

Air



Urea Manufacture

Emission Test Report Agrico Chemical Company Blytheville, Arkansas

REPORT ON PROCESS EMISSIONS TESTS
AT THE AGRICO CHEMICAL COMPANY
UREA MANUFACTURING FACILITY
IN BLYTHEVILLE, ARKANSAS
(DECEMBER 1978)
REPORT 79-NHF-13a

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PREFACE

The work reported herein was performed by personnel from TRC Environmental Consultants, Inc. (TRC), the GCA/Technology Division (GCA), Agrico Chemical Company, Blytheville, Arkansas (Agrico), and the U.S. Environmental Protection Agency (EPA).

The scope of work, issued under EPA Contract No. 68-02-2820, Work Assignment Number 11, was under the supervision of the TRC Project Manager, Mr. Willard A. Wade III. Mr. Eric A. Pearson of TRC was responsible for summarizing the test and analytical data presented in this report. Sample analysis was performed at the Agrico, Blytheville, Arkansas plant under the direction of Ms. Margaret M. Fox, and at the TRC laboratory in Wethersfield, Connecticut under the direction of Ms. Joanne M. Marchese.

Stephen K. Harvey of GCA was responsible for monitoring the process operations during the emissions testing program. GCA personnel were also responsible for preparing Section 3.0, Process Description and Operations, and Appendix G of this report.

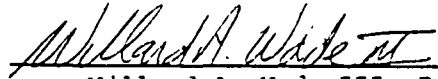
Personnel of Agrico Chemical Company, Blytheville, Arkansas, whose assistance and guidance contributed greatly to the success of this emissions testing program included Mr. Jesse Boggan, Environmental Coordinator, Mr. James Kilpatrick, Chief Chemist, and Mr. Deryl Beiard, Chemist.

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

Mr. Gary D. McAlister, Office of Air Quality Planning and Standards, Emission Measurement Branch, EPA, served as Lead Chemical Engineer and was responsible for developing and evaluating the analytical procedures used on this program.

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.

TRC-Environmental Consultants, Inc.

A handwritten signature in cursive script, reading "Willard A. Wade III", is written over a horizontal line.

Willard A. Wade III, P.E.
Project Manager

July 31, 1980

NOTE: Mention of trade names or commercial products in this publication does not constitute endorsement or recommendation for use by the Environmental Protection Agency.

TABLE OF CONTENTS

<u>SECTION</u>		<u>PAGE</u>
1.0	INTRODUCTION	1
1.1	Background	1
1.2	Measurement Program	1
1.3	Description of Report Sections	4
2.0	SUMMARY OF RESULTS	5
2.1	Granulator "C" Scrubber Outlet Gas Stream	5
2.2	Comparison of TRC and Agrico Scrubber Outlet Gas Stream Analyses	9
2.3	Scrubber Liquor Sampling Results	15
2.4	Urea Audit Samples - Comparison of TRC and Agrico Analyses	17
3.0	PROCESS DESCRIPTION AND OPERATIONS	20
3.1	Process Equipment	20
3.2	Process Operation	22
4.0	LOCATION OF SAMPLING POINTS	31
4.1	Granulator C Scrubber Outlet	31
4.2	Scrubber Liquor Sampling Locations	31
5.0	SAMPLING AND ANALYSIS METHODS	34
5.1	EPA Reference Methods Used in This Program	34
5.2	Urea and Ammonia Sampling and Analysis	35
5.2.1	Sampling	35
5.2.2	Sample Recovery and Preparation	37
5.2.3	Sample Analysis	38
5.2.3.1	Analysis by TRC	38
5.2.3.2	Analysis by Agrico	40
5.3	Formaldehyde Sampling and Analysis	41
5.4	Insoluble Particulate Sampling and Analysis	41
5.5	Scrubber Liquor Sampling and Analysis	42
5.5.1	Sampling, Sample Recovery and Preparation	42
5.5.2	Sample Analysis	43
5.6	Urea Audit Samples - TRC and Agrico Analysis	44
5.6.1	Analysis by TRC	44
5.6.2	Analysis by Agrico	45

APPENDICES

A	Computer Printout Test Results
A-1	Granulator C Scrubber Outlet
A-2	Sample Equations and Example Calculations
B	Field Data Sheets

TABLE OF CONTENTS (Continued)

APPENDICES (Continued)

C	Sampling and Analysis Procedures
C-1	Urea Procedures
C-2	Ammonia Procedures
C-3	Formaldehyde Procedures
D	Analytical Data
D-1	Data Analysis Summaries
D-2	Chemical Laboratory Notebook
D-3	Scrubber Liquor Analysis Times
E	TRC/Agrico Joint Analyses
E-1	Agrico Field Sample Analyses
E-2	TRC Audit Sample Analyses
E-3	Agrico Audit Sample Analyses
F	Sampling Train Calibration Data
G	Process Operators Log
H	Project Participants
I	Scope of Work
	Work Assignments
	Technical Directives

LIST OF TABLES

<u>TABLE</u>		<u>PAGE</u>
2-1a	Summary of Urea, Ammonia and Formaldehyde Emissions from the C Granulator Scrubber Outlet at Agrico Chemical Company (English Units)	6
2-1b	Summary of Urea, Ammonia and Formaldehyde Emissions from the C Granulator Scrubber Outlet at Agrico Chemical Company (Metric Units)	7
2-2	Insoluble Particulate Analysis Results from the C Granulator Scrubber Outlet at Agrico Chemical Company .	10
2-3	TRC and Agrico <u>Urea</u> Analysis Results from the C Granulator Scrubber Outlet at Agrico Chemical Company . .	11
2-4	TRC and Agrico <u>Ammonia</u> Analysis Results from the C Granulator Scrubber Outlet at Agrico Chemical Company . .	13
2-5	C Granulator Scrubber Liquor Analysis Results from Agrico Chemical Company	16
2-6	Results from Urea Audit Sample Analyses Performed by TRC and Agrico at Agrico Chemical Company, Blytheville, Arkansas	18
3-1	Average Values and Ranges for Process and Control Equipment Operating Parameters During Emission Test Runs at Agrico Chemical Company, Blytheville, Arkansas	24
3-2	Variability of Three Process Operating Parameters During Emission Test Runs at Agrico Chemical Company, Blytheville, Arkansas	26
3-3	Comparison of Product Rates Calculated by Equation 1 and Production Rates Calculated from Corrected Totalizer Readings During the October 1978 Emission Tests at Agrico Chemical Company, Blytheville, Arkansas	29

LIST OF FIGURES

<u>FIGURE</u>		<u>PAGE</u>
1-1	Granulator Exhaust Ducting and Scrubbers at Agrico Chemical Company, Blytheville, Arkansas	3
3-1	Urea Manufacturing - Agrico Chemical Company, Blytheville, Arkansas	21
3-2	Joy Tubulaire Scrubber - Agrico Chemical Company, Blytheville, Arkansas	23
4-1	Locations of C Granulator Scrubber Outlet Test Ports and Points at Agrico Chemical Company in Blytheville, Arkansas	32
4-2	Joy Tubulaire Scrubber - Agrico Chemical Company, Blytheville, Arkansas	33
5-1	Modified EPA Particulate Sampling Train August 18, 1977, <u>Federal Register</u>	36

1.0 INTRODUCTION

1.1 Background

Section 111 of the Clean Air Act of 1970 charges the Administrator of the United States 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. Emission data, collected from controlled sources in the particular industry of concern, provide a portion of the data base used by EPA to develop SPNSS.

EPA's Office of Air Quality Planning and Standards (OAQPS) selected the Agrico Chemical Company urea manufacturing plant in Blytheville, Arkansas, as a site for an emissions test program. The program was designed to provide a portion of the emission data base required for SPNSS. In addition, emission samples obtained during this program were used as part of a urea analysis method investigation. The results of this investigation are presented in the EPA report 79-NHF-13 "Development of Analytical Procedures for the Determination of Urea from Urea Manufacturing Facilities".

EPA engaged TRC to measure urea, ammonia and formaldehyde in the exhaust gas of the granulator "C" scrubber at the Agrico urea plant. This report presents the results of this sampling program conducted under EPA contract #68-02-2820 and Technical Directives #1 and #2.

1.2 Measurement Program

The measurement program consisted of emissions tests performed by TRC at the Agrico Chemical Company urea manufacturing facility in Blytheville,

Arkansas, on December 18 and 19, 1978.

The Agrico plant produces granulated urea for industrial and fertilizer use. The urea is produced by three Spherodizer[®] granulators which operate continuously 24 hours a day, 7 days a week, as production demands. Each granulator has its own impingement-type water scrubber. Granulator exhaust air is ducted through the scrubber and fan and then discharged from a stack. Air 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 granulators' exhaust gas ducting and emission control system is shown in Figure 1-1.

The measurement program consisted specifically of the following:

1. Six one-hour emissions tests at the "C" granulator scrubber outlet. Sampling was performed for urea, ammonia, formaldehyde and insoluble particulate in the outlet gas stream.
2. Sampling of the scrubber inlet and outlet liquor at the beginning and end of each emissions test run.

The scrubber outlet gas stream and scrubber liquor samples were analyzed within 24 hours for urea and ammonia and within 20 days for formaldehyde and insoluble particulate. The urea and ammonia analyses of the gas stream samples were performed by TRC and Agrico, for comparison purposes. Urea analyses were performed using the Kjeldahl (with preliminary distillation) method.

Two identical sets of twelve urea audit samples were prepared by TRC according to specific EPA instructions. One set was analyzed by TRC, the other by Agrico; both analyses took place within 12 hours of sample preparation. While both analyses were performed using the Kjeldahl total nitrogen method (without preliminary distillation), the final ammonia content (from which the urea content was calculated) was determined by nesslerization

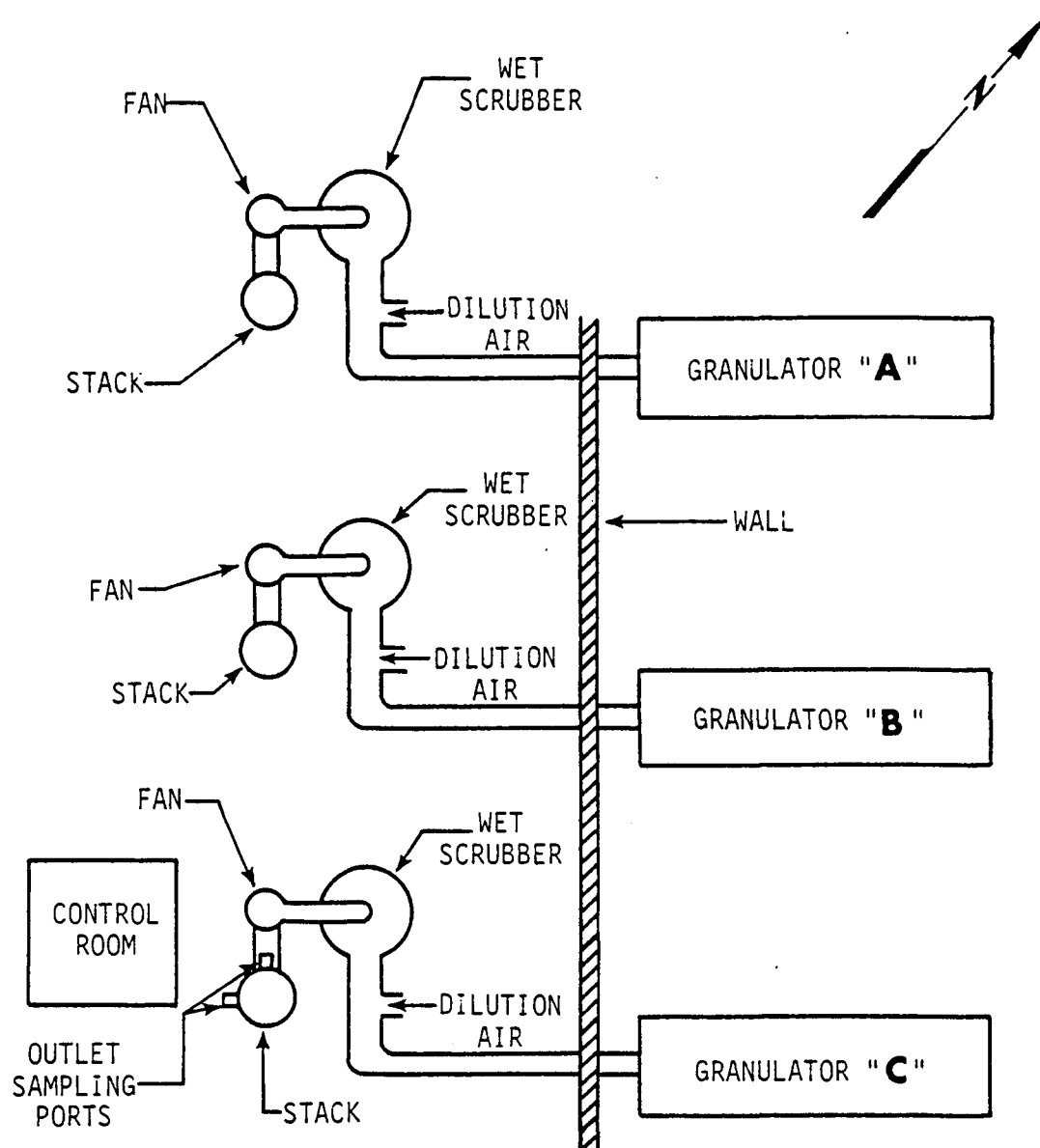


FIGURE 1-1: GRANULATOR EXHAUST DUCTING AND SCRUBBERS AT
 AGRICO CHEMICAL COMPANY IN
 BLYTHEVILLE, ARKANSAS

by TRC, and by titration by Agrico.

All sampling and measurements made at this facility were performed during times of normal urea production process operation, as described in Section 3.0, Process Description and Operations. The urea production rate from the "C" granulator during these tests was approximately 400 tons/day. TRC personnel were responsible for performing the above emissions testing and sampling. Concurrently, GCA was responsible for monitoring and recording pertinent process operation parameters. During the testing program the plant was producing fertilizer grade urea.

1.3 Description of Report Sections

The following sections of this report contain the summary of results (Section 2.0), process description and operations (Section 3.0), location of sampling points (Section 4.0), and descriptions of sampling and analysis methods (Section 5.0). Audit sample results are contained in Section 2.0. Detailed information on methods and procedures, and all field and laboratory data, are contained in their associated appendices, as noted in the Table of Contents.

2.0 SUMMARY OF RESULTS

This section presents the results of the emissions tests performed in December 1978 at the Agrico Chemical Company urea manufacturing plant in Blytheville, Arkansas. Testing was performed on the gas stream exiting, and on the liquor streams entering and exiting, the granulator "C" scrubber.

2.1 Granulator "C" Scrubber Outlet Gas Stream

The data from the granulator "C" scrubber outlet gas stream emissions tests are shown in Table 2-1. The urea and ammonia data represent the analyses performed by TRC at the Agrico laboratory within 24 hours of sample collection; the formaldehyde analyses were performed at TRC within 20 days of sample collection.

The urea and ammonia analyses included a common preliminary distillation step during which hydrolysis of some urea to ammonia is known to occur. The commonly used conversion factor is: 7 percent of the urea converts to ammonia during this preliminary distillation⁽¹⁾. The data in Table 2-1 are appropriately corrected to account for this conversion, using the 7 percent factor.

These scrubber outlet gas stream data differ considerably from the data obtained by TRC during emissions tests on the granulator "A" scrubber at this facility in October 1978. While the average ammonia gas stream concentration (grains/DSCF) in December is about 80% that in October, the urea concentrations in December are 3 times those of October; and the December formaldehyde concentrations are 16 times those of October. These differences may result in large part from differences in the granulators at this Agrico

(1) Standard Methods of Water and Wastewater Analysis, APHA, AWWA, WPCF, 14th edition, 1975 p. 408.

TABLE 2-1a (English)

SUMMARY OF UREA, AMMONIA, AND FORMALDEHYDE EMISSIONS FROM THE C GRANULATOR
SCRUBBER OUTLET AT AGRICO CHEMICAL COMPANY, BLYTHEVILLE, ARKANSAS

Run Number Date	Run 1 12-18-78	Run 2 12-19-78	Run 3 12-19-78	Run 4 12-19-78	Run 5 12-19-78	Run 6 12-19-78	Average
Volume of Gas Sampled (DSCF) ^a	34.93	34.44	32.62	33.14	32.41	33.62	33.53
Volumetric Flowrate (DSCFM) ^b	55180	54720	51130	52910	51730	53750	53237
Average Gas Temperature (°F)	92	102	104	103	105	104	102
Percent Moisture	6.0	3.8	5.1	4.9	3.1	3.8	4.5
Percent Isokinetic	107.2	106.7	108.2	106.2	106.2	106.1	106.8
Production Rate (Tons/Hour)	15.46	15.08	15.08	15.08	15.08	15.08	15.14
<u>Urea Data</u> ^c							
Total Sample Weight (Milligrams)	63.0	96.3	36.0	51.5	30.8	50.3	54.7
Grains/DSCF	0.02779	0.04306	0.01697	0.02391	0.01464	0.02304	0.02511
Pounds/Hour	13.14	20.19	7.438	10.85	6.492	10.61	11.46
Pounds/Ton	0.850	1.339	0.4932	0.7195	0.4305	0.7036	0.7569
<u>Ammonia Data</u> ^d							
Total Sample Weight (Milligrams)	420.7	324.4	591.5	346.2	320.7	303.5	384.5
Grains/DSCF	0.1855	0.1451	0.2792	0.1609	0.1524	0.1390	0.1766
Pounds/Hour	87.72	68.02	122.36	72.95	67.56	64.04	80.57
Pounds/Ton	5.674	4.511	8.114	4.837	44.80	4.247	5.322
<u>Formaldehyde Data</u> ^e							
Total Sample Weight (Milligrams)	3.90	4.70	3.30	4.24	2.05	3.14	3.56
Grains/DSCF	0.001719	0.002102	0.001558	0.001970	0.000974	0.001438	0.001635
Pounds/Hour	0.8131	0.9856	0.6827	0.8934	0.4318	0.6625	0.7460
Pounds/Ton	0.0526	0.06536	0.04527	0.05924	0.02863	0.04393	0.04927

^a Dry standard cubic feet @ 68°F and 29.92 inches Hg.

^b Dry standard cubic feet per minute.

^c Kjeldahl Analysis method with preliminary distillation, corrected for urea to ammonia conversion.

^d Nessler analysis method with preliminary distillation, corrected for urea to ammonia conversion.

^e Chromotropic Acid Analysis Method.

TABLE 2-1b (Metric)
SUMMARY OF UREA, AMMONIA, AND FORMALDEHYDE EMISSIONS FROM THE C GRANULATOR
SCRUBBER OUTLET AT AGRICO CHEMICAL COMPANY, BLYTHEVILLE, ARKANSAS

Run Number Date	Run 1 12-18-78	Run 2 12-19-78	Run 3 12-19-78	Run 4 12-19-78	Run 5 12-19-78	Run 6 12-19-78	Average
Volume of Gas Sampled (Nm ³) ^a	0.98922	0.97534	0.92380	0.93852	0.91785	0.95212	0.94957
Volumetric Flowrate (Nm ³ /min) ^b	1562.7	1549.7	1448.0	1498.4	1464.99	1522.2	1507.7
Average Gas Temperature (°C)	33	39	40	39	41	40	39
Percent Moisture	6.0	3.8	5.1	4.9	3.1	3.8	4.5
Percent Isokinetic	107.2	106.7	108.2	106.2	106.2	106.1	106.8
Production Rate (Mg/Hour)	14.025	13.681	13.681	13.681	13.681	13.681	13.735
<u>Urea Data</u>							
Total Sample Weight (mg)	63.0	96.3	36.0	51.5	30.8	50.3	54.7
Grams/Nm ³	0.06356	0.09851	0.03883	0.05472	0.03349	0.05271	0.05746
Kg/Hour	5.958	9.159	3.374	4.921	2.945	4.813	5.198
Kg/Mg	0.425	0.669	0.247	0.360	0.215	0.352	0.378
<u>Ammonia Data</u> ^d							
Total Sample Weight (mg)	420.7	324.4	591.5	346.2	320.7	303.5	384.5
Grams/Nm ³	0.4244	0.3320	0.6389	0.3681	0.3488	0.3180	0.4041
Kg/Hour	39.79	30.86	55.50	33.09	30.64	29.05	36.55
Kg/Mg	2.837	2.256	4.057	2.419	2.240	2.124	2.661
<u>Formaldehyde Data</u> ^e							
Total Sample Weight (mg)	3.90	4.70	3.30	4.24	2.05	3.14	3.56
Grams/Nm ³	0.00393	0.00481	0.00356	0.00451	0.00223	0.00329	0.00374
Kg/Hour	0.36882	0.44707	0.30967	.40525	0.19586	0.30051	0.33839
Kg/Mg	0.01315	0.03268	0.02264	0.02962	0.01432	0.02197	0.02464

^a Normal cubic meters @ 20°C, 760 mm Hg.

^b Normal cubic meters per minute.

^c Kjeldahl analysis method, corrected for urea to ammonia conversion.

^d Nessler analysis method with preliminary distillation, corrected for urea to ammonia conversion.

^e Chromotropic Acid analysis method.

plant. The three granulators (A, B, and C) in operation at this facility are not identical and, according to Agrico personnel, do have different operating characteristics. In particular, the lifting flights in granulator "C" are larger than those of "B" and "A". These devices help move the prills along inside granulators, and the larger ones in granulator "C" may have contributed to the noticeably higher plume opacity from "C" than from "A" and "B", as noted by Agrico personnel. The higher opacity presumably reflects different granulator operating characteristics.

The sampling train used during the December tests differed from that of the October tests in that the December impingers contained only water, while water and acid impingers were used in October. As a result, the ammonia collection efficiency may have been less than optimum during the December tests. If so, then the actual December ammonia concentrations themselves may equal or exceed those of October.

In December the ammonia analyses were performed both by direct nesslerization and by nesslerization with preliminary distillation⁽¹⁾; the two methods agreed within 10 percent (see Section 2.2). In October, direct nesslerization was used.

The same formaldehyde analysis method was used in December and in October (chromotropic acid method). A probable reason for the higher December formaldehyde results is contaminated distilled water. The water used in December for impinger charging and sample analysis was deionized through a resin which subsequently was found to contain significant amounts of formaldehyde.

The urea analysis methods differed between October and December: the Kjeldahl method was used in December, and the p-dimethylaminobenzaldehyde method was used in October. The differences between these two methods,

(1) *ibid.* pp. 407 ff.

however, would not account for more than a very small fraction of the observed disparity between the October and December urea concentrations.

The insoluble particulate analysis results of the granulator "C" scrubber outlet gas stream tests are shown in Table 2-2. These data indicate that the insoluble particulate content of the outlet gas stream is insignificant.

2.2 Comparison of TRC and Agrico Scrubber Outlet Gas Stream Analysis

The TRC and Agrico granulator "C" scrubber outlet analysis results are shown together in Tables 2-3 (urea results) and 2-4 (ammonia results). The TRC urea data were obtained directly using the Kjeldahl with preliminary distillation method⁽¹⁾. The Agrico urea data were obtained indirectly through separate Kjeldahl (total nitrogen)⁽¹⁾ and distillation/titrimetric (ammonia nitrogen)⁽²⁾ analyses; urea was then calculated by subtracting ammonia nitrogen from total nitrogen. Both corrected and uncorrected data are shown in Tables 2-3 and 2-4 (corrected for conversion of urea to ammonia during distillation, as discussed in Section 2.1).

The urea data in Table 2-3 show that on the average the Agrico results are 30% higher than the TRC results. Run by run, however, there is no consistency between the TRC and Agrico data; the Agrico results vary from much higher to much lower than the TRC results. There is no immediately evident reason for the differences between the two sets of data. The indirect method of analysis used by Agrico is susceptible to inaccuracy, since errors in the component analysis (for total nitrogen and ammonia nitrogen) may be compounded when urea nitrogen is calculated by subtraction. The Agrico analysis data (Appendix E) show that relatively small titrant volumes were used in these titration analyses: the total nitrogen titrant volumes ranged from 5.8 ml to 13.5 ml;

(1) *ibid.* pp. 437 ff.

(2) *ibid.* pp. 417 ff.

TABLE 2-2

INSOLUBLE PARTICULATE ANALYSES RESULTS FROM THE
 "C" GRANULATOR SCRUBBER OUTLET GAS STREAM AT
 AGRICO CHEMICAL COMPANY, BLYTHEVILLE, ARKANSAS

Run Number	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Average
Date	12-18-78	12-19-78	12-19-78	12-19-78	12-19-78	12-19-78	12-19-78
Volume of Gas Sampled (DSCF) ^a	34.93	34.44	32.62	33.14	32.41	33.62	33.53
Volumetric Flowrate (DSCFM) ^b	55180	54720	51130	52910	51730	53750	53237
Total Sample Weight (Milligrams)	2.08	1.82	0	0.18	0	1.13	0.87
Pounds/Hour	<0.001	<0.001	0	<0.001	0	<0.001	<0.001

^aDry Standard Cubic Feet @ 68°F, 29.92 inches Hg.

^bDry Standard Cubic Feet per minute.

TABLE 2-3

TRC AND AGRICO UREA ANALYSIS RESULTS
FROM "C" GRANULATOR SCRUBBER OUTLET GAS-STREAM
AT AGRICO CHEMICAL COMPANY, BLYTHEVILLE, ARKANSAS

Run Number	Run 1				Run 2			
Date	12-18-78				12-19-78			
Volume of Gas Sampled (DSCF) ^a	34.93				34.44			
Volumetric Flowrate (DSCFM) ^b	55180				54720			
Production Rate (Tons/hour)	15.46				15.08			
Urea Analysis By:	TRC ^c		Agrico ^d		TRC		Agrico	
	Uncorrected	Corrected	Uncorrected	Corrected	Uncorrected	Corrected	Uncorrected	Corrected
Total Sample Weight (Milligrams)	58.9	63.0	175.5	188.7	90.0	96.3	11.8	12.7
Grains/DSCF	0.02597	0.02779	0.07754	0.08338	0.04024	0.04306	0.00529	0.00569
Pounds/Hour	12.28	13.14	36.67	39.43	18.87	20.19	2.480	2.667
Pounds/Ton	0.794	0.850	2.372	2.551	1.251	1.339	0.164	0.176
Run Number	Run 3				Run 4			
Date	12-19-78				12-19-78			
Volume of Gas Sampled (DSCF) ^a	32.62				33.14			
Volumetric Flowrate (DSCFM) ^b	51130				52910			
Production Rate (Tons/hour)	15.08				15.08			
Urea Analysis By:	TRC ^c		Agrico ^d		TRC		Agrico	
	Uncorrected	Corrected	Uncorrected	Corrected	Uncorrected	Corrected	Uncorrected	Corrected
Total Sample Weight (Milligrams)	33.6	36.0	26.4	28.3	48.1	51.5	104.8	112.7
Grains/DSCF	0.01586	0.01697	0.01249	0.01343	0.02235	0.02391	0.04880	0.05247
Pounds/Hour	6.951	7.438	5.474	5.886	10.14	10.85	22.13	23.80
Pounds/Ton	0.461	0.493	0.363	0.390	0.672	0.719	1.468	1.578

^aDry standard cubic feet @ 68°F, 29.92 inches Hg.

^bDry standard cubic feet per minute.

^cTRC urea analysis by Kjeldahl with preliminary distillation. Corrected = uncorrected * 1.07.

^dAgrico urea analysis by total Kjeldahl nitrogen minus ammonia nitrogen = urea nitrogen. See Section 3.2 for details on data reduction and correction.

TABLE 2-3 (Cont.)
TRC AND AGRICO UREA ANALYSIS RESULTS
FROM "C" GRANULATOR SCRUBBER OUTLET GAS-STREAM
AT AGRICO CHEMICAL COMPANY, BLYTHEVILLE, ARKANSAS

Run Number	Run 5				Run 6			
Date	12-19-78				12-19-78			
Volume of Gas Sampled (DSCF) ^a	32.41				33.62			
Volumetric Flowrate (DSCFM) ^b	51730				53750			
Production Rate (Tons/hour)	15.08				15.08			
Urea Analysis By:	TRC ^c		Agrico ^d		TRC		Agrico	
	Uncorrected	Corrected	Uncorrected	Corrected	Uncorrected	Corrected	Uncorrected	Corrected
Total Sample Weight (Milligrams)	28.8	30.8	19.7	21.2	47.0	50.3	60.3	64.8
Grains/DSCF	0.01368	0.01464	0.00938	0.01009	0.02153	0.02304	0.02768	0.02976
Pounds/Hour	6.067	6.492	4.159	4.472	9.917	10.61	12.75	13.71
Pounds/Ton	0.402	0.430	0.276	0.297	0.658	0.704	0.846	0.910

Run Number	Average			
Volume of Gas Sampled (DSCF) ^a	33.53			
Volumetric Flowrate (DSCFM) ^b	53237			
Production Rate (Tons/hour)	15.14			
Urea Analysis By:	TRC		Agrico	
	Uncorrected	Corrected	Uncorrected	Corrected
Total Sample Weight (Milligrams)	51.1	54.7	66.4	71.4
Grains/DSCF	0.02347	0.02511	0.03056	0.03286
Pounds/Hour	10.71	11.46	13.95	15.00
Pounds/Ton	0.707	0.757	0.921	0.990

^aDry standard cubic feet @ 68°F, 29.92 inches Hg.

^bDry standard cubic feet per minute.

^cTRC urea analysis by Kjeldahl with preliminary distillation. Corrected = uncorrected * 1.07.

^dAgrico urea analysis by total Kjeldahl nitrogen minus ammonia nitrogen = urea nitrogen. See Section 3.2 for details on data reduction and correction.

TABLE 2-4

TRC AND AGRICO AMMONIA ANALYSIS RESULTS
FROM "C" GRANULATOR SCRUBBER OUTLET GAS-STREAM
AT AGRICO CHEMICAL COMPANY, BLYTHEVILLE, ARKANSAS

Run Number Date	Run 1 12-18-78					Run 2 12-19-78				
Volume of Gas Sampled (DSCF) ^a	34.93					34.44				
Volumetric Flowrate (DSCFM) ^b	55180					54720				
Production Rate (Tons/hour)	15.46					15.08				
Ammonia Analysis By:	TRC ^c					Agrico ^d				
	DN	Dist.-N Uncorrected	Dist.-N Corrected	Dist.-T Uncorrected	Dist.-T Corrected	DN	Dist.-N Uncorrected	Dist.-N Corrected	Dist.-T Uncorrected	Dist.-T Corrected
Total Sample Weight (Milligrams)	403.7	423.2	420.7	464.1	456.6	332.6	328.2	324.4	484.7	484.2
Grains/DSCF	0.1780	0.1866	0.1855	0.2050	0.2017	0.1487	0.1468	0.1451	0.2172	0.2170
Pounds/Hour	84.17	88.24	87.72	96.98	95.41	69.74	68.82	68.02	101.9	101.8
Pounds/Ton	5.444	5.708	5.674	6.273	6.172	4.625	4.564	4.511	6.755	6.748
Run Number Date	Run 3 12-19-78					Run 4 12-19-78				
Volume of Gas Sampled (DSCF) ^a	32.62					33.14				
Volumetric Flowrate (DSCFM) ^b	51130					52910				
Production Rate (Tons/hour)	15.08					15.08				
Ammonia Analysis By:	TRC					Agrico				
	DN	Dist.-N Uncorrected	Dist.-N Corrected	Dist.-T Uncorrected	Dist.-T Corrected	DN	Dist.-N Uncorrected	Dist.-N Corrected	Dist.-T Uncorrected	Dist.-T Corrected
Total Sample Weight (Milligrams)	369.6	592.9	591.5	381.5	380.4	362.8	348.2	346.2	369.4	364.9
Grains/DSCF	0.1745	0.2799	0.2792	0.1805	0.1800	0.1686	0.1618	0.1609	0.1720	0.1699
Pounds/Hour	76.46	122.65	122.36	79.10	78.9	76.45	73.37	72.95	78.01	77.06
Pounds/Ton	5.070	8.133	8.114	5.245	5.230	5.070	4.865	4.837	5.173	5.110

^a Dry standard cubic feet @ 68°F, 29.92 inches Hg.

^b Dry standard cubic feet per minute.

^c TRC ammonia analysis done by direct nesslerization (DN) and distillation/nesslerization (Dist.-N). Correction is for urea to ammonia conversion. Corrected = uncorrected - 0.07 * corrected urea/1.765.

^d Agrico ammonia analysis done by distillation/titration (Dist.-T). Correction is for urea to ammonia conversion. See Section 3.2 for details on data reduction and correction.

TABLE 2-4 (Cont.)

TRC AND AGRICO AMMONIA ANALYSIS RESULTS
FROM "C" GRANULATOR SCRUBBER OUTLET GAS-STREAM
AT AGRICO CHEMICAL COMPANY, BLYTHEVILLE, ARKANSAS

Run Number	Run 5					Run 6				
Date	12-19-78					12-19-78				
Volume of Gas Sampled (DSCF), ^a	32.41					33.62				
Volumetric Flowrate (DSCFM), ^b	51730					53750				
Production Rate (Tons/hour)	15.08					15.08				
Ammonia Analysis by:										
	TRC ^c			Agrico ^d		TRC			Agrico	
	DN	Dist.-N Uncorrected	Dist.-N Corrected	Dist.-T Uncorrected	Dist.-T Corrected	DN	Dist.-N Uncorrected	Dist.-N Corrected	Dist.-T Uncorrected	Dist.-T Corrected
Total Sample Weight (Milligrams)	341.6	321.9	320.7	353.7	352.9	301.5	305.5	303.5	300.7	298.1
Grains/DSCF	0.1623	0.1530	0.1524	0.1684	0.1680	0.1381	0.1399	0.1390	0.1380	0.1368
Pounds/Hour	71.96	67.81	67.56	74.68	74.51	63.62	64.46	64.04	63.59	63.04
Pounds/Ton	4.772	4.497	4.480	4.952	4.941	4.219	4.275	4.247	4.217	4.181

Run Number	Average				
Volume of Gas Sampled (DSCF), ^a	33.53				
Volumetric Flowrate (DSCFM), ^b	53237				
Production Rate (Tons/hour)	15.14				
Ammonia Analysis By:					
	TRC ^c			Agrico ^d	
	DN	Dist.-N Uncorrected	Dist.-N Corrected	Dist.-T Uncorrected	Dist.-T Corrected
Total Sample Weight (Milligrams)	352.0	386.7	384.5	392.4	389.6
Grains/DSCF	0.1617	0.1776	0.1766	0.1806	0.1793
Pounds/Hour	73.76	81.03	80.57	82.41	81.82
Pounds/Ton	4.872	5.352	5.322	5.465	5.426

^a Dry standard cubic feet @ 68°F, 29.92 inches Hg.

