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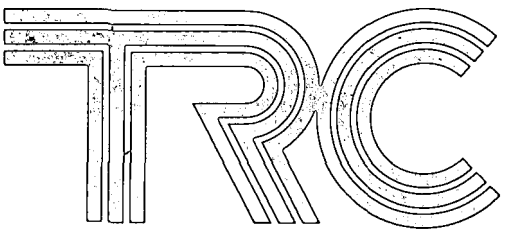
Air

EPA

Urea Manufacture

Emission Test Report
C. F. Industries
Donaldsonville, Louisiana

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



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NOTE:

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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 Donaldsonvlle, 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.

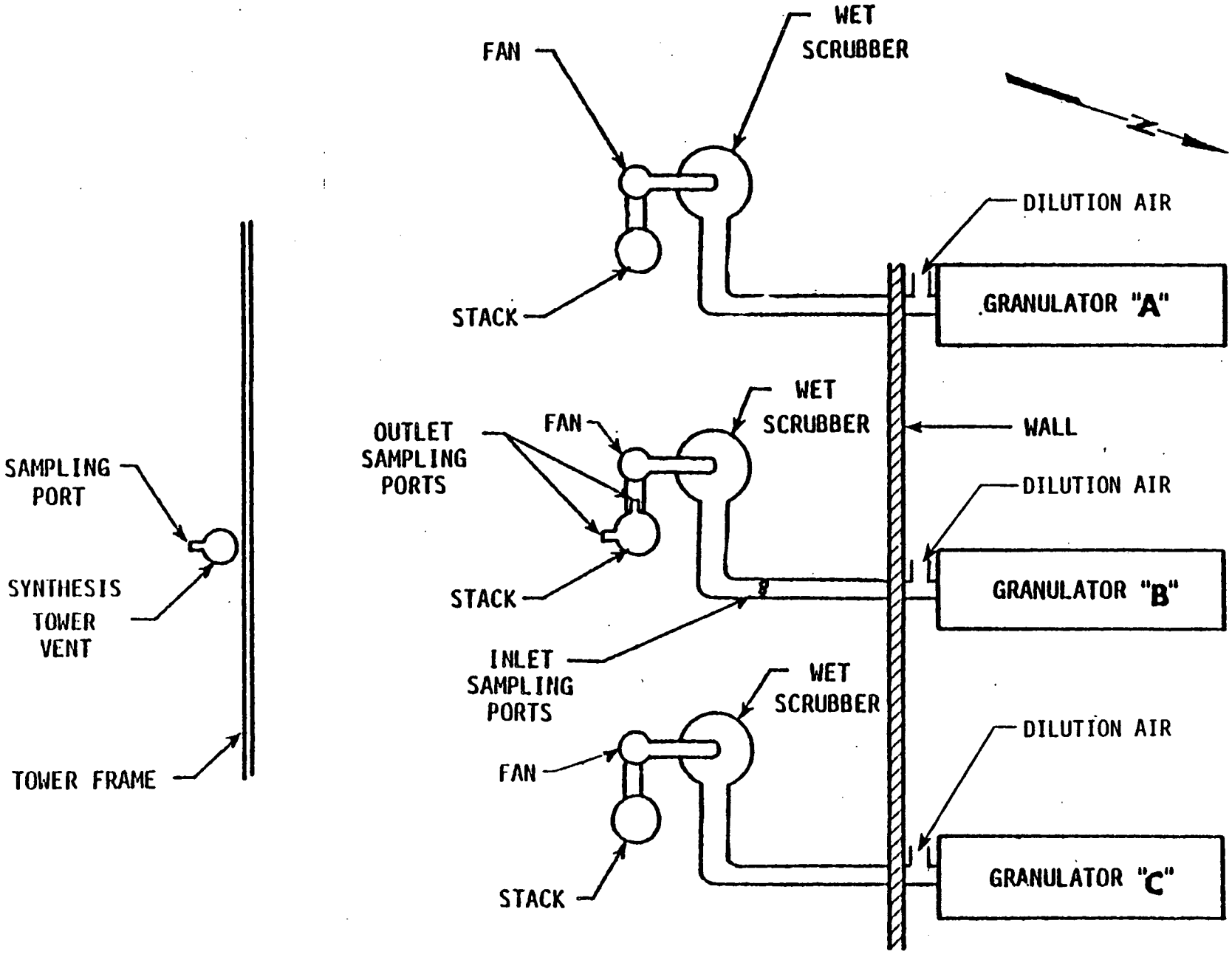

FIGURE 1-1: OVERHEAD VIEW SHOWING LOCATIONS OF SYNTHESIS TOWER VENT & GRANULTORS A, B & C EXHAUST DUCTING, SCRUBBERS & SAMPLING POINTS AT CF INDUSTRIES, INC., DONALDSONVILLE, LOUISIANA

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 1-1

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

Clock Time	Production Rate tons/hour: Granulator	Production Rate tons/hour: Syn. Tower	"B" Granulator Scrubber Urea Particulate: Inlet	"B" Granulator Scrubber Urea Particulate: Outlet	Stack Visible Emissions	Scrubber Liquid Inlet: pH	Scrubber Liquid Inlet: Temp (°F)	Scrubber Liquid Outlet: pH	Scrubber Liquid Outlet: Temp (°F)	Pressure Drop ("H_2O)	Ambient Conditions: Temp (°F)	Ambient Conditions: R.H. (%)	Ambient Conditions: Pressure ("Hg)	Process Sampling
1200	Confidential		Start Run 1											
1201	Per CFI			Start Run 1										
1202	Request				Start									
1207										21.8				
1210			Pitot Plugged											
1212										21.6				
1215						9.50	135	8.15	126					
1217										20.9				
1222			Run Continued											
1223			Probe Plugged											
1226			Run Continued											
1227										20.7				
1230											69	76	-	
1232										20.7				
1237										20.7				
1242										21.1				
1245											69	76	-	
1247										21.1				
1252										20.7				
1255						9.25	154	8.10	124					
1257										20.5				
1301				Complete 1st Traverse							65	76	30.40	
1306														
1310			Complete 1st Traverse											
1316				Start 2nd Traverse										
1318			Start 2nd Traverse Nozzle Plugged											
1320											69	76	30.40	
1321										20.5				
1326										20.7				
1327			Run Continued											
1331						9.15	164	8.10	124	20.9				
1333														
1336										20.9				
1341										20.7				
1343														Unscreened Product
1346										20.7				
1351										20.7				
1356										20.9	69	79	30.39	

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 Urea Particulate		Stack Visible Emissions	Scrubber Liquid				Pressure Drop ("H_2O)	Ambient Conditions			Process Sampling
						Inlet		Outlet						
	Granulator	Syn. Tower	Inlet	Outlet		pH	Temp (°F)	pH	Temp (°F)		Temp (°F)	R.H. (%)	Pressure ("Hg)	
1401	Confidential									20.7				
1406	Per CFI									20.5				
1411	Request									20.5				
1416				Complete Run 1		9.10	152	7.95	120	20.5				
1417														
1422														
1423			Complete Run 1		Stop									
1448											70	74	30.39	
1523											69	79	30.39	
1548			Start Run 2											
1549				Start Run 2						20.2				
1550					Start	9.7	166	8.3	129					
1554										20.2				
1559										20.2				
1604										20.8				
1609										20.9				
1614										20.9				
1619										20.7				
1620						9.5	183	8.2	128					
1624										20.8				
1629										20.9				
1634										21.0				
1635											67	85	30.39	
1637					Stop									
1639										21.0				
1644			Complete 1st Traverse							20.2				
1649				Complete 1st Traverse										
1653				Start 2nd Traverse										
1655						9.2	174	8.2	129					
1657			Start 2nd Traverse Probe Plugged											
1658										20.4				

