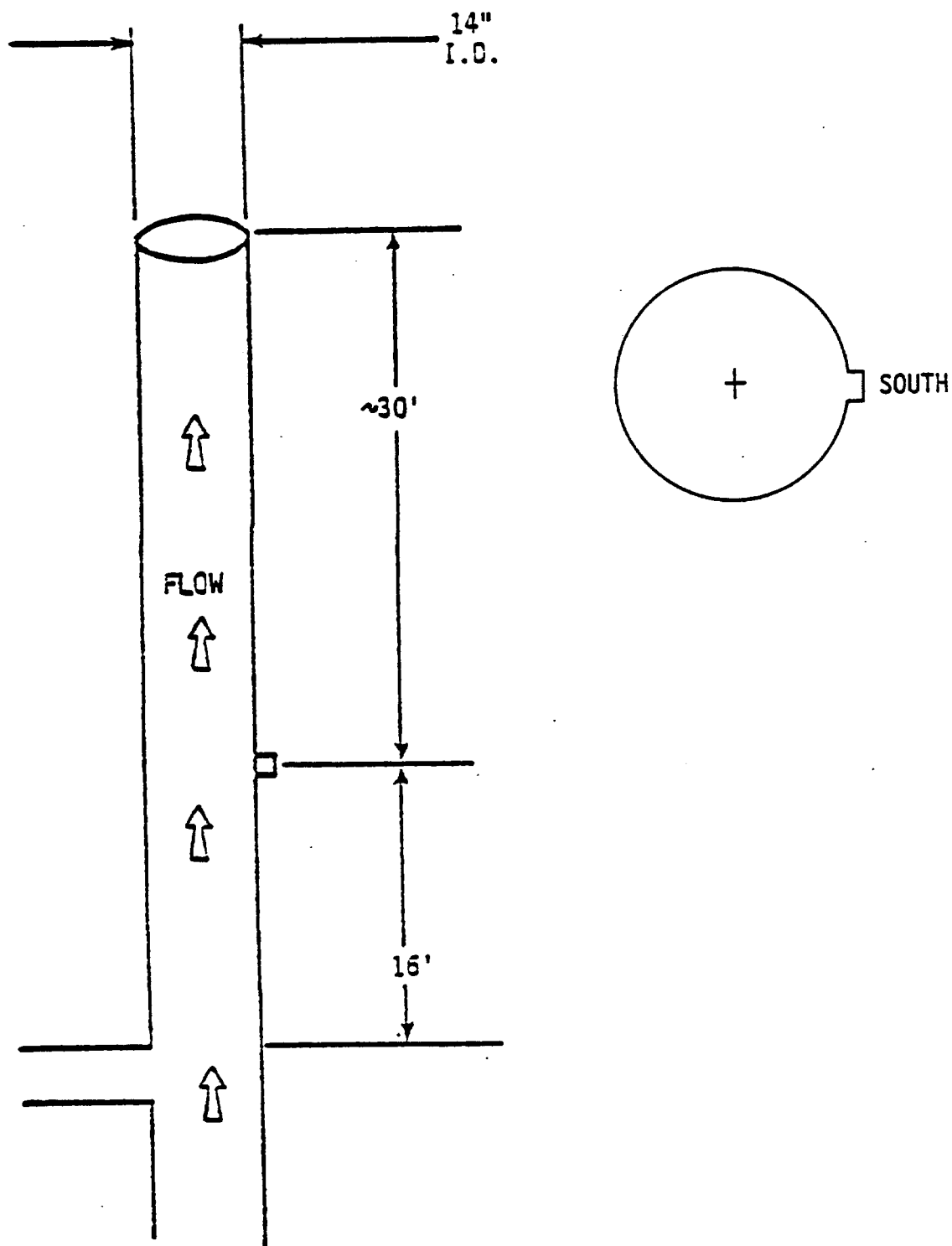


FIGURE 4-5:

LOCATION OF SYNTHESIS TOWER SOLUTION
VENT SAMPLING PORT AT CF INDUSTRIES INC.,
DONALDSONVILLE, LOUISIANA

SECTION 5

SAMPLING AND ANALYSIS METHODOLOGIES

This section presents the description of the sampling and analysis methodologies employed at the CF Industries, Inc. urea manufacturing facility in Donaldsonville, Louisiana during January 15-19, 1979. Sampling and Analysis Methodologies are categorized as:

- A - Urea, Formaldehyde, and Ammonia "B" Granulator Scrubber
- B - Visible Emissions "B" Granulator Scrubber
- C - Particle Size "B" Granulator System Inlet
- D - Scrubber Liquid "B" Granulator
- E - Process Samples "B" Granulator
- F - Urea and Ammonia Synthesis Tower Main Vent

The EPA designated methods and any deviations from these methods are contained in Appendices J and K. This section presents general descriptions of the methods along with discussions of problems encountered during the test program.

UREA, FORMALDEHYDE, AND AMMONIA METHODS USED ON "B" GRANULATOR SCRUBBER

Urea, formaldehyde, and ammonia in the "B" Granulator Scrubber inlet and outlet gas streams were sampled at points identified by EPA Method 1 in accordance with the relationship of the sampling ports to upstream and downstream flow disturbances. The velocity of the duct gas was measured using calibrated S-type pitot tubes in accordance with EPA Method 2. Construction and calibration of the S-type pitot tubes was consistent with EPA Method 2. The complete methods for sampling and analysis for urea, formaldehyde and ammonia are contained in Appendix K.

The sampling train is shown schematically in Figure 5-1 and consists of nozzle, probe, Teflon line, six impingers, vacuum pump, dry gas meter, and an orifice flow meter. The nozzle (1) is stainless steel and is of buttonhook shape. It was connected to a 5/8" stainless steel glass lined probe (2) that is wrapped with nichrome heating wire and jacketed. The probe temperature was maintained at approximately 190°F at the inlet and approximately 110°F at the outlet in order to prevent condensation of the sampled gas. Following the probe, the gas stream passed through a 3/8" I.D. Teflon line (3) into an ice bath/impinger system (4).

The first and second impingers in the ice bath contained deionized distilled water (100 ml), the third and fourth contained 100 ml of 1N H₂SO₄ each, the fifth was dry, and the sixth contained silica gel (200 grams) to remove any remaining moisture. Leaving the last impinger, the sample stream flowed through flexible tubing (5), a vacuum gauge (6), needle valve (7), pump (8), and a dry gas meter (9). A calibrated orifice and inclined manometer (10) complete the sampling train. The stack velocity pressure was measured using a pitot tube (11) and inclined manometer. Stack temperature was monitored by a thermocouple attached to the probe and connected to potentiometer (12). A nomograph was used to rapidly determine the orifice pressure drop required for any pitot velocity pressure and stack temperature in order to maintain isokinetic sampling conditions.

The recorded test data included test time, sampling duration at each traverse point, pitot pressure, stack temperature, meter volume, meter inlet-outlet temperature and orifice pressure drop. At completion of each run, the nozzle and probe contents were tripled washed and brushed into a glass sample jar. Next, the Teflon line between the probe and first impinger was also rinsed three times with distilled, deionized water into the same glass sample jar which was then sealed

with a Teflon lined cap. The sample jars and impingers were returned to the sample train prep-and-clean-up room.

At the same prep-and-clean-up room, the contents of the impingers were transferred to tare-weighed sample jars. The sample jars were used as follows:

- Jar #1 - contents of the first and second water impingers, the water wash of these impingers and their connecting glassware, and the nozzle, probe and teflon line washes.
- Jar #2 - contents of third and fourth acid impingers, the water wash of these impingers, and their connecting glassware.
- Jar #3 - silica gel from the sixth impinger.

The contents of jars 1 and 2 were analyzed by a TRC chemist, Ms. Margaret Fox, at the CF Industries laboratories and also at the TRC laboratories. Ammonia concentrations were determined by two methods: 1) direct Nesslerization (on-site) and 2) preliminary distillation (on-site) followed by Nesslerization (at TRC). Urea concentrations were determined by the Kjeldahl analysis method, as follows: the portion of sample remaining after the preliminary distillation was then digested and redistilled (on-site) and then brought to TRC for completion of the urea analysis. Urea and ammonia concentrations were in all cases determined colorimetrically with a spectrophotometer at a wave length of 405 nm.

The analysis for formaldehyde, done after transporting the samples "on-ice" back to the TRC laboratories, consisted of mixing an aliquot of the sample solution with the chromotropic-sulfuric acid reagent to form a purple chromogen. This colored solution was analyzed colorimetrically using a spectrophotometer at 580 nm.

In all these analyses, standard solutions were prepared and analyzed, and calibration curves of absorbance vs. concentration were drawn. These calibration curves were used to determine the urea, formaldehyde and ammonia concentrations of the samples. The sample concentration indicated by the calibration curve was multiplied by the sample volume to determine the total mass of urea, formaldehyde or ammonia collected.

The distillation-and-Nesslerization analysis method was done in addition to the direct-Nesslerization method for ammonia analysis because of potential interference from several species (including formaldehyde and urea) on ammonia concentrations determined by the direct Nesslerization method.¹ But there also exists evidence that about 7 percent of any urea in a sample is converted to ammonia during distillation.¹ Uncertainty in the exact mechanism and rate of urea conversion, however, leads to uncertainty in indicated ammonia concentrations, especially so in samples with high urea concentrations. This is evident in the analyses of the gases entering the "B" granulator scrubber (Table 2-2) and the liquid exiting this scrubber (Table 2-7). In these cases, the application of the standard 7 percent correction factor yielded negative "corrected" ammonia concentrations. Because of the uncertainty in the urea conversion factor, more confidence is placed in the direct-Nesslerization results.

The main sampling problem during these scrubber gas test runs occurred at the scrubber inlet. The large size and high concentration of the urea particles at this location caused plugging of the pitot, nozzle and probe. Plugs were removed from the pitot by pumping air via a purge line through the pitot tube side facing

¹Standard Methods for the Examination of Water and Wastewater, 14th Edition, APHA, AWWA, WPCF, 1975, p. 408.

upstream; this purge line would then be removed and the pitot reconnected to the manometer. The frequent nozzle and probe plugging required shutting down the sampling train and cleaning the nozzle and probe. Cleaning was quickly accomplished by squirting water into the nozzle to dissolve the plug, as recommended by the EPA Technical Manager. Plugging was minimized by inserting the probe into the duct with the nozzle facing downstream. The probe was then rotated 180 degrees into the flow stream immediately before the initiation of sampling. The probe was removed from the duct in reverse order. In addition, the largest possible nozzle was used as a precaution to minimize plugging problems.

The water used to clean the nozzle and probe did, however, contribute to the total volume of water collected by the impingers during the inlet test runs. For this reason and because of the large mass of urea collected at the inlet, a separate test run was performed at the scrubber inlet to determine the moisture content of the scrubber inlet gas stream. Details of this moisture run and calculations are contained in Appendix A.

VISIBLE EMISSIONS METHODS USED ON "B" GRANULATOR

The visible emissions measurements were conducted by a certified visible emission evaluator in accordance with EPA Reference Method 9. The readings were taken at 15 second intervals. Since the plume was white, it was necessary to read the emissions against a dark background. The dark background selected was the railroad tracks and the dark dirt and rock ground surface in that area.

PARTICLE SIZE METHODS USED ON "B" GRANULATOR INLET

A Sierra Model 266 multi-stage cascade impactor was operated in its in-stack mode. Sampling was performed isokinetically from a single point at the center of

the scrubber inlet duct. Prior to the initiation of sampling the impactor was leak tested and placed in the duct for 20 minutes to allow it to heat to duct temperature to prevent condensation. Sampling was initiated immediately upon rotation of the nozzle into the flow stream. The brief sampling time at the inlet necessitated presetting the sample valve so that when the nozzle was pointed into the flow stream and the pump started, only a minimal adjustment of the valve was necessary to achieve isokinetic flow.

The impactor was loaded before each run with preweighed glass fiber collection substrates. After sampling the impactor was removed to the clean up room and the substrates placed in plastic petri dishes and sealed. The cyclone contents were brushed into a sample jar and sealed. These samples were weighed on an analytical balance to 0.1 mg. Additional information on the particle sizing is presented in Appendix B.

SCRUBBER LIQUID SAMPLING METHODS USED ON "B" GRANULATOR

Half-liter samples of the scrubbing liquid streams entering and exiting the "B" granulator scrubber were collected at approximately 30 minute intervals during the granulator scrubber tests. Sample temperatures were recorded immediately upon collection. The pH of each sample was measured using a pH meter.

All the individual samples collected during a run were combined into one inlet and one outlet sample. The composite samples were analyzed for urea, ammonia, and formaldehyde concentration in accordance with the procedure described in Appendices J and K.

PROCESS SAMPLING AND PRODUCT ANALYSIS

Grab samples of the urea melt and unscreened product were collected at their respective locations in the process.

The urea melt and unscreened product samples were prepared for analysis by dissolving a known weight (approximately 1.0g) into 100 ml of distilled deionized water. The solution was then analyzed for urea, ammonia and formaldehyde using the procedures previously described above and delineated in Appendices J and K. A portion of each of the unscreened product samples were also subjected to bulk density and sieve analyses. Six sieve sizes were used in the sieve analysis, and the mass of material required to fill a 250 ml volume was measured for the bulk density analysis.

UREA AND AMMONIA SAMPLING METHODS USED ON SYNTHESIS TOWER MAIN VENT

Urea and ammonia in the Synthesis Tower Main Vent Stack were sampled and analyzed using the methodology and equipment described in the prior subsection entitled Urea and Ammonia "B" Granulator with the following modifications:

1. An in-stack orifice was used to permit isokinetic sampling of a stream with a moisture content greater than 50%. The orifice measures the sample stream at the same moisture conditions as exist in the stack and therefore changes in moisture will not affect isokinetic sampling.
2. A filter was not used in the sampling train.
3. Two extra impingers were added to achieve complete condensation of the moisture and collection of ammonia. Impingers one through three contained 100 ml each of distilled deionized water, impingers four through six each contained 100 ml of 10 N H_2SO_4 , impinger seven was empty, and impinger eight contained silica gel. The 10 N H_2SO_4 was necessary because of the high ammonia concentration and served to trap ammonia by condensation and neutralization.
4. Only one sampling point (at center of stack) was used because of physical restrictions imposed by the in-stack orifice nozzle.

The total volume of water collected in these synthesis tower test runs consisted of water condensed from vapor and liquid water droplets that were extracted from the gas stream. The existence of liquid water in the gas stream was confirmed by observation and by the data shown in Appendix F. These data show that the total volume of water collected during each run exceeds the volume of water present in a saturated gas stream at the stack temperatures.

PRESSURE DROP MEASUREMENTS ACROSS "B" GRANULATOR SCRUBBER

The pressure drop measurements across the "B" granulator scrubber were made with a vertical U-tube water manometer which was connected to pressure taps at the scrubber inlet and outlet. The pressure drop across the scrubber was recorded at 5 to 15 minute intervals during the tests for urea, ammonia and formaldehyde at the B granulator scrubber.

AMBIENT AIR TEMPERATURE AND RELATIVE HUMIDITY MEASUREMENTS

Ambient air temperature and relative humidity measurements were taken at the CFI chemical laboratory with a Bendix psychron at 15 to 30 minute intervals during each test run.

APPENDIX A

COMPUTER PRINTOUT TEST RESULTS WITH EQUATIONS AND EXAMPLE CALCULATIONS

A-1 Computer Printout Test Results

1. Inlet to "B" Granulator Scrubber (TP-2)
2. Outlet From "B" Granulator Scrubber (TP-2)

A-2 Sample Equations and Example Calculations

APPENDIX A-1

COMPUTER PRINTOUT TEST RESULTS

1. Inlet to "B" Granulator Scrubber (TP-1)
2. Outlet from "B" Granulator Scrubber (TP-2)

INLET TO "B" GRANULATOR SCRUBBER (TP-1)

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 1
TRC PROJECT 22988-20

UNIT TESTED UREA GRANULATOR SCRUBBER 9
DATE AND TIME OF TEST JANUARY 17, 1979 1200-1423 HRS
SAMPLING LOCATION INLET DUCT
NAME OF FIRM C. F. INDUSTRIES
LOCATION OF FIRM DONALDSONVILLE, LA
POLLUTANTS SAMPLED UREA, FORMALDEHYDE, AMMONIA

VOLUME OF WATER IN IMPINGERS BACK CALCULATED FROM SEPARATE MOISTURE RUN DETERMINATION BECAUSE LAB UREA AND AMMONIA ANALYSTS DATA FOR THE TEST RUNS IS NOT APPLICABLE TO THE CORRECTION OF THE IMPINGER WEIGHT GAIN DATA FOR THESE TESTS.

BAROMETRIC PRESSURE, IN HG 30.39
DUCT AREA, SQ FT 13.36
NOZZLE DIAMETER, IN 0.188
PITOT CALIBRATION COEFFICIENTS 1 0.820
2 0.834
3 0.823
DRY GAS METER CALIBRATION FACTOR, Y 1.000
FINAL LEAK RATE, CFM 0.002
TONS PER HOUR, PRODUCT 0.000

COMPOSITION OF DUCT GAS, % BY VOLUME DRY BASIS
CARBON DIOXIDE 0.00
OXYGEN 20.50
CARBON MONOXIDE 0.00
NITROGEN 79.50

UREA, AMMONIA, FORMALDEHYDE COLLECTED, MG

	UREA	AMMONIA-DIR	AMMONIA-DIST	FORMALDEHYDE
420 IMPINGERS	0.2390E 05	0.3020E 03	0.7780E 03	0.2353E 01
42504 IMPINGERS	0.1500E 02	0.1390E 03	0.1100E 03	0.4260E 00
TOTAL	0.2392E 05	0.4400E 03	0.8880E 03	0.2476E 01

AMOUNT OF WATER COLLECTED, GRAMS
IMPINGERS 19.1
SILICA GEL 11.9

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 1
TRC PROJECT 82989-2C

POINT	TIME	VEL HEAD IN H2O	ORIFICE PRESS IN H2O	METER INLET DEG F	TEMP EXIT DEG F	DUCT STAT PRESS IN H2O	DUCT TEMP DEG F	INITIAL METER VOL CU FT	P R	CYC AVG S
1	3.0	0.3800	0.3900	72.	71.	-2.30	183.	123.64	1	0.
2	3.0	0.5400	0.5500	73.	71.	-2.30	189.	121.82	1	0.
3	3.0	0.5300	0.5400	72.	73.	-2.30	195.	123.05	1	0.
4	3.0	0.9800	0.9900	72.	73.	-2.30	180.	124.28	1	0.
5	3.0	1.0500	1.0800	71.	73.	-2.30	186.	126.35	1	0.
6	3.0	1.3000	1.3300	71.	73.	-2.30	196.	128.05	1	0.
7	3.0	1.3500	1.4000	71.	73.	-2.30	196.	129.72	1	0.
8	3.0	1.3500	1.4000	71.	73.	-2.30	196.	131.91	1	0.
9	3.0	1.4000	1.4200	71.	73.	-2.30	195.	133.87	1	0.
10	3.0	1.2500	1.2800	71.	73.	-2.30	196.	135.81	1	0.
11	3.0	1.1500	1.1800	72.	74.	-2.30	197.	137.69	1	0.
12	3.0	1.1000	1.1100	72.	73.	-2.30	197.	139.50	1	0.
13	3.0	1.1500	1.1800	72.	73.	-2.30	196.	141.22	1	0.
14	3.0	1.1500	1.1800	72.	74.	-2.30	197.	143.00	1	0.
15	3.0	1.1500	1.1800	72.	74.	-2.30	197.	144.77	1	0.
16	3.0	1.2000	1.2100	72.	74.	-2.30	197.	146.60	1	0.
17	3.0	1.1500	1.1800	72.	74.	-2.30	197.	148.37	1	0.
18	3.0	1.1000	1.1200	72.	74.	-2.30	196.	150.15	1	0.
1	3.0	0.5200	0.5300	72.	73.	-2.70	153.	151.72	1	0.
2	3.0	1.0000	1.0000	71.	73.	-2.70	153.	153.36	1	0.
3	3.0	1.1500	1.1800	71.	73.	-2.70	180.	155.04	1	0.
4	3.0	1.2000	1.2200	71.	73.	-2.70	195.	156.77	1	0.
5	3.0	1.2000	1.2200	71.	72.	-2.70	196.	158.58	1	0.
6	3.0	1.2000	1.2200	71.	72.	-2.70	196.	160.40	1	0.
7	3.0	1.2000	1.2200	71.	72.	-2.70	197.	162.22	1	0.
8	3.0	1.2500	1.2800	71.	73.	-2.70	196.	164.04	1	0.
9	3.0	1.2500	1.2800	71.	72.	-2.70	196.	165.91	1	0.
10	3.0	1.1000	1.1200	71.	73.	-2.70	196.	167.80	1	0.
11	3.0	1.1500	1.1800	71.	73.	-2.70	197.	169.55	1	0.
12	3.0	1.1000	1.1200	71.	73.	-2.70	196.	171.32	1	0.
13	3.0	1.1000	1.1200	71.	73.	-2.70	196.	173.07	1	0.
14	3.0	1.1000	1.1200	71.	73.	-2.70	196.	174.85	1	0.
15	3.0	1.0500	1.0800	71.	73.	-2.70	196.	176.56	1	0.
16	3.0	1.0500	1.0800	71.	73.	-2.70	196.	178.25	1	0.
17	3.0	0.9300	0.9500	71.	72.	-2.70	195.	179.94	1	0.
18	3.0	0.9300	0.9500	71.	73.	-2.70	195.	182.10	1	0.

FINAL METER VOLUME

183.17

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 1
TRC PROJECT 82983-20

C. F. INDUSTRIES
UREA GRANULATOR SCRUBBER 3 INLET DUCT
JANUARY 17, 1979 1200-1423 HRS

STANDARD CONDITION TEMPERATURE, DEG F	0.6800E 02
STANDARD CONDITION PRESSURE, IN HG	0.2992E 02
TOTAL SAMPLING TIME, MINUTES	0.1080E 03
AVERAGE SQUARE ROOT VELOCITY HEAD, IN H2O EX ² .5	0.1030E 01
AVERAGE ORIFICE PRESSURE DROP, IN H2O	0.1100E 01
AVERAGE METER TEMPERATURE, DEG F	0.7217E 02
AVERAGE DUCT STATIC PRESSURE, IN H2O	-0.2500E 01
AVERAGE DUCT TEMPERATURE, DEG F	0.1921E 03
TOTAL SAMPLE VOLUME, DACF	0.6253E 02
TOTAL SAMPLE VOLUME, DSCF	0.6318E 02
WATER VAPOR VOLUME, DSCF	0.1459E 01
MOISTURE CONTENT OF DUCT GAS, PERCENT	0.2257E 01
MOLE FRACTION DRY GAS	0.9774E 00
MOLECULAR WEIGHT - DRY STACK GAS	0.2882E 02
MOLECULAR WEIGHT - STACK GAS	0.2858E 02
AVERAGE STACK PRESSURE, IN HG	0.3021E 02
DUCT VOLUMETRIC FLOW, ACFM	0.5030E 05
DUCT VOLUMETRIC FLOW, DSCFM	0.4018E 05
AVERAGE DUCT VELOCITY, FPM	0.3765E 04
EXCESS AIR, PERCENT	0.4201E 04
AVERAGE DUCT GAS DENSITY, LBS/ACF	0.6062E-01
ISOINETIC FACTOR, PERCENT	0.1014E 03

EMISSION DATA

	GR/ ACF	GR/ DSCF	LBS/ HR
UREA IN H2O	0.4664E 01	0.5838E 01	0.2011E 04
UREA IN H2SO4	0.2927E-02	0.3663E-02	0.1262E 01
TOTAL UREA	0.4667E 01	0.5842E 01	0.2012E 04
AMMONIA-DIRECT-IN H2O	0.5873E-01	0.7375E-01	0.2540E 02
AMMONIA-DIRECT-IN H2SO4	0.2107E-01	0.2637E-01	0.9084E 01
TOTAL AMMONIA-DIRECT	0.8000E-01	0.1001E 00	0.3449E 02
AMMONIA-DISTILLED-IN H2O	0.1518E 00	0.1900E 00	0.6544E 02
AMMONIA-DISTILLED-IN H2SO4	0.2146E-01	0.2686E-01	0.9252E 01
TOTAL AMMONIA-DISTILLED	0.1733E 00	0.2169E 00	0.7469E 02
FORMALDEHYDE IN H2O	0.4000E-03	0.5006E-03	0.1724E 00
FORMALDEHYDE IN H2SO4	0.8312E-04	0.1040E-03	0.3583E-01
TOTAL FORMALDEHYDE	0.4831E-03	0.6047E-03	0.2083E 00

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 1
TRC PROJECT 92988-20

C. F. INDUSTRIES
UREA GRANULATOR SCRUBBER 3 INLET DUCT
JANUARY 17, 1979 1200-1423 HRS

STANDARD CONDITION TEMPERATURE, DEG C	0.2000E 02
STANDARD CONDITION PRESSURE, MM HG	0.7600E 03
TOTAL SAMPLING TIME, MINUTES	0.1080E 03
AVERAGE SQUARE ROOT VELOCITY HEAD, MM H2O EXP.5	0.5190E 01
AVERAGE ORIFICE PRESSURE DROP, MM H2O	0.2793E 02
AVERAGE METER TEMPERATURE, DEG C	0.2231E 02
AVERAGE DUCT STATIC PRESSURE, MM H2O	-0.6350E 02
AVERAGE DUCT TEMPERATURE, DEG C	0.9995E 02
TOTAL SAMPLE VOLUME, DM3	0.1771E 01
TOTAL SAMPLE VOLUME, DNH3	0.1789E 01
WATER VAPOR VOLUME, DNH3	0.4132E-01
AVERAGE STACK GAS PRESSURE, MM HG	0.7672E 03
DUCT VOLUMETRIC FLOW, AM3/M	0.1424E 04
DUCT VOLUMETRIC FLOW, DNH3/M	0.1138E 04
AVERAGE DUCT VELOCITY, M/M	0.1147E 04
AVERAGE DUCT GAS DENSITY, KG/AM3	0.9710E 00

EMISSION DATA

	MG/ AM3	MG/ DNH3	KG/ HR
UREA IN H2O	0.1067E 05	0.1336E 05	0.9129E 03
UREA IN H2SO4	0.6698E 01	0.8383E 01	0.5726E 00
TOTAL UREA	0.1068E 05	0.1337E 05	0.9134E 03
AMMONIA-DIRECT-IN H2O	0.1348E 03	0.1688E 03	0.1153E 02
AMMONIA-DIRECT-IN H2SO4	0.4822E 02	0.6036E 02	0.4124E 01
TOTAL AMMONIA-DIRECT	0.1931E 03	0.2291E 03	0.1566E 02
AMMONIA-DISTILLED-IN H2O	0.3474E 03	0.4348E 03	0.2971E 02
AMMONIA-DISTILLED-IN H2SO4	0.4912E 02	0.6147E 02	0.4201E 01
TOTAL AMMONIA-DISTILLED	0.3965E 03	0.4963E 03	0.3391E 02
FORMALDEHYDE IN H2O	0.9153E 00	0.1146E 01	0.7828E-01
FORMALDEHYDE IN H2SO4	0.1902E 00	0.2381E 00	0.1627E-01
TOTAL FORMALDEHYDE	0.1106E 01	0.1384E 01	0.9455E-01

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 2
TRC PROJECT 82938-20

UNIT TESTED UREA GRANULATOR SCRUBBER B
DATE AND TIME OF TEST JANUARY 17, 1979 1548-1756 HRS
SAMPLING LOCATION INLET DUCT
NAME OF FIRM C. F. INDUSTRIES
LOCATION OF FIRM DONALDSONVILLE, LA
POLLUTANTS SAMPLED UREA, FORMALDEHYDE, AMMONIA

VOLUME OF WATER IN IMPINGERS BACK CALCULATED FROM SEPARATE MOISTURE RUN DETERMINATION BECAUSE LAB UREA AND AMMONIA ANALYSIS DATA FOR THE TEST RUNS IS NOT APPLICABLE TO THE CORRECTION OF THE IMPINGER WEIGHT GAIN DATA FOR THESE TESTS.

BAROMETRIC PRESSURE, IN HG 30.39
DUCT AREA, SQ FT 13.36
NOZZLE DIAMETER, IN 0.187
PITOT CALIBRATION COEFFICIENTS 1 0.820
2 0.834
3 0.823
DRY GAS METER CALIBRATION FACTOR, Y 1.000
FINAL LEAK RATE, CFM 0.003
TONS PER HOUR, PRODUCT 0.000

COMPOSITION OF DUCT GAS, % BY VOLUME DRY BASIS
CARBON DIOXIDE 0.00
OXYGEN 20.50
CARBON MONOXIDE 0.00
NITROGEN 79.50

UREA, AMMONIA, FORMALDEHYDE COLLECTED, MG

	UREA	AMMONIA-DIR	AMMONIA-DIST	FORMALDEHYDE
420 IMPINGERS	0.2606E 05	0.3910E 03	0.3900E 03	0.1310E 01
42504 IMPINGERS	0.2700E 02	0.5010E 02	0.5120E 02	0.3800E-01
TOTAL	0.2609E 05	0.4411E 03	0.9412E 03	0.1348E 01

AMOUNT OF WATER COLLECTED, GRAMS
IMPINGERS 18.5
SILICA GEL 13.8

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 2
TRC PROJECT 92988-20

POINT	TIME	VEL HEAD IN H2O	ORIFICE PRESS IN H2O	METER INLET DEG F	TEMPS EXIT DEG F	DUCT STAT PRESS IN H2O	DUCT TEMP DEG F	INITIAL METER VOL CU FT	P R	CYC R AVG
1	3.0	1.0500	1.0800	70.	70.	-2.70	191.	183.33	3	0.
2	3.0	1.0500	1.0800	70.	70.	-2.70	193.	185.05	3	0.
3	3.0	1.0500	1.0800	70.	71.	-2.70	194.	186.77	3	0.
4	3.0	1.1500	1.1800	70.	71.	-2.70	194.	189.49	3	0.
5	3.0	1.1500	1.1800	70.	71.	-2.70	195.	190.30	3	0.
6	3.0	1.1500	1.1800	70.	71.	-2.70	194.	192.10	3	0.
7	3.0	1.2000	1.2200	70.	71.	-2.70	196.	193.98	3	0.
8	3.0	1.2000	1.2200	70.	71.	-2.70	196.	195.74	3	0.
9	3.0	1.2000	1.2200	70.	71.	-2.70	197.	197.60	3	0.
10	3.0	1.2000	1.2200	70.	71.	-2.70	196.	199.48	3	0.
11	3.0	1.2000	1.2200	70.	71.	-2.70	197.	201.28	3	0.
12	3.0	1.2000	1.2200	70.	71.	-2.70	197.	203.13	3	0.
13	3.0	1.2500	1.2900	70.	71.	-2.70	194.	204.96	3	0.
14	3.0	1.2500	1.2900	70.	71.	-2.70	195.	206.87	3	0.
15	3.0	1.1500	1.1800	70.	71.	-2.70	189.	208.75	3	0.
16	3.0	1.0500	1.0800	70.	71.	-2.70	185.	210.57	3	0.
17	3.0	0.7600	0.7800	70.	71.	-2.70	170.	212.30	3	0.
18	3.0	0.7600	0.7800	70.	71.	-2.70	130.	213.79	3	0.
1	3.0	1.1000	1.1200	69.	70.	-2.70	189.	215.33	3	0.
2	3.0	1.1500	1.1800	69.	70.	-2.70	196.	217.86	3	0.
3	3.0	1.2000	1.2200	69.	70.	-2.70	196.	219.66	3	0.
4	3.0	1.2000	1.2200	69.	70.	-2.70	196.	221.50	3	0.
5	3.0	1.1000	1.1200	69.	70.	-2.70	196.	223.32	3	0.
6	3.0	1.1500	1.1800	69.	69.	-2.70	197.	225.07	3	0.
7	3.0	1.1000	1.1200	68.	69.	-2.70	196.	225.97	3	0.
8	3.0	1.0500	1.0800	68.	69.	-2.70	196.	226.65	3	0.
9	3.0	1.2000	1.2200	68.	69.	-2.70	197.	230.42	3	0.
10	3.0	1.2500	1.2900	67.	69.	-2.70	197.	232.20	3	0.
11	3.0	1.2500	1.2900	67.	69.	-2.70	196.	234.20	3	0.
12	3.0	1.2500	1.2900	67.	69.	-2.70	197.	236.01	3	0.
13	3.0	1.1500	1.1800	68.	69.	-2.70	196.	237.91	3	0.
14	3.0	1.1000	1.1200	68.	69.	-2.70	193.	239.72	3	0.
15	3.0	1.0500	1.0800	68.	69.	-2.70	191.	241.53	3	0.
16	3.0	0.9800	1.0000	69.	70.	-2.70	192.	243.29	3	0.
17	3.0	0.9800	1.0000	70.	70.	-2.70	184.	245.00	3	0.
18	3.0	0.9800	1.0000	71.	72.	-2.70	140.	246.63	3	0.

FINAL METER VOLUME

249.31

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 2
TRC PROJECT 82988-20

C. F. INDUSTRIES
UREA GRANULATOR SCRUBBER 3 INLET DUCT
JANUARY 17, 1979 1548-1756 HRS

STANDARD CONDITION TEMPERATURE, DEG F	0.6900E C2
STANDARD CONDITION PRESSURE, IN HG	0.2992E 02
TOTAL SAMPLING TIME, MINUTES	0.1080E 03
AVERAGE SQUARE ROOT VELOCITY HEAD, IN H2O EXP .5	0.1056E 01
AVERAGE ORIFICE PRESSURE DROP, IN H2O	0.1145E 01
AVERAGE METER TEMPERATURE, DEG F	0.6974E C2
AVERAGE DUCT STATIC PRESSURE, IN H2O	-0.2700E C1
AVERAGE DUCT TEMPERATURE, DEG F	0.1902E 03
TOTAL SAMPLE VOLUME, DACF	0.6498E 02
TOTAL SAMPLE VOLUME, DSCF	0.6597E 02
WATER VAPOR VOLUME, DSCF	0.1520E C1
MOISTURE CONTENT OF DUCT GAS, PERCENT	0.2253E C1
MOLE FRACTION DRY GAS	0.9775E C0
MOLECULAR WEIGHT - DRY STACK GAS	0.2982E 02
MOLECULAR WEIGHT - STACK GAS	0.2958E C2
AVERAGE STACK PRESSURE, IN HG	0.3019E 02
DUCT VOLUMETRIC FLOW, ACFM	0.5170E 05
DUCT VOLUMETRIC FLOW, DSCFM	0.4141E 05
AVERAGE DUCT VELOCITY, FPM	0.3870E C4
EXCESS AIR, PERCENT	0.4201E C4
AVERAGE DUCT GAS DENSITY, LBS/ACF	0.6077E-C1
ISOKINETIC FACTOR, PERCENT	0.1034E 03

EMISSION DATA

	GR/ ACF	GR/ DSCF	LBS/ HR
UREA IN H2O	0.4883E 01	0.6096E 01	0.2164E 04
UREA IN H2SO4	0.5058E-02	0.6315E-02	0.2241E 01
TOTAL UREA	0.4888E 01	0.6103E 01	0.2166E 04
AMMONIA-DIRECT-IN H2O	0.7325E-01	0.9146E-01	0.3246E 02
AMMONIA-DIRECT-IN H2SO4	0.9386E-02	0.1172E-01	0.4159E 01
TOTAL AMMONIA-DIRECT	0.8264E-01	0.1032E 00	0.3662E 02
AMMONIA-DISTILLED-IN H2O	0.1667E 00	0.2082E 00	0.7388E 02
AMMONIA-DISTILLED-IN H2SO4	0.9592E-02	0.1198E-01	0.4250E 01
TOTAL AMMONIA-DISTILLED	0.1763E 00	0.2202E 00	0.7613E 02
FORMALDEHYDE IN H2O	0.2829E-03	0.3532E-03	0.1234E 00
FORMALDEHYDE IN H2SO4	0.7119E-05	0.8888E-05	0.3155E-02
TOTAL FORMALDEHYDE	0.2900E-03	0.3621E-03	0.1285E 00

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 2
TRC PROJECT 62988-20

C. F. INDUSTRIES
UREA GRANULATOR SCRUBBER 3 INLET DUCT
JANUARY 17, 1979 1546-1756 HPS

STANDARD CONDITION TEMPERATURE, DEG C	0.2300E 02
STANDARD CONDITION PRESSURE, MM HG	0.7600E 03
TOTAL SAMPLING TIME, MINUTES	0.1080E 03
AVERAGE SQUARE ROOT VELOCITY HEAD, MM H2O EXP.5	0.5322E 01
AVERAGE ORIFICE PRESSURE DROP, MM H2O	0.2908E 02
AVERAGE METER TEMPERATURE, DEG C	0.2096E 02
AVERAGE DUCT STATIC PRESSURE, MM H2O	-0.6858E 02
AVERAGE DUCT TEMPERATURE, DEG C	0.8790E 02
TOTAL SAMPLE VOLUME, DM3	0.1840E 01
TOTAL SAMPLE VOLUME, DNMS	0.1868E 01
WATER VAPOR VOLUME, DNMS	0.4306E-01
AVERAGE STACK GAS PRESSURE, MM HG	0.7669E 03
DUCT VOLUMETRIC FLOW, AM3/M	0.1464E 04
DUCT VOLUMETRIC FLOW, DNMS/M	0.1173E 04
AVERAGE DUCT VELOCITY, M/M	0.1179E 04
AVERAGE DUCT GAS DENSITY, KG/AM3	0.9734E 00

EMISSION DATA

	MG/ AM3	MG/ DNMS	KG/ HR
UREA IN H2O	0.1117E 05	0.1395E 05	0.9823E 03
UREA IN H2SO4	0.1158E 02	0.1445E 02	0.1018E 01
TOTAL UREA	0.1119E 05	0.1397E 05	0.9833E 03
AMMONIA-DIRECT-IN H2O	0.1676E 03	0.2093E 03	0.1474E 02
AMMONIA-DIRECT-IN H2SO4	0.2148E 02	0.2682E 02	0.1888E 01
TOTAL AMMONIA-DIRECT	0.1691E 03	0.2361E 03	0.1662E 02
AMMONIA-DISTILLED-IN H2O	0.3816E 03	0.4764E 03	0.3354E 02
AMMONIA-DISTILLED-IN H2SO4	0.2195E 02	0.2741E 02	0.1930E 01
TOTAL AMMONIA-DISTILLED	0.4035E 03	0.5039E 03	0.3547E 02
FORMALDEHYDE IN H2O	0.6474E 00	0.8083E 00	0.5691E-01
FORMALDEHYDE IN H2SO4	0.1629E-01	0.2034E-01	0.1432E-02
TOTAL FORMALDEHYDE	0.6637E 00	0.8286E 00	0.5834E-01

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 3
IRC PROJECT 82988-20

UNIT TESTED UREA GRANULATOR SCRUBBER B
DATE AND TIME OF TEST JANUARY 18, 1979 0947-1210 HRS
SAMPLING LOCATION INLET DUCT
NAME OF FIRM C. F. INDUSTRIES
LOCATION OF FIRM DONALDSONVILLE, LA
POLLUTANTS SAMPLED UREA, FORMALDEHYDE, AMMONIA

VOLUME OF WATER IN IMPINGERS BACK CALCULATED FROM SEPARATE MOISTURE RUN
DETERMINATION BECAUSE LAB UREA AND AMMONIA ANALYSIS DATA FOR THE TEST RUNS IS
NOT APPLICABLE TO THE CORRECTION OF THE IMPINGER WEIGHT GAIN DATA FOR THESE
TESTS.

BAROMETRIC PRESSURE, IN HG 30.27
DUCT AREA, SQ FT 13.36
NOZZLE DIAMETER, IN 0.197
PITOT CALIBRATION COEFFICIENTS 1 0.820
2 0.834
3 0.823
DRY GAS METER CALIBRATION FACTOR, Y 1.000
FINAL LEAK RATE, CFM 0.001
TONS PER HOUR, PRODUCT 0.000

COMPOSITION OF DUCT GAS, % BY VOLUME DRY BASIS
CARBON DIOXIDE 0.00
OXYGEN 20.50
CARBON MONOXIDE 0.00
NITROGEN 79.50

UREA, AMMONIA, FORMALDEHYDE COLLECTED, MG

	UREA	AMMONIA-DIR	AMMONIA-DIST	FORMALDEHYDE
420 IMPINGERS	0.2622E 05	0.4980E 03	0.9260E 03	0.3000E 01
42504 IMPINGERS	0.4050E 02	0.8200E 01	0.1780E 02	0.9100E-01
TOTAL	0.2626E 05	0.5062E 03	0.9438E 03	0.3091E 01

AMOUNT OF WATER COLLECTED, GRAMS
IMPINGERS 19.1
SILICA GEL 13.5

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 3
TRC PROJECT 82988-20

POINT	TIME	VEL HEAD IN H2O	ORIFICE PRESS IN H2O	METER INLET DEG F	TEMPS EXIT DEG F	DUCT PRESS IN H2O	STAT DUCT TEMP DEG F	INITIAL METER VOL CU FT	P R	CYC AVG
1	3.0	1.3000	1.3600	60.	61.	-2.30	182.	248.50	3	0.
2	3.0	1.2000	1.2400	61.	61.	-2.30	190.	250.30	3	0.
3	3.0	1.2000	1.2400	61.	62.	-2.30	190.	252.10	3	0.
4	3.0	1.2000	1.2400	61.	62.	-2.30	190.	254.12	3	0.
5	3.0	1.1000	1.1300	62.	62.	-2.30	191.	255.94	3	0.
6	3.0	1.2000	1.2400	62.	62.	-2.30	191.	257.73	3	0.
7	3.0	1.2000	1.2400	62.	63.	-2.30	191.	259.58	3	0.
8	3.0	1.0500	1.0900	62.	63.	-2.30	191.	261.42	3	0.
9	3.0	1.1000	1.1300	63.	63.	-2.30	191.	263.13	3	0.
10	3.0	1.0000	1.0200	63.	63.	-2.30	191.	264.91	3	0.
11	3.0	1.2500	1.3000	63.	63.	-2.30	184.	266.59	3	0.
12	3.0	1.3000	1.3700	64.	64.	-2.30	195.	268.54	3	0.
13	3.0	1.2000	1.2400	63.	64.	-2.30	189.	270.47	3	0.
14	3.0	1.2000	1.2400	64.	65.	-2.30	186.	272.34	3	0.
15	3.0	1.1000	1.1300	64.	65.	-2.30	176.	274.20	3	0.
16	3.0	1.0000	1.0200	64.	65.	-2.30	164.	275.92	3	0.
17	3.0	0.9000	0.9300	65.	66.	-2.30	164.	277.93	3	0.
18	3.0	0.9000	0.9300	65.	66.	-2.30	130.	279.55	3	0.
1	3.0	1.1000	1.1300	66.	66.	-2.50	170.	281.14	3	0.
2	3.0	1.1000	1.1300	66.	66.	-2.50	184.	283.10	3	0.
3	3.0	1.1500	1.2000	67.	68.	-2.50	189.	284.90	3	0.
4	3.0	1.1000	1.1300	67.	68.	-2.50	189.	286.70	3	0.
5	3.0	1.1000	1.1300	67.	68.	-2.50	189.	288.49	3	0.
6	3.0	1.2000	1.2400	67.	68.	-2.50	189.	290.28	3	0.
7	3.0	1.2000	1.2400	67.	68.	-2.50	190.	292.13	3	0.
8	3.0	1.2000	1.2400	67.	66.	-2.50	191.	294.00	3	0.
9	3.0	1.2000	1.2400	67.	68.	-2.50	191.	295.84	3	0.
10	3.0	1.2000	1.2400	67.	68.	-2.50	191.	297.70	3	0.
11	3.0	1.1500	1.2000	67.	68.	-2.50	191.	299.53	3	0.
12	3.0	1.1500	1.2000	68.	68.	-2.50	190.	301.33	3	0.
13	3.0	1.1000	1.1400	68.	69.	-2.50	189.	303.15	3	0.
14	3.0	1.1000	1.1400	68.	69.	-2.50	189.	304.95	3	0.
15	3.0	1.2000	1.2400	68.	70.	-2.50	184.	306.75	3	0.
16	3.0	1.1500	1.1900	68.	70.	-2.50	181.	308.58	3	0.
17	3.0	0.9000	0.9300	69.	70.	-2.50	160.	310.41	3	0.
18	3.0	0.9000	0.9300	69.	70.	-2.50	150.	312.10	3	0.
FINAL METER VOLUME								313.68		

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 3
TRC PROJECT 82988-20

C. F. INDUSTRIES
UREA GRANULATOR SCRUBBER 3 INLET DUCT
JANUARY 18, 1979 C947-1210 4PS

STANDARD CONDITION TEMPERATURE, DEG F	0.6800E 02
STANDARD CONDITION PRESSURE, IN HG	0.2992E 02
TOTAL SAMPLING TIME, MINUTES	0.1080E 03
AVERAGE SQUARE ROOT VELOCITY HEAD, IN H2O EXP .5	0.1061E 01
AVERAGE ORIFICE PRESSURE DROP, IN H2O	0.1166E 01
AVERAGE METER TEMPERATURE, DEG F	0.6547E 02
AVERAGE DUCT STATIC PRESSURE, IN H2O	-0.2400E 01
AVERAGE DUCT TEMPERATURE, DEG F	0.1931E 03
TOTAL SAMPLE VOLUME, DSCF	0.6518E 02
TOTAL SAMPLE VOLUME, DSCF	0.6645E 02
WATER VAPOR VOLUME, DSCF	0.1534E 01
MOISTURE CONTENT OF DUCT GAS, PERCENT	0.2257E 01
MOLE FRACTION DRY GAS	0.9774E 00
MOLECULAR WEIGHT - DRY STACK GAS	0.2882E 02
MOLECULAR WEIGHT - STACK GAS	0.2858E 02
AVERAGE STACK PRESSURE, IN HG	0.3009E 02
DUCT VOLUMETRIC FLOW, ACFM	0.5174E 05
DUCT VOLUMETRIC FLOW, DSCFM	0.4176E 05
AVERAGE DUCT VELOCITY, FPM	0.3872E 04
EXCESS AIR, PERCENT	0.4201E 04
AVERAGE DUCT GAS DENSITY, LBS/ACF	0.6124E-01
ISOINETIC FACTOR, PERCENT	0.1033E 03

EMISSION DATA

	GR/ ACF	GR/ DSCF	LBS/ HR
UREA IN H2O	0.4914E 01	0.6089E 01	0.2179E 04
UREA IN H2SO4	0.7591E-02	0.9405E-02	0.3366E 01
TOTAL UREA	0.4922E 01	0.6099E 01	0.2182E 04
AMMONIA-DIRECT-IN H2O	0.9334E-01	0.1156E 00	0.4139E 02
AMMONIA-DIRECT-IN H2SO4	0.1537E-02	0.1904E-02	0.5815E 00
TOTAL AMMONIA-DIRECT	0.9488E-01	0.1175E 00	0.4207E 02
AMMONIA-DISTILLED-IN H2O	0.1736E 00	0.2150E 00	0.7696E 02
AMMONIA-DISTILLED-IN H2SO4	0.3336E-02	0.4133E-02	0.1479E 01
TOTAL AMMONIA-DISTILLED	0.1769E 00	0.2192E 00	0.7844E 02
FORMALDEHYDE IN H2O	0.5623E-03	0.6966E-03	0.2493E 00
FORMALDEHYDE IN H2SO4	0.1706E-04	0.2113E-04	0.7563E-02
TOTAL FORMALDEHYDE	0.5793E-03	0.7178E-03	0.2569E 00

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 3
TRC PROJECT 82988-20

C. F. INDUSTRIES
UREA GRANULATOR SCRUBBER 3 INLET DUCT
JANUARY 18, 1977 0947-1210 HRS

STANDARD CONDITION TEMPERATURE, DEG C	0.2900E 02
STANDARD CONDITION PRESSURE, MM HG	0.7600E 03
TOTAL SAMPLING TIME, MINUTES	0.1080E 03
AVERAGE SQUARE ROOT VELOCITY HEAD, MM H2O EXP.5	0.5346E 01
AVERAGE ORIFICE PRESSURE DROP, MM H2O	0.2961E 02
AVERAGE METER TEMPERATURE, DEG C	0.1860E 02
AVERAGE DUCT STATIC PRESSURE, MM H2O	-0.6096E 02
AVERAGE DUCT TEMPERATURE, DEG C	0.3395E 02
TOTAL SAMPLE VOLUME, DM3	0.1846E 01
TOTAL SAMPLE VOLUME, DM3	0.1882E 01
WATER VAPOR VOLUME, DM3	0.4346E-01
AVERAGE STACK GAS PRESSURE, MM HG	0.7644E 03
DUCT VOLUMETRIC FLOW, AM3/M	0.1465E 04
DUCT VOLUMETRIC FLOW, DM3/M	0.1183E 04
AVERAGE DUCT VELOCITY, M/M	0.1180E 04
AVERAGE DUCT GAS DENSITY, KG/AM3	0.9809E 00

EMISSION DATA

	MG/ AM3	MG/ DM3	KG/ HR
UREA IN H2O	0.1125E 05	0.1393E 05	0.7893E 03
UREA IN H2SO4	0.1737E 02	0.2152E 02	0.1528E 01
TOTAL UREA	0.1126E 05	0.1395E 05	0.7908E 03
AMMONIA-DIRECT-IN H2O	0.2136E 03	0.2646E 03	0.1879E 02
AMMONIA-DIRECT-IN H2SO4	0.3517E 01	0.4357E 01	0.3094E 00
TOTAL AMMONIA-DIRECT	0.2171E 03	0.2690E 03	0.1910E 02
AMMONIA-DISTILLED-IN H2O	0.3972E 03	0.4921E 03	0.3494E 02
AMMONIA-DISTILLED-IN H2SO4	0.7635E 01	0.9459E 01	0.6716E 00
TOTAL AMMONIA-DISTILLED	0.4048E 03	0.5015E 03	0.3561E 02
FORMALDEHYDE IN H2O	0.1297E 01	0.1594E 01	0.1132E 00
FORMALDEHYDE IN H2SO4	0.3903E-01	0.4836E-01	0.3434E-02
TOTAL FORMALDEHYDE	0.1326E 01	0.1643E 01	0.1166E 00

OUTLET FROM "B" GRANULATOR SCRUBBER (TP-2)

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 1
TRC PROJECT 82928-20

UNIT TESTED UREA GRANULATOR SCRUBBER B
DATE AND TIME OF TEST JANUARY 17, 1979 1202-1416 HRS
SAMPLING LOCATION OUTLET STACK
NAME OF FIRM C.F. INDUSTRIES
LOCATION OF FIRM DONALDSONVILLE, LA
POLLUTANTS SAMPLED UREA, FORMALDEHYDE, AMMONIA

BAROMETRIC PRESSURE, IN HG 30.40
DUCT AREA, SQ FT 19.63
NOZZLE DIAMETER, IN 0.257
PITOT CALIBRATION COEFFICIENTS 1 0.820
2 0.834
3 0.823
DRY GAS METER CALIBRATION FACTOR, Y 1.000
FINAL LEAK RATE, CFM 0.000
TONS PER HOUR, PRODUCT 0.000

COMPOSITION OF DUCT GAS, % BY VOLUME DRY BASIS
CARBON DIOXIDE 0.00
OXYGEN 20.50
CARBON MONOXIDE 0.00
NITROGEN 79.50

UREA, AMMONIA, FORMALDEHYDE COLLECTED, MG

	UREA	AMMONIA-DIR	AMMONIA-DIST	FORMALDEHYDE
420 IMPINGERS	0.6590E 02	0.2180E 03	0.2060E 03	0.2110E 01
42504 IMPINGERS	0.4700E 01	0.2320E 03	0.2320E 03	0.4310E 03
TOTAL	0.7060E 02	0.4500E 03	0.4380E 03	0.2541E 01

AMOUNT OF WATER COLLECTED, GRAMS
IMPINGERS 105.3
SILICA GEL 22.0

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 1
TRC PROJECT 82988-20

POINT	TIME	VEL HEAD IN H2O	ORIFICE PRESS IN H2O	METER INLET DEG F	TEMPS EXIT DEG F	DUCT PRESS IN H2O	STAT DUCT TEMP DEG F	INITIAL METER VOL CU FT	P R	CYC AVG 9
1	5.0	0.6100	2.3000	69.	73.	-0.35	101.	43.91	2	0.
2	5.0	0.6000	2.2500	72.	73.	-0.35	99.	53.00	2	0.
3	5.0	0.6500	2.4500	72.	72.	-0.35	99.	57.12	2	0.
4	5.0	0.6500	2.4500	72.	72.	-0.35	99.	61.63	2	0.
5	5.0	0.6800	2.6000	71.	72.	-0.35	99.	65.93	2	0.
6	5.0	0.6800	2.6000	71.	72.	-0.35	101.	70.50	2	0.
7	5.0	0.6000	2.2500	72.	72.	-0.35	102.	75.05	2	0.
8	5.0	0.6100	2.3000	71.	71.	-0.35	103.	79.37	2	0.
9	5.0	0.4700	1.7900	71.	72.	-0.35	103.	83.70	2	0.
10	5.0	0.4800	1.8200	71.	71.	-0.35	103.	84.30	2	0.
11	5.0	0.4000	1.5100	71.	72.	-0.35	103.	91.90	2	0.
12	5.0	0.4200	1.6000	71.	72.	-0.35	103.	95.50	2	0.
1	5.0	0.5500	2.1000	72.	72.	-0.35	99.	99.23	2	0.
2	5.0	0.6000	2.2500	71.	72.	-0.35	99.	103.14	2	0.
3	5.0	0.6100	2.3000	71.	71.	-0.35	99.	107.60	2	0.
4	5.0	0.6300	2.3800	71.	71.	-0.35	100.	111.90	2	0.
5	5.0	0.6900	2.6200	71.	72.	-0.35	99.	116.30	2	0.
6	5.0	0.6700	2.5500	71.	72.	-0.35	99.	120.71	2	0.
7	5.0	0.6200	2.3200	71.	72.	-0.35	101.	125.45	2	0.
8	5.0	0.6200	2.3200	71.	72.	-0.35	103.	130.40	2	0.
9	5.0	0.5500	2.0800	71.	72.	-0.35	102.	134.20	2	0.
10	5.0	0.5500	2.0800	71.	72.	-0.35	102.	133.39	2	0.
11	5.0	0.4000	1.5100	71.	72.	-0.35	101.	142.54	2	0.
12	5.0	0.4000	1.5100	71.	72.	-0.35	99.	146.20	2	0.