^b Dry standard cubic feet per minute.

^c TRC ammonia analysis done by direct nesslerization (DN) and distillation/nesslerization (Dist.-N). Correction is for urea to ammonia conversion. Corrected = uncorrected - 0.07 * corrected urea/1.765.

^d Agrico ammonia analysis done by distillation/titration (Dist.-T). Correction is for urea to ammonia conversion. See Section 3.2 for details on data reduction and correction.

the ammonia nitrogen titrant volumes ranged from 5.4 ml to 11.5 ml. In order to minimize titration errors, TRC has found that titrant volumes of at least 20 ml should be used. For these reasons, and because the TRC data are more consistent, the TRC urea data are considered more accurate.

The ammonia data in Table 2-4 show that on the average, the TRC and Agrico results are in close agreement. TRC utilized two analysis methods: direct nesslerization and nesslerization with preliminary distillation. Agrico utilized the titration method with preliminary distillation.

2.3 Scrubber Liquor Sampling Results

Two samples were collected from both the inlet and the outlet liquor streams of the granulator "C" scrubber during each emission test run. At the end of each test run the individual samples obtained during that run were combined into two composite samples: one inlet sample and one outlet sample. These were then analyzed by TRC for urea and ammonia at the Agrico laboratory, and for formaldehyde and insoluble particulate at TRC. The analysis results are shown in Table 2-5. Procedural difficulties precluded obtaining any reliable insoluble particulate data. The same analysis methods used on the scrubber gas stream samples were also used on the scrubber liquor samples. And the same distillation correction factor was applied to the urea and distilled ammonia data. Because the urea concentrations in the outlet liquor greatly exceed the ammonia concentrations, the "corrected" outlet ammonia concentrations are negative. This result illustrates the potential inaccuracy inherent in this correction method when it is applied to samples containing large concentrations of urea.

The urea, direct nesslerization ammonia and formaldehyde data in Table 2-5 generally agree with the data obtained during the October 1978 emissions tests on the granulator "A" scrubber at this Agrico facility. Two exceptions are,

TABLE 2-5
 "C" GRANULATOR SCRUBBER LIQUOR ANALYSIS RESULTS
 FROM AGRICO CHEMICAL COMPANY, BLYTHEVILLE, ARKANSAS

Run Number Date	INLET (ppm)						Average
	1 12-18-78	2 12-19-78	3 12-19-78	4 12-19-78	5 12-19-78	6 12-19-78	
Urea Data ^a							
Uncorrected	29387	38830	38830	28858	35079	35962	34491
Corrected	31444	41548	41548	30878	37535	38479	36905
Ammonia Data							
Direct Nesslerization	7300	*	*	5900	*	*	6600
Dist. - N (uncorrected) ^b	8167	6800	7000	6050	6600	6200	6803
Dist. - N (corrected) ^c	6920	5152	5352	4825	5111	4674	5339
Formaldehyde Data ^d	18.25	38.00	38.00	14.25	16.89	14.63	23.34

Run Number Date	OUTLET (ppm)						Average
	1 12-18-78	2 12-19-78	3 12-19-78	4 12-19-78	5 12-19-78	6 12-19-78	
Urea Data ^a							
Uncorrected	458900	434630	498610	423600	483170	454490	458900
Corrected	491020	46510	53350	453250	516990	486300	491020
Ammonia Data							
Direct Nesslerization	2110	*	*	2400	*	*	2255
Dist. - N (uncorrected) ^b	14650	10650	8800	11400	9200	8350	10508
Dist. - N (corrected) ^c	**	**	**	**	**	**	**
Formaldehyde Data ^d	<0.05	0.21	0.21	0.19	0.30	0.19	0.22

^a Kjeldahl with preliminary distillation analysis method. Correction applied for urea to ammonia conversion. Corrected = uncorrected * 1.07.

^b Nessler analysis method with preliminary distillation.

^c Correction for urea to ammonia conversion. Corrected = uncorrected - 0.07 * corrected urea/1.765.

^d Chromotropic Acid Analysis method.

* Analysis not performed.

** Correction for urea to ammonia conversion yields negative values.

Note: Insoluble particulate measurements were not accurate and are not presented. See Section 3.2 for details.

however, worthy of note:

- o Inlet ammonia concentration - in October the average inlet ammonia concentration was 13900 ppm; the average in Table 2-5 is 6600 ppm (direct nesslerization).
- o Outlet urea concentration - in October the average outlet urea concentration was 689,400 ppm; the average in Table 2-5 is 458,900 ppm (uncorrected).

The higher outlet gas stream urea grain loading in these December tests compared to the October tests should be reflected in a higher scrubber liquor urea concentration. If, however, scrubber "C" is less efficient than scrubber "A", then the urea data are reasonable. The December and October gas stream ammonia data are comparable, which would tend to indicate that the liquor ammonia results should also be comparable. If, however, much of the ammonia in the liquor comes from the breakdown of urea, then the ammonia liquor data are reasonable. The inlet and outlet liquor ammonia data (direct nesslerization) also show evidence of ammonia stripping, whereby ammonia in the liquor is transferred (presumably) to the gas stream.

2.4 Urea Audit Samples - Comparison of TRC and Agrico Analyses

TRC and Agrico each analyzed a different set of twelve urea samples, each set prepared by TRC according to specific EPA instructions. Both analyses were performed at the Agrico laboratory within 12 hours of sample preparation. The TRC audit sample set was analyzed using the total Kjeldahl nitrogen method with no preliminary distillation, ending with nesslerization⁽¹⁾. The Agrico audit sample set was analyzed using the same total Kjeldahl nitrogen method, but ending with titration. The results of the urea audit sample analyses are shown in Table 2-6.

⁽¹⁾ *ibid.* pp. 437 ff.

TABLE 2-6

RESULTS OF UREA AUDIT SAMPLE ANALYSES
PERFORMED BY TRC AND AGRICO
AT AGRICO CHEMICAL COMPANY, BLYTHIEVILLE, ARKANSAS

Audit Sample	TRC Analysis*			AGRICO Analysis**			
	Actual Urea Sample Weight (mg)	As Measured (mg)	Error ^a (%)	Actual Urea Sample Weight (mg)	Measured As Nitrogen (mg)	Equivalent Urea ^b (mg)	Error ^c (%)
	A	B		C	D	E	
1	100.71	94.04	-6.6	100.54	96.3	206.4	105
2	311.98	288.90	-7.4	292.78	281.1	602.4	106
3	598.36	568.75	-4.9	598.08	582.4	1248.0	109
4	5.64	5.44	-3.5	5.26	3.6	7.7	46.4
5	11.60	11.15	-3.9	9.64	11.8	25.3	162
6	40.40	38.69	-4.2	42.48	38.6	82.7	94.7
7	2.60	2.43	-6.5	2.04	1.1	2.4	17.6
8	6.84	6.49	-5.1	6.16	5.0	10.7	73.7
9	9.42	8.96	-4.9	9.54	9.5	20.4	114
10	5.40	4.90	-9.3	5.96	5.3	11.4	91.3
11	4.30	3.93	-8.6	4.18	3.9	8.4	101
12	30.16	27.93	-7.4	31.32	27.4	58.7	87.4
Average			-6.0				92.9

^a Percent error = $(100 * B/A) - 100$

^b $E = D * 60/28$

^c Percent error = $(100 * E/C) - 100$

* TRC Analysis by total Kjeldahl nitrogen method, ending with Nesslerization. No preliminary distillation.

** Agrico analysis by total Kjeldahl nitrogen method, ending with Titration. No preliminary distillation.

The TRC analysis results average 6.0 percent lower than the actual urea sample weights, and each sample analysis is less than the actual. It was initially thought that the consistently low results were due to the blank correction. Discounting the blank correction in the analysis calculation however, yields an overall +5.0 percent error, indicating that factors other than the blank correction may also be involved in the consistently low (blank corrected) results.

The Agrico analysis results average 92.9 percent higher than the actual urea sample weights and the reason for this large error is not immediately evident. These analyses were concluded with with titration, and the Agrico analysis data (Appendix E) indicate that very low titration volumes were often used (seven of the twelve titrations required less than 6 ml of titrant). TRC has found that larger titrant volumes (at least 20 ml) are necessary in order to help minimize errors during titration. A disadvantage of the titration method is that the entire sample is used for one titration; consequently, if an error is made or if a result is suspect, there is no possibility of re-analysis.

Because the titration results are reported as mg nitrogen, conversion of mg nitrogen to mg urea is required and is performed stoichiometrically: 2 moles (28 grams) of nitrogen are contained in 1 mole (60 grains) of urea. The underlying assumption for using this conversion (and for not using preliminary distillation, for that matter) is that all the nitrogen in the samples originated as urea.

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3.0 PROCESS DESCRIPTION AND OPERATIONS

3.1 Process Equipment

This Agrico urea manufacturing facility employs three rotary drum granulators designed by C&I Girdler as the solids forming devices. A single urea solution synthesis process supplies all three granulators. A schematic of the urea manufacturing process is shown in Figure 3-1, showing one of the three granulators and related equipment.

The concentrated molten urea, referred to as melt, leaves the solution synthesis process and is pumped to the granulators. The molten urea is sprayed onto a bed of solid urea "seed" particles at the higher end of the inclined granulator. Lifting flights 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 collection 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 conveyed to a bulk storage warehouse.

The airstream through the granulators entrains significant quantities of urea and recovery of this material is essential for this solids formation technique to be economically viable. A Joy Turbulaire "Type D" scrubber is employed with each granulator to remove most of the particulate from the granulator exhaust. After passing through the granulator, the air is drawn by a fan through the scrubber and out a stack.

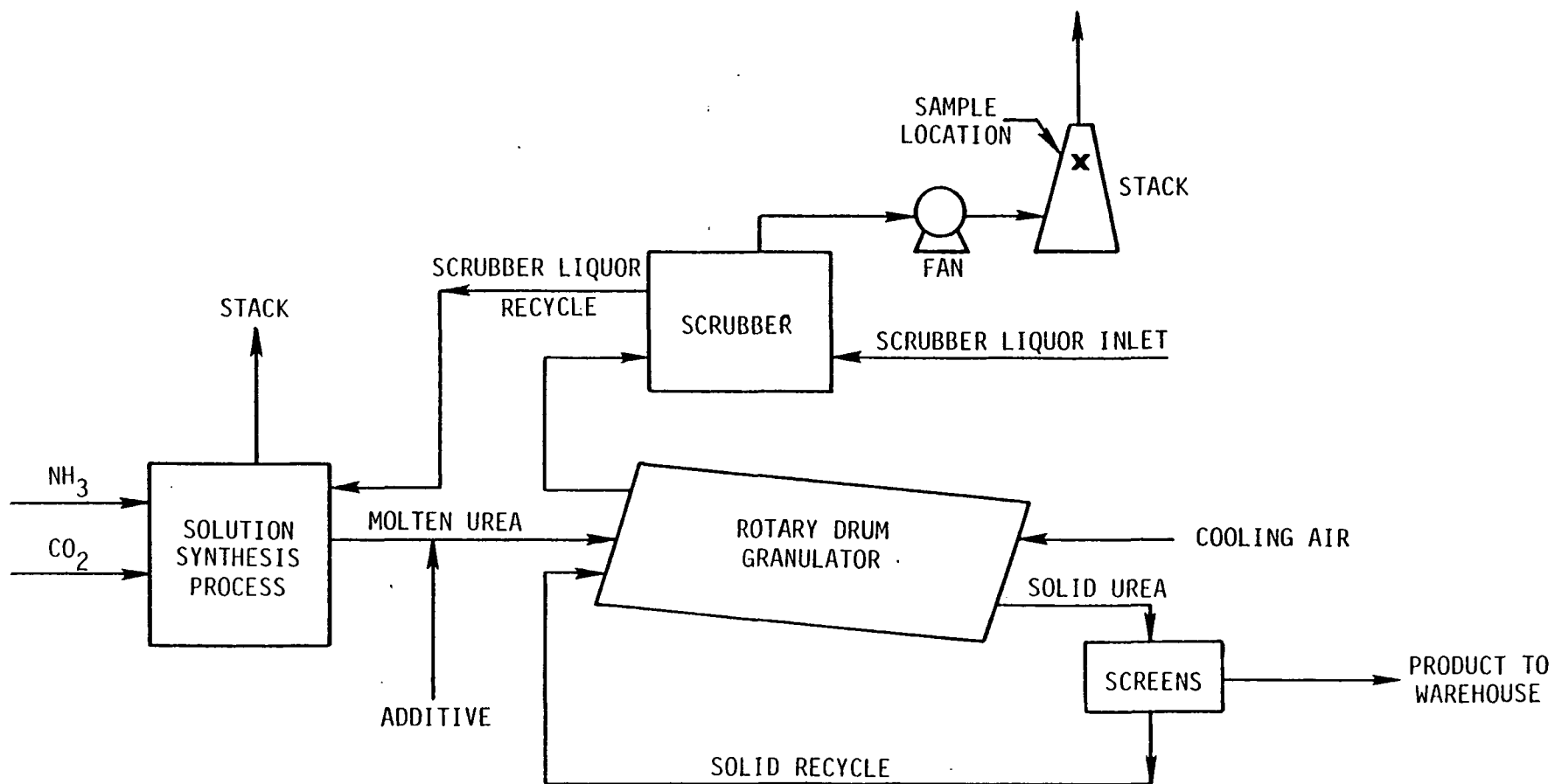


FIGURE 3-1: UREA MANUFACTURING - AGRICO CHEMICAL COMPANY,
BLYTHEVILLE, ARKANSAS

This scrubber can be operated at varied pressure drops by adjusting the scrubber liquor level. In order to meet particulate emission limitations, this plant operates the scrubbers at a pressure drop in excess of 14 inches W.G. Cleaned process condensate from the urea synthesis operation is used as make-up scrubber liquor. The urea concentration of the liquor is maintained at 45 percent to 50 percent. Scrubber liquor is returned to the solution synthesis process for urea recovery. A schematic of the scrubber, depicting air and liquor flow streams, is shown in Figure 3-2.

3.2 Process Operation

Emission testing was conducted by TRC on the exhaust from the "C" granulator scrubber. During each emission test run, GCA monitored and recorded process and control equipment operating parameters to ensure that the process operated at representative, steady-state conditions. GCA also obtained composite scrubber inlet and outlet liquor samples from the "C" granulator scrubber during the test runs.

During the emissions testing on December 18 and 19, 1978, fourteen process parameters were monitored in order to determine granulator production rate and process stability. Relative parameter values, expressed as a percent of the mean value over the two-day testing period, are shown in Table 3-1. Urea melt temperature and the "C" granulator inlet and outlet air temperature values are considered confidential. Appendix G contains all raw data values.

The data in Table 3-1 show that some parameters remained relatively constant, while others varied considerably over the two test periods. The parameters which varied the most are the Urea Solution Tank Level on the 19th, the Additive Feed Rate on both days, and the Scrubber Liquor Level on both days. The high value for the Spray Nozzle Pressure was 12.4 and 14.0 percent

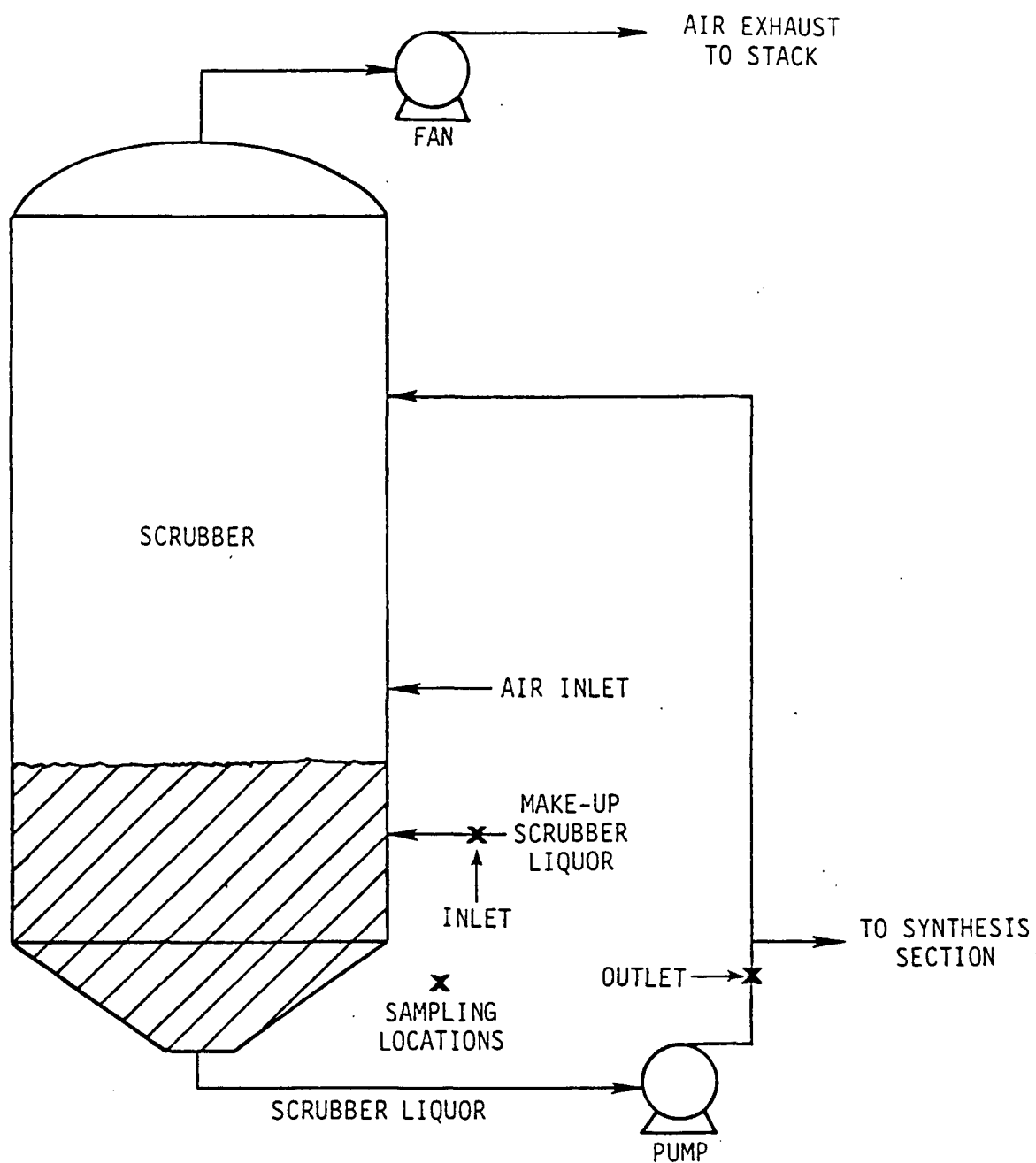


FIGURE 3-2: JOY TURBULAIRE SCRUBBER - AGRICO CHEMICAL COMPANY,
BLYTHEVILLE, ARKANSAS

TABLE 3-1

AVERAGE VALUES AND RANGES FOR PROCESS AND CONTROL EQUIPMENT
OPERATING PARAMETERS DURING EMISSION TEST RUNS
AT AGRICO CHEMICAL COMPANY, BLYTHEVILLE, ARKANSAS

Parameter	Symbol	12/18/78	1:55p-4:10p	12/19/78	9:05a-5:20p
		Mean*	Range*	Mean*	Range*
Ammonia Feed Rate	NH ₃ Feed	98	98-102	101	98-103
Urea Solution Tank Level	TK-101	93	91-94	102	91-124
Additive Feed Rate	AFR	95	82-106	102	78-116
Urea Melt Temperature	UMT	†	†	†	†
Granulator Spray Nozzle Pressure	GSPC	103	97-109	99	93-106
Granulator Inlet Air Temperature	AIGT	†	†	†	†
Granulator Outlet Air Temperature	AOGT	†	†	†	†
Scrubber Liquor Level	SLI	104	90-111	98	85-104
Scrubber Fan Amps	SFA	100	99-102	100	99-102
Scrubber Liquor Temperature	SLT	93	93-94	102	100-107
Scrubber Liquor Feed Rate	ISLF	*	*	*	*
Scrubber Outlet Air Temperature	AOS	93	89-93	103	100-105

*Values expresses as percentages of overall mean values for both test periods.

†Confidential Readings.

*Readings were inaccurate or monitoring device was broken during test period.

higher than the low reading on the 18th and 19th, respectively. Since melt throughput is proportional to the square-root of the pressure drop, the highest throughputs were only 6.0 percent and 6.9 percent higher than the lowest throughput for each day.

The recorded values for Urea Solution Tank Level, Additive Feed Rate, and Scrubber Liquor Levels varied enough to merit further scrutiny. Mean values, standard deviations, and variation ranges of these three parameters during the six sampling runs are shown in Table 3-2. Although all three exhibit significant fluctuations in mean value from run to run, only the Additive Feed Rate readings varied substantially over the course of a single run (a single run lasted 1 hour).

It is important to point out that readings for all three of these parameters are uncalibrated values. In the case of the Additive Feed Rate, the value is followed to maintain steady conditions; for the two liquid levels, the plant attempts to keep the readings at values which they know from experience correspond to the design levels. It is not known to what extent fluctuations in the readings reflect variations in the actual parameters. For instance, does a 10 percent change in the Scrubber Liquor Level reading reflect a 10 percent change in actual scrubber liquor depth or does the monitoring device scale cover only a fraction of the total depth? In this case, the actual fluctuation in the liquor depth is far less than that depicted by the readings. The extent to which fluctuations in Scrubber Liquor Depth readings affect the air passage above the sump, and hence the airstream velocity, is not known.

Production rate data initially appeared ambiguous. The production totalizer readings for the "C" granulator, when corrected using the correction factor developed during tests conducted October 9 to 13, 1978, yielded

TABLE 3-2

VARIABILITY OF THREE PROCESS OPERATING PARAMETERS DURING EMISSION TESTS RUNS
AT AGRICO CHEMICAL COMPANY, BLYTHEVILLE, ARKANSAS

Run Number	Date	Time Span†	TK-101 Urea Solution Tank Level			AFR Additive Feed Rate			SLL Scrubber Liquor Level		
			Mean	Standard Deviation	Range*	Mean	Standard Deviation	Range*	Mean	Standard Deviation	Range*
1	12/18	1:55p-3:00p	-	-	-	2.8	0.29	82-106	42.2	0.51	107-111
2	12/19	9:05a-10:20a	16.1	0.45	94-100	3.3	0.11	106-117	39.1	0.19	101-102
3	12/19	11:00a-12:00p	15.3	0.24	95-98	3.2	0.13	106-117	40.0	0.32	102-104
4	12/19	1:10p-2:10p	16.1	0.20	96-99	2.6	0.16	80-97	38.0	0.89	99-106
5	12/19	2:55p-3:55p	17.3	0.68	99-112	2.8	0.27	81-108	39.1	0.86	97-103
6	12/19	4:10--5:20p	19.5	0.65	112-124	3.0	0.24	85-106	35.4	1.37	85-95

*Range values are expressed as percentages of the overall average for the entire testing period.

†Time spans are meant to encompass the period when sampling occurred and are not start and finish times for the actual sampling.

unrealistic production rates. It was evident that the "C" totalizer had been adjusted since those tests. A new correction factor was therefore developed for the "C" granulator totalizer (as detailed in Appendix G), and production rates were recalculated. These calculated production rates appeared to be more reasonable but were not used because they are valid only if the correction factor for the "A" granulator totalizers did not change. Product totalizers are not considered to be accurate production rate indicators by plant personnel, who use them mostly to indicate changes in production rates. Spray nozzle pressure was then selected as a more valid indicator of production rate. It is a reasonably good method if the physical characteristics of the urea melt do not change significantly from day to day and if the characteristics of the spray nozzles do not change substantially due to wear or urea buildup.

One of the important concepts on which the original correction factors were based was that the urea melt spray conformed to the orifice equation and that, therefore, the flow rate through each nozzle was proportional to the square root of the pressure drop across the nozzle. Carrying this concept one step further, and applying the assumptions of constant melt properties and constant nozzle characteristics, production rates can be calculated using the simplified orifice equation:

$$G = K \sqrt{\Delta P} \quad (1)$$

where

G = Melt flowrate, tons/minute

K = Empirical constant, tons/(Minute · psig^{1/2})

ΔP = Pressure drop across nozzles, psig.

The constant K is a function of fluid, nozzle, and flow properties which are assumed constant for this system. The constant K was calculated to be

0.0434 based on data collected at Agrico during the October 9-13, 1978 tests.

A comparison of production rates as calculated by totalizer readings and production rates calculated from nozzle pressure readings is presented in Table 3-3 for granulators A, B and C for the October emissions tests. The average difference between these production measurement methods was 2.6% for all granulators and 2.7% for granulator "C".

Assuming that no significant change occurred in nozzle or melt characteristics between the October 1978 and the December 1978 test dates, the value of 0.0434 can be used in Equation (1) to calculate average production rates for 18 December and 19 December, 1978. The results are shown below:

PRODUCTION RATES OF "C" GRANULATOR DURING DECEMBER 1978 TESTS
BASED ON EQUATION (1)

<u>Date</u>	<u>Time</u>	<u>Average P, psig</u>	<u>G Tons/Min.</u>	<u>G Ton/Day</u>
Dec. 18, 1978	1:55p-4:10p	35.2	0.257	371
Dec. 19, 1978	9:05a-5:20p	33.6	0.252	362

To assure that the scrubber on the "C" granulator was operating properly during testing, scrubber liquor samples were taken during each emission test run. Agrico preferred that their personnel draw the necessary scrubber liquor samples. GCA observed the sample collection and took immediate custody of the samples. Inlet and outlet liquor samples were taken at the beginning and end of each test run and these samples were then analyzed for urea, ammonia, formaldehyde and percent solids. The sampling locations are shown in Figure 3-2. The actual times that the samples were collected are listed in Appendix G.

TABLE 3-3

COMPARISON OF PRODUCTION RATES CALCULATED BY EQUATION 1
AND PRODUCTION RATES CALCULATED FROM CORRECTED TOTALIZER READINGS
DURING THE 9-13 OCTOBER 1978 EMISSIONS TESTS
AT AGRICO CHEMICAL COMPANY, BLYTHEVILLE, ARKANSAS

"A" GRANULATOR				"B" GRANULATOR				"C" GRANULATOR			
G† (tons/min)	ΔP Spray nozzles (psig)	G* (tons/min)	Error (%)	G† (tons/min)	ΔP Spray nozzles (psig)	G* (tons/min)	Error (%)	G† (tons/min)	ΔP Spray nozzles (psig)	G* (tons/min)	Error (%)
0.270	40.5	0.276	2.2	0.275	41.0	0.278	1.1	0.278	41.0	0.278	0.0
0.273	40.5	0.276	1.1	0.279	40.5	0.276	1.1	0.280	41.0	0.278	0.7
0.270	40.0	0.274	1.5	0.278	39.0	0.271	2.5	0.270	40.5	0.276	2.2
0.276	40.0	0.274	0.7	0.285	40.0	0.274	3.9	0.292	43.0	0.285	2.4
0.277	40.5	0.276	0.4	0.281	40.5	0.276	1.8	0.286	42.0	0.281	1.7
0.294	41.0	0.278	5.4	0.289	42.0	0.281	2.8	0.285	42.0	0.281	1.4
0.248	37.5	0.266	7.3	0.254	35.0	0.257	1.2	0.267	36.5	0.262	1.9
0.270	35.5	0.259	4.1	--	--	--	--	0.276	38.0	0.268	2.9
0.273	40.5	0.276	1.1	0.265	39.5	0.273	3.0	0.264	40.0	0.274	3.8
0.260	38.0	0.268	3.1	0.260	34.5	0.255	1.9	0.278	36.5	0.262	5.8
0.247	37.0	0.264	6.9	0.253	33.5	0.251	0.8	0.277	35.5	0.259	6.5

G† - Production rate based on corrected totalizer readings.

G* - Production rate based on empirical equation using pressure drop across spray nozzles,
(See Equation 1).

$$\text{Error} = \left| \left(\frac{G^*}{G^\dagger} \right) - 1 \right| \times 100$$

Scrubber operating parameters were also recorded during the emission test runs in order to monitor the stability of this device. The variation of these parameters is shown in Table 3-1. The higher operating temperatures recorded on December 19 probably reflect the higher ambient air temperature that occurred that day. The ambient air temperature on December 19 was 15-20° higher than on December 18. The affect of temperature on collection efficiency is not known.

4.0 LOCATION OF SAMPLING POINTS

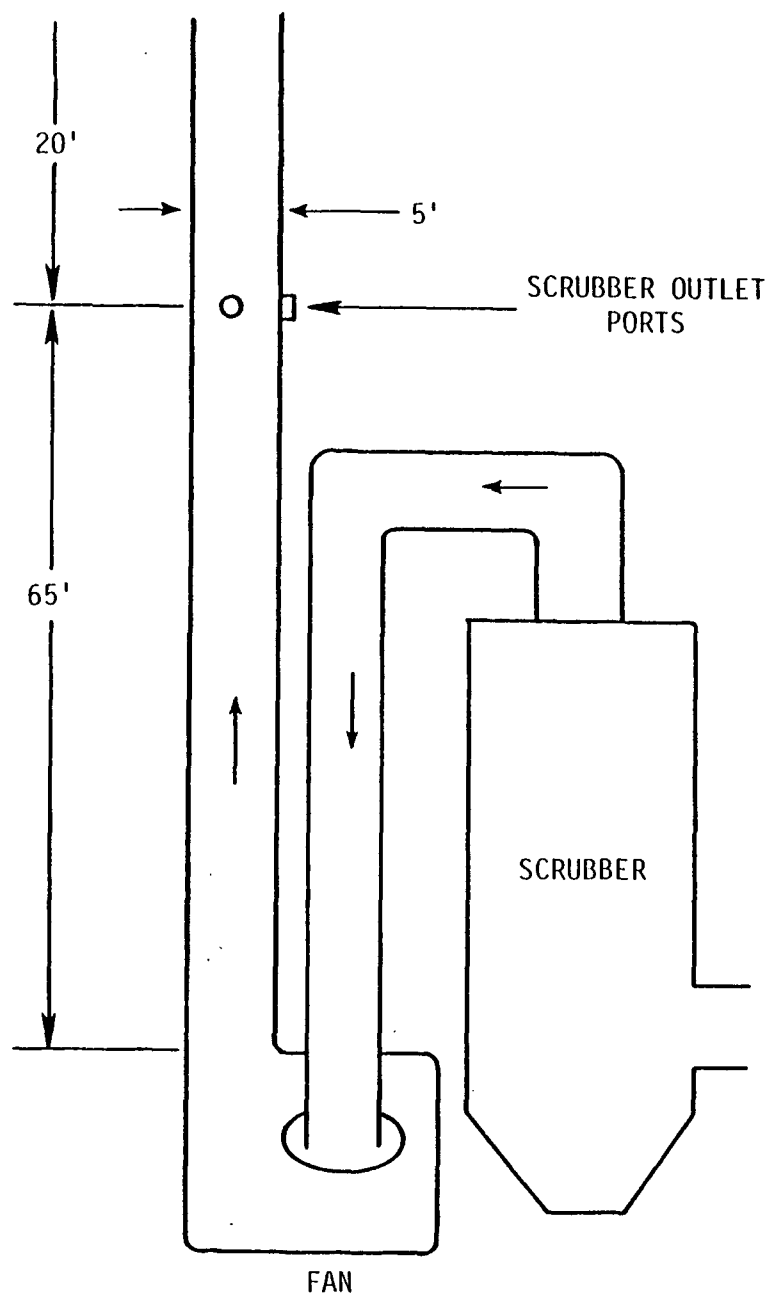
This section presents descriptions of the sampling locations used during the emissions testing program at the Agrico Chemical Company urea manufacturing plant in Blytheville, Arkansas on December 18 and 19, 1978.

4.1 Granulator C Scrubber Outlet

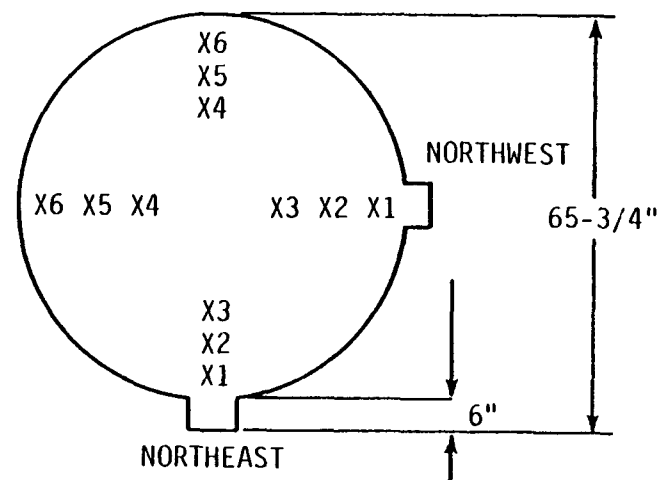
The cleaned gases exiting the scrubber unit are ducted to an induced draft fan adjacent to the emission control unit. The fan discharge is directed vertically through a steel stack to the atmosphere. The "C" scrubber 60-inch I.D. outlet stack was fitted with two 4-inch I.D. pipe-flanged sampling ports positioned 90 degrees apart in a horizontal plane. The two ports were located 65 feet (13 stack diameters) downstream of the fan outlet, and 20 feet (4 stack diameters) upstream of the stack discharge. Since these port locations met the "eight and two diameters" criteria for distance from flow disturbances, six sampling points were chosen for each axis traverse, for a total of twelve sampling points as specified by EPA Reference Method 1. Figure 4-1 shows a cross-sectioned view of the duct at the sampling location and lists the exact distance of each sampling point from the outside flange edge.

