

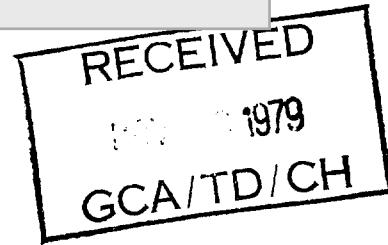
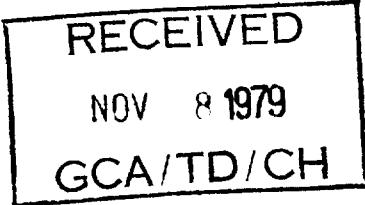


# Urea Manufacture

## Emission Test Report Agrico Chemical Company Blytheville, Arkansas

Note: This is a reference cited in AP 42, *Compilation of Air Pollutant Emission Factors, Volume I Stationary Point and Area Sources*. AP42 is located on the EPA web site at [www.epa.gov/ttn/chief/ap42/](http://www.epa.gov/ttn/chief/ap42/)

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01-799

REPORT ON "A" GRANULATOR SCRUBBER TESTS  
FOR UREA, AMMONIA AND FORMALDEHYDE EMISSIONS  
AND REMOVAL EFFICIENCIES, OPACITY AND  
PARTICLE SIZE AND SYNTHESIS TOWER SOLUTION  
VENT TESTS FOR UREA AND AMMONIA EMISSIONS AT  
THE AGRICO CHEMICAL COMPANY  
UREA FERTILIZER PLANT IN  
BLYTHEVILLE, ARKANSAS

# TRC

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## PREFACE

The work reported herein was conducted by personnel from TRC - The Research Corporation of New England (TRC), The GCA/Technology Division (GCA), The Agrico Chemical Company, Blytheville, Arkansas, and the U.S. Environmental Protection Agency (EPA).

The scope of work issued under EPA Contract No. 68-02-2820, Work Assignment No. 6 was under the supervision of the TRC Project Manager, Mr. Willard A. Wade, III. Mr. Reed W. Cass of TRC served as Project Engineer and was responsible for summarizing the test and analytical data in this report. Analysis of the samples was performed at the TRC labs located in Wethersfield, Connecticut under the direction of Ms. Joanne J. Marchese and at the Agrico Chemical, Blytheville, Arkansas labs under the direction of Mr. Jesse Boggan.

Mr. Stephen V. Capone and Mr. Stephen K. Harvey of GCA were responsible for monitoring the process operations during the testing program. GCA personnel were also responsible for writing the Process Description and Operations Section along with Appendix J of this report.

Members of Agrico Chemical, Blytheville, Arkansas whose assistance and guidance contributed greatly to the accomplishment of the test program, include 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 Project Engineer and was responsible for coordinating the process operations monitoring.

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

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October 30, 1979

NOTE:

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

## SECTION 1

### INTRODUCTION

Section 111 of the Clean Air Act of 1970 charges the Administrator of the U.S. Environmental Protection Agency (EPA) with the responsibility of establishing Federal standards of performance for new stationary sources which may significantly contribute to air pollution. When promulgated, these standards of performance for new stationary sources (SPNSS) are to reflect the degree of emission limitation achievable through application of the best demonstrated emission control technology. To assemble this background information, EPA utilizes emission data obtained from controlled sources involved in the particular industry under consideration.

Based on the above criteria, EPA's Office of Air Quality Planning and Standards (OAQPS) selected the Agrico Chemical Company's urea manufacturing plant at Blytheville, Arkansas as a site for an emission test program. The test program was designed to provide a portion of the emission data base required for SPNSS for the processes associated with the production of urea.

The Agrico Chemical Company's urea manufacturing plant at Blytheville, Arkansas produces granulated urea for industrial and fertilizer usage. The urea is manufactured by three Spherodizers® or granulators which operate continuously, 24 hours a day and 7 days a week as production demands. Emissions sampling was conducted on the "A" granulator exhaust emissions while its urea production rate was approximately 400 ton/day.

Each granulator has its own impingement type water scrubber. The granulator exhaust is ducted through a scrubber and fan prior to being discharged from a stack. Air flow through the granulator is controlled with a dilution damper which varies the ratio of dilution air to exhaust gas flowing to the constant

flow scrubber. A schematic of the granulators' exhaust gas ducting and control systems are presented as Figure 1. Emission sampling was also conducted on the main exhaust vent atop the urea synthesis tower (see Figure 1). This vent combines the various solution synthesis process gases into one common stack before exhausting them to the atmosphere.

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

The testing of the solids formation process was designed to characterize and quantify uncontrolled and controlled drum granulator emissions as well as determine the control equipment efficiency.

The synthesis process testing was designed to characterize and quantify emissions from one of the two newest processes expected to be installed for future urea solution capacity expansion in the industry.

The measurement program which was conducted at the Agrico Chemical Company facility in Blytheville, Arkansas during the week of October 9 through October 13, 1978 consisted of the following:

#### "A" Granulator Scrubber Measurements

1. Urea and Ammonia in Gas Streams: Three repetitions of concurrent inlet and outlet test runs were performed. The tests were conducted in accordance with the prescribed EPA method for urea and ammonia and provided velocity, moisture, ammonia and urea emission data.
2. Formaldehyde in Gas Streams: Three repetitions of concurrent inlet and outlet test runs were conducted. The tests were conducted in accordance with the prescribed EPA method for determination of formaldehyde and provided velocity, moisture, ammonia, urea particulate, and formaldehyde emissions data.

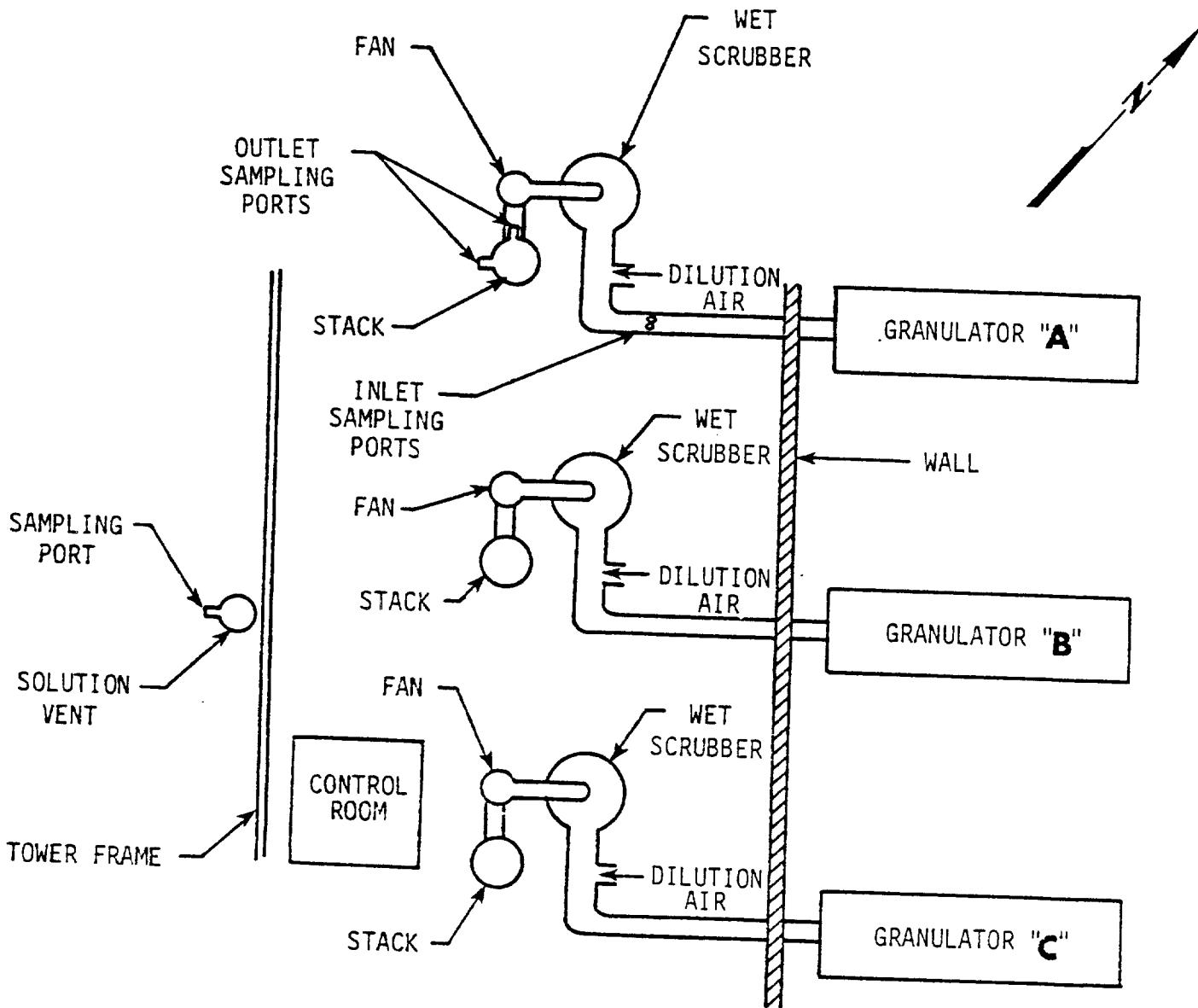


FIGURE 1: PLAN VIEW SHOWING LOCATIONS OF SOLUTION VENT AND GRANULATORS A, B AND C EXHAUST DUCTING, SCRUBBERS AND SAMPLING POINTS AT AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

3. Particle Size Distributions in Gas Streams: Three repetitions of inlet and outlet test runs were conducted. The tests were performed using the prescribed procedures as instructed by the manufacturer which are applicable to cascade impactors.
4. Visible Emissions from Stack: Approximately eight hours of visible observations were recorded on the stack discharge. Observations were performed in accordance with EPA Method 9 guidelines.
5. Gas Pressure Drop Across Scrubber: Pressure drop measurements were recorded approximately every 15 minutes during the testing periods.
6. pH, Temperature and Urea, Ammonia and Formaldehyde Content of Scrubber Liquor: Samples of inlet and outlet scrubber aqueous solutions were collected approximately every 30 minutes during the urea and ammonia testing. The pH and temperature of each sample was recorded. The samples were composited into 3 inlet and 3 outlet samples which were analyzed for urea, ammonia and formaldehyde.
7. Urea, Ammonia and Formaldehyde Content of Product: Single grab samples of the urea melt, formaldehyde additive, and the granulator product (unscreened and screened) were collected during the urea, ammonia, and formaldehyde tests. The formaldehyde additive sample was retained for future analysis. In addition the product samples were analyzed for moisture content.

#### Urea Synthesis Tower Vent Measurements

1. Urea and Ammonia in Gas Stream: Three repetitious test runs were performed according to procedures prescribed in EPA methods for urea and ammonia and for sampling streams with high moisture content.

TRC personnel were responsible for collecting and measuring the above emission parameters. Simultaneously, GCA was responsible for monitoring and recording necessary process and control equipment operating parameters.

Most of the test runs were discontinuous due to excessive loading at the granulator inlet sampling location. These interruptions which also delayed the simultaneous outlet sampling were encountered throughout the test program as indicated in Tables 1 through 5 (Daily Summary Logs).

The following sections of this report cover the summary of results, process description and operation, location of sampling points, and sampling and analytical procedures. In addition, Appendix N contains the summary of results of

TABLE 1. DAILY SUMMARY LOG FOR "A" GRANULATOR SAMPLING ON OCTOBER 10, 1978  
 AT AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Clock Time | Production Rate <sup>a</sup> (tons/day) | Area                   |                      | Stack Visible Emissions | Inlet pH | Inlet Temperature, °C | pH | Outlet Temperature, °C | pH | Liquid Temperature, °C | Pressure Drop, in H <sub>2</sub> O |
|------------|---|------------------------|----------------------|-------------------------|----------|-----------------------|----|------------------------|----|------------------------|------------------------------------|
|            |   | Inlet                  | Particulate b Outlet |                         |          |                       |    |                        |    |                        |                                    |
| 1115       | 399 <sup>c</sup>                        | Started Run 1 (Test 2) |                      | Started                 |          |                       |    |                        |    |                        | 18.25                              |
| 1130       | 394 <sup>d</sup>                        |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1131       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1135       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1139       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1145       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1150       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1157       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1200       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1215       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1230       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1234       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1235       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1240       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1245       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1250       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1300       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1310       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1315       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1328       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1330       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1345       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1353       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1400       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1415       | 399                                     |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1455       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1500       | 397                                     |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1510       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1515       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1525       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1530       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1531       |   | 393                    |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1538       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1540       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1545       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |
| 1550       |   |                        |                      |                         |          |                       |    |                        |    |                        |                                    |

TABLE 1 (Continued)

| Clock<br>Time | Product<br>ion<br>Rate<br>(tons/day) | Urea  |       | Particulate<br>b |        | Stack<br>Visible<br>Emissions | pH   | Scrubber Liquid |     | Outlet<br>Temperature,<br>°C | Pressure<br>Drop,<br>in H <sub>2</sub> O |
|---------------|--------------------------------------|-------|-------|------------------|--------|-------------------------------|------|-----------------|-----|------------------------------|--|
|               |                                      | Inlet | Inlet | Inlet            | outlet |                               |      | inlet           | pH  |                              |  |
| 1600          |                                      |       |       |                  |        |                               | 10.1 | 38              | 8.9 | 29                           | 16.25                                    |
| 1612          |                                      |       |       |                  |        |                               | 10.1 | 37              | 8.9 | 28                           | 16.25                                    |
| 1615          |                                      |       |       |                  |        |                               | 10.1 | 38              | 8.9 | 29                           | 16.25                                    |
| 1620          |                                      |       |       |                  |        |                               | 10.1 | 37              | 8.9 | 28                           | 16.25                                    |
| 1630          |                                      |       |       |                  |        |                               | 10.1 | 38              | 8.9 | 29                           | 16.25                                    |
| 1631          |                                      |       |       |                  |        |                               | 10.1 | 38              | 8.9 | 29                           | 16.25                                    |
| 1637          |                                      |       |       |                  |        |                               | 10.1 | 38              | 8.9 | 29                           | 16.25                                    |
| 1630          | c                                    | 391   | d     | 393              |        |                               |      |                 |     |                              |  |
| 1700          |                                      |       |       |                  |        |                               |      |                 |     |                              |  |
| 1737          |                                      |       |       |                  |        |                               |      |                 |     |                              |  |

d Average production rate value for each test run.

b Urea particulate samples analyzed for ammonia content.

c Inlet test runs production rates. Supplied by GCA.

d outlet test runs production rates. Supplied by GCA.

TABLE 2. DAILY SUMMARY LOG FOR "A" GRANULATOR SAMPLING ON OCTOBER 11, 1978  
AT AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Clock Time | Production Rate (tons/day) | Urea Particulates <sup>b</sup> Inlet | Stack Ventilations | Scrubber Liquid |           |          | Formaldehyde <sup>c</sup> Inlet | Formaldehyde <sup>c</sup> Outlet | Pressure Drop in, H <sub>2</sub> O | Process Material Samples |            |
|------------|----------------------------|--------------------------------------|--------------------|-----------------|-----------|----------|---------------------------------|----------------------------------|------------------------------------|--------------------------|------------|
|            |                            |                                      |                    | Inlet pH        | Outlet pH | Temp, °C |                                 |                                  |                                    | Wt Granulator            | Unscreened |
| 0918       | 309 <sup>e</sup>           | 199 <sup>f</sup>                     |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 0920       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 0924       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 0938       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 0947       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 0948       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 0956       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 0958       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 1004       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 1020       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 1028       |                            | 399                                  |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 1035       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 1040       |                            | 399                                  |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 1044       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 1050       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 1115       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 1120       |                            | 405                                  |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 1127       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 1129       |                            | 402                                  |                    |                 |           |          |                                 |                                  |                                    |                          |            |
| 1140       |                            |                                      |                    |                 |           |          |                                 |                                  |                                    |                          |            |

TABLE 2 (Continued)