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 Granulator	Production Rate tons/hour Syn. Tower	"B" Granulator Scrubber Urea Particulate Inlet	"B" Granulator Scrubber Urea Particulate Outlet	Stack Visible Emissions	Scrubber Liquid Inlet pH	Scrubber Liquid Inlet Temp (°F)	Scrubber Liquid Outlet pH	Scrubber Liquid Outlet Temp (°F)	Pressure Drop ("H_2O)	Ambient Conditions Temp (°F)	Ambient Conditions R.H. (%)	Ambient Conditions Pressure ("Hg)	Process Sampling
1702	Confidential		Run Continued											
1703	Per CFI									20.4				
1708	Request									20.4				
1713										20.2				
1715											68	80	30.39	
1718										20.3				
1723										20.4				
1725						9.2	167	8.1	128					
1728										20.6				
1730														Unscreened Product
1733										20.6				
1738										20.6				
1743										20.4				
1748										20.4				
1753				Complete Run 2						20.2				
1756			Complete Run 2											
1800						9.0	172	8.0	129					
1815											66	85	30.38	

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 Scrubber Urea Particulate		Stack Visible Emissions	Scrubber Liquid Inlet		Scrubber Liquid Outlet		Pressure Drop ("H_2O)	Ambient Conditions			Process Sampling	Synthesis Tower Sampling
	Granulator	Syn. Tower	Inlet	Outlet		pH	Temp (°F)	pH	Temp (°F)		Temp (°F)	R.H. (%)	Pressure ("Hg)		
0945	Confidential										62	94	30.39		
0946	Per CFI			Start Run 3						21.6					
0947	Request		Start Run 3												
0951										21.2					
0955						9.2	180	8.3	128						
0956										21.0					
1001										21.1					
1005											62	94	30.39		
1006										21.0					
1011										21.2					
1016										21.0					
1017			Pitot Plugged												
1020											64	90	30.39		
1021			Continue - Probe Plugged							20.7					
1026										20.5					
1030			Continue			9.3	172	8.25	126						
1031										20.7					
1036										20.6					
1040										20.7	64.5	87	30.38		
1041															
1045														Unscreened Product	
1046				Complete 1st Traverse											
1047			Probe Plugged												
1055			Continue								66	82	30.38		
1102			Complete 1st Traverse												
1105						9.3	177	8.25	129						
1107			Start 2nd Traverse												
1108			Probe Plugged												
1112			Continue												
1115											67.5	85	30.36		
1117				Start 2nd Traverse											
1122										20.5					

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	Production Rate tons/hour: Syn. Tower	"B" Granulator Scrubber Urea Particulate: Inlet	"B" Granulator Scrubber Urea Particulate: Outlet	Stack Visible Emissions	Scrubber Liquid: Inlet pH	Scrubber Liquid: Inlet Temp (°F)	Scrubber Liquid: Outlet pH	Scrubber Liquid: Outlet Temp (°F)	Pressure Drop ("H_2O)	Ambient Conditions: Temp (°F)	Ambient Conditions: R.H. (%)	Ambient Conditions: Pressure ("Hg)	Process Sampling	Synthesis Tower Sampling
1127	Confidential Per CFI Request									20.5					
1132										20.4					
1137										20.6					
1140						9.3	173	8.20	127		67	83	30.36		
1142										20.3					
1147										20.3					
1152										20.2					
1157										20.1					
1200											68	85	30.36		
1202										19.8					
1207										20.0					
1210			Complete Run 3												
1212										19.7					
1215						9.2	173	8.2	124						
1217				Complete Run 3						19.5					
1244					Start										
1344					Stop										
1435											69	85	30.28		
1500											71	77	30.28		
1525											73	73	30.27		
1528			Start Cascade Run 1												
1540											71	77	30.27		Start Run 1 (Test 4)
1543			Complete Cascade Run 1												
1555															Complete Run1 (Test 4)
1615			Start Moisture Run												
1649			Complete Moisture Run												

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		"B" Granulator Scrubber Urea Particulate		Stack Visible Emissions	Scrubber Liquid Inlet		Scrubber Liquid Outlet		Pressure Drop ("H_2O)	Ambient Conditions			Process Sampling	Synthesis Tower Sampling
	Granulator	Syn. Tower	Inlet	Outlet		pH	Temp (°F)	pH	Temp (°F)		Temp (°F)	R.H. (%)	Pressure ("Hg)		
0953	Confidential				Start										
1000	Per CFI		Start Cascade Run 2								68	90	30.17		
	Request														
1015			Complete Cascade Run 2												
1020											68	90	30.17		
1030															Start Run 2 (Test 5)
1040											70	86	30.17		
1046															Complete Run2
1100											70	86	30.17		
1120											70	86	30.14		
1125			Start Cascade Run 3												
1135															Start Run 3 (Test 6)
1140			Complete Cascade Run 3								75	70	30.12		
1150															Complete Run3
1153					Stop										
1200											77	66	30.10		
1230											73	75	30.10		

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

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

collected isokinetically from 12 traverse points located in accordance with EPA Reference Method 1.

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 seperately 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

[2] Standard 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" GRANUALTOR SCRUBBER ON JANUARY 17 AND 18, 1979 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
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)								
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	20690	73.1	26258	62.3	25423	68.7
gr/DSCF[e]	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 seperate 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	Run 1		Run 2		Run 3		Average	
Date	01-18-79		01-19-79		01-19-79			
Location	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^3M[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[e]	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	0.000572
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) Grams 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		Run 2		Run 3		Average	
Date	01-17-79		01-17-79		01-18-79			
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 Flowrate-DSCFM[c]	40180		41410		41760		41117	
Scrubber Liquid pH	9.25		9.32		9.26		9.28	
Production Rate* (Tons/hr)								
Percent Isokinetic	104.5		105.0		102.9		104.1	
Net Sampling Time (Minutes)	111		110		111		111	
UREA DATA	e	f						
Kjeldahl Analysis Method:	Uncorrected	Corrected	Uncorrected	Corrected	Uncorrected	Corrected	Uncorrected	Corrected
Total Sample Weight (mg)	23920	25594	26090	27916	26258	28096	25423	27203
gr/DSCF[d]	5.830	6.238	6.090	6.516	6.085	6.511	6.005	6.425
lb/hr	2007	2147	2161	2312	2178	2330	2116	2264
lb/ton	111.5	119.3	120.1	128.5	125.2	134.0	118.9	127.2
AMMONIA DATA	g	h	i					
Nesslerization Analysis Method:	Direct	Uncorrected Distilled	Corrected Distilled	Direct	Uncorrected Distilled	Corrected Distilled	Direct	Uncorrected Distilled	Corrected Distilled	Direct	Uncorrected Distilled	Corrected Distilled
Total Sample Weight (mg)	410	888	-127	441	941	-166	514	944	-170	455	924	-155
gr/DSCF	0.0999	0.2164		0.1029	0.2196		0.1191	0.2187		0.1073	0.2179	
lb/hr	34.40	74.50		36.52	77.93		42.62	78.27		37.85	76.86	
lb/ton	1.90	4.12		2.03	4.33		2.45	4.50		2.13	4.33	
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