4.2 Scrubber Liquor Sampling Locations

Granulator C scrubber liquor samples were collected from the liquor make-up line (cleaned process condensate from the urea synthesis operation) and from the return liquor line downstream from the circulating pump. Figure 4-2 shows these sampling locations.



A - LOCATION OF TEST PORTS



TRAVERSE POINT NO.	TRAVERSE POINT DISTANCE FROM OUTSIDE EDGE OF NIPPLE (IN.)
1	8-5/8
2	14-3/4
3	23-3/4
4	48-1/8
5	57
6	53-1/8

B - LOCATION OF TEST POINTS

FIGURE 4-1: LOCATIONS OF "C" GRANULATOR SCRUBBER OUTLET TEST PORTS AND POINTS AT AGRIC CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

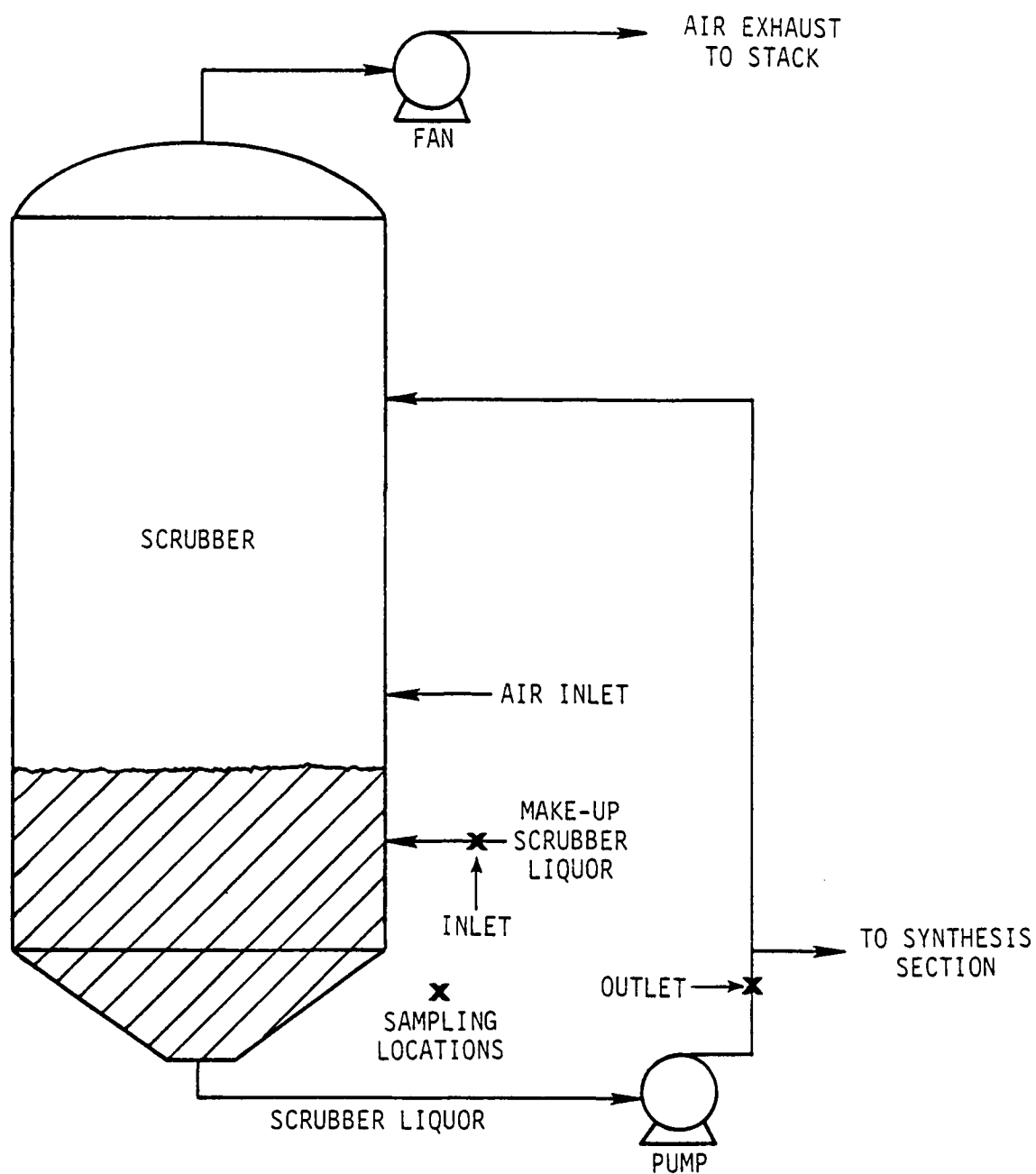


FIGURE 4-2: JOY TURBULAIRE SCRUBBER - AGRICO CHEMICAL COMPANY,
BLYTHEVILLE, ARKANSAS

5.0 SAMPLING AND ANALYSIS METHODS

This section presents general descriptions of sampling and analysis procedures employed during the emissions testing program conducted at the Agrico Chemical Company, Blytheville, Arkansas, urea manufacturing facility during December 18 and 19, 1978. Details of sampling and analysis procedures are contained in Appendices C and D.

5.1 EPA Reference Methods Used in This Program

The following EPA Reference Methods were used during this emission testing program. These methods are taken from "Standards of Performance for New Stationary Sources", Appendix A, Federal Register, Volume 42, No. 160, Thursday, August 18, 1977, pp 41755 ff.

- o Method 1 - Sample and Velocity Traverses for Stationary Sources

This method specifies the number and location of sampling points within a duct, taking into account duct size and shape and local flow disturbances. In addition, this method discusses the pitot-nulling technique used to establish the degree of cyclonic flow in a duct.

- o Method 2 - Determination of Stack Gas Velocity and Volumetric Flowrate

This method specifies the measurement of gas velocity and flowrate using a pitot tube, manometer and temperature sensor. The physical dimensions of the pitot tube and its spatial relationship to the temperature sensor and any sample probe are also specified.

- o Method 4 - Determination of Moisture Content in Stack Gases

This method describes the extraction of a gas sample from a stack and the removal and measurement of the moisture in that sample by condensation impingers. The assembly and operation of the required sampling train is specified.

- o Method 5 - Determination of Particulate Emissions from Stationary Sources

This method specifies the isokinetic sampling of particulate matter from a gas stream utilizing techniques introduced in the above three methods. Sample collection and recovery, sampling train cleaning and calibration, and gas stream flowrate calculation procedures are specified.

5.2 Urea and Ammonia Sampling and Analysis

5.2.1 Sampling

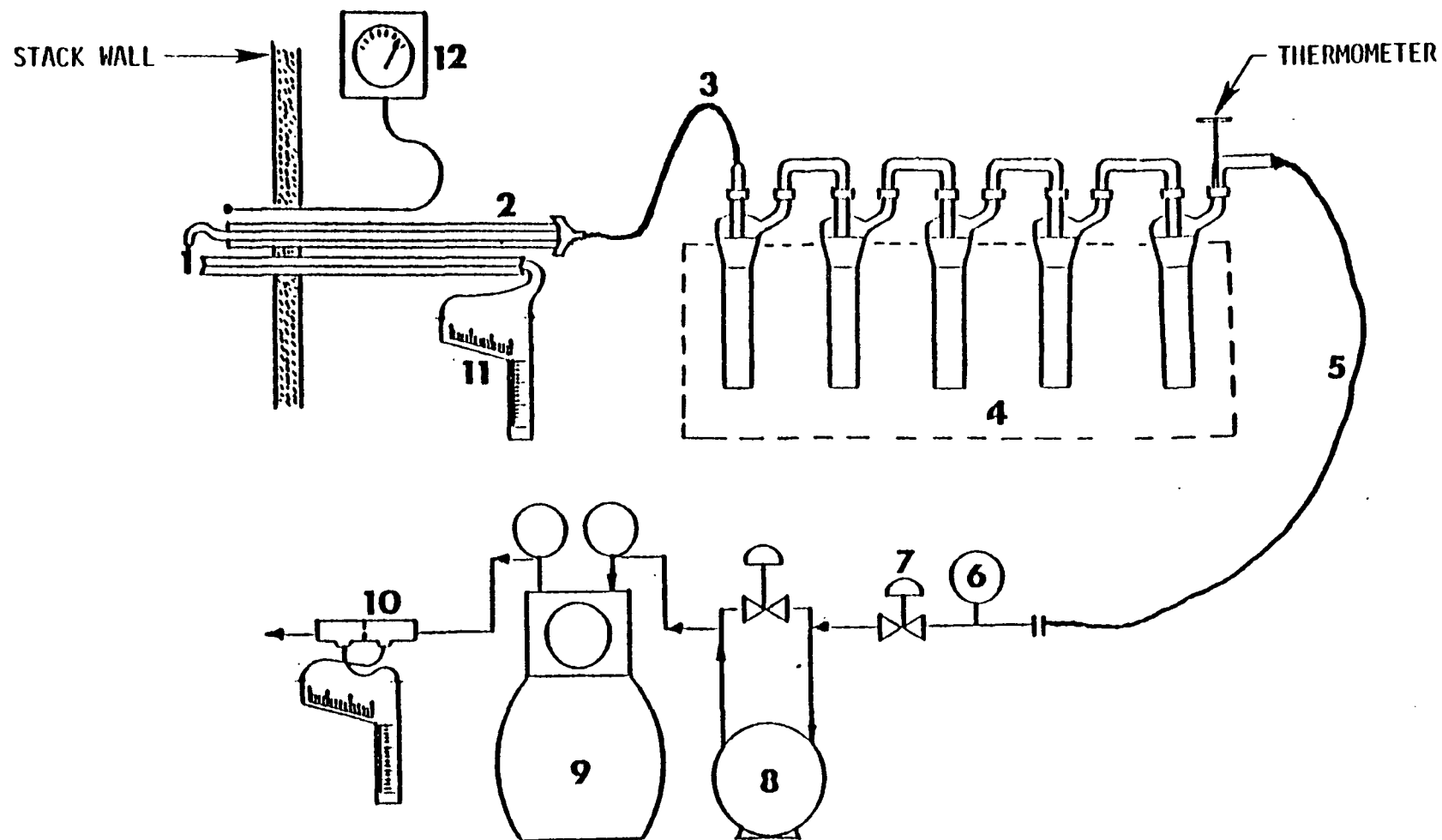
The outlet gas stream of the granulator C scrubber was sampled at points located in accordance with the relationship, detailed by EPA Method 1, of the sampling ports to upstream and downstream flow disturbances. The velocity of the duct gas was measured using S-type pitot tubes constructed and calibrated in accordance with EPA Method 2.

The sampling train used on this sampling program is shown in Figure 5-1 and is a modification of the standard EPA Method 5 particulate sampling train. The modifications used were: altered impinger sequence, absence of a filter, use of a teflon line and maintenance of the probe temperature at about 10°F above stack temperature.

The sampling train shown in Figure 5-1 consists of a nozzle, probe, teflon line, five impingers, vacuum pump, dry gas meter, and an orifice flow meter. The nozzle is stainless steel and of buttonhook shape. The nozzle was connected to a 5/8-inch stainless steel glass-lined probe. Following the probe, the gas stream passed through a 3/8" I.D. Teflon line into an ice bath/impinger system.

The first three impingers each contained 100 ml of deionized distilled water. The fourth impinger remained empty while the fifth was filled with 200 grams of indicating silica gel to remove any remaining moisture.

Leaving the last impinger, the sample gas stream flowed through flexible tubing, a vacuum gauge, needle valve, pump, and dry gas meter. A calibrated orifice and inclined manometer completed the sampling train. The stack velocity pressure was measured with an inclined manometer and an S-type pitot tube constructed, calibrated and used in accordance with EPA Reference Method 2. Stack temperature was monitored by a thermocouple attached to the probe and connected to a potentiometer. A nomograph was used to determine the



LEGEND

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

FIGURE 5-1: MODIFIED EPA PARTICULATE SAMPLING TRAIN
AUGUST 18, 1977, FEDERAL REGISTER

orifice pressure drop required for any measured pitot velocity pressure and stack temperature in order to maintain isokinetic sampling conditions.

Test data recorded included test time, sampling duration at each traverse point, pitot pressure, stack temperature, meter volume, meter inlet-outlet temperature, and orifice pressure drop.

5.2.2 Sample Recovery and Preparation

At the completion of each test run the train was leak-checked. The impinger sample volumes were measured and then the nozzle, probe, flexible teflon line, the first four impingers and their connecting glassware were rinsed with distilled deionized water. The impinger samples were combined with these washes and placed in a glass jar with a teflon-line cap.

At the Agrico laboratory, the silica gel from the fifth impinger was weighed to ± 0.1 g. The combined impinger sample was filtered through a pre-weighed glass-fiber filter. The filter was rinsed with distilled deionized water to prevent solids from drying out. The filtrate and filter rinses were then combined and the total volume was measured in a graduated cylinder.

Approximately 100 ml of this sample was set aside for formaldehyde analysis at TRC. Another portion was removed for immediate analysis (within 24 hours of collection) for urea and ammonia at the Agrico laboratory by both TRC and Agrico. The remaining sample was itself split into two portions; these latter portions were returned to TRC for additional urea analysis method investigations. These investigations are described in the EPA Report 79-NHF-13 "Development of Analytical Procedures for the Determination of Urea from Urea Manufacturing Facilities".

5.2.3 Sample Analysis

5.2.3.1 Analysis by TRC

A portion of each of the emission tests samples was analyzed for urea and ammonia by TRC at the Agrico laboratory within 24 hours of sample collection. The urea analysis was done with the Kjeldahl method (with preliminary distillation, ending with nesslerization); the ammonia analysis was done by direct nesslerization and by nesslerization with preliminary distillation.

The preliminary distillation was a step common to the Kjeldahl urea and distillation/nesslerization ammonia analyses. Sodium borate and sodium hydroxide were added to a portion of the sample to act as a buffer and to bring the pH to 9.5 or greater. The sample was then distilled, and the distillate (containing the ammonia) was collected in a boric acid solution. To this solution was added the nessler reagent, and after full color development the absorbance of this solution was measured with a spectrophotometer. To the distillation residue was added the Kjeldahl digestion reagent which converts organic nitrogen (urea) to ammonia. This (converted) ammonia was then distilled into an acid solution and analyzed by nesslerization as above.

Sample absorption measurements were converted to ammonia concentration through a calibration curve prepared with a series of standard ammonia solutions. Urea concentrations were calculated by multiplying the organic nitrogen ammonia concentrations by the stoichiometric factor 60/34.

Direct nesslerization ammonia measurements were made by adding the nessler reagent directly to a portion of the sample, awaiting full color development, and taking the absorbance reading with the spectrophotometer. A separate calibration curve was prepared for the direct nesslerization measurements.

One complication of the preliminary distillation step to remove ammonia is the hydrolysis of urea to ammonia that occurs during the distillation. It has

been estimated that about 7 percent of the urea in a sample is converted to ammonia during the preliminary distillation step.⁽¹⁾ Therefore, the indicated urea concentration multiplied by 1.07 equals the actual urea concentration. At the same time, the indicated ammonia concentration must be reduced by a stoichiometrically equivalent amount. Since 2 moles (34 grams) of ammonia are formed from the hydrolysis of 1 mole (60 grams) of urea, the ammonia correction equation is as follows:

$$A_a = A_i - (U_a * 0.07 * 34/60)$$

where A_a = actual ammonia concentration

A_i = indicated ammonia concentration

U_a = actual urea concentration

If the actual urea concentration is small relative to the ammonia concentration, then these corrections are insignificant. However, if urea concentrations are large (as, for example, in scrubber liquor streams) compared to ammonia concentrations, then the ammonia corrections are unrealistic, and result in negative actual ammonia concentrations (see Section 2.3 and Section 5.5).

Because urea was the species of concern in this emissions testing program, the impingers in the sampling train contained only water. In order to most efficiently capture ammonia, however, the gas stream should be bubbled through an acid solution; in a neutral or basic solution ammonia will tend to remain as a gas and will tend to leave the solution. For this reason, the ammonia

(1) Standard Methods of Water and Wastewater Analysis, APHA, AWWA, WPCF, 14th edition, 1975 p.408

collection efficiency of this sampling train may have been less than optimum, and the ammonia concentrations shown in Sections 2.1 and 2.2 may be less than the ammonia concentrations that actually existed in the scrubber outlet gas stream.

5.2.3.2 Analysis by Agrico

A portion of the same samples analyzed by TRC were analyzed for urea and ammonia by Agrico personnel at the Agrico laboratory within 24 hours of sample collection. The urea analyses were done with the indirect Kjeldahl method, ending with distillation and titration; the ammonia analyses were done by distillation and titration.

For these analyses two equal aliquots of sample were used. The first aliquot was buffered and distilled into a boric acid solution in the same manner as was done by TRC. Color indicator was then added to the distillate solution, and this solution was then titrated with standard 0.02N sulfuric acid until the proper indicator color was obtained. The sample ammonia nitrogen (N_a) concentration is calculated directly from the volume of standard acid used in this titration.

The second aliquot was digested with the Kjeldahl digestion reagent to convert all organic nitrogen to ammonia. This solution was then distilled into a boric acid solution, and this distillate solution was then titrated and the total nitrogen (N_t) concentration of the sample was calculated from the titrant volume, as described above.

The sample urea concentration was calculated by subtracting the ammonia nitrogen concentration from the total nitrogen concentration, and converting this difference (organic nitrogen) to urea stoichiometrically. The calculation procedure, including corrections for the conversion of urea to

ammonia during distillation, is as follows:

$$N_u = N_t - N_a = \text{mg urea nitrogen (uncorrected)}$$

$$N_u * 60/28 = \text{mg urea (uncorrected)}$$

$$U = (N_u * 60/28)(1-k) = \text{mg urea (corrected)}$$

$$\text{where } k = 0.07$$

$$\text{and } 60/28 = \text{stoichiometric factor.}$$

$$N_a * 17/14 = \text{mg ammonia (uncorrected)}$$

$$A = (N_a * 17/14) - (k * U/1.765) \text{ mg ammonia (corrected)}$$

$$\text{where } 1.765 = 60/34 = \text{stoichiometric factor.}$$

The factor k represents the standard 7 percent correction for urea to ammonia conversion during distillation.

As was noted in Section 2.2, the titrant volumes used by Agrico were relatively small (ranging from 5.8 ml to 13.5 ml for the total nitrogen analyses and from 5.4 ml to 11.5 ml for ammonia nitrogen). Larger titrant volumes (at least 20 ml) are recommended in order to minimize titration errors.

5.3 Formaldehyde Sampling and Analysis

The same samples collected, recovered and prepared as described in Sections 5.2.1 and 5.2.2 were analyzed for formaldehyde as well as urea and ammonia. The sample portions set aside for formaldehyde measurement were analyzed at TRC within 20 days of sample collection using the chromotropic acid method.

5.4 Insoluble Particulate Sampling and Analysis

The combined impinger samples (probe and glassware rinses and impinger contents) were filtered through a pre-weighed glass-filter at the Agrico

laboratory. The filters were returned to TRC in sealed petri dishes. They were then desiccated for at least 24 hours and then weighed to a constant weight. Constant weight is defined as two consecutive weighings, taken at least 6 hours apart, which agree within 0.5 mg. This analysis took place within 20 days of sample collection.

5.5 Scrubber Liquor Sampling and Analysis

5.5.1 Sampling, Sample Recovery and Preparation

During each of the six emissions test runs performed on the granulator C scrubber outlet, scrubber liquor inlet and outlet samples were collected in glass jars with teflon-lined caps. The jars were half-filled about 15 minutes into a test run, and then the remaining half was filled about 15 minutes before the end of the run.

Because of time constraints, only samples from test runs 1 and 4 were filtered (to remove all undissolved solids) and analyzed for urea and ammonia at the Agrico laboratory within 24 hours of sample collection. All samples were then returned to TRC, and the samples from test runs 2, 3, 5, and 6 were filtered and analyzed for urea and ammonia with 72 hours of sample collection.

A portion of each sample was set aside for formaldehyde analysis; these analyses were performed along with the formaldehyde analyses of the scrubber gas stream samples within 20 days of sample collection.

A change in the work assignment scope of work resulted in there being an insufficient supply of pre-weighed glass-fiber filters to filter all the samples as quickly as possible after sample collection. In some cases, therefore, inlet and outlet liquor samples were filtered through the same filter; filtrates were kept separate and these twice-used filters were rinsed thoroughly between sample filtrations. Samples 3, 5, and 6 were filtered in

this way. The exact volume of each filtered sample was not measured, so solids concentration calculations were based on the approximate volume of the sample jars (about 400 ml). For these reasons little confidence is placed in the measured insoluble particulate concentrations of the scrubber liquor samples.

5.5.2 Sample Analysis

The scrubber liquor samples were analyzed for urea, ammonia and formaldehyde in the same manner and with the same analysis methods as the scrubber outlet gas stream samples (Sections 5.2 and 5.3). Much larger dilutions were required for the liquor samples, however, because of the much greater ammonia and urea concentrations in the liquor than in the gas stream (see Appendix D for dilution factors). Consequently, errors or inaccuracies inherent in the analysis procedures may be magnified in the liquor sample analyses.

Because the urea concentrations in the outlet liquor samples are much greater than the ammonia concentrations, the corrected ammonia concentrations (corrected for conversion of urea to ammonia during the preliminary distillation step) for the outlet samples are negative. This result indicates that the 7 percent correction factor (as discussed in Section 5.2) is inappropriate for high concentration urea samples. The actual rate of hydrolysis of urea may be a function of the absolute urea concentration or of the relative urea to ammonia concentration. Further investigation of this problem, over a wide range of urea concentrations, is needed.

5.6 Urea Audit Samples - TRC and Agrico Analyses

Two sets of twelve urea audit samples, each set ranging from about 2 mg to about 600 mg, were weighed at TRC in tared vials on a 5-place analytical balance and then brought to Agrico for analysis during the December 1978 emissions test program. TRC and Agrico each analyzed one set of the sample sets. The TRC analyses were performed with the Kjeldahl method ending with nesslerization; the Agrico analyses were performed with the Kjeldahl method ending with titration. In both cases, no preliminary distillation was performed since the only source of nitrogen in the audit samples was urea.

The analyses were performed within 12 hours of dilution of the urea samples. In each set, the first six samples were diluted with 400 ml distilled, deionized water; the last six were diluted with 250 ml 1N sulfuric acid. This was done to simulate the water and acid impingers normally used in a urea particulate sampling train.

5.6.1 Analysis by TRC

The TRC audit sample set was prepared and analyzed at the Agrico laboratory during the December 1978 field program. Kjeldahl digestion reagent was added to an aliquot of each audit sample solution, converting all organic nitrogen to ammonia. The ammonia was then distilled into a boric acid solution, nessler reagent was added and the absorbance of the distillate solution was measured in a spectrophotometer. Absorbance was converted to ammonia concentration with a calibration curve prepared from the absorbances of standard ammonia solutions. A reagent blank was analyzed in the same manner as the audit samples.

The measured ammonia concentrations were converted to urea concentrations

as follows:

$$\text{urea (mg)} = \text{ammonia (mg)} * 60/34,$$

utilizing the stoichiometric relationship between moles of ammonia and moles of urea.

As noted in Section 2.4, the TRC analysis results agreed with the actual audit sample weights within 6 percent, on the average. The measured urea contents were all less than the actual contents, ranging from 3.9 percent to 9.3 percent lower. Eliminating the blank correction brought the average error to +5 percent, ranging from -5.9 percent to +22.3 percent. The blank correction is therefore considered appropriate. There is no noticeable difference between the analysis results of the first six samples (water diluted) and the last six (acid diluted). A breakdown of each sample analysis is shown in Appendix E.

5.6.2 Analysis by Agrico

The Agrico audit sample set was prepared and analyzed at the Agrico laboratory on January 4 and 5, 1979. The Agrico analyst diluted each sample to one liter with the appropriate diluent (water and acid). The Kjeldahl digestion and distillation was performed in the same way as the TRC analysis. Final total nitrogen content was determined by adding a color indicator to the distillate solution and titrating with standard acid. The indicated mg nitrogen were then converted to mg urea as follows:

$$\text{mg urea} = \text{mg nitrogen} * 60/28,$$

utilizing the stoichiometric relationship between moles of nitrogen and moles of urea.

The Agrico results averaged 92.9 percent higher than the actual audit sample urea content, ranging from 17.6 percent higher to 162 percent higher.

These results could reflect errors in several areas, including standardization of the titration acid and contamination during digestion and distillation. Only one blank was run, and this may not have been representative of the entire sample set analyzed over 2 days. Seven of the twelve analyses had titrant volumes less than 6 ml; usually a titration should utilize at least 20 ml in order to minimize the possibility of error. A variation in the indicated blank titrant volume (1.7 ml) would significantly effect the results of the low titrant volume samples.

APPENDIX A
COMPUTER PRINTOUT TEST RESULTS

Includes:

- A.1 Granulator C Scrubber Outlet
- A.2 Sample Equations and Example Calculations

APPENDIX A.1

GRANULATOR C SCRUBBER OUTLET

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 1
TRC PROJECT 82988-01

UNIT TESTED	UNIT C
DATE AND TIME OF TEST	DEC 18 1978 1500 TO 1607
SAMPLING LOCATION	SCRUBBER OUTLET
NAME OF FIRM	AGRICO -EPA
LOCATION OF FIRM	BLYTHEVILLE ARK
POLLUTANTS SAMPLED	UREA AND AMMONIA

BAROMETRIC PRESSURE. IN HG	29.84
DUCT AREA. SQ FT	18.98
NOZZLE DIAMETER. IN	0.185
PITOT CALIBRATION COEFFICIENTS 1	0.839
2	0.000
3	0.000
DRY GAS METER CALIBRATION FACTOR. Y	0.990
FINAL LEAK RATE. CFM	0.014
TONS PER HOUR, PRODUCT	0.000

COMPOSITION OF DUCT GAS. % BY VOLUME DRY BASIS

CARBON DIOXIDE	0.00
OXYGEN	21.00
CARBON MONOXIDE	0.00
NITROGEN	79.00

UREA, AMMONIA, FORMALDEHYDE COLLECTED. MG

	UREA	AMMONIA-DIR	AMMONIA-DIST	FORMALDEHYDE
H2O IMPINGERS	0.6040E 02	0.4037E 03	0.4232E 03	0.3900E 01
H2SO4 IMPINGERS	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL	0.6040E 02	0.4037E 03	0.4232E 03	0.3900E 01

AMOUNT OF WATER COLLECTED. GRAMS

IMPINGERS	35.0
SILICA GEL	12.0

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 1
TRC PROJECT 82988-01

INT	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 CYC R ANG B
1	5.0	0.9100	1.0500	53.	55.	-0.40	94.	154.23	1 0.
2	5.0	0.9400	1.0800	53.	53.	-0.40	92.	157.00	1 0.
3	5.0	0.9000	1.0200	53.	53.	-0.40	91.	159.92	1 0.
4	5.0	0.9000	1.0200	52.	52.	-0.40	89.	162.20	1 0.
5	5.0	0.8400	0.9700	52.	51.	-0.40	86.	165.56	1 0.
6	5.0	0.6300	0.8500	51.	51.	-0.40	86.	168.52	1 0.
1	5.0	0.9600	1.1000	54.	53.	-0.48	91.	171.18	1 0.
2	5.0	0.9900	1.1500	54.	53.	-0.48	94.	174.12	1 0.
3	5.1	0.9700	1.1400	54.	54.	-0.48	95.	177.10	1 0.
4	5.0	0.8600	1.0100	54.	54.	-0.48	95.	180.20	1 0.
5	5.0	0.8300	0.9600	53.	53.	-0.48	94.	182.97	1 0.
6	5.0	0.6900	0.8100	54.	54.	-0.48	94.	185.76	1 0.

FINAL METER VOLUME

188.52

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 1
TRC PROJECT 82988-01

UNIT C AGRICO -EPA
SCRUBBER OUTLET
DEC 18 1978 1500 TO 1607

STANDARD CONDITION TEMPERATURE, DEG F	0.6800E	02
STANDARD CONDITION PRESSURE, IN HG	0.2992E	02
TOTAL SAMPLING TIME, MINUTES	0.6010E	02
AVERAGE SQUARE ROOT VELOCITY HEAD, IN H2O EXP .5	0.9300E	00
AVERAGE ORIFICE PRESSURE DROP, IN H2O	0.1013E	01
AVERAGE METER TEMPERATURE, DEG F	0.5304E	02
AVERAGE DUCT STATIC PRESSURE, IN H2O	-0.4400E	00
AVERAGE DUCT TEMPERATURE, DEG F	0.9175E	02
TOTAL SAMPLE VOLUME, DACF	0.3429E	02
TOTAL SAMPLE VOLUME, DSCF	0.3493E	02
WATER VAPOR VOLUME, DSCF	0.2212E	01
MOISTURE CONTENT OF DUCT GAS, PERCENT	0.5956E	01
MOLE FRACTION DRY GAS	0.9404E	00
MOLECULAR WEIGHT - DRY STACK GAS	0.2884E	02
MOLECULAR WEIGHT - STACK GAS	0.2819E	02
AVERAGE STACK PRESSURE, IN HG	0.2981E	02
DUCT VOLUMETRIC FLOW, ACFM	0.6155E	05
DUCT VOLUMETRIC FLOW, DSCFM	0.5518E	05
AVERAGE DUCT VELOCITY, FPM	0.3243E	04
EXCESS AIR, PERCENT	-0.1458E	05
AVERAGE DUCT GAS DENSITY, LBS/ACF	0.6976E	-01
ISOKINETIC FACTOR, PERCENT	0.1072E	03

EMISSION DATA

	GR/ ACF	GR/ DSCF	LBS/ HR	LBS TON
UREA IN H2O	0.2392E-01	0.2668E-01	0.1262E 02	0.1262
UREA IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL UREA	0.2392E-01	0.2668E-01	0.1262E 02	0.1262
AMMONIA-DIRECT-IN H2O	0.1599E 00	0.1783E 00	0.8435E 02	0.8435
AMMONIA-DIRECT-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL AMMONIA-DIRECT	0.1599E 00	0.1783E 00	0.8435E 02	0.8435
AMMONIA-DISTILLED-IN H2O	0.1676E 00	0.1869E 00	0.8842E 02	0.8842
AMMONIA-DISTILLED-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL AMMONIA-DISTILLED	0.1676E 00	0.1869E 00	0.8842E 02	0.8842
FORMALDEHYDE IN H2O	0.1545E-02	0.1723E-02	0.8148E 00	0.8148
FORMALDEHYDE IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL FORMALDEHYDE	0.1545E-02	0.1723E-02	0.8148E 00	0.8148

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 1
TRC PROJECT 82988-01

UNIT C AGRICO -EPA
SCRUBBER OUTLET
DEC 18 1978 1500 TO 1607

STANDARD CONDITION TEMPERATURE, DEG C	0.2000E 02
STANDARD CONDITION PRESSURE, MM HG	0.7600E 03
TOTAL SAMPLING TIME, MINUTES	0.6010E 02
AVERAGE SQUARE ROOT VELOCITY HEAD, MM H2O EXP.5	0.4687E 01
AVERAGE ORIFICE PRESSURE DROP, MM H2O	0.2574E 02
AVERAGE METER TEMPERATURE, DEG C	0.1169E 02
AVERAGE DUCT STATIC PRESSURE, MM H2O	-0.1118E 02
AVERAGE DUCT TEMPERATURE, DEG C	0.3319E 02
TOTAL SAMPLE VOLUME, DM3	0.9711E 00
TOTAL SAMPLE VOLUME, DNM3	0.9892E 00
WATER VAPOR VOLUME, DNM3	0.6265E-01
AVERAGE STACK GAS PRESSURE, MM HG	0.7571E 03
DUCT VOLUMETRIC FLOW, AM3/M	0.1743E 04
DUCT VOLUMETRIC FLOW, DNM3/M	0.1563E 04
AVERAGE DUCT VELOCITY, M/M	0.9884E 03
AVERAGE DUCT GAS DENSITY, KG/AM3	0.1117E 01

EMISSION DATA

	MG/ AM3	MG/ DNM3	KG/ HR	KG MT
UREA IN H2O	0.5474E 02	0.6106E 02	0.5729E 01	0.252
UREA IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL UREA	0.5474E 02	0.6106E 02	0.5729E 01	0.252
AMMONIA-DIRECT-IN H2O	0.3659E 03	0.4081E 03	0.3829E 02	0.1687
AMMONIA-DIRECT-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL AMMONIA-DIRECT	0.3659E 03	0.4081E 03	0.3829E 02	0.1687
AMMONIA-DISTILLED-IN H2O	0.3836E 03	0.4278E 03	0.4014E 02	0.1768
AMMONIA-DISTILLED-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL AMMONIA-DISTILLED	0.3836E 03	0.4278E 03	0.4014E 02	0.1768
FORMALDEHYDE IN H2O	0.3535E 01	0.3942E 01	0.3699E 00	0.1630
FORMALDEHYDE IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL FORMALDEHYDE	0.3535E 01	0.3942E 01	0.3699E 00	0.1630

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 2
TRC PROJECT 82988-01

UNIT TESTED UNIT C
DATE AND TIME OF TEST DEC 19 1978 0905 TO 1011
SAMPLING LOCATION SCRUBBER OUTLET
NAME OF FIRM AGRICO -EPA
LOCATION OF FIRM BLYTHEVILLE ARK
POLLUTANTS SAMPLED UREA AND AMMONIA

BAROMETRIC PRESSURE. IN HG 29.71
DUCT AREA. SQ FT 18.98
NOZZLE DIAMETER. IN 0.185
PITOT CALIBRATION COEFFICIENTS 1 0.839
2 0.000
3 0.000
DRY GAS METER CALIBRATION FACTOR. Y 0.990
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 21.00
CARBON MONOXIDE 0.00
NITROGEN 79.00

UREA, AMMONIA, FORMALDEHYDE COLLECTED. MG

	UREA	AMMONIA-DIR	AMMONIA-DIST	FORMALDEHYDE
H2O IMPINGERS	0.9000E 02	0.3330E 03	0.3290E 03	0.4700E 01
H2SO4 IMPINGERS	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL	0.9000E 02	0.3330E 03	0.3290E 03	0.4700E 01

AMOUNT OF WATER COLLECTED. GRAMS
IMPINGERS 23.0
SILICA GEL 5.6

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 2
TRC PROJECT 82988-01

IT	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 B	CYC AVG
1	5.0	0.9700	1.1900	58.	59.	-0.41	98.	193.43	1	0.
2	5.0	0.9500	1.1500	58.	59.	-0.41	102.	196.30	1	0.
3	5.0	0.9000	1.0900	60.	60.	-0.41	103.	199.31	1	0.
4	5.0	0.8400	1.0000	61.	59.	-0.41	102.	202.26	1	0.
5	5.0	0.7500	0.9100	60.	59.	-0.41	101.	205.11	1	0.
6	5.0	0.6300	0.7600	60.	60.	-0.41	100.	207.86	1	0.
7	5.0	0.9300	1.1100	60.	59.	-0.38	101.	210.51	1	0.
8	5.0	0.9500	1.1500	61.	60.	-0.38	103.	213.39	1	0.
9	5.0	0.9300	1.1100	62.	61.	-0.38	104.	216.44	1	0.
10	5.0	0.8100	0.9800	61.	61.	-0.38	102.	219.47	1	0.
11	5.0	0.7400	0.9000	62.	62.	-0.38	102.	222.36	1	0.
12	5.0	0.6900	0.8200	60.	61.	-0.38	101.	225.10	1	0.