TABLE 2 (Continued)

| Clock<br>Time | Production<br>Rate<br>(tons/day) | Urea<br>Inlet | Particulates<br>Inlet | Scrubber Liquid              |   |                      |                       | Formaldehyde <sup>c</sup><br>Inlet | Pressure<br>Drop<br>in. H <sub>2</sub> O | Process Material Samples <sup>d</sup> |      |           |          |
|---------------|----------------------------------|---------------|-----------------------|------------------------------|---|----------------------|-----------------------|------------------------------------|--|---------------------------------------|------|-----------|----------|
|               |                                  |               |                       | pH                           | Stack<br>Visible<br>Emissions               | Inlet<br>Temp,<br>°C | Outlet<br>Temp,<br>°C |                                    |  | "A" Granulator                        | Melt | Unscrubed | Screened |
| 1608          |                                  |               |                       |                              | Cont'd                                      | 9.9                  | 40                    | 8.6                                | 29                                       |                                       |      |           |          |
| 1610          |                                  |               |                       |                              |   | 10.0                 | 40                    | 8.5                                | 29                                       |                                       |      |           |          |
| 1615          | 352                              | e             | f                     | Started<br>Run 3<br>(Test 7) | Started<br>Run 3<br>(Test 7)                | 10.0                 | 40                    | 8.6                                | 29                                       |                                       |      |           |          |
| 1620          |                                  |               |                       |                              | Stepped                                     |                      |                       |                                    |  |                                       |      |           |          |
| 1626          |                                  |               |                       |                              | Awaiting<br>Inlet to<br>correct<br>Problems |                      |                       |                                    |  |                                       |      |           |          |
| 1630          |                                  |               |                       |                              |   |                      |                       |                                    |  |                                       |      |           |          |
| 1631          |                                  |               |                       |                              | Stopped<br>Nozzle                           |                      |                       |                                    |  |                                       |      |           |          |
| 1636          |                                  |               |                       |                              | Plunged<br>Cont'd                           |                      |                       |                                    |  |                                       |      |           |          |
| 1639          |                                  |               |                       |                              | Stopped<br>Nozzle                           | 10.0                 | 38                    | 8.6                                | 29                                       |                                       |      |           |          |
| 1640          |                                  |               |                       |                              | Plugged<br>Cont'd                           |                      |                       |                                    |  |                                       |      |           |          |
| 1641          |                                  |               |                       |                              | Cont'd                                      |                      |                       |                                    |  |                                       |      |           |          |
| 1644          |                                  |               |                       |                              |   |                      |                       |                                    |  |                                       |      |           |          |
| 1645          |                                  |               |                       |                              |   |                      |                       |                                    |  |                                       |      |           |          |
| 1657          |                                  |               |                       |                              | Stopped<br>Nozzle                           | 9.8                  | 38                    | 8.6                                | 29                                       |                                       |      |           |          |
| 1659          |                                  |               |                       |                              | Plunged<br>Cont'd                           |                      |                       |                                    |  |                                       |      |           |          |
| 1652          |                                  |               |                       |                              | Stopped<br>Switched<br>Ports                | 9.9                  | 38                    | 8.7                                | 29                                       |                                       |      |           |          |
| 1707          |                                  |               |                       |                              | Cont'd                                      |                      |                       |                                    |  |                                       |      |           |          |
| 1710          |                                  |               |                       |                              |   |                      |                       |                                    |  |                                       |      |           |          |
| 1717          |                                  |               |                       |                              |   |                      |                       |                                    |  |                                       |      |           |          |
| 1720          |                                  |               |                       |                              | Stopped<br>Nozzle                           | 10.0                 | 38                    | 8.7                                | 29                                       |                                       |      |           |          |
| 1723          |                                  |               |                       |                              | Plugged<br>Cont'd                           |                      |                       |                                    |  |                                       |      |           |          |
| 1731          |                                  |               |                       |                              | Stopped<br>Switched<br>Ports                | 10.0                 | 39                    | 8.6                                | 29                                       |                                       |      |           |          |
| 1732          |                                  |               |                       |                              | Cont'd                                      | 9.9                  | 38                    | 8.6                                | 28                                       |                                       |      |           |          |
| 1735          |                                  |               |                       |                              |   |                      |                       |                                    |  |                                       |      |           |          |
| 1740          |                                  |               |                       |                              |   |                      |                       |                                    |  |                                       |      |           |          |
| 1800          |                                  |               |                       |                              |   |                      |                       |                                    |  |                                       |      |           |          |

TABLE 2 (Continued)

| Clock<br>Time | Production<br>Rate <sup>a</sup><br>(tons/day) | Urea<br>particulates <sup>b</sup> |        | Stack<br>Visible<br>indications |         | Scrubber Liquid |                       | Process<br>Material Sample <sup>d</sup><br>Unscreened | Process<br>Material Sample <sup>d</sup><br>Screened |
|---------------|---|-----------------------------------|--------|---------------------------------|---------|-----------------|-----------------------|---|---|
|               |   | Inlet                             | Outlet | Inlet                           | Outlet  | Inlet<br>pH     | Outlet<br>Temp,<br>°C |   |   |
| 1802          |   |                                   |        |                                 |         | 10.0            | 48                    | Composite   | Composite   |
| 1810          |   |                                   |        |                                 |         | 10.0            | 39                    | Composite   | Composite   |
| 1825          |   |                                   |        |                                 |         |                 |                       |   |   |
| 1832          | 152   | e                                 | f      | Completed<br>Run 3<br>(Test 7)  | Stopped | 8.6             | 28                    |   |   |
| 1850          |   |                                   |        | Completed<br>Run 1<br>(Test 7)  |         | 8.6             | 28                    |   |   |
| 1950          |   |                                   |        |                                 |         |                 |                       |   |   |

<sup>a</sup>Average production rate value for each test run.<sup>b</sup>Urea particulate samples analyzed for ammonia content.<sup>c</sup>Formaldehyde samples also analyzed for urea and ammonia content.<sup>d</sup>Process samples analyzed for urea, ammonia, and formaldehyde content.<sup>e</sup>Inlet test runs production rates. Supplied by GCA.<sup>f</sup>Outlet test runs production rates. Supplied by GCA.

TABLE 3. DAILY SUMMARY LOG FOR "A" GRANULATOR SAMPLING ON  
OCTOBER 12, 1978 AT AGRICO CHEMICAL COMPANY IN  
BLYTHEVILLE, ARKANSAS

| Mean Time | Production Rate <sup>a</sup><br>(tons/day) | Particle Size      |                    | Stack<br>Visible<br>Emissions | Pressure<br>Drop<br>in, H <sub>2</sub> O |
|-----------|--|--------------------|--------------------|-------------------------------|--|
|           |  | Inlet              | Outlet             |                               |  |
| 0919      | 396 <sup>b</sup>                           | Started<br>Run 1   |                    |                               |  |
| 0920      |  | Completed<br>Run 1 |                    |                               |  |
| 0930      |  |                    |                    |                               | 16.75                                    |
| 1100      |  |                    |                    |                               | 16.75                                    |
| 1109      | 410 <sup>c</sup>                           |                    | Started<br>Run 1   |                               |  |
| 1110      |  |                    |                    |                               | 16.50                                    |
| 1130      |  |                    |                    |                               | 15.75                                    |
| 1200      |  |                    |                    |                               | 15.25                                    |
| 1230      |  |                    |                    |                               | 15.75                                    |
| 1300      |  |                    |                    |                               | 15.75                                    |
| 1330      |  |                    |                    |                               | 15.75                                    |
| 1420      |  |                    |                    |                               | 15.00                                    |
| 1445      |  |                    |                    |                               | 14.75                                    |
| 1509      | 410  |                    | Completed<br>Run 1 | Started                       |  |
| 1530      |  |                    |                    | Stopped                       |  |
| 1629      | 397  |                    | Started<br>Run 2   |                               |  |
| 1830      |  |                    |                    |                               | 15.25                                    |
| 1900      |  |                    |                    |                               | 15.25                                    |
| 1930      |  |                    |                    |                               | 15.50                                    |
| 2000      |  |                    |                    |                               | 15.00                                    |
| 2029      |  |                    | Completed<br>Run 2 |                               |  |
| 2030      |  |                    |                    |                               | 15.00                                    |

<sup>a</sup>Average production rate for each test run.

<sup>b</sup>Inlet test runs production rates. Supplied by GCA/Technology.

<sup>c</sup>Outlet test runs production rates. Supplied by GCA/Technology.

TABLE 4. DAILY SUMMARY LOG FOR "A" GRANULATOR SAMPLING ON  
OCTOBER 13, 1978 AT AGRICO CHEMICAL COMPANY IN  
BLYTHEVILLE, ARKANSAS

| Mean Time | Production Rate <sup>a</sup><br>(tons/day) | Particle Size   |                 | Pressure Drop<br>in, H <sub>2</sub> O |
|-----------|--|-----------------|-----------------|---------------------------------------|
|           |  | Inlet           | Outlet          |                                       |
| 0820      |  |                 |                 |                                       |
| 0855      | 359 <sup>c</sup>                           |                 |                 | 16.00                                 |
| 0900      |  |                 | Started Run 3   |                                       |
| 0915      |  |                 |                 | 16.50                                 |
| 0930      |  |                 |                 | 15.50                                 |
| 1000      |  |                 |                 | 16.00                                 |
| 1030      |  |                 |                 | 15.50                                 |
| 1100      |  |                 |                 | 15.50                                 |
| 1130      |  |                 |                 | 15.50                                 |
| 1140      |  |                 |                 | 16.00                                 |
| 1200      |  |                 |                 | 16.00                                 |
| 1230      |  |                 |                 | 16.50                                 |
| 1255      | 359  |                 | Completed Run 3 | 16.25                                 |
| 1300      |  |                 |                 |                                       |
| 1316      |  | Started Run 2   |                 | 16.50                                 |
| 1317      | 350 <sup>b</sup>                           | Completed Run 2 |                 |                                       |
| 1330      |  |                 |                 |                                       |
| 1400      |  |                 |                 | 17.00                                 |
| 1430      |  |                 |                 | 16.50                                 |
| 1500      |  |                 |                 | 18.00                                 |
| 1508      |  | Started Run 3   |                 | 16.50                                 |
| 1509      | 371 <sup>b</sup>                           | Completed Run 3 |                 |                                       |

<sup>a</sup>Average production rate value for each test run.

<sup>b</sup>Inlet test runs production rates. Supplied by GCA/Technology.

<sup>c</sup>Outlet test runs production rates. Supplied by GCA/Technology.

TABLE 5. DAILY SUMMARY LOG FOR SYNTHESIS TOWER VENT SAMPLING ON  
OCTOBER 13, 1978 AT AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Clock Time | Production <sup>a</sup> Rate (tons/day) | Urea <sup>b</sup> Particulates | Process Sample Formaldehyde Additive |
|------------|---|--------------------------------|--------------------------------------|
| 0930       |   | Started Run 1                  |                                      |
| 0945       | 1100                                    | Completed Run 1                |                                      |
| 1030       |   | Started Run 2                  |                                      |
| 1046       | 1100                                    | Completed Run 2                |                                      |
| 1130       |   | Started Run 3                  | Grab Sample Collected                |
| 1145       | 1100                                    | Completed Run 3                |                                      |

<sup>a</sup>Production rate, tons/day of 99.5 percent urea solution based on feed rate of ammonia to synthesis process.

<sup>b</sup>Two trial runs were conducted on October 12, 1978, however, they were not analyzed for urea and ammonia content.

cleanup evaluations performed on the sample collectors used for the test program. The Quality Assurance Audit samples analysis results supplied by the EPA are included in Appendix 0. Detailed descriptions of methods and procedures, field and laboratory data, and calculations are presented in various appendices, as noted.

## SECTION 2

### SUMMARY AND DISCUSSION OF RESULTS

#### INTRODUCTION

This section presents the results of a testing program conducted during the week of October 9 through 13, 1978 at the Agrico Chemical Company Facility in Blytheville, Arkansas. Testing was performed on gas and water streams entering and exiting the "A" Granulator Scrubber and on the gas stream venting from the Synthesis Tower.

The inlet gas sampling location for the "A" Granulator Scrubber (designated TP-1) was in a 30-foot straight section of horizontal duct. The integrated gas samples for the urea, ammonia and formaldehyde tests were collected isokinetically from 24 traverse points which were determined in accordance with EPA Reference Method 1.<sup>1</sup> The gas samples for the particle sizing tests were collected from a single point located at the centroid of the duct's cross sectional area.

The outlet gas sampling locations for the "A" Granulator Scrubber (designated TP-2) was in the 85-foot vertical stack. The integrated gas samples for the urea, ammonia and formaldehyde tests were collected isokinetically from 12 traverse points which were also determined from EPA Reference Method 1. The gas samples for the particle sizing tests were collected from the centroid of the duct's cross sectional area.

The gas sampling location for the Solution Tower Vent was in the 45-foot straight section of vertical duct. The samples were extracted from a single point located at the centroid of the duct's cross sectional area.

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

The following sections present summary tables of results and narrative on the testing. The Appendices contain all the pertinent data and information on the testing. Appendix N contains the results of the analyses of samples of the pretest cleanup of the sampling train and the deionized distilled water blank.

#### UREA AND AMMONIA TESTS ON "A" GRANULATOR SCRUBBER

The summaries of the urea and ammonia results at the "A" Granulator inlet (TP-1) and outlet (TP-2) are contained in Tables 6 and 7. It should be noted that the Run Numbers and TRC Test Numbers do not coincide. This is because there were two types of tests performed on the gases entering and leaving "A" Granulator. One type of test was primarily for urea and ammonia and the other type was primarily for formaldehyde. The Run Numbers signify the position of the tests in the sequence for each type of test. The TRC Test Numbers signify the position of the tests in the sequence of all the urea and ammonia and formaldehyde testing on "A" Granulator. TRC Test Number 1 was performed as a preliminary test to determine the moisture content of the gas streams.

The calculated urea removal efficiency of the "A" Granulator Scrubber (Tables 6 and 7) averaged 99.9%. The calculated ammonia removal efficiency of the "A" Granulator Scrubber (Tables 6 and 7) was always less than zero. A check of the ammonia concentration of the water solution entering and leaving the scrubber (Table 13) shows the water entering the scrubber to have a higher ammonia concentration than that leaving and thus indicates that some of the ammonia was stripped from the scrubber water, thus increasing its concentration in the air stream.

TABLE 6. SUMMARY OF RESULTS OF UREA AND AMMONIA TESTS ON OCTOBER 10 AND 11, 1978  
OF GASES ENTERING AND EXITING THE "A" GRANULATOR SCRUBBER AT AGRICO  
CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Run Number (TRC Test Number)   | Location                | Run 1 (Test 2)          | Run 2 (Test 3)          | Run 3 (Test 7)          |
|--|-------------------------|-------------------------|-------------------------|-------------------------|
|  |                         | Inlet<br>Outlet         | Inlet<br>Outlet         | Inlet<br>Outlet         |
| Batch Time   | 10-10-78<br>11:30-14:15 | 10-10-78<br>11:31-13:36 | 10-10-78<br>15:00-17:20 | 10-11-78<br>16:16-18:32 |
| Volume of Gas Sampled <sup>a</sup> -BSCF <sup>b</sup>  | 88.09                   | 107.8                   | 71.05                   | 56.68                   |
| Percent Moisture by Volume   | 1.6                     | 4.3                     | 2.1                     | 4.1                     |
| Average Stack Temperature--°F  | 161                     | 100                     | 163                     | 98                      |
| Stack Volumetric Flow Rate-BSCFM <sup>b</sup>  | 48,970                  | 52,020                  | 50,020                  | 53,090                  |
| Stack Volumetric Flow Rate-ACFM <sup>c</sup>   | 58,700                  | 57,410                  | 60,480                  | 58,260                  |
| Pressure Drop Across Scrubber--in. H <sub>2</sub> O  |                         | 17.9                    | 16.3                    | 61,940                  |
| Scrubber Liquid--pH  | 9.9                     | 8.9                     | 10                      | 8.9                     |
| Percent Isokinetic   | 96.7                    | 97.9                    | 95.4                    | 97.8                    |
| Percent Opacity Average  |                         | 5.0                     | No Readings             | 100.2                   |
| Production Rate--Tons/day  | 197                     | 392                     | 387                     | 391                     |
| Net Sampling Time-- Minutes  | 120                     | 120                     | 96                      | 72                      |
| Urea Concentrations and Mass Flow Rates<br>(Analyses Procedures Colorimetric)                  |                         |                         |                         |                         |
| mg<br>gr/BSCF <sup>d</sup>   | 64,280                  | 39.52                   | 60,090                  | 73.94                   |
| gr/ACFM <sup>c</sup>   | 11.26                   | 0.005659                | 13.05                   | 0.01038                 |
| lb/hr  | 9,394                   | 0.005127                | 10.79                   | 0.009458                |
| lb/ton   | 4,726                   | 2.523                   | 5,594                   | 4.723                   |
| Collection Efficiency, Percent   | 286.1                   | 0.1564                  | 347.2                   | 0.2898                  |
| Ammonia Concentrations and Mass<br>Flow Rates<br>(Analyses Procedure-Direct<br>Nesslerization) |                         |                         |                         |                         |
| mg<br>gr/BSCF  | 533.0                   | 1,352                   | 416.4                   | 2,572                   |
| gr/ACFM  | 0.09337                 | 0.1936                  | 0.0942                  | 0.361                   |
| lb/hr  | 0.07789                 | 0.1754                  | 0.07478                 | 0.329                   |
| lb/ton   | 39.19                   | 36.31                   | 38.77                   | 164.3                   |
| Collection Efficiency, Percent   | 2,372                   | 5.282                   | 2,406                   | 10.08                   |
|  | 0 <sup>f</sup>          | 0                       | <0                      | <0                      |
|  |                         |                         |                         | 99.9                    |
|  |                         |                         |                         | 99.9                    |

<sup>a</sup>Dry standard cubic feet at 68°F, 29.92 in H<sub>2</sub>O.

<sup>b</sup>Dry standard cubic feet per minute at 68°F, 29.92 in H<sub>2</sub>O.

<sup>c</sup>Actual cubic feet per minute.

<sup>d</sup>Grains per dry standard cubic foot at 68°F, 29.92 in H<sub>2</sub>O.

<sup>e</sup>Grains per actual cubic foot.

<sup>f</sup>Ammonia collection efficiency <0 because ammonia was stripped from scrubber water which contained the ammonia concentration in the scrubber outlet air.