TABLE 2-3

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

Run Number	Run 1			Run 2			Run 3			Average		
Date	01-17-79			01-17-79			01-17-79					
Volume of Gas Sampled - DSCF[a]	102.5			102.7			102.4			102.5		
Percent Moisture by Volume	5.522			5.733			5.608			5.621		
Average Gas Temperature (°F)	100.5			100.7			102.5			101.2		
Stack Volumetric Flowrate - DSCFM[b]	46260			45590			45760			45870		
Scrubber Liquid pH	8.08			8.16			8.24			8.16		
Production Rate* (tons/hr)												
Percent Isokinetic	100.7			102.4			101.6			101.6		
Pressure Drop Across Scrubber (inches H_2O)	20.8			20.5			20.6			20.6		
Net Sampling Time (minutes)	120			120			120			120		
UREA DATA		d	e									
Kjeldahl Analysis Method:		uncorrected	corrected		uncorrected	corrected		uncorrected	corrected		uncorrected	corrected
Total Sample Weight (mg)		70.6	75.5		73.1	78.2		62.3	66.7		68.7	73.5
gr/DSCF[c]		0.0106	0.0113		0.0110	0.0118		0.0094	0.0101		0.0103	0.0110
lb/hr		4.205	4.499		4.283	4.583		3.674	3.931		4.058	4.342
lb/ton		0.232	0.248		0.238	0.255		0.211	0.226		0.228	0.244
AMMONIA DATA												
Nesslerization Analysis Method:	f	g uncorrected	h corrected		uncorrected	corrected		uncorrected	corrected		uncorrected	corrected
	direct	distilled	distilled	direct	distilled	distilled	direct	distilled	distilled	direct	distilled	distilled
Total Sample Weight (mg)	450	438	435	694	426	423	387	388	385	510	417	414
gr/DSCF	0.0676	0.0658	0.0653	0.1041	0.0639	0.0635	0.0582	0.0584	0.0579	0.0766	0.0626	0.0622
lb/hr	26.80	26.08	25.91	40.17	24.96	24.79	22.82	22.88	22.70	30.10	24.61	24.43
lb/ton	1.48	1.44	1.43	2.26	1.39	1.38	1.31	1.31	1.30	1.68	1.37	1.36
FORMALDEHYDE DATA												
Chromotropic Acid Analysis Method												
Total Sample Weight (mg)		2.523			1.172			1.296			1.664	
gr/DSCF		0.000379			0.000176			0.000195			0.000250	
lb/hr		0.1503			0.0687			0.0764			0.0983	
lb/ton		0.0083			0.0038			0.0044			0.0055	

[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/1.765)
* 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 seperate 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

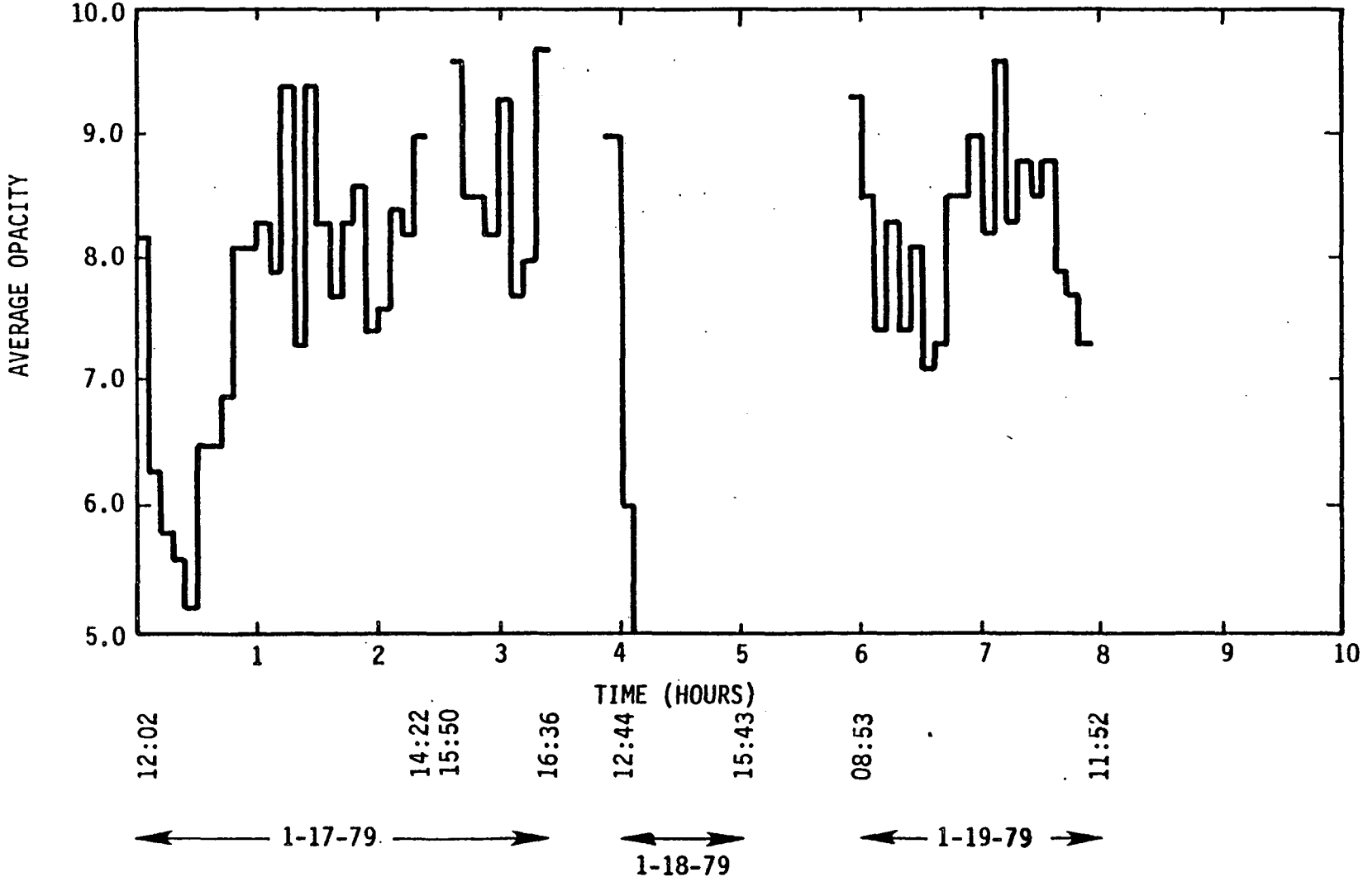

TABLE 2-4

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	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	

* 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	8.8	
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	

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, μm	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