AL METER VOLUME

227.85

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 2
TRC PROJECT 82988-01

UNIT C AGRICO -EPA
SCRUBBER OUTLET
DEC 19 1978 0905 TO 1011

STANDARD CONDITION TEMPERATURE, DEG F	0.6800E 02
STANDARD CONDITION PRESSURE, IN HG	0.2992E 02
TOTAL SAMPLING TIME, MINUTES	0.6000E 02
AVERAGE SQUARE ROOT VELOCITY HEAD, IN H2O EXP .5	0.9149E 00
AVERAGE ORIFICE PRESSURE DROP, IN H2O	0.1014E 01
AVERAGE METER TEMPERATURE, DEG F	0.6012E 02
AVERAGE DUCT STATIC PRESSURE, IN H2O	-0.3950E 00
AVERAGE DUCT TEMPERATURE, DEG F	0.1016E 03
TOTAL SAMPLE VOLUME, DACF	0.3442E 02
TOTAL SAMPLE VOLUME, DSCF	0.3444E 02
WATER VAPOR VOLUME, DSCF	0.1346E 01
MOISTURE CONTENT OF DUCT GAS, PERCENT	0.3762E 01
MOLE FRACTION DRY GAS	0.9624E 00
MOLECULAR WEIGHT - DRY STACK GAS	0.2884E 02
MOLECULAR WEIGHT - STACK GAS	0.2843E 02
AVERAGE STACK PRESSURE, IN HG	0.2968E 02
DUCT VOLUMETRIC FLOW, ACFM	0.6096E 05
DUCT VOLUMETRIC FLOW, DSCFM	0.5472E 05
AVERAGE DUCT VELOCITY, FPM	0.3212E 04
EXCESS AIR, PERCENT	-0.1458E 05
AVERAGE DUCT GAS DENSITY, LBS/ACF	0.6882E-01
ISOKINETIC FACTOR, PERCENT	0.1067E 03

EMISSION DATA

	GR/ ACF	GR/ DSCF	LBS/ HR	LBS/ TON
UREA IN H2O	0.3620E-01	0.4033E-01	0.1891E 02	0.1891E 02
UREA IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL UREA	0.3620E-01	0.4033E-01	0.1891E 02	0.1891E 02
AMMONIA-DIRECT-IN H2O	0.1339E 00	0.1492E 00	0.6998E 02	0.6998E 02
AMMONIA-DIRECT-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL AMMONIA-DIRECT	0.1339E 00	0.1492E 00	0.6998E 02	0.6998E 02
AMMONIA-DISTILLED-IN H2O	0.1323E 00	0.1474E 00	0.6914E 02	0.6914E 02
AMMONIA-DISTILLED-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL AMMONIA-DISTILLED	0.1323E 00	0.1474E 00	0.6914E 02	0.6914E 02
FORMALDEHYDE IN H2O	0.1890E-02	0.2106E-02	0.9877E 00	0.9877E 00
FORMALDEHYDE IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL FORMALDEHYDE	0.1890E-02	0.2106E-02	0.9877E 00	0.9877E 00

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 2
TRC PROJECT 82988-01

UNIT C AGRICO -EPA
SCRUBBER OUTLET
DEC 19 1978 0905 TO 1011

STANDARD CONDITION TEMPERATURE. DEG C	0.2000E 02
STANDARD CONDITION PRESSURE. MM HG	0.7600E 03
TOTAL SAMPLING TIME, MINUTES	0.6000E 02
AVERAGE SQUARE ROOT VELOCITY HEAD. MM H2O EXP.5	0.4611E 01
AVERAGE ORIFICE PRESSURE DROP. MM H2O	0.2576E 02
AVERAGE METER TEMPERATURE. DEG C	0.1562E 02
AVERAGE DUCT STATIC PRESSURE. MM H2O	-0.1003E 02
AVERAGE DUCT TEMPERATURE. DEG C	0.3866E 02
TOTAL SAMPLE VOLUME. DM3	0.9748E 00
TOTAL SAMPLE VOLUME. DNM3	0.9752E 00
WATER VAPOR VOLUME. DNM3	0.3812E-01
AVERAGE STACK GAS PRESSURE. MM HG	0.7539E 03
DUCT VOLUMETRIC FLOW. AM3/M	0.1726E 04
DUCT VOLUMETRIC FLOW. DNM3/M	0.1550E 04
AVERAGE DUCT VELOCITY. M/M	0.9790E 03
AVERAGE DUCT GAS DENSITY. KG/AM3	0.1102E 01

EMISSION DATA

	MG/ AM3	MG/ DNM3	KG/ HR	KG/ MT
UREA IN H2O	0.8284E 02	0.9229E 02	0.8587E 01	0.3783E 02
UREA IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL UREA	0.8284E 02	0.9229E 02	0.8587E 01	0.3783E 02
AMMONIA-DIRECT-IN H2O	0.3065E 03	0.3415E 03	0.3177E 02	0.1400E 03
AMMONIA-DIRECT-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL AMMONIA-DIRECT	0.3065E 03	0.3415E 03	0.3177E 02	0.1400E 03
AMMONIA-DISTILLED-IN H2O	0.3028E 03	0.3374E 03	0.3139E 02	0.1383E 03
AMMONIA-DISTILLED-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL AMMONIA-DISTILLED	0.3028E 03	0.3374E 03	0.3139E 02	0.1383E 03
FORMALDEHYDE IN H2O	0.4326E 01	0.4819E 01	0.4484E 00	0.1975E 01
FORMALDEHYDE IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL FORMALDEHYDE	0.4326E 01	0.4819E 01	0.4484E 00	0.1975E 01

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 3
TRC PROJECT 82988-01

UNIT TESTED	UNIT C
DATE AND TIME OF TEST	DEC 19 1978 1100 TO 1205
SAMPLING LOCATION	SCRUBBER OUTLET
NAME OF FIRM	AGRICO -EPA
LOCATION OF FIRM	BLYTHEVILLE ARK
POLLUTANTS SAMPLED	UREA AND AMMONIA

BAROMETRIC PRESSURE. IN HG	29.68
DUCT AREA. SQ FT	18.98
NOZZLE DIAMETER. IN	0.185
PITOT CALIBRATION COEFFICIENTS 1	0.839
2	0.000
3	0.000
DRY GAS METER CALIBRATION FACTOR. Y	0.990
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	21.00
CARBON MONOXIDE	0.00
NITROGEN	79.00

UREA, AMMONIA, FORMALDEHYDE COLLECTED. MG

	UREA	AMMONIA-DIR	AMMONIA-DIST	FORMALDEHYDE
H2O IMPINGERS	0.3360E 02	0.3700E 03	0.5420E 03	0.3300E 01
H2SO4 IMPINGERS	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL	0.3360E 02	0.3700E 03	0.5420E 03	0.3300E 01

AMOUNT OF WATER COLLECTED. GRAMS	
IMPINGERS	32.0
SILICA GEL	5.6

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 3
TRC PROJECT 82988-01

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 AVG
1	5.0	0.9400	1.1300	64.	64.	-0.43	102.	227.94	1	0
2	5.0	0.9500	1.1500	65.	64.	-0.43	105.	230.80	1	0
3	5.0	0.8700	1.0600	65.	65.	-0.43	105.	233.80	1	0.
4	5.0	0.6800	0.8300	64.	65.	-0.43	105.	236.69	1	0
5	5.0	0.6800	0.8300	61.	63.	-0.43	102.	239.35	1	0
6	5.0	0.6200	0.7600	61.	64.	-0.43	102.	241.96	1	0.
1	5.0	0.7500	0.9100	65.	65.	-0.36	103.	244.62	1	0
2	5.0	0.8000	0.9600	65.	65.	-0.36	104.	247.23	1	0.
3	5.0	0.7600	0.9300	65.	65.	-0.36	106.	250.10	1	0.
4	5.0	0.7500	0.9100	65.	65.	-0.36	106.	252.78	1	0
5	5.0	0.6800	0.8300	65.	65.	-0.36	107.	255.55	1	0.
6	5.0	0.6000	0.7300	65.	65.	-0.36	105.	258.15	1	0.

FINAL METER VOLUME

260.85

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 3
TRC PROJECT 82988-01

UNIT C AGRICO -EPA
SCRUBBER OUTLET
DEC 19 1978 1100 TO 1205

STANDARD CONDITION TEMPERATURE, DEG F	0.6800E 02
STANDARD CONDITION PRESSURE, IN HG	0.2992E 02
TOTAL SAMPLING TIME, MINUTES	0.6000E 02
AVERAGE SQUARE ROOT VELOCITY HEAD, IN H2O EXP .5	0.8676E 00
AVERAGE ORIFICE PRESSURE DROP, IN H2O	0.9192E 00
AVERAGE METER TEMPERATURE, DEG F	0.6437E 02
AVERAGE DUCT STATIC PRESSURE, IN H2O	-0.3950E 00
AVERAGE DUCT TEMPERATURE, DEG F	0.1043E 03
TOTAL SAMPLE VOLUME, DACF	0.3291E 02
TOTAL SAMPLE VOLUME, DSCF	0.3262E 02
WATER VAPOR VOLUME, DSCF	0.1770E 01
MOISTURE CONTENT OF DUCT GAS, PERCENT	0.5147E 01
MOLE FRACTION DRY GAS	0.9485E 00
MOLECULAR WEIGHT - DRY STACK GAS	0.2884E 02
MOLECULAR WEIGHT - STACK GAS	0.2828E 02
AVERAGE STACK PRESSURE, IN HG	0.2965E 02
DUCT VOLUMETRIC FLOW, ACFM	0.5813E 05
DUCT VOLUMETRIC FLOW, DSCFM	0.5113E 05
AVERAGE DUCT VELOCITY, FPM	0.3063E 04
EXCESS AIR, PERCENT	-0.1458E 05
AVERAGE DUCT GAS DENSITY, LBS/ACF	0.6806E-01
ISOKINETIC FACTOR, PERCENT	0.1082E 03

EMISSION DATA

	GR/ ACF	GR/ DSCF	LBS/ HR	LB TO
UREA IN H2O	0.1398E-01	0.1590E-01	0.6965E 01	0.696
UREA IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL UREA	0.1398E-01	0.1590E-01	0.6965E 01	0.696
AMMONIA-DIRECT-IN H2O	0.1539E 00	0.1750E 00	0.7670E 02	0.767
AMMONIA-DIRECT-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL AMMONIA-DIRECT	0.1539E 00	0.1750E 00	0.7670E 02	0.767
AMMONIA-DISTILLED-IN H2O	0.2255E 00	0.2564E 00	0.1124E 03	0.112
AMMONIA-DISTILLED-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL AMMONIA-DISTILLED	0.2255E 00	0.2564E 00	0.1124E 03	0.112
FORMALDEHYDE IN H2O	0.1373E-02	0.1561E-02	0.6841E 00	0.684
FORMALDEHYDE IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL FORMALDEHYDE	0.1373E-02	0.1561E-02	0.6841E 00	0.684

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 3
TRC PROJECT 82988-01

UNIT C AGRICO -EPA
SCRUBBER OUTLET
DEC 19 1978 1100 TO 1205

STANDARD CONDITION TEMPERATURE, DEG C	0.2000E 02
STANDARD CONDITION PRESSURE, MM HG	0.7600E 03
TOTAL SAMPLING TIME, MINUTES	0.6000E 02
AVERAGE SQUARE ROOT VELOCITY HEAD, MM H2O EXP.5	0.4373E 01
AVERAGE ORIFICE PRESSURE DROP, MM H2O	0.2335E 02
AVERAGE METER TEMPERATURE, DEG C	0.1799E 02
AVERAGE DUCT STATIC PRESSURE, MM H2O	-0.1003E 02
AVERAGE DUCT TEMPERATURE, DEG C	0.4019E 02
TOTAL SAMPLE VOLUME, DM3	0.9320E 00
TOTAL SAMPLE VOLUME, DNM3	0.9237E 00
WATER VAPOR VOLUME, DNM3	0.5012E-01
AVERAGE STACK GAS PRESSURE, MM HG	0.7531E 03
DUCT VOLUMETRIC FLOW, AM3/M	0.1646E 04
DUCT VOLUMETRIC FLOW, DNM3/M	0.1448E 04
AVERAGE DUCT VELOCITY, M/M	0.9336E 03
VVERAGE DUCT GAS DENSITY, KG/AM3	0.1090E 01

EMISSION DATA

	MG/ AM3	MG/ DNM3	KG/ HR	K
UREA IN H2O	0.3199E 02	0.3637E 02	0.3162E 01	0.157
UREA IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL UREA	0.3199E 02	0.3637E 02	0.3162E 01	0.157
AMMONIA-DIRECT-IN H2O	0.3523E 03	0.4005E 03	0.3482E 02	0.153
AMMONIA-DIRECT-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL AMMONIA-DIRECT	0.3523E 03	0.4005E 03	0.3482E 02	0.153
AMMONIA-DISTILLED-IN H2O	0.5160E 03	0.5868E 03	0.5101E 02	0.224
AMMONIA-DISTILLED-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL AMMONIA-DISTILLED	0.5160E 03	0.5868E 03	0.5101E 02	0.224
FORMALDEHYDE IN H2O	0.3142E 01	0.3572E 01	0.3106E 00	0.174
FORMALDEHYDE IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL FORMALDEHYDE	0.3142E 01	0.3572E 01	0.3106E 00	0.174

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO
TRC PROJECT 82988-01

4

UNIT TESTED	UNIT C
DATE AND TIME OF TEST	DEC 19 1978 1300 TO 1412
SAMPLING LOCATION	SCRUBBER OUTLET
NAME OF FIRM	AGRICO -EPA
LOCATION OF FIRM	BLYTHEVILLE ARK
POLLUTANTS SAMPLED	UREA AND AMMONIA

BAROMETRIC PRESSURE. IN HG	29.68
DUCT AREA. SQ FT	18.98
NOZZLE DIAMETER. IN	0.185
PITOT CALIBRATION COEFFICIENTS 1	0.839
2	0.000
3	0.000
DRY GAS METER CALIBRATION FACTOR. Y	0.990
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	21.00
CARBON MONOXIDE	0.00
NITROGEN	79.00

UREA, AMMONIA, FORMALDEHYDE COLLECTED. MG

	UREA	AMMONIA-DIR	AMMONIA-DIST	FORMALDEHYDE
H2O IMPINGERS	0.4810E 02	0.3630E 03	0.3490E 03	0.4240E 01
H2SO4 IMPINGERS	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL	0.4810E 02	0.3630E 03	0.3490E 03	0.4240E 01

AMOUNT OF WATER COLLECTED. GRAMS

IMPINGERS	30.0
SILICA GEL	5.9

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO
TRC PROJECT 82988-01

4

JOINT	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 AVG
1	5.0	0.6100	0.7300	68.	68.	-0.42	103.	261.00	1	0.
2	5.0	0.7800	0.9200	68.	68.	-0.42	106.	263.35	1	0.
3	5.0	0.9000	1.0500	67.	68.	-0.42	106.	266.07	1	0.
4	5.0	0.7600	0.9100	65.	67.	-0.42	101.	268.95	1	0.
5	5.0	0.7300	0.8700	64.	66.	-0.42	99.	271.74	1	0.
6	5.0	0.6200	0.7400	65.	66.	-0.42	100.	274.43	1	0.
1	5.0	0.9300	1.0900	67.	68.	-0.48	103.	277.06	1	0.
2	5.0	0.9800	1.1500	67.	68.	-0.48	104.	279.94	1	0.
3	5.0	0.9600	1.1400	67.	68.	-0.48	104.	283.01	1	0.
4	5.0	0.8500	1.0000	68.	68.	-0.48	105.	286.08	1	0.
5	5.0	0.8000	0.9400	67.	67.	-0.48	105.	289.00	1	0.
6	5.0	0.7400	0.8800	67.	67.	-0.48	103.	291.78	1	0.

FINAL METER VOLUME

294.61

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO
TRC PROJECT 82988-01

4

UNIT C AGRICO -EPA
SCRUBBER OUTLET
DEC 19 1978 1300 TO 1412

STANDARD CONDITION TEMPERATURE, DEG F	0.6800E 02
STANDARD CONDITION PRESSURE, IN HG	0.2992E 02
TOTAL SAMPLING TIME, MINUTES	0.6000E 02
AVERAGE SQUARE ROOT VELOCITY HEAD, IN H2O EXP .5	0.8948E 00
AVERAGE ORIFICE PRESSURE DROP, IN H2O	0.9517E 00
AVERAGE METER TEMPERATURE, DEG F	0.6704E 02
AVERAGE DUCT STATIC PRESSURE, IN H2O	-0.4500E 00
AVERAGE DUCT TEMPERATURE, DEG F	0.1032E 03
TOTAL SAMPLE VOLUME, DACF	0.3361E 02
TOTAL SAMPLE VOLUME, DSCF	0.3314E 02
WATER VAPOR VOLUME, DSCF	0.1690E 01
MOISTURE CONTENT OF DUCT GAS, PERCENT	0.4851E 01
MOLE FRACTION DRY GAS	0.9515E 00
MOLECULAR WEIGHT - DRY STACK GAS	0.2884E 02
MOLECULAR WEIGHT - STACK GAS	0.2831E 02
AVERAGE STACK PRESSURE, IN HG	0.2965E 02
DUCT VOLUMETRIC FLOW, ACFM	0.5987E 05
DUCT VOLUMETRIC FLOW, DSCFM	0.5291E 05
AVERAGE DUCT VELOCITY, FPM	0.3154E 04
EXCESS AIR, PERCENT	-0.1458E 05
AVERAGE DUCT GAS DENSITY, LBS/ACF	0.6826E-01
ISOKINETIC FACTOR, PERCENT	0.1062E 03

EMISSION DATA

	GR/ ACF	GR/ DSCF	LBS/ HR	LB TO
UREA IN H2O	0.1979E-01	0.2239E-01	0.1015E 02	0.101
UREA IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL UREA	0.1979E-01	0.2239E-01	0.1015E 02	0.101
AMMONIA-DIRECT-IN H2O	0.1494E 00	0.1690E 00	0.7664E 02	0.766
AMMONIA-DIRECT-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL AMMONIA-DIRECT	0.1494E 00	0.1690E 00	0.7664E 02	0.766
AMMONIA-DISTILLED-IN H2O	0.1436E 00	0.1625E 00	0.7368E 02	0.736
AMMONIA-DISTILLED-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL AMMONIA-DISTILLED	0.1436E 00	0.1625E 00	0.7368E 02	0.736
FORMALDEHYDE IN H2O	0.1745E-02	0.1974E-02	0.8951E 00	0.895
FORMALDEHYDE IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL FORMALDEHYDE	0.1745E-02	0.1974E-02	0.8951E 00	0.895

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO
TRC PROJECT 82988-01

4

UNIT C

AGRICO -EPA
SCRUBBER OUTLET
DEC 19 1978 1300 TO 1412

STANDARD CONDITION TEMPERATURE. DEG C	0.2000E 02
STANDARD CONDITION PRESSURE. MM HG	0.7600E 03
TOTAL SAMPLING TIME, MINUTES	0.6000E 02
AVERAGE SQUARE ROOT VELOCITY HEAD. MM H2O EXP.5	0.4510E 01
AVERAGE ORIFICE PRESSURE DROP. MM H2O	0.2417E 02
AVERAGE METER TEMPERATURE. DEG C	0.1947E 02
AVERAGE DUCT STATIC PRESSURE. MM H2O	-0.1143E 02
AVERAGE DUCT TEMPERATURE. DEG C	0.3958E 02
TOTAL SAMPLE VOLUME. DM3	0.9518E 00
TOTAL SAMPLE VOLUME. DNM3	0.9387E 00
WATER VAPOR VOLUME. DNM3	0.4786E-01
AVERAGE STACK GAS PRESSURE. MM HG	0.7530E 03
DUCT VOLUMETRIC FLOW. AM3/M	0.1695E 04
DUCT VOLUMETRIC FLOW. DNM3/M	0.1498E 04
AVERAGE DUCT VELOCITY. M/M	0.9614E 03
AVERAGE DUCT GAS DENSITY. KG/AM3	0.1093E 01

EMISSION DATA

	MG/ AM3	MG/ DNM3	KG/ HR	K M
UREA IN H2O	0.4529E 02	0.5124E 02	0.4610E 01	0.205
UREA IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL UREA	0.4529E 02	0.5124E 02	0.4610E 01	0.205
AMMONIA-DIRECT-IN H2O	0.3418E 03	0.3867E 03	0.3479E 02	0.153
AMMONIA-DIRECT-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL AMMONIA-DIRECT	0.3418E 03	0.3867E 03	0.3479E 02	0.153
AMMONIA-DISTILLED-IN H2O	0.3286E 03	0.3718E 03	0.3345E 02	0.147
AMMONIA-DISTILLED-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL AMMONIA-DISTILLED	0.3286E 03	0.3718E 03	0.3345E 02	0.147
FORMALDEHYDE IN H2O	0.3992E 01	0.4517E 01	0.4064E 00	0.179
FORMALDEHYDE IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.000
TOTAL FORMALDEHYDE	0.3992E 01	0.4517E 01	0.4064E 00	0.179

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 5
TRC PROJECT 82988-01

UNIT TESTED	UNIT C
DATE AND TIME OF TEST	DEC 19 1978 1450 TO 1553
SAMPLING LOCATION	SCRUBBER OUTLET
NAME OF FIRM	AGRICO -EPA
LOCATION OF FIRM	BLYTHERVILLE ARK
POLLUTANTS SAMPLED	UREA AND AMMONIA

BAROMETRIC PRESSURE. IN HG	29.63
DUCT AREA. SQ FT	18.98
NOZZLE DIAMETER. IN	0.185
PITOT CALIBRATION COEFFICIENTS 1	0.839
2	0.000
3	0.000
DRY GAS METER CALIBRATION FACTOR. Y	0.990
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	21.00
CARBON MONOXIDE	0.00
NITROGEN	79.00

UREA, AMMONIA, FORMALDEHYDE COLLECTED. MG

	UREA	AMMONIA-DIR	AMMONIA-DIST	FORMALDEHYDE
H2O IMPINGERS	0.2840E 02	0.3420E 03	0.3230E 03	0.2050E 01
H2SO4 IMPINGERS	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL	0.2840E 02	0.3420E 03	0.3230E 03	0.2050E 01

AMOUNT OF WATER COLLECTED. GRAMS	
IMPINGERS	18.0
SILICA GEL	3.9

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 5
TRC PROJECT 82988-01

JINT	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 B	CYC ANG
1	5.0	0.9400	1.1100	67.	67.	-0.39	105.	294.70	1	0.
2	5.0	0.9200	1.0900	67.	67.	-0.39	105.	297.58	1	0.
3	5.0	0.8500	1.0000	67.	67.	-0.39	105.	300.60	1	0.
4	5.0	0.6200	0.7400	67.	67.	-0.39	104.	303.50	1	0.
5	5.0	0.5900	0.7000	67.	67.	-0.39	105.	306.10	1	0.
6	5.0	0.5000	0.6000	67.	67.	-0.39	103.	308.55	1	0.
1	5.0	0.7000	0.8300	68.	69.	-0.42	105.	310.96	1	0.
2	5.0	0.7800	0.9200	69.	69.	-0.42	105.	313.49	1	0.
3	5.0	0.9100	1.0800	69.	69.	-0.42	105.	316.25	1	0.
4	5.0	0.8200	0.9700	69.	69.	-0.42	106.	319.22	1	0.
5	5.0	0.7700	0.9200	69.	69.	-0.42	106.	322.10	1	0.
6	5.0	0.6300	0.7400	69.	69.	-0.42	104.	324.91	1	0.

FINAL METER VOLUME

327.68

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 5
TRC PROJECT 82988-01

UNIT C AGRICO -EPA
SCRUBBER OUTLET
DEC 19 1978 1450 TO 1553

STANDARD CONDITION TEMPERATURE, DEG F	0.6800E 02
STANDARD CONDITION PRESSURE, IN HG	0.2992E 02
TOTAL SAMPLING TIME, MINUTES	0.6000E 02
AVERAGE SQUARE ROOT VELOCITY HEAD, IN H2O EXP .5	0.8637E 00
AVERAGE ORIFICE PRESSURE DROP, IN H2O	0.8917E 00
AVERAGE METER TEMPERATURE, DEG F	0.6796E 02
AVERAGE DUCT STATIC PRESSURE, IN H2O	-0.4050E 00
AVERAGE DUCT TEMPERATURE, DEG F	0.1048E 03
TOTAL SAMPLE VOLUME, DACF	0.3298E 02
TOTAL SAMPLE VOLUME, DSCF	0.3241E 02
WATER VAPOR VOLUME, DSCF	0.1031E 01
MOISTURE CONTENT OF DUCT GAS, PERCENT	0.3083E 01
MOLE FRACTION DRY GAS	0.9692E 00
MOLECULAR WEIGHT - DRY STACK GAS	0.2884E 02
MOLECULAR WEIGHT - STACK GAS	0.2951E 02
AVERAGE STACK PRESSURE, IN HG	0.2960E 02
DUCT VOLUMETRIC FLOW, ACFM	0.5772E 05
DUCT VOLUMETRIC FLOW, DSCFM	0.5173E 05
AVERAGE DUCT VELOCITY, FPM	0.3041E 04
EXCESS AIR, PERCENT	-0.1458E 05
AVERAGE DUCT GAS DENSITY, LBS/ACF	0.6842E-01
ISOKINETIC FACTOR, PERCENT	0.1062E 03

EMISSION DATA

	GR/ ACF	GR/ DSCF	LBS/ HR	LB/ TON
UREA IN H2O	0.1212E-01	0.1352E-01	0.5995E 01	0.5995
UREA IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL UREA	0.1212E-01	0.1352E-01	0.5995E 01	0.5995
AMMONIA-DIRECT-IN H2O	0.1459E 00	0.1628E 00	0.7220E 02	0.7220
AMMONIA-DIRECT-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL AMMONIA-DIRECT	0.1459E 00	0.1628E 00	0.7220E 02	0.7220
AMMONIA-DISTILLED-IN H2O	0.1378E 00	0.1538E 00	0.6819E 02	0.6819
AMMONIA-DISTILLED-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL AMMONIA-DISTILLED	0.1378E 00	0.1538E 00	0.6819E 02	0.6819
FORMALDEHYDE IN H2O	0.8748E-03	0.9760E-03	0.4328E 00	0.4328
FORMALDEHYDE IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL FORMALDEHYDE	0.8748E-03	0.9760E-03	0.4328E 00	0.4328

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 5
TRC PROJECT 82988-01

UNIT C AGRICO -EPA
SCRUBBER OUTLET
DEC 19 1978 1450 TO 1553

STANDARD CONDITION TEMPERATURE, DEG C	0.2000E 02
STANDARD CONDITION PRESSURE, MM HG	0.7600E 03
TOTAL SAMPLING TIME, MINUTES	0.6000E 02
AVERAGE SQUARE ROOT VELOCITY HEAD, MM H2O EXP.5	0.4353E 01
AVERAGE ORIFICE PRESSURE DROP, MM H2O	0.2265E 02
AVERAGE METER TEMPERATURE, DEG C	0.1998E 02
AVERAGE DUCT STATIC PRESSURE, MM H2O	-0.1029E 02
AVERAGE DUCT TEMPERATURE, DEG C	0.4046E 02
TOTAL SAMPLE VOLUME, DM3	0.9340E 00
TOTAL SAMPLE VOLUME, DNM3	0.9178E 00
WATER VAPOR VOLUME, DNM3	0.2919E-01
AVERAGE STACK GAS PRESSURE, MM HG	0.7518E 03
DUCT VOLUMETRIC FLOW, AM3/M	0.1635E 04
DUCT VOLUMETRIC FLOW, DNM3/M	0.1465E 04
AVERAGE DUCT VELOCITY, M/M	0.9269E 03
VVERAGE DUCT GAS DENSITY, KG/AM3	0.1096E 01

EMISSION DATA

	MG/ AM3	MG/ DNM3	KG/ HR	KG/ M3
UREA IN H2O	0.2773E 02	0.3094E 02	0.2722E 01	0.1117
UREA IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL UREA	0.2773E 02	0.3094E 02	0.2722E 01	0.1117
AMMONIA-DIRECT-IN H2O	0.3340E 03	0.3726E 03	0.3278E 02	0.1441
AMMONIA-DIRECT-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL AMMONIA-DIRECT	0.3340E 03	0.3726E 03	0.3278E 02	0.1441
AMMONIA-DISTILLED-IN H2O	0.3154E 03	0.3519E 03	0.3096E 02	0.1361
AMMONIA-DISTILLED-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL AMMONIA-DISTILLED	0.3154E 03	0.3519E 03	0.3096E 02	0.1361
FORMALDEHYDE IN H2O	0.2002E 01	0.2234E 01	0.1965E 00	0.8655
FORMALDEHYDE IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL FORMALDEHYDE	0.2002E 01	0.2234E 01	0.1965E 00	0.8655

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO
TRC PROJECT 82988-01

6

UNIT TESTED	UNIT C
DATE AND TIME OF TEST	DEC 19 1978 1608 TO 1715
SAMPLING LOCATION	SCRUBBER OUTLET
NAME OF FIRM	AGRICO -EPA
LOCATION OF FIRM	BLYTHEVILLE ARK
POLLUTANTS SAMPLED	UREA AND AMMONIA

BAROMETRIC PRESSURE. IN HG	29.63
DUCT AREA. SQ FT	18.98
NOZZLE DIAMETER. IN	0.185
PITOT CALIBRATION COEFFICIENTS 1	0.839
2	0.000
3	0.000
DRY GAS METER CALIBRATION FACTOR. Y	0.990
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	21.00
CARBON MONOXIDE	0.00
NITROGEN	79.00

UREA, AMMONIA, FORMALDEHYDE COLLECTED. MG

	UREA	AMMONIA-DIR	AMMONIA-DIST	FORMALDEHYDE
H2O IMPINGERS	0.4700E 02	0.3020E 03	0.3060E 03	0.3140E 01
H2SO4 IMPINGERS	0.0000E 00	0.0000E 00	0.0000E 00	0.0000E 00
TOTAL	0.4700E 02	0.3020E 03	0.3060E 03	0.3140E 01

AMOUNT OF WATER COLLECTED. GRAMS

IMPINGERS	23.0
SILICA GEL	5.5

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 6
TRC PROJECT 82988-01

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 ANG B
1	5.0	0.9700	1.1500	68.	68.	-0.44	100.	329.00	1	0.	
2	5.0	0.9400	1.1100	67.	68.	-0.44	105.	330.73	1	0.	
3	5.0	0.9100	1.0800	67.	68.	-0.44	106.	333.90	1	0.	
4	5.0	0.7200	0.8600	67.	67.	-0.44	105.	336.82	1	0.	
5	5.0	0.6900	0.8100	67.	67.	-0.44	104.	339.60	1	0.	
6	5.0	0.6100	0.7300	66.	66.	-0.44	103.	342.30	1	0.	
1	5.0	0.9500	1.1200	66.	66.	-0.49	104.	344.82	1	0.	
2	5.0	0.9400	1.1100	66.	66.	-0.49	105.	347.77	1	0.	
3	5.0	0.9800	1.1600	66.	66.	-0.49	105.	350.82	1	0.	
4	5.0	0.8100	0.9600	66.	66.	-0.49	106.	353.90	1	0.	
5	5.0	0.7200	0.8600	65.	66.	-0.49	105.	356.80	1	0.	
6	5.0	0.6100	0.7300	65.	65.	-0.49	103.	359.50	1	0.	