TABLE 7. SUMMARY OF RESULTS OF FORMALDEHYDE, UREA, AND AMMONIA TESTS ON OCTOBER 11, 1978  
OF GASES ENTERING AND EXITING "A" GRANULATOR SCRUBBER AT AGRICO CHEMICAL COMPANY  
IN BLYTHEVILLE, ARKANSAS

| Run Number (IRGC Test Number)<br>Location               | Run 1 (Test 4)<br>Inlet<br>Outlet | Run 2 (Test 5)<br>Inlet<br>Outlet | Run 3 (Test 6)<br>Inlet<br>Outlet | Average<br>Outlet     |
|---|-----------------------------------|-----------------------------------|-----------------------------------|-----------------------|
| Time<br>Rate  | 0920-1040<br>10-11-78             | 0918-1028<br>10-11-78             | 1120-1242<br>10-11-78             | 1335-1516<br>10-11-78 |
| Volume of Gas Sampled--BSGF <sup>a</sup>                | 58.86                             | 59.04                             | 58.06                             | 55.9                  |
| Percent Moisture by Volume                              | 1.6                               | 5.4                               | 2.4                               | 2.7                   |
| Average Duct Temperature--°F                            | 157                               | 99                                | 162                               | 168                   |
| Duct Volumeetric Flow Rate--BSGF <sup>b</sup>           | 52.410                            | 55.350                            | 50.270                            | 54.380                |
| Duct Volumeetric Flow Rate--ACFM <sup>c</sup>           | 62.760                            | 61.960                            | 61.290                            | 60.560                |
| Pressure Drop Across Scrubber--in Hg                    | 15.50                             |                                   | 14.9                              | 14.1                  |
| Percent Isokinetic                                      |                                   |                                   | 103.5                             | 103.2                 |
| Net Sampling Time--Minutes                              | 100.6                             | 100.8                             | 72                                | 60                    |
| Percent Opacity Average                                 | 5.0                               |                                   | 3.7                               | 3.7                   |
| Production Rate--Tons/day                               | 397                               | 397                               | 400                               | 405                   |
| <b>Formaldehyde Concentrations and Mass Flow Rates</b>  |                                   |                                   |                                   |                       |
| (Analyses Procedure--Colorimetric)                      |                                   |                                   |                                   |                       |
| mg<br>gr/ACF <sup>d</sup>                               | 1.240                             | 0.304                             | 3.47                              | 1.610                 |
| gr/BSGF <sup>e</sup>                                    | 0.0002714                         | 0.00006626                        | 0.0001131                         | 0.0003452             |
| lb/hr   | 0.000325                          | 0.00008079                        | 0.0001260                         | 0.000267              |
| lb/ton  | 0.146                             | 0.035                             | 0.05873                           | 0.03282               |
| Collection Efficiency, Percent                          | 0.008828                          | 0.002091                          | 0.003525                          | 0.01059               |
| <b>Treated Concentrations and Mass Flow Rates</b>       |                                   |                                   |                                   |                       |
| (Analyses Procedure--Colorimetric)                      |                                   |                                   |                                   |                       |
| mg<br>gr/ACF <sup>d</sup>                               | 42,000                            | 40,420                            | 46.9                              | 38,111                |
| gr/BSGF <sup>e</sup>                                    | 9,194                             | 8,811                             | 0.01129                           | 8,172                 |
| lb/hr   | 11.01                             | 10.74                             | 0.01257                           | 10.10                 |
| lb/ton  | 4.946                             | 4.629                             | 5.86                              | 4.376                 |
| Collection Efficiency, Percent                          | 299                               | 278                               | 0.3518                            | 250.8                 |
| Ammonia Concentrations and Mass Flow Rates <sup>f</sup> |                                   |                                   |                                   |                       |
| (Analyses Procedure--Direct<br>Nesslerization)          |                                   |                                   |                                   |                       |
| mg<br>gr/ACF <sup>d</sup>                               | 157                               | 231                               | 500                               | 243.3                 |
| gr/BSGF <sup>e</sup>                                    | 0.07814                           | 0.05035                           | 0.1204                            | 0.05217               |
| lb/hr   | 0.09358                           | 0.06139                           | 0.1340                            | 0.06449               |
| lb/ton  | 42.04                             | 26.45                             | 62.48                             | 27.94                 |
| Collection Efficiency, Percent                          | 2,541                             | 1,589                             | 3.75                              | 1,601                 |
| Ammonia Concentrations and Mass Flow Rates <sup>f</sup> |                                   |                                   |                                   |                       |
| (Analyses Procedure--Direct<br>Nesslerization)          |                                   |                                   |                                   |                       |
| mg<br>gr/ACF <sup>d</sup>                               |                                   |                                   |                                   |                       |
| gr/BSGF <sup>e</sup>                                    |                                   |                                   |                                   |                       |
| lb/hr   |                                   |                                   |                                   |                       |
| lb/ton  |                                   |                                   |                                   |                       |
| Collection Efficiency, Percent                          |                                   |                                   |                                   |                       |

<sup>a</sup>Dry standard cubic feet at 68°F, 29.92 in Hg.

<sup>b</sup>Dry standard cubic feet per minute at 68°F, 29.92 in Hg.

<sup>c</sup>Actual cubic feet per minute.

<sup>d</sup>Grains per actual cubic foot.

<sup>e</sup>Grains per dry standard cubic foot.

<sup>f</sup>Ammonia results should be less for these runs because 100% capture was not achieved (No H<sub>2</sub>SO<sub>4</sub> in trap).

<sup>g</sup>Ammonia collection efficiency 50 because ammonia was stripped from scrubber water which enriched the ammonia concentration in the scrubber outlet air.

<sup>h</sup>Results voided - portion of sample lost.

## UREA RESULTS AT "A" GRANULATOR SCRUBBER INLET (TP-1)

A summary of the urea results of the urea and ammonia tests of gases sampled at the "A" Granulator Scrubber Inlet (TP-1) is presented in Table 8. The urea results are presented based on two methods of analysis. The colorimetric analysis was performed at the TRC Laboratory in Wethersfield, Connecticut by TRC chemists and the Kjeldahl analysis was performed at Agrico in Blytheville, Arkansas by Agrico chemists. Comparison of averages (Table 8) of the urea results shows the colorimetric results to be 8% higher than the Kjeldahl results. The average urea mass flow rates at TP-1 as determined by the colorimetric and Kjeldahl analyses were 5,024 and 4,853 pounds per hour, respectively.

A summary of the urea results of the formaldehyde tests at TP-1 is presented in Table 9. The average urea mass flow rates at TP-1 was 4,650 pounds per hour. The approximately 350 lb per hour difference between the urea mass flow rates measured during the urea and ammonia tests and the formaldehyde tests is most difficult to explain. The urea production rate averaged 380 tons/day for the urea and ammonia tests versus 408 tons/day for the formaldehyde tests. This would indicate that a higher urea mass flow rate should be present during the formaldehyde tests. The inlet sampling trains were modified for the formaldehyde tests by removing the  $H_2SO_4$  impingers. However, the maximum percent of the total urea mass collected in the  $H_2SO_4$  impingers was only 0.002% for the urea and ammonia tests, making their contribution insignificant.

## UREA RESULTS AT "A" GRANULATOR SCRUBBER OUTLET (TP-2)

A summary of the urea results for the urea and ammonia tests at the "A" Granulator Scrubber Outlet (TP-2) is presented in Table 10. The outlet urea results are also presented on the colorimetric analysis and the Kjeldahl analysis. Comparison of the averages (Table 10) of the urea results shows the Kjeldahl

TABLE 8. SUMMARY OF RESULTS OF UREA AND AMMONIA TESTS ON OCTOBER 10 AND 11, 1978  
OF GASES SAMPLED AT THE "A" GRANULATOR SCRUBBER INLET (TP-1) AT AGRICO  
CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Run Number (TRC Test Number)  | Run 1 (Test 2) |              |              | Run 2 (Test 3) |              |              | Run 3 (Test 7) |              |              | Average      |
|---|----------------|--------------|--------------|----------------|--------------|--------------|----------------|--------------|--------------|--------------|
|   | 10-10-78       | 10-10-78     | 10-10-78     | 10-10-78       | 10-10-78     | 10-10-78     | 10-10-78       | 10-10-78     | 10-10-78     |              |
| Rate  |                |              |              |                |              |              |                |              |              |              |
| Volume of Gas Sampled--dSCF <sup>a</sup>  | 88.09          | 71.05        | 56.68        | 56.68          | 56.68        | 56.68        | 56.68          | 56.68        | 56.68        | 71.94        |
| Percent Moisture of Volume  | 1.6            | 2.1          | 2.8          | 2.8            | 2.8          | 2.8          | 2.8            | 2.8          | 2.8          | 2.2          |
| Average Stack Temperature--°F   | 161            | 163          | 161          | 161            | 161          | 161          | 161            | 161          | 161          | 162          |
| Stack Volumetric Flow Rate--dSCF <sup>b</sup>                                   | 48.970         | 50.020       | 50.670       | 50.670         | 50.670       | 50.670       | 50.670         | 50.670       | 50.670       | 49.890       |
| Stack Volumetric Flow Rate--ACFM <sup>c</sup>                                   | 58.700         | 60.480       | 61.940       | 61.940         | 61.940       | 61.940       | 61.940         | 61.940       | 61.940       | 60.370       |
| Percent Isokinetic  | 96.7           | 95.4         | 100.2        | 100.2          | 100.2        | 100.2        | 100.2          | 100.2        | 100.2        | 97.4         |
| Net Sampling Time--Minutes  | 120.           | 96           | 72.          | 72.            | 72.          | 72.          | 72.            | 72.          | 72.          | 96.0         |
| Production Rate--tons/day   | 497            | 387          | 350          | 350            | 350          | 350          | 350            | 350          | 350          | 378          |
| Urea Concentrations and Mass <sup>d</sup>                                       |                |              |              |                |              |              |                |              |              |              |
| Flow Rates<br>(Analysis Procedure) <sup>d</sup>                                 | Colorimetric   | Kjeldahl     | Colorimetric | Kjeldahl       | Colorimetric | Kjeldahl     | Colorimetric   | Kjeldahl     | Kjeldahl     | Colorimetric |
| mg  | 64.280         | 62.984       | 60.090       | 58.241         | 40.200       | 37.638       | 54.860         | 52.954       | 52.954       |              |
| gr/dSCF <sup>e</sup>  | 11.26          | 11.11        | 13.05        | 12.57          | 10.94        | 10.24        | 11.75          | 11.35        | 11.35        |              |
| gr/ACFM <sup>f</sup>  | 9.394          | 9.27         | 10.79        | 10.40          | 8.952        | 8.384        | 9.712          | 9.39         | 9.39         |              |
| lb/hr   | 4.726          | 4.664        | 5.594        | 5.390          | 4.753        | 4.451        | 5.024          | 4.853        | 4.853        |              |
| lb/ton  | 296.1          | 282.4        | 347.2        | 334.6          | 325.7        | 305          | 319.7          | 308.8        | 308.8        |              |
| Ammonia Concentrations and Mass Flow Rates<br>(Analysis Procedure) <sup>g</sup> |                |              |              |                |              |              |                |              |              |              |
| Flow Rates<br>(Analysis Procedure) <sup>g</sup>                                 | Distillation   | Distillation | Distillation | Distillation   | Distillation | Distillation | Distillation   | Distillation | Distillation | Distillation |
| mg  | 533            | 387          | 416.4        | 416.4          | 298.4        | 298.4        | 415.9          | 415.9        | 415.9        | 415.9        |
| gr/dSCF <sup>e</sup>  | 0.09337        | 0.06784      | 0.0942       | 0.0942         | 0.08125      | 0.08125      | 0.0896         | 0.0896       | 0.0896       | 0.0896       |
| gr/ACFM <sup>f</sup>  | 0.07789        | 0.05666      | 0.07478      | 0.07478        | 0.06644      | 0.06644      | 0.0730         | 0.0730       | 0.0730       | 0.0730       |
| lb/hr   | 39.19          | 28.47        | 38.77        | 38.77          | 35.28        | 35.28        | 37.75          | 37.75        | 37.75        | 37.75        |
| lb/ton  | 2.372          | 1.723        | 2.406        | 2.406          | 2.418        | 2.418        | 2.399          | 2.399        | 2.399        | 2.399        |

<sup>a</sup>dry standard cubic feet at 68°F, 29.92 in. Hg.

<sup>b</sup>dry standard cubic feet per minute at 68°F, 29.92 in. Hg.

<sup>c</sup>Actual cubic feet per minute.

<sup>d</sup>Colorimetric analysis conducted by TRC personnel; Kjeldahl analysis conducted by Agrico personnel.

<sup>e</sup>Grains per dry standard cubic foot, 68°F, 29.92 in. Hg.

<sup>f</sup>Grains per actual cubic foot.

<sup>g</sup>Direct Nesslerization analysis conducted by TRC personnel; distillation followed by titrimetric determination conducted by Agrico personnel.

<sup>h</sup>Negative values (see Table I for additional information).

TABLE 9. SUMMARY OF RESULTS OF FORMALDEHYDE, UREA, AND AMMONIA TESTS ON OCTOBER 11, 1978  
OF GASES SAMPLED AT THE "A" GRANULATOR SCRUBBER INLET (TP-1) AT AGRICO CHEMICAL  
COMPANY IN BLYTHEVILLE, ARKANSAS

| Run Number (TRC Test Number)                            | Run 1 (Test 4) | Run 2 (Test 5) | Run 3 (Test 6) | Average   |
|---|----------------|----------------|----------------|-----------|
| Date  | 10-11-78       | 10-11-78       | 10-11-78       | 58.38     |
| Volume of Gas Sampled-- $\text{dSCF}^{\text{a}}$        | 58.86          | 58.06          | 58.21          | 2.3       |
| Percent Moisture by Volume                              | 1.6            | 2.4            | 2.7            |           |
| Average Duct Temperature-- $^{\circ}\text{F}$           | 157            | 162            | 168            | 162.33    |
| Duct Volumetric Flow Rate-- $\text{dSCFM}^{\text{b}}$   | 52,410         | 50,270         | 50,550         | 51,080    |
| Duct Volumetric Flow Rate-- $\text{ACFM}^{\text{c}}$    | 62,760         | 6,1290         | 62,480         | 62,180    |
| Pressure Drop Across Scrubber--in. $\text{H}_2\text{O}$ | 15.5           | 14.9           | 14.1           | 14.83     |
| Percent Isokinetic                                      | 100.6          | 103.5          | 103.2          | 102.4     |
| Net Sampling Time--Minutes                              | 72             | 72             | 72             | 72        |
| Production Rate--Tons/day                               | 397            | 400            | 419            | 405       |
| <u>Formaldehyde Concentrations and Mass Flow Rates</u>  |                |                |                |           |
| (Analysis Procedure-Colorimetric)                       |                |                |                |           |
| $\text{mg}^{\text{d}}$                                  | 1,260          | 0.304          | 1,610          | 1,055     |
| $\text{gr}/\text{dSCF}^{\text{d}}$                      | 0.000325       | 0.00008079     | 0.0004267      | 0.0002775 |
| $\text{gr}/\text{ACFM}^{\text{e}}$                      | 0.0002714      | 0.00006626     | 0.0003452      | 0.0002276 |
| $\text{lb}/\text{hr}$                                   | 0.146          | 0.035          | 0.1849         | 0.1220    |
| $\text{lb}/\text{ton}$                                  | 0.008828       | 0.002091       | 0.01059        | 0.007170  |
| <u>Urea Concentrations and Mass Flow Rates</u>          |                |                |                |           |
| (Analysis Procedure-Colorimetric)                       |                |                |                |           |
| $\text{mg}^{\text{d}}$                                  | 42,000         | 40,420         | 38,111         | 40,180    |
| $\text{gr}/\text{dSCF}^{\text{d}}$                      | 11.01          | 10.74          | 10.1           | 10.62     |
| $\text{gr}/\text{ACFM}^{\text{e}}$                      | 9.194          | 8.811          | 8.172          | 8.726     |
| $\text{lb}/\text{hr}$                                   | 4,946          | 4,629          | 4,376          | 4,650     |
| $\text{lb}/\text{ton}$                                  | 299            | 278            | 250.8          | 275.9     |
| <u>Ammonia Concentrations and Mass Flow Rates</u>       |                |                |                |           |
| (Analysis Procedure-Direct Nesslerization)              |                |                |                |           |
| $\text{mg}^{\text{d}}$                                  | 357            | 231            | 243.3          | 277.1     |
| $\text{gr}/\text{dSCF}^{\text{d}}$                      | 0.09358        | 0.06139        | 0.06449        | 0.07315   |
| $\text{gr}/\text{ACFM}^{\text{e}}$                      | 0.07814        | 0.05035        | 0.05217        | 0.06022   |
| $\text{lb}/\text{hr}$                                   | 42.06          | 26.45          | 27.94          | 32.14     |
| $\text{lb}/\text{ton}$                                  | 2,541          | 1,589          | 1,601          | 1,910     |

<sup>a</sup>Dry standard cubic feet @ 68°F, 29.92 in. Hg.

<sup>b</sup>Dry standard cubic feet per minute @ 68°F, 29.92 in. Hg.

<sup>c</sup>Actual cubic feet per minute.

<sup>d</sup>Grains per dry standard cubic foot.

<sup>e</sup>Grains per actual cubic foot.

<sup>f</sup>Ammonia results should be less for these runs because 100% capture was not achieved (No  $\text{H}_2\text{SO}_4$  in train).