FINAL METER VOLUME

149.95

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 1
TRC PROJECT 22988-20

C.F. INDUSTRIES
UREA GRANULATOR SCRUBBER 3 OUTLET STACK
JANUARY 17, 1979 1202-1416 HRS

STANDARD CONDITION TEMPERATURE, DEG F	0.6800E 02
STANDARD CONDITION PRESSURE, IN HG	0.2992E 02
TOTAL SAMPLING TIME, MINUTES	0.1200E 03
AVERAGE SQUARE ROOT VELOCITY HEAD, IN H2O EXP .5	0.7540E 00
AVERAGE ORIFICE PRESSURE DROP, IN H2O	0.2164E 01
AVERAGE METER TEMPERATURE, DEG F	0.7152E 02
AVERAGE DUCT STATIC PRESSURE, IN H2O	-0.3500E 00
AVERAGE DUCT TEMPERATURE, DEG F	0.1005E 03
TOTAL SAMPLE VOLUME, DSCF	0.1010E 03
TOTAL SAMPLE VOLUME, DSCF	0.1025E 03
WATER VAPOR VOLUME, DSCF	0.5992E 01
MOISTURE CONTENT OF DUCT GAS, PERCENT	0.5522E 01
MOLE FRACTION DRY GAS	0.9448E 00
MOLECULAR WEIGHT - DRY STACK GAS	0.2882E 02
MOLECULAR WEIGHT - STACK GAS	0.2922E 02
AVERAGE STACK PRESSURE, IN HG	0.3037E 02
DUCT VOLUMETRIC FLOW, ACFM	0.5120E 05
DUCT VOLUMETRIC FLOW, DSCFM	0.4626E 05
AVERAGE DUCT VELOCITY, FPM	0.2608E 04
EXCESS AIR, PERCENT	0.4201E 04
AVERAGE DUCT GAS DENSITY, LBS/ACF	0.7005E-01
ISOKINETIC FACTOR, PERCENT	0.1007E 03

EMISSION DATA

	GR/ ACF	GR/ DSCF	LBS/ HR
UREA IN H2O	0.8962E-02	0.9919E-02	0.3932E 01
UREA IN H2SO4	0.6392E-03	0.7074E-03	0.2805E 00
TOTAL UREA	0.9601E-02	0.1063E-01	0.4213E 01
AMMONIA-DIRECT-IN H2O	0.2965E-01	0.3261E-01	0.1301E 02
AMMONIA-DIRECT-IN H2SO4	0.3155E-01	0.3492E-01	0.1384E 02
TOTAL AMMONIA-DIRECT	0.6120E-01	0.6773E-01	0.2685E 02
AMMONIA-DISTILLED-IN H2O	0.2801E-01	0.3101E-01	0.1229E 02
AMMONIA-DISTILLED-IN H2SO4	0.3155E-01	0.3492E-01	0.1384E 02
TOTAL AMMONIA-DISTILLED	0.5956E-01	0.6593E-01	0.2614E 02
FORMALDEHYDE IN H2O	0.2869E-03	0.3176E-03	0.1259E 00
FORMALDEHYDE IN H2SO4	0.5861E-04	0.6487E-04	0.2572E-01
TOTAL FORMALDEHYDE	0.3456E-03	0.3825E-03	0.1516E 00

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 1
TRC PROJECT 82988-23

C.F. INDUSTRIES
UREA GRANULATOR SCRUBBER 3 OUTLET STACK
JANUARY 17, 1979 1202-1416 HRS

STANDARD CONDITION TEMPERATURE, DEG C	0.2000E 02
STANDARD CONDITION PRESSURE, MM HG	0.7600E 03
TOTAL SAMPLING TIME, MINUTES	0.1200E 03
AVERAGE SQUARE ROOT VELOCITY HEAD, MM H2O EXP.5	0.3800E 01
AVERAGE ORIFICE PRESSURE DROP, MM H2O	0.5497E 02
AVERAGE METER TEMPERATURE, DEG C	0.2196E 02
AVERAGE DUCT STATIC PRESSURE, MM H2O	-0.8890E 01
AVERAGE DUCT TEMPERATURE, DEG C	0.3906E 02
TOTAL SAMPLE VOLUME, DM3	0.2861E 01
TOTAL SAMPLE VOLUME, DNMS	0.2903E 01
WATER VAPOR VOLUME, DNMS	0.1697E 00
AVERAGE STACK GAS PRESSURE, MM HG	0.7715E 03
DUCT VOLUMETRIC FLOW, AM3/M	0.1450E 04
DUCT VOLUMETRIC FLOW, DNMS/M	0.1310E 04
AVERAGE DUCT VELOCITY, M/M	0.7949E 03
AVERAGE DUCT GAS DENSITY, KG/AM3	0.1122E 01

EMISSION DATA

	MG/ AM3	MG/ DNMS	KG/ HR
UREA IN H2O	0.2051E 02	0.2270E 02	0.1785E 01
UREA IN H2SO4	0.1463E 01	0.1619E 01	0.1273E 00
TOTAL UREA	0.2197E 02	0.2432E 02	0.1913E 01
AMMONIA-DIRECT-IN H2O	0.6784E 02	0.7509E 02	0.5906E 01
AMMONIA-DIRECT-IN H2SO4	0.7220E 02	0.7991E 02	0.6285E 01
TOTAL AMMONIA-DIRECT	0.1400E 03	0.1550E 03	0.1219E 02
AMMONIA-DISTILLED-IN H2O	0.6411E 02	0.7095E 02	0.5581E 01
AMMONIA-DISTILLED-IN H2SO4	0.7220E 02	0.7991E 02	0.6285E 01
TOTAL AMMONIA-DISTILLED	0.1363E 03	0.1509E 03	0.1187E 02
FORMALDEHYDE IN H2O	0.5566E 00	0.7268E 00	0.5716E-01
FORMALDEHYDE IN H2SO4	0.1341E 00	0.1485E 00	0.1168E-01
TOTAL FORMALDEHYDE	0.7908E 00	0.8752E 00	0.6884E-01

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 2
TRC PROJECT 32988-20

UNIT TESTED UREA GRANULATOR SCRUBBER B
DATE AND TIME OF TEST JANUARY 17, 1979 1549-1753 HRS
SAMPLING LOCATION OUTLET STACK
NAME OF FIRM C.F. INDUSTRIES
LOCATION OF FIRM DONALDSONVILLE, LA
POLLUTANTS SAMPLED UREA, FORMALDEHYDE, AMMONIA

BAROMETRIC PRESSURE, IN HG 30.38
DUCT AREA, SQ FT 19.63
NOZZLE DIAMETER, IN 0.257
PITOT CALIBRATION COEFFICIENTS 1 0.820
2 0.834
3 0.823
DRY GAS METER CALIBRATION FACTOR, Y 1.000
FINAL LEAK RATE, CFM 0.000
TONS PER HOUR, PRODUCT 0.000

COMPOSITION OF DUCT GAS, % BY VOLUME DRY BASIS
CARBON DIOXIDE 0.00
OXYGEN 20.50
CARBON MONOXIDE 0.00
NITROGEN 79.50

UREA, AMMONIA, FORMALDEHYDE COLLECTED, MG

	UREA	AMMONIA-DIR	AMMONIA-DIST	FORMALDEHYDE
420 IMPINGERS	0.6580E 02	0.3710E 03	3.1830E 03	0.1140E 01
42504 IMPINGERS	0.7300E 01	0.3230E 03	0.2430E 03	0.3600E-01
TOTAL	0.7310E 02	0.6940E 03	0.4260E 03	0.1176E 01

AMOUNT OF WATER COLLECTED, GRAMS
IMPINGERS 112.1
SILICA GEL 20.6

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 2
TRC PROJECT 82958-20

POINT	TIME	VEL HEAD IN H2O	ORIFICE PRESS IN H2O	METER INLET DEG F	TEMPS EXIT DEG F	DUCT PRESS IN H2O	STAT DUCT TEMP DEG F	INITIAL METER VOL CU FT	P R	CYC AVG
1	5.0	0.5800	2.2000	73.	74.	-0.31	103.	150.10	2	0.
2	5.0	0.5800	2.2000	71.	72.	-0.31	100.	154.20	2	0.
3	5.0	0.6500	2.4500	70.	72.	-0.31	102.	153.60	2	0.
4	5.0	0.6500	2.4500	70.	72.	-0.31	102.	162.90	2	0.
5	5.0	0.6700	2.5200	70.	72.	-0.31	101.	167.32	2	0.
6	5.0	0.6700	2.5200	70.	71.	-0.31	102.	171.89	2	0.
7	5.0	0.5600	2.1100	70.	71.	-0.31	103.	175.45	2	0.
8	5.0	0.5600	2.1100	69.	71.	-0.31	102.	183.77	2	0.
9	5.0	0.4800	1.8200	69.	70.	-0.31	103.	185.00	2	0.
10	5.0	0.4700	1.7900	68.	70.	-0.31	102.	189.00	2	0.
11	5.0	0.3500	1.3200	68.	69.	-0.31	103.	193.00	2	0.
12	5.0	0.3500	1.3200	68.	69.	-0.31	99.	196.35	2	0.
1	5.0	0.5600	2.1100	67.	68.	-0.38	103.	199.88	2	0.
2	5.0	0.5800	2.2000	67.	68.	-0.38	100.	203.93	2	0.
3	5.0	0.6200	2.3000	66.	68.	-0.38	101.	203.24	2	0.
4	5.0	0.6300	2.3500	66.	67.	-0.38	100.	212.70	2	0.
5	5.0	0.6700	2.5200	65.	67.	-0.38	97.	217.20	2	0.
6	5.0	0.6800	2.5900	66.	67.	-0.38	99.	221.52	2	0.
7	5.0	0.6200	2.3000	66.	67.	-0.38	103.	226.20	2	0.
8	5.0	0.6200	2.3000	65.	66.	-0.38	102.	233.63	2	0.
9	5.0	0.5400	2.0200	65.	66.	-0.38	103.	235.02	2	0.
10	5.0	0.5400	2.0200	66.	66.	-0.38	102.	239.21	2	0.
11	5.0	0.4000	1.5100	66.	67.	-0.38	99.	243.60	2	0.
12	5.0	0.4000	1.5100	66.	67.	-0.38	103.	247.09	2	0.
FINAL METER VOLUME								253.82		

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST 10 2
TRC PROJECT 32988-20

C.F. INDUSTRIES
UREA GRANULATOR SCRUBBER 3 OUTLET STACK
JANUARY 17, 1979 1549-1753 HRS

STANDARD CONDITION TEMPERATURE, DEG F	0.6800E 02
STANDARD CONDITION PRESSURE, IN HG	0.2992E 02
TOTAL SAMPLING TIME, MINUTES	0.1203E 03
AVERAGE SQUARE ROOT VELOCITY HEAD, IN H ₂ O EXP .5	0.7448E 00
AVERAGE ORIFICE PRESSURE DROP, IN H ₂ O	0.2106E 01
AVERAGE METER TEMPERATURE, DEG F	0.6342E 02
AVERAGE DUCT STATIC PRESSURE, IN H ₂ O	-0.3450E 00
AVERAGE DUCT TEMPERATURE, DEG F	0.1007E 03
TOTAL SAMPLE VOLUME, DSCF	0.1007E 03
TOTAL SAMPLE VOLUME, DSCF	0.1027E 03
WATER VAPOR VOLUME, DSCF	0.6246E 01
MOISTURE CONTENT OF DUCT GAS, PERCENT	0.5733E 01
MOLE FRACTION DRY GAS	0.9427E 00
MOLECULAR WEIGHT - DRY STACK GAS	0.2882E 02
MOLECULAR WEIGHT - STACK GAS	0.2820E 02
AVERAGE STACK PRESSURE, IN HG	0.3035E 02
DUCT VOLUMETRIC FLOW, ACFM	0.5062E 05
DUCT VOLUMETRIC FLOW, DSCFM	0.4559E 03
AVERAGE DUCT VELOCITY, FPM	0.2579E 04
EXCESS AIR, PERCENT	0.4201E 04
AVERAGE DUCT GAS DENSITY, LBS/ACF	0.6792E-01
ISOINETIC FACTOR, PERCENT	0.1024E 03

EMISSION DATA

	GR/ ACF	GR/ DSCF	LBS/ HR
UREA IN H ₂ O	0.9903E-02	0.9385E-02	0.3862E 01
UREA IN H ₂ SO ₄	0.9877E-03	0.1097E-02	0.4295E 00
TOTAL UREA	0.9891E-02	0.1098E-01	0.4291E 01
AMMONIA-DIRECT-IN H ₂ O	0.5020E-01	0.5574E-01	0.2178E 02
AMMONIA-DIRECT-IN H ₂ SO ₄	0.4370E-01	0.4852E-01	0.1896E 02
TOTAL AMMONIA-DIRECT	0.9390E-01	0.1043E 00	0.4074E 02
AMMONIA-DISTILLED-IN H ₂ O	0.2476E-01	0.2749E-01	0.1074E 02
AMMONIA-DISTILLED-IN H ₂ SO ₄	0.3288E-01	0.3651E-01	0.1426E 02
TOTAL AMMONIA-DISTILLED	0.5764E-01	0.6400E-01	0.2501E 02
FORMALDEHYDE IN H ₂ O	0.1542E-03	0.1713E-03	0.6692E-01
FORMALDEHYDE IN H ₂ SO ₄	0.4271E-05	0.5408E-05	0.2113E-02
TOTAL FORMALDEHYDE	0.1591E-03	0.1767E-03	0.6903E-01

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 2
TRC PROJECT 82988-20

C.F. INDUSTRIES
UREA GRANULATOR SCRUBBER 3 OUTLET STACK
JANUARY 17, 1979 1549-1753 HRS

STANDARD CONDITION TEMPERATURE, DEG C	0.2000E 02
STANDARD CONDITION PRESSURE, MM HG	0.7600E 03
TOTAL SAMPLING TIME, MINUTES	0.1200E 03
AVERAGE SQUARE ROOT VELOCITY HEAD, MM H2O EXP.5	0.3754E 01
AVERAGE ORIFICE PRESSURE DROP, MM H2O	0.5349E 02
AVERAGE METER TEMPERATURE, DEG C	0.2023E 02
AVERAGE DUCT STATIC PRESSURE, MM H2O	-0.8763E 01
AVERAGE DUCT TEMPERATURE, DEG C	0.3915E 02
TOTAL SAMPLE VOLUME, DM3	0.2952E 01
TOTAL SAMPLE VOLUME, DNMS	0.2909E 01
WATER VAPOR VOLUME, DNMS	0.1769E 00
AVERAGE STACK GAS PRESSURE, MM HG	0.7710E 03
DUCT VOLUMETRIC FLOW, AM3/M	0.1433E 04
DUCT VOLUMETRIC FLOW, DNMS/M	0.1291E 04
AVERAGE DUCT VELOCITY, M/M	0.7859E 03
AVERAGE DUCT GAS DENSITY, KG/AM3	0.1120E 01

EMISSION DATA

	MG/ AM3	MG/ DNMS	KG/ HR
UREA IN H2O	0.2037E 02	0.2262E 02	0.1754E 01
UREA IN H2SO4	0.2260E 01	0.2510E 01	0.1945E 00
TOTAL UREA	0.2263E 02	0.2513E 02	0.1948E 01
AMMONIA-DIRECT-IN H2O	0.1149E 03	0.1273E 03	0.9887E 01
AMMONIA-DIRECT-IN H2SO4	0.1000E 03	0.1110E 03	0.8608E 01
TOTAL AMMONIA-DIRECT	0.2149E 03	0.2386E 03	0.1849E 02
AMMONIA-DISTILLED-IN H2O	0.5666E 02	0.6291E 02	0.4877E 01
AMMONIA-DISTILLED-IN H2SO4	0.7524E 02	0.8354E 02	0.6476E 01
TOTAL AMMONIA-DISTILLED	0.1319E 03	0.1465E 03	0.1135E 02
FORMALDEHYDE IN H2O	0.3530E 00	0.3919E 00	0.3038E-01
FORMALDEHYDE IN H2SO4	0.1115E-01	0.1239E-01	0.9594E-03
TOTAL FORMALDEHYDE	0.3641E 00	0.4043E 00	0.3134E-01

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 3
TRC PROJECT 82988-20

UNIT TESTED	UREA GRANULATOR SCRUBBER B
DATE AND TIME OF TEST	JANUARY 18, 1979 0946-1217 HRS
SAMPLING LOCATION	OUTLET STACK
NAME OF FIRM	C.F. INDUSTRIES
LOCATION OF FIRM	DONALDSONVILLE, LA
POLLUTANTS SAMPLED	UREA, FORMALDEHYDE, AMMONIA

BAROMETRIC PRESSURE, IN HG	30.27
DUCT AREA, SQ FT	19.63
NOZZLE DIAMETER, IN	0.257
PITOT CALIBRATION COEFFICIENTS 1	0.820
2	0.834
3	0.823
DRY GAS METER CALIBRATION FACTOR, Y	1.000
FINAL LEAK RATE, CFM	0.000
TONS PER HOUR, PRODUCT	0.000

COMPOSITION OF DUCT GAS, % BY VOLUME DRY BASIS	
CARBON DIOXIDE	0.00
OXYGEN	20.50
CARBON MONOXIDE	0.00
NITROGEN	79.50

UREA, AMMONIA, FORMALDEHYDE COLLECTED, MG

	UREA	AMMONIA-DIR	AMMONIA-DIST	FORMALDEHYDE
420 IMPINGERS	0.5710E 02	0.1250E 03	0.1250E 03	0.1250E 01
42504 IMPINGERS	0.5200E 01	0.2620E 03	0.2630E 03	0.4600E-01
TOTAL	0.6230E 02	0.3870E 03	0.3880E 03	0.1296E 01

AMOUNT OF WATER COLLECTED, GRAMS	
IMPINGERS	110.3
SILICA GEL	18.9

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 3
TRC PROJECT 82968-20

POINT	TIME	VEL HEAD IN H2O	ORIFICE PRESS IN H2O	METER INLET DEG F	TEMPS EXIT DEG F	DUCT PRESS IN H2O	STAT TEMP DEG F	DUCT TEMP DEG F	INITIAL METER VOL CU FT	P R	CYC AVG
1	3.0	0.6200	2.3000	63.	63.	-0.33	103.		251.04	2	0.
2	3.0	0.5800	2.2000	63.	63.	-0.33	101.		253.50	2	0.
3	3.0	0.6500	2.4500	63.	63.	-0.33	102.		259.70	2	0.
4	3.0	0.6500	2.4500	63.	63.	-0.33	101.		263.19	2	0.
5	3.0	0.6700	2.5100	63.	63.	-0.33	103.		267.65	2	0.
6	3.0	0.6500	2.4500	63.	63.	-0.33	104.		272.20	2	0.
7	3.0	0.5600	2.1100	63.	63.	-0.33	104.		276.65	2	0.
8	3.0	0.5600	2.1100	63.	63.	-0.33	105.		280.93	2	0.
9	3.0	0.4900	1.8400	63.	63.	-0.33	103.		285.13	2	0.
10	3.0	0.5000	1.8800	63.	63.	-0.33	104.		289.09	2	0.
11	3.0	0.3700	1.4000	63.	63.	-0.33	102.		293.02	2	0.
12	3.0	0.3700	1.4000	64.	64.	-0.33	101.		296.55	2	0.
1	3.0	0.6000	2.2500	66.	66.	-0.30	102.		303.14	2	0.
2	3.0	0.5800	2.2000	66.	66.	-0.30	101.		304.24	2	0.
3	3.0	0.6400	2.4000	66.	66.	-0.30	101.		305.95	2	0.
4	3.0	0.6500	2.4500	66.	66.	-0.30	101.		312.96	2	0.
5	3.0	0.6800	2.5800	67.	67.	-0.30	102.		317.50	2	0.
6	3.0	0.6800	2.5800	66.	66.	-0.30	101.		322.07	2	0.
7	3.0	0.6000	2.2500	66.	67.	-0.30	103.		326.71	2	0.
8	3.0	0.6000	2.2500	67.	67.	-0.30	105.		331.08	2	0.
9	3.0	0.5200	1.9500	67.	67.	-0.30	104.		335.45	2	0.
10	3.0	0.5200	1.9500	67.	67.	-0.30	103.		339.54	2	0.
11	3.0	0.4200	1.5900	68.	68.	-0.30	102.		343.58	2	0.
12	3.0	0.4200	1.5900	68.	68.	-0.30	102.		347.27	2	0.

FINAL METER VOLUME

351.11

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 3
 TPC PROJECT 82988-20

C.F. INDUSTRIES
 UREA GRANULATOR SCRUBBER 3 OUTLET STACK
 JANUARY 18, 1979 0946-1217 HRS

STANDARD CONDITION TEMPERATURE, DEG F	0.6800E 02
STANDARD CONDITION PRESSURE, IN HG	0.2992E 02
TOTAL SAMPLING TIME, MINUTES	0.1209E 03
AVERAGE SQUARE ROOT VELOCITY HEAD, IN H2O EXP .5	0.7494E 00
AVERAGE ORIFICE PRESSURE DROP, IN H2O	0.2131E 01
AVERAGE METER TEMPERATURE, DEG F	0.6490E 02
AVERAGE DUCT STATIC PRESSURE, IN H2O	-0.3150E 00
AVERAGE DUCT TEMPERATURE, DEG F	0.1025E 03
TOTAL SAMPLE VOLUME, DACF	0.1001E 03
TOTAL SAMPLE VOLUME, DSCF	0.1024E 03
WATER VAPOR VOLUME, DSCF	0.6081E 01
MOISTURE CONTENT OF DUCT GAS, PERCENT	0.5609E 01
MOLE FRACTION DRY GAS	0.9439E 00
MOLECULAR WEIGHT - DRY STACK GAS	0.2882E 02
MOLECULAR WEIGHT - STACK GAS	0.2921E 02
AVERAGE STACK PRESSURE, IN HG	0.3025E 02
DUCT VOLUMETRIC FLOW, ACFM	0.5109E 05
DUCT VOLUMETRIC FLOW, DSCFM	0.4576E 05
AVERAGE DUCT VELOCITY, FPM	0.2603E 04
EXCESS AIR, PERCENT	0.4201E 04
AVERAGE DUCT GAS DENSITY, LBS/ACF	0.6748E-01
ISOINETIC FACTOR, PERCENT	0.1016E 03

EMISSION DATA

	GR/ ACF	GR/ DSCF	LBS/ HR
UREA IN H2O	0.7709E-02	0.8607E-02	0.3376E 01
UREA IN H2SO4	0.7021E-03	0.7838E-03	0.3074E 00
TOTAL UREA	0.8411E-02	0.9391E-02	0.3683E 01
AMMONIA-DIRECT-IN H2O	0.1688E-01	0.1984E-01	0.7391E 01
AMMONIA-DIRECT-IN H2SO4	0.3537E-01	0.3949E-01	0.1549E 02
TOTAL AMMONIA-DIRECT	0.5225E-01	0.5833E-01	0.2288E 02
AMMONIA-DISTILLED-IN H2O	0.1688E-01	0.1884E-01	0.7391E 01
AMMONIA-DISTILLED-IN H2SO4	0.3551E-01	0.3964E-01	0.1555E 02
TOTAL AMMONIA-DISTILLED	0.5239E-01	0.5848E-01	0.2294E 02
FORMALDEHYDE IN H2O	0.1688E-03	0.1884E-03	0.7391E-01
FORMALDEHYDE IN H2SO4	0.6211E-05	0.6934E-05	0.2720E-02
TOTAL FORMALDEHYDE	0.1750E-03	0.1954E-03	0.7663E-01

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 3
TRC PROJECT 82938-2C

C.F. INDUSTRIES
UREA GRANULATOR SCRUBBER 3 GUTLET STACK
JANUARY 18, 1979 0946-1217 HRS

STANDARD CONDITION TEMPERATURE, DEG C	0.2000E 02
STANDARD CONDITION PRESSURE, MM HG	0.7600E 03
TOTAL SAMPLING TIME, MINUTES	0.1200E 03
AVERAGE SQUARE ROOT VELOCITY HEAD, MM H2O EXP.5	0.3777E 01
AVERAGE ORIFICE PRESSURE DROP, MM H2O	0.5412E 02
AVERAGE METER TEMPERATURE, DEG C	0.1328E 02
AVERAGE DUCT STATIC PRESSURE, MM H2O	-0.8001E 01
AVERAGE DUCT TEMPERATURE, DEG C	0.3917E 02
TOTAL SAMPLE VOLUME, DM3	0.2834E 01
TOTAL SAMPLE VOLUME, DM3	0.2899E 01
WATER VAPOR VOLUME, DM3	0.1722E 00
AVERAGE STACK GAS PRESSURE, MM HG	0.7683E 03
DUCT VOLUMETRIC FLOW, AM3/M	0.1447E 04
DUCT VOLUMETRIC FLOW, DM3/M	0.1296E 04
AVERAGE DUCT VELOCITY, M/M	0.7933E 03
AVERAGE DUCT GAS DENSITY, KG/AM3	0.1113E 01

EMISSION DATA

	MG/ AM3	MG/ DM3	KG/ HR
UREA IN H2O	0.1764E 02	0.1970E 02	0.1533E 01
UREA IN H2SO4	0.1607E 01	0.1794E 01	0.1396E 00
TOTAL UREA	0.1925E 02	0.2149E 02	0.1672E 01
AMMONIA-DIRECT-IN H2O	0.3862E 02	0.4312E 02	0.3355E 01
AMMONIA-DIRECT-IN H2SO4	0.8095E 02	0.9037E 02	0.7033E 01
TOTAL AMMONIA-DIRECT	0.1196E 03	0.1335E 03	0.1039E 02
AMMONIA-DISTILLED-IN H2O	0.3862E 02	0.4312E 02	0.3355E 01
AMMONIA-DISTILLED-IN H2SO4	0.3126E 02	0.9072E 02	0.7060E 01
TOTAL AMMONIA-DISTILLED	0.1199E 03	0.1338E 03	0.1041E 02
FORMALDEHYDE IN H2O	0.3862E 00	0.4312E 00	0.3355E-01
FORMALDEHYDE IN H2SO4	0.1421E-01	0.1587E-01	0.1235E-02
TOTAL FORMALDEHYDE	0.4004E 00	0.4470E 00	0.3479E-01

APPENDIX A-2

SAMPLE EQUATIONS AND EXAMPLE CALCUALTIONS

EMISSION CALCULATION SYMBOLS

- La - Allowable leak rate, cfm
- V_{m total} - Total meter sample volume, ft³
- T_{total} - Total sampling time, min
- L_p - Final leak rate of sampling train, cfm
- V_{mc total} - Total volume sampled corrected for excessive leakage, ft³
- Y - Dry gas meter calibration factor, dimensionless
- T_{std} - Standard temperature, °F
- T_{m avg.} - Average dry gas meter temperature, °F
- P_{bar} - Barometric pressure, "Hg
- ΔH_{avg.} - Average orifice pressure drop, "H₂O
- P_{std} - Standard pressure, "Hg
- V_I - Volume of liquid collected in impingers, ml
- V_{SG} - Volume of liquid collected in silica gel, grams
- M_S - Molecular weight of stack gas, lb/lb-mole
- %CO₂ - Percent CO₂ by volume (dry basis), %
- %CO - Percent CO by volume (dry basis), %
- %N₂ - Percent N₂ by volume (dry basis), %
- %O₂ - Percent O₂ by volume (dry basis), %
- D_{st} - Average duct gas density, lbs/ft³
- P_{s avg} - Average duct static pressure, "H₂O
- T_{s avg} - Average duct temperature, °F
- EA - Excess air, %
- V_s - Average duct velocity, ft/min
- C_p - Pitot tube coefficient, dimensionless
- (√ΔP)_{avg} - Average square root of velocity head, √"H₂O
- A_s - Cross-sectional area of duct, ft²

EMISSION CALCULATION SYMBOLS (cont'd)

Q - Duct volumetric flow rate, acfm

Q_{std} - Duct volumetric flow rate, corrected to dry standard conditions, dscfm

D_n - Nozzle diameter, inches

F - F factor, DSCF/MM BTU

ZH - Percent by weight of hydrogen in fuel

ZC - Percent by weight of carbon in fuel

ZS - Percent by weight of sulfur in fuel

ZN - Percent by weight of nitrogen in fuel

ZO - Percent by weight of oxygen in fuel

GCV - Gross calorific value of fuel, BTU/lb.

C - Actual particulate concentration, grains/acf

C_s - Particulate concentration, grains/dscf

ER - Particulate emission rate, lbs/hr

E - Particulate emissions, lbs/MM BTU

C_s @ 12% CO₂ - Particulate concentration, grains/dscf @ 12% CO₂

C_s @ 50% EA - Particulate concentration, grains/dscf @ 50% EA

C_{Lb} - Particulate concentration, lbs/1000 duct gas

C_{Lb} @ 12% CO₂ - Particulate concentration, lbs/1000 lbs @ 12% CO₂

C_{Lb} @ 50% EA - Particulate concentration, lbs/1000 lbs @ 50% EA

M_n - Total particulate collected, mg

Sample Equations and Example Calculations For
Test No. 1 Scrubber "B" Outlet

1. Allowable Leak Rate

$$La = 0.02 \text{ cfm or } 0.04 \frac{V_m \text{ total}}{T_{\text{total}}} \quad \text{which ever is less.}$$

$$\frac{0.04 V_m \text{ total}}{T_{\text{total}}} = \frac{0.04 \times 102.5}{12.0} = 0.03$$

$$La = 0.02 \text{ cfm}$$

2. Correction for Excessive Leak Rate

$$Lp = 0.00 \text{ cfm}$$

if $Lp > La$ use V_{mc} total in place of V_m total in all subsequent equations.

$$V_{mc} \text{ total} = V_m \text{ total} - (Lp - La) T_{\text{total}}$$

$$V_{mc} \text{ total} = \quad - (\quad - \quad) \quad = \quad \text{ft}^3$$

3. Volume of Sample Measured by Dry Gas Meter, Corrected to Standard Conditions

$$V_m \text{ total (std)} = V_m \text{ total} \times \left(\frac{T_{\text{std}} + 460}{T_m \text{ avg} + 460} \right) \left[\frac{P_{\text{bar}} + \frac{\Delta H \text{ avg}}{13.6}}{P_{\text{std}}} \right]$$

$$V_m \text{ total (std)} = 101.0 \times 1.00 \left(\frac{68 + 460}{71.5 + 460} \right) \left[\frac{30.40 + \frac{2.16}{13.6}}{29.92} \right]$$

$$V_m \text{ total (std)} = 102.5 \text{ dscf}$$

4. Moisture Content of Duct Gas

$$\% H_2O = \frac{0.04707 (V_I + V_{SG})}{V_m \text{ total (std)} + 0.04707 (V_I + V_{SG})} \times 100$$

$$\% H_2O = \frac{0.04707 (105.3 + 22.0)}{102.5 + 0.04707 (105.3 + 22.0)} \times 100$$

$$\% H_2O = 55 \%$$

5. Molecular Weight of Stack Gas

$$M_s = \left[(0.44 \times \% \text{CO}_2) + (0.28 \times \% \text{CO}) + (0.28 \times \% \text{N}_2) + (0.32 \times \% \text{O}_2) \right] \left(1 - \frac{\% \text{H}_2\text{O}}{100} \right) + 0.18 (\% \text{H}_2\text{O})$$

$$M_s = \left[(0.44 \times 0.0) + (0.28 \times 0.0) + (0.28 \times 79.5) + (0.32 \times 20.5) \right] \left(1 - \frac{5.5}{100} \right) + 0.18 (5.5)$$

$$M_s = 28.59 \text{ lb/lb-mole}$$

6. Average Duct Gas Density

$$D_{st} = 0.0458 \times M_s \left(\frac{P_{bar} + \frac{P_{s \text{ avg}}}{13.6}}{T_{s \text{ avg}} + 460} \right)$$

$$D_{st} = 0.0458 \times 28.59 \left(\frac{30.40 + \frac{-0.35}{13.6}}{100.5 + 460} \right)$$

$$D_{st} = 0.0710 \text{ lbs/ft}^3$$

7. Excess Air NOT APPLICABLE

$$EA = 100 \left[\frac{\% \text{O}_2 - 0.5 \% \text{CO}}{0.264 \% \text{N}_2 - (\% \text{O}_2 - 0.5 \% \text{CO})} \right]$$

$$EA = 100 \left[\frac{-0.5 x}{0.264 x - (-0.5 x)} \right]$$

$$EA = \quad \quad \quad \%$$

8. Average Duct Velocity

$$V_s = 5129.4 C_p (\sqrt{\Delta P})_{avg} \sqrt{\frac{T_{s \text{ avg}} + 460}{\left(P_{bar} + \frac{P_{s \text{ avg}}}{13.6} \right) M_s}}$$

$$V_s = 5129.4 \times 0.834 \times 0.754 \sqrt{\frac{100.5 + 460}{\left(30.40 + \frac{-0.35}{13.6} \right) 28.59}}$$

$$V_s = 2591. \text{ ft/min}$$

9. Duct Volumetric Flow Rate

$$Q = V_s \times A_s$$

$$Q = 2591. \times 19.63$$

$$Q = 50861. \text{ acfm}$$

10. Duct Volumetric Flow Rate, Corrected to Dry Standard Conditions

$$Q_{\text{std}} = Q \left(1 - \frac{\% \text{ H}_2\text{O}}{100} \right) \left(\frac{T_{\text{std}} + 460}{T_s \text{ avg} + 460} \right) \left(\frac{P_{\text{bar}} + \frac{P_s \text{ avg}}{13.6}}{P_{\text{std}}} \right)$$

$$Q_{\text{std}} = 50861. \left(1 - \frac{5.5}{100} \right) \left(\frac{68 + 460}{100.5 + 460} \right) \left(\frac{30.40 + \frac{-0.35}{13.6}}{29.92} \right)$$

$$Q_{\text{std}} = 45964. \text{ dscfm}$$

11. Isokinetic Factor

$$I = \frac{5.67 (T_s \text{ avg} + 460) (V_m \text{ std})}{\left(P_{\text{bar}} + \frac{P_s \text{ avg}}{13.6} \right) V_s \times T_{\text{total}} \left(1 - \frac{\% \text{ H}_2\text{O}}{100} \right) \left(\frac{(D_n)^2 \times 0.7854}{144} \right)}$$

$$I = \frac{5.67 (100.5 + 460) (102.5)}{\left(30.40 + \frac{-0.35}{13.6} \right) 2591 \times 120 \left(1 - \frac{5.5}{100} \right) \left(\frac{(0.257)^2 \times 0.7854}{144} \right)}$$

$$I = 101. \%$$

12. F - Factor NOT APPLICABLE

$$F = \frac{10^6 (3.64\% \text{ H} + 1.53\% \text{ C} + 0.57\% \text{ S} + 0.14\% \text{ N} - 0.46\% \text{ O})}{\text{GCV}}$$

$$F = \frac{10^6 (3.64 \times +1.53 \times +0.57 \times +0.14 \times -0.46 \times)}{\text{GCV}}$$

$$F = \text{DSCF/MM BTU}$$

13. Actual Particulate Concentration

$$C = \frac{0.01543 \times Mn \left(\frac{T_{std} + 460}{T_{s\ avg} + 460} \right) \left(\frac{P_{bar} + \frac{P_{s\ avg}}{13.6}}{P_{std}} \right) \left(1 - \frac{\%H_2O}{100} \right)}{V_{m\ std}}$$

(UREA AMMONIA FORMALDEHYDE) $\rightarrow$ $\left(\frac{70.6}{450.0} \right)$

$$C = \frac{0.01543 \times \left(\frac{70.6}{450.0} \right) (68 + 460) \left(\frac{30.40 + \frac{-0.35}{13.6}}{29.92} \right) \left(1 - \frac{5.5}{100} \right)}{102.5 (100.5 + 460) (29.92)}$$

$$C = \left(\begin{matrix} 0.0096 \\ 0.0012 \\ 0.00034 \end{matrix} \right) \text{ grains/acf}$$

14. Particulate Concentration, Corrected to Dry Standard Conditions

$$C_s = 0.01543 \times \frac{Mn}{V_{m\ std}}$$

(UREA AMMONIA FORMALDEHYDE) $\rightarrow$ $\left(\frac{70.6}{450.0} \right)$

$$C_s = 0.01543 \times \frac{\left(\frac{70.6}{450.0} \right)}{102.5}$$

$$C_s = \left(\begin{matrix} 0.0106 \\ 0.0077 \\ 0.00038 \end{matrix} \right) \text{ grains/dscf}$$

15. Particulate Emission Rate

$$ER = 0.008571 \times C_s \times Q_{std}$$

(UREA AMMONIA FORMALDEHYDE) $\rightarrow$ $\left(\begin{matrix} 0.0106 \\ 0.0077 \\ 0.00038 \end{matrix} \right)$

$$ER = 0.008571 \times \left(\begin{matrix} 0.0106 \\ 0.0077 \\ 0.00038 \end{matrix} \right) \times 45964.$$

$$ER = \left(\begin{matrix} 4.18 \\ 26.7 \\ 0.15 \end{matrix} \right) \text{ lbs/hr.}$$

16. Particulate Emission NOT APPLICABLE

$$E = 0.0001429 C_s \times F \left(\frac{20.9}{20.9 - \%O_2} \right)$$

$$E = 0.0001429 \times \quad \times \quad \left(\frac{20.9}{20.9 -} \right)$$

$$E = \quad \text{lbs/MM BTU}$$

17. Particulate Concentration Corrected to Dry Standard Conditions and 12% CO₂
NOT APPLICABLE

$$C_s @ 12\% CO_2 = \frac{12}{\% CO_2} \times C_s$$

$$C_s @ 12\% CO_2 = \frac{12}{\quad} \times$$

$$C_s @ 12\% CO_2 = \quad \text{grains/dscf @ 12\% CO}_2$$

18. Particulate Concentration Corrected to Dry Standard Conditions and 50% Excess Air NOT APPLICABLE

$$C_s @ 50\% EA = \frac{\% EA + 100}{150} \times C_s$$

$$C_s @ 50\% EA = \frac{+ 100}{150} \times$$

$$C_s @ 50\% EA = \text{grains/dscf @ 50\% EA}$$

19. Particulate Concentration Based on Duct Gas Weight

$$C_{Lb} = 0.1429 \times \frac{C}{D_{st avg}}$$

(UREA
AMMONIA
FORMALDEHYDE) $\rightarrow$ $\begin{pmatrix} 0.0096 \\ 0.0612 \\ 0.00034 \end{pmatrix}$
 $C_{Lb} = 0.1429 \times \frac{0.0710}{0.0710}$

$$C_{Lb} = \begin{pmatrix} 0.019 \\ 0.123 \\ 0.00068 \end{pmatrix} \text{ lbs/1000 lbs duct gas (uncorrected)}$$

20. Particulate Concentration Based on Duct Gas Weight Corrected to 12% CO₂ NOT APPLICABLE

$$C_{Lb} @ 12\% CO_2 = \frac{C_s @ 12\% CO_2 \times 0.104 (T_{std} + 460)}{(0.44 \times \% CO_2) + (0.28(\% CO + \% N_2)) + (0.32 \times \% O_2)}$$

$$C_{Lb} @ 12\% CO_2 = \frac{\times 0.104 (528)}{(0.44 \times) + (0.28(+)) + (0.32 \times)}$$

$$C_{Lb} @ 12\% CO_2 = \text{lbs/1000 lbs dry corrected to 12\% CO}_2$$

21. Particulate Concentration Based on Duct Gas Weight Corrected to 50% Excess Air NOT APPLICABLE

$$C_{Lb} @ 50\% EA = \frac{C_s @ 50\% EA \times 0.104 (T_{std} + 460)}{(0.44 \times \% CO_2) + (0.28(\% CO + \% N_2)) + (0.32 \times \% O_2)}$$

$$C_{Lb} @ 50\% EA = \frac{\times 0.104 (528)}{(0.44 \times) + (0.28(+ .)) + (0.32 \times)}$$

$$C_{Lb} @ 50\% EA = \text{lbs/1000 lbs dry @ 50\% EA}$$

APPENDIX B
PARTICLE SIZE TESTS RESULTS

INCLUDES:

- B-1. DISCUSSION OF PARTICLE SIZE TESTING**
- B-2. PARTICLE SIZE FIELD DATA SHEETS**
- B-3. LAB WEIGHING DATA**

APPENDIX B-1
DISCUSSION OF PARTICLE SIZE TESTING

DISCUSSION OF PARTICLE SIZE TESTING

The problem of probe plugging during the particle sizing scrubber inlet tests dictated use of a 1/8" I.D. sampling nozzle. The nozzle size, therefore, governed the sampling rate through the impactor necessary to maintain isokinetic sampling conditions. If the plugging was not a problem at the scrubber inlet, it would have been preferable to use a smaller nozzle; this would have allowed a longer sampling time and a larger range of measured particle sizes.

It was not possible to plot cumulative size distribution curves because 99.9% of the particulate matter was collected in the cyclone preseparator. Therefore, it is only possible to conclude that essentially all of the particulate matter collected had diameters greater than the size cutoff of the impactor.

A Sierra Model 226 multi-stage cascade impactor was operated in its instack mode and attached directly to the front end of a standard EPA Method 5 train. Sampling was performed isokinetically, and data was recorded in much the same manner as for the EPA Method 5 train with the exception that sampling was performed at only one point (center of the inlet duct).

The impactor unit consists of a cyclone preseparator and six impactor stages followed by a built-in backup filter stage. All parts are constructed of stainless steel. The unit is designed to hold seven preweighed radially slotted glass fiber filters and one glass fiber backup filter.

An impactor with particle collector filters on each stage was used because it offered low tare weights and caused minimal particle reentrainment (compared to steel plate collection). The sampled air stream accelerates through four radial

slots on every stage. A particle will pass through each stage until it acquires sufficient momentum to impact on one of the collector filters. The backup filter is designed to capture all particulates which do not deposit on a stage filter.

The particle size cutoff for the preseparator is calculated from the manufacturer's calibration data. The cutoffs of each impactor stage depend on the operating conditions during a test. The equations used to calculate these impactor stage cutoffs are as follows:

$$D_{p,50} = (D_{p,50})_r \frac{M C_r P_{pr} Q_r^{1/2}}{m_r C P_p Q}$$

Where

- $D_{p,50}$ = measured particle dia, μm
- r = calibration values from impactor manual
- η = gas viscosity, micropoise
- C = Cunningham slip correction factor, dimensionless
- ρ_p = particle mass density, gm/cc
- Q = volumetric flow rate, acfm

and

$$\mu = \frac{BT^{3/2}}{T+S}$$

where

- B = $14.58 \mu\text{g sec}^{-1}\text{cm}^{-1} (^\circ\text{K})^{-1/2}$
- S = 110.4°K
- T = $^\circ\text{K}$

and

$$C = 1 + \frac{2\lambda}{d} (0.257 + 0.400 \exp \{-1.10d/2\lambda\})$$

Where

d = particle diam, μm

λ = mean free path, μm

and

$$\lambda = 112.96 \frac{n}{p} T^{\frac{1}{2}}$$

where

T = gas temp, $^{\circ}\text{K} = ^{\circ}\text{C} + 273$

P = pressure, atmospheres

m = gas molecular wt (wet), g/g-mole.

Note that graviational constant, gc, is not shown

and

$$Q = \frac{V_m}{DGF} \frac{T_s}{P_s} \frac{P_m}{T_m} \frac{1}{5}$$

where

Q = volumetric flow rate through impactor, acfm

V_m = dry gas meter volume, ft^3

DGF = dry gas fraction

T_s = stack temp, $^{\circ}\text{R}$

P_s = stack pressure, "Hg

T_m = meter temp, $^{\circ}\text{R}$

P_m = meter press., "Hg

t = time, min.

and

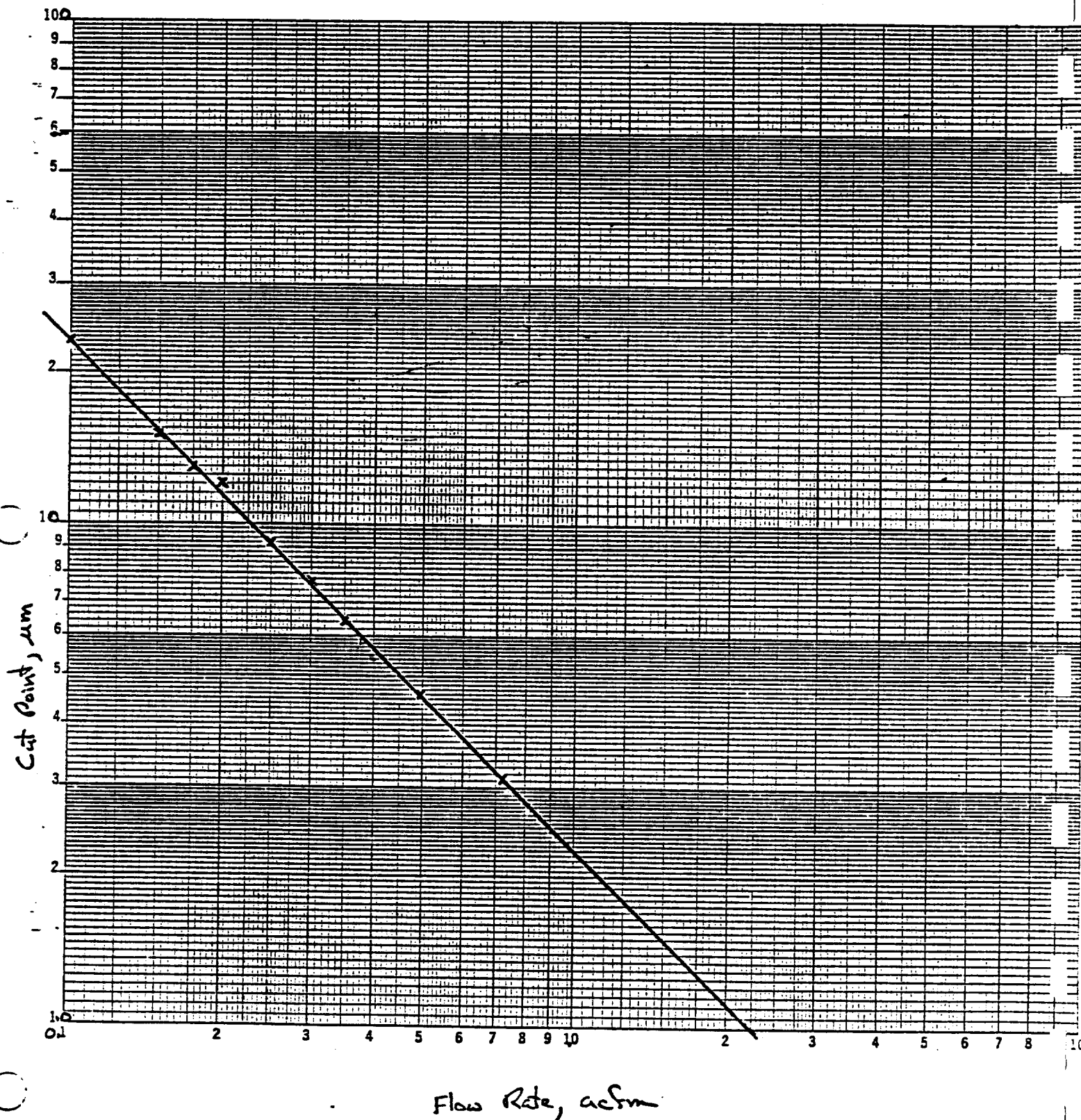
$$DSF = 1 - \%H_2O/100$$

The cutoff size for the cyclone preseparator was determined from Figure B1-

1. The actual stack flow rate, Q_{acfm} , used to determine the cutoff size, was

Figure B1-1

Sierra 220 Cyclone Preseparator cut points as a function of Flow rate*



* data from Sierra Bulletin No 678-220 and Sierra Series 210 Cascade Impactor Cut Points sheet dated April 1, 1978.

calculated by a computer program, whose results are presented in Table B1-1.

The cascade impactor determines aerodynamic particle size distribution. Aerodynamic particle sizing differs from geometric sizing in that a mass of particles containing various shapes and densities is fractionated and collected according to each particles aerodynamic characteristics. A particle collected on a given stage is aerodynamically equivalent in size to the unit density sphere (1g/cc) collected on that stage during calibration.

Particle size distributions are normally plotted cumulatively on log-normal graph paper. In this format, the mass of particulate collected on a given stage and all stages of smaller D_p is plotted against the D_p of that stage as a percent of the total mass of particulate collected on all stages (including the backup filter). The size is typically represented as an "Equivalent Aerodynamic Diameter": the size of a spherical particle of density 1 g/cc which has the same terminal settling velocity as the sampled particle.

Each of the eight filters was placed in a petri dish and transferred to the laboratory for analysis. Each filter was dessicated and weighed repeatedly until a constant weight was obtained. The contents of the cyclone preseparator sample container were collected in a glass jar, transferred to a pre-weighed beaker, dessicated, and weighed to a constant weight. Results are reported to the nearest 0.01 mg.

While standard procedures were followed in these tests, a more detailed sampling method would be required in order to obtain a meaningful particle size distribution. For example, several points across the duct should be sampled in light of evidence from the scrubber gas sampling traverses that the particle distribution across the duct was not uniform. In addition, larger particle size cutoffs would be needed.

CASCADE IMPACTOR
Table B1-1 : DATA REDUCTION INPUTS/OUTPUTS

Inputs	Test #1	Test #2	Test #3
M _T , mg	1321.99	1118.42	271.26
Vm, ft ³	4.59	4.61	4.50
P _{bar} , "Hg	30.27	30.17	30.14
Δh, "H ₂ O	0.28	0.27	0.262
Tm, °F	80.7	67	72.7
DGF	0.97	0.977	0.977
Is, °F	197	201	203.3
Ps, "H ₂ O	-2.5	-2.5	-2.5
Time, min	15	15	15
m, g/g-mole	28.6	28.6	28.6
ρ, g/cc	1.0	1.0	1.0
ρ, g/cc			
Outputs			
Vm, dscf	4.521	4.643	4.479
C, gr/dscf	4.486	3.695	0.929
Q, acfm	0.3755	0.3972	0.3850
η, micropoise	214.2	215.1	215.7
λ, μm	0.0870	0.0876	0.0880
Cyclone μm	6.00	5.70	5.85
Cutoff Stage 1 (ρ=1.0) μm	15.4	15.0	15.3
Cutoff Stage 2 (ρ=1.0) μm	9.4	9.2	9.4
Cutoff Stage 3 (ρ=1.0) μm	3.7	3.6	3.7
Cutoff Stage 4 (ρ=1.0) μm	2.3	2.2	2.2
Cutoff Stage 5 (ρ=1.0) μm	1.4	1.4	1.4
Cutoff Stage 6 (ρ=1.0) μm	0.8	0.7	0.8
Cyclone			
Stage 0	1321.2 mg	1117.65 mg	270.67 mg
Stage 1	nd.	0.20 mg	0.12 mg
Stage 2	0.43 mg	0.34 mg	nd.
Stage 3	0.17 mg	0.05 mg	0.24 mg
Stage 4	nd.	nd.	nd.
Stage 5	nd.	nd.	nd.
Stage 6	nd.	nd.	nd.
Filter	0.19 mg	0.18 mg	0.23 mg
mg, TOTAL wt	1321.99	1118.42	271.26
gr, TOTAL wt	20.40	17.26	4.11
gr/dscf, CONCENTRATION	4.512	3.717	0.935

APPENDIX B-2

PARTICLE SIZE FIELD DATA SHEETS

FIELD DATA SHEETS		Nozzle No. & Dia.		0.125		Homograph	
Pump No.		12				In. C Factor	
Plant Location		Depueville		D-6		Pitot Coefficient	
Test No. #1		Cascade Properties		75		0.829	
Sampling Location		Down the street		30.27		min. Orifice All	
Pitot Project No.		82985-20		35		1.88	
Date		1/18/79		Probe Heater Setting		min. Test Start Time	
Tester		JFK-DK		Filter Temp. Setting		3:28 PM	
Duct Area		(48" dia)		Probe Heater Setting		amps Test End Time	
				Filter Temp. Setting		3:43	
				Dry Gas Meter Y		Leak Test Start	
				1.50		0.01 CFME	
						Leak Test Final	
						0.001 CFME	

Port	Point	Time H:m	Velocity AP in H ₂ O	ΔH in H ₂ O	T _M in °F	T _M Out °F	P _{STACK} in H ₂ O	T _{STACK} °F	Initial Meter Volume cu. ft.	P R Q S E	Cyc. Angle	Heater Box Temp.	Temp. of Gas Leaving Condenser °F	Pump vac. in Hg	Probe Temp. °F	O ₂ Conc. %
R	MA	501	40	028	80	81	-250	197	313	5		-	68	1	-	
	MA	501	40	028	80	81	-250	197	315	5		-	70	1	-	
	MA	501	40	028	79	80	-250	197	316	5		-	70	1	-	
									318	5						

B-10

Probe Wash _____

Silica Gel _____

Silica Gel Cond. _____

Cleaning Patch _____

H₂O Collected _____

H₂O Condition _____

Firm Name CF Ind Pump No. 12 FIELD DATA SHEETS Nozzle No. & Dia. I-6 (0.1276) Nomograph In. C Factor 0.98
 Plant Location Dallas Orifice No. D-6 Assumed Moisture 4 Pitot Coefficient 0.823
 Test No. 3 - Canada Inspector Ambient Temp 65 °F Test Duration 15 min. Orifice Alt. 1.88
 Sampling Location Scrubber inlet Bar Press 30.14 "Hg Traverse Point Interval 15 min. Test Start Time 11:25 AM
 Hg Project No. 82988-20 Probe Ident. No. 35 Probe Heater Setting NA amps Test End Time 11:40 AM
 Date 11/9/79 Filter Ident. No. NA Filter Temp. Setting NA
 Tester DEF DAK Duct Area (49 1/2" dia) ft² Dry Gas Meter Y 1.50 Leak Test Start 0.000 CFME "Hg 8
 Leak Test Final 0.001 CFME "Hg 5

horizontal duct

Port	Point	3	4	5	6	7	8	9	10	12	13	14	15	18	19	20	23	24	25	26	27	28	29	30	32	33	34	35	40	41	42	43	44	45	50	52	53	Heater Box Temp.	Temp. of Gas Leaving Condenser °F	Pump Vac. in Hg	Probe Temp. °F	O ₂ Conc. %																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																
		Time Min			Velocity ΔP in H ₂ O						All in H ₂ O			T _M in °F		T _M Out °F		P STACK in H ₂ O			T STACK °F			Initial Meter Volume cu. ft.			P R O E			Cyc. Angle °																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																												
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Probe Wash _____ Cleaning Patch _____
 Silica Gel _____ H₂O Collected _____
 Silica Gel Cond. _____ H₂O Condition _____

ORSAT Measurements
 Time 1 2 3
 CO₂ O₂ CO N₂

Orsat Bag Sample Triplicate Analysis
 CO₂ O₂ CO N₂

horizontal duct
 Inspector prefabricated in stock
 10 minutes. returned
 authentication

APPENDIX B-3
LAB WEIGHING DATA

CASCADE IMPACTOR SUBSTRATES

TEST #	CI-1	CI-2	CI-3
STAGE 0 filter #	706	713	720
1 "	705	712	719
2 "	704	711	718
3 "	703	710	717
4 "	702	709	716
5 "	701	708	715
6 "	700	707	714
backup "	300	301	302

✓ used
EMS/2/79

FILTER WEIGHTS

Type Filter Slotted cascades

Filter Number	Initial Weight (g)	Initials:	Date Weighed
✓ 700	.06690	EMS	1-9-79
✓ 701	.06819		
✓ 702	.06954		
✓ 703	.06707		
✓ 704	.06821		
✓ 705	.06791		
✓ 706	.06844		
✓ 707	.06789		
✓ 708	.06930		
✓ 709	.06769		
✓ 710	.06938		
✓ 711	.06808		
✓ 712	.06788		
✓ 713	.06690		
✓ 714	.06775		
✓ 715	.06929		
✓ 716	.06862		
✓ 717	.06742		
✓ 718	.06701		
✓ 719	.06834		
✓ 720	.06971		
✓ 721	.06930		
✓ 722	.06754	✓	✓

FILTER WEIGHTS

Back-up's

plain

Type Filter

cascade

Back-up's

Filter Number	Initial Weight (g)	Initials	Date Weighed
✓ 300	.08341	EMS	1-9-79
✓ 301	.08536		
302 ✓	.08383 ✓		
303 ✓	.08172		
304	.08148		
305	.08221		
306	.08188		
307	.08309		
308	.08619		
309	.08206		
310	.08690	↓	↓
✓ 311	.08690	EMS	1-23-79
used EMS 2/1 ✓ 312	.08425		
✓ 313	.08214		
314	.08186		
315	.08270		
316	.08650		
317	.08617		
318	.08276		
319	.08379		
320	.08236	↓	↓

Client: EPA - CF Industries
 Contract No: 82988-20
 Reviewed by: V. Manchesse
 Report to: Rud.

Laboratory No: 48-77(1)
 Date Received: 1-23-79
 Date Reported: 2-15-79

Type Sample: Filter Impactor Fuel Oil Sediment Impinger Other

Sample Number	Location	Analysis No. 1 <i>blue mg</i>	Analysis No. 2	Analysis No. 3	Analysis No. 4	Analysis No. 6	TLV
CT-1	Probe + Nozzle	1.3212 grams	1321.2 <i>mg</i>				
filter 300		0.19 mg					
700		n.d. ↓					
701		n.d.					
702		n.d.					
703		n.d.					
704		0.17					
705		0.43					
706		n.d.					
CT-2	Probe + Nozzle	1.11765 grams					
filter 301		0.18 mg					
707		n.d. ↓					
708		n.d.					
709		n.d.					