DATE	RUN	CLOCK TIME	ΔP, "H_2O	DATE	RUN	CLOCK TIME	ΔP "H_2O
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
01/17/79		1321	20.5			1748	20.5
		1326	20.7			1753	20.2
		1331	20.9				
		1336	20.9			Average	20.5
		1341	20.7				
		1346	20.7	01/18/79	3	0946	21.6
		1351	20.7			0951	21.2
		1356	20.9			0956	21.0
		1401	20.7			1001	21.1
		1406	20.5			1006	21.0
		1411	20.5			1011	21.2
		1416	20.5			1016	21.0
						1021	20.7
		Average	20.9			1026	20.5
						1031	20.7
01/17/79	2	1549	20.2			1036	20.6
		1554	20.2			1041	20.7
		1559	20.2	01/18/79		1122	20.5
		1604	20.8			1127	20.5
		1609	20.9			1137	20.6
		1614	20.9			1142	20.3
		1619	20.7			1147	20.3
		1624	20.8			1152	20.2
		1629	20.9			1157	20.1
		1634	21.0			1202	19.8
		1639	21.0			1207	20.0
						1212	19.7
						1217	19.5
						Average	20.6

each sample was collected. The pH was measured once the sample had cooled to approximately 70^{o} 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	
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

[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 insoluable 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

[a]Composite sample

[b]Kjeldahl analysis results uncorrected for urea conversion during distillation.

[c]Kjeldahl analysis results corrected for urea conversion. (corrected = uncorrected x 1.07)

[d]Distillation/Nesslerzation (D/N) analysis results uncorrected for urea conversion during distillation.

[e]D/N analysis results corrected for urea conversion. (corrected = uncorrected - 0.07*corrected urea/1.765.

[f]Average of measurements by CF Industries Inc.

[g]Turbidity 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	Cumulative Percent of Total Mass	Mass gm	Percent of Total Mass	Cumulative Percent of Total Mass	Mass, gm	Percent of Total Mass	Cumulative 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

TABLE 2-12

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

Run Number	Run 1			Run 2			Run 3			Average		
Date	01-18-79			01-19-79			01-19-79					
Volume of Gas Sampled (including ammonia)-DSCF[a]	5.07			5.30			5.14			5.17		
Percent Moisture by Volume	67.0			57.8			64.5			63.1		
Average Gas Temperature (°F)	193			186			191			190.0		
Stack Volumetric Flowrate (including ammonia)-DSCFM[b]	306			402			339			349		
Production Rate* (tons/hr)												
Percent Isokinetic	84.2			66.5			77.9			76.2		
Net Sampling Time (minutes)	15			15			15			15		
UREA DATA		d	e									
Kjeldahl Analysis Method:		uncorrected	corrected		uncorrected	corrected		uncorrected	corrected		uncorrected	corrected
Total Sample Weight (milligrams)		189	202		198	212		185	198		191	204
gr/DSCF (including ammonia)[c]		0.574	0.614		0.575	0.615		0.554	0.593		0.580	0.619
lb/hr		1.51	1.62		1.98	2.12		1.61	1.72		1.70	1.82
lb/ton		0.0253	0.0271		0.0352	0.0377		0.0286	0.0306		0.0297	0.0317
AMMONIA DATA	f	g	h									
Nesslerization Analysis Method:	direct	uncorrected distilled	corrected distilled	direct	uncorrected distilled	corrected distilled	direct	uncorrected distilled	corrected distilled	direct	uncorrected distilled	corrected distilled
Total Sample Weight (milligrams)	37720	37920	37910	45840	48620	48610	57810	58230	58220	41720	48260	48250
gr/DSCF (including ammonia)	114.6	115.2	115.2	133.2	141.3	141.3	173.2	174.5	174.5	140.3	143.7	143.7
lb/hr	300.5	302.1	302.0	458.9	486.7	486.6	503.2	506.9	506.8	420.9	431.1	431.0
lb/ton	5.06	5.09	5.09	8.15	8.64	8.64	8.94	9.00	9.00	7.83	8.02	8.02

(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 (NH_2 CO_2 NH_4). The liquid ammonium carbamate is subsequently decomposed to urea ($CO(NH_2)_2$) 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.

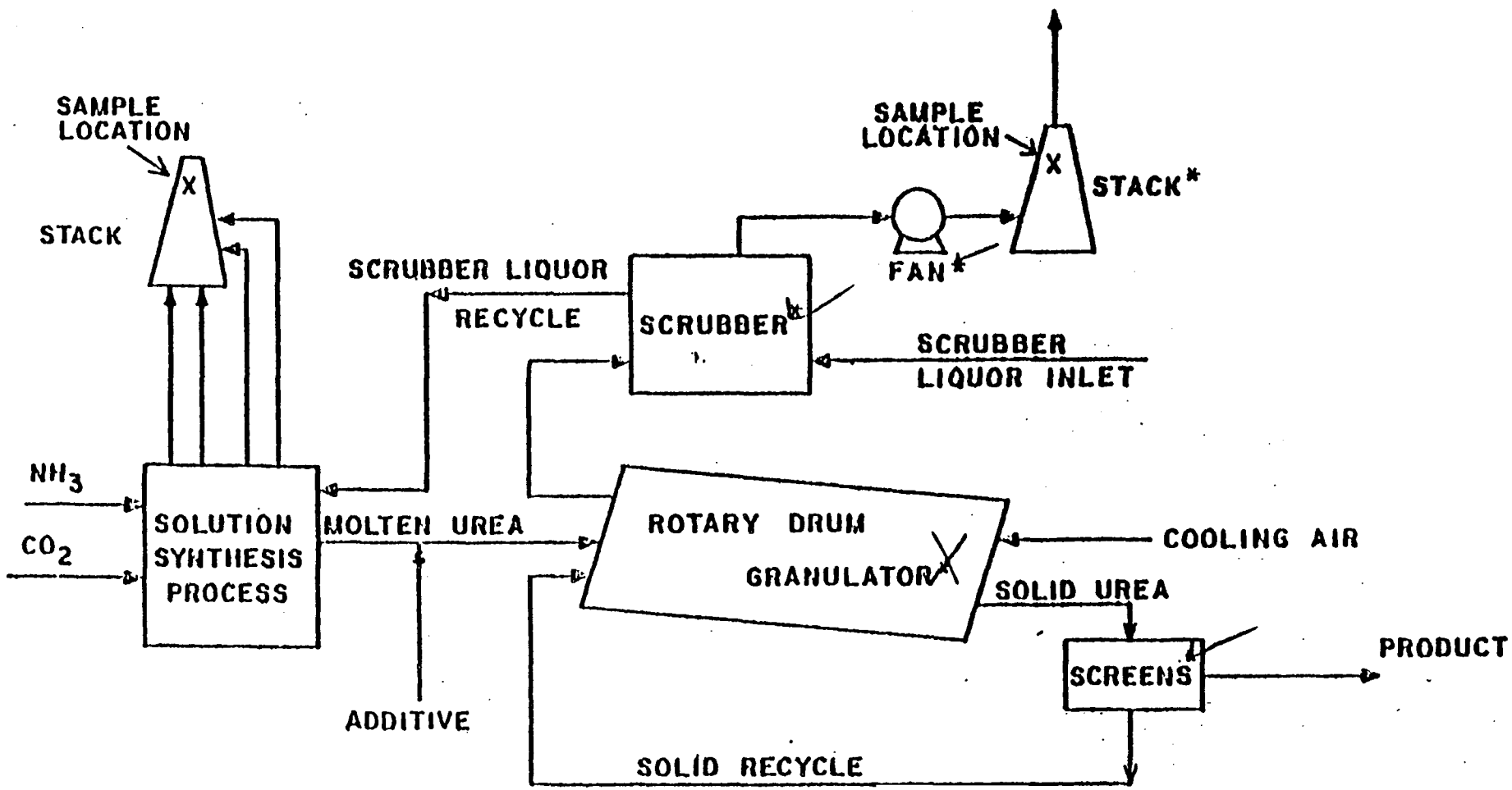

FIGURE 3-1 TYPICAL UREA MANUFACTURING PROCESS AS DESIGNED BY C & I GIRDLER

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. . . . . .[a]Note 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 . . . . . .[b]Note 4 . . . . . scrubbers . . . . . .[c]Note 5 . . . . . On line No. 1, each scrubber exhausts through its own stack.