FINAL METER VOLUME

362.11

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO
TRC PROJECT 82988-01

6

UNIT C AGRICO -EPA
SCRUBBER OUTLET
DEC 19 1978 1608 TO 1715

STANDARD CONDITION TEMPERATURE, DEG F	0.6800E 02
STANDARD CONDITION PRESSURE, IN HG	0.2992E 02
TOTAL SAMPLING TIME, MINUTES	0.6000E 02
AVERAGE SQUARE ROOT VELOCITY HEAD, IN H2O EXP .5	0.9027E 00
AVERAGE ORIFICE PRESSURE DROP, IN H2O	0.9733E 00
AVERAGE METER TEMPERATURE, DEG F	0.6646E 02
AVERAGE DUCT STATIC PRESSURE, IN H2O	-0.4650E 00
AVERAGE DUCT TEMPERATURE, DEG F	0.1042E 03
TOTAL SAMPLE VOLUME, DACF	0.3411E 02
TOTAL SAMPLE VOLUME, DSCF	0.3362E 02
WATER VAPOR VOLUME, DSCF	0.1341E 01
MOISTURE CONTENT OF DUCT GAS, PERCENT	0.3837E 01
MOLE FRACTION DRY GAS	0.9616E 00
MOLECULAR WEIGHT - DRY STACK GAS	0.2884E 02
MOLECULAR WEIGHT - STACK GAS	0.2842E 02
AVERAGE STACK PRESSURE, IN HG	0.2960E 02
DUCT VOLUMETRIC FLOW, ACFM	0.6039E 05
DUCT VOLUMETRIC FLOW, DSCFM	0.5375E 05
AVERAGE DUCT VELOCITY, FPM	0.3182E 04
EXCESS AIR, PERCENT	-0.1458E 05
AVERAGE DUCT GAS DENSITY, LBS/ACF	0.6828E-01
ISOKINETIC FACTOR, PERCENT	0.1061E 03

EMISSION DATA

	GR/ ACF	GR/ DSCF	LBS/ HR	LBS TON
UREA IN H2O	0.1920E-01	0.2157E-01	0.3937E 01	0.9937
UREA IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL UREA	0.1920E-01	0.2157E-01	0.3937E 01	0.9937
AMMONIA-DIRECT-IN H2O	0.1234E 00	0.1386E 00	0.6385E 02	0.6385
AMMONIA-DIRECT-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL AMMONIA-DIRECT	0.1234E 00	0.1386E 00	0.6385E 02	0.6385
AMMONIA-DISTILLED-IN H2O	0.1250E 00	0.1404E 00	0.6470E 02	0.6470
AMMONIA-DISTILLED-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL AMMONIA-DISTILLED	0.1250E 00	0.1404E 00	0.6470E 02	0.6470
FORMALDEHYDE IN H2O	0.1283E-02	0.1441E-02	0.6639E 00	0.6639
FORMALDEHYDE IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL FORMALDEHYDE	0.1283E-02	0.1441E-02	0.6639E 00	0.6639

TEST DATA -- UREA, AMMONIA, FORMALDEHYDE -- TEST NO 6
TRC PROJECT 82988-01

UNIT C AGRICO -EPA
SCRUBBER OUTLET
DEC 19 1978 1608 TO 1715

STANDARD CONDITION TEMPERATURE, DEG C	0.2000E 02
STANDARD CONDITION PRESSURE, MM HG	0.7600E 03
TOTAL SAMPLING TIME, MINUTES	0.6000E 02
AVERAGE SQUARE ROOT VELOCITY HEAD, MM H2O EXP.5	0.4550E 01
AVERAGE ORIFICE PRESSURE DROP, MM H2O	0.2472E 02
AVERAGE METER TEMPERATURE, DEG C	0.1914E 02
AVERAGE DUCT STATIC PRESSURE, MM H2O	-0.1181E 02
AVERAGE DUCT TEMPERATURE, DEG C	0.4014E 02
TOTAL SAMPLE VOLUME, DM3	0.9660E 00
TOTAL SAMPLE VOLUME, DNM3	0.9521E 00
WATER VAPOR VOLUME, DNM3	0.3799E-01
AVERAGE STACK GAS PRESSURE, MM HG	0.7517E 03
DUCT VOLUMETRIC FLOW, AM3/M	0.1710E 04
DUCT VOLUMETRIC FLOW, DNM3/M	0.1522E 04
AVERAGE DUCT VELOCITY, M/M	0.9698E 03
VVERAGE DUCT GAS DENSITY, KG/AM3	0.1094E 01

EMISSION DATA

	MG/ AM3	MG/ DNM3	KG/ HR	KG/ MI
UREA IN H2O	0.4394E 02	0.4936E 02	0.4512E 01	0.1904
UREA IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL UREA	0.4394E 02	0.4936E 02	0.4512E 01	0.1904
AMMONIA-DIRECT-IN H2O	0.2823E 03	0.3172E 03	0.2899E 02	0.1277
AMMONIA-DIRECT-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL AMMONIA-DIRECT	0.2823E 03	0.3172E 03	0.2899E 02	0.1277
AMMONIA-DISTILLED-IN H2O	0.2861E 03	0.3214E 03	0.2937E 02	0.1291
AMMONIA-DISTILLED-IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL AMMONIA-DISTILLED	0.2861E 03	0.3214E 03	0.2937E 02	0.1291
FORMALDEHYDE IN H2O	0.2935E 01	0.3298E 01	0.3014E 00	0.1322
FORMALDEHYDE IN H2SO4	0.0000E 00	0.0000E 00	0.0000E 00	0.0000
TOTAL FORMALDEHYDE	0.2935E 01	0.3298E 01	0.3014E 00	0.1322

F\$01 STOP 00000 00000006

APPENDIX A.2

SAMPLE EQUATIONS AND EXAMPLE CALCULATIONS

EMISSION CALCULATION SYMBOLS

L_a - Allowable leak rate, cfm

V_{m_total} - Total meter sample volume, ft^3

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, ft^3

Y - Dry gas meter calibration factor, dimensionless

T_{std} - Standard temperature, $^{\circ}F$

$T_{m_avg.}$ - Average dry gas meter temperature, $^{\circ}F$

P_{bar} - Barometric pressure, "Hg

$\Delta H_{avg.}$ - 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, $^{\circ}F$

EA - Excess air, %

V_s - Average duct velocity, ft/min

C_p - Pitot tube coefficient, dimensionless

$(\sqrt{\Delta P})_{avg}$ - Average square root of velocity head, $\sqrt{"H_2O}$

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

$\%H$ - Percent by weight of hydrogen in fuel

$\%C$ - Percent by weight of carbon in fuel

$\%S$ - Percent by weight of sulfur in fuel

$\%N$ - Percent by weight of nitrogen in fuel

$\%O$ - 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

$C_s @ 12\% CO_2$ - Particulate concentration, grains/dscf @ 12% CO_2

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

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

$C_{Lb} @ 12\% CO_2$ - Particulate concentration, lbs/1000 lbs @ 12% CO_2

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

M_n - Total particulate collected, mg

Calculations for Test No. 2 Agnew (Aermet) Company
December 19, 1973

1. Allowable Leak Rate

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

$$\frac{0.04 V_m \text{ total}}{T_{\text{total}}} = \frac{0.04 \times 34.42}{60.0} = 0.023$$

$$La = 0.020 \text{ cfm}$$

2. Correction for Excessive Leak Rate

$$Lp = 0.000 \text{ cfm}$$

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

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

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

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

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

$$V_m \text{ total (std)} = 34.42 \times \left(\frac{68 + 460}{60.1 + 460} \right) \left[\frac{29.71 + \frac{1.01}{13.6}}{29.92} \right] = 34.42 \times 1.0106$$

$$V_m \text{ total (std)} = 34.78 \text{ dscf}$$

4. Moisture Content of Duct Gas

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

$$\% H_2O = \frac{0.04707 (23.0 + 5.6)}{34.78 + 0.04707 (23.0 + 5.6)} \times 100$$

$$\% H_2O = 3.73 \%$$

5. Molecular Weight of Stack Gas

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

$$M_s = \left[(0.44 \times 0) + (0.28 \times 0) + (0.28 \times 79) + (0.32 \times 21) \right] \left(1 - \frac{3.73}{100} \right) + 0.18 (3.73)$$

$$M_s = 28.37 \text{ lb/lb-mole}$$

6. Average Duct Gas Density

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

$$D_{st} = 0.0458 \times \left(\frac{29.71 + \frac{-0.40}{13.6}}{101.6 + 460} \right) \times 28.37$$

$$D_{st} = 0.06867 \text{ lbs/ft}^3$$

7. Excess Air

NA

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

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

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

8. Average Duct Velocity

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

$$V_s = 5129.4 \times 0.839 \times 0.9149 \sqrt{\frac{101.6 + 460}{\left(\frac{29.71 + \frac{-0.40}{13.6}}{m_s} \right)}}$$

$$V_s = 3215.51 \text{ ft/min}$$

9. Duct Volumetric Flow Rate

$$Q = V_s \times A_s$$

$$Q = 3215.5 \times 18.98$$

$$Q = 61030.4 \text{ acfm}$$

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

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

$$Q_{\text{std}} = 61030.4 \left(1 - \frac{3.73}{100} \right) \left(\frac{68 + 460}{101.6 + 460} \right) \left(\frac{29.71 + \frac{-0.40}{13.6}}{29.92} \right)$$

$$Q_{\text{std}} = 54796.8 \text{ dscfm}$$

11. Isokinetic Factor

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

$$I = \frac{5.67 (101.6 + 460) (34.78)}{\left(29.71 + \frac{-0.40}{13.6} \right) 3215.5 \times 60.0 \left(1 - \frac{3.73}{100} \right) \left(\frac{(18.5)^2 \times 0.7854}{144} \right)}$$

$$I = 107.6 \%$$

12. F - Factor

NA

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

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

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

13. Actual ^{UREA} Particulate Concentration

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

$$C = \frac{0.01543 \times 96.3 (68 + 460) (29.71 + \frac{-0.40}{13.6}) (1 - \frac{3.73}{100})}{34.78 (101.6 + 460) (29.92)}$$

$$C = 0.0334 \text{ grains/acf}$$

14. Particulate Concentration, Corrected to Dry Standard Conditions

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

$$C_s = 0.01543 \times \frac{96.3}{34.78}$$

$$C_s = 0.0427 \text{ grains/dscf}$$

15. Particulate Emission Rate

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

$$ER = 0.008571 \times 0.0427 \times 54796.8$$

$$ER = 20.05 \text{ lbs/hr.}$$

16. Particulate Emission

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

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

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

17. Particulate Concentration Corrected to Dry Standard Conditions and 12% CO₂

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

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

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

18. Particulate Concentration Corrected to Dry Standard Conditions and 50% Excess Air

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

NA

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

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

19. Particulate Concentration Based on Duct Gas Weight

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

NA

$$C_{Lb} = 0.1429 \times$$

$$C_{Lb} = \text{lbs/1000 lbs duct gas (uncorrected)}$$

20. Particulate Concentration Based on Duct Gas Weight Corrected to 12% CO₂

NA

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

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

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

21. Particulate Concentration Based on Duct Gas Weight Corrected to 50% Excess Air

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

NA

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

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

APPENDIX B
FIELD DATA SHEETS

TITLE Agrico Blytheville Ark.

om Page No. — 12/18/78, Arrived at plant @ 0800 and located Equip. Proceeded to set up equipment but encountered several problems. Pump head leakage, Broken Probe liner #44

3 Broken "U" tubes, Pump to impinger line missing connector, Manometer levels Low, No silica gel or grease, Poor electrical connections.

By the time these problems were resolved and we were ready to start the first test, it was 1400.

With a 1/4" nozzle we were unable to pull the 3.75" Δh required so we switched to a .185 nozzle and started test 1 at 1600. After test 1 ~~I tightened~~ the pump head and found the ~~the~~ gans were cracked. ~~to R.~~

12/19/78 replaced pump head gans but still had leak. I found leak in pump ~~(5)~~ itself and changed pumps. No leakage. Started test 2 @ 0905 Completed Test 6 @ 1715. Test 4 Port 1, pthz had a possible pitot blockage. Because there was no silica gel, we used Agricos Dryrite and Prewegged the entire impinger with dryrite.

Test	1	2	3	4	5	6				
Dryrite	736.2	741.8	723.5	721.7	727.4	727.2	—	—	—	Final
	724.2	736.2	717.9	715.8	723.5	721.7	—	—	—	Initial
	12	5.6	5.6	5.9	3.9	5.5	—	—	—	Total
Water	335	323	332	330	318	323	—	—	—	Final
	300	300	300	300	300	300	—	—	—	Initial
	35	23	32	30	18	23	—	—	—	Total
Total gain	4.7	28.6	37.6	35.9	21.9	28.5				

Stephen Richardson
12/20/78

To Page No.

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

FIELD DATA SHEETS

Firm Name Agrico Pump No. 5 Probe Diameter 1850 (4-1) in. C Factor .96 Nomograph Tm 50

Plant Location Blytheville Ark Orifice No. D-3 Assumed Moisture 5 Pitot Coefficient .889

Test No. 1 Ambient Temp 50 °F Test Duration 60 min. Orifice All 1.84

Sampling Location Scrubber Cut Bar Press 29.84 "Hg Traverse Point Interval 5 min. Test Start Time ~~1500~~ 1500

Purpose of Test Urea Probe Ident. No. 37 Probe Heater Setting 1.0 amps Test End Time 1607

Date 12/18/78 Filter Ident. No. N/A Box Temp Setting N/A Leak Test Start .004 CFHG 15 "Hg

Tester SFR Leak Test Final .004 CFHG 5 "Hg

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	Pump Vac. in Hg	Heater Box Temp.	Temp. of Gas Leaving Condenser
		Time Min	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 Meter Volume cu. ft.	P R O B E																												
1	1	50	97							1050				53			55										94		154.22	1	4.0	NA	43					
	2	50	94							1080				53			53										92		157.00	1	4.0		46					
	3	50	90							1020				53			53										91		159.92	1	4.0		50					
	4	50	80							1020				52			52										89		162.20	1	4.0		52					
	5	50	84							0970				52			51										86		165.56	1	4.0		52					
	6	50	63							0850				51			51										86		168.52	1	3.5		5.2					
2	1	50	96							1100				54			53										91		171.18	1	4.8		48					
	2	50	99							1150				54			53										94		174.12	1	5.0		52					
	3	51	97							1140				54			54										95		177.10	1	5.0		52					
	4	50	86							1010				54			54										95		180.20	1	4.2		54					
	5	50	83							0960				53			53										94		182.97	1	4.0		54					
	6	50	69							0810				54			54										94		185.76	1	3.2		54					

REMARKS

Probe Wash PW-1
 Int 724.2 - 736.2
 Silica Gel 12 g.
 Silica Gel Cond. Blue Puck

Cleaning Patch: N/A
H₂O Collected: 35
H₂O Condition: Clear

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

ORSAT Bag Sample			
CO ₂	O ₂	CO	N ₂

FIELD DATA SHEETS

Firm Name AGRICO Pump No. 21 Probe Diameter 185 Nomograph in. C Factor .99
 Plant Location Blytheville Ark. Orifice No. D-3 Assumed Moisture 5 Pitot Coefficient .839
 Test No. 2 Ambient Temp 60 °F Test Duration 60 min. Orifice ΔH 1.84
 Sampling Location Scrubber Out Bar Press 29.71 "Hg Traverse Point Interval 5 min. Test Start Time 0905
 Purpose of Test Area Probe Ident. No. 37 Probe Heater Setting 1.0 amps Test End Time 1011
 Date 12/19/78 Filter Ident. No. N/A Box Temp Setting N/A Leak Test Start 0.00 CFHQ 5 "Hg
 Tester SFR Leak Test Final 0.00 CFHQ 5 "Hg

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	Pump Vac. In Hg	Heater Box Temp.	Temp. of Gas Leaving Condenser
		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 O B E							
1	1	50	9	7	0					1	1	9	0	5	8		5	9							9	8	1	9	3	4	3	1	2.5	N/A	50.0			
	2	50	9	5	0					1	1	5	0	5	8		5	9							1	0	2	1	9	6	3	0	1	2.5		48		
	3	50	9	0	0					1	0	9	0	6	0		6	0								1	0	3	1	9	9	3	1	1	2.3		48	
	4	50	8	4	0					1	0	0	0	6	1		5	9								1	0	2	2	0	2	6	1	2.1		48		
	5	50	7	5	0					0	9	1	0	6	0		5	9								1	0	1	2	0	5	1	1	1	2.0		48	
	6	50	6	8	0					0	7	6	0	6	0		6	0								1	0	0	2	0	7	8	6	1	2.0		48	
			9	3	0					0	7	6	0															2	1	0	5	1	1	2.2				
2	1	50	9	3	0					1	1	1	0	6	0		5	9								1	0	1	2	1	0	5	1	1	2.2		48	
	2	50	9	5	0					1	1	5	0	6	1		6	0								1	0	3	2	1	3	3	9	1	2.2		48	
	3	50	9	3	0					1	1	1	0	6	2		6	1								1	0	4	2	1	6	4	4	1	2.2		48	
	4	50	8	1	0					0	9	8	0	6	1		6	1								1	0	2	2	1	4	4	7	1	2.0		48	
	5	50	7	4	0					0	9	0	0	6	2		6	2								1	0	2	2	2	3	6	1	2.0		48		
	6	50	6	9	0					0	8	2	0	6	0		6	1								1	0	1	2	2	5	1	0	1	2.0		48	
																												2	2	7	8	5	1					

REMARKS

Probe Wash N/A Cleaning Patch N/A
 Silica Gel 50C H₂O Collected 23
 Silica Gel Cond. 11 H₂O Condition 11

ORSAT Measurements

Time	CO ₂	O ₂	CO	N ₂
1				
2				

ORSAT Bag Sample

CO ₂	O ₂	CO	N ₂

Firm Name Agrico Pump No. 21 FIELD DATA SHEETS Probe Diameter .195 Nomograph 9.9
 Plant Location Blytheville Ark Orifice No. D-3 Assumed Moisture 5 Pitot Coefficient .899
 Test No. 3 Ambient Temp 64 °F Test Duration 60 min. Orifice ΔH 1.84
 Sampling Location Scrubber out Bar Press 29.68 "Hg Traverse Point Interval 5 min. Test Start Time 1100
 Purpose of Test Urea Probe Ident. No. 37 Probe Heater Setting 1.0 amps Test End Time 1205
 Date 12/19/78 Filter Ident. No. N/A Box Temp Setting N/A Leak Test Start 000 CFM @ 5 "Hg
 Tester SFR Leak Test Final 000 CFM @ 5 "Hg

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	Pump Vac. In Hg	Heater Box Temp.	Temp. of Gas Leaving Condenser
		Time Min	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 Meter Volume cu. ft.	P R O B E																												
1	1	50	940							1130				64			64									102		22794	1	2.5	N/A			48				
	2	50	950							1150				65			64									105		23080	1	2.5				48				
	3	50	870							1060				65			65									105		23380	1	2.2				53				
	4	50	680							0830				64			65									105		23669	1	2.0				53				
	5	50	680							0830				61			63									102		23935	1	2.0				53				
	6	50	620							0760				61			64									102		24196	1	2.0				56				
		50																										24462	1									
2	1	50	750							0910				65			65									103		24462	1	2.0				56				
	2	50	800							0960				65			65									104		24723	1	2.0				56				
	3	50	760							0930				65			65									106		25010	1	2.0				58				
	4	50	750							0910				65			65									106		25278	1	2.0				56				
	5	50	680							0830				65			65									107		25555	1	2.0				56				
	6	50	600							0730				65			65									105		25815	1	1.5				56				
																												26085	1									

REMARKS

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Probe Wash PW 3 Cleaning Patch N/A
 Silica Gel 5.1 H₂O Collected 32
 Silica Gel Cond. 1.14 H₂O Condition 5

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

ORSAT Bag Sample			
CO ₂	O ₂	CO	N ₂

100

Leak Test Start 0,000 CFHE 5 "Hg
Leak Test Final 0,000 CFHE 5 "Hg

REMARKS

* Possible
Pitot
Blockage

ORSAT Measurements

ORSAT Bag Sample

CO ₂	O ₂	CO	N ₂

FIELD DATA SHEETS

Firm Name AgriCo Pump No. 21 Probe Diameter .185 (4-1) in. C Factor 1.00
 Plant Location Blytheville Ark Orifice No. D-3 Assumed Moisture 4 Pitot Coefficient .839
 Test No. 6 Ambient Temp 67 °F Test Duration 60 min. Orifice All 1.84
 Sampling Location Scraper Out Bar Press 29.63 "Hg Traverse Point Interval 5 min. Test Start Time 1608
 Purpose of Test Urea Probe Ident. No. 37 Probe Heater Setting 1.0 amps Test End Time 1715
 Date 12/19/79 Filter Ident. No. N/A Box Temp Setting N/A Leak Test Start 0.000 CFME 5 "Hg
 Tester SFR Leak Test Final 0.000 CFME 5 "Hg

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	Pump Vac. in Hg	Heater Box Temp.	Temp. of Gas Leaving Condenser
		Time Min	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 Meter Volume cu. ft.				P R O B E															
1	1	50	970	970	970	970	1150	1150	1150	68	68	68	68	68	68	68	68	68	68	68	68	68		68	68	100	32	800	1	2.5	N/A	48						
	2	50	940	940	940	940	1110	1110	1110	67	67	67	67	67	67	67	67	67	67	67	67	67	67	67	105	33	073	1	2.5		48							
	3	50	910	910	910	910	1080	1080	1080	67	67	67	67	67	67	67	67	67	67	67	67	67	67	67	106	33	390	1	2.5		48							
	4	50	720	720	720	720	0860	0860	0860	67	67	67	67	67	67	67	67	67	67	67	67	67	67	67	105	33	682	1	2.1		48							
	5	50	670	670	670	670	0810	0810	0810	67	67	67	67	67	67	67	67	67	67	67	67	67	67	67	104	33	960	1	2.0		48							
	6	50	610	610	610	610	0730	0730	0730	66	66	66	66	66	66	66	66	66	66	66	66	66	66	66	103	34	230	1	1.9		48							
		50																									34	482	1									
2	1	50	950	950	950	950	1120	1120	1120	66	66	66	66	66	66	66	66	66	66	66	66	66	66	66	104	34	482	1	2.5		48							
	2	50	940	940	940	940	1110	1110	1110	66	66	66	66	66	66	66	66	66	66	66	66	66	66	66	105	34	777	1	2.5		46							
	3	50	980	980	980	980	1160	1160	1160	66	66	66	66	66	66	66	66	66	66	66	66	66	66	66	105	35	082	1	2.5		49							
	4	50	810	810	810	810	0960	0960	0960	66	66	66	66	66	66	66	66	66	66	66	66	66	66	66	106	35	390	1	2.2		49							
	5	50	720	720	720	720	0860	0860	0860	65	65	65	65	65	65	65	65	65	65	65	65	65	65	65	105	35	680	1	2.0		50							
	6	50	610	610	610	610	0730	0730	0730	65	65	65	65	65	65	65	65	65	65	65	65	65	65	65	103	35	950	1	2.0		50							
																												36	211	1								

REMARKS

Probe Wash PN G Cleaning Patch N/A
 Silica Gel 2.2 H₂O Collected 23
 Silica Gel Cond. 12 H₂O Condition 12

ORSAT Measurements

Time	CO ₂	O ₂	CO	N ₂
1				
2				

ORSAT Bag Sample

CO ₂	O ₂	CO	N ₂

APPENDIX C

SAMPLING AND ANALYSIS PROCEDURES

Includes:

- C.1 Urea Procedures
- C.2 Ammonia Procedures
- C.3 Formaldehyde Procedures

APPENDIX C.1
UREA PROCEDURES

5/12/80

APPENDIX A - REFERENCE TEST METHOD

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METHOD 28 - DETERMINATION OF PARTICULATE (UREA) EMISSIONS FROM UREA PLANTS

1. Applicability and Principle

1.1 Applicability. This method applies to the determination of particulate emissions as urea from urea manufacturing facilities.

1.2 Principle. A gas sample is extracted isokinetically from the stack. The ammonia is removed from the sample by boiling, and the particulate emissions are determined as urea by a colorimetric procedure.

2. Apparatus

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 28-1; it is similar in 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 impingers connected in series as shown in Figure 28-1. For the second and third impinger, the tester shall use the Greenburg-Smith design with standard tips. For the first, fourth, and fifth impingers, the tester may use the Greenburg-Smith design, modified by replacing the tips with a 1.25 cm (0.5 in.) ID

glass tube extending to 1.25 cm (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 Containers, 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. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester.

2.2.3 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 Polyethylene.

2.3 Analysis. For analysis, the following equipment is needed.

2.3.1 Pipettes. Volumetric type, 0.5-ml, 2-ml, 5-ml, 8-ml, 10-ml, 20-ml, and 25-ml.

2.3.2 Volumetric Flasks. 25-ml, 100-ml, 250-ml, 500-ml, and 1000-ml.

2.3.3 Graduated Cylinder. 100-ml.

2.3.4 Distillation Apparatus.

2.3.4.1 Flasks or Beakers. At least two, 800-ml.

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.

3.1 Sampling and Sample Recovery. The reagents used in sampling and sample recovery are as follows:

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. 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.

3.2 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. Same as 3.1.3.

3.3.8 Ethyl Alcohol, 95 percent.

3.3.9 p-dimethylaminobenzaldehyde.

3.3.10 Hydrochloric Acid, Concentrated.

3.3.11 Urea Solution, 2.5 mg/ml. Dissolve 2.500 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. Dissolve 2.000 g of p-dimethylaminobenzaldehyde in a mixture of 100 ml of 95 percent ethyl alcohol and 10 ml of concentrated 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 three impingers, place 100 ml of 1 N H_2SO_4 in the fourth impinger, and place the preweighed silica gel in the fifth impinger. Assemble the train as shown in Figure 28-1.

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 Training 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. Begin proper cleanup procedure 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, thus drawing water from the impingers into the probe.

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.

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

4.2.1 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 clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

4.2.2 Container No. 2. Mark the liquid level of the container to determine later if leakage occurred during shipment. Cap and seal the containers and identify. Measure to the nearest ± 1 ml and record the volume of the first three impingers. Then transfer the contents to the container. Rinse the first three 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.

4.2.3 Impinger No. 4. Measure to the nearest ± 1 ml and record the volume of the fourth impinger. Discard the liquid.

4.2.4 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. The tester may use a funnel and rubber policeman as aids in transferring the silica gel. 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, the tester may follow the procedure for container No. 3 in section 4.3.2.

4.2.5 Water Blank. 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."

4.3 Analysis. Record the data required on a sheet such as the one shown in Figure 5-3 of Method 5. Handle each sample container as follows:

4.3.1 Containers No. 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 ± 0.5 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.

4.3.4.1 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: Conduct this step 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. Treat the sample and blank as follows: Pipette 10 ml 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 of 420 nm using the blank solution as a zero reference. If the absorbance exceeds that of the 5.00- $\mu\text{g/ml}$ 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, 1.0, 5.0, 10.0, 15.0, 20.0 and 25.0 ml of the 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, 0.100, 0.500, 1.00, 1.50, 2.00, and 2.50 mg respectively. The calibration procedure must be repeated each day that samples are analyzed. Calculate the spectrophotometer calibration factor as follows:

$$K_c = 0.100 \frac{A_1 + 5A_2 + 10A_3 + 15A_4 + 20A_5 + 25A_6}{A_1^2 + A_2^2 + A_3^2 + A_4^2 + A_5^2 + A_6^2}$$

Where:

K_c = Calibration factor.

A_1 = Absorbance of the 0.100 mg standard.

A_2 = Absorbance of the 0.500 mg standard.

A_3 = Absorbance of the 1.00 mg standard.

A_4 = Absorbance of the 1.50 mg standard.

A_5 = Absorbance of the 2.0 mg standard.

A_6 = Absorbance of the 2.50 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 28-1.

$$m = K_c \left(A_s \frac{V_{\text{soln}}}{V_{\text{al}}} - A_w \frac{V_w}{V_b} \right) \quad \text{Eq. 28-1}$$

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_{soln} = Total volume of solution in which the sample is contained, ml.

6.3 Particulate Concentration: Calculate the particulate (urea) concentration as follows:

$$c = K_2 \frac{m}{V_{\text{m(std)}}} 10^{-3} \quad \text{Eq. 28-2}$$

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_2 = 1.0 for metric units.
= 0.4370 for English units.

7. Bibliography

1. American Public Health Association. Standards Methods for the Examination of Water and Wastewater, 13th Edition. Washington, D.C. 1974. pp. 226-232.
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 1 through 9 of section 7.

NITROGEN, KJELDAHL, TOTAL

Method 351.3 (Colorimetric; Titrimetric; Potentiometric)

STORET NO. 00625

1. Scope and Application

1.1 This method covers the determination of total Kjeldahl nitrogen in drinking, surface and saline waters, domestic and industrial wastes. The procedure converts nitrogen components of biological origin such as amino acids, proteins and peptides to ammonia, but may not convert the nitrogenous compounds of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines.

1.2 Three alternatives are listed for the determination of ammonia after distillation: the titrimetric method which is applicable to concentrations above 1 mg N/liter; the Nesslerization method which is applicable to concentrations below 1 mg N/liter; and the potentiometric method applicable to the range 0.05 to 1400 mg/l.

1.3 This method is described for macro and micro glassware systems.

2. Definitions

2.1 Total Kjeldahl nitrogen is defined as the sum of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate $(\text{NH}_4)_2\text{SO}_4$, under the conditions of digestion described below.

2.2 Organic Kjeldahl nitrogen is defined as the difference obtained by subtracting the free-ammonia value (Method 350.2, Nitrogen, Ammonia, this manual) from the total Kjeldahl nitrogen value. This may be determined directly by removal of ammonia before digestion.

3. Summary of Method

3.1 The sample is heated in the presence of conc. sulfuric acid, K_2SO_4 and HgSO_4 and evaporated until SO_3 fumes are obtained and the solution becomes colorless or pale yellow. The residue is cooled, diluted, and is treated and made alkaline with a hydroxide-thiosulfate solution. The ammonia is distilled and determined after distillation by Nesslerization, titration or potentiometry.

4. Sample Handling and Preservation

4.1 Samples may be preserved by addition of 2 ml of conc. H_2SO_4 per liter and stored at 4°C. Even when preserved in this manner, conversion of organic nitrogen to ammonia may occur. Preserved samples should be analyzed as soon as possible.

5. Interference

5.1 High nitrate concentrations (10X or more than the TKN level) result in low TKN values. The reaction between nitrate and ammonia can be prevented by the use of an anion exchange resin (chloride form) to remove the nitrate prior to the TKN analysis.

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6. Apparatus
 - 6.1 Digestion apparatus: A Kjeldahl digestion apparatus with 800 or 100 ml flasks and suction takeoff to remove SO_2 fumes and water.
 - 6.2 Distillation apparatus: The macro Kjeldahl flask is connected to a condenser and an adaptor so that the distillate can be collected. Micro Kjeldahl steam distillation apparatus is commercially available.
 - 6.3 Spectrophotometer for use at 400 to 425 nm with a light path of 1 cm or longer.
7. Reagents
 - 7.1 Distilled water should be free of ammonia. Such water is best prepared by the passage of distilled water through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin. Regeneration of the column should be carried out according to the manufacturer's instructions.
NOTE 1: All solutions must be made with ammonia-free water.
 - 7.2 Mercuric sulfate solution: Dissolve 8 g red mercuric oxide (HgO) in 50 ml of 1:4 sulfuric acid (10.0 ml conc. H_2SO_4 : 40 ml distilled water) and dilute to 100 ml with distilled water.
 - 7.3 Sulfuric acid-mercuric sulfate-potassium sulfate solution: Dissolve 267 g K_2SO_4 in 1300 ml distilled water and 400 ml conc. H_2SO_4 . Add 50 ml mercuric sulfate solution (7.2) and dilute to 2 liters with distilled water.
 - 7.4 Sodium hydroxide-sodium thiosulfate solution: Dissolve 500 g NaOH and 25 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in distilled water and dilute to 1 liter.
 - 7.5 Mixed indicator: Mix 2 volumes of 0.2% methyl red in 95% ethanol with 1 volume of 0.2% methylene blue in ethanol. Prepare fresh every 30 days.
 - 7.6 Boric acid solution: Dissolve 20 g boric acid, H_3BO_3 , in water and dilute to 1 liter with distilled water.
 - 7.7 Sulfuric acid, standard solution: (0.02 N) 1 ml = 0.28 mg $\text{NH}_3\text{-N}$. Prepare a stock solution of approximately 0.1 N acid by diluting 3 ml of conc. H_2SO_4 (sp. gr. 1.84) to 1 liter with CO_2 -free distilled water. Dilute 200 ml of this solution to 1 liter with CO_2 -free distilled water. Standardize the approximately 0.02 N acid so prepared against 0.0200 N Na_2CO_3 solution. This last solution is prepared by dissolving 1.060 g anhydrous Na_2CO_3 , oven-dried at 140°C , and diluting to 1 liter with CO_2 -free distilled water.
NOTE 2: An alternate and perhaps preferable method is to standardize the approximately 0.1 N H_2SO_4 solution against a 0.100 N Na_2CO_3 solution. By proper dilution the 0.02 N acid can be prepared.
 - 7.8 Ammonium chloride, stock solution: 1.0 ml = 1.0 mg $\text{NH}_3\text{-N}$. Dissolve 3.819 g NH_4Cl in water and make up to 1 liter in a volumetric flask with distilled water.
 - 7.9 Ammonium chloride, standard solution: 1.0 ml = 0.01 mg $\text{NH}_3\text{-N}$. Dilute 10.0 ml of the stock solution (7.8) with distilled water to 1 liter in a volumetric flask.
 - 7.10 Nessler reagent: Dissolve 100 g of mercuric iodide and 70 g potassium iodide in a small volume of distilled water. Add this mixture slowly, with stirring, to a cooled solution of 160 g of NaOH in 500 ml of distilled water. Dilute the mixture to 1 liter. The solution is stable for at least one year if stored in a pyrex bottle out of direct sunlight.

NOTE 3: Reagents 7.7, 7.8, 7.9, and 7.10 are identical to reagents 6.8, 6.2, 6.3, and 6.6 described under Nitrogen, Ammonia (Colorimetric; Titrimetric; Potentiometric-Distillation Procedure, Method 350.2).

8. Procedure

8.1 The distillation apparatus should be pre-steamed before use by distilling a 1:1 mixture of distilled water and sodium hydroxide-sodium thiosulfate solution (7.4) until the distillate is ammonia-free. This operation should be repeated each time the apparatus is out of service long enough to accumulate ammonia (usually 4 hours or more).