Analyzed by: EMS
 Form CL-9012

Client: Et. - CF Industries
 Contract No: 82968-20
 Reviewed by: U. Marchese
 Report to: RUC
 Laboratory No: 48-79(1)
 Date Received: 1-23-79
 Date Reported: 2-15-79

Type Sample: Filter Impaction Fuel Oil Sediment Impinger Other

Sample Number	Location	Analysis No. 1 <i>total mg</i>	Analysis No. 2	Analysis No. 3	Analysis No. 4	Analysis No. 6	TLV
<i>filter</i> 710		<i>n.d.</i>					
711		<i>0.05 mg</i>					
712		<i>0.34</i>					
713		<i>0.20</i>					
<i>Q-I-3</i>	<i>Probe + nozzle</i>	<i>270.67</i>					
<i>filter</i> 702		<i>0.23</i>					
714		<i>n.d.</i>					
715		<i>n.d.</i>					
716		<i>n.d.</i>					
717		<i>n.d.</i>					
718		<i>0.24</i>					
719		<i>n.d.</i>					
720		<i>0.12</i>					

APPENDIX C
VISIBLE EMISSIONS RESULTS

INCLUDES:

- C-1 Visible Emissions Summary Tables for Outlet Stack "B" Granulator**
- C-2 Visual Emission Recertification Certificate**
- C-3 Visible Emission Field Data Sheets**
- C-4 Guidelines for EPA Method 9**

APPENDIX C-1

VISIBLE EMISSIONS SUMMARY TABLES

FOR

OUTLET STACK "B" GRANULATOR

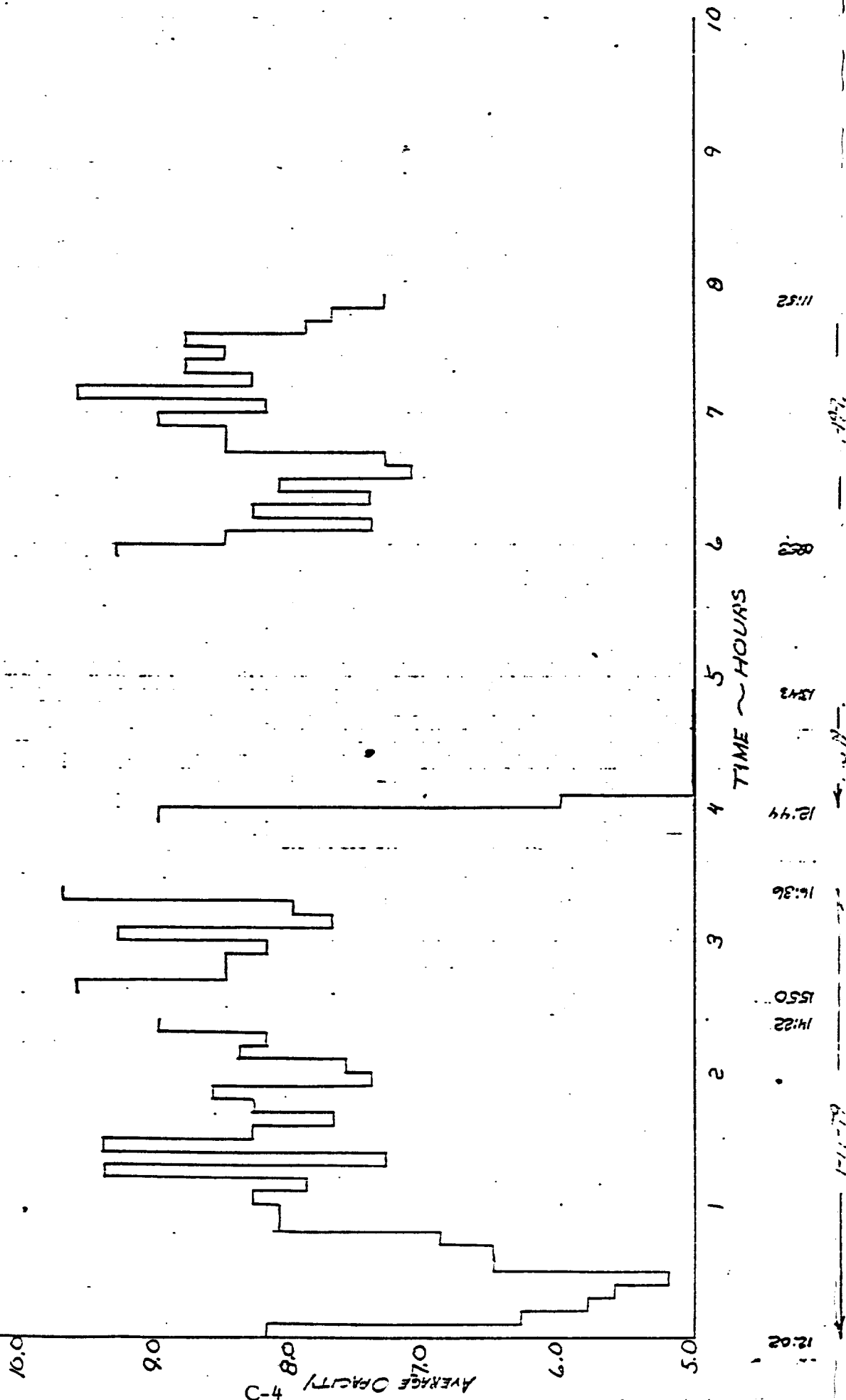
SIX MINUTE ARITHMETRIC AVERAGES OF JANUARY 17, 18 AND 19, 1979
 OPACITY READINGS ON "B" GRANULATOR SCRUBBER STACK AT CF INDUSTRIES, INC.,
 IN DONALDSONVILLE, LOUISIANA

TIME	AVG. OPACITY FOR 6 MIN.	DATE
12:02 - 12:07	8.2	1-17-79
12:08 - 12:13	6.3	
12:14 - 12:19	5.8	
12:20 - 12:25	5.6	
12:26 - 12:31	5.2	
12:32 - 12:37	6.5	
12:38 - 12:43	6.5	
12:44 - 12:49	6.9	
12:50 - 12:55	8.1	
12:56 - 13:01	8.1	
13:02 - 13:07	8.3	
13:08 - 13:13	7.9	
13:14 - 13:19	9.4	
13:20 - 13:25	7.3	
13:26 - 13:31	9.4	
13:32 - 13:37	8.3	
13:38 - 13:43	7.7	
13:44 - 13:49	8.3	
13:50 - 14:55	8.6	
13:56 - 14:01	7.4	
14:02 - 14:07	7.6	
14:08 - 14:13	8.4	
14:14 - 14:19	8.8	
14:20 - 14:22*	9.0	
15:50 - 15:55	9.6	
15:56 - 16:01	8.5	
16:02 - 16:07	8.5	
16:08 - 16:13	8.2	
16:14 - 16:19	9.3	
16:20 - 16:25	7.7	
16:26 - 16:31	8.0	
16:32 - 16:36*	9.7	
12:44 - 12:49	9.0	1-18-79
12:50 - 12:55	6.0	

TIME	AVG. OPACITY FOR 6 MIN.	DATE
12:56 - 13:01	5.0	1-18-79
13:02 - 13:07	5.0	
13:08 - 13:13	5.0	
13:14 - 13:19	5.0	
13:20 - 13:25	5.0	
13:26 - 13:31	5.0	
13:32 - 13:37	5.0	
13:38 - 13:43	5.0	
09:53 - 09:58	9.3	1-19-79
09:59 - 10:04	8.5	
10:05 - 10:10	7.4	
10:11 - 10:16	8.3	
10:17 - 10:22	7.4	
10:23 - 10:28	8.1	
10:29 - 10:34	7.1	
10:35 - 10:40	7.3	
10:41 - 10:46	8.5	
10:47 - 10:52	8.5	
10:53 - 10:58	9.0	
10:59 - 11:04	8.2	
11:05 - 11:10	9.6	
11:11 - 11:16	8.3	
11:17 - 11:22	8.8	
11:23 - 11:28	8.5	
11:29 - 11:34	8.8	
11:35 - 11:40	7.9	
11:41 - 11:46	7.7	
11:47 - 11:52	7.3	

* Less than 6 min average

Six Minute Arithmetic Averages of January 17, 18 and 19, 1979 Opacity Readings
on "1/3" Granular Scatterer Stud at CP Industries, Inc. in Donaldsonville, Louisiana



APPENDIX C-2
VISUAL EMISSION RECERTIFICATION CERTIFICATE



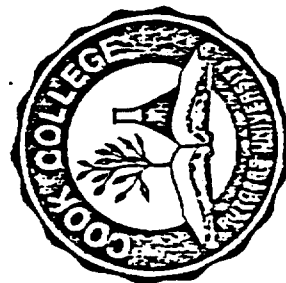
COOK COLLEGE
New Brunswick, New Jersey

THIS IS TO CERTIFY THAT

CARL EKROTH

HAS COMPLETED THE SHORT COURSE
VISUAL EMISSIONS EVALUATION RECERTIFICATION

SEPTEMBER 28, 1978



Grant H. Halton

Dean

APPENDIX C-3
VISIBLE EMISSIONS FIELD DATA SHEETS

SUMMARY
RECORD OF VISIBLE EMISSIONS

Type of Plant UREA

Date 1-17-79

Company Name CF INDUSTRIES

Hours of Observation 1202 - 1423

Plant Address DONALDSONVILLE, LA.

Observer CSF

Type of Discharge STACK OTHER _____

Discharge Location N

Height of Point of Discharge ~80'

Observer's Location:

Distance to Discharge Point 150'

Height of Observation Point ~~160~~ 90'

Direction from Discharge Point S

Background Description RAIL BED, GROUND

Weather: Clear Overcast Partly Cloudy Other _____ Color _____

Wind Direction S Wind Velocity 10 mi/hr

Plume Description:

Detached: Yes No

Color: Black White Other _____

Plume Dispersion Behavior: Looping Coning Fanning

Lofting Fumigating Other _____

Estimated Distance Plume Visible 10'

Summary of Observations:

Opacity	Aggregate Time @ Opacity		Opacity	Aggregate Time @ Opacity
	min.	sec.		
0			55	
5			60	
10			65	
15			70	
20			75	
25			80	
30			85	
35			90	
40			95	
45			100	
50				

RECORD OF VISIBLE EMISSIONS

①

Company Name CF INDUSTRIESDate 1-17-79Plant Address DONALDSONVILLE, LA.Observer CSEStack Location N

Observer's

Location SeUTWeather Conditions MC, HZ, WIND-10 MPH

		TIME				COMMENTS	
HR	MIN	SECONDS					
		00	15	30	45		
1202	00	10	15	15	10	Plume from cooling towers sometimes obscures stack plume. in this case I will use a slash to indicate this. Periodically the sky becomes more overcast blocking out the sun altogether, at these times it is nearly impossible to get an accurate reading.	
	01	10	10	5	5		
	02	10	10	5	—		
	03	—	5	5	5		
1206	04	5	10	10	10		
	05	5	10	5	5		
	06	5	5	10	10		
	07	5	5	10	10		
1210	08	5	5	5	5		
	09	5	5	10	5		
	10	5	5	5	5		
	11	5	10	5	5		
	12	5	5	5	10		
	13	10	5	5	5		
	14	5	5	10	5		sky now heavily overcast has been for last 6 minutes.
	15	5	5	5	5		
16	5	5	5	5			
17	5	10	5	5			
	18	5	10	10	5		
	19	5	10	5	5		
	20	5	5	5	5		
	21	5	5	5	5		
1222	22	5	5	5	5		
	23	5	5	5	5		
	24	5	5	5	5		
	25	5	5	5	5		
1226	26	5	5	5	5		
	27	10	5	5	5		
	28	5	5	5	5		
	29	5	5	5	5		
1231							

RECORD OF VISIBLE EMISSIONS

Company Name CF INDUSTRIES
 Plant Address DOVALDENVILLE, LA
 Stack Location N
 Weather Conditions HVY OVERCAST

Date 1-17-79
 Observer CSF
 Observer's Location S

②

HR	MIN	TIME				COMMENTS
		00	15	30	45	
1232	30	5	5	5	5	sky still heavily overcast. Wind is
	31	5	10	10	5	still about the same direction & speed
	32	5	10	5	5	
	33	5	5	5	5	
1236	34	10	10	10	5	
	35	5	10	5	5	
	36	5	5	10	10	
1239	37	10	10	10	10	
	38	10	5	5	5	
	39	5	5	5	5	
	40	5	5	5	5	
1243	41	5	5	5	5	
	42	10	5	10	5	
	43	5	10	5	10	
1246	44	5	5	10	10	
	45	5	5	5	10	
	46	10	5	5	5	
1249	47	5	5	5	10	
	48	10	5	10	10	
	49	5	5	5	10	
1252	50	10	10	10	10	
	51	5	10	10	5	
	52	10	10	5	5	wind beginning to shift around more from
1255	53	5	10	10	10	SSW now
	54	10	5	5	5	sky is now brightening a little
	55	10	10	5	10	
1258	56	10	10	10	5	
	57	5	10	10	10	
1300	58	5	10	10	10	
	59	10	10	5	5	

RECORD OF VISIBLE EMISSIONS

3

Company Name _____

Date _____

Plant Address DONALDSONVILLE, LA

Observer CSF

Stack Location N

Observer's Location _____

Weather Conditions HVY OVERCAST

Location S

	TIME					COMMENTS	
	HR	MIN	SECONDS				
			00	15	30		45
1302		00	5	5	5	5	
		01	5	10	10	5	
		02	5	10	10	10	
1305		03	5	10	10	10	
		04	10	10	10	10	
		05	10	10	10	10	
1308		06	10	10	5	10	
		07	5	10	10	10	
		08	10	10	10	10	
1311		09	5	5	5	5	sky getting dark again
		10	5	10	10	5	
		11	5	10	10	5	
1314		12	5	10	10	10	
		13	10	15	10	10	
		14	10	10	10	10	plume seems to be swirling around
		15	15	10	10	10	mouth of stack
1318		16	10	10	5	5	
		17	10	5	5	10	
		18	10	10	10	10	
		19	10	10	10	10	
1322		20	5	10	5	5	
		21	5	5	5	10	
		22	5	5	5	5	
1325		23	5	5	5	10	
		24	10	10	10	10	
		25	10	10	10	10	
		26	5	10	5	10	
		27	10	10	10	10	
1330		28	10	5	10	10	
		29	10	10	10	10	

RECORD OF VISIBLE EMISSIONS

4

Company Name CE INDUSTRIES

Date 1-17-79

Plant Address Daytonville, LA.

Observer CSZ

Stack Location N

Observer's Location S.

Weather Conditions overcast

HR	MIN	TIME				COMMENTS
		00	15	30	45	
1332	30	10	10	5	10	
	31	5	10	5	10	
	32	10	10	5	10	wind appears to have swung around to the SSE.
1335	33	10	10	10	5	
	34	10	10	10	5	
	35	10	10	5	5	
	36	5	5	5	5	
1339	37	5	10	10	10	sun trying to break through
	38	5	5	10	10	
	39	10	5	5	10	
	40	10	5	10	10	
	41	10	5	10	10	
	42	5	5	5	10	
1345	43	5	5	5	5	
	44	10	10	10	10	
	45	10	10	10	10	
	46	10	10	10	10	
	47	10	10	5	—	
1350	48	10	10	5	10	
	49	10	10	10	10	
	50	10	10	10	10	
	51	5	10	—	10	
	52	—	5	5	5	
1355	53	5	—	10	10	
	54	5	10	5	5	
	55	5	10	5	5	
	56	5	5	5	5	
	57	5	5	5	10	
1400	58	5	5	—	—	
	59	—	5	10	10	

RECORD OF VISIBLE EMISSIONS

Company Name CE INDUSTRIES

Date 1-17-79

Plant Address DONALDSONVILLE, LA.

Observer CSE

Stack Location N

Observer's Location S

Weather Conditions OURCST

HR	MIN	TIME				COMMENTS
		SECONDS	00	15	30	45
1402	00	10	10	10	—	
	01	5	10	10	10	
	02	10	5	—	5	
1405	03	10	5	—	—	
	04	—	—	5	5	
	05	10	—	5	5	
	06	10	10	10	10	
	07	10	5	5	5	
	08	5	—	—	—	
	09	5	10	10	10	
	10	—	—	—	—	
	11	10	10	—	10	
	12	5	—	10	10	
1415	13	10	10	5	—	
	14	—	—	—	10	
	15	10	10	—	—	
	16	5	10	5	10	
	17	10	5	10	5	
1420	18	—	10	10	10	
	19	10	—	10	10	
	20	10	10	5	5	ended test.
	21					
	22					
	23					
	24					
	25					
	26					
	27					
	28					
	29					

SUMMARY RECORD OF VISIBLE EMISSIONS

Type of Plant UREA

Date 1-17-79

Company Name CF INDUSTRIES

Hours of Observation 1550-1638

Plant Address DONALDSONVILLE, LA.

Observer CSE

Type of Discharge STACK OTHER _____

Discharge Location NORTH

Height of Point of Discharge 80'

Observer's Location:

Distance to Discharge Point 150'

Height of Observation Point 90'

Direction from Discharge Point South

Background Description Rail Bed, Ground

Weather: Clear Overcast Partly Cloudy Other _____ Color _____

Wind Direction ESSE Wind Velocity 5 mi/hr

Plume Description:

Detached: Yes No

Color: Black White Other _____

Plume Dispersion Behavior: Looping Coning Fanning

Lofting Fumigating Other _____

Estimated Distance Plume Visible _____

Summary of Observations:

Opacity	Aggregate Time @ Opacity		Opacity	Aggregate Time @ Opacity
	min.	sec.		
0			55	
5			60	
10			65	
15			70	
20			75	
25			80	
30			85	
35			90	
40			95	
45			100	
50				

RECORD OF VISIBLE EMISSIONS

①

Company Name CE INDUSTRIES

Date 1-17-79

Plant Address DONALDSONVILLE, LA.

Observer CSE

Stack Location N

Observer's Location 5

Weather Conditions HYV OVCST, HAZY

TIME						COMMENTS
HR	MIN	SECONDS				
		00	15	30	45	
1550	00	10	10	10	10	Sky is heavy overcast and due to the lateness of the hour, the light is fading, making it difficult to obtain any real accurate readings.
	01	10	10	10	10	
	02	10	10	10	10	
	03	10	10	10	10	
	04	10	10	10	10	
1555	05	5	5	10	-	The plume from cooling towers periodically obscures stack plume, these readings indicated by slash.
	06	5	10	10	10	
	07	5	10	10	10	
	08	5	10	10	10	
	09	10	10	10	10	
1600	10	5	5	5	10	
	11	10	10	10	5	
	12	10	10	10	10	
	13	5	10	10	10	
	14	10	10	10	5	
1605	15	10	10	5	5	
	16	5	5	5	-	
	17	10	10	10	10	
	18	5	10	5	-	
	19	10	5	-	10	
1610	20	10	10	10	10	
	21	10	-	-	10	
	22	5	5	-	5	
	23	5	10	10	10	
	24	-	10	10	10	
1615	25	10	5	10	10	
	26	10	5	5	-	
	27	10	10	10	10	
	28	10	-	10	10	
	29	10	10	-	10	

RECORD OF VISIBLE EMISSIONS

Company Name C F INDUSTRIES

Date _____

Plant Address DONALDSONVILLE, LA

Observer CSE

Stack Location N

Observer's Location S

Weather Conditions HVY OVRCAST, BKN HZ

TIME						COMMENTS
HR	MIN	SECONDS				
		00	15	30	45	
1620	30	-	10	10	-	
	31	-	-	10	10	
	32	5	5	-	-	
	33	10	5	-	5	
	34	5	5	-	-	
1625	35	5	10	10	10	
	36	-	10	5	5	
	37	10	5	10	10	
	38	10	10	5	5	
	39	10	10	5	10	
1630	40	5	5	10	10	
	41	10	5	10	10	
	42	10	10	10	10	
	43	10	10	5	-	
	44	10	10	10	10	
1635	45	10	10	10	10	
	46	10	10	10	10	too dark to get any more readings
	47					
	48					
	49					
	50					
	51					
	52					
	53					
	54					
	55					
	56					
	57					
	58					
	59					

SUMMARY
RECORD OF VISIBLE EMISSIONS

Type of Plant UREA

Date 1-18-79

Company Name CF INDUSTRIES

Hours of Observation 1244-1344

Plant Address DONALDSONVILLE, LA

Observer C. S. Ekroth

Type of Discharge STACK OTHER _____

Discharge Location N

Height of Point of Discharge 80'

Observer's Location:

Distance to Discharge Point 150'

Height of Observation Point 90'

Direction from Discharge Point S

Background Description Rail Bed / ground

Weather: Clear Overcast Partly Cloudy Other _____ Color Light

Wind Direction N NE Wind Velocity 0-5 mi/hr

Plume Description:

Detached: Yes No

Color: Black White Other _____

Plume Dispersion Behavior: Looping Coning Fanning

Lofting Fumigating Other _____

Estimated Distance Plume Visible 10'

Summary of Observations:

Opacity	Aggregate Time @ Opacity		Opacity	Aggregate Time @ Opacity
	min.	sec.		
0			55	
5			60	
10			65	
15			70	
20			75	
25			80	
30			85	
35			90	
40			95	
45			100	
50				

RECORD OF VISIBLE EMISSIONS

①

Company Name CF INDUSTRIESDate 1-18-79Plant Address DONALDSONVILLE, LAObserver CSSStack Location NObserver's Location S

Weather Conditions _____

HR	MIN	TIME				COMMENTS
		SECONDS	00	15	30	45
1244	00	10	10	10	10	Sky is high overcast, with haze, clouds are beginning to break up. Sun is trying to come out. Sky is very bright. Wind is calm.
1245	01	10	10	10	10	
	02	10	5	10	10	
	03	10	10	5	10	
	04	5	5	10	10	
	05	10	10	5	10	
1250	06	5	5	10	10	
	07	10	10	5	10	
	08	5	5	5	5	
	09	5	5	5	5	sun is brighter now. Background is hazy which accounts for lower readings
	10	5	5	5	5	
1255	11	5	5	5	5	
	12	5	5	5	5	
	13	5	5	5	5	
	14	5	5	5	5	
	15	5	5	5	5	
1300	16	5	5	5	5	
	17	5	5	5	5	
	18	5	5	5	5	
	19	5	5	5	5	
	20	5	5	5	5	
1305	21	5	5	5	5	
	22	5	5	5	5	
	23	5	5	5	5	
	24	5	5	5	5	
	25	5	5	5	5	
1310	26	5	5	5	5	
	27	5	5	5	5	
	28	5	5	5	5	
	29	5	5	5	5	

RECORD OF VISIBLE EMISSIONS

Company Name CF INDUSTRIES

Date 1-18-79

Plant Address DEPAULSVILLE, LA

Observer CSE

Stack Location N

Observer's

Location S

Weather Conditions _____

HR	MIN	TIME				COMMENTS
		00	15	30	45	
1314	30	5	5	5	5	Sky conditions still the same, making for difficulty in getting readings
1315	31	5	5	5	5	
	32	5	5	5	5	
	33	5	5	5	5	
	34	5	5	5	5	
	35	5	5	5	5	
1320	36	5	5	5	5	
	37	5	5	5	5	
	38	5	5	5	5	
	39	5	5	5	5	
	40	5	5	5	5	Brigntness has diminished slightly
1325	41	5	5	5	5	
	42	5	5	5	5	
	43	5	5	5	5	
	44	5	5	5	5	
	45	5	5	5	5	
1330	46	5	5	5	5	
	47	5	5	5	5	
	48	5	5	5	5	What little ice there is now is coming out of the NE.
1333	49	5	5	5	5	
	50	5	5	5	5	
1335	51	5	5	5	5	
	52	5	5	5	5	
	53	5	5	5	5	
	54	5	5	5	5	
	55	5	5	5	5	
1340	56	5	5	5	5	
	57	5	5	5	5	
	58	5	5	5	5	
1343	59	5	5	5	5	

RECORD OF VISIBLE EMISSIONS

Company Name CF INDUSTRIES

Date 1-19-79

Plant Address Donaldsonville, LA

Observer CSZ

Stack Location N

Observer's Location S

Weather Conditions See Below

HR	MIN	TIME				COMMENTS
		SECONDS				
		00	15	30	45	
0953	00	10	10	10	5	heavy over cast, wind from east ^{west} 12-15 mph.
	01	10	10	10	10	High humidity and foggy haze.
0955	02	10	5	10	10	
	03	10	10	10	10	
	04	10	10	10	-	Plume from cooling tower periodically obscures
	05	10	10	10	5	stack plume, these instances are indicated
	06	5	5	10	10	by slash.
1000	07	10	-	10	10	
	08	10	10	10	10	
	09	10	5	10	5	
	10	-	10	10	10	
	11	10	5	10	5	
1005	12	5	5	5	10	
	13	5	10	5	10	
	14	5	10	10	10	
	15	10	-	5	5	
	16	5	10	10	10	
1010	17	5	5	10	5	
	18	5	10	10	10	
	19	5	5	5	10	
	20	10	5	5	5	
	21	10	10	10	10	
1015	22	-	5	5	10	
	23	10	5	10	10	
	24	5	10	10	5	
	25	-	10	5	10	
	26	10	5	10	5	
1020	27	5	5	10	10	
	28	5	5	5	10	
	29	10	5	5	5	

RECORD OF VISIBLE EMISSIONS

②

Company Name CF INDUSTRIESDate 1-19-79Plant Address DONALDSONVILLE, LA.Observer CS 2Stack Location NorthObserver's
Location SouthWeather Conditions OVERCAST. HAZY

HR	MIN	TIME				COMMENTS
		00	15	30	45	
	00	10	10	10	5	
	01	5	10	5	10	
1025	02	10	5	10	10	
	03	5	5	10	10	
	04	10	5	10	5	
	05	5	10	10	10	
	06	5	5	5	10	
1030	07	5	5	10	10	
	08	10	5	5	10	
	09	5	10	5	5	
	10	5	10	10	10	
	11	10	5	5	5	
1035	12	5	10	10	5	
	13	10	10	10	5	
	14	5	5	10	10	
	15	—	10	5	5	
	16	10	10	5	5	
1040	17	10	10	5	5	
	18	5	10	10	10	
	19	10	10	5	5	
	20	5	10	10	5	
	21	10	5	5	10	
1045	22	10	10	10	10	
	23	10	10	10	10	
	24	5	10	5	5	
	25	10	10	10	5	
	26	10	10	10	10	
1050	27	5	5	10	5	
	28	10	—	10	10	
	29	10	10	10	10	

RECORD OF VISIBLE EMISSIONS

Company Name CE INDUSTRIES

Date 1-19-79

Plant Address RODALSONVILLE, LA

Observer CSE

Stack Location N

Observer's

Weather Conditions _____

Location S

TIME						COMMENTS
HR	MIN	SECONDS				
		00	15	30	45	
	30	10	10	5	5	
	31	10	5	10	10	
1055	32	10	10	10	10	
	33	10	10	10	10	
	34	10	10	10	5	
	35	10	10	5	10	
1059	36	10	10	10		heavy steam plume coming from stack
1100	37					some more doing something to fan.
	38	5	10	5	10	
	39	5	10	10	5	
	40	10	10	10	10	
	41	10	10	10	5	
1105	42	10	10	10	10	
	43	10	10	10	10	
	44	10	10	10	10	
	45	5	10	10	5	
	46	10	10	10	10	
1110	47	10	10	10	10	
	48	10	10	5	5	
	49	10	10	5	10	
	50	10	10	5	10	
	51	10	10	10	10	
1115	52	10	5	5	5	
	53	10	5	10	10	
	54	5	10	10	10	
	55	10	5	10	10	
	56	10	10	10	10	
1120	57	10	10	10	5	
	58	10	5	10	5	
	59	10	5	10	10	

RECORD OF VISIBLE EMISSIONS

4

Company Name CE INDUSTRIES

Date 1-19-79

Plant Address DONALDSONVILLE, LA

Observer CSE

Stack Location N

Observer's Location S

Weather Conditions HVY OVCST

HR	MIN	TIME				COMMENTS
		00	15	30	45	
	00	10	10	5	10	
	01	10	10	5	5	
1125	02	10	10	10	10	
	03	10	5	5	10	
	04	10	10	10	5	
	05	10	5	10	10	
	06	10	10	10	5	Sky is becoming a little brighter
1130	07	5	10	10	10	
	08	10	5	5	—	
	09	10	10	10	10	
	10	10	10	10	10	
	11	10	10	10	10	sun is trying to break through
1135	12	5	10	5	5	
	13	5	10	10	10	
	14	10	10	10	5	
	15	5	10	10	5	
	16	5	5	10	5	
1140	17	10	10	10	10	
	18	10	10	10	5	
	19	10	10	10	5	
	20	5	10	5	5	
	21	5	5	5	5	
1145	22	5	10	10	10	
	23	10	10	10	5	
	24	10	5	10	10	
	25	5	5	5	5	
	26	5	10	10	5	
1150	27	5	10	10	10	
	28	10	5	10	5	
1152	29	10	5	5	5	

APPENDIX C-4

GUIDELINES FOR EPA METHOD 9

DRAFT COPY

SEP 6 1974

GUIDELINES FOR USE OF METHOD 9
FOR DETERMINATION OF NSPS

DRAFT COPY

I. Purpose

To establish basic procedures for visually determining the opacity of emissions for the purpose of attaining baseline opacity data for NSPS. These general guidelines are to aid the observer in ensuring representative data is collected.

II. Observer Qualification

Prior to performing work for Emission Standards and Engineering Division, the observer must meet the following qualifications:

1. He must have a current certification from a smoke school conducted by either the Environmental Protection Agency, State air pollution agency or local air pollution agency.

2. He must have attended, at one time, the classroom portion of the smoke school.

Certificates indicating the above requirements have been met, will be submitted to the Emission Measurement Branch and filed.

III. General

1. The procedures set forth in Method 9 shall be followed.
2. The data should be recorded on the attached forms. Note that observations are made and recorded at 15 second intervals. It is suggested a digital clock be used for timekeeping. Use of a stopwatch is helpful for exactly timing short periods of peak emissions such as during rapping, soot-blowing, bag cleaning, etc.
3. Each observer should monitor only one emission source at a time.

DRAFT COPY

RECORD OF VISIBLE EMISSIONS

Company Name _____ Date _____

Plant Address _____ Observer _____

Stack Location _____ Observer's Location _____

Weather Conditions _____

		TIME				COMMENTS
HR	MIN	SECONDS				
		00	15	30	45	
	00					
	01					
	02					
	03					
	04					
	05					
	06					
	07					
	08					
	09					
	10					
	11					
	12					
	13					
	14					
	15					
	16					
	17					
	18					
	19					
	20					
	21					
	22					
	23					
	24					
	25					
	26					
	27					
	28					
	29					

RECORD OF VISIBLE EMISSIONS

Company Name _____

Date _____

Plant Address _____

Observer _____

Stack Location _____

Observer's Location _____

Weather Conditions _____

		TIME				COMMENTS
HR	MIN	SECONDS				
		00	15	30	45	
-----	30					
	31					
	32					
	33					
	34					
	35					
	36					
	37					
	38					
	39					
	40					
	41					
	42					
	43					
	44					
	45					
	46					
	47					
	48					
	49					
	50					
	51					
	52					
	53					
	54					
	55					
	56					
	57					
	58					
	59					

SUMMARY RECORD OF VISIBLE EMISSIONS

Type of Plant _____ Date _____

Company Name _____ Hours of Observation _____

Plant Address _____ Observer _____

Type of Discharge STACK OTHER _____

Discharge Location _____

Height of Point of Discharge _____

Observer's Location:

Distance to Discharge Point _____

Height of Observation Point _____

Direction from Discharge Point _____

Background Description _____

Weather: Clear Overcast Partly Cloudy Other _____ Color _____

Wind Direction _____ Wind Velocity _____ mi/hr

Plume Description:

Detached: Yes No

Color: Black White Other _____

Plume Dispersion Behavior: Looping Coning Fanning

Lofting Fumigating Other _____

Estimated Distance Plume Visible _____

Summary of Observations:

Opacity	Aggregate Time @ Opacity		Opacity	Aggregate Time @ Opacity	
	min.	sec.			
0			55		
5			60		
10			65		
15			70		
20			75		
25			80		
30			85		
35			90		
40			95		
45			100		
50					

NAME _____ HOURS OF OBSERVATION _____

DATE _____ OBSERVER _____

NUMBER _____ OBSERVER CERTIFICATION DATE _____

_____ OBSERVER AFFILIATION _____

FACILITY _____ Point of Emissions _____

PL DEVICE _____ Height of Discharge Point _____

Record the following information prior to and upon completion of observations at each source; if observations are made over an extended period of time, additional recordings should be made as applicable.

Final

Initial

TIME _____

LOCATION: _____

Distance To Discharge _____

Direction From Discharge _____

Point of Observation Point _____

WIND DESCRIPTION: _____

WIND CONDITIONS: _____

Direction _____

Wind Speed _____

Air Temperature _____

RELATIVITY: _____

Bar, overcast, % cloud) _____

DESCRIPTION: _____

Distance Visible _____

DATA: _____

Notes of Compliance _____

APPENDIX D
FIELD DATA SHEETS FOR "B" GRANULATOR TESTING

INCLUDES:

Field Data Sheets for:

- D-1 Scrubber Inlet Location (TP-1)
- D-2 Scrubber Outlet Location (TP-2)

APPENDIX D-1

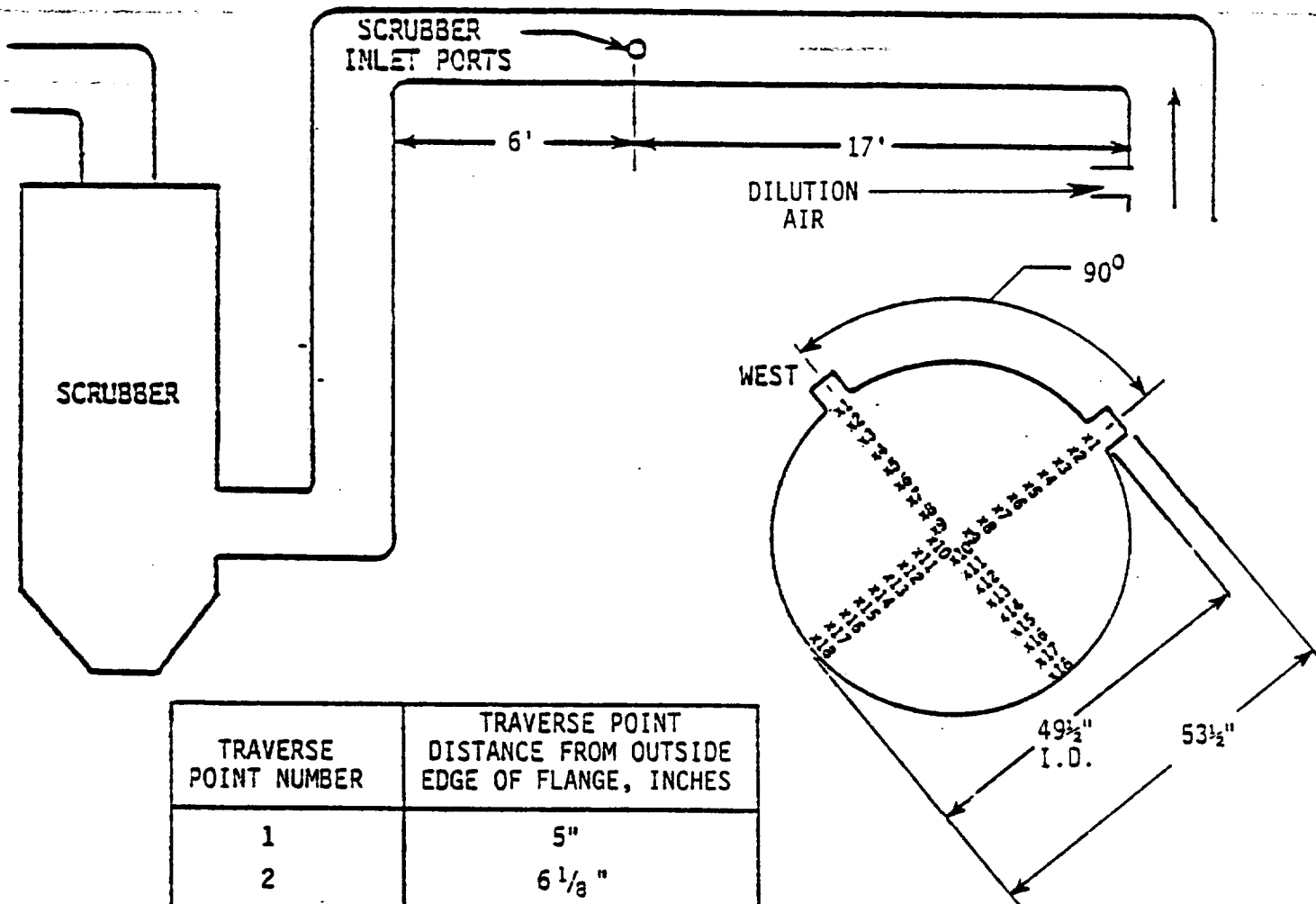
SCRUBBER INLET LOCATION (TP-1)
FIELD DATA SHEETS

TRAVERSE POINT LOCATION FOR CIRCULAR DUCTS

PLANT CF Industries Inc., Donaldsonville, Louisiana
 DATE 11/17/79
 SAMPLING LOCATION Granulator "B" scrubber inlet
 INSIDE OF FAR WALL TO
 OUTSIDE OF NIPPLE, (DISTANCE A) 53 1/2"
 INSIDE OF NEAR WALL TO
 OUTSIDE OF NIPPLE, (DISTANCE B) 4"
 STACK I.D., (DISTANCE A - DISTANCE B) 49 1/2"
 NEAREST UPSTREAM DISTURBANCE 17'
 NEAREST DOWNSTREAM DISTURBANCE 6'
 CALCULATOR J. KIRZEC, TRC

SCHEMATIC OF SAMPLING LOCATION

TRAVERSE POINT NUMBER	FRACTION OF STACK I.D.	STACK I.D.	PRODUCT OF COLUMNS 2 AND 3 (TO NEAREST 1/8 INCH)	DISTANCE B	TRAVERSE POINT LOCATION FROM OUTSIDE OF NIPPLE (SUM OF COLUMNS 4 & 5)
1	0.014	49 1/2"	1"	4"	5"
2	0.044		2 1/8"		6 1/8"
3	0.075		3 3/4"		7 3/4"
4	0.109		5 3/8"		9 3/8"
5	0.146		7 1/4"		11 1/4"
6	0.188		9 1/4"		13 1/4"
7	0.236		11 5/8"		15 5/8"
8	0.296		14 5/8"		18 5/8"
9	0.382		18 7/8"		22 7/8"
10	0.618		30 5/8"		34 5/8"
11	0.704		34 7/8"		38 7/8"
12	0.764		37 7/8"		41 7/8"
13	0.812		40 1/4"		44 1/4"
14	0.854		42 1/4"		46 1/4"
15	0.891		44 1/8"		48 1/8"
16	0.925		45 3/4"		49 3/4"
17	0.956		47 3/8"		51 3/8"
18	0.986	↓	48 1/2"	↓	52 1/2"



TRAVERSE POINT NUMBER	TRAVERSE POINT DISTANCE FROM OUTSIDE EDGE OF FLANGE, INCHES
1	5"
2	6 ¹ / ₈ "
3	7 ³ / ₄ "
4	9 ² / ₃ "
5	11 ¹ / ₄ "
6	13 ¹ / ₄ "
7	15 ⁵ / ₈ "
8	18 ⁵ / ₈ "
9	22 ⁷ / ₈ "
10	34 ⁵ / ₈ "
11	38 ⁷ / ₈ "
12	41 ⁷ / ₈ "
13	44 ¹ / ₄ "
14	46 ¹ / ₄ "
15	48 ¹ / ₈ "
16	49 ³ / ₄ "
17	51 ² / ₃ "
18	52 ¹ / ₂ "

LOCATIONS OF "B" GRANULATOR SCRUBBER INLET
TEST PORTS AND POINTS AT CF INDUSTRIES INC.,
DONALDSONVILLE, LOUISIANA

NOMOGRAPH DATA

PLANT CF Industries Inc, Donaldsonville, Louisiana

DATE 1/17/79

SAMPLING LOCATION Granulator "B" scrubber inlet

CALIBRATED PRESSURE DIFFERENTIAL ACROSS ORIFICE, in. H ₂ O	ΔH_0	1.88
AVERAGE METER TEMPERATURE (AMBIENT + 20°F), °F	$T_{m\text{ avg.}}$	~70°F
PERCENT MOISTURE IN GAS STREAM BY VOLUME	B_{wo}	~4%
BAROMETRIC PRESSURE AT METER, in. Hg	P_m	30.39
STATIC PRESSURE IN STACK, in. Hg ($P_m \pm 0.073 \times \text{STACK GAUGE PRESSURE in in. H}_2\text{O}$)	P_s	30.21
RATIO OF STATIC PRESSURE TO METER PRESSURE	P_s/P_m	0.994
AVERAGE STACK TEMPERATURE, °F	$T_{s\text{ avg.}}$	~195
AVERAGE VELOCITY HEAD, in. H ₂ O	$\Delta p_{\text{ avg.}}$	~1.15
MAXIMUM VELOCITY HEAD, in. H ₂ O	$\Delta p_{\text{ max.}}$	~1.30
C FACTOR		0.97
CALCULATED NOZZLE DIAMETER, in.		0.21"
ACTUAL NOZZLE DIAMETER, in.		0.1876"
REFERENCE Δp , in. H ₂ O		1.80

VELOCITY TRAVERSE DATA FORM

Client CF Industries Stack Ident. No. scrub. inlet Stack I.D. 4 Ft. 1 1/2
 Location Donaldsonville LA Pitot Ident. No. 44 Stack O.D. _____ Ft. _____
 Date 11/16/79 Test by JEK Pitot Cal. No., F_s _____
 Barometric Pressure, P_B 30.30 "Hg

Run 1

Run 1 *after flow*

Port	Point	VP "H ₂ O	SP "H ₂ O	T _F °F	T _R °F	$\sqrt{VP \cdot T_R}$	Port	Point	VP "H ₂ O	SP "H ₂ O	T _F °F	T _R °F	$\sqrt{VP \cdot T_R}$
L	1	0.78		70			R	1	0.47		164		—
	2	0.95		160				2	0.58		164		—
	3	1.05		166				3	0.64		168		0
	4	1.30		171				4	0.84		172		5
	5	1.40		173				5	1.05		174		15
	6	1.30		174				6	1.10		174		2
	7	1.35		175				7	1.30		175		0
	8	1.45		175				8	1.40		176		0
	9	1.55		175				9	1.50		176		0
	10	1.60		175				10	1.60		176		0
	11	1.65		175				11	1.70	-2.8	176		0
	12	1.70		175				12	1.75		176		0
	13	1.70		175				13	1.70		176		0
	14	1.70		176				14	1.70		176		5
	15	1.65		175				15	1.45		175		5
	16	1.60		174				16	1.40		174		0
	17	1.55		175				17	1.40		176		0
	18	1.50		176				18	1.40		176		0
Total							Total						
Average							Average						
							Overall Average						

Absolute Stack Pressure, P_s

$$P_s = P_B + SP/13.6 = \underline{\hspace{2cm}} + \underline{\hspace{2cm}} = \underline{\hspace{2cm}}$$

* unmeasurable
due to embair
leaking in.

Velocity, V_s

$$V_s = 2.90 \cdot F_s \cdot \sqrt{VP \cdot T_R} \cdot \sqrt{\frac{29.92}{P_s} \cdot \frac{29.84}{M_s}}$$

M_s = molecular weight of
stack gas (wet basis)

$$V_s = \underline{\hspace{2cm}} \text{ FPS}$$

Volume, standard conditions T_{std} = _____ °R

$$Q = V_s \cdot A \cdot 60 \cdot T_{std}/T_R \cdot P_s/29.92 = \underline{\hspace{2cm}} \cdot \underline{\hspace{2cm}} \cdot 60 \cdot \underline{\hspace{2cm}}$$

SCFM

D-6

Firm Name CR Ind. Plant Location Donsdaleville LA Test No. 1 Sampling Location before smelter TRC Project No. 82988-20 Date 11/17/79 Tester DEK-PAY

Pump No. 112 FILED ON TA Sheet No. 5-2 Assumed Moisture 4 Test Duration 108 Test Start Time 12 Noon Probe Heater Setting 170°F Filter Temp. Setting NA Dry Gas Meter Y 1.00

Orifice No. D-6 Orifice Dia. 0.876 Pitot Coefficient 0.820 (reg) min. Orifice All 1.88 min. Test Start Time 12 Noon Probe Ident. No. 44 Test End Time 2:23 Leak Test Start 0.01 CFME "lig 15

Ambient Temp 70 °F Traverse Point Interval 3 Test Temp. Setting NA Leak Test Final 0.002 CFME "lig 5

Duct Area (49.5" dia) 13.36 ft²

Probe Wash 1-PW-I

Silica Gel 1-S-I

Silica Gel Cond _____

Cleaning Patch N/A

H₂O Collected _____

H₂O Condition _____

ORSAT Measurements

Time	CO ₂	O ₂	CO	N ₂
1				
2				
3				

Great Bag Sample Triplicate Analysis

CO ₂	O ₂	CO	N ₂

12:10 stop to
 ck robots
 1st ppd
 returned 12:10

Firm Name CF Ind Pump No. 12 FIELD DATA SHEETS Nozzle No. & Dia. 5-2 (0.1876) In. C Factor 0.97 Monograph
 Plant Location Danville Orifice No. D-6 Assumed Moisture 4 % Pitot Coefficient 0.820 (rev)
 Test No. 1 Ambient Temp 70 °F Test Duration 108 min. Orifice A_A 1.88
 Sampling Location Substation Bar Press 30.39 "Hg Traverse Point Interval 3 min. Test Start Time 12 Noon
 TRC Project No. 87A88-2a Probe Ident. No. 44 Probe Heater Setting 170°F Temp Test End Time 2:23 PM
 Date 11/7/79 Filter Ident. No. NA Filter Temp. Setting NA Leak Test Start 0.01 CFHE 15 "Hg
 Tester DEF DAK Duct Area (49.5" DIA) 13.36 ft² Dry Gas Meter Y 1.00 "Hg Leak Test Final 0.008 CFHE 5 "Hg

80 ft air
 used to
 imply probe

2 of 3

Port	Point	Time Min	Velocity AP in H ₂ O	Alt in H ₂ O	T _M in °F	T _M Out °F	P _{STACK} in H ₂ O	T _{STACK} °F	Initial Meter Volume cu. ft.	P R E	Cyc. Angle °	Heater Box Temp.	Temp. of Gas Leaving Condenser °F	Temp vac. in Hg	Probe Temp. °F	O ₂ Conc. %
R	17	30	115	118	72	74	-230	197	14837	1		NA	60	3	172	
	18	30	110	112	72	74	-230	196	15015	1			60	3	171	
									15192	1						
L	1	30	052	053	72	73	-270	153	15192	1		NA	60	2	168	
	2	100	100	100	71	73		153	15336	1			60	3	169	
	3	115	115	118	71	73		180	15504	1				3	169	
	4	120	120	122	71	73		195	15677	1				3	169	
	5	120	120	122	71	72		196	15858	1				3	168	
	6	120	120	122	71	72		196	16040	1				3	168	
	7	120	120	122	71	72		197	16222	1				3 1/2	167	
	8	125	125	128	71	73		196	16404	1				3 1/2	170	
	9	125	125	128	71	72		196	16591	1				3 1/2	168	
	10	110	110	112	71	73		196	16780	1				3	169	
	11	115	115	118	71	73		197	16955	1				3	169	
	12	110	110	112	71	73		196	17132	1				3	169	

Orsat Bag Sample Triplicate Analysis

Time	CO ₂	O ₂	CO	N ₂
1				
2				
3				

ORSAT Measurements

Time	CO ₂	O ₂	CO	N ₂
1				
2				
3				

Cleaning Patch N/A

Probe Wash 1-PW-I

Silica Gel 1-S-I

Silica Gel Condition

FIELD DATA SHEETS

Firm Name CF 2nd Pump No. 12 Nozzle No. & Dia. 5.2 0.1876

Monograph

097

Homograph
in: C Factor

SIGNALS AND DATA

Pump No. 2

Firm Name **CF Ind**

Plant Location Dona Allenville LA
 Office No. D-6
 Ambient Temp 70 °F
 Bar Press 30.39 "Hg
 Sampling Location Sinabonville
 Project No. 8988-20
 Date 11/17/79
 Assumed Moisture 4 % Pitot Coefficient 0.820 (rev)
 Test Duration 168 min. Office Alt 188
 Traverse Point Interval 3 min. Test Start Time 12 Noon
 Probe Heater Setting 170°F Probe Test End Time 2:23
 Filter Temp. Setting NA Leak Test Start 0.01 CFM @ 15 "Hg
 Dry Gas Meter Y 1.00 Leak Test Final 0.008 CFM @ 5 "Hg
 Duct Area (49.5" dia) 13.36 ft²
 Tester DEK DAE

3-f3

Original War Sample Triplicate Analysis

[illegible]

ORSAT Measurements

ONCART requirements				
Time	CO ₂	O ₂	CO	N ₂
1				
2				
3				

VIA

1-PW-T

11.0 Collected

1-S-I

H₂O Condition

Stillen Gel Cond.

Firm Name	CF Ind	Pump No.	12	FIELD DATA SHEETS	Nozzle No. & Dia.	5-2 (0.1876)	in. C Factor	0.97	Monograph
Plant Location	Donaldsonville LA	Office No.	D-6	Assumed Moisture	4	Pitot Coefficient	0.872 (rev)		
Test No.	2	Ambient Temp	70	Test Duration	108	min. Orifice ΔH_A	188		
Sampling Location	Senubambula	Bar Press	30.39	Traverse Point Interval	3	min. Test Start Time	3:48 PM	0.71 ft ³ /an	
THC Project No.	82988-70	Probe Ident. No.	4437	Probe Heater Setting	1800 1800	Test End Time	5:56 PM	westb	
Date	11/29	Filter Ident. No.	NA	Filter Temp. Setting	NA	Leak Test Start	0:01 CME 15	imply	
Tester	DEF DAF	Duct Area	(49.5" DIA) 13.36	Dry Gas Meter Y	1.00	Leak Test Final	0:00 CME 5	probe	

Orsat Eng Sample Triplicate Analysis			
CO ₂	O ₂	CO	N ₂

ONSAT Measurements				
Time	CO ₂	O ₂	CO	N ₂
1				
2				
3				

Pre-Rinse Wash	2-PW-I	Cleaning Patch	N/A
Silica Gel	2-B-I	H ₂ O Collected	
Silica Gel Cond.		H ₂ O Cond.	

Firm Name CF Ind Pump No. 12 FIELD DATA SHEETS Hozzle No. & Dia. 5-2 (0.1876) In. C Factor 0.97
 Plant Location Donaldsonville, LA Office No. D-6 Assumed Moisture 4 Pitot Coefficient 0.833 (rev)
 Test No. 2 Ambient Temp 70 °F Test Duration 108 min. Office Alt. 1.88 0.71 ft in
 Sampling Location Grubbs rd Bar Press 30.39 "Hg Traverse Point Interval 3 min. Test Start Time 3:48 PM
 TRC Project No. 828870 Probe Ident. No. 4437 Probe Heater Setting 170°F (1/2A) Probe-Test End Time 5:56 PM
 Date 11/17/74 Filter Ident. No. NA Filter Temp. Setting NA Leak Test Start 0.01 CPM 15 "Hg
 Tester AEK-DAC Duct Area (4.95' dia) 13.36 ft² Dry Gas Meter Y 1.00 Leak Test Final 0.003 CPM 5 "Hg

Port Point	Time Min	Velocity AP in H ₂ O	Alt in H ₂ O	T _H in °F	T _H Out °F	P _{STACK} in H ₂ O	T _{STACK} °F	Initial Meter Volume cu. ft.	Cyc. Angle	Heater Box Temp.	Temp. of Gas Leaving Condenser °F	Imp. vac. in Hg	Probe Temp. °F	O ₂ Conc. %
17	3:00	0.76	0.78	70	71	-2.70	170	21.230	3	NA	60	2	170	
18	3:00	0.86	0.78	70	71		130	21.379	3			2	170	
1	3:00	1.12	1.12	69	70	-2.70	189	21.533	3	NA	60	3	170	
2	3:00	1.15	1.18	69	70		196	21.786	3			3		
3	3:00	1.20	1.22	69	70		196	21.966	3			3		
4	3:00	1.20	1.22	69	70		196	22.150	3			3		
5	3:00	1.15	1.18	69	70		196	22.332	3			3		
6	3:00	1.10	1.12	69	70		196	22.507	3			3		
7	3:00	1.10	1.12	68	69		196	22.687	3			3		
8	3:00	1.25	1.28	68	69		196	22.867	3			3		
9	3:00	1.25	1.29	67	69		197	23.042	3			3		
10	3:00	1.25	1.29	67	69		196	23.220	3			3		
11	3:00	1.25	1.29	67	69		196	23.420	3			3		
12	3:00	1.25	1.29	67	69		197	23.601	3			3		

Probe Wash 2-PW-I Cleaning Patch N/A
 Silica Gel 2-S-I H₂O Collected
 Silica Gel Cond. H₂O Condition

ORSAT Measurements
 Time CO₂ O₂ CO N₂
 1
 2
 3

Orsat Bag Sample Triplicate Analysis
 CO₂ O₂ CO N₂
 1
 2
 3

20% R-1 po
 See bag tag

FIELD DATA SHEETS
 Firm Name CFR Pump No. 12 Nozzle No. & Dia. 5-2 (0.1876) In. C Factor 0.97
 Plant Location Donaldsonville LA Office No. D-6 Assumed Moisture 4 % Pilot Coefficient 0.823 (rev)
 Test No. 3 Ambient Temp 63 °F Test Duration 108 min. Orifice All. 158
 Sampling Location South of the road "Hg Traverse Point Interval 3 min. Test Start Time 9:47 AM
 TRC Project No. 82988-20 Probe Ident. No. 37 Probe Heater Setting 1.5 amps Test End Time 12:10 PM
 Date 1/18/79 Filter Ident. No. NA Filter Temp. Setting NA Leak Test Start 0.01 CFM 18 "Hg
 Tester DEK DAK Duct Area (49 1/2" dia) ft² Dry Gas Meter Y 1.00 Leak Test Final 0.001 CFM 5 "Hg

1 of 3

Port	Point	Time	Velocity	ΔP	in H ₂ O	ΔH	in H ₂ O	T _H	in °F	T _H	Out °F	P _{STACK}	in H ₂ O	T _{STACK}	°F	Initial	Water Volume	cu. ft.	50	52	53	Heater	Box	Temp.	Temp.	of Gas	Leaving	Condenser	Pump	Var.	in Hg	Probe	Temp.	O ₂	Conc.	%
R	1	30	130	0	130	130	60	61	61	-2	30	182	182	248	50	3	50	3	50	NA	50	3 1/2	170													
	2	30	120	0	120	124	61	61	61			190	190	250	30	3						3 1/2														
	3	30	120	0	120	124	61	62	62			190	190	252	10	3						3 1/2														
	4	30	120	0	120	113	61	62	62			190	190	254	12	3						3 1/2														
	5	30	110	0	110	124	62	62	62			191	191	255	9	3						3 1/2														
	6	30	120	0	120	124	62	62	62			191	191	257	7	3						3 1/2														
	7	30	120	0	120	109	62	63	63			191	191	259	5	3						3 1/2														
	8	30	105	0	105	113	62	63	63			191	191	261	4	3						3														
	9	30	110	0	110	102	63	63	63			191	191	263	1	3						3														
	10	30	100	0	100	130	63	63	63			184	184	264	9	3						3														
	11	30	125	0	125	137	63	64	64			195	195	266	5	3						3 1/2														
	12	30	130	0	130	124	64	64	64			188	188	268	4	3						3 1/2														
	13	30	120	0	120	124	64	65	65			186	186	270	4	3						3 1/2														
	14	30	120	0	120	113	64	65	65			176	176	272	3	3						3 1/2														
	15	30	110	0	110	102	64	65	65			164	164	274	2	3						3 1/2														
	16	30	100	0	100	102	64	65	65			164	164	275	9	3						3 1/2														

Orsat Bag Sample Triplicate Analysis

Time	CO ₂	O ₂	CO	N ₂
1				
2				
3				

Orsat Measurements

Time	CO ₂	O ₂	CO	N ₂
1				
2				
3				

Cleaning Patch N/A

Probe Wash 3-PW-I

Silica Gel 3-S-I

Silica Gel Cond.

Firm Name CF Indr Pump No. 12 FIELD DATA SHEETS Hozzle No. & Dia. 5-2 (01876) in. C Factor 0.97
 Plant Location Donaldsonville LA Office No. D-6 Assumed Moisture 4 Pitot Coefficient 0.823 (ver)
 Test No. 3 Ambient Temp 63 Test Duration 108 min. Orifice ΔH_A 1.88
 Sampling Location Sandstone let Bar Press 30.27 Traverse Point Interval 3 min. Test Start Time 9:47 AM
 TRC Project No. 82988-20 Probe Ident. No. 37 Probe Heater Setting 1.5 amps Test End Time 12:10 PM
 Date 11/8/79 Filter Ident. No. NA Filter Temp. Setting NA Leak Test Start 0.01 CFHE 18 "Hg
 Tester DEK DAE Duct Area (49 1/2 in²) ft² Dry Gas Meter Y 1.00 Leak Test Final 0.001 CFHE 5 "Hg

Potentiometer
 returned to
 workshop
 probe.

2 of 3

Port	Point	Time Min	Velocity ΔP in H ₂ O	ΔH in H ₂ O	T _M in °F	T _M Out °F	P _{STACK} in H ₂ O	T _{STACK} °F	Initial Meter Volume cu. ft.	P R Angle °	Cyc. Angle °	Heater Box Temp.	Temp. of Gas Leaving Condenser °F	Pump Vac. in Hg	Probe Temp. °F	O ₂ Conc. %
A	17	30	0.90	0.93	65	66	-2.50	164	277.93	3	50	NA	50	3	170	
	18	30	0.90	0.93	65	66	-2.50	130	279.55	3				5		
	1	30	1.10	1.13	66	68	-2.50	170	281.14	3				3		
	2	30	1.10	1.13	66	68	-2.50	184	281.14	3				3		
	3	30	1.15	1.20	67	68	-2.50	189	283.10	3				3		
	4	30	1.10	1.13	67	68	-2.50	189	284.90	3				3		
	5	30	1.10	1.13	67	68	-2.50	189	286.70	3				3		
	6	30	1.20	1.24	67	68	-2.50	189	288.49	3				3		
	7	30	1.20	1.24	67	68	-2.50	190	290.28	3				3		
	8	30	1.20	1.24	67	68	-2.50	191	292.13	3				3 1/2		
	9	30	1.20	1.24	67	68	-2.50	191	294.00	3				3 1/2		
	10	30	1.20	1.24	67	68	-2.50	191	295.84	3				3 1/2		
	11	30	1.15	1.20	67	68	-2.50	191	297.70	3				3 1/2		
	12	30	1.15	1.20	68	68	-2.50	190	299.63	3				3 1/2		
		30	1.15	1.20	68	68	-2.50	190	301.33	3				3 1/2		

Probe Wash 3-PW-I Cleaning Patch NA
 Silica Gel 3-S-I H₂O Collected
 Silica Gel Cond. H₂O Condition

OHSAT Measurements
 Time CO₂ O₂ O₁ N₂
 1
 2
 3

Orsat Bag Sample Triplicate Analysis
 CO₂ O₂ CO N₂

APPENDIX D-2

**SCRUBER OUTLET LOCATION TP-2
FIELD DATA SHEETS**

TRAVERSE POINT LOCATION FOR CIRCULAR DUCTS

PLANT CF Industries Inc., Donaldsonville, Louisiana

DATE 11/17/79

SAMPLING LOCATION Granulator "B" scrubber outlet

INSIDE OF FAR WALL TO

OUTSIDE OF NIPPLE. (DISTANCE A) 62"

INSIDE OF NEAR WALL TO

OUTSIDE OF NIPPLE. (DISTANCE B) 2"

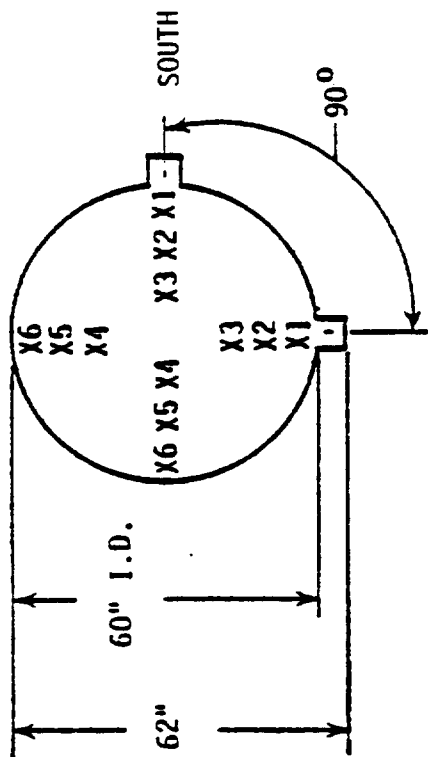
STACK I.D., (DISTANCE A - DISTANCE B) 60"

NEAREST UPSTREAM DISTURBANCE 40'

NEAREST DOWNSTREAM DISTURBANCE 10'

CALCULATOR S. Richardson

SCHEMATIC OF SAMPLING LOCATION



TRAVERSE POINT NO.	TRAVERSE POINT DISTANCE FROM OUTSIDE EDGE OF NIPPLE (INCHES)
1	4 ⁵ / ₈ "
2	10 ³ / ₄ "
3	19 ³ / ₄ "
4	44 ¹ / ₄ "
5	53 ¹ / ₄ "
6	59 ³ / ₈ "

LOCATIONS OF "B" GRANULATOR SCRUBBER OUTLET
TEST PORTS AND POINTS AT CF INDUSTRIES, INC.,
DONALDSONVILLE, LOUISIANA

NOMOGRAPH DATA

PLANT CF Industries Inc., Donaldsonville, Louisiana

DATE 11/17/79

SAMPLING LOCATION Granulator "B" scrubber outlet

CALIBRATED PRESSURE DIFFERENTIAL ACROSS ORIFICE, in. H ₂ O	ΔH_0	1.68
AVERAGE METER TEMPERATURE (AMBIENT + 20 °F), °F	$T_{m\text{ avg.}}$	~ 70 °F
PERCENT MOISTURE IN GAS STREAM BY VOLUME	B_{wo}	~ 4%
BAROMETRIC PRESSURE AT METER, in. Hg	P_m	30.4
STATIC PRESSURE IN STACK, in. Hg ($P_m \pm 0.073 \times \text{STACK GAUGE PRESSURE in in. H}_2\text{O}$)	P_s	30.37
RATIO OF STATIC PRESSURE TO METER PRESSURE	P_s/P_m	0.999
AVERAGE STACK TEMPERATURE, °F	$T_{s\text{ avg.}}$	~ 100 °F
AVERAGE VELOCITY HEAD, in. H ₂ O	$\Delta p_{\text{ avg.}}$	~ 0.57
MAXIMUM VELOCITY HEAD, in. H ₂ O	$\Delta p_{\text{ max.}}$	~ 0.69
C FACTOR		0.88
CALCULATED NOZZLE DIAMETER, in.		0.246
ACTUAL NOZZLE DIAMETER, in.		0.2575
REFERENCE Δp , in. H ₂ O		0.40

Velocity Check

FIELD DATA SHEETS

Homograph
In. G Factor

Pump No. _____

Plant Location Donaldsonville

Orifice No. _____

Assumed Moisture _____

Test No. V

Ambient Temp _____

min. Orifice ΔH_A _____

Sampling Location Scrubber Out

Bar Press _____

min. Test Start Time _____

Probe Heater Setting _____

Filter Temp. Setting _____

Leak Test Start _____

Leak Test Final _____

Leak Test Initial _____

Leak Test End Time _____

Leak Test Start _____

Leak Test Final _____

Leak Test Initial _____

Leak Test End Time _____

Leak Test Start _____

Leak Test Final _____

Leak Test Initial _____

Leak Test End Time _____

Leak Test Start _____

Leak Test Final _____

Port	Point	Time Min	Velocity AP in H ₂ O	ΔH in H ₂ O	T _H in °F	T _H Out °F	P _{STACK} in H ₂ O	T _{STACK} °F	Initial Meter Volume cu. ft.	P R Q R	Cyc. Angle °	Heater Box Temp.	Temp. of Gas Leaving Condenser °F	Imp. vac. in. Hg	Probe Temp. °F	O ₂ Conc. %
		3														
		4														
		5														
		6														
		7														
		8														
		9														
		10														
		11														
		12														
		13														
		14														
		15														
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		42														
		43														
		44														
		45														
		46														
		47														
		48														
		49														
		50														
		51														
		52														
		53														

Probe Weight _____

Silica Gel _____

Silica Gel Cond. _____

Cleaning Patch _____

H₂O Collected _____

H₂O Condition _____

URSAT Measurements

Time	CO ₂	O ₂	CH ₄	N ₂
1				
2				
3				

Orsat Bag Sample Triplicate Analysis

CO ₂	O ₂	CH ₄	N ₂

[illegible][illegible]

STANDARD INCREASING CEMENTS				
Time	CO ₂	O ₂	C ₂	N ₂
1				
2				
3				

11.2.0 Conclusion

Sullen (el) Cond.