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[a]Note 3 - See Item 3, Confidential Addendum, Contact Eric Noble, EPA (919) 541-5213.

[b]Note 4 - See Item 4, Confidential Addendum, Contact Eric Noble, EPA (919) 541-5213.

[c]Note 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]

[a]Note 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]

[a]Note 7 - See Item 7, Confidential Addendum, Contact Eric Noble, EPA (919) 541-5213.

During emissions testing of the solution production equipment, CO_2 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 Granulator Emissions			Solution Tower Emissions Tests		
	1/17; 1200-1415		1/17; 1545-1803		1/18; 0945-1217							
	Avg.	Std. Dev.	Avg.	Std. Dev.	Avg.	Std. Dev.	1/18 1530-1545	1/18 1600-1018	1/19 1115-1147	1/18 1545-1603	1/19 1030-1047	1/19 1130-1200
Production Rate Parameters												
Urea Melt Feed to Granulator A	100	0.9	83	0.6	111	0.7	113	100	100	111	100	100
Urea Melt Feed to Granulator B	101	0.8	100	0.0	97	0.7	98	100	100	98	100	100
Urea Melt Feed to Granulator C	105	0.5	90	0.0	108	0.5	105	94	94	102	94	94
Total Solids from A, B, and C	100	-	92	-	104	-	119	98	97	80	101	102
B Granulator Operating Parameters												
Air Inlet Temperature	101	1.0	100	1.7	92	1.5						
Air Outlet Temperature	100	0.3	100	0.6	98	0.5						
Urea Inlet Temperature	100	0.7	100	0.6	100	0.3						
Solids Outlet Temperature	101	1.7	99	0.7	94	0.7						
Formaldehyde Addition Rate	106	4.7	104	0.0	100	3.3						
Scrubber Operating Parameters												
Liquor Level	101	1.0	101	1.0	101	1.0						
Liquor Temperature	99	0.5	104	0.5	99	0.0						
Liquor Feed Rate	105	0.0	107	5.6	113	9.2						
Liquor Blowdown Rate	70	68.	120	63.	136	136.						
Blowdown Urea Content	101	0.8	98	2.0	106	4.2						
Solution Production Operating Parameters												
Ammonia Feed Pump Rate (RPM)	100	1.1	102	1.0	100	2.1	98	99	99	98	99	99
CO_2 Compressor Rate (SCFM)										102	99	99
T105 Overheads Pressure										101	99	98
Solution Feed to Evaporator										97	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, CO_2 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	

[a]Note 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 CO_2 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, Lousiana Region, CF Industries, Inc., Donaldsonville, Louisiana, May 28, 1979.

SECTION 4

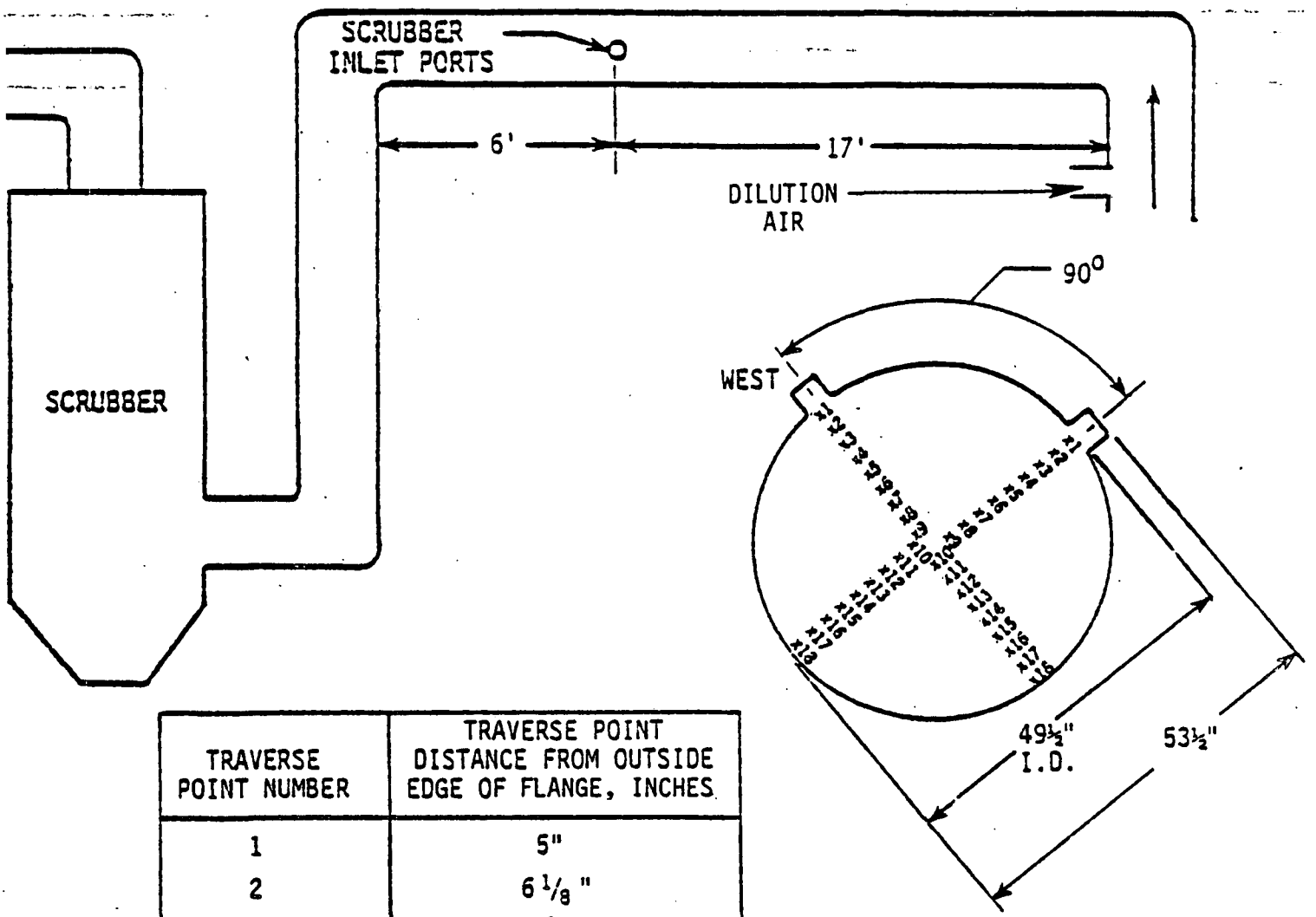
LOCATION OF SAMPLING POINTS

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.