8.2 Macro Kjeldahl system

8.2.1 Place a measured sample or the residue from the distillation in the ammonia determination (for Organic Kjeldahl only) into an 800 ml Kjeldahl flask. The sample size can be determined from the following table:

Kjeldahl Nitrogen in Sample, mg/l	Sample Size ml
0-5	500
5-10	250
10-20	100
20-50	50.0
50-500	25.0

Dilute the sample, if required, to 500 ml with distilled water, and add 100 ml sulfuric acid-mercuric sulfate-potassium sulfate solution (7.3). Evaporate the mixture in the Kjeldahl apparatus until SO_3 fumes are given off and the solution turns colorless or pale yellow. Continue heating for 30 additional minutes. Cool the residue and add 300 ml distilled water.

8.2.2 Make the digestate alkaline by careful addition of 100 ml of sodium hydroxide - thiosulfate solution (7.4) without mixing.

NOTE 5: Slow addition of the heavy caustic solution down the tilted neck of the digestion flask will cause heavier solution to underlay the aqueous sulfuric acid solution without loss of free-ammonia. Do not mix until the digestion flask has been connected to the distillation apparatus.

8.2.3 Connect the Kjeldahl flask to the condenser with the tip of condenser or an extension of the condenser tip below the level of the boric acid solution (7.6) in the receiving flask.

8.2.4 Distill 300 ml at the rate of 6-10 ml/min., into 50 ml of 2% boric acid (7.6) contained in a 500 ml Erlenmeyer flask.

8.2.5 Dilute the distillate to 500 ml in the flask. These flasks should be marked at the 350 and the 500 ml volumes. With such marking, it is not necessary to transfer the distillate to volumetric flasks. For concentrations above 1 mg/l, the ammonia can be determined titrimetrically. For concentrations below this value, it is determined colorimetrically. The potentiometric method is applicable to the range 0.05 to 1400 mg/l.

8.3 Micro Kjeldahl system

- 8.3.1 Place 50.0 ml of sample or an aliquot diluted to 50 ml in a 100 ml Kjeldahl flask and add 10 ml sulfuric acid-mercuric sulfate-potassium sulfate solution (7.3). Evaporate the mixture in the Kjeldahl apparatus until SO_3 fumes are given off and the solution turns colorless or pale yellow. Then digest for an additional 30 minutes. Cool the residue and add 30 ml distilled water.
- 8.3.2 Make the digestate alkaline by careful addition of 10 ml of sodium hydroxide-thiosulfate solution (7.4) without mixing. Do not mix until the digestion flask has been connected to the distillation apparatus.
- 8.3.3 Connect the Kjeldahl flask to the condenser with the tip of condenser or an extension of the condenser tip below the level of the boric acid solution (7.6) in the receiving flask or 50 ml short-form Nessler tube.
- 8.3.4 Steam distill 30 ml at the rate of 6–10 ml/min., into 5 ml of 2% boric acid (7.6).
- 8.3.5 Dilute the distillate to 50 ml. For concentrations above 1 mg/l the ammonia can be determined titrimetrically. For concentrations below this value, it is determined colorimetrically. The potentiometric method is applicable to the range 0.05 to 1400 mg/l.

8.4 Determination of ammonia in distillate: Determine the ammonia content of the distillate titrimetrically, colorimetrically, or potentiometrically, as described below.

- 8.4.1 Titrimetric determination: Add 3 drops of the mixed indicator (7.5) to the distillate and titrate the ammonia with the 0.02 N H_2SO_4 (7.7), matching the endpoint against a blank containing the same volume of distilled water and H_3BO_3 (7.6) solution.
- 8.4.2 Colorimetric determination: Prepare a series of Nessler tube standards as follows:

<u>ml of Standard</u> <u>1.0 ml = 0.01 mg $\text{NH}_3\text{-N}$</u>	<u>mg $\text{NH}_3\text{-N}/50.0$ ml</u>
0.0	0.0
0.5	0.005
1.0	0.010
2.0	0.020
4.0	0.040
5.0	0.050
8.0	0.080
10.0	0.10

Dilute each tube to 50 ml with ammonia free water, add 1 ml of Nessler Reagent (7.10) and mix. After 20 minutes read the absorbance at 425 nm against the blank. From the values obtained for the standards plot absorbance vs. mg $\text{NH}_3\text{-N}$ for the standard curve. Develop color in the 50 ml diluted distillate in exactly the same manner and read mg $\text{NH}_3\text{-N}$ from the standard curve.

- 8.4.3 Potentiometric determination: Consult the method entitled Nitrogen, Ammonia: Potentiometric, Ion Selective Electrode Method, (Method 350.3) in this manual.
- 8.4.4 It is not imperative that all standards be treated in the same manner as the samples. It is recommended that at least 2 standards (a high and low) be digested, distilled,

and compared to similar values on the curve to insure that the digestion-distillation technique is reliable. If treated standards do not agree with untreated standards the operator should find the cause of the apparent error before proceeding.

9. Calculation

9.1 If the titrimetric procedure is used, calculate Total Kjeldahl Nitrogen, in mg/l, in the original sample as follows:

$$\text{TKN, mg/l} = \frac{(A - B)N \times F \times 1,000}{S}$$

where:

A = milliliters of standard 0.020 N H_2SO_4 solution used in titrating sample.

B = milliliters of standard 0.020 N H_2SO_4 solution used in titrating blank.

N = normality of sulfuric acid solution.

F = milliequivalent weight of nitrogen (14 mg).

S = milliliters of sample digested.

If the sulfuric acid is exactly 0.02 N the formula is shortened to:

$$\text{TKN, mg/l} = \frac{(A - B) \times 280}{S}$$

9.2 If the Nessler procedure is used, calculate the Total Kjeldahl Nitrogen, in mg/l, in the original sample as follows:

$$\text{TKN, mg/l} = \frac{A \times 1,000}{D} \times \frac{B}{C}$$

where:

A = mg $\text{NH}_3\text{-N}$ read from curve.

B = ml total distillate collected including the H_3BO_3 .

C = ml distillate taken for Nesslerization.

D = ml of original sample taken.

9.3 Calculate Organic Kjeldahl Nitrogen in mg/l, as follows:

Organic Kjeldahl Nitrogen = TKN - ($\text{NH}_3\text{-N}$.)

- 9.4 Potentiometric determination: Calculate Total Kjeldahl Nitrogen, in mg/l, in the original sample as follows:

$$\text{TKN, mg/l} = \frac{B}{D} \times A$$

where:

A = mg $\text{NH}_3\text{-N/l}$ from electrode method standard curve.

B = volume of diluted distillate in ml.

D = ml of original sample taken.

10. Precision

- 10.1 Thirty-one analysts in twenty laboratories analyzed natural water samples containing exact increments of organic nitrogen, with the following results:

Increment as Nitrogen, Kjeldahl mg N/liter	Precision as Standard Deviation mg N/liter	Accuracy as	
		Bias, %	Bias, mg N/liter
0.20	0.197	+ 15.54	+ 0.03
0.31	0.247	+ 5.45	+ 0.02
4.10	1.056	+ 1.03	+ 0.04
4.61	1.191	- 1.67	- 0.08

(FWPCA Method Study 2, Nutrient Analyses)

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 437, Method 421 (1975).
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APPENDIX C.2
AMMONIA PROCEDURES

NITROGEN, AMMONIA

Method 350.2 (Colorimetric; Titrimetric; Potentiometric - Distillation Procedure)

STORET NO. Total 00610
Dissolved 00608

1. Scope and Application
 - 1.1 This distillation method covers the determination of ammonia-nitrogen exclusive of total Kjeldahl nitrogen, in drinking, surface and saline waters, domestic and industrial wastes. It is the method of choice where economics and sample load do not warrant the use of automated equipment.
 - 1.2 The method covers the range from about 0.05 to 1.0 mg $\text{NH}_3\text{-N/l}$ for the colorimetric procedure, from 1.0 to 25 mg/l for the titrimetric procedure, and from 0.05 to 1400 mg/l for the electrode method.
 - 1.3 This method is described for macro glassware; however, micro distillation equipment may also be used.
2. Summary of Method
 - 2.1 The sample is buffered at a pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanates and organic nitrogen compounds, and is then distilled into a solution of boric acid. The ammonia in the distillate can be determined colorimetrically by nesslerization, titrimetrically with standard sulfuric acid with the use of a mixed indicator, or potentiometrically by the ammonia electrode. The choice between the first two procedures depends on the concentration of the ammonia.
3. Sample Handling and Preservation
 - 3.1 Samples may be preserved with 2 ml of conc. H_2SO_4 per liter and stored at 4°C.
4. Interferences
 - 4.1 A number of aromatic and aliphatic amines, as well as other compounds, both organic and inorganic, will cause turbidity upon the addition of Nessler reagent, so direct nesslerization (i.e., without distillation), has been discarded as an official method.
 - 4.2 Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which distillation is carried out. Volatile alkaline compounds, such as certain ketones, aldehydes, and alcohols, may cause an off-color upon nesslerization in the distillation method. Some of these, such as formaldehyde, may be eliminated by boiling off at a low pH (approximately 2 to 3) prior to distillation and nesslerization.
 - 4.3 Residual chlorine must also be removed by pretreatment of the sample with sodium thiosulfate before distillation.

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5. Apparatus

- 5.1 An all-glass distilling apparatus with an 800–1000 ml flask.
- 5.2 Spectrophotometer or filter photometer for use at 425 nm and providing a light path of 1 cm or more.
- 5.3 Nessler tubes: Matched Nessler tubes (APHA Standard) about 300 mm long, 17 mm inside diameter, and marked at 225 mm \pm 1.5 mm inside measurement from bottom.
- 5.4 Erlenmeyer flasks: The distillate is collected in 500 ml glass-stoppered flasks. These flasks should be marked at the 350 and the 500 ml volumes. With such marking, it is not necessary to transfer the distillate to volumetric flasks.

6. Reagents

- 6.1 Distilled water should be free of ammonia. Such water is best prepared by passage through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin. Regeneration of the column should be carried out according to the manufacturer's instructions.

NOTE 1: All solutions must be made with ammonia-free water.

- 6.2 Ammonium chloride, stock solution: 1.0 ml = 1.0 mg $\text{NH}_3\text{-N}$. Dissolve 3.819 g NH_4Cl in distilled water and bring to volume in a 1 liter volumetric flask.
- 6.3 Ammonium chloride, standard solution: 1.0 ml = 0.01 mg. Dilute 10.0 ml of stock solution (6.2) to 1 liter in a volumetric flask.
- 6.4 Boric acid solution (20 g/l): Dissolve 20 g H_3BO_3 in distilled water and dilute to 1 liter.
- 6.5 Mixed indicator: Mix 2 volumes of 0.2% methyl red in 95% ethyl alcohol with 1 volume of 0.2% methylene blue in 95% ethyl alcohol. This solution should be prepared fresh every 30 days.

NOTE 2: Specially denatured ethyl alcohol conforming to Formula 3A or 30 of the U.S. Bureau of Internal Revenue may be substituted for 95% ethanol.

- 6.6 Nessler reagent: Dissolve 100 g of mercuric iodide and 70 g of potassium iodide in a small amount of water. Add this mixture slowly, with stirring, to a cooled solution of 160 g of NaOH in 500 ml of water. Dilute the mixture to 1 liter. If this reagent is stored in a Pyrex bottle out of direct sunlight, it will remain stable for a period of up to 1 year.

NOTE 3: This reagent should give the characteristic color with ammonia within 10 minutes after addition, and should not produce a precipitate with small amounts of ammonia (0.04 mg in a 50 ml volume).

- 6.7 Borate buffer: Add 88 ml of 0.1 N NaOH solution to 500 ml of 0.025 M sodium tetraborate solution (5.0 g anhydrous $\text{Na}_2\text{B}_4\text{O}_7$ or 9.5 g $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ per liter) and dilute to 1 liter.
- 6.8 Sulfuric acid, standard solution: (0.02 N, 1 ml = 0.28 mg $\text{NH}_3\text{-N}$). Prepare a stock solution of approximately 0.1 N acid by diluting 3 ml of conc. H_2SO_4 (sp. gr. 1.84) to 1 liter with CO_2 -free distilled water. Dilute 200 ml of this solution to 1 liter with CO_2 -free distilled water.

NOTE 4: An alternate and perhaps preferable method is to standardize the approximately 0.1 N H_2SO_4 solution against a 0.100 N Na_2CO_3 solution. By proper dilution the 0.02 N acid can then be prepared.

6.8.1 Standardize the approximately 0.02 N acid against 0.0200 N Na_2CO_3 solution. This last solution is prepared by dissolving 1.060 g anhydrous Na_2CO_3 , oven-dried at 140°C , and diluting to 1000 ml with CO_2 -free distilled water.

6.9 Sodium hydroxide, 1 N: Dissolve 40 g NaOH in ammonia-free water and dilute to 1 liter.

6.10 Dechlorinating reagents: A number of dechlorinating reagents may be used to remove residual chlorine prior to distillation. These include:

a. Sodium thiosulfate (1/70 N): Dissolve 3.5 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in distilled water and dilute to 1 liter. One ml of this solution will remove 1 mg/l of residual chlorine in 500 ml of sample.

b. Sodium arsenite (1/70 N): Dissolve 1.0 g NaAsO_2 in distilled water and dilute to 1 liter.

7. Procedure

7.1 Preparation of equipment: Add 500 ml of distilled water to an 800 ml Kjeldahl flask. The addition of boiling chips which have been previously treated with dilute NaOH will prevent bumping. Steam out the distillation apparatus until the distillate shows no trace of ammonia with Nessler reagent.

7.2 Sample preparation: Remove the residual chlorine in the sample by adding dechlorinating agent equivalent to the chlorine residual. To 400 ml of sample add 1 N NaOH (6.9), until the pH is 9.5, checking the pH during addition with a pH meter or by use of a short range pH paper.

7.3 Distillation: Transfer the sample, the pH of which has been adjusted to 9.5, to an 800 ml Kjeldahl flask and add 25 ml of the borate buffer (6.7). Distill 300 ml at the rate of 6–10 ml/min. into 50 ml of 2% boric acid (6.4) contained in a 500 ml Erlenmeyer flask.

NOTE 5: The condenser tip or an extension of the condenser tip must extend below the level of the boric acid solution.

Dilute the distillate to 500 ml with distilled water and nesslerize an aliquot to obtain an approximate value of the ammonia-nitrogen concentration. For concentrations above 1 mg/l the ammonia should be determined titrimetrically. For concentrations below this value it is determined colorimetrically. The electrode method may also be used.

7.4 Determination of ammonia in distillate: Determine the ammonia content of the distillate titrimetrically, colorimetrically or potentiometrically as described below.

7.4.1 Titrimetric determination: Add 3 drops of the mixed indicator to the distillate and titrate the ammonia with the 0.02 N H_2SO_4 , matching the end point against a blank containing the same volume of distilled water and H_3BO_3 solution.

7.4.2 Colorimetric determination: Prepare a series of Nessler tube standards as follows:

ml of Standard 1.0 ml = 0.01 mg NH ₃ -N	mg NH ₃ -N/50.0 ml
0.0	0.0
0.5	0.005
1.0	0.01
2.0	0.02
3.0	0.03
4.0	0.04
5.0	0.05
8.0	0.08
10.0	0.10

Dilute each tube to 50 ml with distilled water, add 2.0 ml of Nessler reagent (6.6) and mix. After 20 minutes read the absorbance at 425 nm against the blank. From the values obtained plot absorbance vs. mg NH₃-N for the standard curve. Determine the ammonia in the distillate by nesslerizing 50 ml or an aliquot diluted to 50 ml and reading the absorbance at 425 nm as described above for the standards. Ammonia-nitrogen content is read from the standard curve.

7.4.3 Potentiometric determination: Consult the method entitled Nitrogen, Ammonia: Selective Ion Electrode Method (Method 350.3) in this manual.

- 7.5 It is not imperative that all standards be distilled in the same manner as the samples. It is recommended that at least two standards (a high and low) be distilled and compared to similar values on the curve to insure that the distillation technique is reliable. If distilled standards do not agree with undistilled standards the operator should find the cause of the apparent error before proceeding.

8. Calculations

8.1 Titrimetric

$$\text{mg/l NH}_3 - \text{N} = \frac{A \times 0.28 \times 1,000}{S}$$

where:

A = ml 0.02 N H₂SO₄ used.

S = ml sample.

8.2 Spectrophotometric

$$\text{mg/l NH}_3 - \text{N} = \frac{A \times 1,000}{D} \times \frac{B}{C}$$

where:

A = mg NH₃-N read from standard curve.

B = ml total distillate collected, including boric acid and dilution.

C = ml distillate taken for nesslerization.

D = ml of original sample taken.

8.3 Potentiometric

$$\text{mg/l NH}_3 - \text{N} = \frac{500}{D} \times A$$

where:

A = mg NH₃-N/l from electrode method standard curve.

D = ml of original sample taken.

9. Precision and Accuracy

9.1 Twenty-four analysts in sixteen laboratories analyzed natural water samples containing exact increments of an ammonium salt, with the following results:

Increment as Nitrogen, Ammonia mg N/liter	Precision as Standard Deviation mgN/liter	Accuracy as	
		Bias, %	Bias, mg N/liter
0.21	0.122	-5.54	-0.01
0.26	0.070	-18.12	-0.05
1.71	0.244	+0.46	+0.01
1.92	0.279	-2.01	-0.04

(FWPCA Method Study 2, Nutrient Analyses)

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1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 410, Method 418A and 418B (1975).
2. Annual Book of ASTM Standards, Part 31, "Water", Standard D1426-74, Method A, p 237 (1976).

APPENDIX C.3
FORMALDEHYDE PROCEDURES

TENTATIVE METHOD FOR **DO NOT QUOTE OR CITE**
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. This 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.

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3. Range:

3.1 Formaldehyde: .05 $\mu\text{g/ml}$ - 2.0 $\mu\text{g/ml}$; Based on impinger solution of 600 ml and 60 Ft^3 gas collected: 6 - 240 ppm; upper limit is easily extended by diluting aliquot taken.

4. Sensitivity: unknown5. Precision:

5.1 Formaldehyde: $\pm 5\%$

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*	(-)

7.2 Methanol; same as above

7.3 Dimethyl ether; unknown

*Use of Aqueous bisulfite solution as the scrubbing medium will reduce interference of nitrogen oxides.

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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.

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8.3.3 Gas chromatograph

8.3.4 Flame ionization detector

8.3.5 Recorder

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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": (10 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 12.7g 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.

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✓ 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 100 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 ^{EXD}

9.5.1 Chromatographic column: 10% triethyl acetyl citrate.

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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 Ft³ 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.

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→ 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 1cm 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.)

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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.

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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.

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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.

$$\text{ppm (volume)} = \frac{(C) \times (S) \times (24.15)}{(2) \times (V_s) \times (MW)}$$

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 = Meter Temp., °F

C = μg of formaldehyde in aliquot (from calibration 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 an aliquot of equal size (previously determined).

This is the μg formaldehyde in the aliquot formed from the oxidation of methanol.

$$12.2.2 \quad M = FM \left(\frac{30.03}{32.04} \right)$$

$$12.2.3 \quad M_C = \frac{(M) (S) (24.15)}{(A) (V_S) (MW)}$$

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.

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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 D
ANALYTICAL DATA

Includes:

- D.1 Data Analysis Summaries
- D.2 Chemical Laboratory Notebook
- D.3 Scrubber Liquor Sampling Times

APPENDIX D.1
DATA ANALYSIS SUMMARIES

UREA AUDIT SAMPLES (TRC)

INITIAL STACK GAS ANALYSES (TRC)

Sample no.	V _r	F	Absorbance	X ₁	17/F = C	V ₀	Blank CORR. mg NH ₃ /dm ³	Digestion Volume (ml)	Mass of Urea mg	Sample no.	V _r	F ₀	Absorbance	X ₁	C	(X ₁ - C) × F ₀ × 1000	Net Blank (mg NH ₃ /dm ³)	
	Sample Volume (ml)	DILUTION FACTORS		mg NH ₃ /dm ³	Blank CORR. mg NH ₃ /dm ³	mg NH ₃ /dm ³	mg	Sample Volume (ml)	DILUTION FACTORS		mg NH ₃ /dm ³	Blank CORR. mg NH ₃ /dm ³		mg				
1	400	5	.241	70	3.4	25	94.04			1	475	10 × 20 10 × 25 4 × 10	11/11/78 " "	0.359 .301 .164	103.5 87.0 48.0	0 0.2 5.2	403.65 413.15 58.92	403.7 424.1 66.1
2	400	1 × 10	.361	104	1.7	25	288.90			2	990	10 × 20 10 × 25 10 × 25	11/11 " "	.285 .226 .462	84.0 66.5 135.0	0 0.2 6.3	332.6 328.2 90.0	332.6 328.2 94.4
3	400	1 × 20	.352	101.5	0.8	25	568.75			3	1100	10 × 20 10 × 25 10 × 25	11/10 " "	.285 .367 .168	84 108 49.5	0 0.2 6.3	369.6 592.9 33.6	369.6 594.0 38.5
4	400	1	.326	94	1.7	100	5.44			4	970	20 × 10 10 × 25 10 × 25	11/10 " "	.319 .244 .261	93.5 72 76.5	0 0.2 6.3	362.8 348.2 48.1	362.8 348.2 52.4
5	400	1	.608	175	1.7	100	11.15			5	1095	20 × 10 10 × 25 10 × 25	11/10 " "	.266 .201 .145	78 59 43	0 0.2 6.3	341.6 321.9 28.8	341.6 321.9 33.7
6	400	1	.535	154	1.7	25	38.69			6	1005	20 × 10 10 × 25 10 × 25	12/10 " "	.255 .206 .248	75 61 72.5	0 0.2 6.3	301.5 305.5 47.0	301.5 306.5 51.5
7	250	1	.247	72	1.7	100	2.43											
8	250	2	.212	84	8.5	100	6.49											
9	250	5/15	.226	66	5.1	100	8.96											
10	250	2	.220	64	8.5	100	4.90											
11	250	2	.181	53	8.5	100	3.93											
12	250	10	.225	65	1.7	100	27.93											
BLANK		1	.055	17														
											V	F ₀		X ₁		(X ₁ - F ₀) × V 4 × 1000 mg NH ₃ (mg)		
										1	475	10	11/10/75	.084	1.6		3.90	
										2	990	10	"	.100	1.9		4.70	
										3	1100	10	"	.100	1.2		3.70	
										4	970	10	"	.090	1.75		4.24	
										5	1095	10	"	.035	0.75		1.05	
											1		"	.100	2.4		6.66	
										6	1005	10	"	.064	1.25		3.14	

INITIAL ANALYSIS
INLET SCRUBBER LIQUOR SAMPLES

OK

OK

INITIAL ANALYSIS
OUTLET SCRUBBER LIQUOR SAMPLES

Sample no.	DILUTION FACTORS	Absorbance	γ_1 mg NH ₃ /gal	Blank Corr. mg NH ₃ /gal	ppm mg/gal		
1 NH ₃ NH ₃ -D VFA	100 x 50 50 x 100 50 x 10 x 5	.253 .166 .570	73 49 166.5	0 .08 .13	7300 8167 29387	11/11/78 11/11/78 11/11/78	11/20
2	50 x 20 x 10 50 x 25 x 10	.128 .330	34 81	NC 0.01	6600 38830	11/11/78	
3	50 x 20 x 10 50 x 15 x 10	.132 .330	35 88	NC 0.01	7000 38830	11/11/78	
4	1 x 10 x 50 50 x 1 x 50 50 x 10 x 5	.200 .413 .558	59 121 163.5	0 0.1 0.125	5900 6056 28858	11/11/78	
5	50 x 20 x 10 50 x 15 x 10	.124 .231	33 79.5	NC 0.01	6600 35079	11/11/78	
6	50 x 20 x 10 50 x 15 x 10	.117 .307	31 81.5	NC 0.01	6200 35962	11/11/78	
NC - no blank corrected because no clearly blank							
xx old blank corrections are negligible							
Sample no.	F ₀	Abs.	γ_1 mg/gal	$\gamma_1 \cdot F_0 / 4$ ppm mg/gal			
1 Form.	10	.365	73	18.25	11/11/78		
2	10	.730	15.2	38.60	"		
3	10	.730	15.2	38.60	"		
4	10	.290	5.7	14.25	"		
5	10	.340	6.75	16.87	"		
6	10	.360	5.85	14.63	"		

Sample no.	DILUTION FACTORS	Absorbance	γ_1 mg NH ₃ /gal	Blank Corr. mg NH ₃ /gal	ppm mg/gal		
1 NH ₃ NH ₃ -D VFA	100 x 10 50 x 100 50 x 100 x 50	.2361 .501 .178	105.5 146.5 52	0 0.05 0.0025	2110 14650 458900	11/20/78 11/20/78 11/20/78	
2	50 x 1 x 10 50 x 50 x 50	.400 .370	106.5 98.5	NC 0.01	10650 474631	11/11/78	
3	50 x 1 x 10 50 x 50 x 50	.332 .426	87 113	NC 0.01	8700 491613	11/11/78	
4	1 x 100 x 10 50 x 1 x 50 50 x 75 x 10	.410 .778 .327	120 228 94	0 0.1 0.005	2400 11401 423600	11/11/78	*
5	50 x 1 x 100 50 x 50 x 50	.346 .411	92 109.5	NC 0.01	9200 483169	11/11/78	
6	50 x 1 x 100 50 x 50 x 50	.313 .387	83.5 103	NC 0.01	8350 454488	11/11/78	
M (reported with additional dilution)							
	50 x 2 x 50	.711	100	0.05	10000	USE average = 10700 ppm	
Sample no.	F ₀	Abs.	γ_1 mg/gal	$\gamma_1 \cdot F_0 / 4$ ppm mg/gal			
1 Form.	1	.1651	—	—	11/11/78		
2	1	.000	0.02	0.05	"		
3	1	.035	0.85	0.21	"		
4	1	.034	0.65	0.21	"		
5	1	.030	0.75	0.19	"		
6	1	.048	1.20	0.30	"		
7	1	.028	0.75	0.19	"		

APPENDIX D.2

CHEMICAL LABORATORY NOTEBOOK

TRC – THE RESEARCH CORPORATION OF NEW ENGLAND

Report of Chemical Analysis Non-Routine Samples

Client: EPA

Laboratory No: _____

Contract No: 82988-01 Africa

Date Received: 12-22-78

Reviewed by: _____

Date Reported: _____

Report to: UAW

Type Sample: Filter _____ Fuel Oil _____ Sediment _____ Impinger _____ Other Salinity 10.68

[illegible]

Analyzed by:

Report of Chemical Analysis: Non-Routine Samples

Laboratory No: _____

Date Received: 12-22-78

Date Reported: _____

Date Reported: _____

Type Sample: Filter _____ Fuel Oil _____ Sediment _____ Impinger _____ Other Normal Aldehyde
p 74 & 75

Analyzed by:

Project No. 82988-01

14

Book No. 82988-1 TITLE AGRICO - RESET

From Page No. —

Scraper Grab Sampler
"Legion"

12-18-78

Test I 2 samples collected @ Outlet + Inlet @ 15:10 } compared
15:50 } to Iran

To Page

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

From Page No. _____

12/19/78

Test 1 - Combined Impinger + PW.

- ① Total Volume 880 ml
 ② " " + ~100 ml of ^{TRC} H₂O to line filter
 Filter # 4027
 Final Volume 975 ml

Filter Inlet & Outlet Sample through Filter # 4028 to
Remove Particulate

Sample Preserved @ 1330 12/19/78

Test 2 - Combined Imp + PW

- ① Total Volume 910 ml
 ② " " + ~1.5 ml TRC H₂O Rinse
 Filter # 4029

12/19/78

$$= \frac{990}{\text{Final Vol}}$$

Preserved @ 18:50

Test 3 Combined Imp + PW

- ① Total Vol 995 ml
 ② " " + ~1.5 ml TRC H₂O Rinse = 1100 ml

Filter # 4030

Preserved @ 18:50

To Page

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

From Page No. —

Analysis of Samples

12/19/78

Sample - Aligned for NH_3 vol. of NH_3 distilled ml. of 4.00 Dist. Water

Dist Blank	500 ml. Dist. H_2O	500	200.
Test 1 - Stack	50 ml	500	200.
Test 1 Inlet grab	10 ml	500 ml	500 ml
Test 1 Outlet grab	10 ml	500 ml	500 ml
Test 2 Stack	50 ml	500	500 ml
Test 3 Stack	50 ml	500 ml	500 ml
Test 4 Stack	50 ml	500	500 ml
Test 5 Stack	50 ml	500	500
T 6	50	500	500
Blank 2	500 500 ml	500	500
Test 4 Inlet	10 ml	500	500
Test 4 Outlet	10 ml	500	500

after digestion, the sample was solid in Bottoming H_2O . I look

To Page No. —

Inessed & Understood by me,

Date

Invented by

Date

Recorded by

age No. _____

Mercurization continued

Sample	Kolby distillate	mls/mercurtube	Abs.	equivalent µg NH ₃	total mg (net uncorrected)
Batch I	as is	distillate 500 500	.359	1035 µg	405.2
NH ₃	500	2 mls	.301	87	431
Urea	200	500 5 mls	.164	48	66.1

Batch I	as is Agrico	50	9.992 = (-.048)	n.d.	
NH ₃	500	50	9.992 (-.018) .015	5 µg	.05 mg/500 ml
Urea	200	50	.179	52 µg	.347 mg/500 ml
	as is TRC	50 ml	(9.992) = (-.018)	n.d.	
Batch I	as is	distillate 500 500 (500 ml)	me in Mercur. 253	73	7300 µg/ml
"	NH ₃	500 ml 100 → 100	.166	49	8167 µg/ml

Today
Batch I as is 50 → 500 500 ml/mercur
" NH₃ 500 ml — 10 ml

Batch Reading after 45 minutes

Sample	Kolby distillate	add dilution	ml in 50.0 ml Mercurtube	Equivalent µg NH ₃	Blank correction	Results
Batch I NH ₃ as is		50 → 100	5.0	103.5	0	403.7 mg NH ₃
NH ₃ distilled	50 → 500		2.0	87	$5.0 \times \frac{2.0}{5.0} = 0.2$	423.2 mg NH ₃
Urea distilled	200		5.0	48	$5.0 \times \frac{5}{5.0} = 5.2$	60.4 mg Urea
						58.9
Batch as is Agrico			50.0	n.d.		EAP
NH ₃ distilled	500		↓	51.0		
Urea distilled	200		↓	52.0		
TRC H ₂ O as is			51.0	n.d.		
Batch I as is		50 → 500	1.0	73.	0	7.300 mg NH ₃ /ml
NH ₃ distilled	100 → 500	100 → 100	3.0	49.	$5.0 \times \frac{3}{5.0} = 0.3$	8.167 mg NH ₃ /ml

Batch I sample to 50 ml with TRC H₂O in Mercur Tube
Batch I sample to 50 ml with TRC H₂O

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

12/20/78

Sample Contention

Test 4

Total Volume - 870 ml

" " plus filter residue =

970 ml Final total Vol

Filter # 4031

Preserved 12:30 12/20/78

Test 5

Total Vol = 995 ml

" " + filter residue =

1095 ml Final total Vol

Filter # 4033

Preserved 12:30 12/20/78

Test 6

Total Vol 910 ml

" " + filter residue =

1005 ml Final Total Vol

Filter # 4034

Preserved 12/20/78

Test 1, Inlet & Outlet Samples

Preserved 12/20/78 12:30

Test 4 Inlet + outlet filter through Filter # 4035

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

Form Page No. _____

Analysis

12-20-78

Standard

total $\mu\text{g NH}_3$ / some $\mu\text{g urea}$	
0 Distilled	.000
20	.067
40	.133
100	.352
160	.546
200	.665

original vol. & aliquots
sample volumes? list ~~list~~ & attachurea apply $1.765 \times$ factor

Sample

Direct Sample

100	.352
200	.690

samples

	Vol of distilled	2nd dilution	mls / test tube	Abs	Abs	equivalent μg	
Test 1 - Dist 1	as is	5 \rightarrow 500 5 \rightarrow 100	5 ml OH	.361	105.5	211.6 μg	
NH_3	500	—	(5 ml high) 1 ml	.501	146.5	146.5 μg	
Urea	500	5 \rightarrow 500	(5 ml high) 1 ml	.178	52	45.9 μg	
Dist 1 - Urea	500	5 \rightarrow 100	10 ml OH	.570	166.5	293.8 μg	
Test 2 - Stack	as is	5 \rightarrow 100	5 ml	.285	84	333.1 μg	
NH_3	500	—	2 ml	.226	66.5	329.6 μg	
Urea	500	—	2.5 ml	.462	135	344.4 μg	
Test 3 - Stack	as is	5 \rightarrow 100	5 ml	.235	84	359.6 μg	
NH_3	500	—	2 ml	.367	108	541.8 μg	
Urea	500	—	2.5 ml	.168	49.5	38.4 μg	
Test 4 - Stack	as is	5 \rightarrow 100	5 ml	.319	93.5	362.8 μg	
NH_3	500	—	2	.244	72.0	349.2 μg	
Urea	500	—	2.5	.261	76.5	504.4 μg	
Test 5 - Stack	as is	5 \rightarrow 100	5 ml	.266	79.0	342.6 μg	
NH_3	500	—	2	.201	57.0	323.6 μg	
Urea	500	—	2.5	.145	43.0	330.6 μg	

To Page No. _____

Witnessed & Understood by me, _____

Date _____

Invented by _____

Date _____

Recorded by _____

From Page No. _____

Sample	Vol. Distilled	2nd Dilution	vol./Nessler Tube	Flas	equivalent μg	
Best 6 Star	as is	5 \rightarrow 100	5 ml	.255	75.0	302
NH ₃	500	-	2	.206	61.0	30
Urea	500	-	25	.248	172.5	51.4
Blank 2 NH ₃	500	-	50.0	.017	5.0	
Urea	500	-	50.0	.047-.042	12.5	
Grab Outlet 4	as is	5 \rightarrow 2500	1 ml	.200	59	
NH ₃	500	-	1 ml	.413	121	
Urea	500	5 \rightarrow 100	10 ml	.558	163.5	
Grab Outlet 4	as is	5 \rightarrow 2500	5 ml	.410	120	
NH ₃	500	-	(5 ml high) 1 ml	.778*	(228)	
Urea	500	2 \rightarrow 2500	5 ml	.327	96.0	
Grab Out 4 NH ₃ + Dilute 1:1				.341	100 μg	

Calculation

$$\text{NH}_3) \text{ equivalent } \mu\text{g NH}_3 \times \frac{\text{total Vol Sample}}{\text{aliquot/neutralizer}} \times \frac{\text{vol 2nd dil}}{\text{ml distilled}} \times \frac{\text{volume distilled}}{\text{aliquot of Sample}} \times \frac{\text{total}}{\text{vol}} = \text{mg NH}_3 \text{ total}$$

Urea calculation = above \times 1.765

Blank Correction Inlet, Outlet = Blank Calculation $\mu\text{g NH}_3$
 $\text{equivalent } \mu\text{g} \div \text{ml in Nessler tube} \times \frac{\text{volume}}{\text{aliquot of Sample}} \times \frac{\text{volume 2nd dil}}{\text{ml distilled}}$
 EAT

To Page No. 36

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

Initial analysis of 2 ml + distilled water sample

Original

250 ml distilled water

250 ml distilled water

Test 2 In 5 ml
out 1

Test 3 In 5 ml
out

Test 5 In
out

Test 6 In
out

Alone distilled NH_3 , then added digestion Soln, digested on 12/26/78 - TAC
Blank TRC 190

Sample filler

Test 2 filler = 4036

3-In 4037

3-out 4038

Test 5 5-In 4039
5-out 4040

Test 6 6-In 4041
6-out 4042

Before date 12/1
PG

From Page No.