FIELD DATA SHEETS
 Pump No. 11 Nozzle No. & Dia. 2575 4-2 In. C Factor .88
 Plant Location Donaldsonville Assumed Moisture 4 Pitot Coefficient .834
 Test No. 1 Ambient Temp 88 °F Test Duration 120 min. Office AH 168
 Sampling Location Scrubber Out Bar Press 30.39 "Hg Traverse Point Interval 10 min. Test Start Time 1201
 TRC Project No. 8295 Probe Ident. No. 38 Probe Heater Setting 120 °F Amps Test End Time 1416
 Date 11/17/78 Filter Ident. No. N/A Filter Temp. Setting N/A Leak Test Start 0.002 CFME "Hg
 Tester SRR DR Duct Area 19.635 ft² Dry Gas Meter Y 5 "Hg
 Leak Test Final 0.000 CFME "Hg

Port	Point	Time Min	Velocity ΔP in H ₂ O	AH in H ₂ O	T _H in °F	T _H Out °F	P _{STACK} in H ₂ O	T _{STACK} °F	Initial Meter Volume cu. ft.	P R E	Cyc. Angle °	Heater Box Temp.	Temp. of Gas Leaving Condenser °F	Pump Vac. in Hg	Probe Temp. °F	Scrubber Temp. °F
2	1	50	550	2100	72	72		98	9923	2		N/A	58	3.8	123	20.5
1	1	50	650	2250	71	71		98	10317	2			58	3.5	123	20.7
2	2	50	610	2380	71	71		98	10260	2			68	3.7	123	20.9
2	2	50	630	2380	71	71		98	11180	2			60	3.9	123	20.9
3	3	50	690	2620	71	72		98	11630	2			60	4.5	118	20.7
3	3	50	670	2550	71	72	-35	98	12071	2			55	4.5	123	20.7
4	4	50	620	2320	71	72		101	12545	2			55	3.9	123	20.7
4	4	50	620	2320	71	72		103	13040	2			55	3.2	122	20.7
5	5	50	550	2080	71	72		102	13420	2			55	3.2	122	20.5
5	5	50	550	2080	71	72		101	13839	2			55	2.0	122	20.5
6	6	50	400	1510	71	72		99	14254	2			55	2.0	122	20.5
6	6	50	400	1510	71	72		99	14620	2			55	2.0	122	20.5
		6754		2164	71.5	71.5	-0.35	100.5	14995	2			55			

ORSAT Measurements

Time	CO ₂	O ₂	CO	N ₂
1				
2				
3				

Orsat Bag Sample Triplicate Analysis

	CO ₂	O ₂	CO	N ₂

Probe Wash 18V-0 Cleaning Patch N/A
 Silica Gel 15-0 H₂O Collected
 Silica Gel Cond. 8 P H₂O Cond.

7-0-2

D-24

Cleaning Patch N/A

H₂O Collected _____

H₂O Condition _____

Silica Gel 2-50

Silica Gel Cond. _____

Probe Wash 2-PW-O

ORSAT Measurements				
Time	CO ₂	O ₂	Cl	N ₂
1				
2				
3				

Greatest Bag Sample Triplicate Analysis

CO ₂	O ₂	CO	N ₂

10 2012

Firm Name CF Pump No. 11 FIELD DATA SHEETS Nozzle No. & Dia. 2575 4-2 In. C Factor 86
Plant Location Donaldsonville Office No. D-5 Assumed Moisture 5 % Pitot Coefficient 0.834
Test No. 3 Ambient Temp 58 °F Test Duration 120 min. Orifice ΔH 1.68
Sampling Location Scrubber Out Bar Press _____ min. Test Start Time 09046
TRC Project No. 82988-20 Probe Ident. No. 38 Traverse Point Interval 10 Probe Heater Setting 120° F amps Test End Time 1217
Date 1/18/79 Filter Ident. No. 1/A Filter Temp. Setting _____ Leak Test Start 0.000 CFME 5 "Hg
Tester SFR DR Duct Area 19.633 ft² Dry Gas Meter Y _____ Leak Test Final 0.000 CFME 5 "Hg

Port	Point	3	4	5	6	7	8	9	10	All			T _H		P _{STACK}			T _{STACK}			Initial					P	50	52	53	Heater Box Temp.	Temp. of Gas leaving Condenser °F	Pump Vac. in Hg	Probe Temp. °F	Scrubber OP or Conc. %
		Time Min	Velocity AP in H ₂ O		in H ₂ O		In °F	Out °F	in H ₂ O		°F		Miter Volume cu. ft.					Cyc. Angle °																
2	1	50	50	50	50	600	600	600	2250	66	66							102	102	300	17	2	17	17	17	45	3.4	121	20.5					
	1	50	50	50	50	580	580	580	2200	66	66							101	101	304	24	2	24	24	24	46	3.3	121	20.5					
	2	50	50	50	50	640	640	640	2400	66	66							101	101	505	85	2				52	3.7	120	20.4					
	2	50	50	50	50	650	650	650	2450	66	66							101	101	312	96	2				54	3.7	120	20.6					
	3	50	50	50	50	680	680	680	2580	67	67							102	102	317	50	2				55	4.0	120	20.3					
	3	50	50	50	50	680	680	680	2580	66	66							101	101	322	07	2				55	4.0	120	20.3					
	4	50	50	50	50	600	600	600	2250	66	66							103	103	326	71	2				55	3.5	120	20.2					
	4	50	50	50	50	600	600	600	2250	67	67							105	105	331	08	2				57	3.5	120	20.1					
	5	50	50	50	50	520	520	520	1950	67	67							104	104	335	45	2				57	2.6	119	19.8					
	5	50	50	50	50	520	520	520	1950	67	67							103	103	339	54	2				57	2.6	120	20.0					
	6	50	50	50	50	420	420	420	1590	68	68							102	102	343	58	2				58	2.0	120	19.7					
	6	50	50	50	50	420	420	420	1590	68	68							102	102	347	27	2				58	2.0	120	19.5					
										6	49									35	11	2												
						075			213																									

Probe Wash 3-PW-0 Cleaning Patch 1/A
Silica Gel 3-S-0 H₂O Collected _____
Silica Gel Cap _____ H₂O Condition _____

URSAT Measurements²

Time	CO ₂	O ₂	(A)	N ₂
1				
2				
3				

Orsat Bag Sample Triplicate Analysis

CO ₂	O ₂	CO	N ₂

Firm Name C7 Ind Pump No. _____ Nozzle No. & Dia. _____ Homograph In. C Factor _____
 Plant Location Danversville, La Assumed Moisture _____ Z Pitot Coefficient _____
 Test No. MOISURE DET CORRECTION °F Test Duration 34 min. Orifice Area _____
 Sampling Location INLET Bar Press _____ °Hg Traverse Point Interval _____ min. Test Start Time 1615
 TRC Project No. 82988-20 Probe Ident. No. _____ Probe Heater Setting _____ amps Test End Time 1649
 Date 1/18/78 Filter Ident. No. _____ Filter Temp. Setting _____ Leak Test Start 0.00 CFM@ 15 "Hg
 Tester DAK Duct Area _____ ft² Dry Gas Meter Y _____ Leak Test Final 0.00 CFM@ 15 "Hg

Port	Point	3	4	5	6	7	8	9	10	12	13	14	15	18	19	20	T _H		P _{STACK}			T _{STACK}			Initial		40	41	42	43	44	45	50	52	53	Heater Box Temp.	Temp. of Gas Leaving Condenser °F	Pump Vac. in Hg	Probe Temp. °F	O ₂ Conc. %
		Time Min	Velocity AP in H ₂ O		All in H ₂ O		in °F		in H ₂ O		°F			Meter Volume cu. ft.		P	R	Q	R	Cyc. Angle °	40																			
		50								25				80			80	80	197						31	9	4	3												
		50								25				80			80	80	197						32	3	7	5												
		50								25				80			80	80	197						32	8	2	5												
		50								25				81			81	81	197						33	2	6	4												
		50								25				81			81	81	197						33	7	1	4												
		50								25				81			81	81	197						34	1	6	0												
		40								25				81			81	81	197						34	6	0	5												
																								34	9	7	7													

Orsat Bag Sample Triplicate Analysis

Time	CO ₂	O ₂	CO	N ₂
1				
2				
3				

Orsat Measurements

Time	CO ₂	O ₂	CO	N ₂
1				
2				
3				

Probe Wash _____ Cleaning Patch _____
 Silica Gel _____ H₂O Collected _____
 Silica Gel Cond. _____ H₂O Condition _____

APPENDIX E

FIELD DATA FOR
MISCELLANEOUS SAMPLES AND OPERATIONS

INCLUDES:

- E-1 Scrubber Water Data (Inlet-Outlet)**
- E-2 Scrubber Pressure Drops**
- E-3 Process Samples Analysis Data**
- E-4 Relative Humidity and Ambient Air
Temperature Measurements**

APPENDIX E-1
SCRUBBER WATER DATA (INLET AND OUTLET)

INCLUDES:

Collection Times, pH, Temp.
and Laboratory Analysis Results

SUMMARY OF JANUARY 17 AND 18, 1979 pH AND TEMPERATURE
MEASUREMENTS ON INDIVIDUAL SAMPLES OF SCRUBBING LIQUID
ENTERING AND EXITING "B" GRANULATOR SCRUBBER AT
CF INDUSTRIES, INC., DONALDSONVILLE, LOUISIANA

Run No.	Date	Sampling Time	Scrubber Inlet Sample		Scrubber Outlet Sample	
			pH	° F	pH	° F
1	01-17-79	1215	9.50	135	8.15	126
		1255	9.25	154	8.10	124
		1331	9.15	164	8.10	124
		1416	9.10	152	7.95	120
2	01-17-79	1550	9.7	166	8.3	129
		1620	9.5	183	8.2	128
		1655	9.2	174	8.2	129
		1725	9.2	167	8.1	128
		1800	9.0	172	8.0	129
3	01-18-79	0955	9.2	180	8.30	128
		1030	9.3	172 172	8.25	126
		1105	9.3	177	8.25	129
		1140	9.3	173	8.20	127
		1215	9.2	173	8.20	124

Project No. CFI DONALDSONVILLE

Book No. _____

TITLE Scrubber inlet/outlet temp, pH

Page No. _____

Test #	Scrubber Inlet			Scrubber Outlet			date LWC 1/17/79
	time	temp, °F	pH	time	temp, °F	pH	
1-1	1216	135	9.50	1214	126	8.15	1/17/79
1-2	1256	154	9.25	1254	124	8.10	"
1-3	1333	164	9.15	1330	124	8.10	"
1-4	1417	152	9.10	1415	120	7.95	"
1-5							
2-1	1550	166	9.7	1550	129	8.3	"
2-2	1620	183	9.5	1620	128	8.2	"
2-3	1655	174	9.2	1655	129	8.2	"
2-4	1725	167	9.2	1725	128	8.1	"
2-5	1800	172	9.0	1800	128	8.0	"
3-1	0955	180	9.2	0955	128	8.3	1/18/79
3-2	1030	172	9.3	1030	126	8.25	"
3-3	1105	177	9.3	1105	129	8.25	"
3-4	1140	173	9.3	1140	127	8.2	"
3-5	1215	173	9.2	1215	124	8.2	"

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SUMMARY OF UREA, AMMONIA AND FORMALDEHYDE MEASUREMENTS ON
THE SCRUBBING LIQUID ENTERING AND LEAVING THE
"B" GRANULATOR SCRUBBER AT CF INDUSTRIES INC.,
DONALDSONVILLE, LOUISIANA

Run Number	Run 1		Run 2		Run 3		Average	
Date	01-17-79		01-17-79		01-18-79			
Location	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Urea Concentration (mg/l) ^e								
Uncorrected ^a	114	516300	83	525100	94	538300	97	526600
Corrected ^b	122	552400	89	561900	101	576000	104	563400
Ammonia Concentration (mg/l)								
Direct Nesslerization	180	f	44	f	23	f	82	-
Distilled Uncorrected ^c	138 ⁱ	14100	52 ^j	12800	31	13100	74	13330
Distilled Corrected ^d	133	-7800	85	-9500	27	-9700	82	-
Formaldehyde Concentration (mg/l)	26	0.01	20	0.08	14	0.05	20	0.05
pH ^g	9.25	8.08	9.32	8.16	9.26	8.24	9.28	8.16
Temperature (°F) ^h	151	124	172	128	175	127	166	126
Percent Solids	1.2	3.9	0.9	4.6	3.6	4.8	1.9	4.4

^aKjeldahl analysis results uncorrected for urea conversion during distillation.

^bKjeldahl analysis results corrected for urea conversion during distillation (corrected = uncorrected x 1.07).

^cDistillation/Nesslerization (D/N) analysis results uncorrected for urea conversion during distillation.

^dD/N analysis results corrected for urea conversion (corrected = uncorrected - 0.07*corrected urea/1.765).

^eMilligrams per liter.

^fAverage spectrophotometric measurement not possible due to turbidity.

^gAverage of several individual liquid samples taken during each run.

^hAverage of individual sample temperatures taken immediately after collection.

ⁱAverage slight turbidity may have given high instrument readings. Visual estimate for this sample is 60 mg/l.

^jAverage slight turbidity may have given high instrument readings. Visual estimate for this sample is 30 mg/l.

APPENDIX E-2
SCRUBBER PRESSURE DROPS

SUMMARY OF JANUARY 17 and 18, 1979 "B" GRANULATOR
SCRUBBER PRESSURE DROP MEASUREMENTS AT CF INDUSTRIES, INC.
IN DONALDSONVILLE, LOUISIANA

<u>DATE</u>	<u>CLOCK TIME</u>	<u>ΔP, "H₂O</u>	<u>DATE</u>	<u>CLOCK TIME</u>	<u>ΔP, "H₂O</u>
1-17-79	1207	21.8	1-17-79	1644	20.2
	1212	21.6		1658	20.4
	1217	21.5		1703	20.4
	1222	20.9		1708	20.4
	1227	20.7		1713	20.2
	1232	20.7		1718	20.3
	1237	20.7		1723	20.4
	1242	21.1		1728	20.6
	1247	21.1		1733	20.6
	1252	20.7		1738	20.6
	1257	20.5		1743	20.4
	1321	20.5		1748	20.4
	1326	20.7		1753	20.2
	1331	20.9	1-18-79	0946	21.6
1-17-79	1336	20.9		0951	21.2
	1341	20.7		0956	21.0
	1346	20.7		1001	21.1
	1351	20.7		1006	21.0
	1356	20.9		1011	21.2
	1401	20.7		1016	21.0
	1406	20.5		1021	20.7
	1411	20.5		1026	20.5
	1416	20.5		1031	20.7
	1549	20.2		1036	20.6
	1554	20.2		1041	20.7
	1559	20.2		1122	20.5
	1604	20.8		1127	20.5
	1609	20.9	1-18-79	1132	20.4
1-17-79	1614	20.9		1137	20.6
	1619	20.7		1142	20.3
	1624	20.8		1147	20.3
	1629	20.9		1152	20.2
	1634	21.0		1157	20.1
	1639	21.0		1202	19.8
				1207	20.0
				1212	19.7
				1217	19.5

APPENDIX E-3

PROCESS SAMPLES ANALYSIS DATA

1. Details Specific to the Analysis of the Scrubber Liquid Samples:

a. Urea

A five ml. aliquot of the scrubber inlet and outlet water sample was digested and distilled to 250 ml. The inlet sample was analyzed in this state, while the outlet scrubber sample distillate was diluted 100 fold, and a 2 ml. aliquot of this was taken for nesslerization.

b. Ammonia

The inlet scrubber waters from Test 1 and 2 were cloudy after the addition of nessler reagent to the distilled samples. Visual estimation of color intensity against the standard solution color intensity yielded "estimated" ammonia concentrations.

The outlet scrubber waters from all three tests were very cloudy when analyzed by the direct nesslerization method, precluding either instrumental or visual determination of ammonia concentrations.

c. Formaldehyde

Dilutions of the liquid samples was not required. The samples were analyzed directly by colorimetric analysis.

The three outlet scrubber water sample concentrations were below the limit of detection for formaldehyde ($<0.05 \mu\text{g/ml.}$)

2. Analysis of the Process Samples:

A. Chemical Analysis

Two solid samples were received for analysis. Samples for analysis were prepared by weighing approximately 1 gram of sample to the nearest 0.1 mg. and then diluting it to 100.0 ml. with deionized distilled water. Aliquots of these solutions were taken for the analyses which were carried out without problems from interferences for ammonia and urea. A problem with the formaldehyde analysis is described in Appendix J.

The laboratory results were reported as the percent of each component present in the sample.

B. Bulk Density Analytical Procedure

The bulk density of the unscreened product was determined using a graduated cylinder and platform balance.

The sample was passed through a riffle and a 300-350 ml portion was obtained. The tare weight of the graduated cylinder was determined. The sample was then passed into the graduated cylinder until it overflowed the cylinder. The sample was then leveled with the top of the cylinder. The cylinder and contents were then reweighed and the bulk density was then determined as a weight of material per unit volume.

C. Sieve Analysis Analytical Procedure

The particulate size of the product was estimated by means of a sieve analysis. This analysis employed a sieve shaker, timer, balance, sample splitter and sieves (sieves 6, 8, 10, 12, 14, and 16 and a pan were used).

A small amount (about 250 grams) from each of the three unscreened product samples was weighed to the nearest 0.2 gram. The sieves were then arranged in numerical order with the smallest sieve number on top and a pan on the bottom. The sample was poured into the top sieve while tapping the stack of sieves. The stack was next vigorously shaken in a rotary horizontal motion for one minute. The sieves were then inserted in the shaker and shaken for five minutes. After shaking, the contents of each sieve and bottom pans were weighed.

The percent in each sieve was calculated as follows:

$$\% \text{ Retained} = \frac{(\text{Weight of material} \times 100)}{(\text{Total Weight})}$$

SUMMARY OF JANUARY 17, AND 18, 1979 SIEVE AND BULK
DENSITY MEASUREMENTS ON THE "B" GRANULATOR PRODUCT BEFORE SCREENING AT CF INDUSTRIES,
INC. IN DONALDSONVILLE, LOUISIANA

	Granulator Unscreened Product Sample #1 Collected @1343 on 01/17/79			Granulator Unscreened Product Sample #2 Collected @1730 on 01/17/79			Granulator Unscreened Product Sample #3 Collected @1045 on 01/18/79		
	Mass, gm	Percent of Total Mass	Cumula- tive Percent of Total Mass	Mass gm	Percent of Total Mass	Cumula- tive Percent of Total Mass	Mass, gm	Percent of Total Mass	Cumula- tive Percent of Total Mass
Total Sample To Sieves	237.36			245.26			224.37		
Sieve No. 6	28.25	11.90	100	24.44	10.03	100	16.55	7.39	100
Sieve No. 8	44.50	18.75	88.10	46.77	19.20	89.97	42.92	19.12	92.63
Sieve No. 10	58.42	24.62	69.34	60.74	24.93	70.77	56.15	25.01	73.51
Sieve No. 12	45.56	19.20	44.73	48.87	20.06	45.84	46.20	20.58	48.49
Sieve No. 14	39.91	16.82	25.52	40.43	16.59	25.78	40.58	18.08	27.91
Sieve No. 16	13.75	5.79	8.71	14.74	6.05	9.19	14.37	6.40	9.83
Bottom	6.92	2.92	2.92	7.64	3.14	3.14	7.70	3.43	3.43
Sum of Mass on Sieves	237.31			243.23			244.47		
Bulk Density, grams per 250 ml	213.11			214.64			213.53		

111

TITLE urea bulk sample analysis (sieve & bulk density)

Book No. _____

From Page No. _____

Sieve No	Granulator unscreened prod Sample #1 collected @ 1343 on 1/17/79			Granulator unscreened product Sample #2 collected @ 1730 on 1/17/79			Granulator unscreened Sample #3 collected @ 1045 on 1/18/79			C p total
	Mass, gm	percent of total mass	Cumulative percent of total mass	Mass, gm	percent of total mass	Cumulative percent of total mass	Mass, gm	percent of total mass		
Sample to sieves	237.36			245.26			224.37			
Sieve No 6	28.25	11.90	100	24.44	10.03	100	16.55	7.39		1
" 8	44.50	18.75	88.10 88.05 RWC	46.77	19.20	89.97	42.92	19.12		9
" 10	58.42	24.62	69.34	60.74	24.93	70.77	56.15	25.07		2
" 12	45.56	19.20	44.73 44.73 RWC	48.87	20.06	45.84	46.20	20.58 20.58 RWC		4
" 14	39.91	16.82	25.52	40.43	16.59	25.78	40.58	18.08		7
" 16	13.75	5.79	8.70	14.74	6.05	9.19	14.37	6.40		9.5
bottom	6.92	2.92	2.92	7.64	3.14	3.14	7.70	3.43		3.
Σ on sieves	237.36			243.63			224.47 223.97 RWC			
Bulk Density grams per 250 ml	213.11			214.64			213.53			

% H₂O of urea product = 0.24 @ 7AM, 1/17/79 ; 0.21 @ 1530, 1/17/79 ; 0.17 @ 0700
1/18/79 and 0.14 @ 1530 1/18/79 - analysis by CF Industries.

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APPENDIX E-4

**RELATIVE HUMIDITY AND AMBIENT AIR
TEMPERATURE MEASUREMENTS**

SUMMARY OF JANUARY 17, 18 AND 19, 1979 AMBIENT TEMPERATURE,
RELATIVE HUMIDITY AND BAROMETRIC PRESSURE MEASUREMENTS
FOR "B" GRANULATOR AT CF INDUSTRIES, INC. DONALDSONVILLE, LOUISIANA

Date	Sampling Time	Ambient Temperature °F	Relative Humidity %	Barometric Pressure Inches Hg
01-17-79	1230	69	76	
	1245	69	76	
	1306	69	76	30.40
	1320	69	76	30.40
	1356	69	79	30.39
	1448	70	74	30.39
	1523	69	79	30.39
	1635	67	85	30.39
	1715	68	80	30.39
	1815	66	85	30.38
01-18-79	0945	62	94	30.39
	1005	62	94	30.39
	1020	64	90	30.39
	1040	64.5	87	30.38
	1055	66	82	30.38
	1115	67.5	85	30.36
	1140	67	83	30.36
	1200	68	85	30.36
	1435	69	85	30.28
	1500	71	77	30.28
	1525	73	73	30.27
	1540	71	77	30.27
01-19-79	1000	68	90	30.17
	1020	68	90	30.17
	1040	70	86	30.17
	1100	70	86	30.17
	1120	70	86	30.14
	1140	75	70	30.12
	1200	77	66	30.10
	1230	73	75	30.10

TITLE Temperature + Relative Humidity Measurements

Project No. CFI DONALDSONVILLE

Book No. _____

From Page No. _____			Dry Bulb °F	Wet Bulb °F	R.H. %	measurement taken @ Place "H ₂ O"	Comments
Test #	Date	Time					
1	1/17	1230	69	64	76	outside inside	measurement taken
"	"	1245	69	64	76	outside inside	outside chem lab
"	"	1306	69	64	76	30.40	"
"	"	1320	69	64	76	30.40	"
"	"	1356	69	64.5	79	30.39	"
"	"	1448	70	64.5	74	30.39	"
"	"	1523	69	64	79	30.39	"
"	"	1635	67	64	85	30.39	"
"	"	1715	68	64	80	30.39	"
"	"	1815	66	63	85	30.38	"
	1/18	0945	62	61	94	30.39	"
	"	1005	62	61	94	30.39	"
	"	1020	64	62	90	30.39	"
	"	1040	64.5	62	87	30.38	"
	"	1055	66	62.5	82	30.38	"
	"	1115	67.5	64.5	85	30.36	"
	"	1140	67	63.5	83	30.36	"
	"	1200	68	64	85	30.36	"
	"	1435	69	66	85	30.28	"
	"	1500	71	66	77	30.28	"
	"	1525	73	66	73	30.27	"
	"	1540	71	65	77	30.27	"
	1/19	1000	68	66	90	30.17	"
	"	1020	68	66	90	30.17	"
	"	1040	70	67	86	30.17	"
	"	1100	70	67	86	30.17	"
	"	1120	70	67	86	30.14	"
	1/19	1140	75	68	70	30.12	"
		1200	77	69	66	30.10	"
		1230	73	67.5	75	30.10	"

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APPENDIX F
SYNTHESIS TOWER SAMPLING DATA
INCLUDES:

- F-1 Draft Method for Testing Ammonium Nitrate Facilities**
- F-2 Field Data Sheets for Synthesis Tower Sampling**
- F-3 Synthesis Tower Test Results Calculations**

APPENDIX F-1

**DRAFT METHOD FOR TESTING
AMMONIUM NITRATE FACILITIES**

DETERMINATION OF PARTICULATE (AMMONIUM NITRATE) EMISSIONS FROM AMMONIUM NITRATE PLANTS

1. Principle and Applicability

1.1 Principle. Particulate emissions are assumed to be ammonium nitrate. A portion of the emission is withdrawn isokinetically from the source using a sampling train. The ammonium nitrate is collected in the impinger water and on the final filter of the sampling train. The total weight of ammonium nitrate collected is calculated from the determination of nitrate ion concentration using a specific ion electrode method.

1.2 Applicability. This method is applicable for the determination of particulate (ammonium nitrate) emissions from neutralizers, evaporators and prilling towers in ammonium nitrate production facilities only when specified by the appropriate test procedure for determining compliance.

2. Range and Sensitivity

2.1 Range. The range of the procedure is limited only by the lower detection limit of nitrate concentration using a specific ion electrode. A typical linear measurement range is 6×10^{-6} to 1.0 M (gm moles/liter) nitrate ion. This converts to a particulate emission concentration range of about 2.5×10^{-4} to 43 grains per standard cubic foot, using a 90 percent water vapor stream for calculation basis.

2.2 Sensitivity. The sensitivity of the technique will vary depending on the particular combination of electrode and readout meter. The manufacturer specifications should be consulted when an estimate is required.

3. Interferences

The following ions concentrations are reported by one vendor to cause a 10 percent error when measuring a 10^{-3} M nitrate concentration:¹

¹ Analytical Methods Guide, Orion Research, May 1975.

10^{-7}M ClO_4^- ; 10^{-5}I^- ; 10^{-4}ClO_3^- ; $6 \times 10^{-4} \text{Br}^-$; $4 \times 10^{-3} \text{HS}^-$, CN^- ;
 $2 \times 10^{-3} \text{NO}_2^-$; $4 \times 10^{-2} \text{HCO}_3^-$, Cl^- ; 10^{-1}OAc^- .

4. Precision, Accuracy, and Stability

A statistically valid estimate has not been made for the precision of the total technique. However, using a limited amount of data collected during the initial demonstration tests resulted in a precision based on a 95 percent confidence interval of 19.7 percent. No standard reference materials are available to form a basis for the determination of accuracy. Samples collected during the demonstration tests were stored for a period of about 30 days with no apparent degradation of the nitrate ion concentration.

5. Apparatus

5.1 Sampling train. See Figure 1. Many of the design specifications of this sampling train are described in APTD-0581.

5.1.1 Nozzle--Stainless Steel (316) with a sharp, tapered leading edge.

5.1.1 Probe--Borosilicate¹ glass or stainless steel (316) with a heating system capable of maintaining a gas temperature of about 121°C (250°F) at the exit of the probe when it is desired to prevent condensation in the probe.

5.1.3 Orifice flow-meter--Stainless steel (316) constructed and calibrated such that sampling rates can be monitored and adjusted as required. Additional information regarding the construction, calibration, and use of this type meter can be obtained from: Emission Measurement Branch, Emission Standards and Engineering Division, Environmental Protection Agency, Research Triangle Park, North Carolina 27711.

5.1.4 Pitot tube--Type S, or equivalent, attached to probe to monitor stack gas velocity.

5.1.5 Impingers--Five impingers connected in series as shown in Figure 1 with ground glass vacuum tight fittings. The first, second, fourth, and fifth impingers are of the Greenburg-Smith design, modified by replacing the tip with a 1.25 cm (1/2 inch) inside diameter glass tube extending to 1.25 cm (1/2 inch) from the bottom of the flask. The third impinger is of the Greenburg-Smith design with a standard tip.

5.1.6 Filter holder--Borosilicate glass with a glass frit filter support and a silicone rubber gasket. The holder design shall provide a positive seal against leakage from the outside or around the filter.

5.1.7 Filter heating system--Capable of maintaining the filter holder at about 121°C (250°F). The filter heating system is required only when necessary to prevent condensation on the filter media.

5.1.8 Metering system--Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (±5°F), dry gas meter with 2 percent accuracy at the required sampling rate, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume.

5.1.9 Barometer--To measure atmospheric pressure to ± 2.5 mm Hg (0.1 in.).

5.2 Sample Recovery

5.2.1 Probe brush--At least as long as the probe.

5.2.2 Wash bottle--Glass or polypropylene.

5.2.3 Sample storage containers--1 liter, polypropylene, wide mouth, with Teflon cap liner.

5.3 Analysis

5.3.1 Glass or polypropylene beaker; 5000 ml.

5.3.2 Balance--Triple beam or dual pan, 1500 gram capacity to measure ± 0.5 g.

5.3.3 Nitrate ion activity sensing electrode.

5.3.4 Reference electrode--Silver/silver chloride. Saturated Calomel electrode, or equivalent, suitable for use with the nitrate electrode and electrometer used.

5.3.5 Electrometer--a pH meter with millivolt scale capable of ± 0.5 mv resolution, or a specific ion meter made especially for specific ion use.

5.3.6 Magnetic stirrer and TFE flouorocarbon coated stirring bars.

6. Reagents

6.1 Sampling

6.1.1 Filters--MSA 1108BH glass fiber, or equivalent, to fit filter holder.

6.1.2 Silica gel--Indicating type, 6-16 mesh, dried at 175°C (350°F) for two hours.

6.1.3 Water--Distilled.

6.1.4 Crushed ice.

6.2 Sample recovery

6.2.1 Water--Distilled, from same container as 6.1.3.

6.3 Analysis

6.3.1 Methyl red indicator.

6.3.2 Water--Distilled, from same container as 6.1.3.

6.3.3 Dibasic potassium phosphate (ACS reagent grade).

6.3.4 Sulfuric acid, 25 percent. Add 143 ml concentrated sulfuric acid to 857 ml distilled water. Cool.

6.3.5 Ammonium hydroxide, concentrated reagent grade.

6.3.6 Ammonium nitrate standard solution, 0.100 M--Weigh 8.005 grams of ACS reagent grade ammonium nitrate. The ammonium nitrate should be dried prior to weighing according to standard laboratory practices to eliminate the presence of water. Add to a 1000 ml volumetric flask, dissolve, and dilute to volume.

7. Procedure

7.1 Sampling

7.1.1 Select the sampling site and the minimum number of sampling points; determine the stack pressure, temperature and range of velocity head as described in Methods 1, 2, and 3 (Federal Register, Vol. 36, No. 247, Thursday, December 23, 1971).

7.1.2 Preparation of sampling train--Place glass fiber filter into filter holder. Weigh approximately 200 gm. of silica gel to the nearest gram. Place 100 ml of distilled water in each of the second and third impingers; leave the first and fourth impingers empty, and place the pre-weighed silica gel in the fifth impinger. Assemble the train as shown without the probe in Figure 1, with the filter between the fourth and fifth impinger.

Leak check the sampling train at the sampling site by plugging the inlet to the train and pulling a 380 mm Hg. (15 inch) vacuum, or the maximum vacuum that is expected to occur during the test run, whichever is greater. A leakage rate not in excess of $0.00057 \text{ m}^3/\text{min}$ ($0.02 \text{ ft}^3/\text{min}$) at a vacuum of 380 mm Hg. (15 inch) is acceptable. Attach the probe and

adjust the probe heater to provide a gas temperature of about 121°C (250°F) at the probe outlet. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 21°C (70°F) or less.

7.1.3 Train operation--For each run, record the data required on the example sheet shown in Figure 2. Take readings at each sample point at least every five minutes, and when changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Sample for the same amount of time at each traverse point. Maintain isokinetic sampling throughout. Turn off the pump at the conclusion of each run and record final readings. Remove the probe and nozzle from the stack and seal for transport.

7.2 Sample recovery. Move the impinger train to the cleanup area. If the sample analysis is to be performed in the field, transfer the train contents directly to the analysis beaker. If samples are to be transported, transfer the impinger contents to the sample storage bottles as follows:

7.2.1 Container 1--Record the container tare. A sample cleanup form is given in Figure 3. Add the contents of the first four impingers to the container if possible. Record weight either when filled to capacity or when all impinger contents have been added. Cap and seal. If additional impinger contents remain, proceed to container 2.

7.2.2 Container 2--Record the container tare. Transfer any remaining impinger contents and record weight. With distilled water, rinse all sample exposed surfaces three times. Rinse and brush the probe three times also.

Add all rinses into container. Disassemble the filter holder and rinse the front half three times and add rinses to container. Record final weight. Now add glass fiber filter to washes in container 2. Cap and seal. If rinses have not been completed before the container is filled proceed to use a third container, recording the tare and final weight.

7.2.3 Silica gel container--Transfer the silica gel from the fifth impinger to this container. Cap and seal.

7.3 Analysis. Treat the contents of each example container as described below:

7.3.1 Containers 1 and 2 (plus any other impinger water or train rinse container)--Quantitatively transfer the contents of each sample and rinse container to a larger mixing container of sufficient volume to hold the entire sample. Record the total volume of transfer rinse water in order to determine the total sample and blank correction volumes.

7.3.2 Silica gel container--Weigh the spent silica gel and report to the nearest gram.

Note: If concentration analysis is to be performed at the sample recovery site, the transfer steps to storage containers can be omitted. The impinger contents and rinses can be placed directly into a large volume mixing container. The appropriate weights of impinger contents and rinses should be recorded as described in 7.2.1 and 7.2.2. The mixed sample should be allowed to stand for at least one-half hour to allow dissolution of the filter catch.

7.3.3 Determination of concentration--Remove three 100 ml aliquots from the sample mixing container and place in separate 250 ml beakers. Add one or two drops of methyl red indicator to each beaker. If sample is yellow, add 25 percent H_2SO_4 dropwise until first red. To the red solution add concentrated

agua ammonia dropwise to first yellow. Add 0.20 grams of dibasic potassium phosphate and stir to dissolve.

The sample should be at the same temperature as the calibration standards when measurements are made. If ambient lab temperature fluctuates more than $\pm 2^{\circ}\text{C}$ from the temperature at which the calibration standards were measured, condition samples and standards in a constant temperature bath before measurement. Stir the sample with a magnetic stirrer during measurement to minimize electrode response time. If the stirrer generates enough heat to change solution temperature, place a piece of temperature insulating material such as cork, between the stirrer and the beaker.

Insert the nitrate and reference electrodes into the solution. When a steady millivolt reading is obtained, record it. Determine the concentration from the calibration curve. Between electrode measurements rinse the electrodes with distilled water and wipe dry. Report the average of the three aliquot determinations as the sample nitrate concentration in gram moles/liter (molarity).

7.3.4 Blank determination--Treat three 100 ml aliquots of the distilled water used for sampling and sample recovery as in 7.3.3. Report the average blank concentration in molarity.

8. Calibrations

8.1 Sampling train. Use methods and equipment which have been approved to calibrate the orifice meter, pitot tube, dry gas meter, and temperature sensors.

8.2 Analytical apparatus.

8.2.1 Estimate the approximate concentration of the samples to be analyzed by using a previous or manufacturer's supplied calibration with a trial aliquot treated as in 7.3.3.

8.2.2 Prepare standardizing solutions that closely bracket this range and provide at least two calibration points between the end points. These can be prepared by appropriate dilution of the 0.1 M standard solution.

8.2.3 Zero and standardize the electrometer according to the manufacturer's instructions using the standards in 8.2.2, treated as in 7.3.5. Begin with the most concentrated and proceed to the most dilute. Reverse the order and repeat the millivolt determination. Record the readings for each concentration. Prepare a calibration curve by plotting the millivolt reading on the linear scale of semi-logarithmic graph paper versus the concentration on the log scale. Calibrate the nitrate electrode daily and check hourly.

9. Calculations

9.1 Average dry gas meter temperature: see data sheet, Figure 2.

9.2 Sample gas volume at standard conditions: Calculate the volume of gas sampled on a wet basis at standard conditions (70°F, 29.92" Hg.) using equation 9-1.

$$V_{ss} = \frac{17.71 V_m P_b}{T_m} + 0.0474 W_c \quad \text{Equation 9-1}$$

Where:

V_{ss} = Volume of gas sampled at standard conditions

V_m = Volume measured by dry gas meter

P_b = Dry gas meter pressure (barometric) in. Hg.

T_m = Average dry gas meter temperature, °R

W_c = Weight of water condensed in the sampling train and collected in the silica gel, grams

17.71 = Conversion to standard conditions, $\frac{530^\circ\text{R}}{29.92'' \text{ Hg.}}$

0.0474 = Conversion factor, ft^3/gm at 70°F, 29.92" Hg.

9.3 Weight of ammonium nitrate collected: Calculate the total weight of ammonium nitrate collected in the sample by Equation 9-2.

$$W_n = 0.08 [v_t C_t - v_b C_b] \quad \text{Equation 9-2}$$

Where:

W_n = Weight of ammonium nitrate collected, grams

v_t = Total volume of mixed sample, ml

C_t = Nitrate molarity of mixed sample, gm moles/liter

v_b = Total volume of water initially placed in train plus all rinse volumes, ml

C_b = Nitrate molarity of blank water, gm moles/liter

0.08 = Conversion factor: gram·liters/ml·gm mole

9.4 Emission concentration. Calculate the particulate (ammonium nitrate) emission concentration at wet standard conditions by Equation 9-3.

$$C_{ns} = \frac{15.43 W_n}{V_{ss}} \quad \text{Equation 9-3}$$

Where:

C_{ns} = Particulate (ammonium nitrate) emission concentration at wet standard conditions, grains/SCF

15.43 = Conversion factor, grains/gram

W_n = Weight of ammonium nitrate collected, grams

9.5 Isokinetic variation. Calculate the average isokinetic variation by Equation 9-4.

$$\%I = \frac{1032 V_{ss} T_s}{V_s P_s D_n^2 t} \quad \text{Equation 9-4}$$

Where:

%I = Isokinetic variation

1032 = Conversion factor

V_{ss} = Total sample volume at standard conditions, SCF

T_s = Average stack temperature, °R

V_s = Average stack velocity, fpm, from Equation 2-2, Method 2,
Federal Register, Vol. 36, No. 247, December 23, 1971

P_s = Stack pressure, in. Hg. absolute

D_n = Nozzle diameter, inches

t = test time, minutes

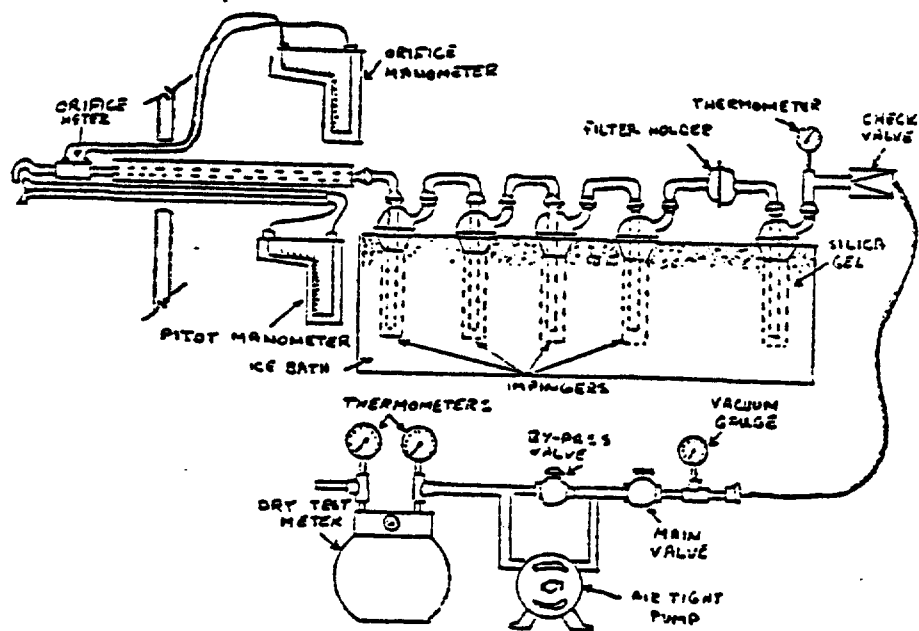


FIGURE 1. NITRATES SAMPLING TRAIN

PROBE LENGTH AND TYPE _____
 NOZZLE I.D. _____
 ASSIGNED MUSTUNE, % _____
 SAMPLE BOX NUMBER _____
 METER BOX NUMBER _____
 METER K_m _____
 C FACTOR _____
 PROBE HEATER SETTING _____
 HEATER BOX SETTING _____
 REFERENCE Δp _____

PLANT _____
DATE _____
SAMPLING LOCATION _____
SAMPLE TYPE _____
RUN NUMBER _____
OPERATOR _____
AMBIENT TEMPERATURE _____
DIAGNOSTIC PRESSURE _____
STATIC PRESSURE, (P_s) _____
FILTER NUMBER (S) _____

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY _____ MINUTES

[illegible]

SUBJECT

1557 (cont.) 235

SAMPLE RECOVERY DATA

Plant: _____

Comments

Date: _____

Sampling Location: _____

Sample Type: _____

Run Number: _____

Sample Box I.D.: _____

Cleanup Operator: _____

	Container 1	Container 2	Container 3
Sample Number	_____	_____	_____
1. Container Tare	_____	_____	_____
2. Weight after adding impinger contents	_____	_____	_____
3. Weight after adding washes	_____	_____	_____
Net liquid weight = (3-1)	_____	_____	_____
Net impinger contents weight = (2-1)	_____	_____	_____
A. Total Net Liquid Weight: _____			
B. Total Impinger Contents: _____			
C. Initial Impinger Contents: _____			
D. Net Condensate: _____			= (B-C)
E.* Sample Transfer Rinse Volume: _____			
F. Final Silica Gel Weight: _____			
G. Initial Silica Gel Weight: _____			
H. Net Silica Gel Weight Gain: _____			= (F-G)
I. Total Water Collected: _____			= (D+H)
J. Total Sample Volume: _____			= (A+E)
K. Total Blank Volume: _____			= (A-B+C+E)

APPENDIX F-2
FIELD DATA SHEETS FOR SYNTHESIS TOWER SAMPLING

TRAVERSE POINT LOCATION FOR CIRCULAR DUCTS

PLANT C F Industries
DATE 1-18-79
SAMPLING LOCATION SOLUTION TOWER VENT
INSIDE OF FAR WALL TO
OUTSIDE OF NIPPLE, (DISTANCE A) 14"
INSIDE OF NEAR WALL TO
OUTSIDE OF NIPPLE, (DISTANCE B) -
STACK I.D., (DISTANCE A - DISTANCE B) 14"
NEAREST UPSTREAM DISTURBANCE 16' (13.7 Dia.)
NEAREST DOWNSTREAM DISTURBANCE ~30' (~26 Dia.)
CALCULATOR S. Richardson

SCHEMATIC OF SAMPLING LOCATION

[illegible]

Firm Name: CF Tool Pump No. 11 FIELD DATA SHEETS 4-4 Monograph NA
 Muzzle No. & Dia. 0.5 in. C Factor
 Plant Location: Pandemonium Office No. instale 0.25" Assumed Moisture NA Pilot Coefficient 0.837
 Test No. 4 Ambient Temp 73 °F Test Duration 15 min. Orifice All NA
 Sampling location: Sub 1 Tower Bar Press 30.27 "Hg Traverse Point Interval 15 min. Test Start Time 1540
 TRC Project No. 62988-20 Probe Ident. No. 3850 Probe Heater Setting 190°F amps Test End Time 1555
 Date 1/18/79 Filter Ident. No. NA Filter Temp. Setting NA Leak Test Start 0.004 CFME -4 "Hg
 Tester RWC / DEB / SFR Duct Area 1.07 ft² Dry Gas Meter Y NA Leak Test Final 0.004 CFME -4 "Hg

$\Delta h = 21.24 \text{ in H}_2\text{O}$

Port	Point	3	4	5	6	7	8	9	10	12	13	14	15	18	19	20	23	24	25	26	27	28	29	30	32	33	34	35	40	41	42	43	44	45	50	52	53	AKO over heater	Temp. Box	Temp. of Gas Leaving Condenser °F	Pump vac. in Hg	Probe Temp. °F	O ₂ Conc. %																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																												
		Time Min		Velocity ΔP in H ₂ O				All in H ₂ O		T _H in °F		T _H Out °F		P _{STACK} in H ₂ O		T _{STACK} °F		Initial Water Volume cu. ft.		P R Q E		Cyc. Angle °																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																	

[illegible][illegible]

ORGANIC PARAMETERS				
Time	CO ₂	O ₂	Ca	N ₂
1				
2				
3				

H₂O Condition

Silica Gel Cond.

APPENDIX F-3
SYNTHESIS TOWER TEST RESULT CALCULATIONS

Synthesis Tower Data Analysis

The impinger catch for these synthesis tower test runs consisted of water mass and ammonia mass; the masses of urea and formaldehyde were negligible. The ammonia mass was therefore subtracted from the total impinger catch to yield the mass of water collected. This water includes both collected condensed vapor and collected liquid water droplets. As discussed in Section 5, the existence of liquid water droplets in the gas stream was confirmed by observation and by the moisture data. As is shown in the following data and calculations, the volume of water collected exceeded the volume that would be carried by a saturated gas stream at the stack temperatures:

<u>Run</u>	<u>Condensed Water Vapor (lb)</u> <u>(Saturated Conditions)</u>	<u>Actual Water</u> <u>collected (lb)</u>
1	0.479	0.65
2	0.339	0.77
3	0.437	0.80

The following pages show the procedure used to calculate urea, formaldehyde and ammonia emissions.

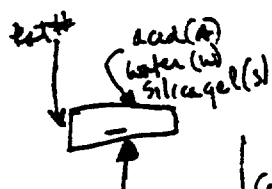


Table F-1: Mass H_2O collected for solution tower tests

Container # code key Container #	A final wt. (container + tower + sample + H_2O) gm	B initial wt. (container + tower) gm	C impinger change weight gm	A-B-C $H_2O + NH_3$ collected gm	D wt NH_3 in sample gm	A-B-C-D wt H_2O collected, gm	total H_2O gm	lb
4S-A	874.18	498.84	381	-5.66	<0.1	-5.66	306.86	0.45
4S-W	1142.54	501.89	300	340.65	37.69	302.96		
4S-S	252.75	243.19	-	9.56	0	9.56		
5S-A	875.73	498.64	381	-3.91	<0.1	-3.91	363.81	0.77
5S-W	1279.5	498.97	300	408.53	45.8	362.73		
5S-S	249.21	244.22	-	4.99	0	4.99		
6S-A	883.65	498.04	381	4.61	~0.1	4.61	380.83	0.84
6S-W	1226.8	501.29	300	425.51	57.81	367.7		
6S-S	251.33	242.81	-	8.52	0	8.52		

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TRC Project # 82988-20 CF Industries, Inc

Calculation of Synthesis Tower Results

Run #1 (Test #4), Run #2 (Test #5), Run #3 (Test #6)

1. Determine Moisture Content of Gas Stream - assumed saturated conditions

Dalton's Law
$$\frac{V_{H_2O}}{V_{total}} = \frac{P_{H_2O}}{P_T}$$

where V_{H_2O} / V_{total} = volume ratio of water vapor to total P_{H_2O} = vapor pressure of H_2O @ T_{stack} P_T = Stack Pressure Total

Run #	$T_{stack}, ^\circ F$	$P_{stack}, "Hg$	VAPOR PRESSURE OF WATER AT T_{stack} $P_{H_2O}, ("Hg)^{**}$	Theoretical % M $(V_{H_2O} / V_{total}) \times 100$	Calculated % M*
1	193	30.27	20.27	67.0	74.2
2	186	30.17	17.44	57.8	76.5
3	191	30.12	19.42	64.5	77.8

* CALCULATED FROM TOTAL VOLUME OF WATER COLLECTED

** "Methods For DETERMINATION OF VELOCITY, VOLUME, DUST, AND MIST CONTENT OF GASES", BY Harold H Haaland, page 35.

2. Calculate Volume of Water Vapor in Sample, V_{H_2O} SCF

$$\left(\frac{V_{H_2O}}{V_T} \right) = \frac{V_{H_2O}}{V_{Air} + V_{NH_3} + V_{H_2O}}$$

$$V_{H_2O} = \frac{\left(\frac{V_{H_2O}}{V_T} \right) (V_{Air} + V_{NH_3})}{\left(1 - \frac{V_{H_2O}}{V_T} \right)}$$

where $V_{NH_3} = \omega_{NH_3} \times \rho$

$$V_{NH_3} = \omega_{NH_3} \times 4.97 \times 10^{-5} \text{ ft}^3 \text{ NH}_3 / \text{mg NH}_3$$

Run #	V_{H_2O}/V_T	V_{Air}, ft^3	ω_{NH_3}, mg	V_{NH_3}, ft^3	V_{H_2O}, ft^3
1	0.6696	3.19	37,720	1.882	10.25
2	0.5780	3.01	45,840	2.287	7.26
3	0.6448	2.26	57,810	2.885	9.34

3 A. Calculate Wet Molecular Weight of Gas Stream, $\bar{M}_w$, lb/lb-mole

$$\bar{M}_w = \frac{1}{\omega_{NH_3} + \omega_{Air} + \omega_{H_2O}} \left(\frac{1}{lb} \right) \left[\omega_{NH_3} \times 17 + \omega_{Air} \times 29 + \omega_{H_2O} \times 18 \right] \left(\frac{lb-lb}{lb-mole} \right)$$

Run #	ω_{NH_3}, lb	ω_{Air}, lb	ω_{H_2O}, lb	$M_w, lb/lb-mole$
1	0.0831	0.240	0.479	21.2
2	0.101	0.226	0.339	21.6
3	0.127	0.170	0.437	20.4

where $\omega (lb) = V (ft^3) \times \frac{M (lb/lb-mole)}{385 (ft^3/lb-mole)}$

$M_{Air} = 29$

$M_{H_2O} = 18$

$M_{NH_3} = 17$

3 B. Calculate Dry Molecular Weight of Gas Stream, $\bar{M}_D$, lb/lb-mole

$$M_D = \frac{1}{\omega_{Air} + \omega_{NH_3}} \left(\frac{1}{lb} \right) \left[\omega_{NH_3} \times 17 + \omega_{Air} \times 29 \right] \left(\frac{lb-lb}{lb-mole} \right)$$

3 B (continued)

Run #	$\bar{M}_D, \text{lb/lb-mole}$
1	25.9
2	25.3
3	23.9

4. Calculate Velocity V , ft/min and Volumetric Flow Rate Q , ft³/min of Gas Stream

$$V = 174 \times C_p \times \sqrt{V_P \times T_R} \times \sqrt{\frac{29.92}{P_s} \times \frac{28.84}{M_w}}$$

$$Q = V (\text{ft/min}) \times A (\text{ft}^2)$$

where

174 = units correction factor

C_p = pitot tube calibration coefficient

$\sqrt{V_P \times T_R}$ = average of square roots of individual V_P and T product for each sampling point

note $T = ^\circ R = ^\circ F + 460$

P_s = Static Pressure, "Hg

$\bar{M}_w$ = Gas Stream Molecular Weight, lb/lb-mole (WET)

A = stack Cross-Sectional Area, ft²

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4 (continued)

Run #	C _p	$\sqrt{VP \times TR}$	P _s , "Hg	M _w , lb/lb-mole	V, ft ³ /min	Q, ft ³ /min
1	0.837	6.26	30.27	21.2	1057	1131
2	0.837	6.48	30.17	21.6	1086	1162
3	0.837	6.25	30.12	20.4	1079	1154

$$A = 1.07 \text{ ft}^2$$

5. Calculate Gas Stream Flow Rate, Q_{STD} dry @ 68°F and 29.92 "Hg

$$Q_{STD} \left[\frac{\text{ft}^3}{\text{min}} \right] = Q_{ACFM} \left[\frac{\text{ft}^3}{\text{min}} \right] \times \left(1 - \frac{\% \text{H}_2\text{O}}{100} \right) \times \frac{528}{T_s + 460} \left(\frac{^{\circ}\text{R}}{^{\circ}\text{R}} \right) \times \frac{P_{STACK} \left(\frac{\text{"Hg}}{\text{"Hg}} \right)}{29.92}$$

Run #	Q _{ACFM}	1 - %H ₂ O/100	T _s + 460, °R	P _s , "Hg	Q _{DSCFM}
1	1131	0.33	653	30.27	306
2	1162	0.42	646	30.17	402
3	1154	0.36	651	30.12	339

6. Calculate Ammonia Emissions, M_{NH_3} (lb/hr)

$$M_{NH_3} (lb/hr) = \frac{\omega_{NH_3}}{\omega_{NH_3} + \omega_{air}} \left(\frac{lb}{lb} \right) \times \phi_{STD} \left(\frac{ft^3}{min} \right) \times M_D (lb/lb-mole) \times 0.156 \left(\frac{min}{ft^3 hr} \right)$$

Run #	ω_{NH_3} , lb	ω_{air} , lb	ϕ_{STD} , ft^3/min	M_D , lb/lb-mole	M_{NH_3} , lb/hr
1	0.0831	0.240	306	25.9	318
2	0.101	0.226	402	25.3	490
3	0.127	0.170	339	23.9	540

7. Calculate Urea Emissions, M_U (lb/hr)

$$M_U (lb/hr) = \frac{\omega_{urea}}{\omega_{NH_3} + \omega_{air}} \left(\frac{lb}{lb} \right) \times \phi_{STD} \left(\frac{ft^3}{min} \right) \times M_D \left(\frac{lb}{lb-mole} \right) \times 0.156 \left(\frac{min}{ft^3 hr} \right)$$

Run #	ω_{urea} , lb	ω_{NH_3} , lb	ω_{air} , lb	ϕ_{STD} , ft^3/min	M_D , lb/lb-mole	M_U , lb/hr
1	4.16×10^{-4}	0.0831	0.240	306	25.9	1.59
2	4.36×10^{-4}	0.101	0.226	402	25.3	2.11
3	4.09×10^{-4}	0.127	0.170	339	23.9	1.74

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8. Calculate Ammonia Concentration, C_A (grains/dscf)

$$C_A (\text{grains/dscf}) = \frac{W_{NH_3}}{V_{air} + V_{NH_3}} \left(\frac{\text{lbs}}{\text{ft}^3} \right) \times 7000 \text{ grains/lb}$$

Run #	W_{NH_3}, lb	V_{air}, ft^3	V_{NH_3}, ft^3	$C_A, \text{gr/dscf}$
1	0.083	3.19	1.882	115
2	0.101	3.01	2.287	134
3	0.127	2.26	2.885	173

9. Calculate Urea Concentration, C_U (grains/dscf)

$$C_U (\text{grains/dscf}) = \frac{W_U}{V_{air} + V_{NH_3}} \left(\frac{\text{lbs}}{\text{ft}^3} \right) \times 7000 \text{ grains/lb}$$

Run #	W_U, lb	V_{air}, ft^3	V_{NH_3}, ft^3	$C_U, \text{gr/dscf}$
1	4.16×10^{-4}	3.19	1.882	0.574
2	4.36×10^{-4}	3.01	2.287	0.576
3	4.09×10^{-4}	2.26	2.885	0.556

CF I Donaldsonville

SOLUTION TOWER ISOKINETIC CALCULATIONS 11/4/79 JEK

test 4

$$T_{SAVG} = 193^{\circ}F \quad V_{mSTD}(\text{air}) = 3.19 \quad P_{bar} = 30.27 \quad P_s = 0.00$$

$$V_s = 1057 \text{ ft/min} \quad T_{total} = 15 \text{ min} \quad \%H_2O = 67.0 \quad D_n^2 = 0.508$$

$$lb \text{ NH}_3 = 0.083$$

$$V_{NH_3 STD} = \frac{lb \text{ NH}_3}{17 lb / lb\text{-mole}} \times \frac{385 \text{ ft}^3 \text{ NH}_3}{lb\text{-mole}} = 1.88 \text{ ft}^3 \text{ at } 68^{\circ}F$$

29.92" Hg

$$\therefore I = \frac{5.67(T_{SAVG} + 460) [V_{mSTD}(\text{air}) + V_{STD}(\text{NH}_3)]}{\left(P_{bar} + \frac{P_{SAVG}}{13.6}\right) V_s T \left(1 - \frac{\%H_2O}{100}\right) \left(\frac{D_n^2 \times 0.7854}{144}\right)}$$

$$= \frac{(5.67)(193 + 460) [3.19 + 1.88]}{(30.27)(1057)(15) \left(1 - \frac{67.0}{100}\right) \left(\frac{(0.508)^2 (0.7854)}{144}\right)}$$

$$= 84.1 \%$$

test 5

$$T_{SAVG} = 186^{\circ}F \quad V_{mSTD}(\text{air}) = 3.008 \quad P_{bar} = 30.17 \quad P_s = 0.00$$

$$V_s = 1086 \quad T_{total} = 15 \text{ min} \quad \%H_2O = 57.8 \quad D_n = 0.508$$

$$lb \text{ NH}_3 = 0.101$$

$$V_{NH_3 STD} = \frac{0.101}{17} \times 385 = 2.29 \text{ ft}^3$$

$$I = \frac{(5.67)(186 + 460) (3.008 + 2.29)}{(30.17)(1086)(15) \left(1 - \frac{57.8}{100}\right) \left(\frac{(0.508)^2 (0.7854)}{144}\right)}$$

$$= 66.5 \%$$

SOLUTION TOWER ISOKINETIC CALCULATIONS 4/4/79 JEK

test 6

$$T_{S,AVG} = 191^{\circ}F \quad V_{M,STD}(\text{air}) = 2.26 \quad P_{bar} = 30.12 \quad P_s = 0.00$$
$$V_s = 1079 \text{ ft}^3/\text{min} \quad T_{total} = 15 \text{ min} \quad \%H_2O = 64.5 \quad D_n = 0.508$$
$$lb \text{ NH}_3 = 0.127$$

$$V_{\text{NH}_3, STD} = \frac{lb \text{ NH}_3}{17 \text{ lb/lb-mole}} \times 385 \frac{\text{ft}^3 \text{ NH}_3}{\text{lb-mole}} = 2.88 \text{ ft}^3 \text{ NH}_3$$

$$I = \frac{(5.67)(191 + 460)(2.26 + 2.88)}{(30.12)(1079)(15)\left(1 - \frac{64.5}{100}\right)\left(\frac{(0.508)^2(0.7854)}{144}\right)}$$
$$= 77.9 \%$$

APPENDIX F-4

IN-STACK ORIFICE DEVELOPMENT

The standard EPA-5 particulate sampling procedure requires the isokinetic collection of a stack gas. The moisture in the wet stack gas is removed in a condenser/dryer after particulate removal. The dried sample stream then passes through a pump, a gas meter for volume measurement, and then finally through an orifice meter for continuous sampling rate measurement and control. The orifice meter differential pressure required to achieve isokinetic conditions at the sampling nozzle, expressed as a function of the pitot tube pressure differential, is:

$$\Delta h = \left\{ 847.8 C_p^2 (1-B)^2 \Delta H_g \left[\frac{M_d}{M_d(1-B) + 18B} \right] \left(\frac{P_s}{P_m} \right) (TM) \right\} \frac{1}{T_s} D_n^4 \Delta p$$

where:

Δh = orifice meter differential pressure, inches H_2O

847.8 = units conversion factor

C_p = pitot tube coefficient, dimensionless

B = stack gas moisture content, volume fraction

ΔH_g = standard orifice meter calibration coefficient

M_d = stack gas dry molecular weight, lb/lb mole

P_s = absolute pressure of stack gas, inches H_g

P_m = absolute pressure of sample stream at orifice meter, inches H_g

T_m = temperature of sample stream at orifice meter, $^{\circ}R$

T_s = temperature of stack gas, $^{\circ}R$

D_n = nozzle diameter, inches

Δp = pitot tube pressure differential, inches H_2O

In order to use this relationship, it is necessary to know prior to sampling the following variables:

- (1) pitot tube coefficient, C_p
- (2) standard orifice calibration coefficient, ΔH_o
- (3) nozzle diameter, D_n
- (4) stack gas pressure P_s
- (5) sample gas temperature and pressure at the orifice, T_m and P_m
- (6) stack gas dry molecular weight, M_d
- (7) stack gas temperature, T_s
- (8) pitot tube pressure differential, Δp
- (9) stack gas moisture content, B

Variables (1) to (3) are determined by the particular combination of pitot tube, orifice meter and nozzle used for sampling. Variables (4) to (6) are usually constant or can be estimated with confidence from a preliminary measurement. Variables (7) and (8) are continuously monitored during testing. This leaves the stack gas moisture content as the remaining variable.