TABLE 10. SUMMARY OF RESULTS OF UREA AND AMMONIA TESTS ON OCTOBER 10 AND 11, 1978  
OF GASES SAMPLED AT THE "A" GRANULATOR SCRUBBER OUTLET (TP-2) AT AGRICO  
CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Run Number (TRC Test Number)                             | Run 1 (Test 2) | Run 2 (Test 3) | Run 3 (Test 7) | Average      |
|--|----------------|----------------|----------------|--------------|
| date   | 10-10-78       | 10-10-78       | 10-11-78       | 110.07       |
| Volume of Gas Sampled--DSCF <sup>a</sup>                 | 107.8          | 109.90         | 115.0          | 115.9        |
| Percent Moisture by Volume                               | 4.3            | 4.1            | 3.4            | 3.93         |
| Average Duct Temperature--°F                             | 100.           | 98.            | 103.           | 100.3        |
| Duct Volumetric Flow Rate--DSCF <sup>b</sup>             | 52,020         | 53,090         | 55,420         | 535.0        |
| Duct Volumetric Flow Rate--ACFM <sup>c</sup>             | 57,410         | 58,260         | 61,370         | 52,020       |
| Pressure Drop Across Scrubber--<br>in., H <sub>2</sub> O | 17.9           | 16.3           | 14.6           | 16.3         |
| Percent Isokinetic                                       | 97.9           | 97.8           | 98.0           | 97.2         |
| Net Sampling Time--Minutes                               | 120.           | 120.           | 120.           | 120.         |
| Percent Opacity Average                                  | 5.0            | 5.0            | 5.1            | 5.1          |
| Production Rate--tons/hay                                | 392            | 391            | 391            | 378          |
| Urea Concentrations and<br>Nesslerization Rates          |                |                |                |              |
| (Analysis Procedure) <sup>d</sup>                        | Colorimetric   | Kjeldahl       | Colorimetric   | Kjeldahl     |
| mg   | 39.52          | 53.1           | 73.94          | 75.5         |
| gr/DSCF <sup>e</sup>                                     | 0.005659       | 0.00786        | 0.01038        | 62.03        |
| gr/ACF <sup>f</sup>                                      | 0.005127       | 0.00712        | 0.009458       | 0.02384      |
| lb/hr  | 2.523          | 3.521          | 4.723          | 0.01634      |
| lb/ton   | 0.1544         | 0.215          | 0.2898         | 0.007516     |
|  |                |                |                | 0.0103       |
|  |                |                |                | 0.00993      |
|  |                |                |                | 0.007637     |
|  |                |                |                | 0.01128      |
|  |                |                |                | 5.513        |
|  |                |                |                | .3495        |
|  |                |                |                | 0.2384       |
| Ammonia Concentrations and<br>Nesslerization Rates       |                |                |                |              |
| (Analysis Procedure) <sup>g</sup>                        | Direct         | Distillation   | Direct         | Distillation |
|  | Nesslerization | Titrimetric    | Nesslerization | Titrimetric  |
| mg   | 1,352          | 1,791          | 2,572          | 3,261        |
| gr/DSCF  | 0.1936         | 0.232          | 0.361          | 0.4571       |
| gr/ACF   | 0.1754         | 0.256          | 0.329          | 0.4166       |
| lb/hr  | 86.31          | 114.17         | 164.3          | 208.1        |
| lb/ton   | 5.282          | 6.987          | 10.08          | 12.76        |
|  |                |                |                | 1.34         |
|  |                |                |                | 0.2547       |
|  |                |                |                | 0.226        |
|  |                |                |                | 0.249        |
|  |                |                |                | 131.3        |
|  |                |                |                | 128.2        |
|  |                |                |                | 8.997        |
|  |                |                |                | 8.181        |
|  |                |                |                | 9.581        |

<sup>a</sup>dry standard cubic feet at 68°F, 29.92 in H<sub>2</sub>O.

<sup>b</sup>dry standard cubic feet per minute at 68°F, 29.92 in H<sub>2</sub>O.

<sup>c</sup>Actual cubic feet per minute.

<sup>d</sup>Colorimetric analysis conducted by TRC personnel; Kjeldahl analysis conducted by Agrico personnel.

<sup>e</sup>Grains per dry standard cubic foot at 68°F, 29.92 in H<sub>2</sub>O.

<sup>f</sup>Grains per actual cubic foot.

<sup>g</sup>Direct nesslerization analysis conducted by TRC personnel; distillation followed by titrimetric determination conducted by Agrico personnel.

results to be 48% higher than the colorimetric results.

The average urea mass flow rates at TP-2 (Table 10) as determined by the colorimetric and Kjeldahl analyses were 3.733 and 5.513 pounds per hour, respectively.

A summary of the urea results of the formaldehyde tests at TP-2 is presented in Table 11. The average urea mass flow rate at TP-2 was 3.715 pounds per hour.

The results show large differences in the urea mass flow rates measured at TP-2 for both the urea and ammonia tests (2.523 to 4.723 lb/hr) and the formaldehyde tests (1.571 to 5.860 lb/hr). These differences are believed to have been the result of the build up and periodic dislodgement of urea on the fan, breeching and stack walls. Water sprays were used twice a day to remove urea from the fan to prevent vibration. Testing was delayed for fan wash down.

#### AMMONIA RESULTS AT "A" GRANULATOR SCRUBBER INLET (TP-1)

A summary of the ammonia results of the urea and ammonia tests of gas sampled at the "A" Granulator Scrubber Inlet (TP-1) is presented in Table 8. The ammonia results are presented based on two methods of analysis. The analysis by direct Nesslerization was performed at the TRC Laboratory in Wethersfield, Connecticut by TRC chemists and the distillation/titrimetric analysis was performed at Agrico in Blytheville, Arkansas by Agrico chemists. The distillation/titrimetric method was done in addition to the direct Nesslerization because of interferences in the direct Nesslerization method. However, it is documented in the literature that during preliminary distillation analysis approximately 7 percent of the urea is converted to ammonia<sup>1</sup>. Since there was such a high concentration of urea (11.75 gr/dscf) compared to ammonia (0.0896 gr/dscf-by direct Nesslerization), a small deviation from the 7 percent conversion factor would mean a

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<sup>1</sup>Standard Methods, 14th Edition, 1975, p. 408

TABLE 11. SUMMARY OF RESULTS OF FORMALDEHYDE, UREA, AND AMMONIA TESTS ON OCTOBER 11, 1978  
OF GASES SAMPLED AT THE "A" GRANULATOR SCRUBBER OUTLET (TP-2) AT AGRICO CHEMICAL  
COMPANY IN BLYTHEVILLE, ARKANSAS

| Run Number (TRC Test Number)                           | Run 1 (Test 4) | Run 2 (Test 5) | Run 3 (Test 6) | Average  |
|--|----------------|----------------|----------------|----------|
| Date   | 10-11-78       | 10-11-78       | 10-11-78       | 57.5     |
| Volume of Gas Sampled--DSCF <sup>a</sup>               | 59.04          | 57.56          | 55.9           | 57.5     |
| Percent Moisture by Volume                             | 5.4            | 4.1            | 4.3            | 4.6      |
| Average Duct Temperature--°F                           | 99             | 102            | 105            | 102      |
| Duct Volumetric Flow Rate--DSCF <sup>d</sup>           | 55,350         | 54,380         | 53,550         | 54,430   |
| Duct Volumetric Flow Rate--ACFM <sup>c</sup>           | 61,960         | 60,560         | 60,060         | 60,860   |
| Pressure Drop Across Scrubber--in H <sub>2</sub> O     | 15.5           | 14.9           | 14.1           | 14.83    |
| Percent Isokinetic                                     | 100.8          | 100            | 98.6           | 99.8     |
| Net Sampling Time--minutes                             | 60             | 60             | 60             | 60       |
| Percent Opacity Average                                | 5.0            | 3.7            | 3.7            | 4.1      |
| Production Rate--Tons/day                              | 397            | 400            | 417            | 405      |
| <u>Formaldehyde Concentrations and Mass Flow Rates</u> |                |                |                |          |
| (Analysis Procedure)                                   |                |                |                |          |
| mg   | 0.47           | 0.259          | 0.365          |          |
| gr/DSCF  | 0.0001260      | 0.00007150     | 0.000069875    |          |
| gr/ACF   | 0.0001131      | 0.00006374     | 0.00006884     |          |
| lb/hr  | f              | 0.05873        | 0.03282        | 0.04578  |
| lb/ton   |                | 0.003525       | 0.001887       | 0.002706 |
| <u>Urea Concentrations and Mass Flow Rates</u>         |                |                |                |          |
| (Analysis Procedure)                                   |                |                |                |          |
| mg   | 46.9           | 12.4           | 29.65          |          |
| gr/DSCF  | 0.01257        | 0.003423       | 0.00800        |          |
| gr/ACF   | 0.01129        | 0.003052       | 0.00717        |          |
| lb/hr  | f              | 5.86           | 1.571          | 3.72     |
| lb/ton   |                | 0.3518         | 0.09035        | 0.2211   |
| <u>Ammonia Concentrations and Mass Flow Rates</u>      |                |                |                |          |
| (Analysis Procedure)                                   |                |                |                |          |
| mg   | 500            | 607.9          | 554            |          |
| gr/DSCF  | 0.1340         | 0.1678         | 0.1509         |          |
| gr/ACF   | 0.1204         | 0.1396         | 0.135          |          |
| lb/hr  | f              | 62.48          | 77.02          | 69.75    |
| lb/ton   |                | 3.75           | 4.429          | 4.090    |

<sup>a</sup>dry standard cubic feet at 68°F, 29.92 in. H<sub>2</sub>O.

<sup>b</sup>dry standard cubic feet per minute.

<sup>c</sup>Actual cubic feet per minute.

<sup>d</sup>Grains per dry standard cubic feet.

<sup>e</sup>Grains per actual cubic feet.

<sup>f</sup>Results voided - portion of sample lost.

considerable change in the amount of ammonia in the sample. When the values measured for ammonia by the distillation/titrimetric method were corrected for the 7 percent conversion of urea, the net ammonia concentrations were negative. Appendix L has additional information concerning conversion of urea to ammonia and the impact on the ammonia results.

The average ammonia mass flow rate at TP-1 as determined by direct Nesslerization was 37.75 lb per hour.

A summary of the ammonia results of the formaldehyde tests as TP-1 is presented in Table 9. The average ammonia mass flow rate was 32.14 lb per hour. It was expected that the ammonia which was collected by the formaldehyde tests would be less than that during the urea and ammonia tests because 1N  $H_2SO_4$  was not used in the formaldehyde train. However, two water impingers (100 ml distilled/deionized  $H_2O$  each) were used in the formaldehyde train while only one water impinger (100 ml distilled/deionized  $H_2O$ ) was used in the urea and ammonia tests. The differences in ammonia emissions as measured by both methods is not significant.

#### AMMONIA RESULTS AT "A" GRANULATOR OUTLET (TP-2)

A summary of the ammonia results of the urea and ammonia tests at the "A" Granulator Scrubber Outlet (TP-2) is presented in Table 10. The samples were analyzed by the direct Nesslerization and distillation/titrimetric methods as explained in the two previous sections. Since the urea in the sample was low (58.5 mg) compared to the ammonia (2009 mg), the correction for the 7 percent conversion of urea to ammonia during distillation was small compared to the amount of ammonia measured and, therefore, did not alter the results. The average ammonia mass flow rates determined by the direct

Nesslerization and distillation/titrimetric methods were 128.2 and 151.2 lb per hour, respectively.

A summary of the ammonia results of the formaldehyde tests at TP-2 is presented in Table 11. The average ammonia mass flow rate at TP-2 was 69.75 lb/hr. Again, as described in the previous section, it was expected that the ammonia measured by the formaldehyde train would be less than that collected in the urea and ammonia train because the formaldehyde train did not have the 1N H<sub>2</sub>SO<sub>4</sub> in the impingers. These outlet ammonia values are effected substantially by the sampling method as anticipated.

#### SAMPLING AND ANALYSIS PROBLEMS WITH UREA AND AMMONIA TESTS

The main sampling problem occurred at the scrubber inlet (TP-1) and was due to the high concentration and large size of the urea particles. This caused plugging of both the pitot tubes and the sample probe and nozzle. The problem of plugging of the pitot tube was alleviated by pumping air through the pitot tube side facing upstream. This purge line was removed and the pitot reconnected to the manometer for velocity readings. The problem of the nozzle plugging was never eliminated and the system had to be frequently shut down and the nozzle cleaned. The plugging was minimized by inserting the probe into the duct with the nozzle facing downstream. The probe was rotated 180° into the flow stream immediately before the initiation of sampling. The probe was removed from the duct in the reverse order. To reduce further the risk of plugging, we used the largest nozzle for which we could still maintain an isokinetic flow rate. This large nozzle increased sample velocities through the probe thus reducing agglomeration at the various fittings in the probe assembly.

The four major problems which were encountered during analysis were interferences, sensitivity of analytical instrumentation, conversion of urea to ammonia and contamination of either glassware and/or sampling and wash water. The interferences showed up on the blank wash up of the clean glassware in that the ammonia samples had high absorbance values but had a green instead of a yellow color which is expected for the ammonia. During analysis, each sample was checked to assure that sample color was consistent with that expected (see Appendix K). Analysis of the water blank found evidence of ammonia contamination at the level of 0.14 mg/l. This was insignificant compared to sample ammonia concentrations. Some of the measurements were made just above the limit of sensitivity of the spectrophotometer and, therefore, the error involved could be substantial. Conversion of urea to ammonia occurs when a sample containing a large amount of urea and a small amount of ammonia (in comparison to the urea) is distilled. It has been documented that approximately 7% of the urea is converted to ammonia. A correction for urea conversion to ammonia was applied to the results of the Agrico analysis by distillation and many of the results were calculated to be negative.

#### FORMALDEHYDE RESULTS AT GRANULATOR "A" SCRUBBER INLET (TP-1)

A summary of the formaldehyde results of gas sampled at the "A" Granulator Scrubber Inlet TP-1 is presented in Table 9. The average formaldehyde mass flow rate was 0.122 lb/hr. Examination of the formaldehyde mass flow rates for each of the three tests reveals that Run #2 had a much lower formaldehyde mass flow rate than either Run #1 or #3 while the Scrubber Outlet (TP-2) mass flow rates for Run #2 and #3 were similar with Run #2 having the highest formaldehyde mass flow rate. However, Run #1 for formaldehyde was voided due to a portion being lost.

## FORMALDEHYDE RESULTS AT GRANULATOR "A" SCRUBBER OUTLET

A summary of the formaldehyde results of gas sampled at the "A" Granulator Scrubber Outlet TP-2 is presented in Table 11. The average formaldehyde mass flow rate was 0.046 lb/hr. The results of the formaldehyde tests are somewhat questionable because the results of the analysis of the clean impinger samples, done prior to testing, indicate contamination of the same magnitude as the concentrations measured during some of the tests. This problem is dealt with more thoroughly in Appendix N.

## SAMPLING AND ANALYSIS PROBLEMS WITH FORMALDEHYDE DESIGNATED TESTS

The only sampling problem encountered was plugging at the scrubber inlet (TP-1) which has already been discussed in the previous subsection. An analysis problem did occur in that the analysis of a sample of the prewash of the sampling train glassware indicated contamination of the glassware of approximately the same magnitude as the amounts of formaldehyde measured at the scrubber outlet. There was also some formaldehyde in the blank H<sub>2</sub>O sample. The blank value was subtracted from the samples in accordance with the volume of water used during sampling and wash up.

## VISIBLE EMISSIONS FROM "A" GRANULATOR SCRUBBER STACK

The opacity of the plume from "A" Granulator Scrubber stack ranged from 0 to 5 percent. The six minute arithmetic averages are represented graphically in Figure 2 and are summarized in Tables 12-14. Most of the observations were made from the synthesis tower with the trees as background because the plume was white and the sky cloudy or completely overcast. Some readings were made on October 10, 1978 when the sun was shining and these were made using the

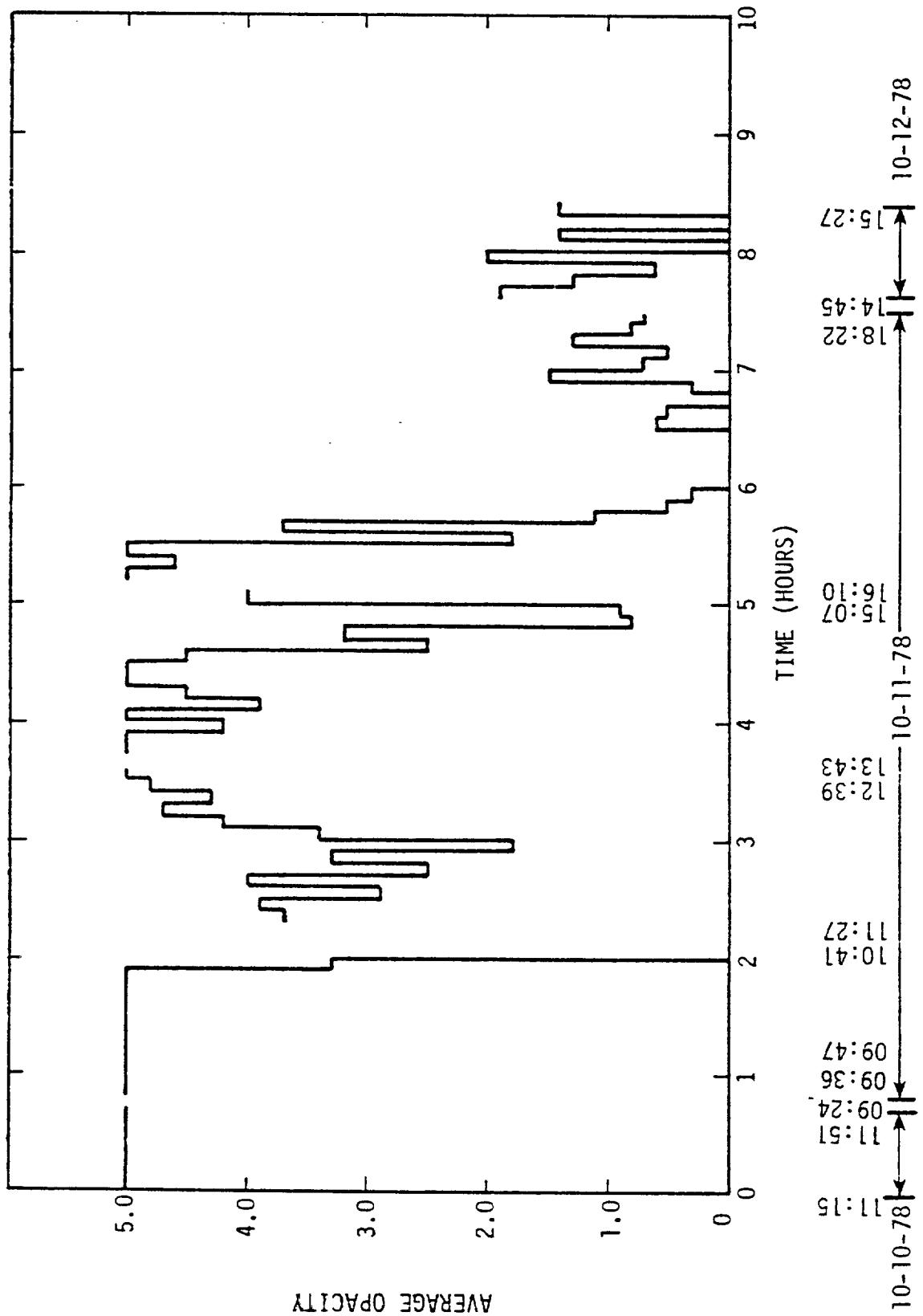


FIGURE 2: SIX MINUTE ARITHMETIC AVERAGES OF OCTOBER 10, 11 AND 13, 1978 OPACITY READINGS ON "A" GRANULATOR SCRUBBER STACK AT AGRICO COMPANY'S BLYTHEVILLE, ARKANSAS FACILITY