1/27/79

Final Weight of filter which was used
to filter inlet & outlet scrubber water samples and stack samples
taken at Agrico 12/15 + 12/19/79

Filter #	final wgt	initial wgt	grams of particulate	Sample	volume	Stack Results total mg Particulate	mg/l Particulate
4027	0.63948	0.63740	.00208	St 1		2.08 ✓	
4028	0.63776	.63736	.00040	In + Out 1	~ 800ml		0.5
4029	0.55948 ✓	.55766	.00182	St 2		1.82	
4030	0.55458	.55478	-.00020	St 3		n.d	
4031	0.55592	.55574	.00018	St 4		0.18	
4032							
4033	0.55 ⁵⁰⁴ 445	.55554	-.00059	St 5		n.d	
4034	0.55728	.55615	.00113	St 6		1.13 ↓	
4035	0.55325	.55180	.00145	In + out St 4	~ 500ml		1.84
4036	0.55513	.55024	.00491	In + out St 2	500ml		6.14
4037	0.56234 ✓	.54992	.01242	In 3	400ml		31.0
4038	0.55112	.55396	-.00284	Out 3			n.d
4039	0.55692	.55750	-.00058	In 5			n.d
4040	0.54856 ✓	.55314	-.00458	Out 5			n.d
4041	0.64390	.64286	.00104	In 6			2.6
4042	0.65888 ✓	.64454	.01344	Out 6			de med report
4043			.01434				

* weight written wrong ^{would be} either .64544
or .64454
4042 particulate appears slight

To Page No

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

Type Filter

4 1/2"

Filter Number	Initial Weight (g)	Initials	Date Weigh
✓ 4014	.91613	EMS	12/11/11
✓ 4015	.91379		
✓ 4016	.91976		
✓ 4017	.91844		
4018	.92389		
✓ 4019	.91831		
4020	.91445		
4021	.92768		
4022	.92233		
✓ 4023	.93589		
4024	.91633		
✓ 4025	.92081		
4026	.93145		
✓ 4027	0.63740 ✓		
4028	0.63736		
4029	0.55766		
again 4030	0.55478		
12/11/11 4031	0.55574		
4032			
4033	0.555 ⁵⁴ 45		
4034	0.55615 ✓		
4035	0.55180		
4036	0.55024		

FILTER WEIGHTS

Type Filter

Filter Number	Initial Weight (g)	Initials	Date
4037	0.54992		
4038	0.55396		
4039	0.55750		
4040	0.55314		
4041	0.64286		
4042	0.64454		
4043	0.64328		
4044	-0.73230	Jmm	12-19-
4045	0.73195		
4046	0.72673		
4047	0.73745		
4048	0.73905		
4049	0.73876		
4050	0.73045	PAK	1-3
4051	0.73089		
4052	0.73830		
4053	0.74414		
4054	0.74259		
4055	0.73829		
4056	0.74395		

om Page No. —

Formaldehyde Analysis (Chromotropic Acid method)

analysis done by SW in Lab Book # IV Salkunzyrnicki p 51+52 1/10/79
and M. Renner in Lab Book 82988-20-4 p 20, 21, 22, 23

SW data

Standards 1000 µg/ml dilute 1/100 ⇒ 10 µg/ml working solution

mls Working Sol'n	total µg CH ₂ O	Abs
0.0	0	.00 - zeroed with DI H ₂ O
0.1	1	.052
0.3	3	.158
0.5	5	.26
0.7	7	.34
1.0	10	.49
2.0	20	.95
3.0	30	1.35

Samples	Dilution	aliquot	Abs	Equivalent µg CH ₂ O	Results µg/ml Formaldehyde
Smelt Scrubber					
1	1 → 10	4.0 ml	.365	9.3	18.2 ✓ OK June
2			.73	15.2	38.0
3			.73	15.2	38.0
4			.29	5.7	14.2
5			.34	6.75	16.9
6			.30	5.85	14.6

Stack Sample	Total Volume	Dilution	aliquot	Abs	Equivalent µg	Results total/mg
1	975 ml	1 → 10	4.0 ml	.084	1.6 µg	3.90
2	990 ml			.10	1.9	4.76
3	1100 ml			.06	1.2	3.30
4	970 ml			.09	1.75	4.24
5	1045 ml			.039	0.75	2.05
6	1005 ml			.064	1.25	3.14 ✓ OK June

Stack calculation total/mg CH₂O = $\frac{\text{equivalent } \mu\text{g}}{4 \text{ ml}} \times \frac{10}{1} \times \text{total volume ml} \times \frac{0.001 \text{ mg}}{1 \mu\text{g}}$

Formaldehyde analysis continued

data from Lab Book 82988-20, p 20 & 22

p 20 1/26/79

total $\mu\text{g CH}_2\text{O}$

abs p 20

abs p 22 1/29/79

0

0

0

1

.048

.068

3

.122

.170

5

.190

.255

7

.290

.348

10

.44

.480

20

.86

.880

p 20

Samples

aliquot

abs p 20

Equivalent μg

Result

$\mu\text{g}/\text{ml CH}_2\text{O}$

Outlet 1

4.0 ml

sample lost

repeat

repeat below

2

.035

0.85

0.21

3

.034

0.85

0.21

4

.030

0.75

0.169

5

.048

0.20

0.30 ✓ de mm

6

.028

0.75

0.19

Stack 5

4.0 ml

.100

2.4 (sample volume 1095 ml) = 0.66 total mg

p 22

Sample Outlet 1

4.0

.000

< .2 μg

< .05 $\mu\text{g}/\text{ml}$

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	

APPENDIX D.3

SCRUBBER LIQUOR SAMPLING TIME

SCRUBBER LIQUOR SAMPLING TIMES
AGRICHO CHEMICAL COMPANY, BLYTHEVILLE, ARKANSAS

<u>Date</u>	<u>Run</u>	<u>Sampling Time (CST)</u>	
		<u>First Sample</u>	<u>Second Sample</u>
12-18-78	1	1405	1445
12-19-78	2	0925	1000
12-19-78	3	1120	1148
12-19-78	4	1320	1400
12-19-78	5	1515	1548
12-19-78	6	1627	1700

APPENDIX E

TRC/AGRICO JOINT ANALYSES

Includes:

- E.1 Agrico Field Sample Analysis
- E.2 TRC Audit Sample Analysis
- E.3 Agrico Audit Sample Analysis

APPENDIX E.1

AGRICO FIELD SAMPLE ANALYSIS

12-19-78

Stack Sampling Analysis

Outlet 0-1

Test #1

$$\text{NH}_3 (7.3 - .3) \cdot 0.2 \times 14000 = 392.0 \text{ ppm}$$

Volume 975

$$392 \text{ ppm} \times 975 = 382.2 \text{ mg / total as NH}_3\text{-N}$$

$$\text{Total } (9.0 - .5) \cdot 0.2 \times 14000 = 476.0 \text{ ppm}$$

Total 476.0

NH₃ 392.0

pH.

$$\text{Org } 84.0 \text{ ppm} \times .975 = 81.9 \text{ mg / total as Org-N}_2$$

Outlet 2 0-2

$$\text{NH}_3 (7.5 - .3) \cdot 0.2 \times 14000 = 403.2 \text{ ppm}$$

Test #2

Volume 990

$$403.2 \times .990 = 399.2 \text{ mg / total as NH}_3\text{-N}_2$$

$$\text{Total } (7.8 - .5) \cdot 0.2 \times 14000 = 405.5 \text{ ppm}$$

Total 408.9

NH₃ 403.2

$$\text{Org } 5.6 \text{ ppm} \times .99 = 5.5 \text{ mg / total as NH}_3\text{-N}_2$$

12-19-78

Stack Sampling Analysis

Outlet 3 0-3

$$\text{NH}_3 (5.4 - .3) \cdot 0.2 \times 1000 = 285.6 \text{ ppm}$$

Test # 3

$$\text{volume } 1100 \text{ cc Total } (5.8 - .5) \cdot 0.2 \times 1000 = 296.8 \text{ ppm}$$

Total 296.8

$$285.6 \times 1.1 = \frac{314.2}{3/4 \text{ mg/total as NH}_3\text{-N}_2$$

NH₃ 285.6

$$\text{Org } 11.2 \text{ ppm} \times 1.1 = \frac{12.3 \text{ EAP}}{10.1 \text{ mg/total as Org-N}_2$$

Outlet 4 0-4

Test #4

$$\text{NH}_3 (11.5 - .3) \cdot 0.2 \times 1000 = 313.6 \text{ ppm}$$

volume 970 cc

$$313.6 \times .97 = 304.2 \text{ mg/total as NH}_3\text{-N}_2$$

$$\text{Total } (13.5 - .5) \cdot 0.2 \times 1000 = 364.0 \text{ ppm}$$

Total 364.0

NH₃ 313.6

$$\text{Org } 50.4 \text{ ppm} \times .97 = 48.9 \text{ mg/total as Org-N}_2$$

Outlet 5 0.5 NH_3 (9.8 - .3) $\frac{.02 \times 1000}{10} = 266.0$ ppm

Test #5

volume 1095 mls $266 \times 1.095 = 291.3$ mg / total as $\text{NH}_3\text{-N}_2$

Total (10.3 - .5) $\frac{.02 \times 1000}{10} = 274.4$ ppm

~~274.4~~

Total 274.4

NH_3 266.0

Oxy 8.4 ppm $\times 1.095 = 9.2$ mg / total as Oxy-N₂

Outlet 6 0.6 NH_3 (9.1 - .3) $\frac{.02 \times 1000}{10} = 246.4$ ppm

Test #6

volume 1005 mls $246.4 \times 1.005 = 247.6$ mg / total as $\text{NH}_3\text{-N}_2$

Total (10.3 - .5) $\frac{.02 \times 1000}{10} = 274.4$ ppm

Total 274.4

NH_3 246.4

Oxy 38.0 ppm $\times 1.005 = 38.1$ mg / total as Oxy-N₂

re required,

solve 134 g
ia-free dis-
ne H₂SO₄.
m prepared
curic oxide.
Dilute the
ep at a tem-
-ent crystal-

tor solution.
um thiosul-
NaOH and
monia-free
11.
See Section

ume: Place
00-ml kjel-
sample size
v:

ple Size
ml
90
50
90
50.0
25.0

e to 300 ml

Add 25 ml
4 until pH
ss beads or
0 ml. If de-
l determine
natively, if
nd by the
residue in

the distilling flask for the organic nitro-
gen determination. For sludge and sedi-
ment samples weigh wet sample in a
crucible or weighing bottle, transfer the
contents to a kjeldahl flask, and deter-
mine total kjeldahl nitrogen. Follow a
similar procedure for ammonia nitrogen
determination and organic nitrogen de-
termined by difference. Determinations
of organic and total kjeldahl nitrogen on
dried sludge and sediment samples are
not accurate because drying results in
loss of ammonium salts.

c. Digestion: Cool and add carefully
50 ml digestion reagent (or substitute 10
ml conc H₂SO₄, 6.7 g K₂SO₄, and 1.5
ml mercuric sulfate solution). If large
quantities of nitrogen-free organic mat-
ter are present, add an additional 50 ml
digestion reagent for each gram of solid
matter in the sample. After mixing, heat
under a hood or with suitable ejection
equipment to fumes of SO₃ and con-
tinue to boil briskly until the solution
clears (becomes colorless or a pale straw
color). Then digest for an additional 30
min. Let flask and contents cool, dilute
to 300 ml with ammonia-free water,
and add 0.5 ml phenolphthalein in-
dicator solution and mix. Tilt the flask
and carefully add sufficient (approx-
imately 50 ml/50 ml digestion reagent
used) hydroxide-thiosulfate reagent to
form an alkaline layer at the bottom of
the flask.

Connect the flask to the steamed-out
distillation apparatus and shake the flask
to insure complete mixing. Add more
hydroxide-thiosulfate reagent in the pre-
scribed manner if a red phenolphthalein
color fails to appear at this stage.

d. Distillation: Distill and collect 200
ml distillate below the surface of 50 ml
boric acid solution. Use plain boric acid
solution when the ammonia is to be de-

termined by nesslerization and use in-
dicating boric acid for a titrimetric fin-
ish. Extend the tip of the condenser well
below the level of boric acid solution and
do not allow the temperature in the con-
denser to rise above 29 C. Lower the
collected distillate free of contact with
the delivery tube and continue dis-
tillation during the last minute or two to
cleanse the condenser.

e. Final ammonia measurement: De-
termine the ammonia by either nessleri-
zation or titration.

1) Nesslerization—Mix the distillate
thoroughly and measure a 50.0-ml por-
tion or less. Complete the determination
as described in Nitrogen (Ammonia),
Section 418B.4b-e.

2) Titration—Titrate the ammonia
in the distillate as described in Nitrogen
(Ammonia), Section 418D.4c.

f. Blank: Carry a blank through all
the steps of the procedure and apply the
necessary correction to the results.

5. Calculation

a. Nesslerization finish:

$$\text{mg/l organic N} = \frac{A \times 1,000}{\text{ml sample}} \times \frac{B}{C}$$

where A = mg N found colorimetrically,
B = ml total distillate collected including
the H₃BO₃, and C = ml distillate taken
for nesslerization.

b. Titrimetric finish:

$$\text{mg/l organic N} = \frac{(D-E) \times 280}{\text{ml sample}}$$

where D = ml H₂SO₄ titration for
sample and E = ml H₂SO₄ titration for
blank.

6. Precision and Accuracy

Three synthetic unknown samples
containing varying organic nitrogen
concentrations and other constituents

$$* \text{Agrico calc } \text{ppm N} = \frac{(D-E) \times 0.02 \times 14,000}{\text{ml sample}}$$

TABLE 421-1. PRECISION AND ACCURACY DATA FOR ORGANIC NITROGEN

Sample	No. of Labora- tories	Organic Nitrogen Concen- tration μg/l	Relative Standard Deviation			Relative Error		
			Nessler Finish %	Titri- metric Finish %	Calculation of Total Kjeldahl N Minus Ammonia N %	Nessler Finish %	Titri- metric Finish %	Calculation of Total Kjeldahl N Minus Ammonia N %
1	26	200	94.8			55.0		
	29			104.4			70.0	
	15				68.8			70.0
2	26	800	52.1			12.5		
	31			44.8			3.7	
	16				52.6			8.7
3	26	1,500	43.1			9.3		
	30			54.7			22.6	
	16				45.9			4.0

APPENDIX E.2

TRC AUDIT SAMPLE ANALYSIS

TRC - THE RESEARCH CORPORATION OF NEW ENGLAND

Report of Chemical Analysis: Non-Routine Samples

Client: EPA

Laboratory No: _____

Contract No: 8 2988-01 agucoDate Received: 12-22-78

Reviewed by: _____

Date Reported: _____

Report to: UAWType Sample: Filter _____ Fuel Oil _____ Sediment _____ Impinger _____ Other Urea Audit Sample

TRC Analysts Sample Number	Location	Analysis No. 1	Analysis No. 2	Analysis No. 3	Analysis No. 4	Analysis No. 6	TLV
<u>Audit Set I</u>		<u>actual</u>	<u>total</u>	<u>total</u>	<u>% error</u>		
		<u>total mg/lua</u>	<u>mg lrea</u>	<u>mg lrea</u>			
			<u>not Blk & control</u>	<u>Blk & control</u>			
<u>1</u>		<u>100.71</u>	<u>98.84</u>	<u>94.9</u>	<u>5.77 %</u>		
<u>2</u>		<u>311.48</u>	<u>305.9</u>	<u>288.9</u>	<u>7.40 %</u>		
<u>3</u>		<u>548.36</u>	<u>573.3</u>	<u>568.8</u>	<u>4.94 %</u>		
<u>4</u>		<u>5.64</u>	<u>6.6</u>	<u>5.44</u>	<u>3.55</u>		
<u>5</u>		<u>11.60</u>	<u>12.36</u>	<u>11.20</u>	<u>3.45</u>		
<u>6</u>		<u>40.40</u>	<u>43.5</u>	<u>38.70</u>	<u>4.21</u>		
<u>7</u>		<u>2.60</u>	<u>3.18</u>	<u>2.43</u>	<u>6.54</u>		
<u>8</u>		<u>6.84</u>	<u>7.24</u>	<u>6.49</u>	<u>5.12</u>		
<u>9</u>		<u>9.42</u>	<u>9.71</u>	<u>8.96</u>	<u>4.88</u>		
<u>10</u>		<u>5.40</u>	<u>5.65</u>	<u>4.90</u>	<u>9.26</u>		
<u>11</u>		<u>4.30</u>	<u>4.68</u>	<u>3.93</u>	<u>8.60</u>		
<u>12</u>		<u>30.16</u>	<u>28.68</u>	<u>27.9</u>	<u>7.49</u>		
					<u>mean = 5.93</u>		

S. Dev = 1.92

Variance = 3.3

Analyzed by: mm. Jop

Form 0012

From Page No. Unal Audit Sample Prep.

12/18/78

1. Steam out distillation with Borate Buffer

2. Samples 1-6 Dilute with 400 ml H_2O 9:30 Dilute I-1 2 3 & 4, 5, 6 to 400 ml H_2O (TRC H_2O)

Sample Aliquot Digested distilled ml

I-1 25 ml

500 ml

I-2 25 ml

I-3 25 ml

I-4 100 ml

I-5 100

I-6 25

Samples I-7 to I-12 dilute with 250 ml 10% H_2SO_4

I-7 100

500 ml

I-8 100

Audit Blank 100 ml 10% H_2SO_4

I-9 100 ml

10 100 ml

* 11 100

* 12 100

1, 2 & 3 add 350 ml Agric. H_2O for distillation, dilute to 500 with Agric. H_2O

I-12 & BL 450 " " "

* may have lost some during etch of distillation

* Distillation here not under surveillance at start of test

To Page 1

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

From Page No. _____

Standard - Stock 3.141 g NH_4Cl / l TPCH_2O = 1mg/ml NH_4Cl
 Dilute 10ml \rightarrow 100 ml

Standards

0 $\mu\text{g}/50$	total $\mu\text{g}/500$	ml of 100 $\mu\text{g}/\text{ml}$ Schick
20	200	2 \pm 50ml
40	400	4
100	1000	10
160	1600	16
200	2000	20

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

From Page No. —

Reorganization of Audit Samples12-15-78

<u>Std</u>	<u>Graph I</u>
<u># Distilled</u>	<u>Abs</u>
0 0 <u>0</u> <u>µg/50ml</u>	.000
20	.060
40	.150
100	.346
160	.559
200	.690 <u>End</u> <u>1.072</u>

<u>Direct</u>	<u>1mg/lr. NH₃ dil to 20µg/lr</u>	<u>Abs</u>
0 <u>µg NH₃/50ml</u>	<u>ml 20µg/lr NH₃</u>	<u>2000-1000</u>
20	2 1	.049
40	4 2	.117
100	10 5	.304
160	16 8	.507
200	20 10	.622

<u>Audit Sample</u>	<u>Val Distilled</u>	<u>Aligned in Multi-tube</u>	<u>Abs</u>	<u>equivalent</u> <u>µg NH₃ Urea</u>	<u>not Blend</u> <u>corrected</u> <u>Total mg</u>
5:15 1	500ml	10	.241	70µg	98.84
2	del. to 10-100	50ml	.361	104µg	305.4 98.84
3	5-100	50ml	.352	101.5	305.4 293.5
4		50	.326	94	573.3 / 10.5
5		50	.608	175	6.6
6		50	.535	154	12.36 ✓ ok 2-
7		50	.247	72	43.5
8		25	.282	82	3.18
23 9		25 15	.226	66	7.24
10		25	.220	64	9.71
11		25	.181	53	5.65 ✓ ok 10
12		25 (high) 5ml	.225	65	4.68
23 Blank		50	.055	17	28.68

Continued p15
total mg Urea

See p19 for Blank corrected data

300 Urea
100µg for 50ml
distilled
for 50ml
in Multi-tube

To Page No. 5

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

TITLE AGRICO - RESET

From Page No. _____

Notes:

12-19-78 - in October Agrico Analyzed Inlet & Outlet
Grab samples by Urease Method

D.B. Began Analyzing Part I Grab Inlet &
Outlet, but these are too high for
the titration method. That
he is using on the Slack Samples
Part II did not call for him to
analyze these Grab Samples

Audit samples		Blanks corrected		from p 17		1/15/79		Blank correction
Sample	2nd dilution	aliquot in μ l	equivalent μ g	Blank correction	Sample Vol	ml digested	total mg urea	
1		10.0 ml	70	-3.4 μ g	400 ml	25.0	94.6	✓
2	10 → 100	50.0	104	-1.7			288.9	✓
3	5 → 100		101.5	-0.8			568.8	
4			94	-17		100	5.44	
5			175	-17		100	11.2	
6			154	-17		25	38.7	
7			72	-17	250 ml	100	2.43	
8		25	82	-8.5 ✓			6.49	
9		15	66	-5.1			8.96	
10		25	64	-8.5			4.90	
11		25	53	-8.5			3.93	
12		5	65	-1.7			27.9	
Blank		50	17					

$$* \text{Blank correction} = \frac{17 \mu\text{g}}{50 \text{ ml}} \times \text{aliquot in merlon tube} \times \frac{\text{aliquot diluted}}{\text{vol of 2nd dilution}}$$

$$\text{total mg urea} = \left(\frac{\text{Equivalent } \mu\text{g NH}_3 - \text{Blank correction}}{\text{aliquot in merlon tube}} \right) \times \frac{\text{Volume of distillate } 500 \text{ ml}}{\text{aliquot digested}} \times \frac{\text{total Volume of sample in ml}}{\text{ml}} \times 1.765 \times .001 \mu\text{g}$$

To Page No. _____

Witnessed & Understood by me, _____

Date _____

Invented by _____

Date _____

Recorded by _____

TITLE AGRICO - RESET

Book No. 82938-1

from Page No. 17+19

Audit Samples I Comparison

	Actual mg	mg Blank Corrected mg Analyzer Urea	mg Urea Blank Corrected from p 19 Retained Error
1	100.71	98.84	94.9 94.0 EAP
2	311.93	305.9	288.9
3	548.36	573.3	568.8
4	5.64	6.6	5.44
5	11.60	12.36	11.20
6	40.40	43.5	38.7
7	2.60	3.18	2.43
8	6.84	7.24	6.49
9	9.42	9.71	8.96
10	5.40	5.65	4.90
11	4.30	4.68	3.93
12	30.16	28.68	27.9
Blank	0.00		

To Page No. _

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

APPENDIX E.3

AGRICO AUDIT SAMPLE ANALYSIS

TRC - THE RESEARCH CORPORATION OF NEW ENGLAND
Report of Chemical Analysis: Non-Routine Samples

Client: EPA

Laboratory No: _____

Contract No: 82988-01 Agrico

Date Received: 12-22-77

Reviewed by: _____

Date Reported: _____

Report to: WPH

Type Sample: Filter _____ Fuel Oil _____ Sediment _____ Impinger _____ Other Urea Audit Sample

<u>Agrico Analyst</u> Sample Number	Location	Analysis No. 1	Analysis No. 2	Analysis No. 3	Analysis No. 4	Analysis No. 6	TLV
<u>Audit Lot II</u>		<u>actual total</u> <u>mg Urea</u>	<u>mg</u> <u>Total Nitrogen</u>	<u>mg</u> <u>Urea</u>	<u>% error</u> <u>in analysis</u>		
1		100.54	96.3	206	104.89 %		
2		292.78	281.1	602	105.42 %		
3		598.08	582.4	1246	108.33 %		
4		5.26	3.6	7.7	46.39 %		
5		9.64	11.8	25.2	161.41 %		
6		42.48	38.6	82.6	94.44 %		
7		2.04	1.1	2.35	15.20 %		
8		6.16	5.0	10.7	73.70 %		
9		9.54	9.5	20.3	112.79 %		
10		5.96	5.3	11.3	89.60 %		
11		4.18	3.9	8.3	98.56 %		
12		31.32	27.4	58.6	87.10 %		
				mean = 91.50			

S. Dev = 36.00
Variance = 1188

Analyzed by: D. J. L. Bland

Form CL-0012

Jan 4, 1979

Radix Samples

II-1 — $(10.3 - 1.7) \cdot 0.22 \times 1000 = 96.3 \text{ ppm}$ Total Nitrogen

$\frac{60}{14}$

II-2 — $(26.8 - 1.7) \cdot 0.22 \times 1000 = 281.1 \text{ ppm}$

II-3 — $(22.5 - 1.7) \cdot 0.22 \times 1000 = 580.4 \text{ ppm}$

II-4 — $(3.0 - 1.7) \cdot 0.22 \times 1000 = 31.6 \text{ ppm}$

II-5 — $(5.9 - 1.7) \cdot 0.22 \times 1000 = 118 \text{ ppm}$

II-6 — $(9.6 - 1.7) \cdot 0.22 \times 1000 = 386 \text{ ppm}$

Conversation with Daryl Baird 4-11-79

The samples were diluted to 1 liter. $\therefore \text{ppm} = \text{mg/l}$ result = mg

Result as ppm Total Nitrogen

To convert to Urea - multiply by $\frac{\text{MW urea}}{\text{wgt N/urea}} = \frac{60}{28}$

Audit Samples

Jan 5, 1979

#-7 - (2.1 - 1.7) $\frac{.02 \times 14000}{100}$ 1.1 ppm

#-8 - (3.5 - 1.7) $\frac{.02 \times 14000}{100}$ 5.0 ppm

#-9 - (5.1 - 1.7) $\frac{.02 \times 14000}{100}$ 9.5 ppm

#-10 - (3.6 - 1.7) $\frac{.02 \times 14000}{100}$ 5.3 ppm

#-11 - (3.1 - 1.7) $\frac{.02 \times 14000}{100}$ 3.9 ppm

#-12 - (6.6 - 1.7) $\frac{.02 \times 14000}{50}$ 27.4 ppm

APPENDIX F

SAMPLING TRAIN CALIBRATION DATA

⑤

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

Actual in 3 Places	Average	Date/Inits	Actual in 3 Places	Average	Date/Inits
.1869-.1870-.1871	.1870	11/30/72 JH			
			Vm = 34.42		
			ΔHAN = 1.019		
			TMAV = 60.125		
			TS AV = 101.58		
			√ ΔP AV = .9199		
			646.9		
			.962		
			28.41 = (37)(18) + (21)		
			(+037) (32) + (79)(.962)(28)		
			100		
			101.58 + 960		
			28.41 (29.21 + .395)		
			.843.23		
			111.5		
			= .0944		
			√ [401.58 + 960] X 39.73		
			(29.68) (32) (.00018) (60) (+ .762)		

☐

□

☒

11

INSPECTOR:

ELFRID MACHINE COMPANY
105 JORDAN LANE
WETHERSFIELD, CONN. 06109
NOV 50 ENT'D

①

DATE 11/29/77

INSPECTION REPORT

ELFRED MACHINE COMPANY

CUSTOMER TRC-THE RESEARCH CORP. CUSTOMER P.O. NO. 9964
 S.O. NUMBER — PURCHASED FROM ELFRED P.O. NO. —
 PART NO. 1-2 NO. PIECES ORDERED 1 NO. PIECES RECEIVED 1
 TOOL NO. SET #1 PART NAME ST/STL NOZZLE
 TOOL NAME —

Actual in 3 Places	Average	Date/Initials	Actual in 3 Places	Average	Date/Initials
2547-2551-2552	2550	11/30/77 JMS	$A_n = .00018$		
2570-2576-2591	2576	1/13/78 H	$\Delta H_{AV} = .95$		
2590-2590-2590	2590	7/3/78 JMS	$T_s + 460 = 563.25$		
2574-2574-2575	2574	10/26/74 JMS	$P_{bar} + \frac{1}{12.6} = 29.64$		
			$V_m = 33.61$		
			$T_m = 67.04 \rightarrow 527.04 K$		
			$\sqrt{\Delta P_{AV}} = .873$		
			$V_{m@STP} = 33.48 = 17.65(33.61)$		
			$\%H_2O = 4.8$		
			$(1-.098) = .952$		
			$M_s 28.32 = \frac{(21)(.952)(32) + (79)(.952)(28) + (4.8)(18)}{100}$		
			$V_s 57.16 = \frac{85.48(.839)(.973)}{(58.25)}$		
			$\sqrt{563.25}$		
			$\sqrt{28.32 \times 29.64}$		
			$98.5 = .0944 \sqrt{563.25 \times 33.48}$		
			$(29.64)(57.16)(.00018)(60)(.952)$		
			(100.28)		

1st PIECE INSPECTION ☐
 PARTIAL INSPECTION ☐
 COMPLETE INSPECTION ☒

ACCEPTED ☒
 REJECTED ☐

INSPECTOR [Signature]
 WETHERSFIELD, CONN. 06182

ELFRED MACHINE COMPANY
 100 JORDAN LANE
 WETHERSFIELD, CONN. 06182
 NOV 30 1977

DATE 11/29/77

INSPECTION REPORT

ELFRED MACHINE COMPANY

CUSTOMER: TRC-THE RESEARCH CORP. CUSTOMER P.O. NO. 9964

S.O. NUMBER _____ PURCHASED FROM ELFREDA P.O. NO. _____

PART NO. 1-3 NO. PIECES ORDERED 1 NO. PIECES RECEIVED 1

PART NAME ST / STL / NOZZLE

TOOL NO. SET #1 TOOL NAME _____

[illegible]

1st PIECE INSPECTION

PARTIAL INSPECTION

COMPLETE INSPECTION

ACCEPTED

REJECTED

INSPECTOR *[Signature]* FILED 165 W. WETHERSFIELD

PRED MACHINE COMPANY
 165 JORDAN LANE
 WETHERSFIELD, CONN. 06109
 MOV 30 ENT3

DATE 11/29/77

INSPECTION REPORT

ELFRED MACHINE COMPANY

CUSTOMER TRC-THE RESEARCH CORP.

CUSTOMER P.O. NO. 9964

S.O. NUMBER

PURCHASED FROM ELFRED P.O. NO.