In the majority of sampling situations encountered, the stack gas moisture content can be measured or estimated to an adequate degree of confidence so that isokinetic sampling can be easily achieved. This is the case when the moisture content is less than about 40 percent by volume and is relatively constant.

However, in those cases where the moisture content is greater than 40 percent, or when the content varies significantly, a single average value is usually an inadequate estimate. Since the moisture content is not a linear variable in the isokinetic equation, the estimation error that is tolerable decreases as the absolute value of moisture content increases.

There have been two different types of sampling situations recently encountered where the moisture content was very high (40 to 90 percent) and highly variable.

In the first of these cases, the sampling location was after a scrubber where the gas temperature was less than 212°F, psychometric charts could be used to estimate saturation contents, and the essentially instantaneous moisture content values were used to solve the isokinetic equation. At a facility where the moisture content was 45 percent, this procedure for estimation of moisture was adequate while at the higher moisture content location it was not. In either case, the technique was cumbersome and marginally reliable.

In the second type of situation, the sampling location was after an aqueous reactor where the vent stream was essentially water vapor at temperatures greater than 212°F. The moisture contents range from 60 to 99 percent by volume. In these ranges, there is no procedure available to adequately estimate the moisture content with the accuracy necessary. A second complication at this type source is that after condensation of moisture in the sample stream there is an inadequate amount of dry gas left to enable accurate sample flow rate measurement of the orifice meter.

In order to enable accurate reliable isokinetic sampling at these types of sources, an in-stack orifice meter system was developed in which the sampling rate is measured at stack conditions prior to any moisture condensation. The development, specifications, calibration and operation of this type meter is discussed below.

A. Meter Design Development

Three potential systems were evaluated for use in the measurement of sampling rate prior to moisture condensation. These were (1) the null-balance probe, (2) venturi meters, and (3) orifice meters.

The primary factors considered were:

- (1) difficulty of fabrication
- (2) calibration requirements
- (3) interchangeability with standard EPA-5 equipment
- (4) usability with standard three-inch sampling ports.

The null-balance probe approach was not used because of the requirement that it be calibrated at each individual source and also because of the large size of most designs.

This left venturi or orifice meters as the potential alternatives. An orifice meter approach was chosen based on the conclusions that an orifice is easier to construct, and that its measurement range can be easily changed by varying the orifice opening.

The first design proposed for the meter assembly is shown in Figure A1. While this assembly was directly mountable on the EPA-5 probe, it was found that it was not usable in three-inch ports. Calibration using this first prototype did confirm that the orifice meter approach was valid and that sampling rates could be controlled as desired.

The second generation design was fabricated so that the orifice meter body and the nozzle were not combined into one unit. In this design, the orifice was located between the gooseneck nozzle and the probe end-union. This allowed use of the assembly in a 3-inch port. The design also allowed for the installation of the orifice in the vertical section between the nozzle

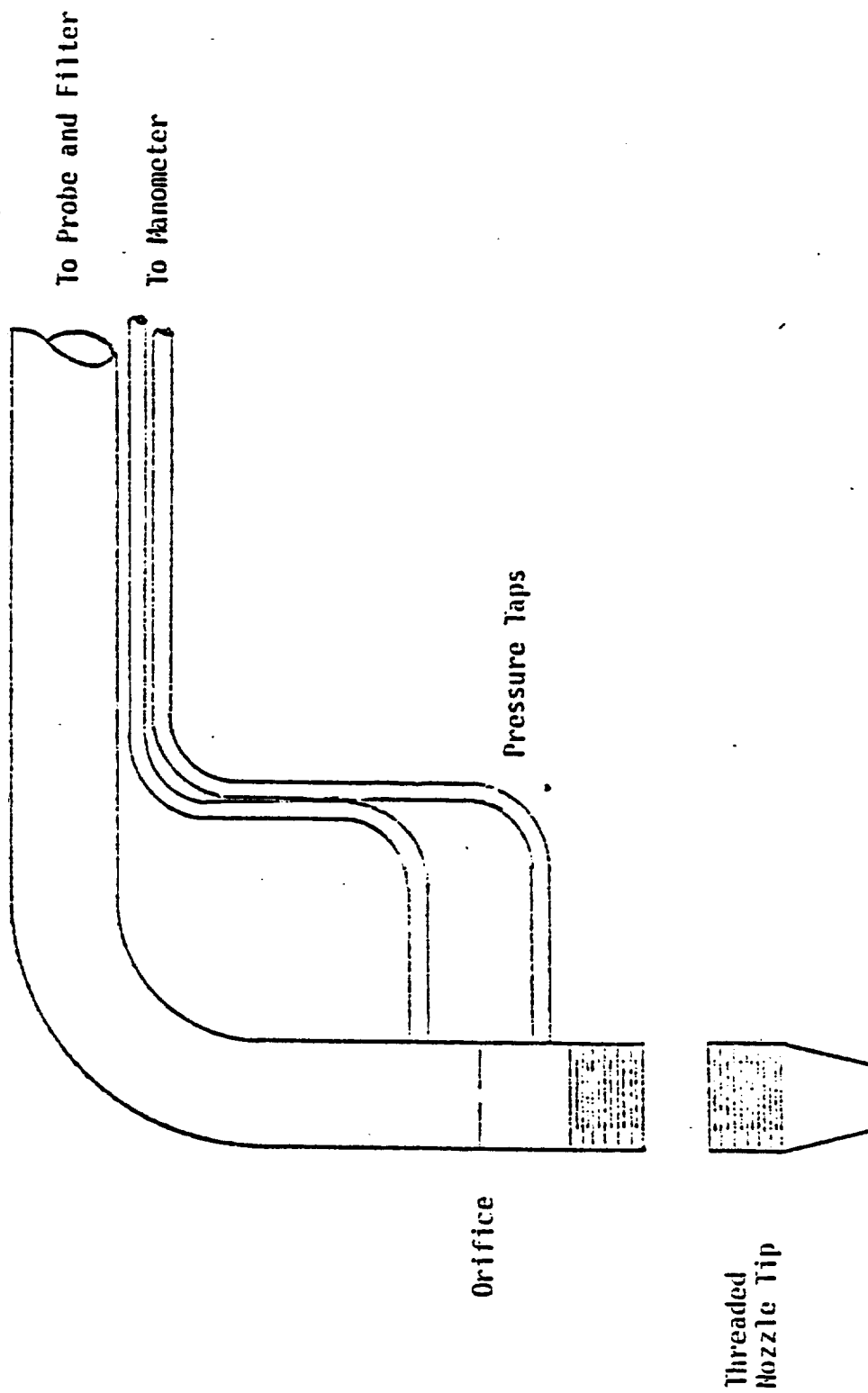


Figure A1. Initial Design, In-stack Orifice and Nozzle

tip and the gooseneck when stack access ports are available to accommodate that configuration. This meter was fabricated from solid stock with the orifice plate machined permanently. This design was found to operate very satisfactorily. The prime disadvantage with this design was that a complete orifice body had to be machined to obtain a different measurement range.

To overcome this time-consuming and expensive problem, a third-generation prototype was fabricated whereby the meter body was separable at the orifice plate and range changing is accomplished by simply installing a washer with a different hole size. This design is illustrated in Figure A2. This entire assembly, including the pressure tap lines, is constructed of stainless steel. This is necessary to maintain an inert surface for sample contact and to prevent erosion and corrosion on the outer surfaces.

B. Orifice Calibration

The standard orifice equation relating volumetric flow rate to orifice pressure differential is:

$$Q_m = K_m \sqrt{\frac{\Delta h T_m}{P_m M_m}}$$

where

Q_m = volumetric flowrate, acfm

K_m = orifice calibration coefficient

Δh = orifice pressure differential, inches H_2O

T_m = gas temperature, $^{\circ}R$

P_m = gas absolute pressure, inches Hg

M_m = gas average molecular weight, lb/lb mole

During the development of the in-stack orifice, two approaches were taken to determine the orifice coefficient. First, the nozzle/meter assembly was attached to a standard EPA-5 probe, umbilical cord, and meter box, and still room air was drawn through the orifice. In the second, case, a condenser

1-8-81 9/30
 2-297-12/64
 1-250-1/4
 1-33-21/64
 1-261-17/64

TO SAMPLING TUBIN

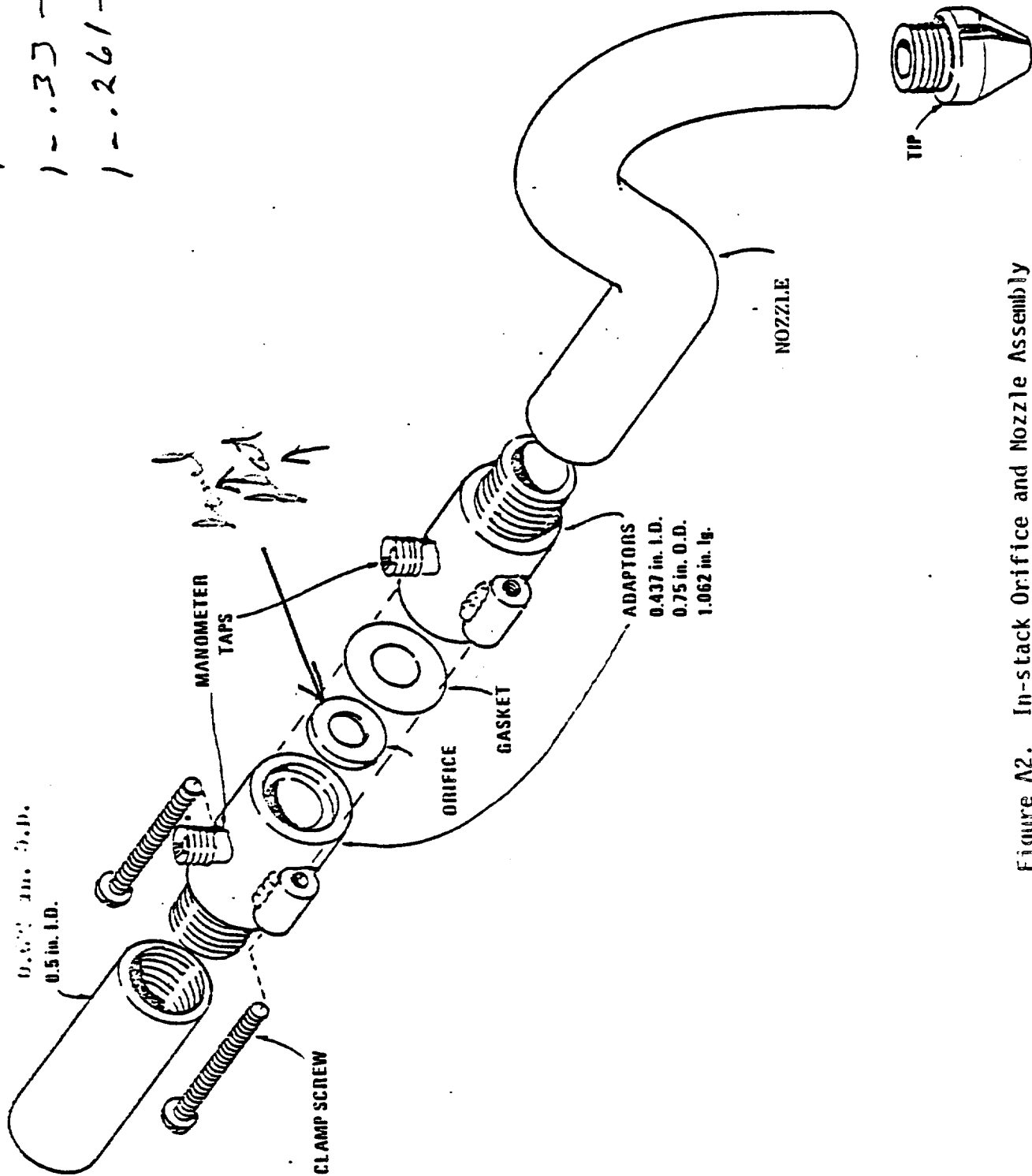


Figure A2. In-stack Orifice and Nozzle Assembly

train was added after the probe and the calibration was carried out in a flowing stream. In the flowing stream, it was possible to perform calibrations at several temperatures.

Calibration calculations were developed based on the combinations of standard equipment used for the procedures described above. These are presented in Figures A3 and A4. A sample of the data sheet developed for calibration studies is shown in Figure A5.

The calibration procedure is essentially identical to that used for the standard orifice in an EPA-5 train. The system is operated at several Δh settings over the range 0.5 to 8 in. H_2O and the average K_m is calculated from the individual K_m values.

During calibrations, the linear operating range of each orifice should be determined. This can be accomplished by comparing the individual meter coefficients at each pressure differential to the average or midrange value. In order to achieve 100 ± 10 percent isokinetic conditions, the maximum allowable deviation in the orifice calibration factor would be ± 10 percent, assuming no errors in any other measurements. Therefore, the maximum linear range of the orifice would be those pressure differentials where the individual meter coefficients are within the range of 90 to 110 percent of the average. It should be recognized that if the meter operation will be near either extreme of the linear range, it is potentially possible to exceed the allowable isokinetic variation. In those cases it is more appropriate to use the calibration factor for that end of the range. This can be avoided if desired by establishing the linear range of the meter at some level less than a ± 10 percent variation in the meter calibration coefficient. For example, a tolerance of ± 5 percent in the meter calibration coefficient would yield

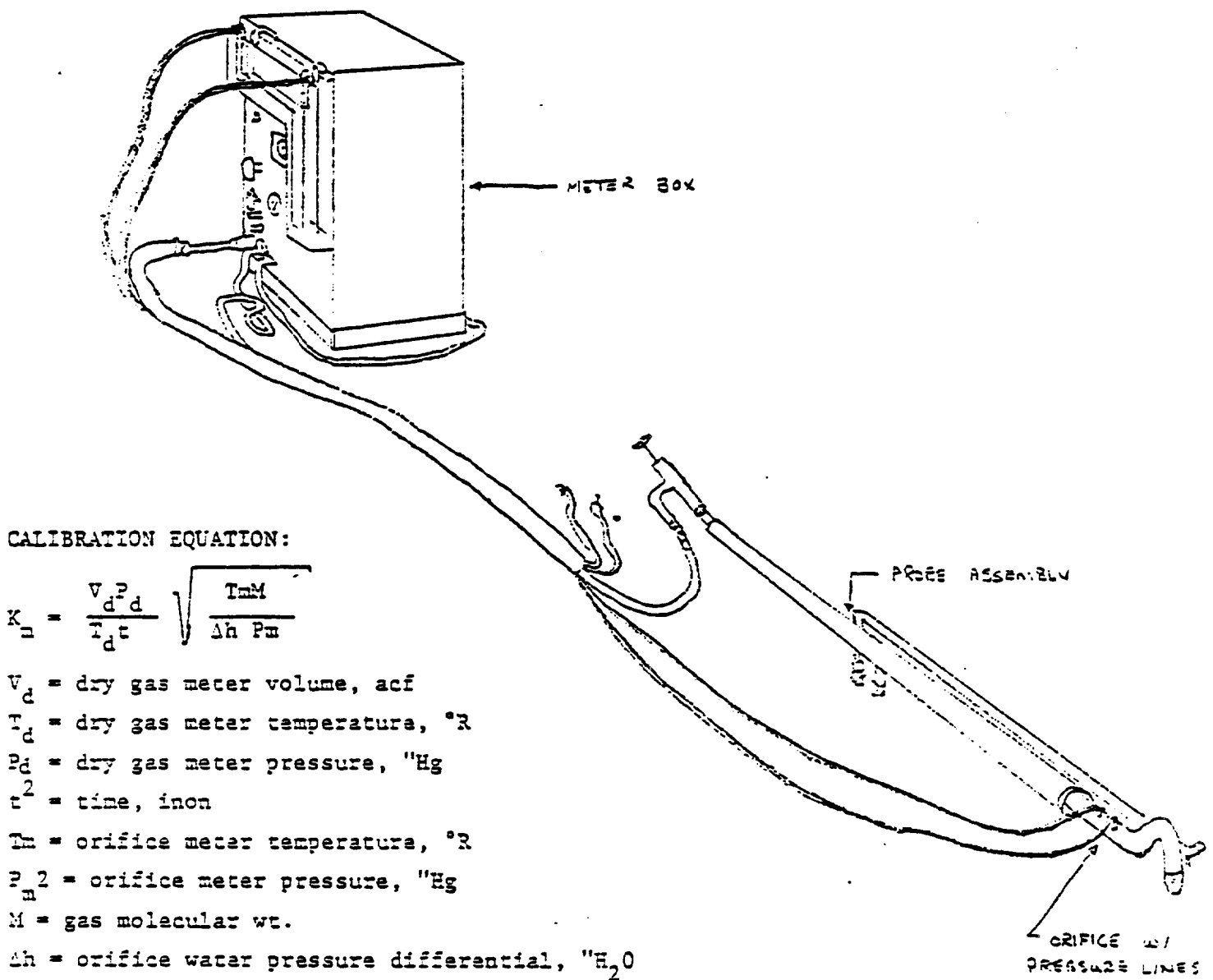


Figure A-3. Calibration Configuration Using Dry Gas

1" — (0) — 3/4"

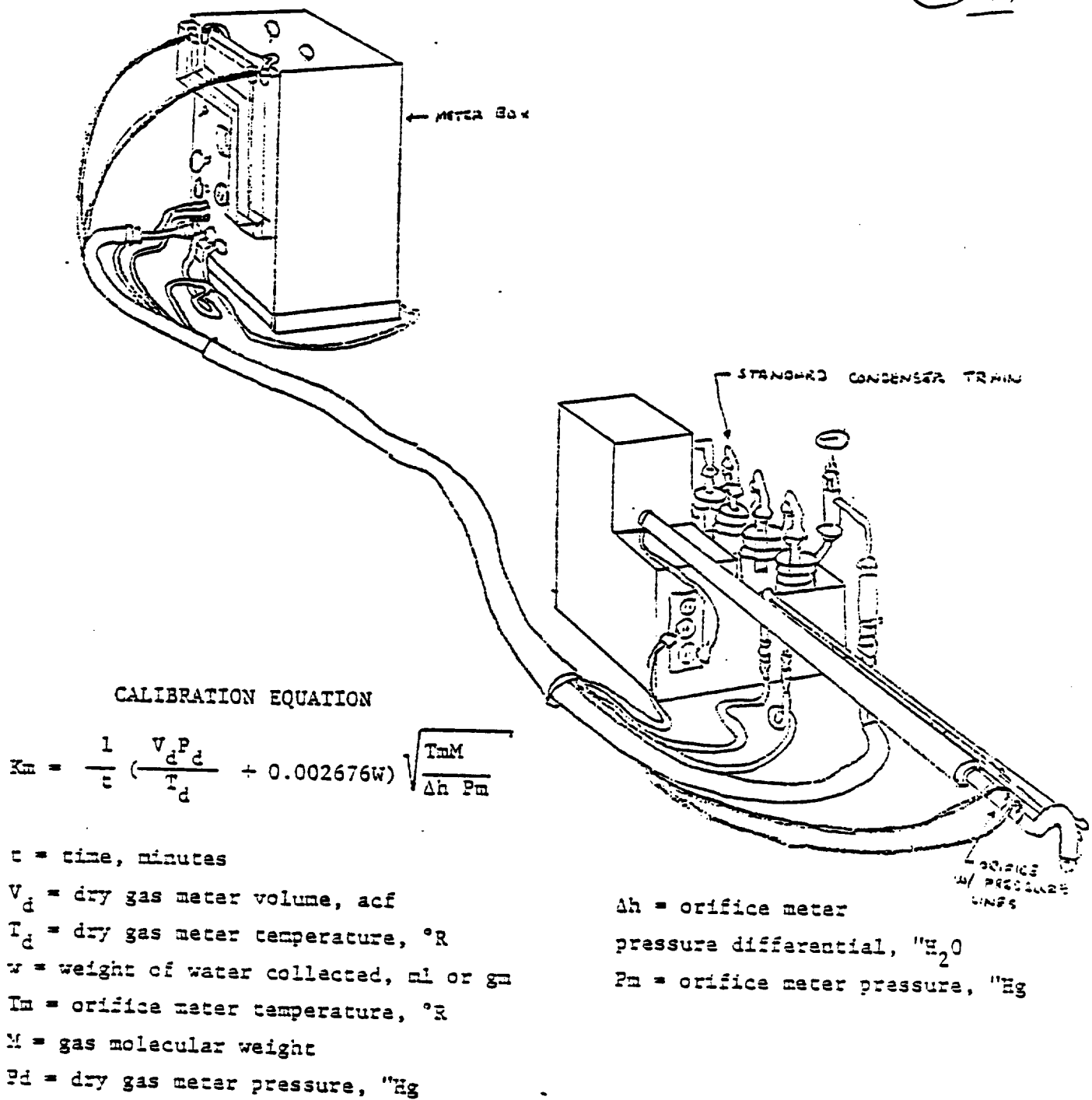


Figure A-4. Calibration Configuration Using Wet Gas

IN-STACK ORIFICE CALIBRATION

Run No: _____

Water collected: _____ ml

Date: _____

Ambient Temp: _____ °F

Barometric Pressure: _____ "Hg

Nozzle Idn.: _____

Time	Volume	Gas meter temp.		Δh	T_m
		Inlet	Outlet		

NOTES: _____

$$K_m = \frac{1}{t} \left(\frac{V_d P_d}{T_d} + 0.002676W \right) \sqrt{\frac{T_m M}{\Delta h P_m}}$$

$K_m =$ _____

Figure A5. Orifice Calibration Data Sheet

a safety factor of 2, but at the same time would decrease the linear operating range. Either alternative can be used so long as the assumptions involved with selecting an average meter coefficient are recognized.

Initial calibration results using the first orifice design indicated a difference in coefficients depending on whether calibration was conducted using still room air or a flowing stream. The specific cause for this effect could not be identified, but it is probably due to the relative closeness of the orifice to the nozzle entrance in that design. Comparative calibrations using the later designs indicate that there is little difference between results using the two procedures.

Calibrations were also performed at temperatures ranging from 80 to 329°F. The results of these tests showed that the orifice coefficient did not change with temperature.

The results of calibration studies with various orifice opening sizes are summarized in Figures A6 and A7. The flow measurement range versus orifice pressure differential is shown in Figure A6. The variation of K_m with orifice opening is shown in Figure A7. These correlations are presented for general information since the specific calibration will depend on the meter design. However, it has been found that duplicate meters of the same design will have essentially identical calibration coefficients for the same size orifice opening.

C. Meter Operation

The operating isokinetic equation for an in-stack orifice meter can be derived from the general EPA-5 operating equation given earlier by substituting the following conditions.

Qm, Flow Rate at Orifice Meter, ACFM

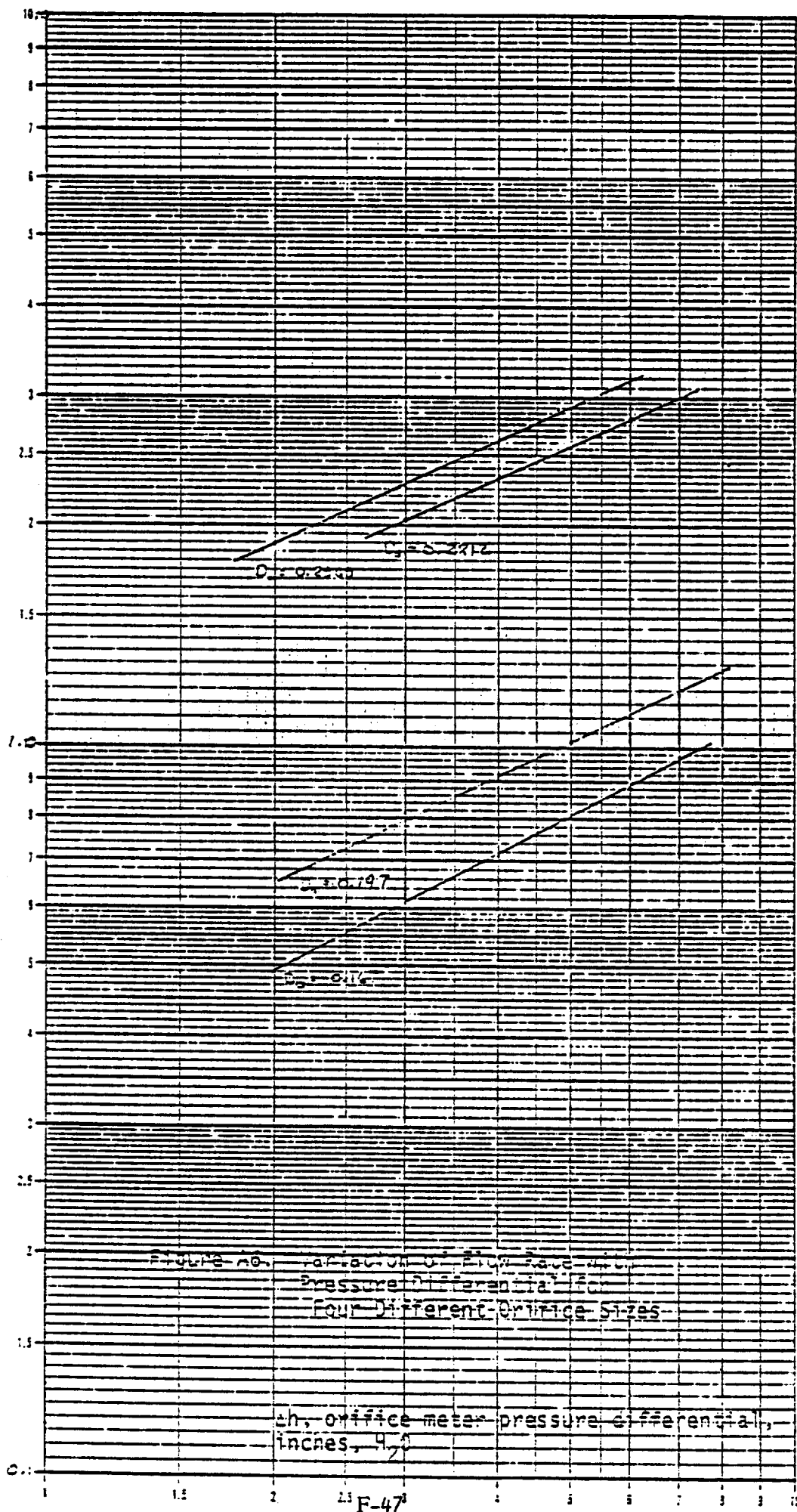
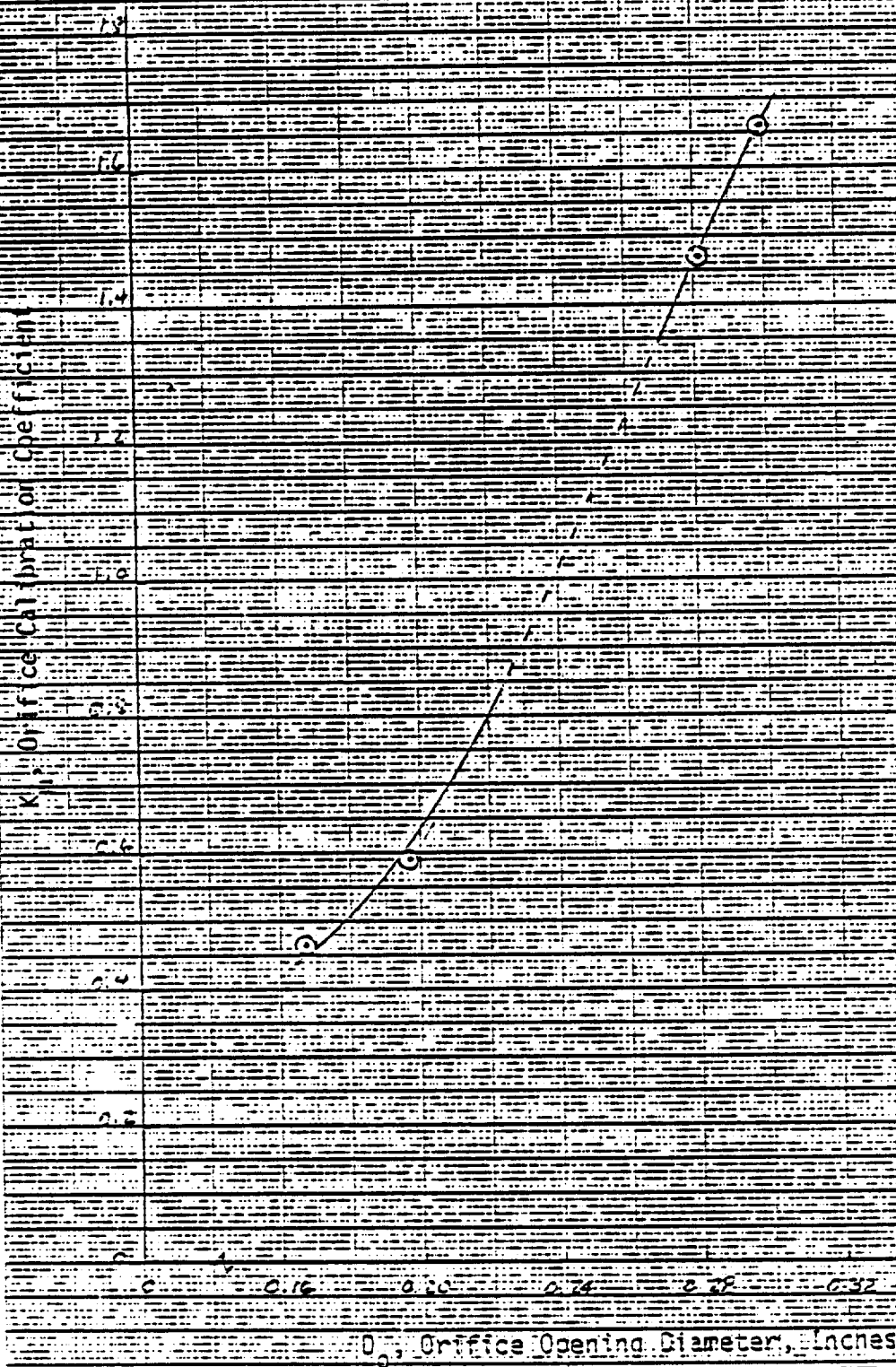


FIGURE A0. Variation of Flow Rate with Pressure Differential for Four Different Orifice Sizes

h, orifice meter pressure differential, inches, 9.75



1. Since the orifice meter is located in the stream, the stack temperature, pressure and molecular weight will equal the meter temperature, pressure, and molecular weight, therefore,

$$\frac{P_s}{P_m} = 1, \frac{T_m}{T_s} = 1, \frac{(1-B)^2 M_d}{M_d(1-B) + 188} = \frac{M_s}{M_m} = 1$$

2. The more convenient form for the meter coefficient is used -

$$\Delta H_{@} = \frac{0.923}{K_m^2}$$

Making these substitutions, the operating equation becomes

$$\Delta h = 782.502 \left(\frac{C_p}{K_m} \right)^2 D_n^4 \Delta p$$

As can be seen, the only independent variable for any one combination of equipment is the pitot tube pressure differential. All of the other variables are removed by placing the orifice in-stack and metering the sample stream at stack conditions. It is necessary, however, to know the approximate stack gas temperature and moisture content in order to properly size the orifice-nozzle combination required.

The standard EPA-5 procedure specifies sampling rates of 0.5 to 1.5 dry standard cubic feet per minute at the orifice meter. For most situations where the use of an in-stack orifice is required, this specification is impractical to achieve. For example, if the moisture content is 50 percent by volume and the stack temperature is 300°F, a sampling rate of 2.35 acfm at the nozzle would be required for a rate of 0.75 dscfm. The multiplication factor becomes even more as the moisture content increases.

A more practical approach is to establish a range of sampling rates at the nozzle. For most cases, a range of from 1.0 to 3.0 acfm will result in adequate total sample volumes. This range will also be compatible with commonly encountered stack velocities. For extremely high velocities, it may be necessary to have a meter with a range as high as 5.0 acfm.

The recommended range of nozzle sizes is from 0.20 to at least 0.325 inches. This range of nozzles combined with one or more orifice ranges should allow sampling in the majority of cases.

The recommended procedure for the selection of an appropriate combination of orifice meter size and nozzle size is as follows:

- (1) Determine from preliminary measurements the stack parameters necessary to calculate the average actual stack velocity. The range of pitot tube differential pressures should also be recorded.

- (2) Choose a nozzle size and multiply by the average stack velocity to obtain the average sampling rate necessary. Usually, for first trial purposes, a nozzle size of about 0.25 inches is appropriate for stack velocities commonly encountered (2500-3000 fpm). On the basis of these calculations, select the orifice size necessary to place the required sampling rate at approximately the middle of the meter's linear operating range. The field calculations necessary in this step are eliminated if a chart is prepared after initial calibrations that express each available orifice meter's linear range in terms of orifice pressure differential (Δh), flowrate (Q), and the applicable stack velocity range for each nozzle size that is available. The stack velocity range is obtained by dividing the upper and lower flowrate limits by the available nozzle areas.

(3) Use the meter calibration coefficient K_m , and the nozzle size as selected above with the pitot tube coefficient to calculate the proportionality constant in the isokinetic equation:

$$\Delta h = 782.50 \left[\frac{C_p}{K_m} \right]^2 D_n^4 \Delta p$$

As a final check, insert the maximum and minimum velocity pressure differential obtained during the preliminary traverse to ascertain that the meter will not be operating outside its linear range. Also, the sampling rate at the dry gas meter should be estimated using the stack moisture and temperature, and an estimate of the dry gas meter temperature. This rate should not exceed the capacity of the meter, which is approximately 2.5 cfm in most cases. After the appropriate orifice size has been selected, all parts should be thoroughly cleaned and assembled. The orifice meter-nozzle assembly can then be attached to a standard EPA-5 probe sheath. A leak check should then be performed by attaching the umbilical cord directly to the probe. A leak rate of zero is attainable at 15 inches Hg vacuum when a metal probe liner is used. However, if a glass liner is used this criteria may not be achievable at vacuums greater than 2-5 inches Hg. In all cases, it must be insured that there are no leaks in the pressure differential lines. The rest of the train assembly and testing are performed similarly to that used in EPA-5.

There are several specific areas that are unique to sampling with this equipment configuration and require comment. These are:

(a) In some sampling cases, there will be a measurable pressure differential across the final orifice in the standard equipment configuration. In order to correct the dry gas meter pressure to standard conditions, this differential should be recorded. Since this would require an extra manometer

and data entry, it is more convenient if the final orifice is replaced with a straight piece of pipe. The second manometer in the meter box can then be used for the instack orifice and the dry gas meter pressure is then ambient or barometric.

(b) In the configuration described, the orifice meter assembly will be out of the stack flow for points located about seven inches or less from the near stack wall. The port should be insulated and/or heated to as near the stack temperature as possible. since it is assumed that the orifice meter temperature is equal to the stack temperature. A general guideline is that the two temperatures expressed in absolute ($^{\circ}\text{R}$) terms must be within about 20 percent of each other, assuming all other variables are measured with no error, to achieve ± 10 percent of isokinetic conditions.

(c) In many cases, the pressure differential taps can become plugged with condensate. This is usually indicated by a reduced pressure differential, a sluggish variation, or no response to flow control valve changes. Sampling should be interrupted and the manometer lines should be momentarily backflushed. Backflushing during sampling is undesirable since the air then becomes part of the gas sampled.

(d) In cases where the moisture content is very high and high sampling rates are required, it may be necessary to add an additional fine metering valve to the pumping system prior to the coarse valve commonly used in EPA-5 systems. Additional metering control is necessary due to the small fraction of dry gas flow that remains after condensation. Since the flow is very small, the coarse valve by-pass combination does not allow an accurate regulation of vacuum. This extra needle valve can be conveniently installed at the quick-correct fitting for the umbilical cord sample hose.

(e) During sampling, the operating equation may be solved by using a slide rule, calculator, graph, etc., since it is simply a constant multiplication. The EPA-5 operating nomograph can also be used if desired. In this case, all scales except the Δh and Δp are ignored. The factor $\left[782.502 \left(\frac{C_p}{K_m} \right)^2 D_n^4 \right]$ is aligned on the Δh scale with 1.0 on the Δp scale to set the pivot point.

After a sampling run has been completed and the train has been transported to the cleanup area, the orifice-nozzle assembly should be removed from the probe for cleanup purposes. All openings on the assembly should be plugged and the outer surface should be thoroughly cleaned prior to recovery of the sample fraction. The meter body can then be separated at the orifice plate and rinsing with the appropriate solvent and brushing can be performed on the two halves of the assembly.

APPENDIX G
SAMPLING TRAIN CALIBRATION DATA
INCLUDES

- G-1. SAMPLING ORIFICE CALIBRATIONS
 - a. Granulator Testing
 - b. Synthesis Tower Testing
- G-2. NOZZLE MEASUREMENTS
 - a. Granulator Testing
 - b. Synthesis Tower Testing
- G-3. PITOT TUBE CALIBRATIONS
 - a. Granulator Testing
 - b. Synthesis Tower

APPENDIX G-1a
SAMPLING ORIFICE CALIBRATIONS
FOR
GRANULATOR TESTING

DATE 12/27/78

ORIFICE NO D6 NAME C EKROTH

BAROMETRIC PRESSURE 30.03 IN HG

INLET, INLET PARTICLE SIZING
DRY GAS METER NO 6

ORIFICE	GAS VOLUME		TEMPERATURE						
MANOMETER SETTING IN WATER	WET TEST METER CU FT	DRY GAS METER CU FT	WET TEST METER F	DRY GAS INLET F	METER OUTLET F	AVERAGE F	TIME MIN	RATIO	DHO
0.5	5.0	5.03	70.4	73.0	74.0	73.5	12.7	1.00	1.80
1.0	5.0	5.03	70.5	72.0	72.0	72.0	9.2	0.99	1.89
2.0	10.0	10.05	70.2	74.0	75.0	74.5	13.0	1.00	1.88
3.0	10.0	10.08	70.0	75.0	75.0	75.0	10.8	0.99	1.94

AVERAGE 1.00 1.88

DATE 1/03/79

ORIFICE NO D5 NAME T ELODUC

BAROMETRIC PRESSURE 29.70 IN HG

OUTLET

DRY GAS METER NO 5

ORIFICE	GAS VOLUME		TEMPERATURE						
MANOMETER SETTING IN WATER	WET TEST METER CU FT	DRY GAS METER CU FT	WET TEST METER F	DRY GAS METER INLET F	OUTLET F	AVERAGE F	TIME MIN	RATIO	CHO

0.5	5.0	4.96	70.0	69.0	69.0	69.0	11.9	1.00	1.61
1.0	5.0	4.96	70.0	72.0	72.0	72.0	8.6	1.01	1.67
2.0	10.0	9.92	70.0	71.0	72.0	71.5	12.3	1.01	1.71
3.0	10.0	9.93	70.0	71.0	72.0	71.5	10.1	1.00	1.73

AVERAGE								1.01	1.68

APPENDIX G-1b

SAMPLING ORIFICE CALIBRATIONS
FOR
SYNTHESIS TOWER TESTING

PRETEST CALIBRATION

Orifice = 0.2500" $P_{bar} = 30.59$ 1/12/79

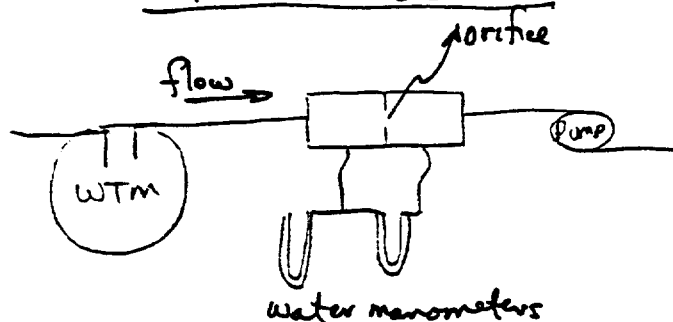
CF_w	time, min.	$\Delta H, "H_2O$	orifice static press. $"H_2O$	$T_w, ^\circ F$	$CF_w/time$	K_m
1.00 ft ³	1.03	1.0	-0.15	71.5	0.97	1.25
1.00 "	0.58	3.0	-0.35	71.5	1.72	1.28
1.00 "	0.45	5.0	-0.60	71.4	2.22	1.28
AUG						1.27

POST TEST CALIBRATION

Orifice = 0.2500" $P_{bar} = 30.06$ 3/20/79

CF_w	time, min.	$\Delta H, "H_2O$	orifice static press. $"H_2O$	$T_w, ^\circ F$	$CF_w/time$	K_m
2.00 ft ³	2.39	0.70	-0.3	72.5	0.84	1.28
2.00 "	1.85	1.0	-0.45	72.5	1.08	1.38
2.00 "	1.76	1.3	-0.4	72.5	1.14	1.28
2.00 "	1.60	1.6	-0.5	72.5	1.25	1.27
AUG						1.30

CALIBRATION SET UP



CALIBRATION CALCULATIONS (see following page)

$$K_m = \frac{CF_w}{time} \sqrt{\frac{(P_{bar} + \frac{static}{13.6}) M_m}{\Delta H (T_w + 460)}}$$

G-6

T_w = temp wettest meter
 M_m = mol. wt air = 28.83 lb/lb-mole
 static = orifice static pressure

Instack Orifice Calculations

For isokinetic flow $V_n = V_s$

where $V_n \equiv$ nozzle velocity, fpm

$V_s \equiv$ stack velocity, fpm

$$V_n = \frac{Q_n}{A_n}$$

where $Q_n \equiv$ volumetric flow rate through nozzle, cfm

$A_n \equiv$ area of nozzle, ft²

assuming that $Q_n = Q_m$

where $Q_m \equiv$ volumetric flow rate through instack orifice, cfm

$$V_n = \frac{Q_m}{A_n}$$

$$Q_m = k_m \sqrt{\frac{\Delta h T_m}{P_m M_m}}$$

where $k_m \equiv$ meter constant

$T_m \equiv$ meter temperature, °R

$P_m \equiv$ meter pressure, "Hg

$M_m \equiv$ meter gas molecular weight, lb/lb-mole

$\Delta h \equiv$ pressure drop across orifice, "H₂O

$$\text{and } A_n = \frac{0.7854 d_n^2}{144 \text{ in}^2/\text{ft}^2}$$

where $d_n \equiv$ nozzle diameter, inches

$$V_s = 5128.8 C_p \sqrt{\frac{T_s \Delta P}{P_s M_s}}$$

where C_p = pitot tube coefficient, dimensionless

T_s = stack temperature, $^{\circ}R$

ΔP = velocity pressure, $"H_2O$

P_s = stack pressure, $"Hg$

M_s = molecular weight of stack gas, lb/lb-mole

$V_n = V_s$ therefore

$$k_m \sqrt{\frac{\Delta h T_m}{P_m \mu_m}} \times \frac{144}{0.7854 d_n^2} = 5128.8 C_p \sqrt{\frac{T_s \Delta P}{P_s M_s}}$$

for in-stack orifice assume $T_m = T_s$, $P_m = P_s$ and $\mu_m = \mu_s$

$$\Delta h = 7825 \left[\frac{C_p}{k_m} \right]^2 d_n^{2/3} \Delta P$$

For CFI Donaldsonville

$$d_n = 0.5"$$

$$C_p = 0.837$$

$$k_m = 1.27$$

$$\Delta h = 21.24 \Delta P$$

APPENDIX G-2a

NOZZLE MEASUREMENTS
FOR
GRANULATOR TOWER TESTING

ELFRED MACHINE COMPANY
150 JORDAN LANE
WETHERSFIELD, CONN. 06092
DEC 29 1970

DATE 1/10/79

INSPECTION REPORT

ELFRED MACHINE COMPANY

CUSTOMER TRC THE RESEARCH CORP.

CUSTOMER P.O. NO. 3790

S.O. NUMBER

PURCHASED FROM ELFRED P.O. NO

PART NO.

NO. PIECES ORDERED 1 NO. PIECES RECEIVED 1

PART NAME

PART NAME ST/STL NOZZLE 1/8

TOOL NO.

TOOL NAME

INLET PARTICLE SIZING

[illegible]

1st PIECE INSPECTION

PARTIAL INSPECTION

COMPLETE INSPECTION

ACCEPTED

G-11

REJECTED

INSPECTOR

CTOP
ELFRED MACHINE COMPANY
185 JORDAN LAKE
WETHERSFIELD, CONN. 06109
IAN 10 ENFO

IAN 10 ENFO

④

ELFRED MACHINE COMPANY

CUSTOMER P.O. NO. 9964

PURCHASED FROM ELFRED P.O. NO.

NO. PIECES ORDERED 1 NO. PIECES RECEIVED 1

PART NAME ST/STL NOZZLE

TOOL NAME _____ OUTLET

WETTER MACHINE COMPANY
185 JORDAN LANE
WETHERFIELD, CONN. 06109

APPENDIX G-2b
NOZZLE MEASUREMENTS
FOR
SYNTHESIS TOWER TESTING

INSPECTION REPORT

ELFRED MACHINE COMPANY

CUSTOMER TRC - THE RESEARCH CORP CUSTOMER P.O. NO. 9767
S.O. NUMBER — PURCHASED FROM ELFRED P.O. NO. —
PART NO. 4-4 NO. PIECES ORDERED 1 NO. PIECES RECEIVED 1
TOOL NO. SET # 4 PART NAME ST/STL NO 22 LE
TOOL NAME SOLUTION TOWER

[illegible]

1st PIECE INSPECTION

☐

PARTIAL INSPECTION

☐

COMPLETE INSPECTION

☒

ACCEPTED
G-14

☒

REJECTED

110

INSPECTOR
155 JUNE
WETHERSFIELD
NO

PAPER MACHINE COMPANY
 155 JOURNAL LANE
 WETHERSFIELD, CONN. 06109
 NOV 30 1975

APPENDIX G-3a

PITOT TUBE CALIBRATIONS
FOR
GRANULATOR TESTING

THE RESEARCH CORPORATION OF NEW ENGLAND

CLIENT EPA

CHARGE NO. 92988

TESTER T BOLDUC

CALIBRATION DATA
TYPE S PILOT TUBE

DATE 01/10/79 TEMP.(F)= 50.0 BARO.PRES.(IN.HG)= 0.00

STANDARD PITOT NO. CE-1

SERIAL NO. 44 INLET

NO. OF SCREENS	STANDARD DELTA P " W.C.	TYPE S DELTA P " W.C.	COEFFICIENT + CP(S)
1	0.051	0.076	0.901
2	0.100	0.145	0.922
3	0.200	0.285	0.929
4	0.310	0.440	0.931
5	0.420	0.599	0.929
6	0.500	0.710	0.931
7	0.610	0.865	0.931
			AVERAGE CP(S) 0.925

REVERSE (S) PITOT AND RERUN TEST

1	0.055	0.081	0.916
2	0.103	0.153	0.912
3	0.201	0.295	0.917
4	0.300	0.435	0.922
5	0.395	0.570	0.924
6	0.505	0.725	0.926
7	0.595	0.857	0.925
			AVERAGE CP(S) 0.920

+ CP(S)=0.9950RT(DELTA P(STANDARD) / DELTA P(S))

F800 EXIT

THE RESEARCH CORPORATION OF NEW ENGLAND

CLIENT EPA

CHARGE NO. 82988

TESTER T BOLDUC

CALIBRATION DATA

TYPE S PILOT TUBE

DATE 01/10/79

TEMP.(F) = 50.0

BARO.PRES.(IN.HG) = 30.00

STANDARD PILOT NO. CE-1

SERIAL NO. 35 INLET PARTICLE SIZING

NO. OF SCREENS	STANDARD DELTA P " W.C.	TYPE S DELTA P " W.C.	COEFFICIENT + CP(S)
1	0.100	0.158	0.738
2	0.205	0.300	0.818
3	0.400	0.560	0.837
4	0.595	0.825	0.841
5	0.795	1.105	0.840
6	0.990	1.375	0.840
7	1.200	1.670	0.839

AVERAGE CP(S) 0.829

REVERSE (S) PILOT AND RERUN TEST

1	0.105	0.165	0.790
2	0.200	0.310	0.795
3	0.405	0.600	0.813
4	0.600	0.875	0.820
5	0.800	1.155	0.824
6	1.010	1.450	0.826
7	1.205	1.750	0.822

AVERAGE CP(S) 0.813

+ CP(S) = 0.99 SQRT(DELTA P(STANDARD) / DELTA P(S))

F\$00 EXIT

THE RESEARCH CORPORATION OF NEW ENGLAND

CLIENT EPA

CHARGE NO. 82938

TESTER BOLDUC

CALIBRATION DATA

TYPE S PILOT TUBE

DATE 01/10/79

TEMP.(F) = 50.0

BARO.PRES.(IN.HG) = 0.00

STANDARD PILOT NO. CE-1

SERIAL NO. 38 OUTLET

* NO. OF SCREENS	STANDARD DELTA P '' W.C.	TYPE S DELTA P '' W.C.	COEFFICIENT + CP(S)
* 1	0.050	0.078	0.793
* 2	0.100	0.148	0.814
* 3	0.195	0.275	0.834
* 4	0.297	0.420	0.833
* 5	0.405	0.570	0.834
* 6	0.505	0.705	0.838
* 7	0.595	0.830	0.838

AVERAGE CP(S) 0.826

REVERSE (S) PILOT AND REVERSE TEST

1	0.050	0.075	0.808
2	0.105	0.148	0.834
3	0.195	0.270	0.841
4	0.295	0.410	0.840
5	0.400	0.550	0.844
6	0.495	0.695	0.835
7	0.635	0.845	0.838

AVERAGE CP(S) 0.834

CP(S) = 0.99 SQR(DLTA P(STANDARD) / DLTA P(S))

FSOC EXIT

APPENDIX G-3B
PITOT TUBE CALIBRATION
FOR
SYNTHESIS TOWER

THE RESEARCH CORPORATION OF NEW ENGLAND

CLIENT SPA

CHARGE NO. 82988

TESTER T BOLDUC

CALIBRATION DATA
TYPE S PILOT TUBE

DATE 01/10/79

TEMP.(F)= 50.0

BARO.PRES.(IN.HG)=30.00

STANDARD PITOT NO. CE-1

SERIAL NO. 50 SOLUTION TOWER

NO. OF SCREENS	STANDARD DELTA P ** W.C.	TYPE S DELTA P ** W.C.	COEFFICIENT +CP(S)
1	0.102	0.148	0.822
2	0.200	0.280	0.837
3	0.395	0.550	0.839
4	0.600	0.830	0.842
5	0.792	1.095	0.842
6	1.005	1.395	0.840
7	1.200	1.680	0.837

AVERAGE CP(S) 0.837

REVERSE (S) PITOT AND RERUN TEST

1	0.100	0.145	0.822
2	0.205	0.290	0.832
3	0.402	0.555	0.843
4	0.606	0.825	0.848
5	0.800	1.085	0.850
6	0.995	1.365	0.845
7	1.200	1.645	0.846

AVERAGE CP(S) 0.841

+ CP(S)=0.79 SQRT(DELTA P(STANDARD) / DELTA P(S))

FS00 EXIT

APPENDIX H

DAILY SAMPLING LOGS

H-1 Sampling Task Logs

H-2 Field Laboratory Logs

APPENDIX H-1

SAMPLING TASK LOGS

SAMPLING TASK LOG

Plant CF Industries Inc

Date

Plant Location Dona Idemville LA
Recorded by E. Pearson

Recorded by EAPearce

[illegible]

SAMPLING TASK LOG

Plant CF Industries Inc
Date 1-18-75

Plant Location Douglasville LA
Recorded by SAFARI

Comments	Run	Sampling Location (Port)	Pollutant	Clock Time Began	Elapsed Time (min)	Sample Nos.
B Granulator Scrubber Inlet						
Start 1st Traverse	3	Scrubber Inlet	Urea Part.	0947		
pitot plugged					30	
continue				1021		
probe plugged					0	
continue				1030		
nozzle plugged					17	
continue				1055		
end 1st traverse				1102	7	
Start 2nd Traverse	3			1107		
probe plugged					1	
continue				1112		
complete 2nd traverse				1210	58	
Start Cascade Impactor	1	Scrubber Inlet	Part.	1328	15	
Procedure Determination	1	Scrubber Inlet	moisture	1615	34	
Urea Synthesis Tower	1	Syn. Tower	Urea/Ammine	1540	15	
(collected 4)						

SAMPLING TASK LOG

Plant CF Industries Inc
Date 1-19-77

Plant Location Dorchesterville LA
Recorded by Effigore

[illegible]

Plant CFF Industries, Inc.
Date 1-17-79 / 1-18-79

Plant Location Dardsboro LA
Recorded by EFH

[illegible]

SAMPLING TASK LOG

plant CF Industries

Date 1-17-75 / 1-18-75

Plant Location

Recorded by E. A. Deane

Donchisonville LA

[illegible]

SAMPLING TASK LOG

Plant (CF Industries Inc

Date 1-17-79 / 1-18-79 ✓ 1-19-79

Plant Location
Recorded by _____

A

FA(2000)

Comments	Run	Sampling location (Port)	Pollutant	Clock Time Began	Elapsed Time (min)	Sample Nos.
Plume Opacity Observations						
1-17-79 Plume Opacity obs made every 15 seconds cloudy to overcast		Top of Syn. Tower	Opacity	1202	81	
Plume Opacity overcast				1550	47	
1-18-79 Plume Opacity overcast				1244	60	
1-19-79 Plume Opacity overcast				0953	120	

SAMPLING TASK LOG

Plant CF INDUSTRIES INC
 Date 1-17-79 / 1-18-79 / 1-19-79

Plant Location Durham, NC
 Recorded by LA

Comments	Run	Sampling location (Port)	Pollutant	Clock Time Began	Elapsed Time (min)	Sample Nos.
→ B Granulator Scrubber Purge Drops						
1-17-79 measurements every 5 minutes		scrubber	pressure	1207	1257	50
				1321	1416	55
				1549	1753	64
1-18-79 measurements every 5 minutes		scrubber	pressure	0946	1041	55
				1122	1217	55
→ Ambient Temp, Pressure, RH						
1-17-79 measurements (10 measurements)		at B Granulator	T, P, RH	1230	1815	
1-18-79 (12 measurements)		at B Granulator	T, P, RH	0945	1540	
1-19-79 (8 measurements)		at B Granulator	T, P, RH	1000	1230	

SAMPLING TASK LOG

Plant Cl Industries Inc
Date 1-17-79 / 1-18-79

Plant Location
Recorded by

Donaldsonville LA

[illegible]

APPENDIX H-2

FIELD LABORATORY LOGS

From Page No. _____			Probe wash tare + wash, gm	H ₂ O imp. tare + H ₂ O + sample H ₂ O tare + sample, gm	H ₂ SO ₄ imp. tare + H ₂ SO ₄ + sample H ₂ SO ₄ tare + sample, gm	H ₂ SO ₄ imp. tare + H ₂ SO ₄ + sample + wash H ₂ SO ₄ tare + sample + wash, gm	total sample vol. ml	S Gel + tare wt + H ₂ O	H ₂ O imp tare + H ₂ O + sample + wash, gm
sample container #	sample container tare wt, gm	Volume H ₂ O or H ₂ SO ₄ or wt S Gel							
B-W-I	328.27	200.0							
B-W-O	326.08	200.0							
B-A-I	326.54	200.0			528.94	617.25			
B-A-O	325.15	200.0	8/A		530.45	647.87			
1-S-O	242.45	200.0						264.48	
1-S-I	244.54	200.0						256.43	
1-PW-I	325.43	200.0	712.44	Rwc 604.00 712.44					
1-PW-O	326.52		597.67						
1-A-I	327.07	204.8 gm			526.35	677.26			677.26
1-A-O	325.08	204.8 gm			527.79	630.99			630.99
1-W-O	327.28	200ml		637.64					691.62
1-W-I	327.70	200ml		604.00					691.41
2-S-O	243.34	200 gm						263.89	
2-S-I	242.68	200 gm						256.52	
2-PW-O	327.24		570.92						
2-PW-I	326.28		746.20						
2-A-O	327.26	204.8 gm			530.98	658.97			
2-A-I	327.40	204.8 gm			518.87	719.71			
2-W-O	328.18	200ml		641.36					749.50
2-W-I	326.28 329.09	200ml		746.20 599.06					687.74
3-S-O	242.88	200 gm						261.74	
3-S-I	245.33	200 gm						258.88	
3-PW-O	325.85		618.36						
3-PW-I	326.68		663.00						
3-A-O	326.23	204.8 gm			531.72	704.70			
3-A-I	327.83	204.8 gm			475.47	701.63			
3-W-O	325.29	200ml		534.86					649.10
3-W-I	326.25	200ml		596.44					722.35
3-PW-I(2)			631.90						

100ml H₂SO₄ (1N) = 102.4 gm

To Page o.