TABLE 12. SIX MINUTE ARITHMETIC AVERAGES OF OCTOBER 10, 1978  
OPACITY READINGS ON "A" GRANULATOR SCRUBBER STACK AT  
AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Time        | Average Opacity<br>for 6 Minutes |
|-------------|----------------------------------|
| 11:15-11:20 | 5                                |
| 11:21-11:26 | 5                                |
| 11:27-11:32 | 5                                |
| 11:33-11:38 | 5                                |
| 11:39-11:44 | 5                                |
| 11:45-11:50 | 5                                |
| 11:51-11:56 | 5                                |

TABLE 13. SIX MINUTE ARITHMETIC AVERAGES OF OCTOBER 11, 1978  
 OPACITY READINGS ON "A" GRANULATOR SCRUBBER STACK AT  
 AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Time         | Average Opacity<br>for 6 Minutes | Time         | Average Opacity<br>for 6 Minutes |
|--------------|----------------------------------|--------------|----------------------------------|
| 09:24-09:29  | 5                                | 14:19-14:24  | 5                                |
| 09:30-09:35  | 5                                | 14:25-14:30  | 5                                |
| 09:36-09:38* | 5                                | 14:31-14:36  | 4.5                              |
| 09:47-09:52  | 5                                | 14:37-14:42  | 2.5                              |
| 09:53-09:58  | 5                                | 14:43-14:48  | 3.2                              |
| 09:59-10:04  | 5                                | 14:49-14:54  | 0.8                              |
| 10:05-10:10  | 5                                | 14:55-15:00  | 0.9                              |
| 10:11-10:16  | 5                                | 15:01-15:06  | 3.2                              |
| 10:17-10:22  | 5                                | 15:07-15:12  | 4                                |
| 10:23-10:28  | 5                                | 16:10-16:15  | 5                                |
| 10:29-10:34  | 3.3                              | 16:16-16:21  | 4.6                              |
| 10:35-10:40  | 0                                | 16:22-16:27  | 5                                |
| 10:41-10:44* | 0                                | 16:28-16:33  | 1.8                              |
|              |                                  | 16:34-16:39  | 3.7                              |
| 11:27-11:32  | 3.7                              | 16:40-16:45  | 1.1                              |
| 11:33-11:38  | 3.9                              | 16:46-16:51  | .5                               |
| 11:39-11:44  | 2.9                              | 16:52-16:57  | .3                               |
| 11:45-11:50  | 4                                | 16:58-17:03  | 0                                |
| 11:51-11:56  | 2.5                              | 17:04-17:09  | 0                                |
| 11:57-12:02  | 3.3                              | 17:10-17:15  | 0                                |
| 12:03-12:08  | 1.8                              | 17:16-17:21  | 0                                |
| 12:09-12:14  | 3.4                              | 17:22-17:27  | 0                                |
| 12:15-12:20  | 4.2                              | 17:28-17:33  | .6                               |
| 12:21-12:26  | 4.7                              | 17:34-17:39  | .5                               |
| 12:27-12:32  | 4.3                              | 16:40-17:45  | 0                                |
| 12:33-12:38  | 4.8                              | 17:46-17:51  | .3                               |
| 12:39-12:43* | 5                                | 17:52-17:57  | 1.5                              |
| 13:43-13:48  | 5                                | 17:58-18:03  | .7                               |
| 13:49-13:54  | 5                                | 18:04-18:09  | .5                               |
| 13:55-14:00  | 4.2                              | 18:10-18:15  | 1.3                              |
| 14:01-14:06  | 5                                | 18:16-18:21  | 0.8                              |
| 14:07-14:12  | 3.9                              | 18:22-18:25* | 0.7                              |
| 14:13-14:18  | 4.5                              |              |                                  |

\*Averaging time less than 6 minutes.

TABLE 14. SIX MINUTE ARITHMETIC AVERAGES OF OCTOBER 12, 1978  
OPACITY READINGS ON "A" GRANULATOR SCRUBBER STACK AT  
AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Time         | Average Opacity<br>for 6 Minutes |
|--------------|----------------------------------|
| 14:45-14:50  | 1.9                              |
| 14:51-14:56  | 1.3                              |
| 14:57-15:02  | 0.6                              |
| 15:03-15:08  | 2                                |
| 15:09-15:14  | 0                                |
| 15:15-15:20  | 1.4                              |
| 15:21-15:26  | 0                                |
| 15:27-15:30* | 1.4                              |

\*Averaging time less than 6 minutes.

synthesis tower as a dark background. The detailed information on the visible emission measurements can be found in Appendix C.

#### PARTICLE SIZING TESTS ON "A" GRANULATOR SCRUBBER

Particle size distribution tests were conducted on the "A" Granulator Scrubber Inlet (TP-1) and Outlet (TP-2). Summaries of the results from the inlet and outlet tests are presented in Table 15. In a cascade impactor the stage which the gas stream enters first has a lower impaction velocity than the subsequent stage. As the impactor velocity increases, the size cutoff of the particulate which will be collected decreases. The aerodynamic size range is determined by the size cutoffs of stages  $n$  and  $n+1$  and corresponds to the mass of particulate collected on stage  $n+1$ . In other words, stage  $n$  collects particles greater than a particular size, stage  $n+1$  collects particles greater than a second particular size. Therefore, particles collected on stage  $n+1$  are  $\geq$  the size cutoff for stage  $n+1$  and the range of sizes of the particulate collected on stage  $n+1$  is defined. The size of the particulates entering the "A" Granulator Scrubber was 100%  $>2.2 \mu\text{m}$  in that essentially all the particulate was collected in the cyclone precollector. Most of the particulate leaving the scrubber was also collected in the cyclone precollector (average 77% of total mass collected). The cumulation size distribution curves for outlet Runs 2 and 3 are depicted in Figure 3. The curve in Figure 3 correspond to the percentage of total mass collected which is  $\leq$  a particular particle size expressed in terms of aerodynamic diameter. A straight line on the log probability graph paper would indicate a normal distribution of particulate.

TABLE 15. SUMMARY OF INLET AND OUTLET PARTICLE SIZING TEST RESULTS ON "A" GRANULATOR SCRUBBER AT AGRICO CHEMICAL IN BLYTHEVILLE, ARKANSAS<sup>a</sup>

| TRC Test No. | Sampling Location | Test Date | Test Time | Particulate Concentration, grains/dscf | Aerodynamic * Size Range, $\mu\text{m}$                    | Mass in Size Range, %                          |
|--------------|-------------------|-----------|-----------|--|--|--|
| 1            | Scrubber Inlet    | 10/12/78  | 0919-0920 | 12.10                                  | >2.2   | 100  |
| 2            | Scrubber Outlet   | 10/12/78  | 1109-1509 | 0.0033                                 | >3.7<br>2.7-3.7<br>1.7-2.7<br>1.0-1.7<br>0.56-1.0<br><0.56 | 98.7<br>0.00<br>0.14<br>0.21<br>0.39<br>0.39   |
| 3            | Scrubber Outlet   | 10/12/78  | 1629-2029 | 0.0044                                 | >3.7<br>2.7-3.7<br>1.7-2.7<br>1.0-1.7<br>0.56-1.0<br><0.56 | 89.35<br>0.00<br>7.32<br>1.31<br>1.05<br>0.97  |
| 4            | Scrubber Outlet   | 10/13/78  | 0855-1255 | 0.014                                  | >3.8<br>2.8-3.8<br>1.7-2.8<br>1.1-1.7<br>0.56-1.1<br><0.56 | 65.34<br>0.00<br>4.54<br>0.67<br>23.89<br>5.56 |
| 5            | Scrubber Inlet    | 10/13/78  | 1316-1317 | 32.67                                  | >2.3   | 100  |
| 6            | Scrubber Inlet    | 10/13/78  | 1508-1509 | 16.94                                  | >2.4   | 100  |

<sup>a</sup>The complete results can be found in Appendix B.

\* As unit density spheres

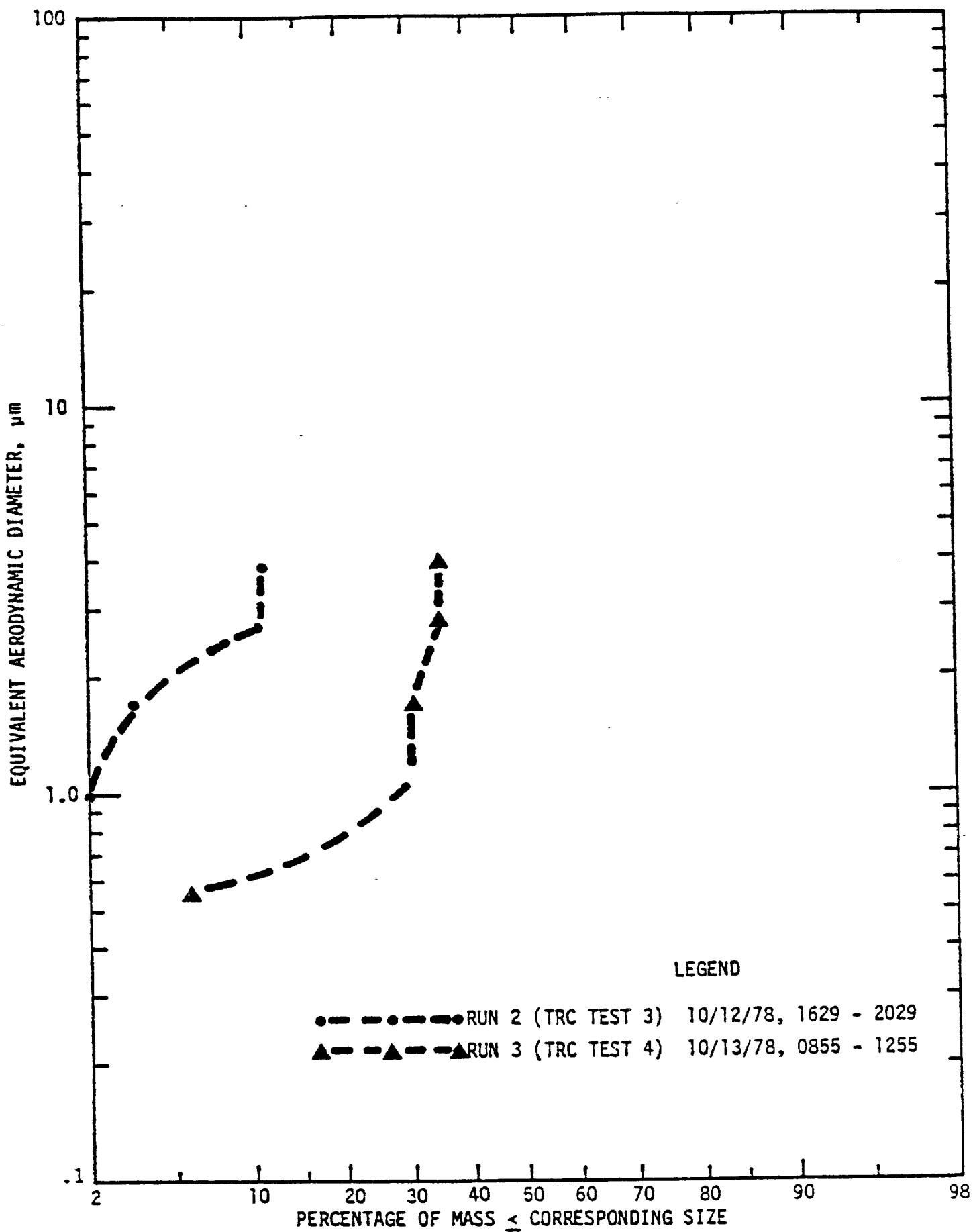


FIGURE 3: CUMULATIVE SIZE DISTRIBUTIONS OF PARTICULATE IN THE "A" GRANULATOR SCRUBBER STACK AT AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

It was not possible to plot cumulative size distribution curves for inlet Runs 1-3 (TRC Tests 1, 5, & 6) or outlet Run 1 (TRC Test 2) because more than 98% of the total mass was collected in the cyclone for these tests.

It was necessary to run the impactors on the inlet at a sampling rate of approximately one cubic foot per minute to maintain isokinetic sampling through the 3/16" nozzle. A smaller nozzle was not used because of problems of nozzle clogging during the urea and ammonia tests. This high flow rate resulted in a smaller size cutoff for the cyclone than desired. The high concentration of particulate at the scrubber inlet required a one minute sampling duration. The short sampling duration necessitated presetting the impactor flow. Each inlet test was begun by turning the impactor nozzle into the flow stream and starting the pump. Only minor adjustments were then required to maintain isokinetic flow.

#### PRESSURE DROP MEASUREMENTS ACROSS "A" GRANULATOR SCRUBBER

The pressure drop measurements across the "A" Granulator Scrubber were made with a vertical U tube water manometer which was connected to pressure taps at the scrubber inlet and outlet. The inlet pressure tap consisted of a stainless steel tube inserted into the middle of the duct through the dilution air damper. The tubing was fixed in place so that the open end was perpendicular to the flow stream. The outlet pressure tap consisted of a hole drilled through the section of ducting between the scrubber outlet and the inlet to the fan. The pressure drop across the scrubber was recorded at approximately 15 minute intervals during the tests for urea, ammonia and formaldehyde. The pressure drop was recorded at approximately 30 minute intervals for the particle sizing tests.

The pressure drops across the "A" Granulator Scrubber ranged from 14.0 to 20.5 inches of vertical water column and are presented in Table 16.

TABLE 16. SUMMARY OF OCTOBER 10, 11, 12, 13, 1978 "A" GRANULATOR SCRUBBER PRESSURE DROP MEASUREMENTS AT AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| <u>Date</u> | <u>Clock Time</u> | <u>ΔP, "H<sub>2</sub>O</u> | <u>Date</u>                         | <u>Clock Time</u> | <u>ΔP, "H<sub>2</sub>O</u> |
|-------------|-------------------|----------------------------|-------------------------------------|-------------------|----------------------------|
| 10/10/78    | 1130              | 18.25                      | 10/12/78                            | 0930              | 16.75                      |
|             | 1145              | 17.75                      |                                     | 1100              | 16.75                      |
|             | 1200              | 17.25                      |                                     | 1110              | 16.50                      |
|             | 1215              | 17.25                      |                                     | 1130              | 15.75                      |
|             | 1230              | 17.25                      |                                     | 1200              | 15.25                      |
|             | 1245              | 17.25                      | Dilution Damper Closed Down 1 Notch |                   |                            |
|             | 1300              | 17.25                      |                                     | 1230              | 15.75                      |
|             | 1315              | 18.00                      |                                     | 1300              | 15.75                      |
|             | 1330              | 18.75                      |                                     | 1330              | 15.00                      |
|             | 1345              | 20.50                      |                                     | 1420              | 14.75                      |
|             | 1400              | 18.00                      |                                     | 1830              | 15.25                      |
|             | 1415              | 17.00                      |                                     | 1900              | 15.25                      |
| 10/10/78    | 1500              | 16.25                      |                                     | 1930              | 15.50                      |
|             | 1515              | 16.25                      |                                     | 2000              | 15.00                      |
|             | 1530              | 16.25                      |                                     | 2030              | 15.00                      |
|             | 1545              | 16.25                      |                                     |                   |                            |
|             | 1600              | 16.25                      | 10/13/78                            | 0820              | 16.00                      |
|             | 1615              | 16.25                      |                                     | 0900              | 16.50                      |
|             | 1630              | 16.25                      |                                     | 0915              | 15.50                      |
|             | 1700              | 16.25                      |                                     | 0930              | 16.00                      |
| 10/11/78    | 1020              | 15.50                      |                                     | 1000              | 15.50                      |
|             | 1035              | 15.50                      |                                     | 1030              | 15.50                      |
|             | 1050              | 15.50                      |                                     | 1100              | 15.50                      |
|             | 1115              | 15.50                      |                                     | 1130              | 16.00                      |
| 10/11/78    | 1120              | 15.00                      |                                     | 1140              | 16.00                      |
|             | 1140              | 15.00                      |                                     | 1200              | 16.50                      |
|             | 1200              | 15.00                      |                                     | 1230              | 16.25                      |
|             | 1220              | 14.60                      |                                     | 1300              | 16.50                      |
|             | 1235              | 14.60                      |                                     | 1330              | 17.00                      |
| 10/11/78    | 1340              | 14.00                      |                                     | 1400              | 16.50                      |
|             | 1355              | 14.00                      |                                     | 1430              | 18.00                      |
|             | 1420              | 14.00                      |                                     | 1500              | 16.50                      |
|             | 1440              | 14.00                      |                                     |                   |                            |
|             | 1455              | 14.40                      |                                     |                   |                            |
|             | 1518              | 14.20                      |                                     |                   |                            |
| 10/11/78    | 1615              | 15.00                      |                                     |                   |                            |
|             | 1630              | 15.50                      |                                     |                   |                            |
|             | 1645              | 14.50                      |                                     |                   |                            |
|             | 1735              | 14.00                      |                                     |                   |                            |
|             | 1800              | 14.00                      |                                     |                   |                            |

UREA, AMMONIA AND FORMALDEHYDE IN SCRUBBING LIQUOR ENTERING AND EXISTING "A" GRANULATOR SCRUBBER

Samples (500 ml) of the scrubbing liquor streams entering and leaving (after the pump) the "A" Granulator Scrubber were collected at approximately 15 minute intervals during the urea and ammonia tests. The solution temperature was measured immediately after each sample was collected. The pH of each sample was also measured. The individual samples were composited into one inlet and one outlet sample and the pH was measured. The composited samples were analyzed for urea, ammonia and formaldehyde. Table 17 presents a summary of the measured urea, ammonia and formaldehyde concentrations in the scrubber liquor. Table 18 contains the pH and temperature readings of each of the individual samples.