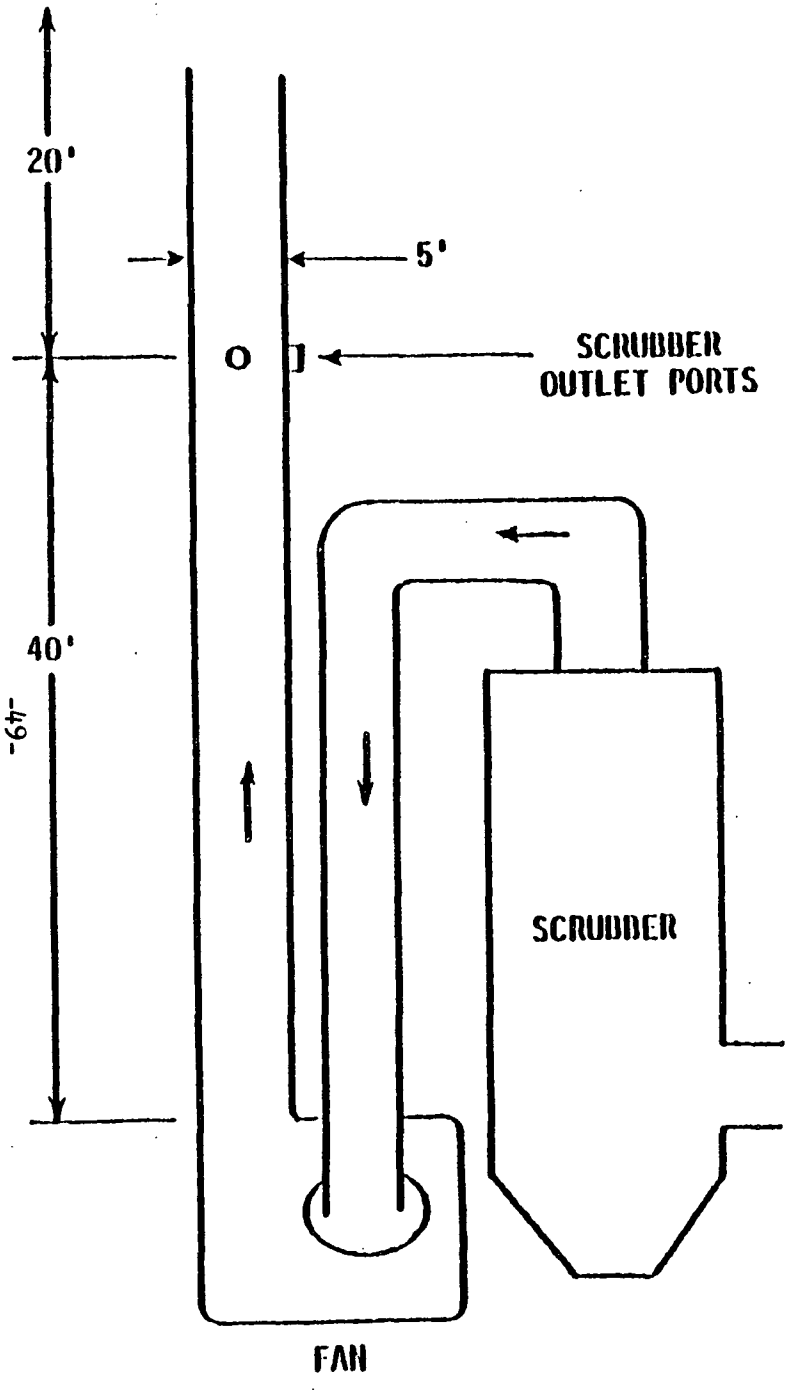
The "B" scrubber 60 inch I.D. outlet stack was fitted with two 3-inch pipe flange sampling ports positioned 90° apart in a horizontal plane. The two ports were located 40 feet (8 stack diameters) downstream from the fan discharge, and 20 feet (4 stack diameters) upstream of the top of the stack. See 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.

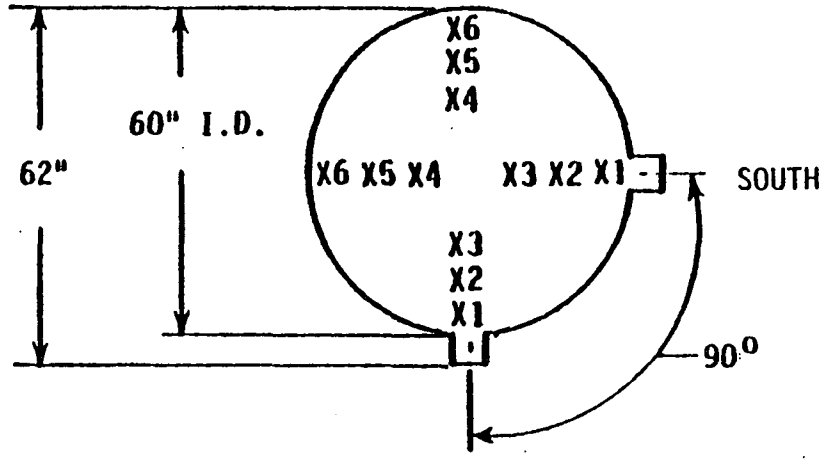
INLET PARTICLE SIZE SAMPLING LOCATION, TP-1

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.