Witnessed & Understood by me,

Date

Invented by

Date

H-12

Recorded by

TITLE CF Industries Dandeeville La

Project No. _____

Book No. _____

From Page No. _____

Sample #	tare weight	Vol. of H ₂ O, H ₂ SO ₄ or SG	Probe Wash tare + Wash (gm)	H ₂ O Imp tare, H ₂ O + Sample	H ₂ SO ₄ Imp tare, H ₂ SO ₄ and sample	H ₂ SO ₄ Imp tare, H ₂ SO ₄ sample, + Wash	Total sample Vol.	SG. tare + H ₂ O	H ₂ O Imp tare, H ₂ O Sample + Wash
4-A									
4s-A	498.84	300ml			879.18	1132.76			
4s-W	501.89	300ml		1142.54					1341.2
4s-PW	328.11	0	531.65						
4s-S	243.19	200gm						252.75	
5s-A	498.64	300ml			875.23	1092.83			
5s-W	498.97	300ml		1279.5					
5s-PW	327.88	0	522.85						
5s-S	244.22	200gm						249.21	
5s-W(2)	327.74	0							this sample container contains only impurities wash. 542.35
6s-A	498.04				883.65	1127.00			
6s-W	501.29			1226.8					
6s-PW	328.56		542.25						
6s-S	242.81							251.33	
6s-W(2)	328.24								559.64 Improv Data C
H ₂ O Test Scrubber Inlet	SG. tare 244.14	SG. tare + H ₂ O 253.1	Vol H ₂ O Imp collected 6ml	Vol H ₂ O in S.C. 8.96	Total H ₂ O collected 14.96				

Witnessed & Understood by me,

Date

Invented by

Date

H-13

Recorded by

To Page No.

Silica gel (s) Inlet (T)
 acc. (A) Outlet (O)
 water (w)

1-S-I

Serialist
 (container for
 + charge
 H₂O) gm

initiallet
 (container
 tare) gm

charge
 wt, gm

Scrubber inlet/outlet tests; sample
 weight gain data.

weight
 gain,
 gm

Anterior # key

EFFICIENCY® LINE NO. 636

	Container	2	3	4	5	6	7	8	9
1									
2	1-S-I	256.43	244.54*	—		11.89			
3									
4	1-A-I	526.35	327.07	204.8		-5.52			
5									
6	1-W-I	604.00	327.70	200		76.3			
7									
8									
9									
10	1-S-O	264.48	242.45*	—		22.03			
11									
12	1-A-O	527.79	325.08	204.8		-2.09			
13									
14	1-W-O	634.64	327.28	200		107.36			
15									
16									
17									
18	2-S-I	256.52	242.68*	—		13.84			
19									
20	2-A-I	518.87	327.40	204.8		-13.33			
21									
22	2-W-I	589.00	329.09	200		59.91			
23									
24									
25									
26	2-S-O	263.89	243.34*	—		20.55			
27									
28	2-A-O	530.98	327.26	204.8		-1.08			
29									
30	2-W-O	641.36	328.18	200		113.18			
31									
32									
33									
34	3-S-I	258.88	245.33*	—		13.55			
35									
36	3-A-I	475.47	327.83	204.8		-57.16			
37									
38	3-W-I	596.44	326.25	200		70.19***			
39									
40									
41									
42	3-S-O	261.74	242.88*	—		18.86			
43									
44	3-A-O	531.72	326.23	204.8		0.69			
45									
46	3-W-O	534.86	325.29	100**		109.57			
47									
48									
49									
50									

total

total

total

total

total

total

H-14

* includes 200 gm silica gel charge

** no H₂O in impinger 2 for Test 3-O

*** H₂SO₄ impinger sample sucked back to H₂O impinger

APPENDIX I

PROCESS OPERATION DATA

This entire Appendix is confidential. See Item 9 - Confidential Addendum, Contact Eric Noble, EPA (919) 541-5213

APPENDIX J
TRC LABORATORY ANALYTICAL DATA

INCLUDES:

- J-1 Analytical Log w/ Discussion
- J-2 Sample and Analysis Results Logs
- J-3 Laboratory Data Sheets

APPENDIX J-1
ANALYTICAL LOG WITH DISCUSSION

Upon arrival at ^{the} ACF Industries laboratory, samples of the five water containers transported from TRC were analyzed for formaldehyde. Results were at the limit of detection, $0.05 \mu\text{g}/\text{ml}$.

Next, a blank analysis was run on the sampling train pretest wash. This is described in detail in APPENDIX L.

Scrubber inlet and outlet probe wash and impinger volumes were measured in a graduated cylinder before the analyses were begun. Within 24 hours of collection, all the samples were analyzed for ammonia by direct nesslerization. Then they were distilled and digested for the distilled ammonia and urea analyses (by nesslerization) that were subsequently performed the following week at ^{the} TRC laboratory. A portion of the sample was kept on ice and analyzed for formaldehyde by the chromatotropic sulfuric acid colorimetric method within twelve days.

The scrubber water solutions were analyzed for suspended solids by passing 100 to 200 ml. of sample, at the temperature of collection, through a preweighed gooch crucible containing a glass fiber filter.

The two solid samples, Feed Melt to Granulator and Granulator Unscreened Product, were prepared immediately before analysis by dissolving an amount weighed to 0.1 mg. in distilled deionized water and then diluting to 100.0 ml in a volumetric flask. The results were reported as percentage.

The Feed Melt sample was a solid at room temperature. A saturated solution of it in water was prepared, and the pH was measured.

1. Direct Ammonia

An aliquot of sample was diluted to 50ml. Then one ml. of nessler reagent was added and the color read on a spectrophotometer at 405 nm. The blank probe wash samples, the outlet scrubber water solutions, and ^{the} solid samples turned cloudy upon the addition of the nessler reagent. The formaldehyde concentration in the blank probe washes was higher than in the impinger blanks which were at the limit of detection ($0.05 \mu\text{g/ml}$). This has been known to cause turbidity like that observed. The interference in the outlet scrubber waters could be due to the high urea concentration - about 50%. The solid samples were prepared as an approximately 1% solution. The limit of detection for this method is $\sim 0.06 \mu\text{g/ml}$.

2. Distilled Ammonia

The blank inlet probe wash and inlet scrubber waters for tests 1 and 2 turned cloudy with nesslerization. The calculated results were high due to this turbidity.

The distillation of 25 ml of sample into a final volume of 250ml. resulted in a ten fold decrease in sensitivity referred to the original sample solution; that is, $\sim 0.6 \mu\text{g/ml}$.

3. Urea

There were no problems with interferences or turbidity for these samples analyzed by the kjeldahl urea method. The limit of detection was $\sim 1 \mu\text{g/ml}$ for this method.

4. Formaldehyde

The solid samples, dissolved in water, were analyzed four to six times. The results are an average of three values run on a particular dissolved sample. The results agree within $\pm 0.1\%$, about 20% of the total.

The sample was diluted from 25 to 100 fold, and an aliquot of this dilution of one to four ml was analyzed so that equal amounts of sample were reacted with the color reagent. The 50 and 100 fold dilutions agreed within about 5%, whereas the 25 fold dilution differed from these values by about $\pm 20-25\%$. These variations indicate that further study should be done on the proper preparation and analysis methodology of solid urea samples for formaldehyde content.

The following pages contain a summary of the analytical laboratory results along with the raw laboratory data.

505
561000 L

UREA

"clean up Act" notebook

A girl who still has a tendency to slip on p. 7 "New-Africa" instead of "New-Africa" and back.

WATSON REVIEW

Not Analyzed For CH₂O

[illegible]

APPENDIX 5

For Appendix 5

7 Nov 74
SAL
DISTRIBUTION

ANTHRA- DIRECT NESTLEIZATION

Sample ID	Sample Volume (ml)	Alcohol Volume (ml)	Alcohol Diluted to (ml)	Dilution Factor D ₁	Added Dilution D ₂	Aldehyde Concentration (mg/ml)	X ₀ Black Count (mg/ml)	X ₀ - X ₆ net count (mg/ml)	X ₆ (X ₁ - X ₆) (mg/ml)	X ₆ (mg/ml)	X ₆ (mg/ml)	X ₆ (mg/ml)
1-S-1	-	2	250	125	-	.022	72/50	72/50	180	-	-	-
1-S-2	-	2	250	125	-	.000	-	-	-	-	-	-
1-A-1	880	1	100	100	-	.051	180/50	180/50	317	108	108	410
1-A-2	781	1	100	100	-	.044	72/50	72/50	406	342	342	410
1-A-3	306	2	50	25	10	.055	157/50	157/50	785	240	240	450
1-A-4	640	2	50	25	10	.038	58/50	58/50	340	216	216	450
2-S-1	-	2	250	125	-	.055	175/50	175/50	438	-	-	-
2-S-2	-	2	250	125	-	.000	-	-	-	-	-	-
2-A-1	353	2	50	25	10	.062	57.5/50	57.5/50	131	50.1	50.1	441
2-A-2	779	2	50	25	10	.050	208/50	208/50	502.5	391	391	441
2-A-3	323	2	50	25	10	.045	208/50	208/50	1000	332	332	654
2-A-4	665	2	50	25	10	.035	111/50	111/50	555	369	369	654
3-S-1	-	2	250	125	-	.000	110/50	110/50	23.2	-	-	-
3-S-2	-	2	250	125	-	.000	-	-	-	-	-	-
3-A-1	374	2	50	25	10	.016	9/50	9/50	71.5	8.4	8.4	514
3-A-2	1035	2	50	25	10	.019	156/50	156/50	430	509	509	514
3-A-3	378	2	50	25	10	.065	106/50	106/50	718	371	371	387
3-A-4	116	2	50	25	10	.022	46/50	46/50	200	123	123	387
4-S-1	634	2	250	125	25	.030	110/50	110/50	55	14.9	14.9	3771
4-S-2	815	2	250	125	25	.005	148/50	148/50	40250	45239	45239	3771
5-S-1	462	2	250	125	25	.035	113/50	113/50	40000	47600	47600	3771
5-S-2	1155	2	250	125	25	.000	-	-	-	-	-	-
6-S-1	502	2	250	125	25	.000	113/50	113/50	50938	59448	59448	3771
6-S-2	1135	2	250	125	25	.055	-	-	-	-	-	-

✓ oxygen with ethanol
Process samples were cloudy
new cap volume present, parameters due to high concentration of BSA plus water, and directly compared the volume. For calculated from sample weights.
A 10 ml and why cap volume is 50 and 100, according to date on 10.7 of chemical assay with BSA

APPENDIX J-2
SAMPLE AND ANALYSIS RESULTS LOG

Report of Chemical Analysis of Non-Routine Samples

Client: EPA CF Industries
Contract No: 82788-20

Laboratory No: 48-79 (1)
Date Received: 1/23/79
Date Reported: _____

Reviewed by: _____
Report to: RUC / WAW

Type Sample: Filter Fuel Oil _____ Sediment _____ Impinger _____ Other _____

Sample Number	Location	Analysis No. 1 <u>Direct wt%</u>	Analysis No. 2 <u>Distillable Wt%</u>	Analysis No. 3 <u>Urease</u>	Analysis No. 4 <u>Potential Volatile</u>	Analysis No. 6 <u>Suspended Solids</u>	pH <u>FTV</u>
B-W-I		0.20 mg/ml	1.4 mg/ml *** 0.60	6.94 mg/ml 4.24	2.05 mg/ml		
B-W-O		0.12 mg/ml	1.32 mg/ml 0.52	4.64 mg/ml 2.82	2.05 mg/ml		
B-A-I		cloudy 1.00 mg/ml	1.20 mg/ml 0.50	4.44 mg/ml 3.04	2.05 mg/ml		
B-A-O		cloudy 1.16 mg/ml	1.40 mg/ml 0.60	3.79 mg/ml 1.94	2.05 mg/ml		
B-PW-I		cloudy	7.3 mg/ml cloudy 6.5	10.24 mg/ml 8.39	0.075 mg/ml		
B-PW-O		cloudy	2.45 mg/ml 1.65	10.3 mg/ml 8.47	0.108 mg/ml		
I-S-I		180 mg/ml	15.4 mg/ml 13.8	15.09 mg/ml	26.0 mg/ml	117 mg/l	
I-S-O		very cloudy	14.50 mg/ml 14.130	516.300 mg/ml	2.04 mg/ml	3.9 mg/l	
I-A-I		108 mg	116 mg 110	27.7 mg 15.0	0.486 mg		
I-A-O		232 mg	233 mg 232	6.87 mg 4.64	0.413 mg		
I-W-I		302 mg	790 mg 774 mg *	23,932 mg 23,905	2.05 mg		
I-W-O		218 mg	208 mg 206	70.6 mg 65.9	2.11 mg		
* upper value is not blank corrected							
** bottom value is blank corrected							

Analyzed by: DPH / DPH
Form CI. 0012

Client: FPA, CF Industries
 Contract No: 82988-20

Laboratory No: 48-79(1)
 Date Received: 1/22/59
 Date Reported: _____

Reviewed by: _____
 Report to: MWC/WAW

Type Sample: Filter _____ Fuel Oil _____ Sediment _____ Impinger _____ Other _____

Sample Number	Location	Analysis No. 1 Direct wt%	Analysis No. 2 Distillable mg	Analysis No. 3 Area	Analysis No. 4 Dissolved mg	Analysis No. 6 Suspended mg	PH TLV
2-S-I		43.8 mg/mg	68 mg/mg 52	120.0 mg/mg 82.96	20.0 mg/mg	0.88 mg/l	
2-S-O		very cloudy	12,800 mg/mg 12,800	525,100 mg/mg 525,100	0.075 mg/mg	4.6 mg/l	
2-A-I		50.1 mg	57.3 mg 51.2	41.1 mg 26.97	0.0382 mg		
2-A-O		32.3 mg	244 mg 243	9.73 mg 7.29	0.0363 mg		
2-W-I		39 mg	902 mg 890	26,090 mg 26,063	1.507 mg		
2-W-O		37 mg	185 mg 183	70.7 mg 65.8	1.136 mg		
3-S-I		23.2 mg/mg	47 mg/mg 31	130.6 mg/mg 93.5	13.5 mg/mg	3.6 mg/l	
3-S-O		very cloudy	13,100 mg/mg 13,100	538,300 mg/mg 538,300	0.05 mg/mg	4.8 mg/l	
3-A-I		3.19 mg	23.7 mg 17.8	53.97 mg 40.47	0.091 mg		
3-A-O		26.2 mg	264 mg 263	7.88 mg 5.17	0.0458 mg		
3-W-I		49.8 mg	943 mg 926	26,253 mg 26,218	2.996 mg		
3-W-O		125 mg	127 mg 125	61.8 mg 57.1	1.25 mg		

Analyzed by: MWC
 Form CL-0012

Laboratory No: 48-79(1)

Date Received: 1/23/79

Date Reported: 11/11/2014

RWC / WAW

Type Sample: _____
Type Sample: Filter _____
Fuel Oil _____
Sediment _____
Impinger _____
Other _____

[illegible]

Approved by: M. J. A.

APPENDIX J-3
LABORATORY DATA SHEETS

From Page No. _____

1/16/79

CF. Lab

Prepare Chromotropic Acid solution
0.10g in 10.0ml H_2O (Water Canister #4)

weighing paper assayed, tare to 0.0000g weight 0.10g

Stock Formaldehyde Solution 4.4703g of formaldehyde solution / 100ml H_2O
(Prepared at TRC 1/9/79 see statute 1000000)

Dilute 1.0 ml $\Rightarrow$ 100 ml $\Rightarrow$ 10 μ g/ml Working Solution

Standard

ml Working Solution	total μ g formaldehyde	Abs	Sample
0	0	.012	
0.1	1	.068	
0.3	3	.2160	
0.5	5	.252	
0.7	7	.348	
1.0	10	.490	
2.0	20	.82 (15.5 μ g)	zero OK

* Brought up to 4.0ml with TRC #4 H_2O (all stds)

Wise TRC Spcl Container ml Sample

ml	4ml	Abs
1		.003
2		.005
3		.012
4		.012
5		.005

$H_2O + H_2SO_4$ on a

500ml Plastic Bottle (just filled) 4.0ml

@ 16:10 hour bottle filled with

TRC #4 H_2O - Sample repeated within 3 minutes

.000 use to zero Spec 21
.008 (zero 0.4)

In order to zero the Spec 21 with a solution that is clear

Witnessed & Understood by me.

Date

Invented by

Date

To Page

Prepared by

From Page No.

ie, not colored by any trace contained, I made up a
 Blank with 4.1 ml #4 TRC H_2O and 6 ml conc H_2SO_4 .
 Use the to zero Spec 21 @ 580 nm (no Chromogenic Acid)

Prepare 1.0 N H_2SO_4 from TRC H_2O #2

Note ① - The first sample of #4 water which was also used as the
 standard 0 μg formaldehyde - turned yellow (0.28 Abs)
 the pretest on p. 1 did not turn yellow
 - why that time?

~ 15:10 PM - Skid being DW #5 in Lab

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

To Page No.

Project No. 82788-20

4

Book No. 11-4 TITLE CE Ord. Donnellville, LA - LRB

From Page No. _____

1/17/79 GA 8:45 AM

- Clean out - Steam out the 6 distillation units.

Prepare Distilled Standards 1000 µg/ester - Dilute 10.0 ml $\Rightarrow$ 100 ml = 100 µg/ml

Working Solution		
total µg in 50.0 ml	total µg NH ₃ in 250.0 ml	ml W.S. distilled into 250.0 ml flask
0 Blank	0 NH ₃	0
20	100	1.0
40	200	2.0
100	500	5.0
160	800	8.0
200	1000 µg NH ₃	10.0
200	1000 - Digestion then Distilled	

Do 0 Blank residue for distillation, add 10.0 ml of 100 µg/ml to 250 ml flask - Digest then Distill - Do compare to 200 µg NH₃ distillation.

M.D. of

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TITLE CP 2nd, Donaldsonville, La.

Project No. 82758-20

Book No. 4

From Page No. —	Sample Distillation and Digestion Prep.			1/17/79
Sample ID	Date	Time	Aliquot Distilled then Digested in ml	final total Sample Volume, ml
B-W-I	1/17	12:15 pm	100.0 ml	
B-W-O				
B-A-I				
B-A-O				
B-P-W-I	1/17	13:20	100.0	
B-P-W-O				
1-I-Water ¹⁻⁵⁰	1/17	17:30	5.0	
1-O-Water ¹⁻⁵⁰			5.0	
1-A-I		17:30	5.0	341 ml
1-A-O			25.0	295 ml
1-W-I*			5.0	745 ml
1-W-O*			25.0	640 ml
2-S-I	1/18	11:55 am	5.0	
2-S-O			5.0	
2-A-I			5.0	
2-A-O			25.0	
2-W-I*			5.0	
2-W-O*			25.0	
3-S-I	1/19	11:35 am	5.0 ml	
3-S-O				
3-A-I				
3-A-O			25.0	
3-W-I*			5.0	
3-W-O*			25.0	

* PW + Water from Imp Combined volume
all Distillations diluted to 250.0 ml

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1/17/79

*Formaldehyde-Blood-Increased Urine
and Wash Bottle**Stock pl 1000 µg/ml Formaldehyde - Dilute 1.0 ml → 100 ml → 10 µg/ml
Working Sol'n**Standards ml WS Abs*

0 µg Formaldehyde	0	.009
1	0.1	.060
3	0.3	.164
5	0.5	.240
7	0.7	.360
10	1.0	.510
20	2.0	.960

*11.0 %T**Result**Samples*

B-W-I	4.0	.010	Equivalent µg
B-W-O	↓	.010	4.2
B-A-I	↓	.006	4.2
B-A-O	↓	.014	4.2

*µg/ml Formaldehyde
4.05**Two Wash Bottle - 19.00 ml
4.0**1000**4.2**Hold for 20 min**since 1/16/79**16:10 hr*

B-PW-I	4.0	.012	.30
B-PW-O	4.0	.024	.35

*.075 µg/ml Formaldehyde**.085 µg/ml Formaldehyde**Zero Spec 21st without chromotropic Acid (0.1 ml)**in the Sample - 4.1 ml H₂O + 6.0 ml H₂SO₄ only**M. J. P.*

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TITLE CF-Data, Donald'sville, La

Project No. 42285-20

Book No. -4

From Page No. 5

1/19/79

Sample Distillation & Digestion - Preparation

Sample ID	Date of analysis	Time NH ₃ distillation started	aliquot of sample distilled, then digested	final total sample volume, ml
4S A	1/19/79	5:30 PM	5.0 ml	495
4S-W	↓	↓	↓	815 ml*
5S A				462 ml
5S W				1155 ml*
6S A				502 ml
6S W**				1135 ml*

* The volume is the combination of the Oxygenizer water and the Probe Wash Water - Mixed well.

** 6SW - NH₃ may be low because the ^{distilling} tube was not under the surface of the Boric Acid at the start of the test. (~10 minutes - just beginning to boil when, placed under surface of H₃BO₃. m/fx

S = Solution From Sample
A = Acid Impregnated
W = Water + Probe Wash Combination

M. Hox

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Project No. 49455-20Book No. -4 TITLE C.F. Ind. Don't knowFrom Page No.

1/18/74

11:20 - final $\text{TRCH}_2\text{O} \pm 5$ - starting #3

Direct Nesslerization

Note: Some samples were turbid ~~before~~ after the Nessler Reagent was added. The absorbance readings indicate higher concentrations than the normal yellow color would indicate. I estimate the $\mu\text{g. Hg}$ in the Sample Nessler tube by comparing the yellow color of the sample to the color of the 6 standards.

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111

TITLE C.F. Ind. Donaldville, La

Project No. 82988-20

Book No. "-4

From Page No. _____

1/18/79

Direct Measurement of Blanket, Test 1 Sample and Test 2

Stock Solution 1000 $\mu\text{g/ml}$ NH_3 Dilute 5ml $\rightarrow$ 250.0 ml $\Rightarrow$ 20.0 $\mu\text{g/ml}$ NH_3
Working Solution

Standard
Total $\mu\text{g NH}_3$
in 50 ml Test Tube

mls Working Sol'n Abs @ 450 nm

0	0.	.000
20.0	1.	.050
40.0	2.	.120
100.0	5.	.306
160.0	8.	.504
200.0	10.	.655 (22.25T)

Sample	Dilution	Aliquot in 50.0 ml Test Tube	Abs	Equivalent $\mu\text{g NH}_3$	Result $\mu\text{g/ml NH}_3$
BWI	none	50.0 ml	.030	10.0 μg	0.20
BWO		50.0 ml	.018	6.0 μg	0.12
BAI		25.0 ml	.076 cloudy*	25	1.00 cloudy
BAO		25.0 ml	.085 cloudy*	29	1.16 cloudy
BPW-I		50.0 ml	0.78 - cloudy*		cloudy
BPW-O		50.0 ml	0.168 *	55.8	cloudy

2:30 PM

1-S-I (2 $\rightarrow$ 250) 2 to low (5.0 ml as is to high) 50.0 ml of 2 $\rightarrow$ 250 OK
 1-S-O (2 $\rightarrow$ 250) (2 to low) 50.0 ml of 2 $\rightarrow$ 250 - cloudy
 1-A-I (1 $\rightarrow$ 100) 2 to low (2.0 ml as is to high) 50.0 ml of 1 $\rightarrow$ 100 OK
 1-A-O (2 $\rightarrow$ 50) 5 OK
 1-W-I (1 $\rightarrow$ 100) 2 to low (2.0 ml as is to high) 50.0 ml of 1 $\rightarrow$ 100 OK
 1-W-O (2 $\rightarrow$ 50) 5 OK (could use 10. ml)

- continue on p 10 - set up Sample 1 aliquots

2-S-I
 2-S-O * visually ~ 5 μg BAI
 2-A-I * " ~ 5 μg BAO
 2-A-O * BPW I visually ~ 5 μg
 2-W-I BPW O visually ~ 13 μg
 2-W-O

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with

Direct Meas.

1/18/79

Sample	Dilution	Alleged in Test Tube (1000 ml. Test Tube)	Abs.	Equivalent $\mu\text{g NH}_3$	Result $\mu\text{g/l. me.}$ NH_3	total volume sample ml	Result total mg NH_3
1-S-I	2-250	50.0	0.220	72	180		
1-S-O	2-250	50.0	very cloudy 1.7 * 1000	158.5	very cloudy		
1-A-I	1-7100	50.0	0.510	158.5		341 ml	108
1-A-O	2-250	5.0	.505	157.0		295	232
1-W-I	1-7100	50.0	.664	203		745	302
1-W-O	2-250	5.0	.208	68		640	216
1-S-O	—	1.0 ml. $\frac{1}{2}$ cloudy	2.0 **				

* initially ~ 60 μg 1-S-O** " ~ 100 μg 1-S-O

Calculation:

for Scrubber Waters

S-I, S-O

$$\mu\text{g/l. me. NH}_3 = \frac{\text{Equivalent } \mu\text{g NH}_3}{\text{ml. in Test Tube}} \times \frac{\text{final Vol. of Dilution ml.}}{\text{ml. diluted}}$$

for Stack Samples A-I, A-O, W-I, W-O

$$\text{total mg NH}_3 = \frac{\text{Equivalent } \mu\text{g NH}_3}{\text{ml. in Test Tube}} \times \frac{\text{final volume of Dilution ml.}}{\text{ml. diluted}} \times \frac{\text{total volume of sample, ml.}}{1000} \times \frac{1000}{\mu\text{g}}$$

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TITLE

C.F. Inductance, Donaldsonville La

Project No. 82983-20

Book No. 12-4

From Page No. —

Direct Neutralization of Test II

1/18/79

16:45

Stds - see p 9 for make up Abs @ 405nm

	ml WS	
0	0	.000
20	1	.060
40	2	.122
100	5	.308
160	8	.515
200	10	.670

.635 end

Sample	Dilution	ml/match	Abs	Equivalent µg NH ₃	total ml of single ml PS	µg/m ² NH ₃	total mg NH ₃
2-S-I	2→250	50.0	.055	17.5		43.8	
2-S 0	↓	50.0	2.0 *			50.1	
2-A I	↓	50.0	.162 * OK	52.5	322	323	
2-A 0	2→50	5.0	.645	200 µg	323	391	
2-B I	2→250	50.0	.650	201	778	371	
2-W 0	2→50	5.0	.345	345 111 µg	668		

* 250 - usually ~ 50-60 µg
2AI - approx match 40 µg Std.

see p 10 for Calculations

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TITLE CFDnd. Donaldsonville, La.

Project No. 82383-20

Book No. "-4"

From Page No.

1/19/19

Scrub Water Inlet and outlet in "4th tank Water"
Should not have Ca or Mg, but may have organics,
- used by products bi and the Urea
- per conversation with Dave Cargot 1/18/19 10:20 a.m.
- attempt to find out why sample S-0 is cloudy.

m. P. of

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From Page No.

1-19-79

Direct Neutralization of Test 3 Samples

Stock 1000 $\mu\text{g}/\text{ml}$ NH_3 Dilute 5.0 ml $\rightarrow$ 250. $\Rightarrow$ 20 μg NH_3/ml
 Working Solution = 40.5

stds

total μg NH_3 in same test tube	mls	Abs
0	0 ml 40.5	1.000
20	1.0	.070, .064
40	2.0	.110
100	5.0	.306 .305 end color
160	8.0	.570
200	10.0	.620 or .61 end color

Sample	Dilution	Aliquot 5.0 ml in test tube	Abs	Equivalent μg NH_3	total volume of sample ml	$\mu\text{g}/\text{ml}$ NH_3	total mg NH_3
3-6-I	none	5.0	.235, .360	116 μg		232 very cloudy	
3-5-0	2 $\rightarrow$ 250	50.0	very cloudy*	2.0			
3-A-I	2 $\rightarrow$ 250	50.0	.09, .026	9 μg	364		8.19
3-A-0	2 $\rightarrow$ 250	3.0	.2, .265	86 μg	366		262
3-W-I	2 $\rightarrow$ 250	25.0	50.0 ml .1, .610	196 μg	1033		445
3-W-0	2 $\rightarrow$ 250	5.0	.0, .122	40 μg	625		125
Rep 3-A-I	none	2.0 ml	.196 visually 0.1	64 μg			

* 3-5-0 visually yellow color when compared to standards
 looks approximately 30 μg or more

See p 10 for Calculations

m. D. P.

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1/20/78

Out of TAC H_2O - Use CFma - Report Water to bring up
Use digestate for distillation into Boric acid
(30 ml/sample)
Also use CF H_2O for Direct Msr. of Sample 4 to 6
- Dilution.

M. Jop

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Project No. 82988-20
Book No. 11-4

17

From Page No. —

Direct Neutralization of Bact 4-6

1/20/79
11 AM

Standard Stock 1000 $\mu\text{g}/\text{ml}$ NH_3
Dilute 5.0 ml $\rightarrow$ 250 ml $\Rightarrow$ 20 μg NH_3/ml (Nobony Solution)

Std	total NH_3 in 5.0 ml Thurber tube	ml U.S.	Ats	Ats after
0		0	.000	
20		1.	.075	.060
40		2.	.145	
100		5.	.350	
160		8.	.530	
200		10.	.680	.66

Sample	1st Dilution	2nd Dilution	Aliquot of final Dilution	Ats	Equivalent μg NH_3	Sample Volume ml	Total mg NH_3	Results $\downarrow$
4S-A	none	none	2.0	.380*	110 μg	495	27.2	✓ ok
4S-W	2-250	2-250	50.0	.505	148 μg	825	37.694	✓ ok
5S-A	none	none	2.0	very cloudy .2		462	cloudy	
5S-W	2-250	2-250	50.0	.438	127 μg	1155	45.837	
6S-A	none	none	2.0	slightly cloudy 1.5 dk		502	cloudy	
6S-W	2-250	2-250	50.0	.555	163 μg	1135	57.814	

* 4S-A visually 90 μg (did not appear cloudy)
5S-A " 100 μg very cloudy
6S-A " 60 μg

see p 10 for calculations

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Formaldehyde Determinations

1/26/79

Standard - stock 1 mg/ml in Solution "B" (ug)	10 ml → 100 ml = 100 ug/ml (Solution "B") mls Soln "B"	abs 580nm
0	0	0
1	0.1	.048
3	0.3	.122
5	0.5	.170, 190
7	0.7	.290 ✓
10	1.0	.440
20	2.0	.860

Sample	Aliquot mls	Abs 580nm sample last
Test 1 outlet gas	4	
Run 2 outlet		.035 ✓
Run 3 "		.034
Test 4 outlet gas		.030
Run 5 outlet		.048
Run 6 outlet		.028
Test 5 Inscr. PWS		.100
1 - J - scrubber H ₂ O		off scale - dilute 1 → 10
1 - O - "		.008
1 - A - I		.208
1 - A - D		.234 ✓
1 - W - I		.470 470
1 - W - O		.565

Agric. Sample
Calculated on p
74-75 Agric. Book

ug CH ₂ O	Sample Volume ml	ug/ml Sample	total mg Form
4.4	1	4.04	
5.0	341	0.	0.426
5.6	245		0.413
11.0	745		2.105
13.2	640		2.11

↓ de sum
↓ sum

Calculations

$$\text{total } \mu\text{g/ml Form.} = \frac{\text{Eq. } \mu\text{g CH}_2\text{O}}{\text{ml sample}} \times \text{dilution factor}$$

$$\text{total mg CH}_2\text{O} = \frac{\text{Eq. } \mu\text{g CH}_2\text{O}}{\text{ml sample}} \times \text{dilution factor} \times \text{ml sample} \times \frac{.001 \text{ mg}}{1 \mu\text{g}}$$

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1/26/79

Prep of Solid Samples for Formaldehyde Analysis

Feed Melt to Granulator 1/18/79

$$\begin{array}{r} 5.8610 \\ - 0.2000 \\ \hline 5.6610 \text{ g / 100 ml H}_2\text{O} \end{array}$$

Unscreened Urea Composite

$$\begin{array}{r} 5.8950 \\ - 0.2000 \\ \hline 5.6950 \end{array}$$

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Standard stock = 1 mg/ml ^{no formaldehyde benzoate} 1.0 ml → 100 ml ⇒ 100 μg/ml formaldehyde (salt is "B")
 Solution "B" (μg) ml soln "B" abo 580 nm

0	0	0.800
1	0.1	0.068 0.80
3	0.3	.170 .80
5	0.5	.255 .260
7	0.7	.348 .347
10	1.0	.480 .475
20	2.0	.880 .80

* See Calculations p20

Samples	Aliquot (mls)	2600 580 nm	Equivalent μg formaldehyde	total sample volume	μg/ml formaldehyde	total μg formaldehyde
Test 1, redist. grad	4 mls - Agree	.000	0.0	Agree		
1-I- scrubber H ₂ O (dil. 5-5) take 4 mls		.490	10.4		26.0	
2-S-I (dil. 5-5) take 4 mls		.390	8.0	-	2.00	
2-S-O		.016	0.3	-	0.075	
2-A-I		.020	0.4	382		0.0382
2-A-O		.022	0.45	323		0.0363
2-W-I		.380	7.75	778		1.507
2-W-O		.340	6.9	668		1.136
3-S-I (dil. 5-5) take 4 mls		.273	5.4	-	13.5	
3-S-O		.010	0.2	-	0.05	
3-A-I		.052	1.0	364		0.091
3-A-O		.026	0.5	366		0.0458
3-W-I		.560	11.6	1033		2.996
3-W-O		.393	8.0	625		1.25
Feed mill to granulator						
1.1819 CF BND	1 ml	1.22	high			
Unscreened urea composite	1 ml	1.20	high			
Feed Mill 2-725	4.0 mls 1.0 ml	0.72	16.1	100.0		20.12
Unscreened 2-725	4.0 mls 1.0	0.94	21	100.0		26.25
Unscreened 2-750	1.0	0.36	7.25	100.0		18.12
Unscreened 3-7200	4.0 mls	.162	3.25	100.0		5.417

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Formaldehyde Determination

1/30/79

Standard (7% formaldehyde bucellite) - stock = 1mg/ml 1.0 → 100 ml ⇒ 100g/ml formal. (Soln. E)

Soln. "B" (g)	mls taken Soln. "B"	Abs. 580nm
0	0	0.00
1	0.1	.023
3	0.3	.0806 .095
5	0.5	.150 .160
7	0.7	.250 .252
10	1.0	.388 .390
20	2.0	.6806 .683

Samples	Code	1st dilution	Aligent	Abs 580nm	Equivalent total mg CH ₂ O	% CH ₂ O in sample
CFIND Feed Rch.	A	2 mls → 50	1 ml	.378	10.7	26.75 0.473
p21 5.661g/100ml	B	2 mls → 100	2 ml	.290	8.7	20.75 0.367
	C	2 mls → 200	4 ml	.308	9.8	22.0 0.389
Unscreened Urea	D	2 mls → 50	1 ml	.440	12.5	31.25 0.599
5.695g/100ml	E	2 mls → 100	2 ml	.590	15.3	38.25 0.612
p21	F	2 mls → 200	4 ml	.538	15.2	38.0 0.667

Calculation

Unscreened & Feed melt were solid samples
- dissolved & known wgt in 100.0 ml H₂O

Calculate total mg CH₂O in 100 ml of sample (as on p 20)

Then divide total mg CH₂O by total mg of sample in 100 ml (p 22)
and multiply by 100 = get %.

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2-16-79

Calculations for Distilled NH_3 and Urea sample
on pp 25-27 p 35

Blank Correction Factor from p 27

NH_3 Reagent Blank 16 μg /50 ml of Distilled in Nessler tube = 0.32 $\frac{\text{NH}_3}{\mu\text{g/ml}}$ in Nessler tube

Urea Reagent Blank 21 μg /50 ml of Distilled in Nessler tube = 0.42 $\frac{\text{NH}_3}{\mu\text{g/ml}}$ in Nessler tube

$$\mu\text{g/ml } \text{NH}_3 = \frac{\text{Equivalent } \mu\text{g } \text{NH}_3}{\text{ml in Nessler tube}} \times \frac{\text{Serial Vol of 2nd dilution}}{\text{ml diluted}} \times \frac{250 \text{ ml}}{\text{ml sample distilled}}$$

$$\mu\text{g/ml Urea} = \text{above} \times 1.765$$

$$\text{total } \mu\text{g } \text{NH}_3 = \frac{\text{Equivalent } \mu\text{g } \text{NH}_3}{\text{ml in Nessler tube}} \times \frac{\text{Serial Vol of 2nd dilution}}{\text{ml diluted}} \times \frac{250 \text{ ml}}{\text{ml sample distilled}} \times \frac{\text{Sample Volume ml}}{\text{Volume ml}} \times \frac{0.01 \text{ mg}}{\mu\text{g}}$$

$$\text{total } \mu\text{g Urea} = \text{above} \times 1.765$$

to apply Blank Correction substitute the below value for Equivalent $\mu\text{g } \text{NH}_3$

$$\text{Equivalent } \mu\text{g } \text{NH}_3 = \left[\begin{array}{l} 0.32 \mu\text{g/ml} \\ 0.42 \mu\text{g/ml} \end{array} \times \text{ml in Nessler tube} \right]$$

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2/1/79

Analysis of Distilled Ammonia Sample.

Results

Sample	Sample Volume	Aliquid distilled to 250ml	2nd Dilution	ml in 50 ml Neutrotite	H ₂ O	viscous observe	Equivalent NH ₃	total mg NH ₃	Blank correct
2-1-79 B-W-I	does not apply	100ml → 250	none	50.0	.094	10.0	28	1.04	0.66
B-W-O					.090	10	26.5	1.32	0.52
B-A-I					.088	10	26.0	1.30	0.50
B-A-O					.094	10	28.	1.40	0.60
B-P-W-I					.495	cloudy	cloudy	7.3	6.5
B-P-W-O					.165	20.0	49	2.45	1.65
1-S-I	—	5.0	—	50.0	.522	cloudy 60-80	cloudy 154	15.4	cloudy
1-S-O	—		5 → 100	(25.0) 100 ml	.480	~50	141.5	14.50	135.0
1-A-I	34/ml	↓	none	10.0	.230	~60	68.0	116	110
1-A-O	275 ml	25.0	↓	2.0	.535		158.	233	232
1-W-I	745 ml	5.0	↓	10.0	.73 repeat		R	R	R
1-W-O	640 ml	25.0	↓	2.0	.22		65	208.	206
2-1-79 2-S-I	—	5.0	—	50.0	.23	cloudy ~40	cloudy 68	68.	52.4
2-S-O	—	↓	5 → 100	10.0	.232		128	12,800	12,800
2-A-I	382	↓	none	10.0	.102		30.	57.3	57.2
2-A-O	328	25.0	↓	2.0	.505		149	244	243
2-W-I	778	5.0	↓	10.0	.84 repeat		R	R	R
2-W-O	668	25.0	↓	2.0	.148		55.5	195	183
3-1-79 3-S-I	—	5.0	—	50.0	0.160	20-30	47	47	31.2
3-S-O	—	↓	5 → 100	10.0	.445		131	13,100	13,100
3-A-I	364	↓	none	10.0	.045		13.	23.7	17.8
3-A-O	366	25.0	↓	2.0	.490		144.5	264.	263
3-W-I	1033	5.0	↓	10.0	.620		182.5	943.5	926.1
3-W-O	625	25.0	↓	2.0	.138		40.5	127	125

+ 150 - 25ml aliquot - form a ~~very~~ cloudy brown precipitate upon neutralization

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Standards - Stock 1000 $\mu\text{g}/\text{ml}$ NH_3
 Dilute 10.0 ml $\rightarrow$ 500 ml $\Rightarrow$ 20 $\mu\text{g}/\text{ml}$ NH_3 Working solution - W.S

total mg NH_3 in same test tube	vol of W.S	Abs.	2/1/79	Blank Test 1 and Test 4, 5, 6
0	0	.010	.010	-
20	1	.068	.065	
40	2	.140	.138	
100	5	.345	.344	
160	8	.540	.538	
200	10	.670	.67	

Standard	
0	.000
20	.068
40	.132
100	.348
160	.545
500	.680

2-2-79

Date _____

From Page No. 27

2-6-79

Multiplication of Urea Samples Diluted @ CPD md.

Sample	Sample vol. ml	Aliquot diluted to 250 ml	2nd dilution	ml in 50 ml	abs	Equivalent $\mu\text{g NH}_3$	mg urea	total mg urea	Blank corrected Results
B-W-I	does not apply	100 $\rightarrow$ 250	none	50.0	.244	69	6.09		4.24 $\mu\text{g/ml}$ Urea
B-W-O					.185	53	4.68		2.82 $\mu\text{g/ml}$ Urea
B-A-I					.198	56	4.94		3.09 $\mu\text{g/ml}$ Urea
B-A-O					.152	43	3.79		1.94 $\mu\text{g/ml}$ Urea
B-PW-I					.410	116	10.24		8.39 $\mu\text{g/ml}$ Urea
B-PW-O					.412	117	10.33		8.47 $\mu\text{g/ml}$ Urea
1-S-I	-	5.0		50	.300	85.5	150.9		113.8 $\mu\text{g/ml}$ Urea
1-S-O	-		2 $\rightarrow$ 200	2	.412	117	516.300	85.5	516.300 $\mu\text{g/ml}$ Urea
1-A-I	341			25	.082	23	27.7		15.0 mg Urea
1-A-O	295	25.0		25	.115	33	6.87		4.69 mg Urea
1-W-I	745	5.0	5 $\rightarrow$ 100	5	.340	91	239.32	239.32	239.05
1-W-O	640	25.0	-	10 $\rightarrow$	.220	62.5	70.6		65.9 mg Urea
2 SI	-	5.0		50	.240	68	120.0		82.96 $\mu\text{g/ml}$ Urea
2 SO	-		2 $\rightarrow$ 200	2	.420	119	525.100		525.100 $\mu\text{g/ml}$ Urea
2 AI	382			25	.108	30.5	41.1		26.97 mg Urea
2 AO	328	25.0		25	.144	42	9.73		7.29 mg Urea
2 WI	778	5.0	5 $\rightarrow$ 100	5	.336	95	240.90	240.90	24.063 mg Urea
2 WO	668	25.0		10	.212	60	70.7		65.8 mg Urea
3 SI	-	5.0		50	.260	74	130.6		93.5 $\mu\text{g/ml}$ Urea
3 SO	-		2 $\rightarrow$ 200	2	.430	122	539.300		539.300 $\mu\text{g/ml}$ Urea
3 AI	364			25	.148	42	53.97		40.47 mg Urea
3 AO	366	25.0		25	.104	30.5	7.86		5.17 mg Urea
3 WI	1033	5.0	5 $\rightarrow$ 100	5	.252	72	26.55	26.55	26.215 mg Urea
3 WO	625	25.0		10	.198	56	61.8		57.1

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Date

Recorded by

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Project No. 52938-20

TITLE CF Lord, Donaldson Lake, La.

Book No. " -4

From Page No. 25

2/6/79

Urea Analysis continued

Sample	Sample volume	Original distilled to 250 ml	2nd distillation	ml in 50 ml Testate	abs.	Equivalent 10% NH ₃	total mg N in blank	Blank correct results
45W	815	50.0	none	50.0	.440	125	179.8	149.6
45A	495	↓	↓	↓	.230	65.5	57.2	38.9
55W	1155	↓	↓	↓	.362	102.5	209.0	166.1
55A	462	↓	↓	↓	.212	60.0	48.9	31.8
65W	1135	↓	↓	↓	.320	91.0	182.3	140.2
65A	502	↓	↓	↓	.252	72	63.8	45.2

Reagent Blanks

					μg/ml
NH ₃	-	50.0	.056	16	0.32
Urea	-	50.0	.063	21	0.42

Note There does not appear to be any problem with turbidity with the neutralized samples run on pp 27, 28 & 29

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2 pages

Project No. 53453-20

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Book No. 11-4 TITLE 2-2nd, Goodwinville, T.

Page No. _____

2-7-74

Preparation of Solid Sample for NH_3 + Urea Analysis

① Feed Melt to Granulator 1/18/74

$$\begin{array}{r} \cancel{1.2069} \\ \cancel{0.2000} \\ \hline \cancel{1.0069} \end{array} \quad \begin{array}{r} 1.2295 \\ 0.2000 \\ \hline 1.0295 \text{ g} / 100 \text{ ml } \text{H}_2\text{O} \end{array}$$

② Unscreamed Urea Composite

$$\begin{array}{r} 1.2069 \\ 0.2000 \\ \hline 1.0069 \text{ g} / 100 \text{ ml } \text{H}_2\text{O} \end{array}$$

① Feed Melt Aliquot Dried to 5.0 ml
② Unsc. Product 5.0 ml

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Analysis of NH_3 + CO_2 in Solid Sample (p30) 2/7/78

See p35

NH_3 Distillation and Aliquot also
 Sample of Relation in Distillation.
 Distilled NH_3
 Melt 5-7250 none 50.0 .58% cloudy
 Unknown 5-7250 none 50.0 .58% cloudy

Distilled
 NH_3

Melt 5-7250 none 50.0 .64
 Unknown 5-7250 none 50.0 .74 repeat

See p35

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Book No. _____

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5/9/79

Suspended Solids in Inlet & Outlet
Scrubber Water

Filter Samples in glassies with Sample at temperature
atly was when the sample was collected

Infos from p4 of Clean up area Notebook 82288-4 SC

Test 1	Inlet Temp °F		Outlet Temp °F	
①	135	151°F ave	126	124°F ave
②	154		124	
③	164		124	
④	152		120	

Test 2				
①	166	172°F	129	128°F
②	183		128	
③	174		129	
④	167		128	
⑤	172		128	

Test 3				
①	188	175°F	128	127°F
②	172		126	
③	177		129	
④	173		127	
⑤	173		124	

To Page 1

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Date

Record is of

From Page No. 33

7/5/77

Suspended Solids of Scrubber Water

Sample	Temp °F ± 100° at which sample filtered	Volume filtered mls	Good ID	Initial wt of Good g	21/2/77 final wt of good g	wt of suspended solids mg	mg/l TSS	Average of a+b TSS mg/l
1 Outlet a	154/124	200	7	23.95076	23.95156	0.90	4	3.9
1 Outlet b		200	8	23.92296	23.92362	0.76	3.8	
1 Outlet a	124/151	150	3	23.11615	23.11630	0.15	1.0	1.17
1 Outlet b		150	4	23.63110	23.63130	0.20	1.33	
2 Outlet a	172	200	5	23.54224	23.54250	0.26	1.30	0.88
2 Outlet b		200	6	23.63253	23.63462	0.09	0.45	
2 Outlet a	128	200	1	23.88590	23.88714	1.24	6.2	4.6
2 Outlet b		200	2	24.24050	24.24116	0.60	3.0	
3 Outlet a	175	100	9	23.19306	23.19352	0.46	4.6	3.6
3 Outlet b		100	10	22.85316	22.85342	0.26	2.6	
3 Outlet a	127	200	11	24.12042	24.12152	1.1	5.5	4.8
3 Outlet b		100	12	23.36191	23.36222	0.41	4.1	

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2/9/79

Analysis of Solid Sample see p30
 for sample prep

Mett 1.0295 g / 100 me H₂O
 Unscrub 1.0069 g / 100 me

Sample Distillation Distillation Aliquot also

					Equipped mg H ₂	total mg H ₂	% N H ₂	Blank correct results
<u>Distilled NH₃</u>								
Mett	5.0 → 7250	none	25.0	.340	97	19.4	1.88	17.9 mg H ₂ 1.73%
Unscrub		none	25.0	.400	114	22.8 total mg H ₂	2.26 % H ₂	21.2 mg H ₂ 2.11% all done
<u>Urea</u>								
Mett		10 → 100 me	5.0	.185	53	935.4	90.9%	932.6 mg H ₂ 90.6%
Unscrub		10 → 100 me	5.0	.1940	55	9708	96.4%	967.9 mg H ₂ 88.1% all done

Standards p 91 Agner

	Alk-ene
0	.000
20	.072
40	.140
100	.350
160	.545
200	.665

p31 - Direct Methylation samples were very cloudy

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		Recorded by	

Project No. 32980-20

Book No. 11-4

TITLE CF Ind. Louisiana, La

Page No.