The urea concentration for the inlet ranged from 21,680 to 49,360 mg/l and averaged 36,590 mg/l. The concentration for the outlet ranged from 506,200 to 1,012,000 mg/l and averaged 689,400 mg/l.

The ammonia concentration for the inlet ranged from 7,420 to 22,780 mg/l and averaged 13,900 mg/l. The concentration for the outlet ranged from 1,350 to 2,550 mg/l and averaged 1,800 mg/l.

The formaldehyde concentration for the inlet ranged from 1.52 to 36.8 mg/l and averaged 21.2 mg/l. The concentration for the outlet averaged 0.0275 mg/l.

Assuming that the scrubber liquid flows into and out of the scrubber are equal, the results of the analysis of the scrubber liquids shows the average urea concentration of the outlet solution to be 18.8 times the inlet solution which would be expected because the urea is being scrubbed out of the gas stream entering the scrubber. The average ammonia concentration of the outlet solution

TABLE 17. SUMMARY OF OCTOBER 10 AND 11, 1978 UREA, AMMONIA AND FORMALDEHYDE MEASUREMENTS ON THE SCRUBBING LIQUOR ENTERING AND EXITING "A" GRANULATOR SCRUBBER AT AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Run Number (AGR Test Number)                  | Run 1 (Test 2) |                 |          | Run 2 (Test 3) |          |          | Run 3 (Test 7)  |                 |                 | Average |
|---|----------------|-----------------|----------|----------------|----------|----------|-----------------|-----------------|-----------------|---------|
|   | Location       | Outlet          | Outlet   | Location       | Outlet   | Outlet   | Location        | Outlet          | Outlet          |         |
| Date  | 10/10/78       | 10/10/78        | 10/10/78 | 10/10/78       | 10/10/78 | 10/10/78 | 10/11/78        | 10/11/78        | 10/11/78        | ...     |
| Urea Concentration, mg/l <sup>a</sup>         | 21,680         | 506,200         | 38,720   | 1,012,000      | 69,360   | 550,000  | 66,590          | 680,400         | ...             | ...     |
| Ammonia Concentration, mg/l <sup>a</sup>      | 7,420          | 1,150           | 22,780   | 2,550          | 11,510   | 1,500    | 11,900          | 11,900          | 11,900          | 1,800   |
| Formaldehyde Concentration, mg/l <sup>a</sup> | 25.7           | ND <sup>d</sup> | 36.8     | ND             | 1.32     | 0.0275   | 21.2            | 21.2            | 21.2            | 0.0275  |
| pH <sup>b</sup>                               | 9.90           | 8.90            | 10.1     | 8.90           | 9.95     | 8.6      | NA <sup>c</sup> | NA <sup>c</sup> | NA <sup>c</sup> | NA      |
| Temperature, °C                               | 40             | 12              | 38       | 29             | 19       | 29       | 39              | 39              | 30              | 30      |
| Solids, mg/l <sup>a</sup>                     | ...            | ...             | ...      | ...            | ...      | ...      | ...             | ...             | ...             | ...     |

<sup>a</sup> milligrams per liter

<sup>b</sup> pH of composite sample

<sup>c</sup> average sample temperatures measured immediately after collection

<sup>d</sup> not detected

<sup>e</sup> not applicable

<sup>f</sup> from samples collected December 19, 1978.

TABLE 18. SUMMARY OF OCTOBER 10 AND 11, 1978 pH AND TEMPERATURE MEASUREMENTS ON INDIVIDUAL SAMPLES OF SCRUBBING LIQUID ENTERING AND EXITING "A" GRANULATOR SCRUBBER AT AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Run No.<br>(TRC Test No.) | Date     | Sampling Time | Scrubber Inlet Sample |    | Scrubber Outlet Sample |    |
|---------------------------|----------|---------------|-----------------------|----|------------------------|----|
|                           |          |               | pH                    | °C | pH                     | °C |
| 1 (2)                     | 10/10/78 | 1135          | 9.85                  | 40 | 8.90                   | 32 |
|                           |          | 1200          | 9.91                  | 40 | 8.90                   | 30 |
|                           |          | 1215          | 9.90                  | 40 | 8.91                   | 31 |
|                           |          | 1235          | 9.88                  | 40 | 8.90                   | 32 |
|                           |          | 1310          | 9.79 <sup>a</sup>     | 40 | 8.84                   | 33 |
|                           |          | 1330          | 9.92                  | 40 | 8.95                   | 33 |
| 2 (3)                     | 10/10/78 | 1455          | 10.05                 | 40 | 8.90                   | 30 |
|                           |          | 1510          | 10.10                 | 36 | 8.90                   | 29 |
|                           |          | 1525          | 10.10                 | 38 | 8.95                   | 29 |
|                           |          | 1540          | 10.10                 | 38 | 8.85 <sup>a</sup>      | 29 |
|                           |          | 1600          | 10.10                 | 38 | 8.90                   | 29 |
|                           |          | 1620          | 10.08 <sup>a</sup>    | 37 | 8.91                   | 28 |
|                           |          | 1650          | 10.08                 | 38 | 8.92                   | 29 |
| 3 (7)                     | 10/11/78 | 1608          | 9.90                  | 40 | 8.60                   | 29 |
|                           |          | 1620          | 9.95                  | 40 | 8.50                   | 29 |
|                           |          | 1640          | 9.95                  | 38 | 8.55                   | 29 |
|                           |          | 1650          | 9.80                  | 38 | 8.55                   | 29 |
|                           |          | 1710          | 9.85                  | 38 | 8.65                   | 29 |
|                           |          | 1720          | 9.98                  | 38 | 8.65                   | 29 |
|                           |          | 1740          | 9.95                  | 39 | 8.60 <sup>a</sup>      | 29 |
|                           |          | 1800          | 9.94                  | 38 | 8.61                   | 28 |
|                           |          | 1810          | 9.95                  | 38 | 8.62                   | 28 |
|                           |          | 1825          | 9.95                  | 39 | 8.60                   | 28 |

<sup>a</sup>pH drifting.

was 0.13 times the inlet solution which indicates that the ammonia is being stripped from the scrubber liquor (the measurements on the inlet and outlet gas streams indicated this -- See Table 6). The average formaldehyde concentration of the outlet solution was 0.0013 times the inlet solution which indicates that the formaldehyde was reacting with the urea since there was no evidence of formaldehyde stripping by the measurements on the inlet and outlet gas streams (see Table 7). The lower pH measured in the scrubber outlet solution is consistent with the lower ammonia concentration and the lower temperature of the outlet solution is indicative of the dissolution of urea into the scrubbing media, which is an endothermic reaction.

#### "A" GRANULATOR PROCESS SAMPLES UREA, AMMONIA, AND FORMALDEHYDE COMPOSITION

Process samples were taken of the urea melt, the urea product before screening, the urea product after screening and the formaldehyde additive. The urea melt, product before screening and product after screening was analyzed for urea, ammonia and formaldehyde. The formaldehyde additive sample is being retained for possible future analysis. The results of the analysis of the process samples are summarized in Table 19.

#### UREA AND AMMONIA TESTS ON SYNTHESIS TOWER MAIN VENT

A summary of the urea and ammonia test results on the Synthesis Tower Main Vent (TP-1) are presented in Table 20. The average urea and ammonia mass flow rates were 0.0713 and 1283.5 pounds per hour, respectively.

TABLE 19. SUMMARY OF OCTOBER 11, 1978 UREA, AMMONIA AND FORMALDEHYDE MEASUREMENTS ON THE "A" GRANULATOR UREA MELT, PRODUCT BEFORE SCREEN AND PRODUCT AFTER SCREEN AT AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

|                                | Urea<br>Melt | Urea Product<br>Before Screen | Urea Product<br>After Screen |
|--------------------------------|--------------|-------------------------------|------------------------------|
| Date                           | 10/11/78     | 10/11/78                      | 10/11/78                     |
| Sampling Time                  | 1510         | 1850                          | 1850                         |
| Urea Percent by Weight         | 98.77        | 93.98                         | 100.0                        |
| Ammonia Percent by Weight      | 0.00         | 0.00                          | 0.00                         |
| Formaldehyde Percent by Weight | 0.15         | 0.50                          | 0.23                         |

TABLE 20. SUMMARY OF RESULTS OF UREA AND AMMONIA TESTS ON OCTOBER 13, 1978  
OF GASES IN THE SYNTHESIS TOWER VENT AT AGRICO CHEMICAL COMPANY,  
BLYTHEVILLE, ARKANSAS

| Run No.  | (TRC Test No.) | 1 (3)        | 2 (4)        | 3 (5)    | Average  |
|--|----------------|--------------|--------------|----------|----------|
| Date   | 10-13-78       | 10-13-78     | 10-13-78     | 10-13-78 | 10-13-78 |
| Time   | 0930 to 0945   | 1030 to 1446 | 1130 to 1145 |          |          |
| Volume of Gas Sampled-DSCF <sup>a</sup>  | 4.31           | 4:41         | 4.70         |          |          |
| % Moisture by Volume   | 87.97          | 88.37        | 90.56        | 88.97    | 88.97    |
| Average Duct Temperature - °F  | 175            | 185          | 185          | 182      | 182      |
| Duct Volumetric  |                |              |              |          |          |
| Flow Rate - DSCFM <sup>b</sup>   | 1248           | 1202         | 990.9        | 1147     | 1147     |
| Duct Volumetric  |                |              |              |          |          |
| Flow Rate - ACFM <sup>c</sup>  | 12458          | 12631        | 12802        | 12630    | 12630    |
| Urea - Concentrations and Mass Flow Rates, Analysis Procedure - Colorimetric           |                |              |              |          |          |
| Mg   | 1.71           | 0.36         | 4.64         | 2.24     | 2.24     |
| gr/DSCF <sup>d</sup>   | 0.0061         | 0.00126      | 0.0152       | 0.00752  | 0.00752  |
| 1b/hr <sup>e</sup>   | 0.065          | 0.13         | 0.130        | 0.0713   | 0.0713   |
| Ammonia Concentrations and Mass Flow Rates, Analysis Procedure - Direct Nesslerization |                |              |              |          |          |
| Mg   | 32730          | 39310        | 40120        | 37390    | 37390    |
| gr/DSCF  | 117.2          | 137.5        | 131.7        | 128.8    | 128.8    |
| 1b/hr  | 1253.6         | 1417.6       | 1179.3       | 1283.5   | 1283.5   |

<sup>a</sup>Dry standard cubic feet @ 68°F, 29.92 in. Hg. includes Air, Ammonia and 'Inerts'.

<sup>b</sup>Dry standard cubic feet per minute @ 68°F, 29.92 in. Hg.

<sup>c</sup>Actual cubic feet per minute.

<sup>d</sup>Grains per dry standard cubic foot @ 68°F, 29.92 in. Hg.

Five tests were conducted on the synthesis tower Main Vent, however, the first two were not used because of evidence of ammonia passing through the sampling system. This problem was alleviated by the use of additional impingers in the sampling train and by using 10N H<sub>2</sub>SO<sub>4</sub>. The Sampling and Analysis Methodologies Section 5 explains in detail the changes which were made to the sampling train.

## SECTION 3

### PROCESS DESCRIPTION AND OPERATION

#### FACILITY DESCRIPTION:

##### I. PROCESS EQUIPMENT

Urea is produced by reacting liquid ammonia ( $\text{NH}_3$ ) with carbon dioxide ( $\text{CO}_2$ ) at elevated temperature and pressure. The reaction is exothermic and spontaneous and results in formation of liquid ammonium carbamate ( $\text{NH}_2\text{CO}_2\text{NH}_4$ ). The liquid ammonium carbamate is subsequently decomposed to urea ( $\text{CO}(\text{NH}_2)_2$ ) and water. The resulting solution of urea in water is concentrated to 98+ percent urea when it will be subsequently solidified.

The Stamicarbon  $\text{CO}_2$  Stripping Process is the urea synthesis method employed at this facility. There are three continuous vents and one intermittent vent from this process which have been combined into one tall stack. The continuous vents are: medium pressure absorber, low pressure scrubber and the flash tank condenser from the vacuum evaporators. The intermittent vent is from the carbamate condenser steam drum. Approximately 25 percent of the time 50 psig steam from this steam drum is vented through the common stack because it is not usable elsewhere in the plant.

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

The concentrated molten urea, referred to as melt, leaves the solution synthesis process and is pumped to the solids formation equipment. This facility employs rotary drum granulators, designed by C&I Girdler, as the solids forming devices.

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

Cooled granules leaving the rotary drum granulator are screened. Over-size 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.

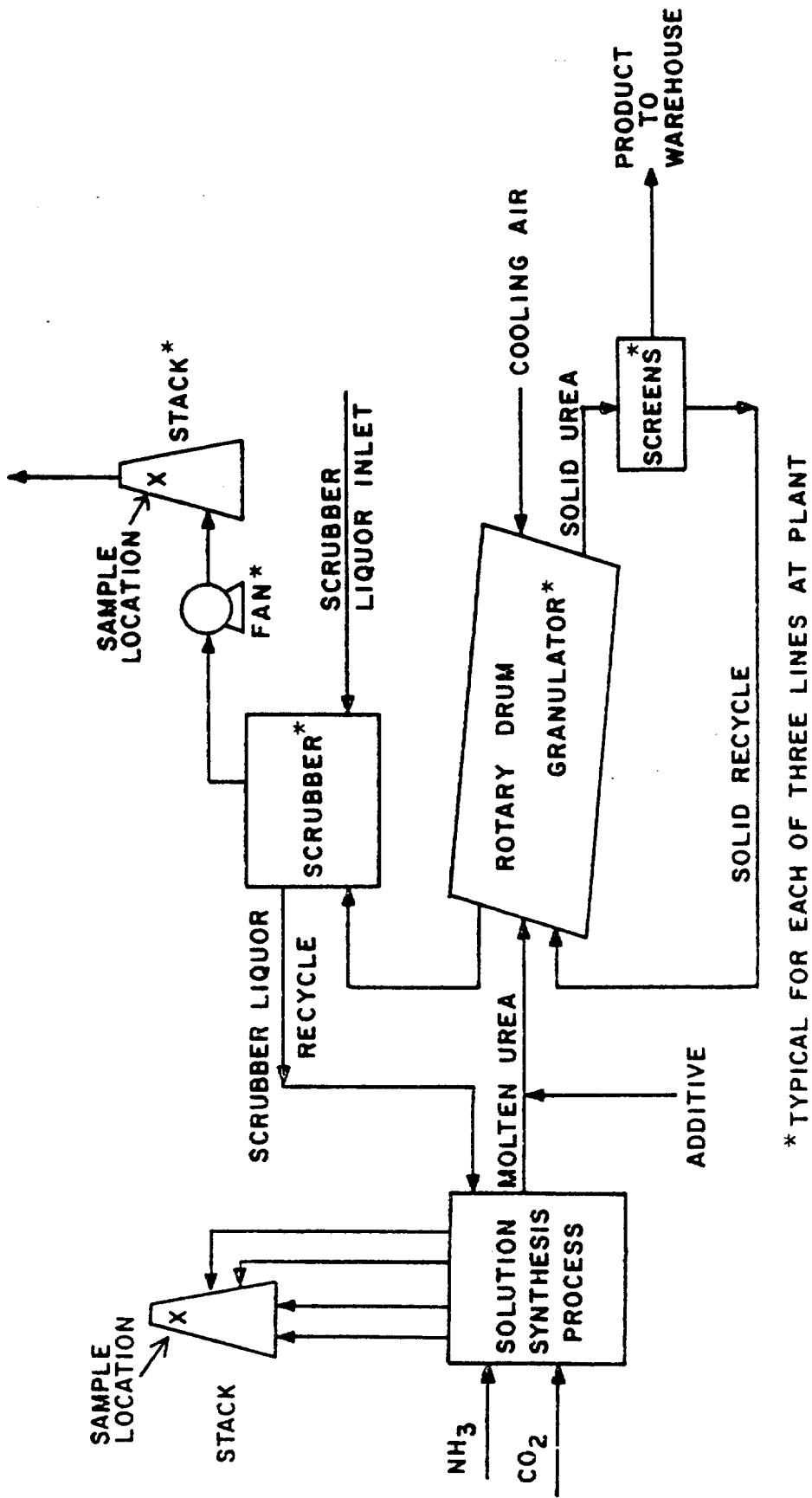
This facility does not coat the product urea granules. Instead, a formaldehyde-based additive is added to the molten urea before it is sprayed in the granulator.

At this facility, emissions result from various vents in the solution synthesis process and from the granulator cooling air exhaust. The solution synthesis process vents are combined into one stack before exhausting to ambient. The granulator cooling air passes through a scrubber and fan before being exhausted out a stack. Scrubber liquid is returned to the solution synthesis process for urea recovery.

This facility employs three parallel granulators, each with its own screens, scrubber, fan, and stack, for solid production. A single solution synthesis process supplies all three granulators (Figure 4).

Plant personnel provided a process drawing during the testing which EPA was allowed to retain. It is entitled:

Utility Flow Diagram  
1000 ST D Urea Plant  
Agrico Chemical Company  
Blytheville, Arkansas  
1320-1-50-15



\* TYPICAL FOR EACH OF THREE LINES AT PLANT

Figure 4 Urea Manufacturing, Agrico Chemical Co., Blytheville, Arkansas.

## II. EMISSIONS AND EMISSIONS CONTROL EQUIPMENT

The Stamicarbon CO<sub>2</sub> Stripping urea synthesis process at this plant incorporates emission reduction and energy recovery techniques in its standard design.

The high and low pressure scrubbers at this facility are typically employed in this process to reduce ammonia losses to the exhaust streams that are necessary for inerts removal from the system. The medium pressure absorber is employed only in those instances when the overheads stream from the reactor has a high enough hydrogen content to make total ammonia removal in the high pressure scrubber unsafe. The exhausts at this facility from the medium pressure absorber and the low pressure scrubber should therefore contain very little ammonia by design. In addition, the process design should result in practically no urea in these exhausts.