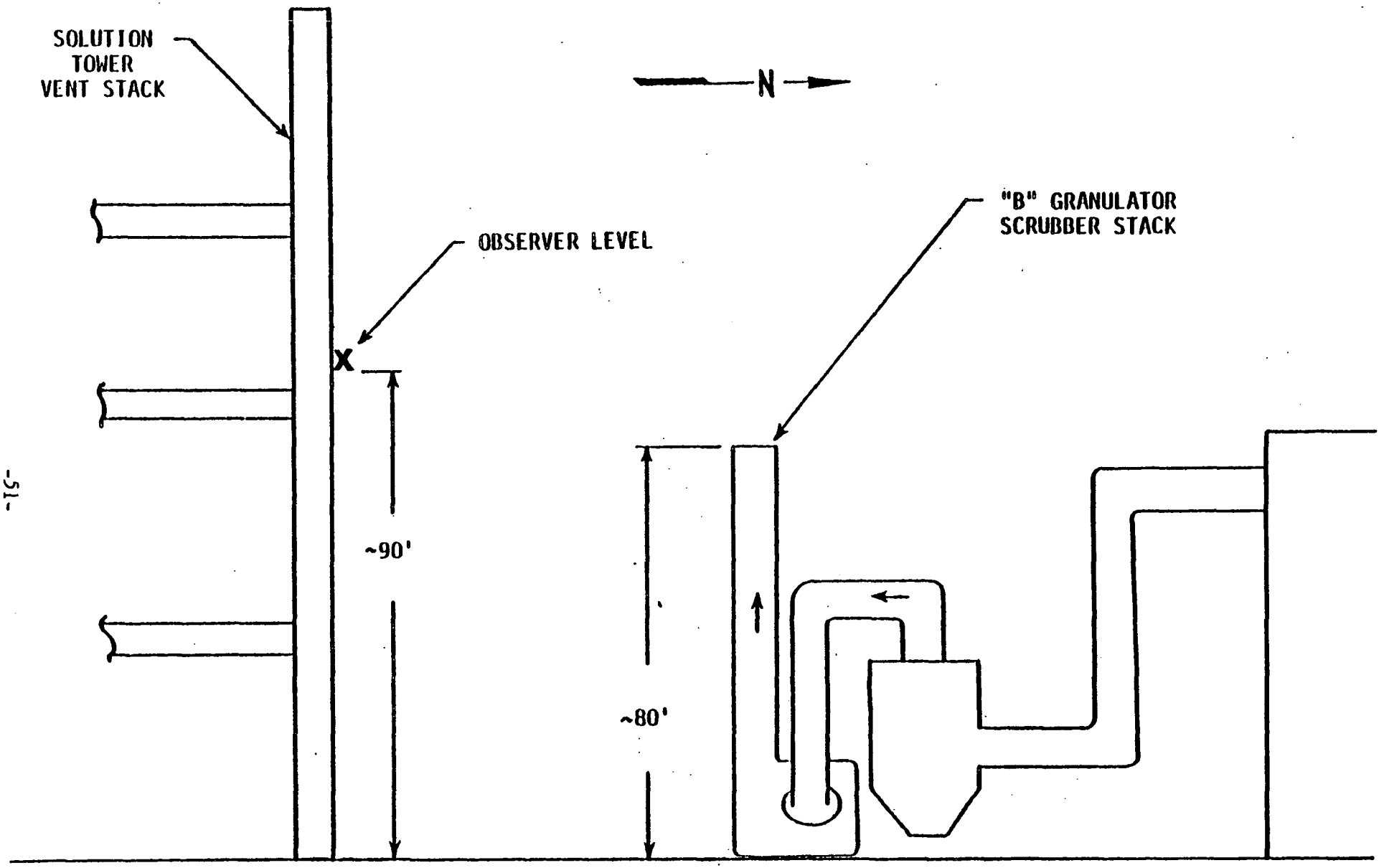


FIGURE 4-3: LOCATION OF SMOKE OBSERVER FOR JANUARY 17 - 19, 1979 OPACITY READINGS ON "B" GRANULATOR SCRUBBER STACK AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

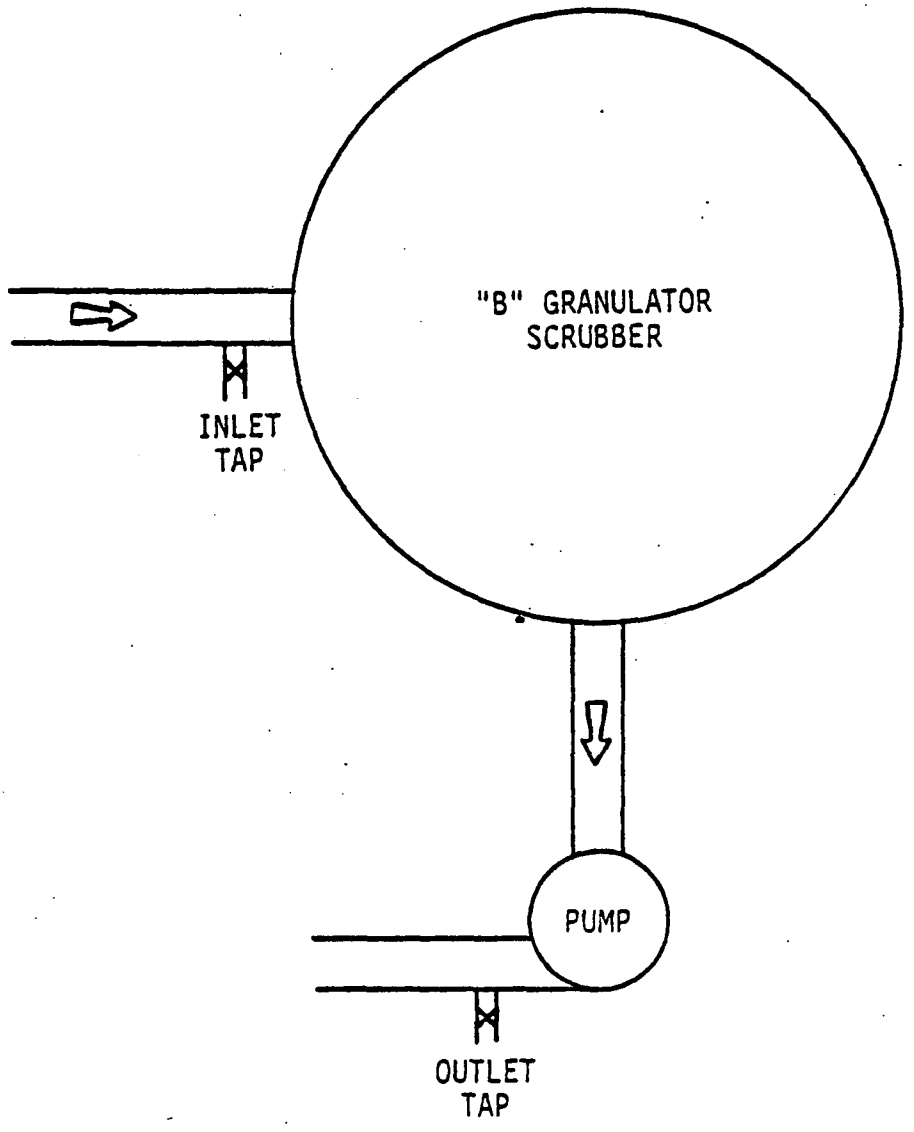


FIGURE 4-4: LOCATIONS OF SCRUBBER LIQUID COLLECTION TAPS FOR JANUARY 17 - 19, 1979 TESTS ON "B" GRANULATOR AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

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.