2/13/77

pH of Free Melt

This sample was "liquid" when collected. It solidified in sample bottle, a pH is requested.
 Gary McAllister said to make up a solution using the least amount of H₂O possible. Then measure pH

72.5g Descent Melt
 51.8g wt. Benton
20.7g Free Melt.

added 100 ml H₂O
 pH of the saturated solution was 9.2

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Date

Invented by

Date

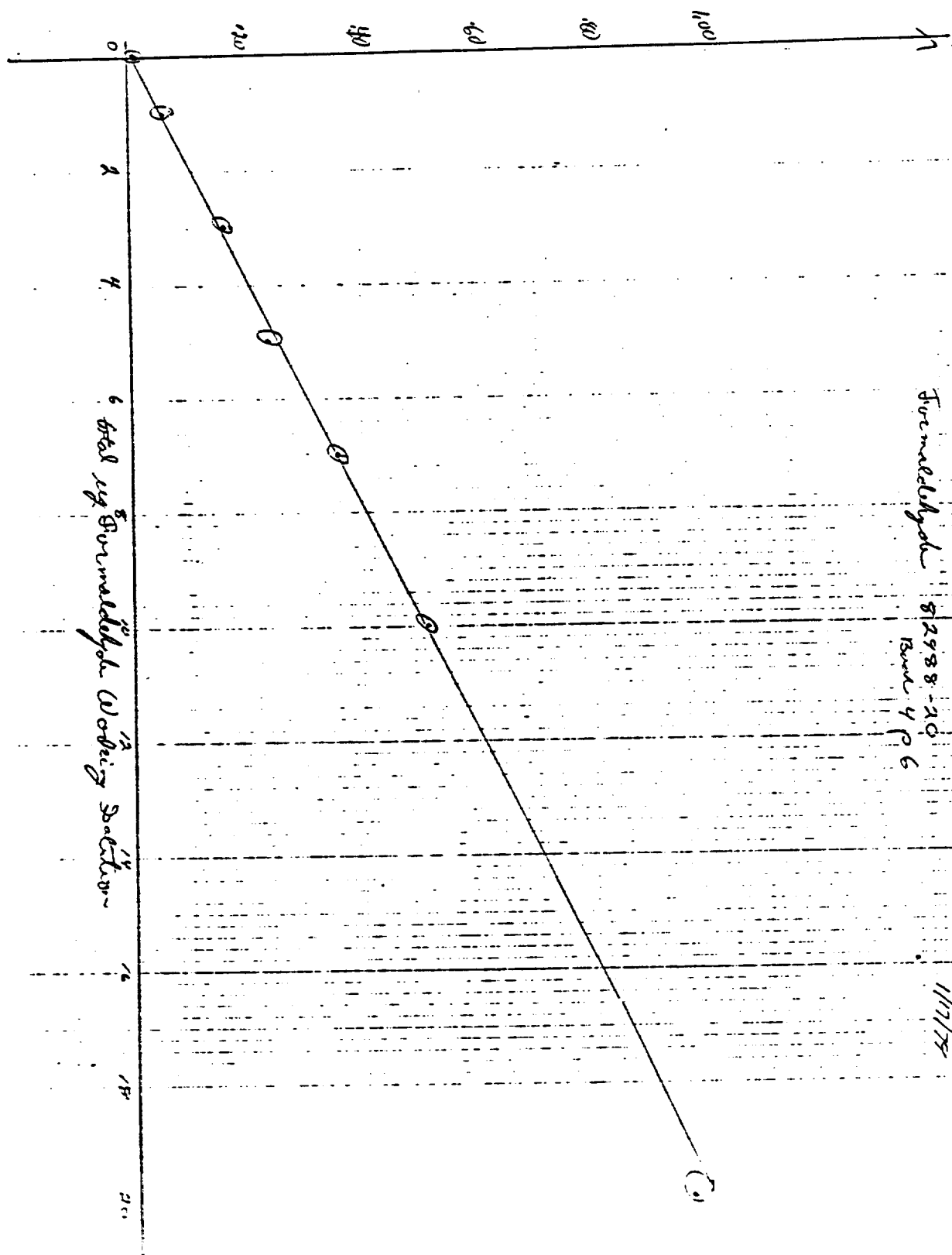
Recorded by

Formaldehyde

82988-20

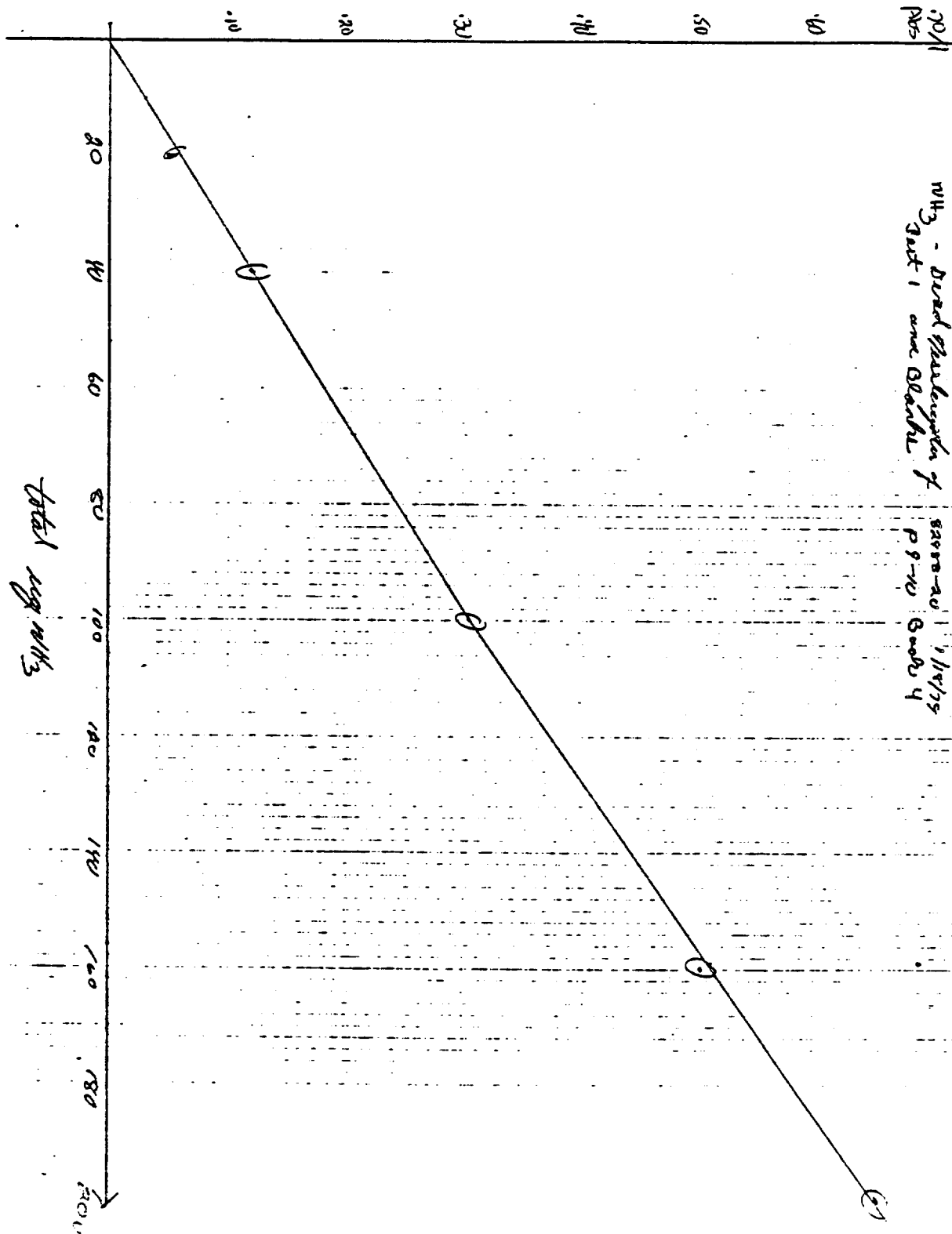
Run 496

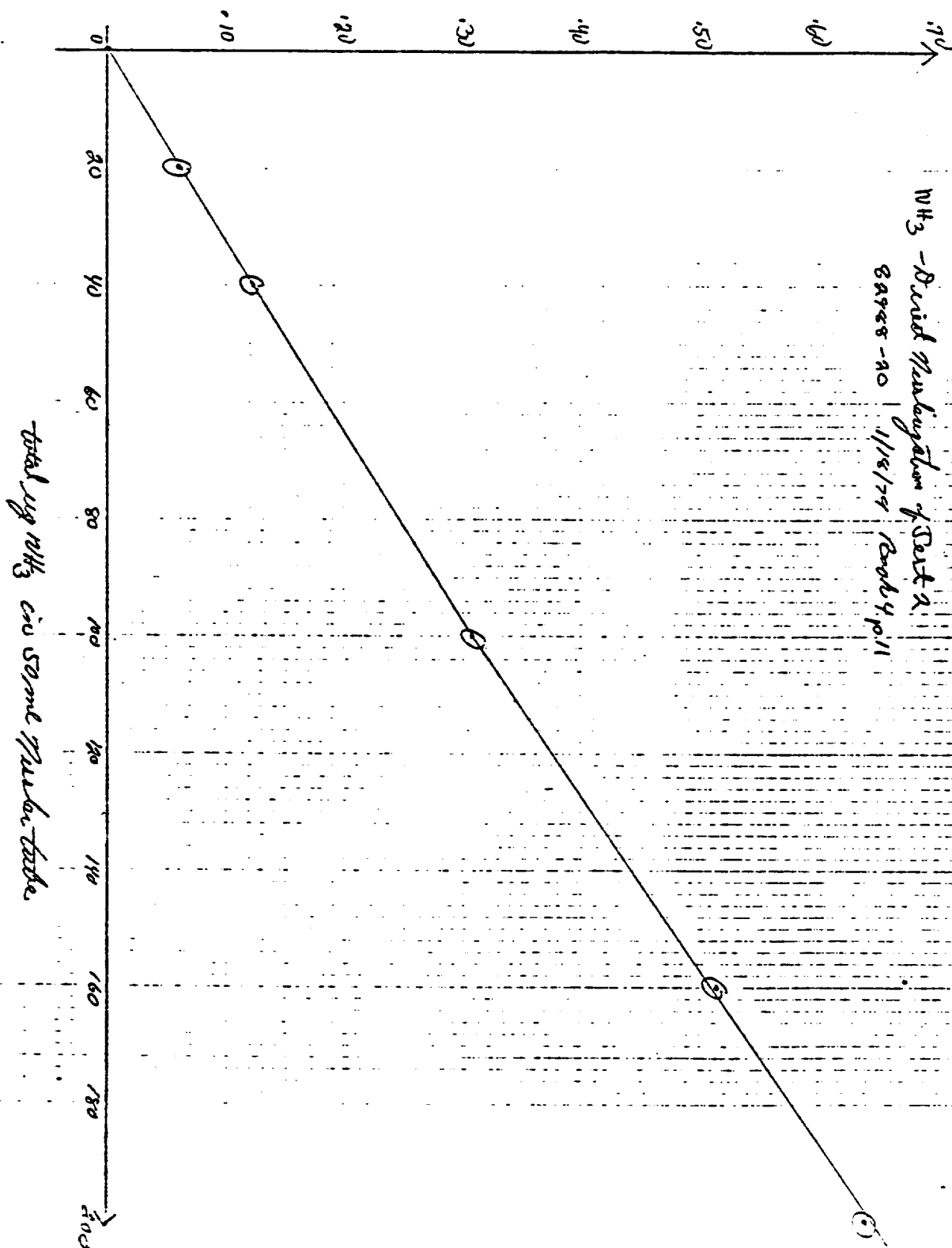
11/17/74



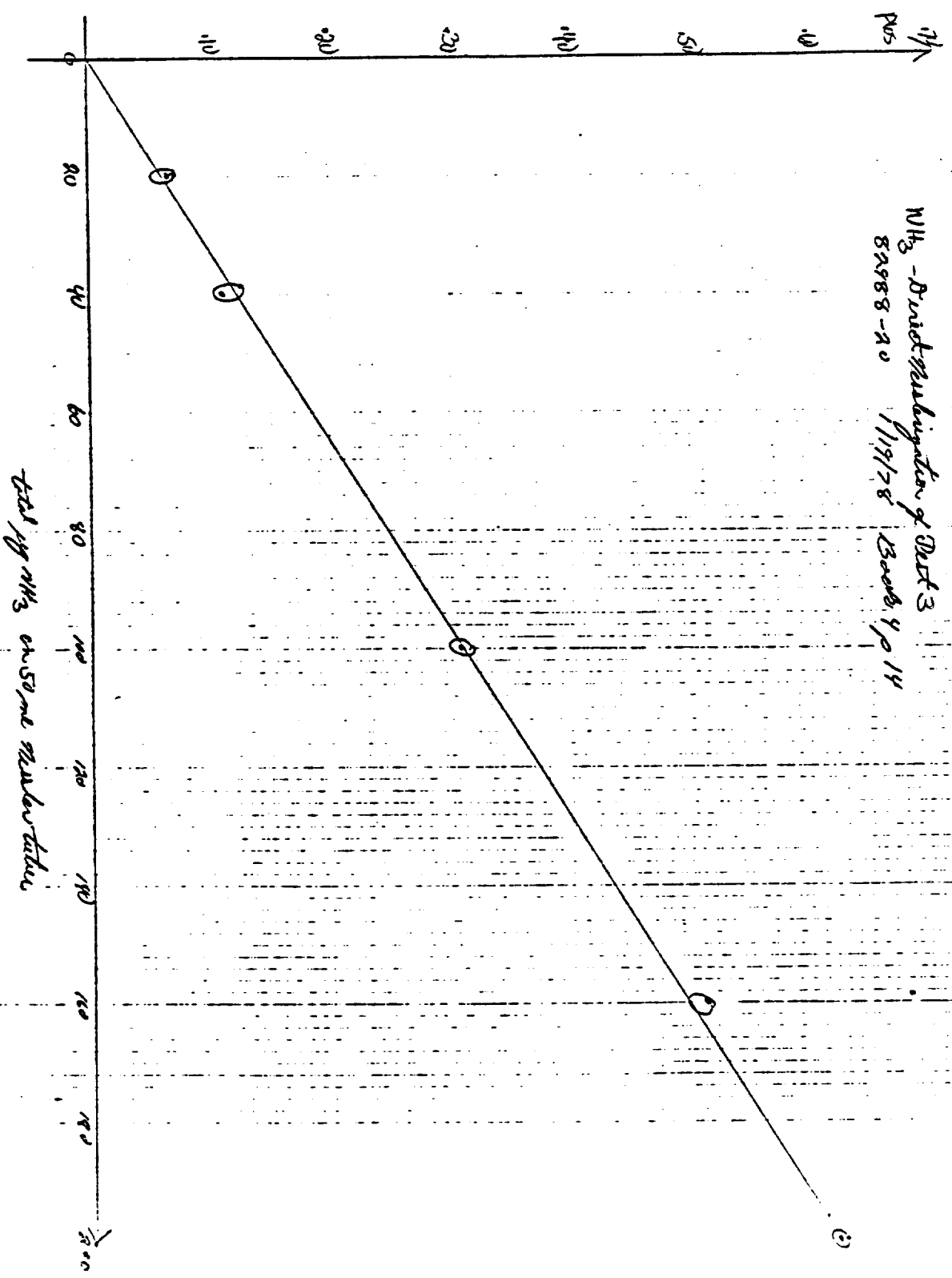
6 bed 14 Formaldehyde Working Station

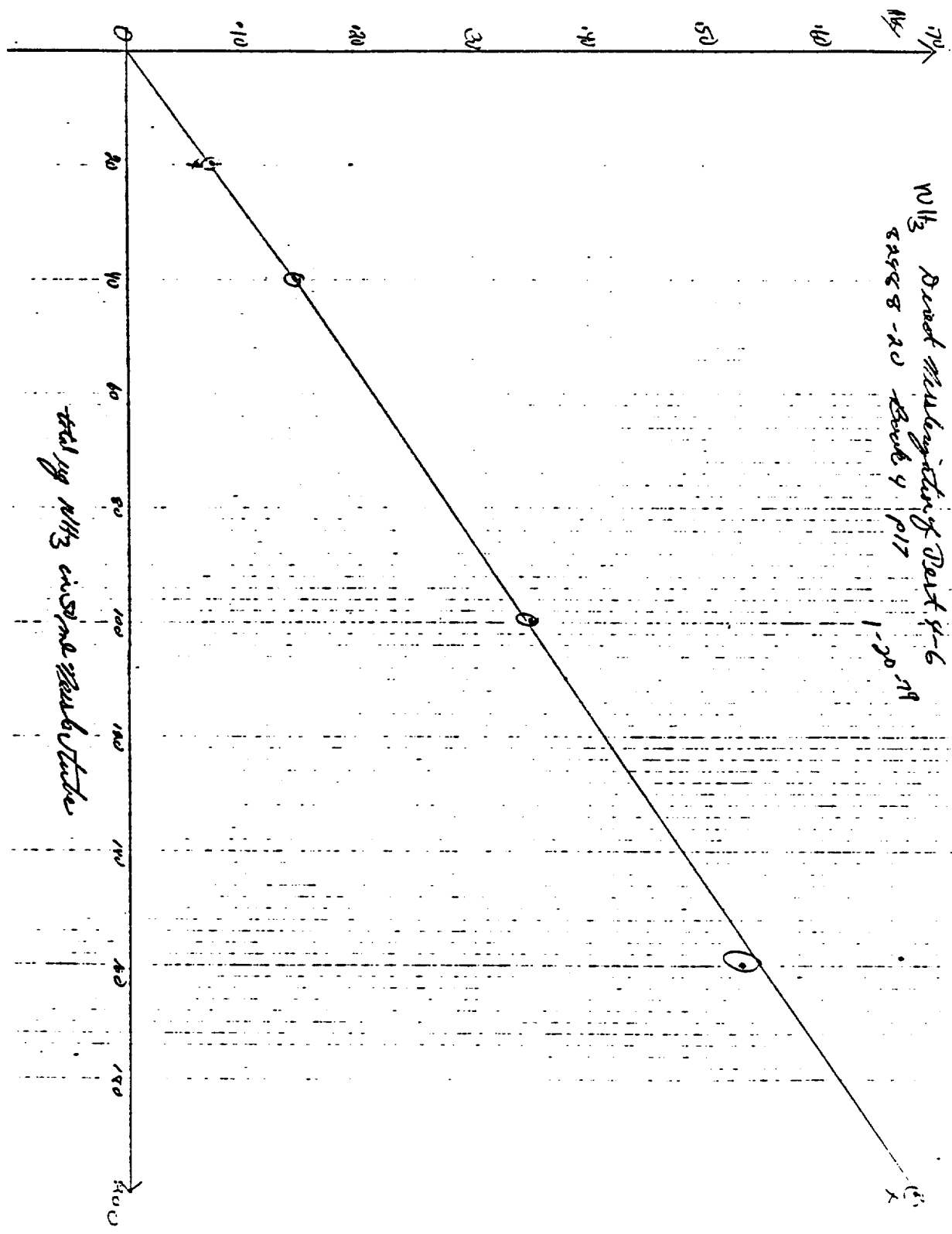
NH_3 - Dead length of
 Post 1 and Dead
 8200-20 11/1/75
 98-10 8000 4

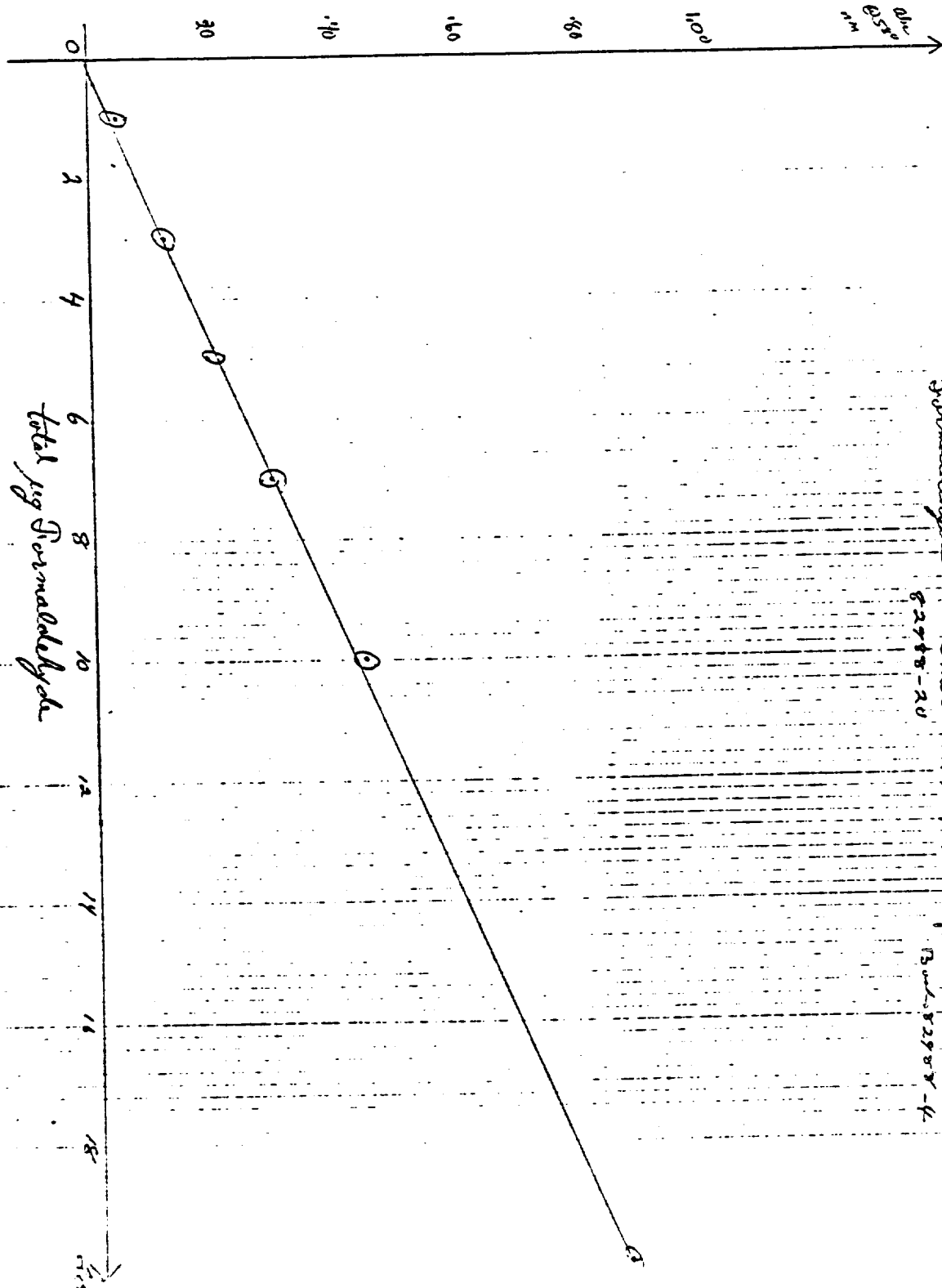




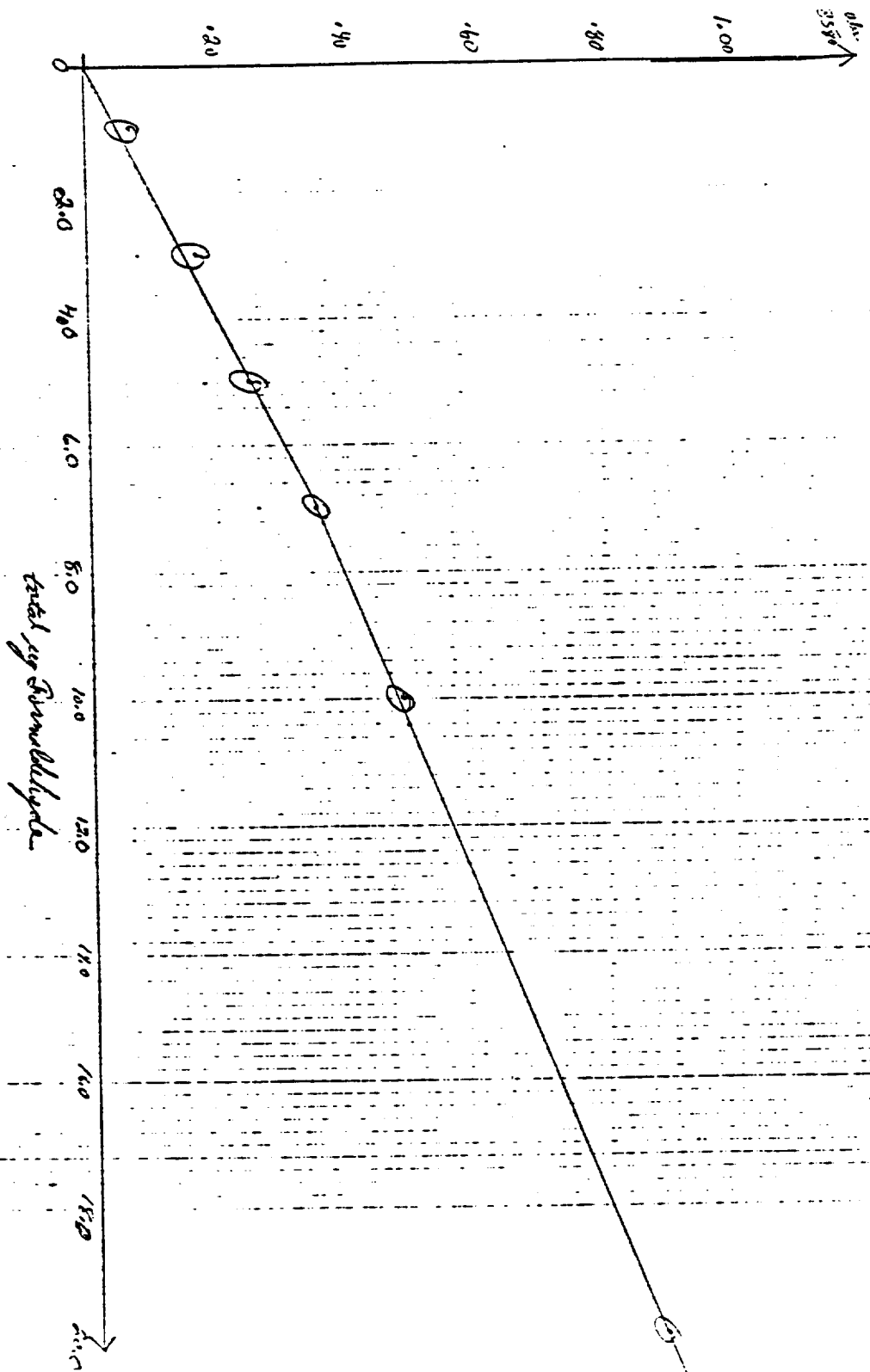
NH_3 - Dividing factor of Dist 3
 82988-20 11/19/78 13008.4 p 14



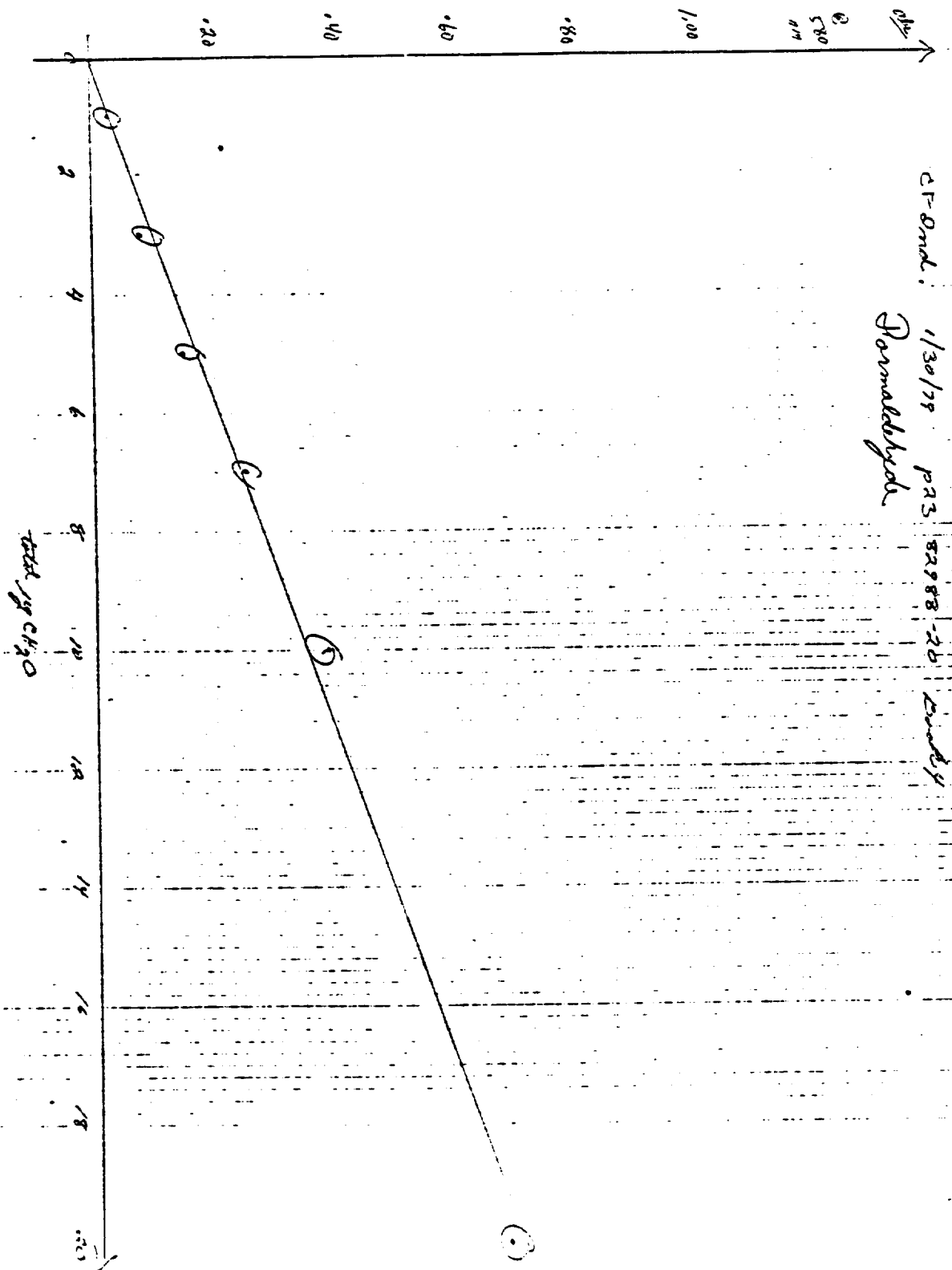


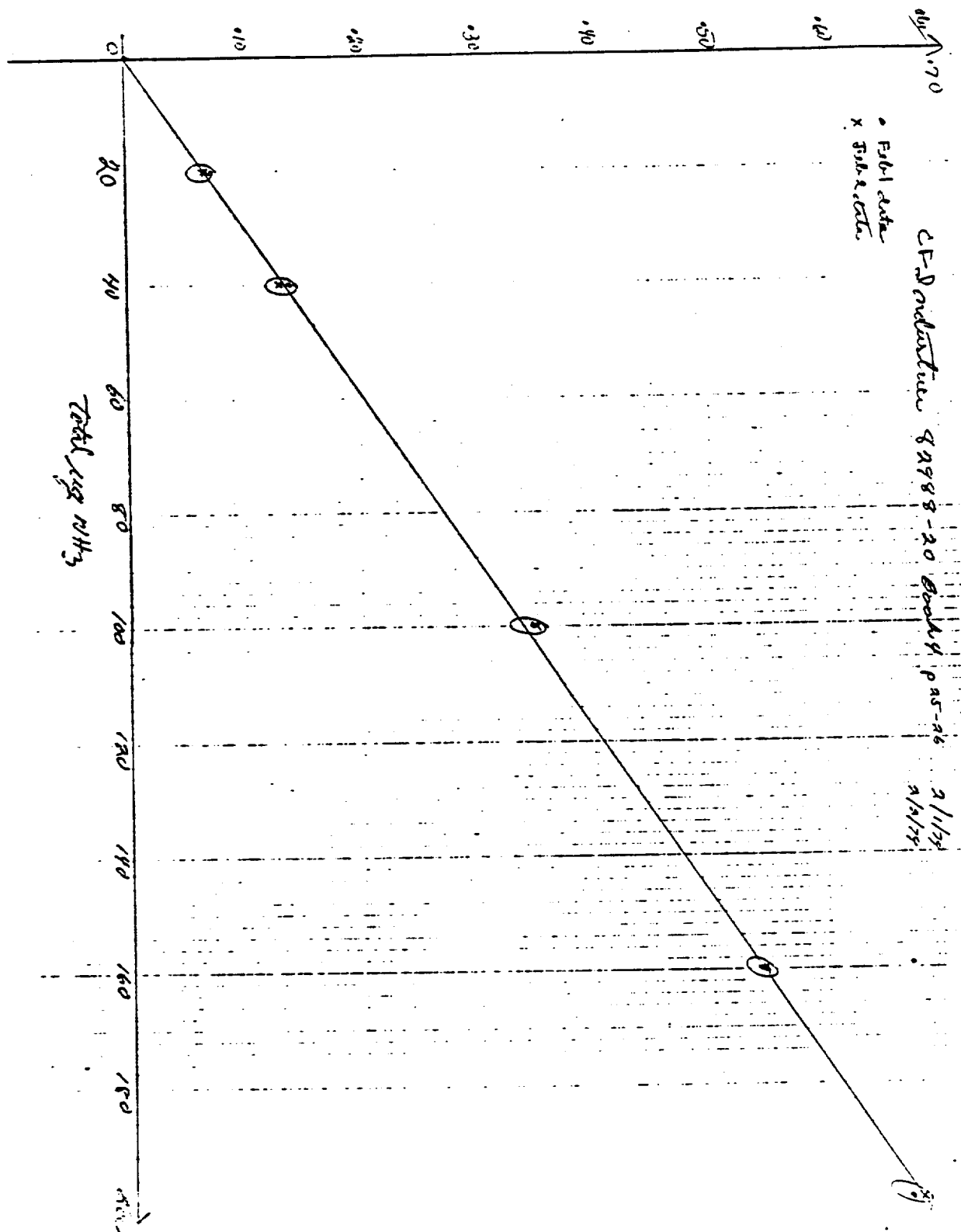


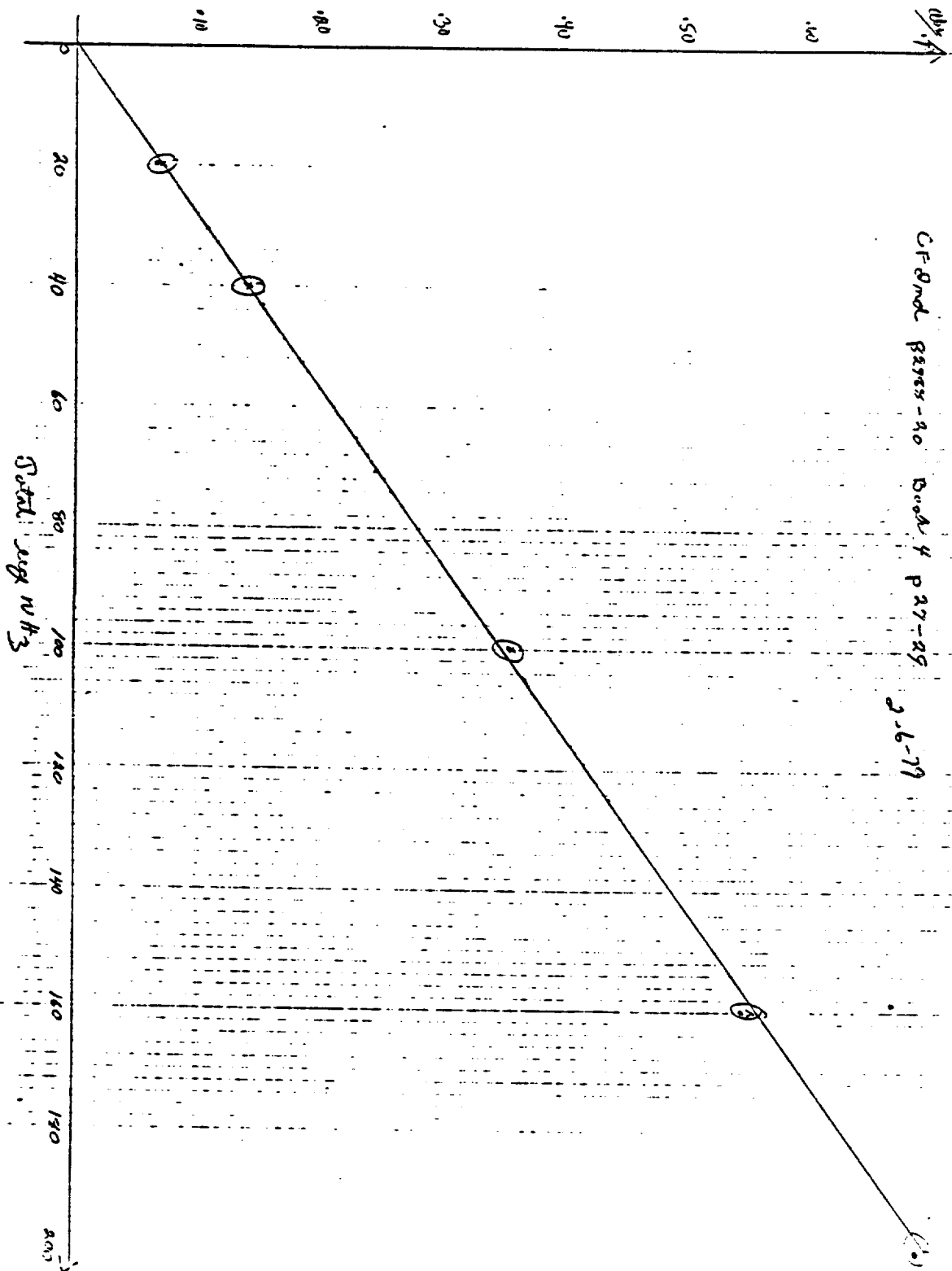
CF Prod. p 22-23 1-22-75
 82928-20
 Bond 4
 Bondedlyde

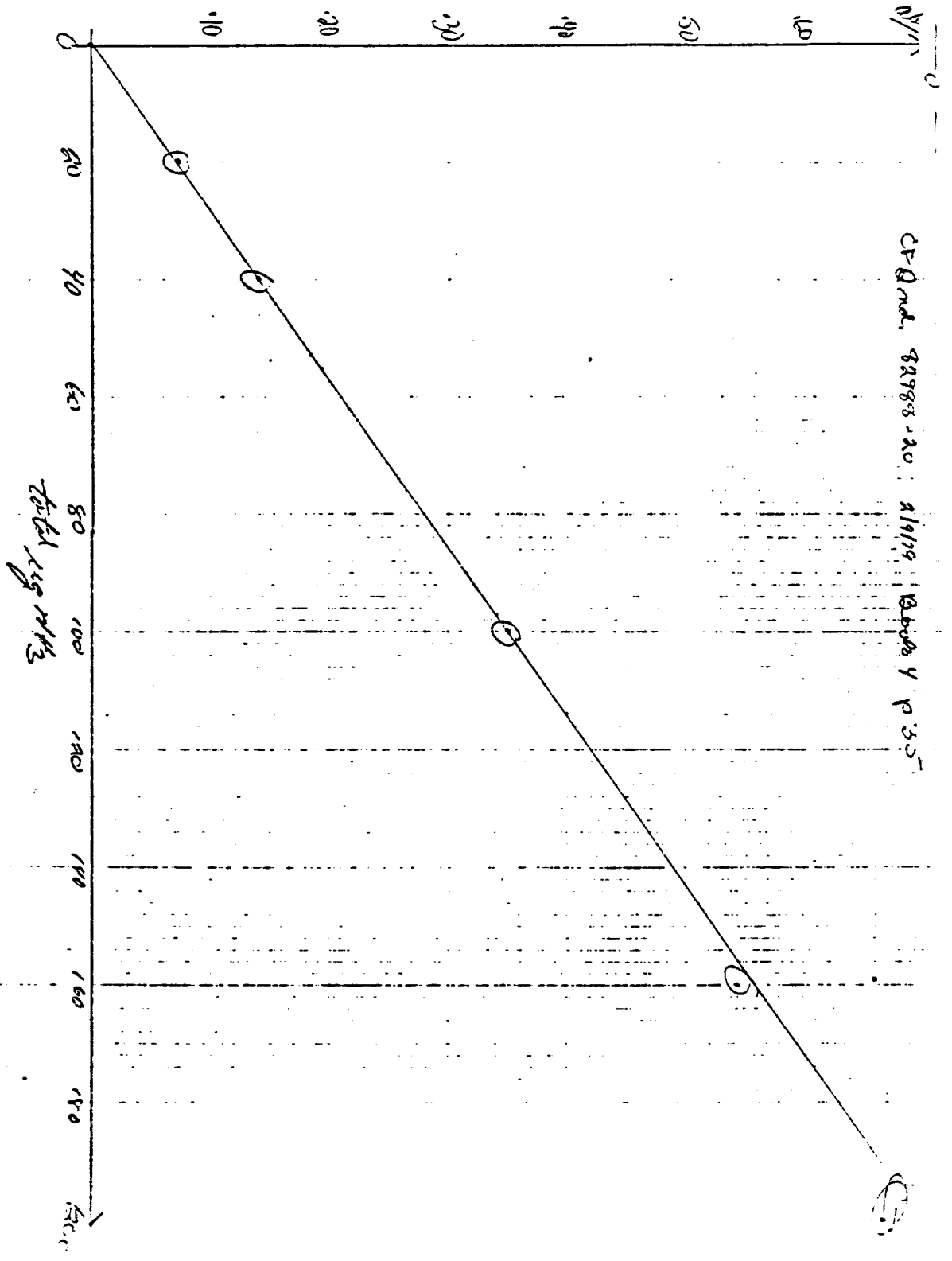


CF-8nd: 1/30/78 P23 52988-20 Exdly
 Dornaldhyde









APPENDIX K

SAMPLING AND ANALYTICAL PROCEDURES INCLUDES PROCEDURES FOR

- K-1 EPA Procedure for Urea**
- K-2 EPA Procedure for Ammonia**
- K-3 EPA Draft Method for Formaldehyde**
- K-4 Discussion of Analytical Procedures**
- K-5 Moisture Determination Procedure**

APPENDIX K-1

EPA PROCEDURES FOR UREA

DETERMINATION OF PARTICULATE, AND UREA
EMISSIONS FROM UREA PLANTS

1-11-80

1. Principle and Applicability

1.1 Principle. A gas sample is extracted isokinetically from the stack. The ammonia is removed from the sample by boiling, and the urea is measured by a colorimetric procedure.

1.2 Applicability. This method is applicable for the determination of urea from urea manufacturing facilities.

2. Apparatus

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 1; it is similar to construction to Method 5. The sampling train consists of the following components.

2.1.1 Probe Nozzle, Probe Liner, Pitot Tube, Differential Pressure Gauge, Metering System, and Barometer. Same as Method 5, sections 2.1.1, 2.1.2, 2.1.3, 2.1.4, 2.1.8, and 2.1.9 respectively. Stainless steel probe liners may also be used.

2.1.2 Impingers--Five as shown in Figure 1. The second and third shall be of the Greenburg-Smith design with standard tips. The first, fourth, and fifth shall be of the Greenburg-Smith design, modified by replacing the insert with an approximately 13 millimeter (0.5 in) I.D. glass tube, having an unstricted tip located 13 mm (0.5 in) from the bottom of the flask. Similar collection systems, which have been approved by the Administrator, may be used.

2.2 Sample Recovery. The following equipment is needed:

2.2.1 Probe-Liner and Probe-Nozzle Brushes, Graduated Cylinder and/or Balance, Plastic Storage Container, and Rubber Policeman. Same as Method 5, sections 2.2.1, 2.2.5, 2.2.6, 2.2.7, respectively.

2.2.2 Wash Bottles--Two. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester.

2.2.3 Glass or Plastic Sample Storage Containers. Chemically resistant, borosilicate glass bottles 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free. (Narrow mouth glass bottles have been found to be less prone to leakage). Alternatively, polyethylene bottles may be used.

2.2.4 Funnel, Glass or Polypropylene.

2.3 Analysis.

2.3.1 Pipettes. Volumetric type 0.5-ml, 2-ml, 5-ml, 8-ml, 10-ml, 20-ml, and 25-ml sizes.

2.3.2 Volumetric Flasks. 25-ml size, 100-ml size, 250-ml size, 500-ml size and 1000-ml size.

2.3.3 Graduated Cylinder. 100-ml size.

2.3.4 Distillation Apparatus.

2.3.4.1 Flasks or Beakers. At least two, 800-ml size.

2.3.4.2 Hot Plate. Capable of heating the distillation flasks to 120°C (248°F).

2.3.5 Spectrophotometer. To measure absorbance at 420 nanometers.

2.3.6 Sample Cells. Two matched absorbance cells to fit the spectrophotometer.

3. Reagents

Use ACS reagent-grade chemicals or equivalent, unless otherwise specified. The reagents used in sampling and sample recovery are as follows:

3.1 Sampling and Sample Recovery.

3.1.1 Silica Gel, Crushed Ice, and Stopcock Grease. Same as Method 5, sections 3.1.2, 3.1.4, 3.1.5, respectively.

3.1.2 Water. Deionized distilled to conform to ASTM specification D 1193-74, type 3. At the option of the analyst, the KMNO_4 test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.1.3 Sulfuric Acid, 1 N. Dilute 28 ml of concentrated sulfuric acid to 1 liter with deionized distilled water.

3.2 Analysis. The reagents need for analysis are listed below.

3.2.1 Water. Same as 3.1.2.

3.2.2 Sodium Hydroxide (NaOH), 10 N. Dissolve 40 g of NaOH in a 100-ml volumetric flask and dilute to exactly 100 ml with deionized distilled water.

3.2.3 Sodium Hydroxide 6 N. Dissolve 240 g of NaOH in 800 ml of deionized distilled water in a 1-liter flask. Dilute to exactly 1 liter with deionized distilled water.

3.2.4 Sodium Hydroxide 1 N. Dissolve 40 g of NaOH in 800 ml of deionized distilled water in a 1-liter flask and dilute to exactly 1 liter with deionized distilled water.

3.2.5 Sodium Hydroxide 0.1 N. Dilute 100 ml of 1 N NaOH to exactly 1 liter with deionized distilled water.

3.2.6 Borate Buffer. Dissolve 2.5 g of sodium tetraborate, $\text{Na}_2\text{B}_4\text{O}_7$, or 4.8 g of the decahydrate $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$, in 500 ml of deionized distilled water in a 1-liter volumetric flask. Add 88 ml of 0.1 N NaOH solution and dilute to exactly 1 liter with deionized distilled water.

3.3.7 Sulfuric Acid 1 N. Slowly add 28 ml of concentrated sulfuric acid to 800 ml of deionized distilled water in a 1-liter flask and dilute to exactly 1 liter with deionized distilled water.

3.3.8 Ethyl Alcohol, 95 percent.

3.3.9 P-dimethylaminobenzaldehyde.

3.3.10 Hydrochloric Acid, Concentrated (36.5 - 38 percent by weight).

3.3.11 Stock Standard Urea Solution. Dissolve 5.000 g of urea in 500 ml of deionized distilled water in a 1-liter flask and dilute to exactly 1 liter with deionized distilled water.

3.3.12 Urea Color Reagent. Prepare the color reagent by dissolving 2.000 g of P-dimethylaminobenzaldehyde in a mixture of 100 ml of 95 percent ethyl alcohol and 10 ml of hydrochloric acid.

4. Procedure

4.1 Sampling. Because of the complexity of this method, testers should be trained and experienced with the test procedure to insure reliable results.

4.1.1 Pretest Preparation. Follow the general procedure given in Method 5, section 4.1.1, except omit the directions for the filter.

4.1.2 Preliminary Determinations. Follow the general procedure given in Method 5, section 4.1.2.

4.1.3 Preparation of Sampling Train. Follow the general procedure given in Method 5, section 4.1.3, except place 100 ml of deionized distilled water in each of the first two impingers, place 100 ml of 1 N H_2SO_4 in the third impinger, leave the fourth impinger empty, and place the preweighed silica gel in the fifth impinger. Assemble the train as shown in Figure 1.

4.1.4 Leak Check Procedures. Follow the leak-check procedures given in Method 5, sections 4.1.4.1 (Pretest Leak Check), 4.1.4.2 (Leak-Check During Sampling Run) and 4.1.4.3 (Post-Test Leak-Check).

4.1.5 Sampling Train Operation. Follow the general procedure given in Method 5, section 4.1.5. For each run, record the data required on a data sheet such as the one shown in Method 5, Figure 5-2.

4.1.6 Calculation of Percent Isokinetic. Same as Method 5, section 4.1.6.

4.2 Sample Recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, thus drawing water from the impingers into the filter holder.

Before moving the sampling train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the impinger inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger or condenser and the probe, disconnect the line at the probe and let any condensed water or liquid drain into the impingers or condenser. Either ground-glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the deionized distilled water used for cleanup as a blank. Take 200 ml of this water directly from the wash bottle being used and place it in a glass sample container labeled "water blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, probe fitting, and probe liner, by washing these components with water and placing the wash in a glass container. Perform the water rinses as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with water from a wash bottle and brushing with a Nylon bristle brush. Brush until the water rinse shows no visible particles, after which make a final rinse of the inside surface with water.

Brush and rinse the inside parts of the Swagelok fitting with water in a similar way until no visible particles remain.

Rinse the probe liner with water by tilting and rotating the probe while squirting water into its upper end so that all inside surfaces will be wetted with water. Let the water drain from the lower end into the sample container. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Follow the water rinse with a probe brush. Hold the probe in an inclined position, squirt water into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any water and particulate matter which is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out with the water or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with water, and quantitatively collect these washings in the sample container. After brushing, make a final water rinse of the probe as described above.

It is recommended that two people be used to clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

Container No. 2. Measure and record the volume of the first two impingers. Then transfer the contents to the container. Rinse the first two impingers and the connecting glassware with water and add the rinse water to the container. Mark the level of the liquid on the container and identify the sample container.

Impingers Nos. 3 and 4. Measure and record the volume of the third and fourth impingers. Discard the liquid.

Container No. 3. Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fifth impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for container No. 3 in section 4.3.

4.3 Analysis. Record the data required on a sheet such as the one shown in Figure 5-3. Handle each sample container as follows:

4.3.1 Container Nos. 1 and 2. Note the level of liquid and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid either volumetrically to ± 1 ml or gravimetrically to ± 1.0 g and record on the data sheet. Combine the contents of both containers in a 500-ml volumetric flask and dilute to exactly 500 ml with deionized distilled water. Distill the sample following the procedure in 4.3.4.

4.3.2 Container No. 3. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field.

4.3.3 "Water Blank" Container. Measure water in this container either volumetrically or gravimetrically and record on the data sheet. Distill the sample following the procedure in 4.3.4.

4.3.4 Sample Distillation. Treat the combined sample 1 and 2 and the water blank as follows:

4.3.4.1 Preparation of Sample. Pipette a 100-ml aliquot of sample into a 1-liter flask or beaker and add 400 ml of deionized distilled water. Then add 25 ml of borate buffer and adjust the pH to 9.5 with 6N NaOH using short-range pH paper to measure the pH. Heat the flask to boiling and boil until the volume is reduced to about 75 ml. (Caution: This step should be conducted under a hood.) Transfer the remaining sample to a

100-ml volumetric flask and dilute to exactly 100 ml with deionized distilled water.

4.3.4.2 Analysis. Pipette 10 ml of this solution into a 25-ml volumetric flask and add 10 ml of the urea color reagent. Dilute to exactly 25 ml with deionized distilled water. Mix well and allow to stand for at least 10 minutes for full color development. Measure the absorbance of the solution at 420 nm using the blank solution (section 5.5) as a zero reference. If the absorbance exceeds that of the 5.00 mg urea standard, prepare another sample using less than a 10-ml aliquot.

5. Calibrations

5.1 Sampling Train. Calibrate the sampling train components according to the indicated section of Method 5. Probe Nozzle (5.1); Pitot Tube (5.2); Metering System (5.3); Temperature Gauge (5.5); Leak-Check of the Metering System (5.6); and Barometer (5.7).

5.2 Determination of Spectrophotometer Calibration Factor K. Add 0.0, 5.0, 10.0, 15.0, 20.0 and 25.0 ml of the stock standard urea solution to a series of six 250-ml volumetric flasks. Then follow the distillation and analysis procedures described for the samples in section 4.3.4 of this method. Each standard at the time of analysis will contain 0, 1.00, 2.00, 3.00, 4.00, and 5.00 mg respectively. The calibration procedure must be repeated each day that samples are analyzed. Calculate the spectrophotometer calibration factor as follows:

$$K_c = 1.00 \frac{A_1 + 2A_2 + 3A_3 + 4A_4 + 5A_5}{A_1^2 + A_2^2 + A_3^2 + A_4^2 + A_5^2}$$

Where:

K_c = Calibration factor.

A_1 = Absorbance of the 1.00 mg standard.

A_2 = Absorbance of the 2.00 mg standard.

A_3 = Absorbance of the 3.00 mg standard.

A_4 = Absorbance of the 4.00 mg standard.

A_5 = Absorbance of the 5.00 mg standard.

6. Calculations

6.1 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop, Dry Gas Volume, Volume of Water Vapor, Moisture Content, Isokinetic Variation, and Acceptable Results. Using data from this test, same as Method 5, sections 6.2, 6.3, 6.4, 6.5, 6.11, and 6.12 respectively.

6.2 Mass of Urea. Calculate the total weight of urea collected in the sample by Equation 1.

$$M = K_c \left(A_s \frac{V_{soln}}{V_{al}} - A_w \frac{V_w}{V_b} \right) \quad \text{Eq. 1.}$$

Where:

- M = Mass of urea collected, mg.
- K_c = Spectrophotometer calibration factor.
- A_s = Absorbance of sample.
- A_w = Absorbance of the water blank
- V_{al} = Volume of sample aliquot analyzed, ml.
- V_b = Volume of water-blank aliquot analyzed, ml.
- V_{soln} = Total volume of solution in which the sample
is contained, ml.
- V_w = Volume of sample returned for analysis, ml.

6.3 Particulate Concentration: Calculate the particulate (urea) concentration as follows:

$$c = K_2 \frac{M}{V_{m(std)}} 10^{-6} \quad \text{Eq. 2}$$

Where:

- c = Particulate (urea) concentration at dry
standard conditions, g/dscm (gr/dscf).
- M = Mass of urea collected, g.
- V_{m(std)} = Volume of gas sample measured by dry gas meter,
corrected to standard conditions, dscm (dscf).
- K = 1.0 for metric units.
= 0.4370 for English units.

7. Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 13th Edition. American Public Health Association, Washington, D.C., pp. 226-232, 1974.
2. Watt, George W. and Joseph D. Chrisp. Spectrophotometric Method for Determination of Urea. Analytical Chemistry. 26:452-453, 1954.
3. Same as Method 5, Citation/through 9 of section 7.

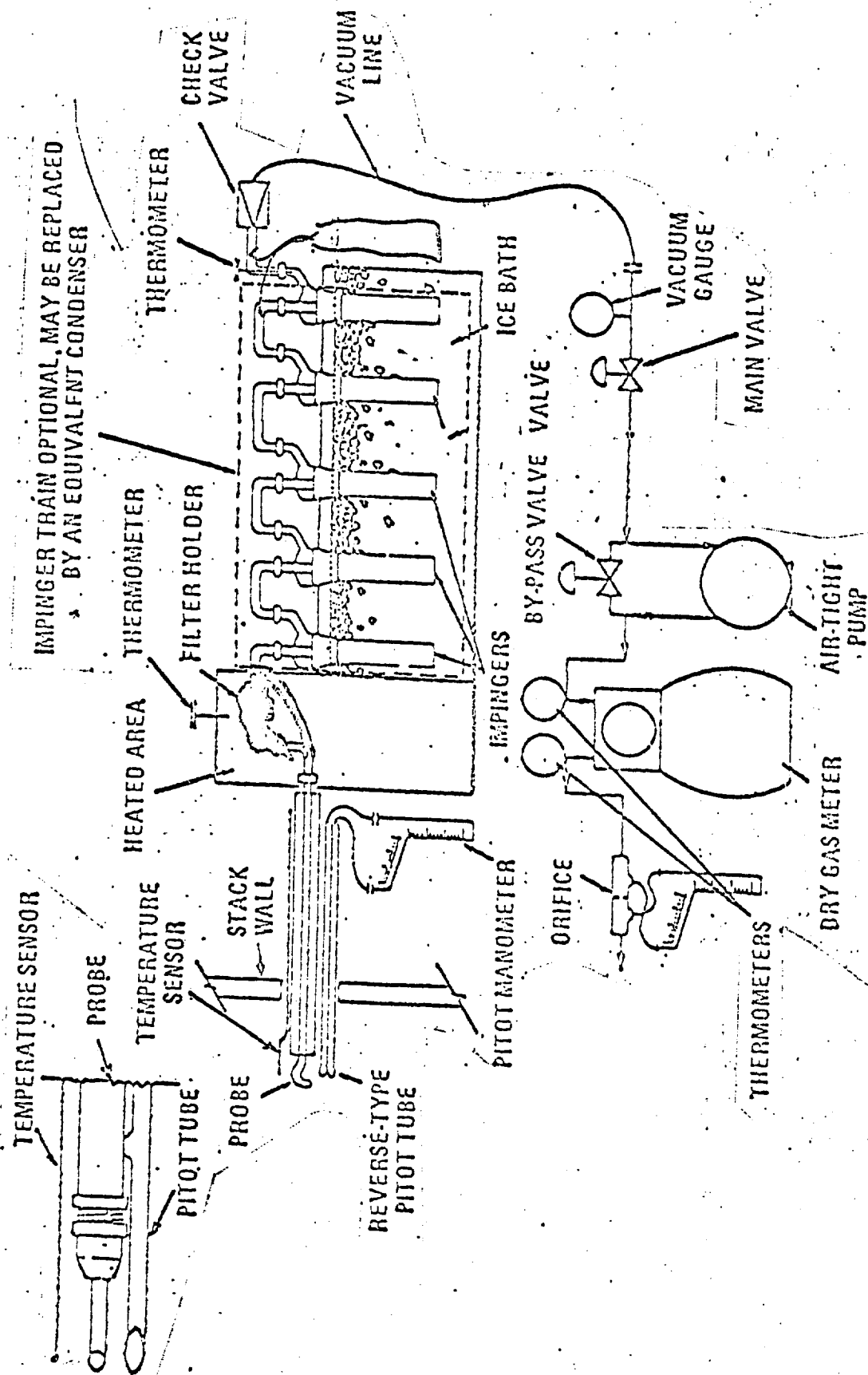
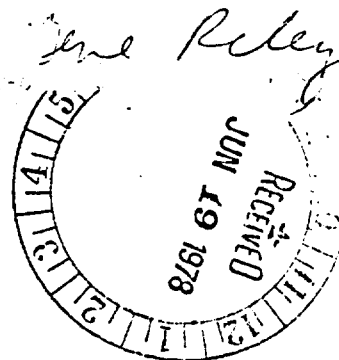


Figure 1. Particulate sampling train

APPENDIX K-2

EPA PROCEDURES FOR AMMONIA

DETERMINATION OF AMMONIA



1.1 Principle and Applicability

1.1 Principle. A gas sample is extracted from the stack and the ammonia is collected in impingers containing sulfuric acid solution. The collected ammonia is reacted to form a colored complex whose intensity is measured by a spectrophotometer.

1.2 This method is applicable for the determination of ammonia emissions from nitrogen fertilizer plants. The minimum detectable ammonia concentration is 30 $\mu\text{g/l}$. The upper limit is 5000 $\mu\text{g/l}$, but this may be extended by diluting the sample.

Possible interferences are calcium, magnesium, iron and sulfide.

2. Apparatus

2.1 Sampling. The sampling train is shown in Figure 1 and component parts are discussed below.

The tester has the option of determining NH_3 simultaneously with particulate matter and moisture determination by replacing the water in a Method 5 impinger system with 1.0 N sulfuric acid.

2.1.1 Probe. Borosilicate glass, or stainless steel (other materials of construction may be used, subject to the approval of the Administrator), approximately 6-mm inside diameter, with a heating system to prevent water condensation and a filter (either in-stack or heated out-stack) to remove particulate matter, including sulfuric acid mist. A ^{1/2 inch} plug of ^{tightly packed} glass wool is a satisfactory filter.

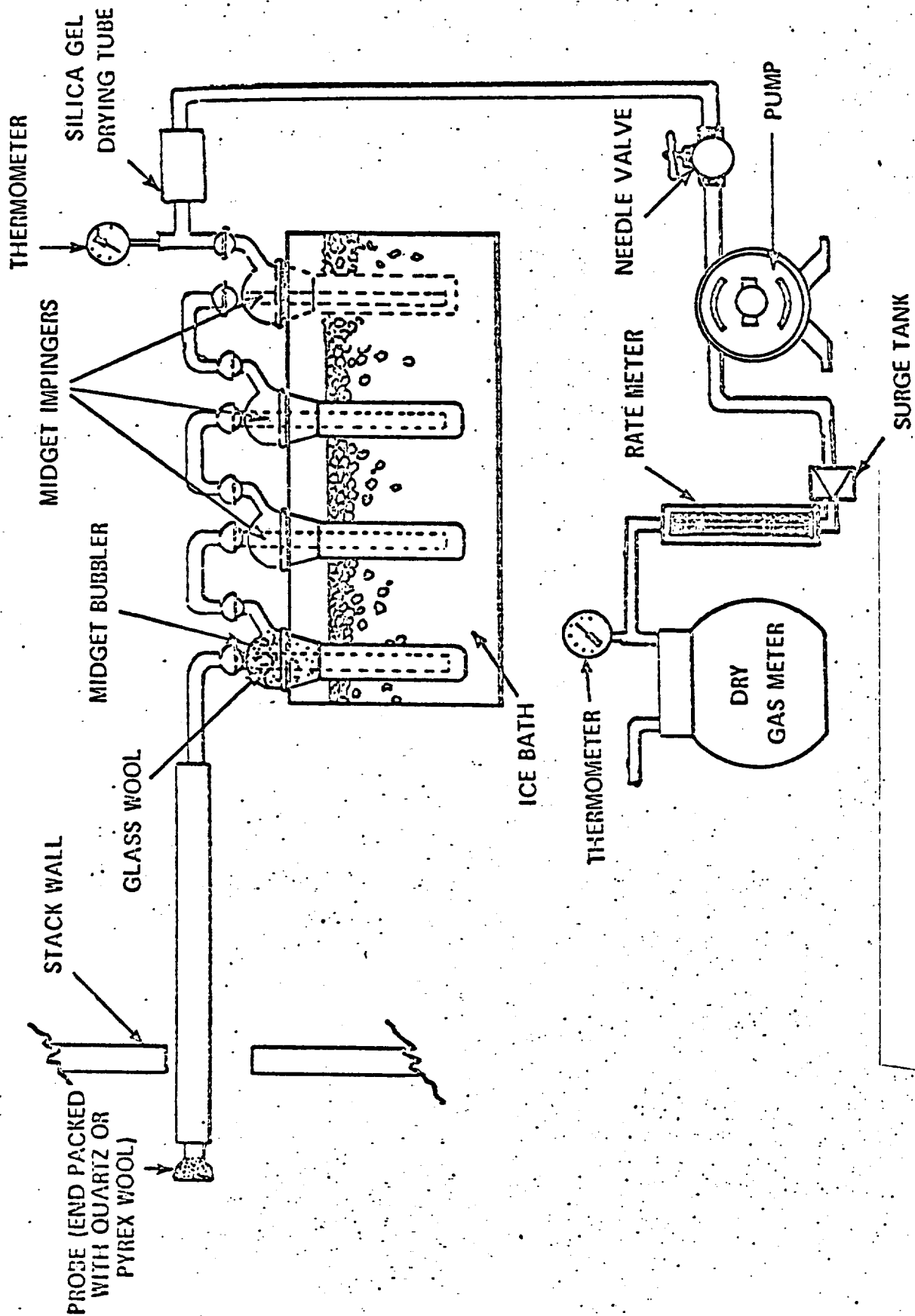


Figure 1, NH_3 sampling train.

2.1.2 Impingers. Four 30-ml midget impingers. The midget impingers must be connected in series with leak-free glass connectors. Silicone grease may be used, if necessary, to prevent leakage.