The exhaust from the steam jet ejector vacuum system on the evaporators should also contain mostly inerts, very little ammonia, and little urea by design. The procedure employed to condense the steam used in this system acts as an emission reduction device.

The steam drum in this process is employed as a heat recovery device. It uses "dirty" process condensate to produce 50 psig steam for use in the process when needed. However, when not usable in the process this "dirty" steam must be vented to atmosphere. Because the production of this steam is an integral heat removal step in the overall process, its production cannot be ceased when there is no demand. When vented, this steam can be expected to contain approximately 100 ppm of ammonia and carbon dioxide and traces of urea.

This plant has combined the above four exhausts into a single tall stack before release to the atmosphere.

The granulators employed at this plant for solids production are typically controlled by scrubbers. These scrubbers must be used on the granulators to reduce the product losses resulting from the cooling air passing through the granulator. This airstream entrains significant quantities of urea as it passes through the granulator and, if exhausted untreated, would significantly effect the economic viability of this solids production technique. The Joy Turbulaire "Type D" impingement scrubbers normally employed can be operated at varied pressure drops by adjusting scrubber liquid level. This plant maintains a pressure drop in excess of 14 in. W.G. to satisfy particulate emission limitations. Cleaned process condensate from the urea synthesis operation is used as make-up scrubber liquid. Specific gravity is monitored to maintain the liquor at 45 to 50 percent urea in the scrubber. Liquor blow-down is returned to the synthesis operation for urea recovery. The designers of the granulation process guarantee emissions of urea particulate to no more than 10 lb/hr from the scrubber exhaust. Some ammonia can be expected in the additive into the urea melt before solidification, formaldehyde may also be present.

FACILITY OPERATION DURING TESTING:

GCA monitored and recorded process and control equipment operating parameters during sampling of the solution and granulation processes by TRC to ensure that samples were collected only during representative, steady-state process operation.

Table 21 presents a list of 30 parameters monitored during testing. The raw data are presented in Appendix J of this report along with summaries of the parameter values.

Table 22 presents a summary of the test runs conducted by TRC, indicating the date and times of testing, the time period over which the process and control

TABLE 21. PARAMETERS MONITORED DURING TESTING AT AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Item No. | Parameter                     | Indication of: | Appendix Column Heading | Conversion factor       | Units                                | Comments                     |
|----------|-------------------------------|----------------|-------------------------|-------------------------|--------------------------------------|------------------------------|
| 1        | Urea Solution Feed Tank Level | PR,PS*         | TK-101                  |                         |                                      | See Note 1                   |
| 2        | Additive Feed Rate            | PS,E*          | AFR                     | Reading x 100 (approx.) | #/HR                                 |                              |
| 3        | Urea Melt Temperature         | PS,E*          | UMT                     | None                    | $\Delta^0\text{F}$ from design valve | Design value is confidential |
| 4        | "A" Granulator Spray Pressure | PR,PS*         | GSP-A                   | None                    | psig                                 |                              |
| 5        | "B" Granulator Spray Pressure | PR,PS*         | GSP-B                   | None                    | psig                                 |                              |
| 6        | "C" Granulator Spray Pressure | PR,PS*         | GSP-C                   | None                    | psig                                 |                              |

(continued)

TABLE 21 (continued).

| Item No. | Parameter   | Indication of: | Appendix Column Heading | Conversion factor | Units                             | Comments   |
|----------|---|----------------|-------------------------|-------------------|-----------------------------------|--|
| 7        | "A" Granulator Inlet Air Temperature              | PS*            | AIGT                    | None              | $\Delta$ °F from standard valve   | Standard value arbitrarily chosen to protect confidentiality of this parameter |
| 8        | "A" Granulator Outlet Air Temperature             | PS*            | AOGT                    | None              | $\Delta$ °F from standard valve   | Same as Above  |
| 9        | Temperature of Urea Solids Exiting "A" Granulator | PS*            | UST                     | None              | °F                                | See Note 2   |
| 10       | "A" Granulator Scrubber Liquor Level              | SO, E*         | SIL                     | None              | % of total gauge                  | See Note 3   |
| 11       | "A" Granulator Scrubber Fan Amperage              | SO*            | SFA                     | None              | Amps                              | Non-recording dial instrument  |
| 12       | "A" Granulator Drive Motor Amperage               | PS*            | GMA                     | None              | $\Delta$ amps from standard valve | Standard value arbitrarily chosen to protect confidentiality of this parameter |

(continued)

TABLE 21 (continued).

| Item No. | Parameter                                     | Indication of: | Appendix Column Heading | Conversion factor         | Units | Comments  |
|----------|---|----------------|-------------------------|---------------------------|-------|---|
| 13       | Weight of Total Solids Leaving "A" Granulator | PR, PS*        | TWOD                    | Reading $\times$<br>1230  | #/HR  | Weigh belt indicator                                |
| 14       | Product Weight to Warehouse                   | PR, PS*        | PWTW                    | Reading $\times$<br>358.8 | #/HR  | Weigh belt indication - sum of three granulators    |
| 15       | "A" Granulator Total Outlet Solids Weight     | PR, PS*        | SOWT-A                  | Reading $\times$<br>0.05  | tons  | Totalizer for TWOD above                            |
| 16       | "B" Granulator Total Outlet Solids Weight     | PR, PS*        | SOWT-B                  | Reading $\times$<br>0.05  | tons  | Totalizer   |
| 17       | "C" Granulator Total Outlet Solids Weight     | PR, PS*        | SOWT-C                  | Reading $\times$<br>0.05  | tons  | Totalizer   |
| 18       | "A" Granulator Product Weight                 | PR, PS*        | PWTA                    | Reading $\times$<br>0.05  | tons  | Totalizer for weigh belt on product leaving screens |

(continued)

TABLE 21 (continued).

| Item No. | Parameter  | Indication of: | Appendix Column Reading | Conversion factor       | Units        | Comments  |
|----------|--|----------------|-------------------------|-------------------------|--------------|---|
| 19       | "B" Granulator Product Weight                    | PR, PS*        | PWTB                    | Reading $\times$ 0.05   | tons         | Totalizer for weigh belt on product leaving screens |
| 20       | "C" Granulator Product Weight                    | PR, PS*        | PWTC                    | Reading $\times$ 0.05   | tons         | Totalizer for weigh belt on product leaving screens |
| 21       | "A" Granulator Scrubber Liquor Temperature       | SO, E*         | SLT                     | None                    | $^{\circ}$ F | Dial thermometer at scrubber                        |
| 22       | "A" Granulator Scrubber Liquor Feed Rate         | SO, E*         | ISLF                    | None                    | gpm          | Dial instrument at scrubber liquor feed line        |
| 23       | "A" Granulator Scrubber Exit Air Temperature     | SO*            | AOS                     | None                    | $^{\circ}$ F | Dial thermometer at scrubber outlet duct            |
| 24       | CO <sub>2</sub> Feed Rate Urea Synthesis Process | PR, PS*        | CO <sub>2</sub> Feed    | Reading $\times$ 80,000 | SCFH         | See Note 4  |

(continued)

TABLE 21 (continued).

| Item No. | Parameter   | Indication of: | Appendix Column Heading | Conversion factor | Units                             | Comments                                |
|----------|---|----------------|-------------------------|-------------------|-----------------------------------|---|
| 25       | "Dirty" Steam Exhaust Flow Rate from Urea Synthesis                         | E*             | DSEF                    | Reading x 5,000   | #/HR                              | Strip Chart                             |
| 26       | "Dirty" Steam Exhaust Valve Actuator and % Valve Open                       | PS*            | DSEP / valve cond.      | See Note 5        | See Note 5                        |   |
| 27       | NH <sub>3</sub> Feed Rate to Urea Synthesis Process                         | PR, PS*        | NH <sub>3</sub> Feed    | Reading x 12.4    | % of Urea Process Design Capacity | Design capacity is 1000TPD of 100% Urea |
| 28       | Vacuum on No. 1 Evaporator  | PS, E*         | EV-1                    | None              | mm Hg vacuum                      |   |
| 29       | Vacuum on No. 2 Evaporator  | PS, E*         | EV-2                    | None              | mm Hg vacuum                      |   |
| 30       | Temperature of Circulating H <sub>2</sub> O to Top of Low Pressure Scrubber | PS, E*         | TI 16                   | None              | °F                                |   |

(continued)

TABLE 21 (concluded).

NOTES

1. This tank is located before the concentrators. In addition to the liquid level in the tank, the concentration is necessary for determination of production rate. However, the liquid level alone indicates increase or decrease in production rate.
2. Inspection of the sensing device indicated that it was not in intimate contact with the urea solids and was, therefore, not indicating actual solids temperature.
3. Scrubber liquid level is controlled by an overflow weir. The weir is set when the scrubber is not operating. Once the scrubber is operating, the liquid level indicator is used as a gross indication of scrubber liquor feed pump operation (i.e., level will fall if pump fails).
4. The meter conversion factor of 80,000 applies for CO<sub>2</sub> at 257° F and 2100 psig which are conditions applicable to the discharge stream of compressors at this plant.
5. These valves are monitored by plant personnel as an additional indication of the amount of "dirty" steam being vented. "Dirty" steam is at 295.9° F and 48 psig.

\*KEY

|     |                      |
|-----|----------------------|
| PR: | Process Rate         |
| PS: | Process Stability    |
| E:  | Emission Fluctuation |
| SO: | Scrubber Operation   |

TABLE 22. SUMMARY OF TESTING AT AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Test type                  |        | Date     | Sampling period | Process parameter averaging period | No. of data points in averaging period | Test designation |
|----------------------------|--------|----------|-----------------|------------------------------------|--|------------------|
| Urea particulate Run 1     | Inlet  | 10/10/78 | 1130 to 1415    | 1130 to 1410                       | 17                                     | UP-1-I           |
|                            | Outlet | 10/10/78 | 1131 to 1336    | 1130 to 1340                       | 14                                     | UP-1-O           |
| Urea particulate Run 2     | Inlet  | 10/10/78 | 1500 to 1700    | 1500 to 1700                       | 13                                     | UP-2-I           |
|                            | Outlet | 10/10/78 | 1531 to 1737    | 1530 to 1730                       | 13                                     | UP-2-O           |
| Urea particulate Run 3     | Inlet  | 10/11/78 | 1616 to 1832    | 1620 to 1820                       | 13                                     | UP-3-I           |
|                            | Outlet | 10/11/78 | 1616 to 1832    | 1620 to 1820                       | 13                                     | UP-3-O           |
| Formaldehyde Run 1         | Inlet  | 10/11/78 | 0920 to 1040    | 0920 to 1030                       | 8                                      | F-1-I            |
|                            | Outlet | 10/11/78 | 0918 to 1028    | 0920 to 1030                       | 8                                      | F-1-O            |
| Formaldehyde Run 2         | Inlet  | 10/11/78 | 1120 to 1242    | 1120 to 1240                       | 9                                      | F-2-I            |
|                            | Outlet | 10/11/78 | 1129 to 1237    | 1130 to 1240                       | 8                                      | F-2-O            |
| Formaldehyde Run 3         | Inlet  | 10/11/78 | 1335 to 1516    | 1325 to 1515                       | 11                                     | F-3-I            |
|                            | Outlet | 10/11/78 | 1348 to 1515    | 1345 to 1515                       | 9                                      | F-3-O            |
| Particle size Run 1        | Inlet  | 10/12/78 | 0919 to 0920    | 0915 to 0932                       | 2                                      | PS-1-I           |
|                            | Outlet | 10/12/78 | 1109 to 1509    | 1059 to 1500                       | 9                                      | PS-1-O           |
| Particle size Run 2        | Inlet  | 10/13/78 | 1316 to 1317    | 1300 to 1325                       | 2                                      | PS-2-I           |
|                            | Outlet | 10/12/78 | 1629 to 2029    | 1630 to 2030                       | 9                                      | PS-2-O           |
| Particle size Run 3        | Inlet  | 10/13/78 | 1508 to 1509    | 1500 to 1515                       | 2                                      | PS-3-I           |
|                            | Outlet | 10/13/78 | 0855 to 1255    | 0915 to 1300                       | 9                                      | PS-3-O           |
| Urea synthesis tower Run 1 | Outlet | 10/13/78 | 0930 to 0945    | 0921 to 0945                       | 2                                      | ST-1-O           |
| Urea synthesis tower Run 2 | Outlet | 10/13/78 | 1030 to 1046    | 0955 to 1057                       | 2                                      | ST-2-O           |
| Urea synthesis tower Run 3 | Outlet | 10/13/78 | 1130 to 1145    | 1125 to 1143                       | 2                                      | ST-3-O           |

equipment parameters were averaged, and assignment of test designations.

Table 23 is a summary of the data monitored and indicates the variations of the parameters during each of the test runs. The columns titled "Maximum Value % Deviation" and "Minimum Value % Deviation" present the difference between the highest and lowest values observed and the average value of the parameter for that run, expressed as a percent of the average value.

Table 24 presents the variation of the average value of each parameter between tests as a percent deviation from the overall average of that parameter for all test runs of the designation.

The average values of each parameter are presented in Appendix J.

#### I. GRANULATOR PARAMETERS MONITORED

Of the 30 parameters monitored, items 1 through 9 and 12 through 20 in Table 21 were recorded specifically as indicators of the granulator production rate, process stability, and possible, emission fluctuation during testing. The third column of Table 21 designates what each parameter indicates.

In addition to the instrument data recorded by GCA, TRC collected samples from various portions of the granulation facility, whose analysis should aid in characterizing the process operation. These samples include: a grab sample of the molten urea feed to the granular for analysis of temperature, pH, percent urea, percent ammonia, and percent formaldehyde; a grab sample of the product leaving the granular for analysis of temperature, moisture, percent urea, percent ammonia, and percent formaldehyde; a second grab sample of the product leaving the granulator for sieve size analysis, and; a grab sample of the additive retained for future analysis.

As indicated by the data in Tables 23 and 24 the granulator ran quite steady during each test run and showed little variation when parameters are compared between runs.

TABLE 23. PARAMETER DEVIATIONS DURING TESTING AT AGRICO  
CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| TEST DESIGNATION UP-1-1               |               | 'DATE 10/10/78  |                                       | TIME 1130 TO 1410                    |                                     |
|---------------------------------------|---------------|---|---------------------------------------|--------------------------------------|-------------------------------------|
| Process parameter                     | Appendix code | Units   | Standard deviation                    | Maximum value                        | Minimum value                       |
|                                       |               |   | % deviation                           | % deviation                          | % deviation                         |
| Urea 70% solution tank level          | TK-101        | % of tank   | 0.33                                  | 1.0%                                 | 0.9%                                |
| Additive feed rate                    | AFR           | lb/hr   | 9.93                                  | 2.9%                                 | 2.4%                                |
| Urea melt temperature                 | UMT           | $\Delta^{\circ}\text{F}$ from design value <sub>1</sub>   | -3.50 $^{\circ}\text{F}$ <sup>5</sup> | -50 $^{\circ}\text{F}$ <sup>6</sup>  | -10 $^{\circ}\text{F}$ <sup>6</sup> |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 1.18                                  | 2.7%                                 | 8.5%                                |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | 1.98                                  | 3.9%                                 | 13.6%                               |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 1.72                                  | 2.9%                                 | 12.0%                               |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +0.50 $^{\circ}\text{F}$ <sup>5</sup> | +30 $^{\circ}\text{F}$ <sup>6</sup>  | -10 $^{\circ}\text{F}$ <sup>6</sup> |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +8.80 $^{\circ}\text{F}$ <sup>5</sup> | +150 $^{\circ}\text{F}$ <sup>6</sup> | +20 $^{\circ}\text{F}$ <sup>6</sup> |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | Standard deviation                        | Maximum value        | Minimum value       |
|--|----------------------|---|---|----------------------|---------------------|
|  |                      |   | % deviation                               | % deviation          | % deviation         |
| Liquid level in "A" granulator scrubber                | SLL                  | N.A. <sup>3</sup>                       | 0.08                                      | 2.8%                 | 2.5%                |
| "A" Granulator scrubber fan amps                       | SFA                  | amps                                    | 1.5                                       | 3.6%                 | 3.6%                |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sub>2</sub> | +2.7 amps <sup>5</sup>                    | +6 amps <sup>6</sup> | 0 amps <sup>6</sup> |
| Liquor temperature in "A" granulator scrubber          | SLT                  | °F                                      | 0.59                                      | 0.6%                 | 1.1%                |
| Liquor feed rate to "A" granulator scrubber            | ISLF                 | gpm                                     | 0.24                                      | 0.3%                 | 4.7%                |
| "A" Granulator scrubber exit air temperature           | AOS                  | °F                                      | 0.64                                      | 1.4%                 | 1.0%                |
| Production rate of urea synthesis process <sup>4</sup> | NH <sub>3</sub> feed | ton/day of 99.5% urea solution          | (Parameter not recorded during this test) |                      |                     |

(continued)