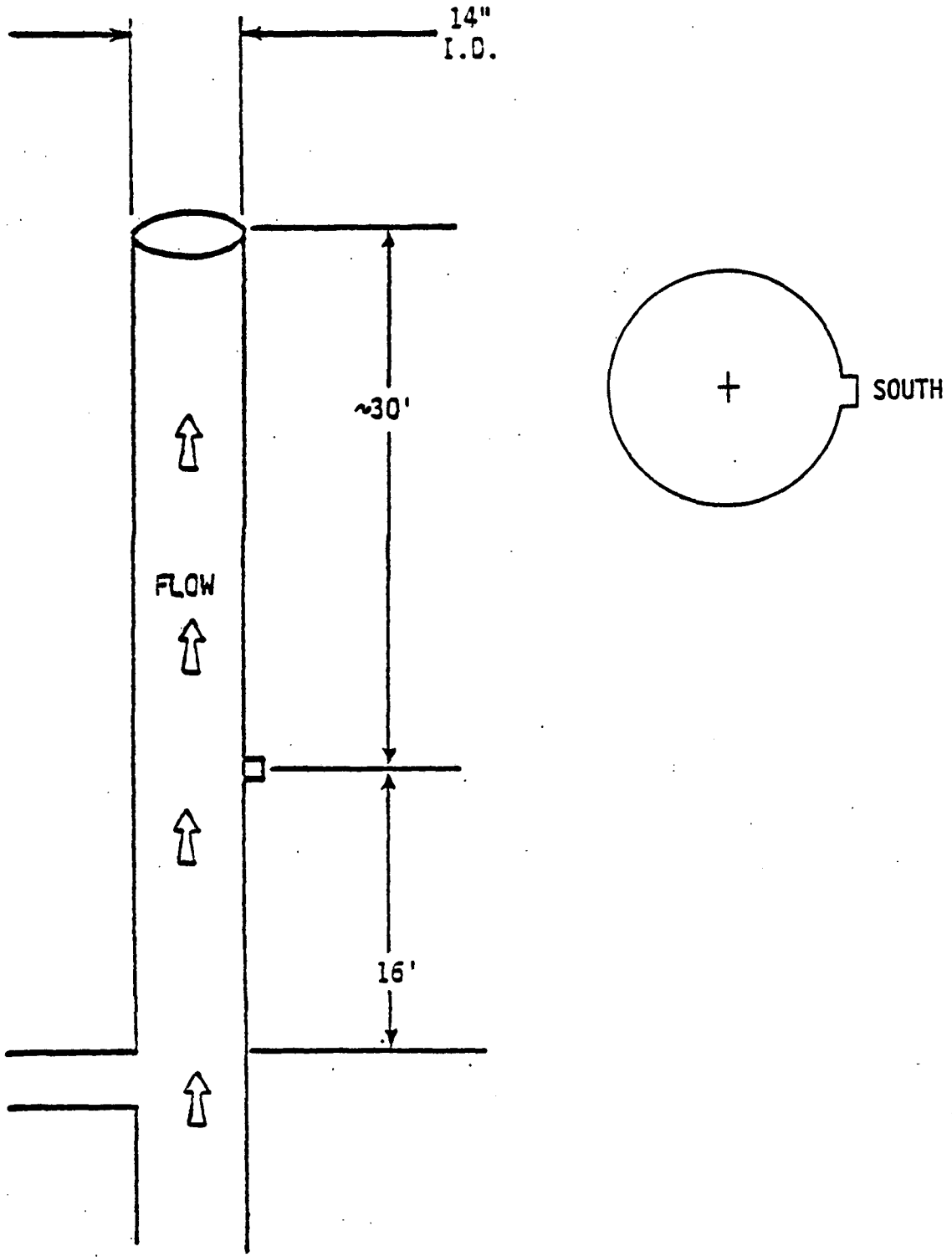


FIGURE 4-5: LOCATION OF SYNTHESIS TOWER SOLUTION VENT SAMPLING PORT AT CF INDUSTRIES INC., DONALDSONVILLE, LOUISIANA

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.

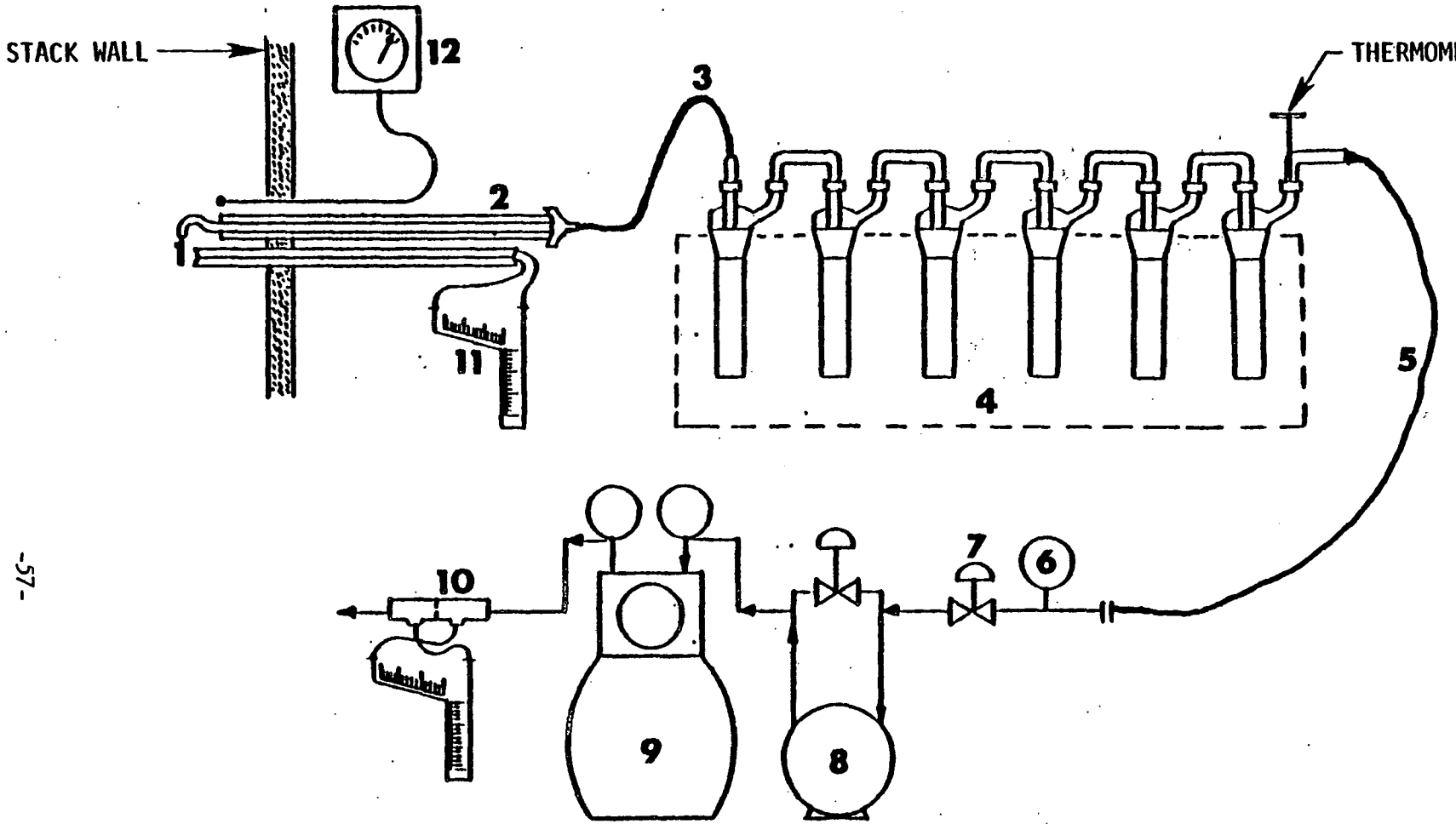
UREA, FORMALDEHYDE, AND AMMONIA METHODS USED ON "B" GRANULATOR SCRUBBER

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 H_2SO_4 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



LEGEND

1 - NOZZLE	7 - NEEDLE VALVE
2 - PROBE	8 - PUMP
3 - TEFLON LINE	9 - DRY GAS METER
4 - ICE BATH	10 - ORIFICE
5 - FLEXABLE LINE	11 - PITOT TUBE & INCLINED MANOMETER
6 - VACUUM GAGE	12 - POTENTIOMETER

FIGURE 5-1: MODIFIED EPA PARTICULATE SAMPLING TRAIN
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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 Kjeldehl 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 chromatropic-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

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[1] Standard 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.