Other collection absorbers and flow rates may be used, but are subject to the approval of the Administrator. Also, collection efficiency must be shown to be at least 99 percent for each test run and must be documented in the report. If the efficiency is found to be acceptable after a series of three tests, further documentation is not required. To conduct the efficiency test, an extra absorber must be added ^{in series} and analyzed separately. This extra absorber must not contain more than 1 percent of the total NH_3 ^{mass}.

2.1.3 Glass Wool. Borosilicate or quartz.

2.1.4 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease may be used, if necessary.

2.1.5 Temperature Gauge. Dial thermometer, or equivalent, to measure temperature of gas leaving impinger train to within 1°C (2°F).

2.1.6 Drying Tube. Tube packed with 6- to 16-mesh indicating-type silica gel, or equivalent, to dry the gas sample and to protect the meter and pump. If the silica gel has been used previously, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to approval of the Administrator.

2.1.7 Valve. Needle valve, to regulate sample gas flow rate.

2.1.8 Pump. Leak-free diaphragm pump, or equivalent, to pull gas through the train. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.1.9 Rate Meter. Rotameter, or equivalent, capable of measuring flow rate to within 2 percent of the selected flow rate of about 1000 cc/min.

2.1.10 Volume Meter. Dry gas meter, sufficiently accurate to measure the sample volume within 2 percent, calibrated at the selected flow rate and conditions actually encountered during sampling, and equipped with a temperature gauge (dial thermometer, or equivalent) capable of measuring temperature to within 3°C (5.4°F).

2.1.11 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.12 Vacuum Gauge and Rotameter. At least 760 mm Hg (30 in. Hg) gauge and 0-40 cc/min rotameter to be used for leak check of the sampling train.

2.2 Sample Recovery.

2.2.1 Wash Bottles. Polyethylene or glass, 500 ml, two.

2.2.2 Storage Bottles. Polyethylene, 100 ml, to store impinger samples (one per sample).

2.3 Analysis.

2.3.1 Pipettes. Volumetric type 0.5-ml, 1-ml, 2-ml, 5-ml, 8.0-ml, 10.0-ml, 20-ml (one per sample), and 25-ml sizes.

2.3.2 Volumetric Flasks. 100-ml size (one per sample), 1000-ml size, and 25-ml size.

2.3.3 Graduated Cylinder. 100-ml size.

2.3.4 Spectrophotometer. To measure absorbance at 405 nanometers.

2.3.5 Sample Cells. Two matched absorbance cells to fit the spectrophotometer.

3. Reagents

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

3.1 Sampling.

3.1.1 Water. Deionized, distilled to conform to ASTM specification D1193-74, Type 3. At the option of the analyst, the KMnO_4 test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

3.1.2 Sulfuric Acid, 1.0 N. Dilute 28 ml of concentrated, ACS grade sulfuric acid to 1 liter with deionized, distilled water.

3.2 Sample Recovery.

3.2.1 Water. Deionized, distilled, as in 3.1.1.

3.2.2 Sulfuric Acid, 1.0 N. As in 3.1.2.

3.3 Analysis.

3.3.1 Water. Deionized, distilled, as in 3.1.1.

3.3.2 Anhydrous Mercuric Iodide (HgI_2). ACS grade.

3.3.3 Potassium Iodide (KI). ACS grade.

3.3.4 Sodium Hydroxide (NaOH). ACS grade.

3.3.5 Stock Standard Ammonium Chloride Solution. Dissolve 3.141 g of ammonium chloride (NH_4Cl) in 1.0N H_2SO_4 in a 1-liter volumetric flask and dilute to exactly 1 liter with 1.0N H_2SO_4 . One milliliter of this solution contains 1.0 mg of ammonia (NH_3).

3.3.6 Working Standard Ammonium Chloride Solution. Dilute 10 ml of the stock standard solution to 1 liter with 1.0 N H_2SO_4 in a 1-liter volumetric flask. One milliliter of this solution contains 10 μg of ammonia (NH_3).

3.3.7 Sodium Hydroxide, 10 N. Dissolve 40 grams of NaOH in a 100-ml volumetric flask and dilute exactly to 100-ml with deionized distilled water.

3.3.8 Nessler Reagent. Dissolve 160 g of NaOH in 500 ml of deionized distilled water in a 1-liter volumetric flask. Allow to cool. Dissolve 100 g of mercuric iodide and 70 g of potassium iodide in a small volume of deionized distilled water and while stirring add to the sodium hydroxide solution. Dilute to exactly 1 liter with deionized, distilled water. This reagent is stable up to 1 year.

4. Procedure

4.1 Sampling.

4.1.1 Preparation of Collection Train. Measure 15 ml of 1.0 N sulfuric acid into each of the first three midget impingers. Leave the final midget impinger dry. Assemble the train as shown in Figure 1. Adjust the probe heater to a temperature sufficient to prevent water condensation. Place crushed ice and water around the impingers.

4.1.2 Leak-check Procedure. A leak check prior to the sampling run is optional; however, a leak check after the sampling run is mandatory. The leak-check procedure is as follows:

Temporarily attach a suitable (e.g., 0-40 cc/min) rotameter to the outlet of the dry gas meter and place a vacuum gauge at or near the probe inlet. Plug the probe inlet, pull a vacuum of at least 250 mm Hg (10 in. Hg), and note the flow rate as indicated by the rotameter. A leakage rate not in excess of 2 percent of the average sampling rate is acceptable. Note: carefully release the probe inlet plug before turning off the pump.

It is suggested (not mandatory) that the pump be leak-checked separately, either prior to or after the sampling run. If done prior to the sampling run, the pump leak-check shall precede the leak check of the sampling train described immediately above; if done after the sampling run, the pump leak-check shall follow the train leak-check. To leak check the pump, proceed as follows: Disconnect the drying tube from the probe-impinger assembly. Place a vacuum gauge at the inlet to either the drying tube or the pump, pull a vacuum of 250 mm (10 in.) Hg, plug or pinch off the outlet of the flow meter and then turn off the pump. The vacuum should remain stable for at least 30 seconds.

4.1.3 Sample Collection. Record the initial dry gas meter reading and barometric pressure. To begin sampling, position the tip of the probe at the sampling point, connect the probe to the bubbler, and start the pump. Adjust the sample flow to a constant rate of approximately 1.0 liter/min as indicated by the rotameter. Maintain this constant rate (± 10 percent) during the entire sampling run. Take readings (dry gas meter, temperatures at dry gas meter and at impinger outlet and rate meter) at least every 5 minutes. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 20°C (68°F) or less. At the conclusion of each run, turn off the pump, remove probe from the stack, and record the final readings. Conduct a leak check as in Section 4.1.2. (This leak check is mandatory.) If a leak is found,

in excess of 2% of the avg sampling rate
 void the test run. Use procedures acceptable to the Administrator to adjust the sample volume for the leakage *below 2% of the avg sample rate.*

4.2 Sample Recovery. Disconnect the impingers after purging. Pour the contents of the midget impinger into a leak-free polyethylene bottle for shipment. Rinse the impinger and connecting glassware with deionized distilled water and add the washings to the same storage container. Mark the fluid level. Seal and identify the sample container.

4.3 Sample Analysis. Note the level of the liquid in the container and confirm whether or not any sample was lost during shipment; note this on the analytical data sheet. If a noticeable amount of leakage has occurred either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

Note level not measure volume

Quantitatively transfer the contents of the shipping container to a 1-liter volumetric flask. Rinse the container and cap with several portions of 1.0N sulfuric acid and transfer to the flask. Dilute to exactly 1 liter with 1.0N sulfuric acid. Pipet 10 ml of the sample from the 1-liter flask into a 500 ml-volumetric flask and dilute to exactly 500 ml with 1.0 N H_2SO_4 . Pipet 20 ml of this solution into a 25-ml volumetric flask. Add 10 N sodium hydroxide dropwise to the flask until the pH is between eight and ten. Then add 0.5 ml of Nessler reagent and dilute to exactly 25 ml with deionized distilled water. Mix well and allow to stand for the same amount of time as the standards used for calibration. Measure the absorbance at 405 nm using the blank solution as a zero reference. Dilute the sample and the blank with equal amounts of deionized distilled water if the absorbance exceeds that of the $100 \mu\text{g}$ NH_3 solution.

5. Calibration

5.1 Metering System.

5.1.1 Initial Calibration. Before its initial use in the field, first leak check the metering system (drying tube, needle valve, pump, rotameter, and dry gas meter) as follows: place a vacuum gauge at the inlet to the drying tube and pull a vacuum of 250 mm (10 in.) Hg; plug or pinch off the outlet of the flow meter, and then turn off the pump. The vacuum shall remain stable for at least 30 seconds. Carefully release the vacuum gauge before releasing the flow meter end.

Next, calibrate the metering system (at the sampling flow rate specified by the method) as follows: connect an appropriately sized

wet test meter (e.g., 1 liter per revolution) to the inlet of the drying tube. Make three independent calibration runs, using at least five revolutions of the dry gas meter per run. Calculate the calibration factor, Y (wet test meter calibration volume divided by the dry gas meter volume, both volumes adjusted to the same reference temperature and pressure), for each run, and average the results. If any Y value deviates by more than 2 percent from the average, the metering system is unacceptable for use. Otherwise, use the average as the calibration factor for subsequent test runs.

5.1.2 Post-Test Calibration Check. After each field test series, conduct a calibration check as in Section 5.1.1 above, except for the following variations: (a) the leak check is not to be conducted, (b) three, or more revolutions of the dry gas meter may be used, and (c) only two independent runs need be made. If the calibration factor does not deviate by more than 5 percent from the initial calibration factor (determined in Section 5.1.1), then the dry gas meter volumes obtained during the test series are acceptable. If the calibration factor deviates by more than 5 percent, recalibrate the metering system as in Section 5.1.1, and for the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

5.2 Thermometers. Calibrate against mercury-in-glass thermometers.

5.3 Rotameter. The rotameter need not be calibrated, but should be cleaned and maintained according to the manufacturer's instruction.

5.4 Barometer. Calibrate against a mercury barometer.

5.5 Determination of Spectrophotometer Calibration Factor K.

Add 0.0, 1.0, 2.0, 5.0, 8.0, and 10.0 ml of working standard ammonium chloride solution to a series of six 25-ml volumetric flasks. Adjust the total volume of solution in each to 20 ml using 1.0 N H_2SO_4 . Adding 10 N NaOH dropwise, adjust the pH to between 8 and 10. Pipette exactly 0.5 ml of Nessler reagent into each flask and dilute to exactly 25 ml with deionized distilled water. Mix well and allow each to stand for 10 to 30 minutes for color development. Note the time allowed for color development of the standards and use the same time for the samples. Measure the absorbance of each standard at 405 nm. The calibration procedure must be repeated each day that samples are analyzed. Calculate the spectrophotometer calibration factor as follows:

$$K_c = 100 \frac{A_1 + 2A_2 + 5A_3 + 8A_4 + 10A_5}{A_1^2 + A_2^2 + A_3^2 + A_4^2 + A_5^2}$$

Where:

K_c = Calibration factor.

A_1 = Absorbance of the 10 μg standard.

A_2 = Absorbance of the 20 μg standard.

A_3 = Absorbance of the 50 μg standard.

A_4 = Absorbance of the 80 μg standard.

A_5 = Absorbance of the 100 μg standard.

6. Calculation

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

6.1 Nomenclature.

A = Absorbance of sample.

C_{NH_3} = Concentration of ammonia dry basis corrected to standard condition, mg/dscm (lb/dscf).

F = Dilution factor (i.e., 25/5, 25/10, etc. required only if sample dilution was needed to reduce the absorbance into the range of calibration).

K_c = Spectrophotometer calibration factor.

m = Mass of ammonia in gas sample, μ g.

P_{bar} = Barometric pressure at the exit orifice of the dry gas meter, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

T_m = Average dry gas meter absolute temperature, $^{\circ}$ K ($^{\circ}$ R).

T_{std} = Standard absolute temperature, 293 $^{\circ}$ K (528 $^{\circ}$ R).

V_a = Volume of sample aliquot analyzed, ml.

V_m = Dry gas volume as measured by the dry gas meter, dcm (dcf).

$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

V_{soln} = Total volume of solution in which the ammonia sample is contained, 1000 ml.

Y = Dry gas meter calibration factor.

6.2 Dry sample gas volume, corrected to standard conditions:

$$V_{m(std)} = V_m Y \frac{T_{std}}{T_m} \frac{P_{bar}}{P_{std}} = K_1 Y \frac{V_m P_{bar}}{T_m}$$

Equation 2

Where:

$$K_1 = 0.3858 \text{ } ^\circ\text{K/mm Hg for metric units.}$$

$$= 17.64 \text{ } ^\circ\text{R/in. Hg for English units.}$$

6.3 Total $\mu\text{g NH}_3$ per sample.

$$m = 2 K_c AF \frac{V_a}{V_{\text{soln}}} \quad \text{Equation 3}$$

6.4 Sample concentration, dry basis, corrected to standard condition:

$$C = K_2 \frac{m}{V_{\text{sc}}}$$

Where:

$$K_2 = 10^3 \frac{\text{mg/m}^3}{\mu\text{g/ml}} \text{ for metric units.}$$

$$= 6.243 \times 10^{-5} \frac{\text{lb/scf}}{\mu\text{g/ml}} \text{ for English units.}$$

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APPENDIX K-3
EPA DRAFT METHOD FOR FORMALDEHYDE

TENTATIVE METHOD FOR
ISOKINETIC DETERMINATION OF POLLUTANT LEVELS
IN THE EFFLUENT OF FORMALDEHYDE MANUFACTURING FACILITIES

1. Principle:

1.1 General: An air sample is drawn isokinetically through an impinger train containing water as the scrubbing medium. Formaldehyde methanol and dimethyl ether are scrubbed from the gas. A glass bomb is connected after the scrubbing impingers and before the silica gel so that any non-condensable pollutants may be collected in a grab sample.

1.2 Formaldehyde: The analysis consists of reacting an aliquot of the impinger solution with chromotropic - sulfuric acid reagent to form a purple chromogen. The resulting solution is analyzed colorimetrically using a spectrophotometer at 580 nm; the absorbance of the colored solution is proportional to the quantity of formaldehyde in the solution.

1.3 Methanol: An aliquot of the scrubber solution is reacted with potassium permanganate oxidizing all methanol present to formaldehyde. The total formaldehyde is then determined colorimetrically. The background formaldehyde content as determined by (1.2) is then subtracted out and the methanol content determined.

1.4 Dimethyl ether: An aliquot of the scrubber solution is analyzed for dimethyl ether using a gas chromatograph with a flame ionization detector.

1.5 Grab sample: Using a Hamilton syringe, 20 ml of water is injected into the glass bomb. The bomb is shaken and the liquid removed and analyzed for methanol, formaldehyde, and dimethyl ether to check impinger efficiency. A sample of the remaining gas is analyzed for dimethyl ether.

2. Applicability:

2.1 This method is applicable for the determination of formaldehyde, methanol and dimethyl ether in the effluent of formaldehyde manufacturing facilities.

3. Range:

3.1 Formaldehyde: .05 µg/ml - 2.0 µg/ml; based on impinger solution of 600 ml and 60 Ft³ gas collected: 6 - 240 ppm; upper limit is easily extended by diluting aliquot taken.

4. Sensitivity: unknown

5. Precision:

5.1 Formaldehyde: ± 5%

6. Collection Efficiency

6.1 Formaldehyde 95%

7. Interferences

7.1 Formaldehyde: This method is specific for formaldehyde although other hydrocarbons in concentrations in excess of formaldehyde to the order of 10:1 will give interferences in absorbance readings:

Saturated Aldehydes	<.01%	(+)
Unsaturated Aldehydes	1 - 2%	(+)
Ethanol, High Alcohols, Olefins		(-)
Phenols (8:1 excess)	10-20%	(-)
Ethylene, Propylene (10:1 excess)	5 - 10	(-)
Aromatics (15:1 excess)	15%	(-)
Methanol (10,000:1 excess)	None	
Nitrogen Oxides*		(-)

*Use of Aqueous bisulfite solution as the scrubbing medium will reduce interference of nitrogen oxides.

7.2 Methanol: same as above

7.3 Dimethyl ether: unknown

8. Apparatus:

8.1 Sampling:

8.1.1 Stainless steel nozzle

8.1.2 Pyrex probe - heated

8.1.3 Pitot tube: s - type

8.1.4 Glass impingers: 2 Greenburg-Smith, 1 modified Greenburg-Smith, 1 silica gel.

8.1.5 Glass sample tube with side adapter for syringe; 250 ml. (Fisher Catalog #11-134-190)

8.1.6 Metering - Vacuum System as required to maintain an isokinetic sampling rate.

8.1.7 Metering - Vacuum System as required to obtain grab sample.

8.2 Sample recovery

8.2.1 Probe brush

8.2.2 Wash bottle

8.2.3 Graduated cylinder

8.2.4 Glass sample storage jars

8.3 Analysis

8.3.1 Spectrometer capable of measuring absorbance of the color developed solution at 580 nm.

8.3.2 Hamilton syringe for removal of sample from grab sample bomb

8.3.3 Gas chromatograph

8.3.4 Flame ionization detector

8.3.5 Recorder

9. Reagents:

9.1 Sampling

9.1.1 Distilled water

9.1.2 Silica gel

9.1.3 Crushed ice

9.2 Sample recovery

9.2.1 Distilled water

9.3 Analysis: Formaldehyde

9.3.1 Chromotropic acid reagent: Dissolve 0.10 g of 4,5 dihydroxy-2, 7 - naphthalene-disulfonic acid disodium salt (Eastman Kodak Co. Cat. No. P230) in water and dilute to 10 ml. Filter, if necessary: store in brown bottle. Make fresh weekly.

9.3.2 Sulfuric acid: Concentrated reagent grade

9.3.3 Formaldehyde standard solution "A": (1 mg/ml). Dissolve 4.4703 g sodium formaldehyde bisulfite (Eastman PG 450) in distilled water and dilute to 1 liter. Stable for one month.

9.3.4 Formaldehyde standard solution "B": (1 µg/ml) Dilute 1 ml of standard solution "A" to 100 ml with distilled water. Make fresh daily.

9.3.5 Iodine (0.1 N, approximate): Dissolve 25 g of potassium iodide in about 25 ml of water. Add 12.7 g of iodine and dilute to 1 liter.

9.3.6 Iodine (0.01 N): Dilute 100 ml of the 0.1 N iodine solution to 1 liter. Standardize against sodium thiosulfate.

9.3.7 Starch solution, 1 percent: Make a paste of 1 g of soluble starch and 2 ml of water. Slowly add the paste to 10 ml of boiling water. Cool, add several ml of chloroform as a preservative, and store in a stoppered bottle. Discard when a mold growth is noticeable.

9.3.8 Sodium carbonate buffer solution: Dissolve 80 g of anhydrous sodium carbonate in about 500 ml of water. Slowly add 20 ml of glacial acetic acid and dilute to 1 liter.

9.3.9 Sodium bisulfite, 1 percent: Dissolve 1 g of sodium bisulfite in 100 ml of water. Prepare fresh weekly.

9.4 Analysis: Methanol

9.4.1 Same as formaldehyde analysis (9.3) plus:

9.4.2 Potassium permanganate solution: Dissolve 1 g A.R. potassium permanganate in water and dilute to 100 ml with water.

9.4.3 Ethanol solution: Prepare a 5 percent (volume) solution of methanol - free ethanol in water.

9.4.4 Dilute phosphoric acid: Dilute 25 ml phosphoric acid (85%) to 100 ml with water.

9.4.5 Hydrogen peroxide solution: Prepare a solution containing approximately 1.5 percent w/v H_2O_2 (5 - volumes peroxide).

9.5 Analysis: Dimethyl ether

9.5.1 Chromatographic column: 10% triethyl acetyl citrate.

10. Procedure:

10.1 Sampling

10.1.1 The sample train is assembled as shown in Figure 4. Each of the two impingers (Greenburg-Smith) is filled with 100 ml distilled water. The third impinger is left dry and the fourth impinger contains approximately 200 gm silica gel.

10.1.2 A minimum sample of 60 Ft^3 is collected isokinetically as per EPA Method 5 at a rate of 0.5 to 1.0 CFM.

10.1.3 Halfway through the sample run the valve to the glass bomb is opened and the glass bomb is purged at a rate of 1 LPM for two minutes. The stopcocks at both ends of the gas sample tube are simultaneously closed. The vacuum source to the sample tube and the valve to the main sample train are closed off.

10.2 Sample Recovery

10.2.1 The gas sample tube is removed from the sample train and stored.

10.2.2 The liquid from each impinger is stored in a separate sample collection jar.

10.2.3 The probe and impingers are sparingly washed with water (It is important to dilute the sample as little as possible) and the wash from each impinger is added to the sample collection jar for that impinger. The probe wash is stored separately.

10.2.4 The weight gain in the silica gel is recorded.

10.3 Analytical: Formaldehyde

10.3.1 Measure and record the volume of each of the sample solutions.

10.3.2 Pipette a 4 ml aliquot from each of the sampling solutions into glass stoppered test tubes. A blank containing 4 ml of distilled water must also be run. (If the formaldehyde content of the aliquot exceeds the limit of the method a smaller aliquot diluted to 4 ml with distilled water is used.)

10.3.3 Add 0.1 ml of 1 percent chromotropic acid reagent to the solution and mix.

10.3.4 To the solution pipette slowly and cautiously 6 ml of concentrated sulfuric acid. The solution becomes extremely hot during the addition of the sulfuric acid. If the acid is not added slowly, some loss of sample could occur due to spattering.

10.3.5 Allow to cool to room temperature. Read at 580 nm in a suitable spectrophotometer using a 1 cm cell.

10.3.6 Determine the formaldehyde content of the sampling solution from a curve previously prepared from standard formaldehyde solutions.

10.4 Analysis: Methanol

10.4.1 Pipette a 4 ml aliquot from each of the sampling solutions into glass stoppered test tubes. A blank containing 4 ml of distilled water must also be run. (If the methanol content exceeds the limit of the method a smaller aliquot diluted to 4 ml with distilled water is used).

10.4.2 Add .5 ml ethanol solution, 2.5 ml potassium permanganate solution, and .5 ml phosphoric acid solution. Mix and allow to stand for 1 hour.

10.4.3 Add hydrogen peroxide solution drop by drop until the solution is colorless.

10.4.4 Proceed with formaldehyde analysis (10.3.3)

10.5 Analysis: Dimethyl ether

10.5.1 Using Hamilton syringe take aliquot of sample solutions and inject into gas chromatograph.

10.6 Analysis: Gas sampling tube

10.6.1 Using Hamilton syringe inject 20 ml of distilled water into the sampling tube. Swirl and shake for 15 minutes.

10.6.2 Remove two 4 ml aliquots using syringe and analyze for formaldehyde and methanol using the already mentioned procedures.

10.6.3 Remove two samples, one liquid and one gas, using the Hamilton syringe and analyze for dimethyl ether by gas chromatography.

11. Calibration:

11.1 Standardization of formaldehyde solution

11.1.1 Pipette 1 ml of formaldehyde standard solution "A" into an iodine flask. Into another flask pipette 1 ml of distilled water. This solution serves as the blank.

11.1.2 Add 10 ml of 1 percent sodium bisulfite and 1 ml of 1 percent starch solution.

11.1.3 Titrate with 0.1 N iodine to a dark blue color.

11.1.4 Destroy the excess iodine with 0.05 N sodium thiosulfate.

11.1.5 Add 0.01 N iodine until a faint blue end point is reached.

11.1.6 The excess inorganic bisulfite is now completely oxidized to sulfate, and the solution is ready for the assay of the formaldehyde bisulfite addition product.

11.1.7 Chill the flask in an ice bath and add 25 ml of chilled sodium carbonate buffer. Titrate the liberated sulfite with 0.01 N iodine, using a microburette, to a faint blue end point. The amount of iodine added in this step must be accurately measured and recorded.

11.1.8 One ml of 0.0100 N iodine is equivalent to 0.15 mg of formaldehyde. Therefore, since 1 ml of formaldehyde standard solution was titrated, the ml of 0.01 N iodine used in the final titration multiplied by 0.15 mg gives the formaldehyde concentration of the standard solution in mg/ml.

11.2 Preparation of standard curve, formaldehyde

11.2.1 Pipette 0, 0.1, 0.3, 0.5, 0.7, 1.0, and 2.0 ml of standard solution "B" into glass stoppered test tubes.

11.2.2 Dilute each standard to 4 ml with distilled water.

11.2.3 Develop the color as described in the analytical procedure (10.3) .

11.2.4 Plot absorbance against micrograms of formaldehyde in the color developed solution.

12. Calculations:

12.1 Formaldehyde

12.1.1 Correct the volume of air sampled to the volume at standard conditions.

$$V_s = V \times \left(\frac{P - P_m}{29.92} \right) \times \left(\frac{530}{T + 460} \right)$$

12.1.2 Calculate concentration of formaldehyde in the sample.

$$\text{ppm (volume)} = \frac{(C) \times (S) \times (24.15)}{(2) \frac{(V_s)}{(MW)}}$$

V	=	Volume Sampled, (Liters)
V _s	=	Volume S.T.P. (Liters)
S.T.P.	=	70°F, 29.92"hg
P	=	Barometric Pressure, "Hg
P ^m	=	Meter Pressure, "hg
T ^m	=	Meter Temp., °F
C	=	μg of formaldehyde in aliquot (from calibrated curve)
S	=	Total ml of sampling solution
A	=	ml of aliquot taken from sampling solution
MW	=	Molecular weight of formaldehyde, 30.03
24.15	=	ml of formaldehyde gas in one millimole @ S.T.P.

12.2 Methanol

12.2.1 The total μg formaldehyde read from the absorbance is equal to the formaldehyde originally in the sample plus the formaldehyde formed from oxidation of methanol. Therefore, from the total μg formaldehyde in the aliquot, subtract the μg of background formaldehyde present in a aliquot of equal size (previously determined).

This is the μg formaldehyde in the aliquot formed from the oxidation of methanol.

12.2.2

$$M = FM \left(\frac{30.03}{32.04} \right)$$

12.2.3

$$M_c = \frac{(M) (S) (24.15)}{(A) (V_s) (MW)}$$

Where:	M	=	Methanol content, μg
	M _c	=	Methanol concentration of air sample (ppm)
	F _m	=	Formaldehyde from oxidation of methanol, μg
	S	=	Total volume sample solution, ml
	A	=	Aliquot taken from sample solution, ml
	V _s	=	Air sample volume @ S.T.P., liters
	M _w	=	Molecular weight of methanol, 32.04

S.T.P. = 70°F, 29.92 "Hg

24.15 = ml of methanol gas in one millimole @ S.T.P.

12.3 Dimethyl ether

12.3.1 Not completed yet.

13. Major References:

13.1 Cares, Janet Walker; "Determination of Formaldehyde by the Chromotropic Acid Method in the Presence of Oxides of Nitrogen;" Amer. Ind. Hyg. Jour; July, 1968.

13.2 "Determination of Formaldehyde: Chromotropic Acid Method," PHS Standard Methods.

13.3 "Tentative Method of Analyses for Formaldehyde Content of the Atmosphere (Colorimetric Method);" Health Life Science Journal;" Vol. 7 #1; January, 1970.

13.4 Walker, J.F.; Formaldehyde; Reinhold Publishing Co.: 3rd Edition; 1964.

13.5 Federal Register, Volume 36, Number 247, Part II, December 23, 1971.

13.6 "Method for the Determination of Toxic Substances in Air: Methanol (Adopted 1949); International Union of Pure and Applied Chemistry, London, 1959.

APPENDIX K-4

DISCUSSION OF ANALYTICAL PROCEDURES

Discussion of Analysis Procedures

Standard Methods for the Examination of Water and Wastewater, 14th edition, contains the methods followed for direct and distilled ammonia analyses and for Kjeldahl nitrogen determination. The following is the basic procedure taken from the text with the modifications included. Example calculations are shown at the end of this appendix for all the methods.

I Direct Ammonia

1. Pipet an aliquot of sample into a 50 ml. nessler tube.
2. If 1 ml. of nessler reagent is not sufficient to raise the pH to >10, adjust the pH of the aliquot to 8-10 before diluting to 50 ml. Alternatively, add 2 ml. of nessler reagent to the sample and standards.
3. To the sample diluted to 50 ml., add 1 ml. of nessler reagent and mix thoroughly.
4. Allow the color to develop for a minimum of 10 minutes. Then read the absorbance on a spectrophotometer at 405 nm.
5. A series of standards is made up in 50 ml. nessler tubes to contain 0, 20, 40, 100, 160, and 200 $\mu\text{g/ml}$. NH_3 is made up of 3.141 g $\text{NH}_4\text{A}/\text{lH}_2\text{O}$.
6. A plot of total μgNH_3 vs. absorbance is prepared from the standards, and equivalent μg for the samples is read from it.
7. Calculations are listed at the end of this appendix.

II Ammonia and Kjeldahl Nitrogen Distillation and Digestion for Nesslerization

1. To a clean Kjeldahl flask, add ~ 300 ml. H_2O , 25 ml. borate buffer, and a few ml. of 1 N NaOH plus a few glass beads.
2. Connect flask to distillation apparatus and distill over 100-200 ml. to clean the set-up.
3. Meanwhile, pipet an aliquot of sample into a 400 ml. beaker. (Use 25.0 ml. for the outlet stack samples and 5.0 ml. for the inlet stack samples and the scrubber water samples.)

4. Add ~ 350 ml. H_2O and 25 ml. borate buffer. Check the pH with pH paper. If necessary, add 1 N $NaOH$ to adjust the pH to 9.5.
5. Prepare a collection volumetric flask by adding 50 ml. boric acid solution to a 250 ml. volumetric flask.
6. After the distillation apparatus is clean, disconnect the Kjeldahl flask. Place a delivery tube under the surface of the boric acid solution in the 250 ml. volumetric flask.
7. Pour out the water in the flask. Then carefully transfer the sample in the beaker into the Kjeldahl flask. Connect to the still and turn on the heat.
8. When ~ 3/4 of the flask is full, raise delivery tube above the surface of the sample. Continue to distill until it is almost full. Remove the 250 ml. volumetric flask and shut off the still.
9. Dilute the sample to 250.0 ml. Use this for the distilled NH_3 analysis.
10. Add 50.0 ml. of the digestion solution to the remaining residue in the Kjeldahl flask.
11. Digest this solution under a hood for ~ 1½ hours.
12. Allow to cool.
13. Carefully add ~ 400 ml. H_2O to the Kjeldahl flask.
14. Prepare a collection flask as in step 5. Place delivery tube under surface of the boric acid.
15. Add 50 ml. hydroxide - thiosulfate solution down the side of the Kjeldahl flask. Do not shake or stir.
16. Connect the Kjeldahl flask to the still. Swirl to mix.
17. Distill over as in step 8.
18. Dilute to 250.0 ml. Use this solution for the urea analysis.
19. Follow steps 5-7 under Direct NH_3 for the preparation of standards and the calibration plot.

III Formaldehyde

The analytical procedure listed in the EPA Method "Isokinetic Determination of Pollutant Levels in the Effluent of Formaldehyde Manufacturing Facilities" was followed. A copy of this method is provided in Appendix K-3.

Calculations:

1. Direct Ammonia

a. stack solutions
$$\text{total mg NH}_3 = \frac{A-B}{C^*} \times F \times 0.001 \frac{\text{mg}}{\mu\text{g}}$$

b. scrubber solutions
$$\text{mg/ml NH}_3 = \frac{A-B}{C^*} \times 0.001 \frac{\text{mg}}{\mu\text{g}}$$

2. Distilled Ammonia

a. stack solution
$$\text{total mg NH}_3 = \frac{A-B}{C} \times \frac{D}{E} \times F \times 0.001 \frac{\text{mg}}{\mu\text{g}}$$

b. scrubber solutions
$$\text{mg/ml NH}_3 = \frac{A-B}{C} \times \frac{D}{E} \times 0.001 \frac{\text{mg}}{\mu\text{g}}$$

3. Kjeldahl Urea

a. stack solutions
$$\text{total mg urea} = \frac{A-B}{C} \times \frac{D}{E} \times F \times 1.765 \times 0.001 \frac{\text{mg}}{\mu\text{g}}$$

b. scrubber solutions
$$\text{mg/ml urea} = \frac{A-B}{C^*} \times \frac{D}{E} \times 1.765 \times 0.001 \frac{\text{mg}}{\mu\text{g}}$$

4. Formaldehyde

a. stack solution
$$\text{total mg CH}_2\text{O} = \frac{A}{C} \times F \times 0.001 \frac{\text{mg}}{\mu\text{g}}$$

b. scrubber solutions
$$\text{ug/mg CH}_2\text{O} = \frac{A}{C}$$

Codes:

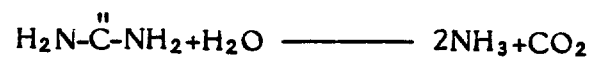
A = μg of NH_3 (or formaldehyde) equivalent to absorbance of sample (from plot of standards vs. absorbance)

B = reagent blank correction = $\frac{A \text{ Blank}}{C \text{ Blank}} \times C \text{ sample}$

C = aliquot of sample or distillate analyzed, ml.

D = volume of distillate, ml.

- E = aliquot of sample digested, ml.
- F = total volume of sample in ml.
- 1.765 = gravimetric factor to convert μgNH_3 to μg urea.
It is based on the following chemical equation



$$\frac{60\text{g urea}}{\text{mole urea}} \longrightarrow \frac{34\text{ grams NH}_3}{2\text{ moles NH}_3}$$

APPENDIX K-5

MOISTURE DETERMINATION PROCEDURE

Moisture determinations of the scrubber gas stream were straightforward matter at the scrubber outlet sampling point. Here the impinger weight gain was essentially all due to water collected during each run.

At the scrubber inlet, however, urea comprised most of the impinger weight gain. The laboratory sample analysis methodology consisted of determining total urea from impingers and probe wash while determining impinger weight gain from only the impinger contents. This methodology precluded simply subtracting total urea from impinger weight gain to obtain water gain. For this reason, and because water was used to clean nozzles and probes plugged by urea particulate (see Section 5), a separate moisture run was performed at the scrubber inlet. Urea was excluded from the impinger catch by use of a simple fiberglass plug in-stack filter arrangement. Ammonia collected in the impingers was a negligible fraction of the water weight collected in this moisture run.

Figure K-1 shows the impinger weight gain data for scrubber gas runs 1 - 3 and Figure K-2 shows the results of the inlet moisture run. Figure K-3 shows the calculated collected water volumes for each scrubber inlet run, assuming the inlet gas stream contained 2.3% water (as determined by the separate moisture run).

FIGURE K-1: SCRUBBER INLET/OUTLET TESTS
SAMPLE WEIGHT GAIN DATA

Test # Acid (A) Inlet (I) Container #	Silicagel (S) Water (W) Outlet (O) Key	Final weight (container tare + charge + H ₂ O) gm	Initial wt., (container tare), gm	Charge wt., gm		Weight gain, gm
Container*		2	3	4	5	6
1-S-I		256.43	244.54*	--		11.89
1-A-I		526.35	327.07	204.8		-5.52
1-W-I		604.00	327.70	200		76.3
					total	82.67
1-S-O		264.48	242.45*			22.03
1-A-O		527.79	325.08	204.8		-2.09
1-W-O		634.64	327.28	200		107.36
					total	127.3
2-S-I		256.52	242.68*	--		13.84
2-A-I		518.87	327.40	204.8		-13.33
2-W-I		589.00	329.09	200-		59.91
					total	60.42
2-S-O		263.89	243.34*	--		20.55
2-A-O		530.88	327.66	204.8		-1.08
2-W-O		641.36	328.18	200		113.18
					total	132.65
3-S-I		258.88	245.33*	--		13.55
3-A-I		475.47	327.83	204.8		-57.16
3-W-I		596.44	326.25	200		70.19***
					total	26.58
3-S-O		261.74	242.88*	--		18.86
3-A-O		531.72	326.23	204.8		0.69
3-W-O		534.86	325.29	100**		109.57
					total	129.12

*includes 200gm silica gel change

**No H₂O in impinger 2 for test 3=0

***H₂SO₄ impinger sample sucked back to H₂O impinger

FIGURE K-2. % MOISTURE TEST

Firm Name CF Industries Test # Moisture TRC Project # 82988-20

Unit Tested Granulator Scrubber Inlet Date 1/18/79 1615-1649

V_m (final-initial dry gas meter reading, ft^3) = 30.34

P_{bar} (barometric pressure, "Hg) = 30.27

ΔH (average orifice pressure drop, "H₂O) = 2.5

T_m (average dry gas meter gas temperature, °F) = 80.5

$$V_m @ \text{STP} = 17.65 V_m \left(\frac{P_{\text{bar}} + \frac{\Delta H}{13.6}}{T_m + 460} \right) = 17.65 \times 30.34 \left(\frac{30.27 + \frac{2.5}{13.6}}{80.5 + 460} \right)$$

$V_m @ \text{STP}' = 30.17 \text{ ft}^3$ corrected to 68°F and 29.92 "Hg.

V_I (total liquid collected in impingers (ml) plus silica gel (gms)) = 14.96

$$\% \text{H}_2\text{O} = \frac{4.71 V_I}{V_m @ \text{STP} + 0.0471 V_I} = \frac{4.71 \times 14.96}{30.17 + 0.0471 \times 14.96}$$

$\% \text{H}_2\text{O} = 2.28 \%$

CO_2 (% CO₂ measured by Orsat or Fyrite, %) =

O_2 (% O₂ measured by Orsat or Fyrite, %) =

CO (% CO measured by Orsat, %) =

N_2 (100 - %CO₂ - % O₂ - % CO) % =

$$M_s = 0.44 (\% \text{CO}_2) + 0.32 (\% \text{O}_2) + 0.28 (\% \text{N}_2 + \% \text{CO}) \left(1 - \frac{\% \text{H}_2\text{O}}{100} + 0.18 (\% \text{H}_2\text{O}) \right)$$

$$M_s = 0.44 () + 0.32 () + 0.28 (+) \left(1 - \frac{ }{100} + 0.18 () \right)$$

$M_s =$ lbs/lb-mole

AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

$$\% \text{H}_2\text{O} = \frac{4.71 (V_{\text{imp}} + V_{\text{sg}})}{V_{\text{m}} @ \text{STP} + 0.0471 (V_{\text{imp}} + V_{\text{sg}})}$$

Test 1 Silica Gel = 11.9g %H₂O = 2.3%
(from moisture run)

$$2.3 = \frac{(4.71)(V_{\text{imp}} + 11.9)}{(63.18) + 0.0471(V_{\text{mp}} + 11.9)}$$

$$2.3 = \frac{4.71 \times 4.71 (11.9)}{63.18 + 0.0471 x + (0.0470) (11.9)}$$

$$2.3 (63.18) + 2.3 (0.0471)X + 2.3 (0.0471) (11.9) = 4.71x + 4.71 (11.9)$$

$$x \cdot 2.3 (0.0471) - 4.71 = (4.71) (11.9) - 2.3 (63.18) - 2.3 (0.0471) (11.9)$$

$$x = \frac{(4.71)(11.9) - 2.3 \cdot 63.18 - 0.0471(11.9)}{2.3(0.0471) - 4.71}$$

$$x = 19.1 \text{ ml}$$

$$\text{Test 2} \quad x = \frac{(4.71)(13.8) - 2.3 \ 65.97 - 0.0471 (13.8)}{2.3 (0.0471) - 4.71}$$

$$x = 18.5 \text{ ml}$$

$$\text{Test 3} \quad x = \frac{(4.71)(13.5) - 2.3 \ 66.45 - 0.0471(13.5)}{2.3(0.0471) - 4.71}$$

$$x = 19.1 \text{ ml}$$

APPENDIX L

CLEANUP EVALUATION RESULTS

APPENDIX L

Prior to sampling, the impingers used for the scrubber inlet, scrubber outlet, and the synthesis tower tests at CF Industries, Donaldsonville, Louisiana, were rinsed with deionized distilled water and then charged with water or acid solution as called for in the text procedure. These impinger contents were then analyzed for urea, ammonia, and formaldehyde to determine if there was any contamination from the glassware. A blank deionized distilled water sample was also analyzed similarly. The results of these analyses are presented in Table L-1. These results are corrected for the water blank value. This table shows the absolute level, in milligrams, of each contaminant, as well as the relative level in percent compared to the test result levels.

The associated laboratory summary sheets on the cleanup results are presented in Table L-2 and Table L-3 of this appendix. Raw laboratory data are contained in Appendix J.

TABLE L-1

RESULTS OF ANALYSES ON CLEAN IMPINGER WASHES
WITH RELATIVE PERCENT VALUES FROM TEST DATA

<u>INLET SAMPLES (mg.)</u>	<u>CLEANUP BLANK</u>	<u>RUN 1</u>	<u>RUN 2</u>	<u>RUN 3</u>	<u>RELATIVE %</u>
NH ₃ - Direct	0.11	410.	441.	506.	<0.027
NH ₃ - Distilled	0.22	888.	941.	944.	<0.025
Urea	4.17	23,920.	26,090.	26,258.	<0.017
Formaldehyde	0.024	2.476	1.545	1.341	<1.8
<u>Outlet Samples (mg.)</u>					
NH ₃ - Direct	0.157	450.	694.	387.	<0.04
NH ₃ - Distilled	0.65	438.	426.	388	<.17
Urea	3.14	70.59	73.09	62.27	<5.
Formaldehyde	0.023	2.523	1.172	1.296	<2.
<u>Synthesis Tower Samples (mg.)</u>					
NH ₃ - Direct	0.157	37,721.	45,839.	57,814.	<0.0004
NH ₃ - Distilled	0.65	37,924.	48,618.	58,232.	<0.002
Urea	3.14	188.5	197.9	185.4	<1.7
Formaldehyde	0.023	--	--	--	--

TABLE L-2

INLET

Clean-up Evaluation Results

<u>INLET SAMPLES</u>	<u>BLANK ID</u>	<u>VOLUME</u>	<u>µg/ml CONC.</u>	<u>TOTAL MG</u>	
Direct NH ₃	BWI	200	0.20 µg/ml vis ~.2	0.04	mg NH ₃ Direct-Inlet
	BAI	200	cloudy vis ~.1	0.04	
	BPWI	322	cloudy	<u>0.03</u> 0.11	
Distilled NH ₃	BWI	200	0.60	0.12	mg NH ₃ Distilled-Inlet
	BAI	200	0.50 vis ~0	0.10	
	BPWI	322	cloudy	<u>~0</u> 0.22	
Urea	BWI	200	4.24	0.848	mg Urea
	BAI	200	3.09	0.618	
	BPWI	322	8.39	<u>2.70</u> 4.17	
Formaldehyde	BWI	200	<.05		mg Formaldehyde
	BAI	200	<.05		
	BPWI	322	0.075	<u>0.024</u> 0.024	

TABLE L-3

OUTLET

Clean up Evaluation Results

<u>Outlet Samples</u>	<u>Blank ID</u>	<u>Volume</u>	<u>µg/ml Conc.</u>	<u>Total mg</u>	
Direct NH ₃	BWO	200	0.12	0.024	mg NH ₃ Direct
	BAO	200	0.20	0.040	
			cloudy vis ~.36		
	BPWO	259	cloudy	0.093 <u>0.157</u>	
Distilled NH ₃	BWO	200	0.52	0.104	mg NH ₃ Distilled
	BAO	200	0.60	0.12	
				0.43	
	BPWO	259	1.65	<u>0.65</u>	
Urea	BWO	200	2.82	0.564	mg Urea
	BAO	200	1.94	0.388	
				2.19	
	BPWO	259	8.47	<u>3.14</u>	
Formaldehyde	BWO	200	<.05		mg Formaldehyde
	BAO	200	<.05		
				0.023	
	BPWO	259	0.088	<u>0.023</u>	

APPENDIX M
PROJECT PARTICIPANTS

Project Participants

CF Industries, Inc.

Donaldsonville, Louisiana

January 15-19, 1979

TRC

Willard A. Wade, III, Senior Project Manager

Reed W. Cass, Project Engineer - Crew Chief

John Kirzec, Senior Technical Specialist - Team Leader

Steven Richardson, Senior Technical Specialist - Team Leader

David Ringquist, Environmental Engineer - Project Engineer/Team Leader

Denise A. Kearns, Technical Specialist - Team Member

James Canora, Technical Specialist - Clean Up

Carl S. Ekroth, Senior Technical Specialist - Visible Emissions

Margaret Fox, Assistant Project Scientist - Chemist

GCA

Stephen A. Capone

Tim Curtin

EPA Emission Measurement Branch

Clyde E. Riley, Technical Manager

EPA Industrial Studies Branch

Eric A. Noble - Test Process Project Engineer

APPENDIX N

WORK ASSIGNMENT AND TECHNICAL DIRECTIVES

- N-1 Copy of Project Work Assignment
- N-3 Copy of Project Technical Directives

APPENDIX N-1

COPY OF PROJECT WORK ASSIGNMENT

68-02-2820

2015.05.15

C.-F. Industries

Company Location:
Donaldsonville, LA

78-NHF-0

See Figure 1

[illegible]

REVIEWS

1. Sampling shall be performed with $\pm 10\%$ isokinetic conditions.

Industry: Urea Manufacturing

Plant: C.F. Industries, Donaldsonville, Louisiana

Process Step: Drum Granulator

Rationale for Selection:

1. This plant is representative of other urea drum granulators and is state of the art for granulators.
2. There are seven drum granulators at this facility. Three are located on one urea solution line and 4 newer units are located on a second urea solution line. The first three units each have separate stacks. The last 4 are combined so that 2 units are vented through one stack and the remaining 2 units are vented through a second stack. It is preferred that the newer vents be tested, however if the duct work does not allow for easy sampling, one of the first three units can be tested. Test only B or C granulator of the first three units.
3. Test can be performed in conjunction with others at this plant.

Comments:

1. Grab samples of the scrubber liquor inlet and outlet should be taken several times during the test. Temperature and pH readings should be taken at the time the solution is sampled. Analyses on the composite sample should include % NH_3 , % urea, ~~and~~ % solids ~~and~~ % Formaldehyde.
2. Test during maximum production rate of the granulator.
3. A grab sample of the refrigerated air should be checked for temperature and moisture.
4. All solid samples will be highly hygroscopic.

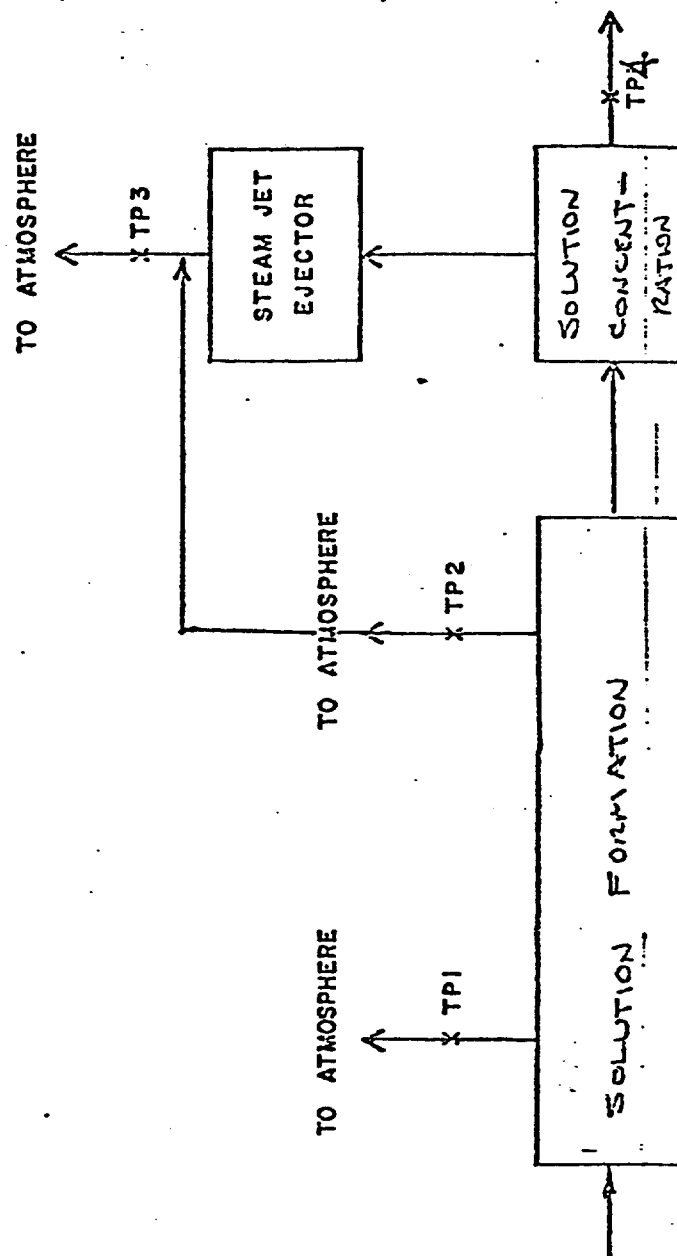


Figure 1. Solution Formation, Urea, C.F. Industries, Donaldsonville, La.

SOURCE SAMPLING AND ANALYSIS SCHEDULE

Continued

See Figure 2

Contract No.: 68-02-2820		Assignment Number: 78-NHF-8									
Company Name: C-F Industries		Company Location: Donaldsonville, LA									
Industry: Ammonium Fertilizer		Control Equipment: Scrubber									
Process: B or C		Rotary Granulator									
Sampling No.	Total No. of Samples	Sample Type	Sampling Method	Sample Collected By	Minimum Sampling Time	Minimum Gas Volume Sampled ft ³	Type	Initial Analysis Method	By	Type	Final Analysis Method
1	3	Pressure Drop	U. Tube Manometer	CTR	Pressure every 15 minutes	drop across scrubber during test periods.					
2	3	Ambient air Temperature	Mercury Thermometer	CTR	Contractor shall measure and record data every 15 minutes during test periods.						
3	3	Ambient air Relative Humidity	Wet, Dry Bulb	CTR	"	"	"	"	"	"	"
Note - Urea and ammonia analysis shall be performed within 24 hours after collection of sample (stack and scrubber waters).											
N-6											

REMARKS:

1. Sampling shall be performed with $\pm 10\%$ isokinetic conditions.
2. Methods are EPA unless indicated otherwise.
3. Impingers and analysis of impinger catch will be per the Federal Register Volume 36, 159, 11, 1 Aug 1971.
4. Sampling time and gas volumes are for each sample.

SOURCE SAMPLING AND ANALYSIS SCHEDULE

See Figure 2

Contract No.: 68-02-2820		Assignment Number: 78-NHF-8								
Company Name: C. E. Industries		Company Location: Donaldsonville, LA								
Industry: Ammonium Fertilizer		Control Equipment: Scrubber								
Process: B or C		Rotary Granulator								
Sampling Point	Total No. of Samples	Sample Type	Sampling Method	Sample Collected By	Minimum Sampling Time	Minimum Gas Volume Sampled ft ³	Initial Analysis		Final Analysis	
							Type	Method	Type	Method
1	1*	Velocity Profile	EPA-2	CTR	N.A.	N.A.	Flow Profile	EPA-2	CTR	
2	1*	"	"	"	"	"	"	"	"	
1	1*	Moisture Content	EPA-4	"	15 min.	10	Percent Moisture	EPA-4	"	
2	1*	"	"	"	15 min.	10	"	"	"	
1	1	Cleanup Evaluation	Modified EPA-5	CTR	N.A.	N.A.	Urea Mass	TBD	CTR	NH ₃ Formaldehyde TBD
2	1	"	"	"	"	"	"	"	"	"
1*	3	Urea Particulate	Modified EPA-5	CTR	2 hrs	60	Urea Mass	TBD	CTR	NH ₃ Formaldehyde TBD
2*	3	"	"	"	"	"	"	"	"	"
1	3	Particle Size	Cascade Impactor	CTR	15 min	N.A.	Particle Distribution	Gravimetric Weighing	CTR	
2*	3	Visible Emissions	EPA-9	CTR	2 hours	N.A.	Summary of 6-min Intervals	EPA-9	CTR	
1*	3	Scrubber Solution In	Composite	CTR	2 hours	N.A.	Tempt, pH	Thermometer pH meter	CTR	% Urea, NH ₃ % Formaldehyde % Solids
2*	3	Scrubber Solution Out	Composite	CTR	2 hours	N.A.	Tempt, pH	"	CTR	TBD
4	3	Feed melt to granulator	Composite	CTR	N.A.	N.A.	pH	TBD	CTR	% Urea % NH ₃ % Formaldehyde
5	3	Granulator effluent	Grab	CTR	N.A.	N.A.	sieve analysis	TBD	CTR	Bulk Density
5	3	"	Composite	CTR	N.A.	N.A.	Tempt, pH % moisture	TBD	CTR	% Urea % Formaldehyde BD
7	1	Formaldehyde Additive	Grab	CTR	N.A.	N.A.	Sample to be retained for future analysis			

REMARKS:

1. Sampling shall be performed with $\pm 10\%$ isokinetic conditions.
2. Methods are EPA unless indicated otherwise.
3. Impingers and analysis of impinger catch will be per the Federal Register, Volume 35, Part 159, Part 11, Appendix A.

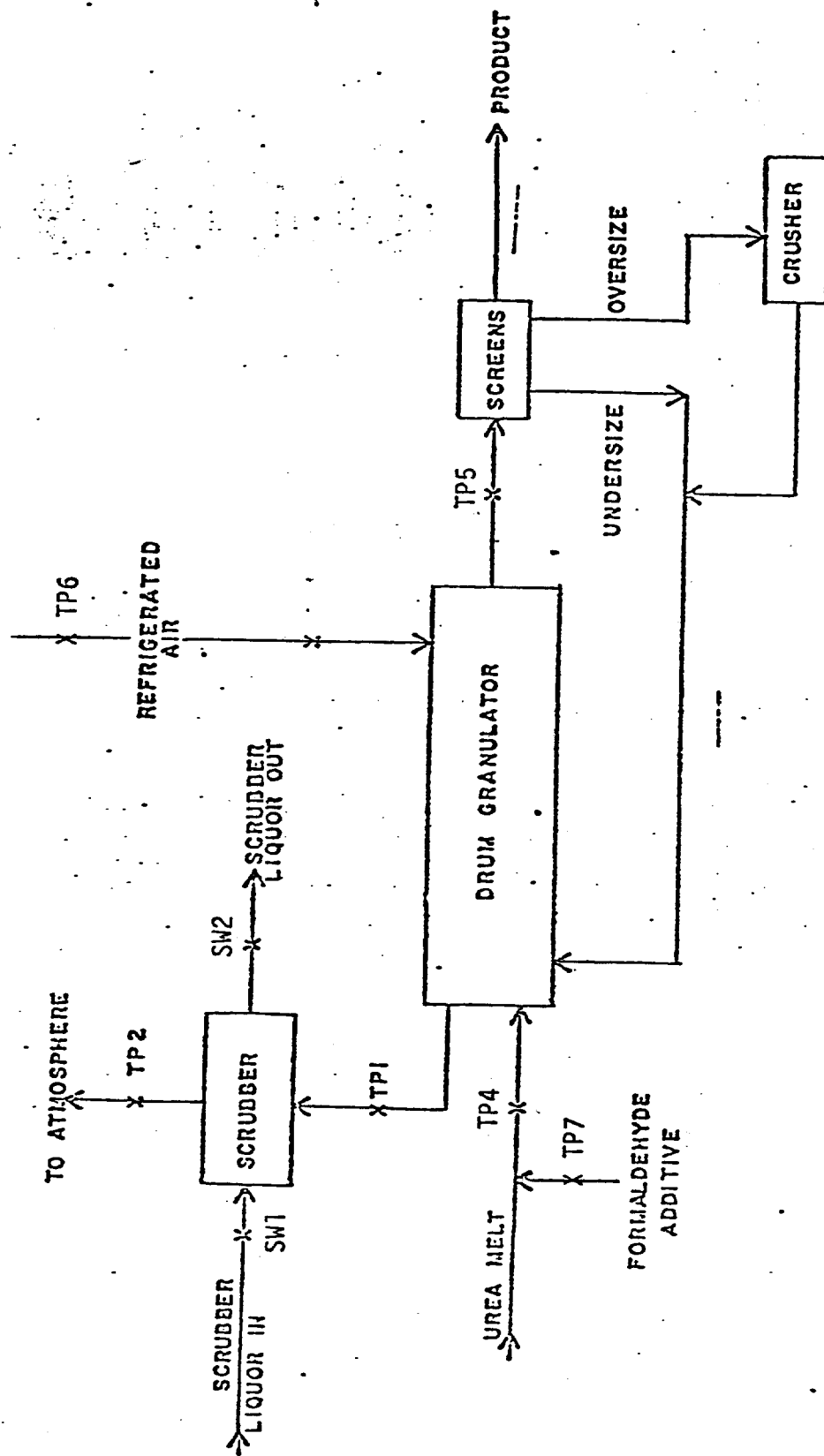


Figure 2. Drum Granulator, Urea, C.F. Industries, Donaldsonville, La.

APPENDIX N-2

COPY OF PROJECT TECHNICIANS DIRECTIVES

EMISSION MEASUREMENT BRANCH

TECHNICAL DIRECTIVE NO. 1

Project Number 78-NHF-8 Date 1-24-79

Contractor TRC of New England

Contract Number 68-02-2820 Work Assignment Number 10

Technical Manager Clyde E. Riley

Verbal Directions Given To Mr. Reed Cass

Directive:

1. Cancel 3 Visible Emission Test to be performed at TP-3 on the main Urea Solution, Formation, and Concentration tower vent.
2. Cancel 1 Formaldehyde Additive sample to be collected at TP-7 of the Rotary Granulator Process.
3. Reduce number of Feed-Melt to Granulator samples at TP-4 from three to one. Single sample shall be analyzed for pH, percent urea, ammonia, and formaldehyde.
4. Reduce number of composite Granulator Unscreened Product samples at TP-5 from three to one. The single sample shall be analyzed for tempt, percent urea, and formaldehyde. Percent moisture and pH data for the unscreened granulator product testing shall be obtained daily from CF Industry personnel.

Clyde E. Riley
Technical Manager, EMB

J. G. Markley
Section Chief, EMB

United States
Environmental Protection
Agency

Office of Air, Noise, and Radiation
Office of Air Quality Planning and Standards
Research Triangle Park, NC 27711

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