PART NO. 1-4

NO. PIECES ORDERED 1 NO. PIECES RECEIVED 1

TOOL NO. SET #1

PART NAME ST/STL NOZZLE

TOOL NAME _____

[illegible]

1st PIECE INSPECTION

☐

PARTIAL INSPECTION

☐

ACCEPTED

☒

COMPLETE INSPECTION

☒

REJECTED

INSPECTOR

TO: **ELATED MACHINE COMPANY.**
165 JORDAN LANE
WETHERSFIELD, CONN. 06109
NOV 30 ENT'D

DATE 11/29/77

INSPECTION REPORT

ELFRED MACHINE COMPANY

CUSTOMER TRC - THE RESEARCH CORP. CUSTOMER P.O. NO. 9964
S.O. NUMBER — PURCHASED FROM ELFRIED P.O. NO. —
PART NO. 1-5 NO. PIECES ORDERED 1 NO. PIECES RECEIVED 1
TOOL NO. SET #1 PART NAME ST/STL NOZZLE
TOOL NAME —

[illegible]

1st PIECE INSPECTION ☐

PARTIAL INSPECTION ☐

COMPLETE INSPECTION ☒

ACCEPTED

REJECTED

INSPECTOR

ALFRED MACHINE COMPANY
165 JORDAN LANE
WETHERSFIELD, CONN. 06109

NOV 30 1961

DATE 12/24/77

INSPECTION REPORT

ELFRED MACHINE COMPANY

CUSTOMER TRC - THE RESEARCH CORP

CUSTOMER P.O. NO. 1547

S.O. NUMBER _____

PURCHASED FROM ELFRED P.O. NO. _____

PART NO. 1-6 (1/8)

NO. PIECES ORDERED 1 NO. PIECES RECEIVED 1

TOOL NO. _____

PART NAME ST/STL NOZZLE

TOOL NAME _____

[illegible]1st PIECE INSPECTION ☐PARTIAL INSPECTION ☐

COMPLETE INSPECTION ☒

ACCEPTED

REJECTED

INSPECTOR

ELFRED MACHINE COMPANY
105 JORDAN LANE
WEATHERFIELD, CONN. 06108
DEC 29 ENTG

DATE 12/11/78

ORIFICE NO D3 NAME SRICHARDSON

BAROMETRIC PRESSURE 30.29 IN HG DRY GAS METER NO 3

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 OUTLET AVERAGE F F F F	TIME MIN	RATIO	DHO
----------------------------------	----------------------------	---------------------------	------------------------	--	-------------	-------	-----

0.5	5.0	4.97	72.5	69.0	67.0	68.0	12.5	1.00	1.76
1.0	5.0	4.99	72.5	68.0	66.0	67.0	9.0	0.99	1.84
2.0	10.0	9.97	72.5	69.0	67.0	68.0	12.9	0.99	1.88
3.0	10.0	9.98	72.4	70.0	68.0	69.0	10.6	0.99	1.88

AVERAGE 0.99 1.84

```

*****
*
*       THE RESEARCH CORPORATION OF NEW ENGLAND
*
*       CLIENT  EPA
*       CHARGE NO. 2988       TESTER TIRONE
*
*****
*
*       CALIBRATION DATA
*       TYPE S PILOT TUBE
*
*       DATE 12/11/78       TEMP.(F)= 55.0       BARO.PRES.(IN.HG)=30.52
*
*       STANDARD PITOT NO. CE-1       SERIAL NO. 44
*       *****
*
*       NO. OF SCREENS      STANDARD      TYPE S      COEFFICIENT
*                          DELTA P °° W.C.  DELTA P °° W.C.  +CP(S)
*
*       1                   0.100         0.140         0.837
*       2                   0.200         0.280         0.837
*       3                   0.300         0.420         0.837
*       4                   0.400         0.460         0.923
*       5                   0.500         0.695         0.840
*
*                               AVERAGE CP(S) 0.855
*
*       REVERSE (S) PITOT AND RERUN TEST
*
*       1                   0.100         0.140         0.837
*       2                   0.200         0.280         0.837
*       3                   0.300         0.410         0.847
*       4                   0.400         0.450         0.933
*       5                   0.500         0.680         0.849
*
*                               AVERAGE CP(S) 0.861
*
* -----
* * CP(S)=0.99 SQRT(DELTA P(STANDARD) / DELTA P(S))
*
*****

```

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*****
*
*   THE RESEARCH CORPORATION OF NEW ENGLAND
*
*   CLIENT   EPA
*   CHARGE NO. 2988      TESTER TIRONE
*
*****
*
*   CALIBRATION DATA
*   TYPE S PILOT TUBE
*
*   DATE 12/11/78      TEMP.(F)= 55.0      BARO.PRES.(IN.HG)=30.52
*
*   STANDARD PITOT NO. CE-1      SERIAL NO. 37
*   *****
*
*   NO. OF SCREENS      STANDARD      TYPE S      COEFFICIENT
*   DELTA P ** W.C.      DELTA P ** W.C.      * CP(S)
*
*   1      0.100      0.139      0.840
*   2      0.200      0.280      0.837
*   3      0.300      0.420      0.837
*   4      0.400      0.560      0.837
*   5      0.500      0.690      0.843
*
*   AVERAGE CP(S) 0.839
*
*   REVERSE (S) PITOT AND RERUN TEST
*
*   1      0.100      0.135      0.852
*   2      0.200      0.270      0.852
*   3      0.300      0.400      0.857
*   4      0.400      0.530      0.860
*   5      0.500      0.655      0.865
*
*   AVERAGE CP(S) 0.857
*
*   -----
*   * CP(S)=0.99 SQR(T(DELTA P(STANDARD) / DELTA P(S)))
*
*****

```

APPENDIX G

AGRICO PROCESS OPERATIONS LOG

LIST OF PARAMETERS RECORDED DURING TESTING

TK-101	- Urea Solution Tank Level
AFR	- Additive Feed Rate
UMT	- Urea Melt Temperature, °F (confidential)
GSP-C	- "C" Granulator Urea Spray Nozzle Pressure, psig
AGT	- Temperature of "C" Granulator Inlet Air, °F (confidential)
AOGT	- Temperature of "C" Granulator Outlet Air, °F (confidential)
SLL	- "C" Granulator Scrubber Liquor Level
SFA	- "C" Granulator Scrubber Exhauster Fan Amps
SOWTC	- Weigh-belt totalizer for "C" Granulator Outlet Urea
PWTC	- Weigh-belt totalizer for "C" Granulator Product Urea
SLT	- Granulator Scrubber Liquor Temperature, °F
ISLF	- "C" Granulator Scrubber Liquor Feed Rate, gpm
AOS	- Temperature of "C" Granulator Scrubber Exit Air, °F
NH ₃ Feed	- Feed Rate of NH ₃ to Urea Synthesis Process

SUMMARY OF PROCESS AND CONTROL EQUIPMENT PARAMETERS

Parameter	Symbol	Units	12/18/78 1:55p-4:10p				12/19/78 9:05a-5:20p			
			Mean Value	Standard Deviation	Minimum Value	Maximum Value	Mean Value	Standard Deviation	Minimum Value	Maximum Value
Urea Solution Tank Level	TK-101	*	15.5	0.15	15.0	15.5	16.9	1.59	15.0	20.5
Additive Feed Rate	AFR	*	2.8	0.26	2.4	3.1	3.0	0.32	2.3	3.4
Urea Melt Temperature	UMT	°F	(-2)†	-	(-6)†	(+1)†	(+0.6)†	-	(-4)†	(+5)‡
Spray Nozzle Pressure	GSPC	psig	35.2	1.21	33	37	33.6	1.35	31.5	36
Granulator Inlet Air Temp.	AICT	°F	(+0.5)†	-	(0)†	(+1)†	(+11.5)†	-	(+8.5)†	(+1.6)‡
Granulator Outlet Air Temp.	AOGT	°F	(-12.4)†	-	(-18)†	(0)†	(-14.5)†	-	(-28)‡	(+3)‡
Scrubber Liquor Level	SLL	*	40.5	2.88	35	43	38.2	1.84	33	40.5
Scrubber Fan Amps	SFA	amps	68.9	0.54	68	70	69.0	0.84	68	70
Scrubber Liquor Temperature	SLT	°F	86.7	0.46	86	87	95.2	1.10	93	96
Scrubber Liquor Feed Rate	ISLF	*	‡	‡	‡	‡	‡	‡	‡	‡
Scrubber Outlet Temp	AOS	°F	83.6	1.20	80	84	92.3	1.30	90	94
Ammonia Feed Rate	NH ₃ Feed	*	8.47	0.115	8.4	8.8	8.65	0.136	8.45	8.9

* Uncalibrated readings, used as check for steady conditions.

† Confidential readings, values listed represent the difference from an arbitrarily chosen confidential base figure.

‡ Readings inaccurate or monitoring device broken during test period.

Sample Calculations

Correction factors for "B" and "C" Granulators

The method for determining the correction factor for these two Granulators was based on the assumption that the correction factor for "A" Granulator was correct for all test periods during 9 October through 13 October 1978. By assuming that the correction factor is correct, we have also accepted the assumptions made in calculating that correction factor; most notably that there is no significant difference between the spray nozzles in the three granulators nor in the melt passing through those nozzles. Furthermore, it is assumed that flow through a spray nozzle is proportional to the square root of the pressure drop across that nozzle. Based on these assumptions, the production rate for a single Granulator can be determined by multiplying the total production rate by the square root fraction, (SRF) where:

$$SRF = \frac{\sqrt{\Delta P_x}}{\sqrt{\Delta P_a} + \sqrt{\Delta P_b} + \sqrt{\Delta P_c}} \quad (A-1)$$

ΔP = pressure drop across nozzle

A,B,C (subscripts) = refer to Granulators "A", "B", and "C"

χ (subscript) = refers to Granulator of interest, "A", "B",
or "C"

Assuming that the correction factor for the "A" Granulator is correct, the total production for a given run can be calculated by multiplying the production rate for "A" based on corrected totalizer readings by the inverse of the SRF for "A" for that run, ($\chi = A$ in Equation A-1). This total production rate is then multiplied by the SRF for "B" and the SRF for "C" to get the actual production rates for those Granulators during this run. These rates are then divided by the production rates based on the uncorrected totalizer readings to yield correction factors for "B" and "C" Granulators. Correction factors, formulated by this technique, were used to calculate the production rates for "B" and "C" Granulators from totalizer readings in Table 2.

Copy of Raw Data Recorded
During Emission Tests

TIME	TEMP	AFK	WIND	CORR	ALT	FLT	WELL	SEA	WIND	WTC	WLT	WLF	WLS	WHS FEED
1/35	15.5	3.0	0	34	0	0	43	68	1117464	2771	27	21 ¹⁴ / ₁₄	84	8.8
2/15	15.5	3.1	-1	35	0	-5	42.5	70	1117692	2834	27	20 ¹⁴ / ₁₄	84	8.5
2/30	15.5	3.0	-1	35	0	-10	42	69	1117850	2850	26	20 ¹⁴ / ₁₄	84	8.4
3/15	15.5	2.5	-6	37	0	-10	42.0	69	1118044	2834	27	20 ¹⁴ / ₁₄	80	8.4
3/30	15.5	2.5	+1	35	0	-15	41.5	68	1118231	2815	27	20 ¹⁴ / ₁₄	84	8.45
4/15	15.5	2.5	-6	20.5	+1	-18	42.0	69	1118454	2815	27	20	84	8.4
5/15	15.5	2.5	-5	35.0	+1	-18	43.0	69	1118593	2817	27	20	84	8.45
6/15	15.5	2.5	0	34.0	+1	-18	35.0	69	1118740	2842	26	20	84	8.45
7/15	15.5	2.7	0	35.5	+1	-15	36.5	69	1118965	3200	26	20	84	8.45
8/15	15.5	2.7	-2	35.5	+1	-15	37.0	69	1119102	3240	27	20	84	8.4
end of 12/12/78										1119152		28		1
9/15	16.5	3.3	+3	34.5	+10	-12	39.5	68	1130852	1921	93	Brkn	90	8.5
10/15	16.5	3.4	-1	33.5	+11	-13	39.0	68	1132030	1967	93		90	8.45
11/15	16.5	3.4	+4	34.0	+11	-15	39.0	70	1132212	2017	93	~20?	90	8.5
12/15	16.0	3.2	-3	33.5	+12	-14	39.0	69	1132394	2069	94	—	90	8.5
13/15	15.5	3.3	-1	35.0	+12	-12	39.0	69	1132581	2121	94	—	91	8.5
14/20	15.5	3.3	-3	36.0	+13	-10	39.0	70	1132773	2175	94	—	91	8.5

12/12/23

* problems w/ scrubber liquid
level indicator (SLI) - appears to be reasonably accurate
during tests on 12/18

also on 12/19

- sight glass on scrubber

5" 2" inch hole : 2 bolts

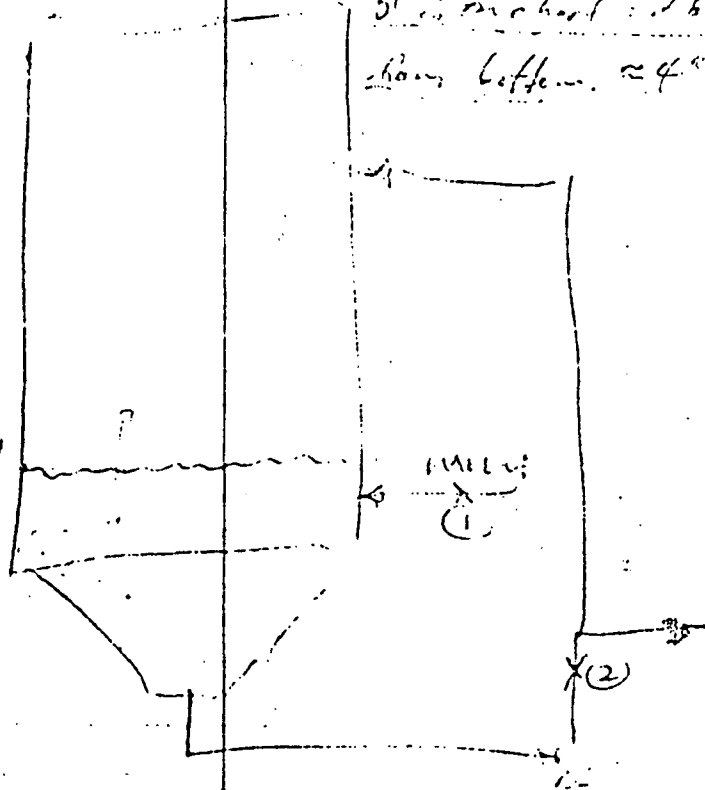
down bottom ~ 4"

3100

Drip to
scrubber ~ 2/8
closed

Total flow to
all three scrubbers

= 53 (gpm)



1 ~~2004~~

2 ~~1004~~

3 ~~2004~~

(1) Defined "Inlet" - (make-up)

(2) Defined "Outlet" - actually

into scrubbing section

cut first 1/2 cuts of samples when @ 205

Send to cut 1/2 cuts of samples when @ 195

32

TIME	TK-101	AFR	UNIT	GSP-C	AKT	NET	SEL	SEA	INT-1	INT-2	SET	FSKF	AOS	NH ₂ FEED
11:00	15.5	3.4	-2	34.5	+135	-5	39.5	69	1133223	2315	94	—	92	8.5
11:15	15.5	3.1	-2	36.0	+14	-3	40.0	69	1133424	2371	94	—	92	8.55
11:30	15.5	3.1	+4	36.0	+14	0	40.0	70	1133664	2424	95	—	92	9.5
11:45	15.0	3.1	0	36.0	+15	+2	40.0	69	1133845	2435	95	—	92	8.5
12:00	15.0	3.3	+2	33.5	+12	+3	40.5	68	1134010	2510	96	—	92	8.5
12:15	16.0	2.6	-3	34.0	+9	-2	36.5	68	1134210	2510	96	—	94	8.75
12:30	16.0	2.5	-1	33.5	+85	-11	32.5	69	1134500	2513	96	—	93	8.75
12:45	16.0	2.5	-4	33.5	+55	-15	33.5	70	1134750	2515	96	—	92	8.75
13:00	16.0	2.6	-2	32.0	+85	-20	36.5	69	1135000	2510	96	—	92	8.75
2:10	16.5	2.3	0	33.5	+10	-25	39.0	68	1135000	2512	96	—	92	8.75
2:35	16.5	2.7	+1	33.5	+9	-22	39.5	68	1135300	2517	96	—	92	8.75
3:10	17.0	2.4	0	34.0	+9	-20	37.5	68	1135500	3143	96	—	93	8.7
3:25	17.0	3.0	+4	33.0	+9	-15	40.0	68	1135700	3120	96	—	93	8.9
3:40	17.5	2.8	+1	33.0	+9	-13	39.0	70	1136000	3120	96	—	94	8.85
3:55	18.5	2.2	-3	32.5	+12	-18	37.5	68	1136300	3275	96	—	94	8.9
4:10	18.5	3.3	+5	31.5	+12	-22	37.0	69	1137044	3325	96	—	94	8.7
4:25	19.0	2.5	+2	32.5	+14	-25	37.0	69	1137225	3330	96	—	93	8.7

[illegible]

12/19/78

Run #2 1st 1/2 samples taken @ 9:25
2nd 1/2 samples taken @ 10:00

Run #3 1st 1/2 samples taken @ 11:20
2nd 1/2 samples taken @ 11:48

Total water to scrubbers @ 11:50 = 58.5

B flow = 10.5, A flow = 9.5

Run #4 1st 1/2 samples taken @ 1:20
2nd 1/2 samples taken @ 2:00

Total water to scrubbers @ 1:30 = 78.0

B flow = 14.5, A flow = 12

2:00 - air chopper to scrubber shut
down completely

Run #5 1st 1/2 samples taken @ 3:15
2nd 1/2 samples taken @ 3:48

Run #6 1st 1/2 samples taken @ 4:27
2nd 1/2 samples taken @ 5:00

APPENDIX H
PROJECT PARTICIPANTS

PROJECT PARTICIPANTS

Agrico Chemical Company
Blytheville, Arkansas
December 18 and 19, 1978

TRC

Willard A. Wade III, Project Manager
Reed W. Cass, Project Engineer
Eric A. Pearson, Project Scientist
Stephen F. Richardson, Test Team Leader
Margaret M. Fox, Chemist
Joanne M. Marchese, Chemist

GCA

Steven K. Harvey

Agrico Chemical Company

Jesse Boggan, Environmental Coordinator
James Kilpatrick, Chief Chemist
Deryl Beiard, Chemist

EPA

Clyde E. Riley, Technical Manager
Daniel Bivins
Eric A. Noble
Gary D. McAlister

APPENDIX I

SCOPE OF WORK

Includes:

Work Assignments
Technical Directives

22988

WORK ASSIGNMENT ENVIRONMENTAL PROTECTION AGENCY Research Triangle Park, N.C. 27711		EPA CONTRACT NO. 68-02-2820
		CONTRACTOR TRC of New England
		ASSIGNMENT NO. 11
		ASSIGNMENT CHANGE NO.
TITLE Conduct Emission Test Program at an Urea Manufacturing Plant		DATE 5 DEC 1978

DESCRIPTION

The Contractor shall perform an emission test program in accordance with the basic contract scope of work for the Emission Measurement Branch, and as set forth in the attached "Source Sampling and Analysis Schedule" at the following site:

Company: Agrico Chemical
 Location: Blytheville, Ark.
 Industry: Ammonium Fertilizer
 Project No.: 79-NHF-13

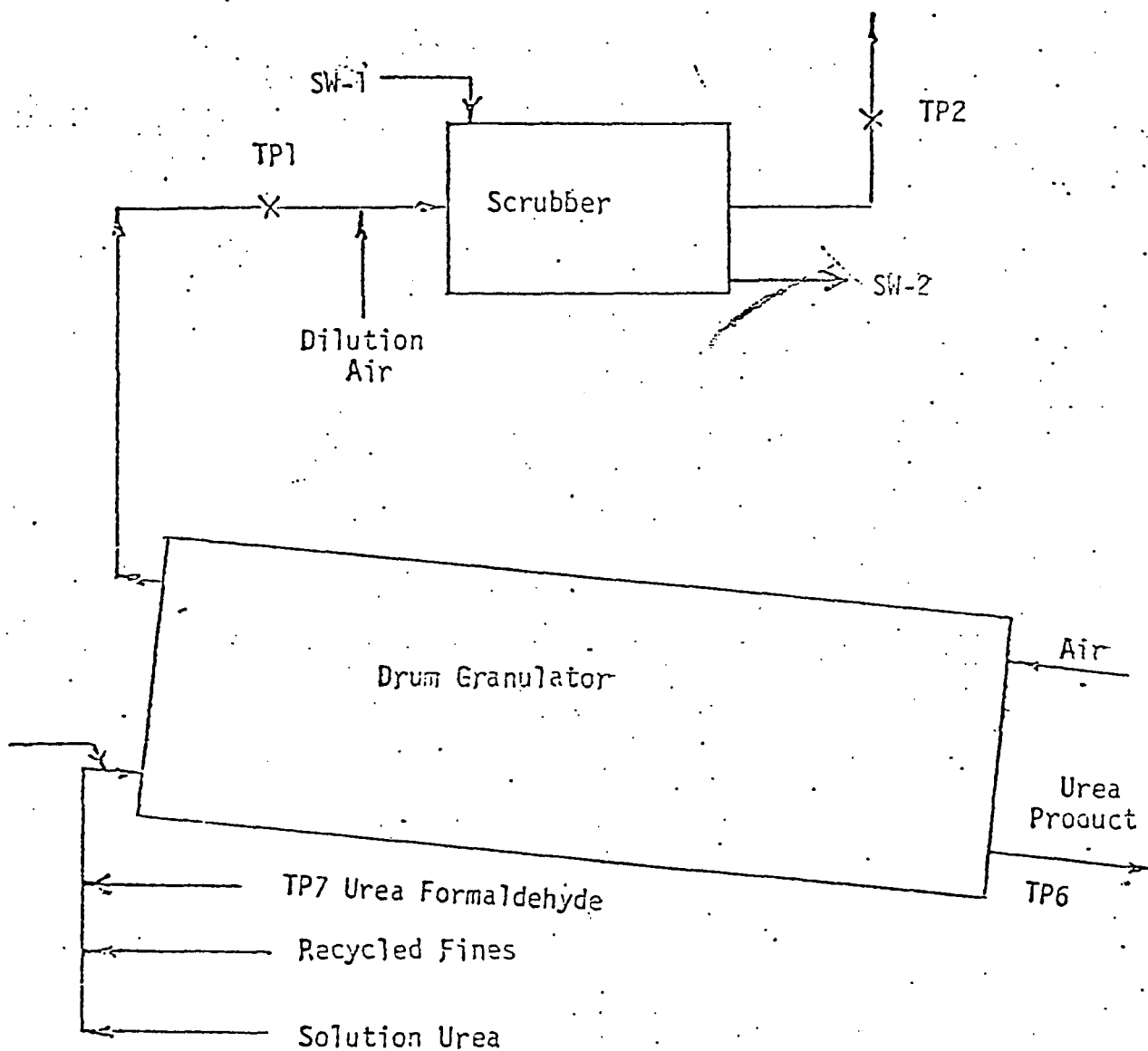
The Emission Measurement Branch's Technical Manager is Clyde E. Riley.
 Mail Drop 13, EMB, ESED, OAQPS, Research Triangle Park, North Carolina 27711.

Upon notification of approval of the proposed source test report, the Contractor shall provide 25 copies of the final report with appendices.

ESTIMATE OF	GOVERNMENT ESTIMATE	CONTRACTOR ESTIMATE
LABOR HOURS	200	200
DURATION OF WORK	3 months	3 months
COMPLETION DATE	March 31, 1979	March 31, 1979
REQUESTER'S SIGNATURE Clyde E. Riley	ORG CODE ESED/EMB	TELEPHONE (919) 541-5243
APPROVALS (as applicable)	SIGNATURE	DATE
BRANCH CHIEF	<i>F. E. McKinley for George W. Hoff</i>	11/30/78
DIVISION CHIEF	<i>Don R. Hadden</i>	12-5-78
PROJECT MANAGER	<i>James M. Bille</i>	12/4/78
CONTRACTING OFFICER	<i>C. Hester</i>	12-5-78

CONTRACTOR'S REPRESENTATIVE ACKNOWLEDGMENT

Figure 1
Agrico Co./The Williams Co., Blytheville, Arkansas



SOURCE SAMPLING AND ANALYSIS SCHEDULE

See Figure 1

Company Name: Agrico Chemical		Company Location: Blytheville, Ark.	
Industry: Ammonium Fertilizer		Process: Urea Granulator	
		Control Equipment: Scrubber	

Sampling Point Location	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	by	Type	Method	
P2	6	Urea Particulate	Modified EPA-5	CTR	60	30	Urea Mass	Kjeldahl	CTR	Ammonia	(EPA) Nessler	CTR
Aliquots shall be collected from each of the above 6 samples							Urea Mass	Kjeldahl	Agrico			
Sample analysis shall be conducted within 24 hours of collection on each of the H ₂ O samples.												
After completion of analysis remaining samples shall be split into 2 equal portions and treated with a stabilizer.												
One portion shall be treated with saturated mercuric chloride (approximately 2 ml per liter of water)												
Second portion shall be treated with concentrated sulfuric acid (approximately 2 ml per liter of water)												
These solutions shall be returned to the IRO lab and allowed to stand at room temperature for a period of 20 days; however they shall be analyzed by the Kjeldahl urea method once every two days for urea content during this period.												
SW-1	6	Scrubber Solution In	Composite	EPA	N. A.	N. A.	Percent Solids	Filtration	CTR	Urea NH ₃	Kjeldahl Nessler	CTR CTR
SW-2	6	Scrubber Solution Out	Composite	EPA	N. A.	N. A.	Percent Solids	Filtration	CTR	Urea NH ₃	Kjeldahl Nessler	CTR CTR

REMARKS:

1. Sampling shall be performed with ± 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, No. 159, Part II, Tuesday, Aug. 17, 1971 unless specifically changed by the Project Office.

A. Urea Method Development Instructions

1. Contractor shall determine stability conditions for the following six urea solution concentrations. TRC shall use the Kjeldahl urea method to analyze for urea content and the Nessler method to determine the ammonia content.
 - a. 40 mg of urea per liter of water
 - b. 100 mg of urea per liter of water
 - c. 40 mg of urea per liter of water with 2 ml of saturated mercuric chloride solution added
 - d. 100 mg of urea per liter of water with 5 ml of saturated mercuric chloride solution added
 - e. 40 mg of urea per liter of water with 2 ml of concentrated sulfuric acid added
 - f. 100 mg of urea per liter of water with 5 ml of concentrated sulfuric acid added.

These solutions shall be allowed to stand at room temperature for a period of 20 days; however, they shall be analyzed once every 2 days for urea and ammonia content. Questions regarding these instructions or the urea and ammonia analysis procedures shall be directed to Mr. Gary McAlister at 919-541-5276.

2. Contractor shall prepare two duplicate sets of "dry" urea audit samples. Each set shall contain 12 individual urea samples. Both sets of samples shall be forwarded to the Agrico Chemical plant in Blytheville, Arkansas, by TRC personnel. One set of samples shall be analyzed by Agrico personnel and the second set shall be analyzed by TRC personnel.

3. Agrico audit sample analysis shall be performed according to methods and procedures employed while analyzing the urea samples generated during the October 9, 1978, EPA test program.
4. TRC audit analysis shall be performed using the Kjeldahl urea method as directed by Mr. Gary McAlister, EPA.
5. Contractor shall specify procedures directing Agrico personnel to dilute the 12 audit samples with solutions of water and/or 1N H_2SO_4 . Audit sample analyses shall be conducted within 12 hours after dilution. The 12 audit samples shall be prepared and diluted as follows:

Dilute With 400 mls H_2O

No. 1	100 mg urea
No. 2	300 mg urea
No. 3	600 mg urea
No. 4	5 mg urea
No. 5	10 mg urea
No. 6	40 mg urea

Dilute with 250 mls 1N H_2SO_4

No. 7	2 mg urea
No. 8	5 mg urea
No. 9	10 mg urea
No. 10	5 mg urea
No. 11	4 mg urea
No. 12	30 mg urea

B. Agrico Test Program

1. Contractor shall collect six urea particulate samples from one of the operating granulator outlet stacks. Samples shall be collected using isokinetic sampling conditions for a period of approximately 1 hour. The collection train shall consist of a probe heated to stack temperature, a flexible teflon line, and five impingers. The first three impingers shall each be prefilled with 100 mls

Dist. H_2O , the fourth shall remain empty, and the fifth shall contain approximately 200 gms of silica gel. The second and third shall be of the Greenburg-Smith design with standard tips. The first, fourth, and fifth shall be modified with a 1/2" tube.

2. Cleanup shall consist of measuring the solution volumes and rinsing the probe, flex line, and impinger several times (3) with Dist. H_2O . Afterwards the water samples shall be filtered through a preweighed fiber glass filter using a Buchner funnel and vacuum pump.

3. Analysis shall consist of weighing the liquid samples initially. Afterward two equal aliquots shall be withdrawn. One aliquot shall be analyzed for urea and ammonia by Agrico personnel using the Kjeldahl urea method. The second aliquot shall be analyzed for urea and ammonia by TRC personnel using the Kjeldahl method as directed by EPA. Sample analysis shall be conducted within 24 hours of collection of all samples.

After the two analysis aliquots have been withdrawn the remaining sample volumes shall be split into two equal portions and treated with a stabilizer solution. One portion shall be combined with a saturated mercuric chloride solution (approximately 2 mls per liter of water). The second portion shall be combined with concentrated sulfuric acid (approximately 2 mls per liter of water).

4. These solutions shall be returned to the TRC laboratory and allowed to stand at room temperature for a period of 20 days; however, they shall be analyzed by the Kjeldahl urea method once every 2 days during this period for urea and ammonia content.
5. The preweighed glass fiber filters used to filter the water solutions shall be returned to the TRC laboratory, dried and weighed for undissolved solids.
6. Contractor shall separate and report all Research and Development data in a separate EPA proposed draft report. These method and evaluation data shall not be included in the Agrico NSPS report. Contractor shall submit 3 copies of the proposed R&D final report directly to Mr. J. E. McCarley, EMB, ESED, Mail Drop 13, Research Triangle Park, N. C. 27711. The separate R&D report shall be entitled "Development of Analytical Procedures for the Determination of Urea from Urea Manufacturing Facilities" and listed under Project No. 79-NHF-13.

EMISSION MEASUREMENT BRANCH

TECHNICAL DIRECTIVE NO. 1

Project Number 79-NHF-13 Date February 16, 1979

Contractor TRC of New England

Contract Number 68-02-2820 Work Assignment Number 11

Technical Manager Clyde E. Riley

Verbal Directions Given To Will Wade

Directive:

1. The Contractor shall perform formaldehyde analysis on each of the six urea particulate samples.

Clyde E. Riley C.E. Riley
Technical Manager, EMB

J. G. McLaughlin
Section Chief, EMB

EMISSION MEASUREMENT BRANCH

TECHNICAL DIRECTIVE NO. 2

Project Number 79-NHF-13 Date March 21, 1979

Contractor TRC of New England

Contract Number 68-02-2820 Work Assignment Number 11

Technical Manager Clyde E. Riley

Verbal Directions Given To Mr. Will Wade

Directive:

See attached pages.

Clyde E. Riley
Technical Manager, EMB

W. M. Ledy
Section Chief, EMB

Contractor shall perform the following evaluation analyses:

1. Prepare an urea standard solution containing 2mg urea/ml H₂O

Weigh 0.2000g of urea into a 100 ml volumetric flask and dilute to 100 ml with deionized, distilled H₂O.

2. Prepare an ammonia standard solution containing 20 mg NH₃/ml H₂O

Weigh 31.4100g of NH₄Cl into a 500 ml volumetric flask and dilute to 500 ml with deionized, distilled H₂O.

3. Prepare nine samples from the above standards as follows:

Sample Nos.	ml of Urea Std.	ml of NH ₃ Std.	Total Volume ml
1	5	0	100
2	5	0	200
3	15	0	200
4	10	0	100
5	10	1	200
6	10	5	200
7	5	5	100
8	5	25	100
9	5	50	100

Note: Samples must be analyzed within 24 hours after being prepared.

4. Analyze the nine samples using the colorimetric (p-aminobenzaldehyde) procedures. Use samples 1,2,3, and 4 to prepare a standard curve.
5. Calculate the measured values for the remaining samples 5 through 9.
6. Data shall be presented in mg urea/ml of solution along with the standard curve.

If additional information is required please contact Mr. Gary McAlister at 919/541-2237.

MD-19

cc: Gary McAlister
File: 79-NHF-13



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Air Quality Planning and Standards
Research Triangle Park, North Carolina 27711

February 13, 1980

Mr. Will Wade
TRC of New England
125 Silas Deane Highway
Wethersfield, Connecticut 06109

Reference: EPA Contract No. 68-02-2820, Assignment No. 11, Agrico Chemical,
Blytheville, Arkansas, EMB Report No. 79-NHF-13

Dear Will:

This correspondence is to document the enclosed Technical Directive instructions for conducting an evaluation of slope linearity for standard urea curves.

It has come to our attention that the standard curve slope may change with low-urea concentrations. In order to verify this conjecture Mr. Gary McAlister has requested that curves for two sets of standard samples be compared. The first set of standard samples will range from 50 mg urea/liter to 250 mg urea/liter. The second set will range from 1 mg urea/liter to 30 mg urea/liter. Standard solutions containing the following urea concentrations will be used to establish the two curves.

Set No. 1

1. 50 mg urea/liter
2. 100 mg urea/liter
3. 150 mg urea/liter
4. 200 mg urea/liter
5. 250 mg urea/liter

Set No. 2

1. 1 mg urea/liter
2. 2 mg urea/liter
3. 5 mg urea/liter
4. 7 mg urea/liter
5. 10 mg urea/liter
6. 20 mg urea/liter
7. 30 mg urea/liter

TRC shall prepare and analyze the standard solutions as follows.

Samples containing urea and deionized distilled water shall be made up in 100 ml volumetric flasks.

Samples shall be analyzed by the P-dimethylaminobenzaldehyde colorimetric procedure. Do not boil off the samples as there should be no impurities present to interfere with the analyses.

Establish calibration curve No. 1 using urea results obtained from Set No. 1 samples.

Determine urea concentrations from calibration curve No. 1 using measured values obtained from Set No. 2 samples.

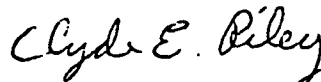
Establish calibration curve No. 2 using urea results obtained from Set No. 2 samples.

Compare the slope of the No. 1 curve to the slope of the No. 2 curve.

Please report your conclusions and recommendations along with a summary of the data to me by March 14, 1980. These data will be used to establish guidelines for the upcoming prill tower test in St. Helens, Oregon.

If you have any questions regarding these instructions or require additional information, please do not hesitate to contact me.

Sincerely yours,



Clyde E. Riley
Field Testing Section
Emission Measurement Branch

Enclosure

cc: Gary McAlister
Marge Fox, TRC

EMISSION MEASUREMENT BRANCH

TECHNICAL DIRECTIVE NO. 4

Project Number 79-NHF-13 Date Feb. 12, 1980
Contractor TRC of New England
Contract Number 68-02-2820 Work Assignment Number 11
Technical Manager Clyde E. Riley
Verbal Directions Given To Mr. Reed Cass

Directive:

Contractor shall determine slope linearity for standard urea curves using the following sets of samples.

Set No. 1

1. 50 mg urea/liter
2. 100 mg urea/liter
3. 150 mg urea/liter
4. 200 mg urea/liter
5. 250 mg urea/liter

Set No. 2

1. 1 mg urea/liter
2. 2 mg urea/liter
3. 5 mg urea/liter
4. 7 mg urea/liter
5. 10 mg urea/liter
6. 20 mg urea/liter
7. 30 mg urea/liter

Contractor shall prepare and analyze samples per instructions presented in February 12, 1980 correspondence to Mr. Will Wade.

Clyde E. Riley 2-12-80
Technical Manager, EMB

J. E. M. [Signature]
Section Chief, EMB