TABLE 23 (continued). TEST DESIGNATION UP-1-0 DATE 10/10/78 TIME 1130 TO 1340

| Process parameter                     | Appendix code | Units   | Standard deviation                   | Maximum value                       | Minimum value                        |
|---------------------------------------|---------------|---|--------------------------------------|-------------------------------------|--------------------------------------|
|                                       |               |   | % deviation                          | % deviation                         | % deviation                          |
| Urea 70% solution tank level          | TK-101        | % of tank   | 0.32                                 | 1.0%                                | 0.8%                                 |
| Additive feed rate                    | AFR           | lb/hr   | 8.70                                 | 2.4%                                | 2.1%                                 |
| Urea melt temperature                 | UMT           | $\Delta^{\circ}\text{F}$ from design value <sup>1</sup>   | -3.7 $^{\circ}\text{F}$ <sup>5</sup> | -5 $^{\circ}\text{F}$ <sup>6</sup>  | -3 $^{\circ}\text{F}$ <sup>6</sup>   |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 1.24                                 | 1.9%                                | 8.2%                                 |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | 2.08                                 | 4.6%                                | 13.1%                                |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 1.83                                 | 3.4%                                | 11.6%                                |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^{\circ}\text{F}$ from standard value <sup>2</sup> | +0.6 $^{\circ}\text{F}$ <sup>5</sup> | +3 $^{\circ}\text{F}$ <sup>6</sup>  | -0.5 $^{\circ}\text{F}$ <sup>6</sup> |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^{\circ}\text{F}$ from standard value <sup>2</sup> | +7.9 $^{\circ}\text{F}$ <sup>5</sup> | +14 $^{\circ}\text{F}$ <sup>6</sup> | +2 $^{\circ}\text{F}$ <sup>6</sup>   |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | Standard deviation     | Maximum value        | Minimum value       | % deviation | % deviation                               |
|--|----------------------|---|------------------------|----------------------|---------------------|-------------|---|
| Liquid level in "A" granulator scrubber                | SLL                  | N.A. <sup>3</sup>                       | 0.07                   | 3.6%                 | 1.8%                |             |   |
| "A" Granulator scrubber fan amps                       | SFA                  | amps                                    | 1.40                   | 3.4%                 | 2.3%                |             |   |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sub>2</sub> | +2.6 amps <sup>5</sup> | +6 amps <sup>6</sup> | 0 amps <sup>6</sup> |             |   |
| Liquor temperature in "A" granulator scrubber          | SLT                  | °F                                      | 0.59                   | 0.5%                 | 1.2%                |             |   |
| Liquor feed rate to "A" granulator scrubber            | ISLF                 | gpm                                     | 0                      | 0                    | 0                   |             |   |
| "A" Granulator scrubber AOS exit air temperature       | OF                   |   | 0.59                   | 1.3%                 | 1.0%                |             |   |
| Production rate of urea synthesis process <sub>4</sub> | NH <sub>3</sub> feed | ton/day of 99.5% urea solution          |                        |                      |                     |             | (Parameter not recorded during this test) |

(continued)

TABLE 23 (continued). TEST DESIGNATION UP-2-I DATE 10/10/78 TIME 1500 TO 1700

| Process parameter                     | Appendix code | Units   | Standard deviation       | Maximum value           | Minimum value           | % deviation | % deviation |
|---------------------------------------|---------------|---|--------------------------|-------------------------|-------------------------|-------------|-------------|
| Urea 70% solution tank level          | TK-101        | % of tank   | 0.88                     | 2.1%                    | 2.6%                    |             |             |
| Additive feed rate                    | AFR           | lb/hr   | 12.79                    | 3.5%                    | 3.3%                    |             |             |
| Urea melt temperature                 | UNT           | $\Delta^{\circ}\text{F}$ from design value <sup>1</sup>   | -4.2 $^{\circ}\text{F}$  | -6 $^{\circ}\text{F}$   | -20 $^{\circ}\text{F}$  |             |             |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 0.63                     | 2.1%                    | 2.9%                    |             |             |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | 1.11                     | 4.1%                    | 6.0%                    |             |             |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 0.89                     | 3.0%                    | 4.5%                    |             |             |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^{\circ}\text{F}$ from standard value <sup>2</sup> | -0.08 $^{\circ}\text{F}$ | +2.5 $^{\circ}\text{F}$ | -1.5 $^{\circ}\text{F}$ |             |             |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^{\circ}\text{F}$ from standard value <sup>2</sup> | +4.2 $^{\circ}\text{F}$  | +10 $^{\circ}\text{F}$  | 0 $^{\circ}\text{F}$    |             |             |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | Standard deviation                        | Maximum value        | Minimum value       |
|--|----------------------|---|---|----------------------|---------------------|
|  |                      |   | % deviation                               | % deviation          | % deviation         |
| Liquid level in "A" granulator scrubber                | SLI.                 | N.A. 3                                  | 0.07                                      | 1.8%                 | 3.5%                |
| "A" Granulator scrubber SFA fan amps                   |                      | amps                                    | 1.01                                      | 2.2%                 | 2.2%                |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sup>2</sup> | +3.1 amps <sup>5</sup>                    | +5 amps <sup>6</sup> | 0 amps <sup>6</sup> |
| Liquor temperature in "A" granulator scrubber          | S.I.T                | °F                                      | 0.30                                      | 0.8%                 | 0.3%                |
| Liquor feed rate to "A" granulator scrubber            | TSLF                 | gpm                                     | 0.49                                      | 3.1%                 | 2.1%                |
| "A" Granulator scrubber exit air temperature           | AOS                  | °F                                      | 0.27                                      | 1.1%                 | 0.1%                |
| Production rate of urea synthesis process <sub>4</sub> | NH <sub>3</sub> feed | ton/day of 99.5% urea solution          | (Parameter not recorded during this test) |                      |                     |

(continued)

TABLE 23 (continued). TEST DESIGNATION UP-2-0 DATE 10/10/78 TIME 1530 TO 1730

| Process parameter                     | Appendix code | Units   | Standard deviation                    | Maximum value                        | Minimum value                        |
|---------------------------------------|---------------|---|---------------------------------------|--------------------------------------|--------------------------------------|
|                                       |               |   | % deviation                           | % deviation                          | % deviation                          |
| Urea 70% solution tank level          | TK-101        | % of tank   | 0.91                                  | 3.4%                                 | 2.3%                                 |
| Additive feed rate                    | AFR           | lb/hr   | 13.94                                 | 3.3%                                 | 3.5%                                 |
| Urea melt temperature                 | UMT           | $\Delta^{\circ}\text{F}$ from design value <sup>1</sup>   | -3.8 $^{\circ}\text{F}$ <sup>5</sup>  | -6 $^{\circ}\text{F}$ <sup>6</sup>   | -2 $^{\circ}\text{F}$ <sup>6</sup>   |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 0.58                                  | 2.3%                                 | 2.7%                                 |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | 0.99                                  | 4.7%                                 | 5.5%                                 |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 0.79                                  | 3.5%                                 | 4.1%                                 |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^{\circ}\text{F}$ from standard value <sup>2</sup> | -0.08 $^{\circ}\text{F}$ <sup>5</sup> | +2.5 $^{\circ}\text{F}$ <sup>6</sup> | -1.5 $^{\circ}\text{F}$ <sup>6</sup> |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^{\circ}\text{F}$ from standard value <sup>2</sup> | +4.77 $^{\circ}\text{F}$ <sup>5</sup> | +10 $^{\circ}\text{F}$ <sup>6</sup>  | +2 $^{\circ}\text{F}$ <sup>6</sup>   |

(continued)

TABLE 23 (continued).

| Process parameter                             | Appendix code        | Units                                   | Standard deviation     | Maximum value                             | Minimum value       |
|---|----------------------|---|------------------------|---|---------------------|
|   |                      |   | % deviation            | % deviation                               | % deviation         |
| Liquid level in "A" granulator scrubber       | SIL                  | N.A. <sup>3</sup>                       | 0.07                   | 1.8%                                      | 3.5%                |
| "A" Granulator scrubber fan amps              | SPA                  | amps                                    | 1.00                   | 2.0%                                      | 2.3%                |
| "A" Granulator drive motor amps               | GMA                  | A amps from standard value <sub>2</sub> | +3.1 amps <sup>5</sup> | +6 amps <sup>6</sup>                      | 0 amps <sup>6</sup> |
| Liquor temperature in "A" granulator scrubber | SLT                  | °F                                      | 0.30                   | 0.8%                                      | 0.3%                |
| Liquor feed rate to "A" granulator scrubber   | ISLF                 | gpm                                     | 0.36                   | 4.2%                                      | 1.0%                |
| "A" Granulator scrubber exit air temperature  | AOS                  | °F                                      | 0                      | 0   | 0                   |
| Production rate of urea synthesis process     | NH <sub>3</sub> feed | ton/day of urea                         | 99.5%                  | (Parameter not recorded during this test) | urea solution       |

(continued)

TABLE 23 (continued). TEST DESIGNATION UP-3-I and UP-3-O DATE 10/11/78 TIME 1620 TO 1820

| process parameter                     | Appendix code | Units   | Standard deviation                   | Maximum value                       | Minimum value                       | deviation % | deviation % |
|---------------------------------------|---------------|---|--------------------------------------|-------------------------------------|-------------------------------------|-------------|-------------|
| Urea 70% solution tank level          | TK-101        | % of tank   | 0.58                                 | 5.6%                                | 5.3%                                |             |             |
| Additive feed rate                    | AFR           | lb/hr   | 14.87                                | 3.2%                                | 4.8%                                |             |             |
| Urea melt temperature                 | UMT           | $\Delta^{\circ}\text{F}$ from design value <sub>1</sub>   | -4.2 $^{\circ}\text{F}$ <sup>5</sup> | -7 $^{\circ}\text{F}$ <sup>6</sup>  | -10 $^{\circ}\text{F}$ <sup>6</sup> |             |             |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 0.38                                 | 1.6%                                | 2.6%                                |             |             |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | 0.69                                 | 2.2%                                | 4.1%                                |             |             |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 0.57                                 | 2.5%                                | 3.4%                                |             |             |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +9.1 $^{\circ}\text{F}$ <sup>5</sup> | +11 $^{\circ}\text{F}$ <sup>6</sup> | +7 $^{\circ}\text{F}$ <sup>6</sup>  |             |             |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +5.0 $^{\circ}\text{F}$ <sup>5</sup> | +9 $^{\circ}\text{F}$ <sup>6</sup>  | 0 $^{\circ}\text{F}$ <sup>6</sup>   |             |             |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | Standard deviation     | Maximum value        | Minimum value       | Deviation % |
|--|----------------------|---|------------------------|----------------------|---------------------|-------------|
| Liquid level in "A" granulator scrubber                | SLI                  | N.A. <sup>3</sup>                       | 0.04                   | 0.4%                 | 12.0%               |             |
| "A" Granulator scrubber fan amps                       | SFA                  | amps                                    | 0.83                   | 2.7%                 | 1.54%               |             |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sub>2</sub> | +2.7 amps <sup>5</sup> | +6 amps <sup>6</sup> | 0 amps <sup>6</sup> |             |
| Liquor temperature in "A" granulator scrubber          | SLT                  | °F                                      | 0.31                   | 0.8%                 | 0.3%                |             |
| Liquor feed rate to "A" granulator scrubber            | ISLF                 | gpm                                     | 0                      | 0                    | 0                   |             |
| "A" Granulator scrubber exit air temperature           | AOS                  | °F                                      | 0.63                   | 1.7%                 | 0.6%                |             |
| Production rate of urea synthesis process <sup>4</sup> | NH <sub>3</sub> feed | ton/day of 99.5% urea solution          | 6.99                   | 1.0%                 | 1.3%                |             |

(continued)

TABLE 23 (continued). TEST DESIGNATION F-1-0 DATE 10/11/78 TIME 0920 TO 1030

| Process parameter                     | Appendix code | Units   | Standard deviation                    | Maximum value                        | Minimum value                      | % deviation | % deviation |
|---------------------------------------|---------------|---|---------------------------------------|--------------------------------------|------------------------------------|-------------|-------------|
| Urea 70% solution tank level          | TK-101        | % of tank   | 0.85                                  | 6.1%                                 | 4.5%                               |             |             |
| Additive feed rate                    | AFR           | lb/hr   | 7.04                                  | 1.9%                                 | 2.0%                               |             |             |
| Urea melt temperature                 | UMT           | $\Delta^{\circ}\text{F}$ from design value <sub>1</sub>   | -3.5 $^{\circ}\text{F}$ <sub>5</sub>  | -4 $^{\circ}\text{F}$ <sub>6</sub>   | -3 $^{\circ}\text{F}$ <sub>6</sub> |             |             |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 0.30                                  | 1.1%                                 | 1.4%                               |             |             |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | 0.35                                  | 1.3%                                 | 1.3%                               |             |             |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 0.35                                  | 1.2%                                 | 1.2%                               |             |             |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +1.75 $^{\circ}\text{F}$ <sub>5</sub> | +3.5 $^{\circ}\text{F}$ <sub>6</sub> | 0 $^{\circ}\text{F}$ <sub>6</sub>  |             |             |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +0.38 $^{\circ}\text{F}$ <sub>5</sub> | +2 $^{\circ}\text{F}$ <sub>6</sub>   | -2 $^{\circ}\text{F}$ <sub>6</sub> |             |             |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | Standard deviation | Maximum value    | Minimum value |
|--|----------------------|---|--------------------|------------------|---------------|
|  |                      |   | % deviation        | % deviation      | % deviation   |
| Liquid level in "A" granulator scrubber                | SLI                  | N.A. 3                                  | 0.03               | 0                | 1.8%          |
| "A" Granulator scrubber fan amps                       | SFA                  | amps                                    | 0.93               | 2.7%             | 1.5%          |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sup>2</sup> | +2.0               | amps 5 +5 amps 6 | 0 amps 6      |
| Liquor temperature in "A" granulator scrubber          | SLT                  | °F                                      | 0.25               | 0.2%             | 0.3%          |
| Liquor feed rate to "A" granulator scrubber            | ISLF                 | gpm                                     | 0.33               | 0.5%             | 4.3%          |
| "A" Granulator scrubber exit air temperature           | AOS                  | °F                                      | 0                  | 0                | 0             |
| Production rate of urea synthesis process <sub>4</sub> | NI <sub>3</sub> feed | ton/day of 99.5% urea solution          | 9.42               | 1.1%             | 1.1%          |

(continued)

TABLE 23 (continued). TEST DESIGNATION F-2-I DATE 10/11/78 TIME 1120 TO 1240

| Process parameter                     | Appendix code | Units   | Standard deviation         | Maximum value              | Minimum value             |
|---------------------------------------|---------------|---|----------------------------|----------------------------|---------------------------|
|                                       |               |   |                            | % deviation                | % deviation               |
| Urea 70% solution tank level          | TK-101        | % of tank   | 1.18                       | 7.7%                       | 7.7%                      |
| Additive feed rate                    | AFR           | lb/hr   | 10.48                      | 1.7%                       | 3.0%                      |
| Urea melt temperature                 | UNT           | $\Delta^{\circ}\text{F}$ from design value <sub>1</sub>   | -4.1 $^{\circ}\text{F}$ 5  | -50 $^{\circ}\text{F}$ 6   | -20 $^{\circ}\text{F}$ 6  |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 0.34                       | 0.7%                       | 1.8%                      |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | 0.58                       | 1.7%                       | 2.2%                      |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 0.62                       | 1.2%                       | 3.5%                      |
| "A" Granulator inlet air temperature  | AICT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +5.11 $^{\circ}\text{F}$ 5 | +7.50 $^{\circ}\text{F}$ 6 | +3.0 $^{\circ}\text{F}$ 6 |
| "A" Granulator outlet air temperature | AOCT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +1.67 $^{\circ}\text{F}$ 5 | +7.00 $^{\circ}\text{F}$ 6 | -1 $^{\circ}\text{F}$ 6   |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | Standard deviation     | Maximum value        | Minimum value       |
|--|----------------------|---|------------------------|----------------------|---------------------|
|  |                      |   | % deviation            | % deviation          | % deviation         |
| Liquid level in "A" granulator scrubber                | SLL                  | N.A. <sup>3</sup>                       | 0.08                   | 1.8%                 | 3.5%                |
| "A" Granulator scrubberSFA fan amps                    |                      | amps                                    | 0.94                   | 2.9%                 | 1.4%                |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sup>2</sup> | +2.8 amps <sup>5</sup> | +5 amps <sup>6</sup> | 0 amps <sup>6</sup> |
| Liquor temperature in SLT "A" granulator scrubber      | o <sub>F</sub>       |   | 0.50                   | 0.4%                 | 1.2%                |
| Liquor feed rate to "A" granulator scrubber            | ISLF                 | gpm                                     | 0                      | 0                    | 0                   |
| "A" Granulator scrubber exit air temperature           | AOS                  | o <sub>F</sub>                          | 0.50                   | 0.5%                 | 0.7%                |
| Production rate of urea synthesis <sub>4</sub> process | NH <sub>3</sub> feed | ton/day of 99.5% urea solution          | 4.11                   | 0.3%                 | 0.8%                |

TABLE 23 (continued). TEST DESIGNATION F-2-0 DATE 10/11/78 TIME 1130 TO 1140

| Process parameter                     | Appendix code | Units   | Standard deviation         | Maximum value             | Minimum value             |
|---------------------------------------|---------------|---|----------------------------|---------------------------|---------------------------|
|                                       |               |   | % deviation                | % deviation               | % deviation               |
| Urea 70% solution tank level          | TK-101        | % of tank   | 1.12                       | 8.8%                      | 6.8%                      |
| Additive feed rate                    | AFR           | lb/hr   | 10.61                      | 1.6%                      | 3.1%                      |
| Urea melt temperature                 | UMT           | $\Delta^{\circ}\text{F}$ from design value <sub>1</sub>   | -4.0 $^{\circ}\text{F}$ 5  | -5 $^{\circ}\text{F}$ 6   | -2 $^{\circ}\text{F}$ 6   |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 0.35                       | 0.8%                      | 1.7%                      |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | 0.56                       | 1.9%                      | 1.9%                      |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 0.63                       | 1.3%                      | 3.4%                      |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +5.63 $^{\circ}\text{F}$ 5 | +7.5 $^{\circ}\text{F}$ 6 | +3.5 $^{\circ}\text{F}$ 6 |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +1 $^{\circ}\text{F}$ 5    | +4 $^{\circ}\text{F}$ 6   | -1 $^{\circ}\text{F}$ 6   |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | Standard deviation     | Maximum value        | Minimum value       |
|--|----------------------|---|------------------------|----------------------|---------------------|
|  |                      |   | % deviation            | % deviation          | % deviation         |
| Liquid level in "A" granulator scrubber                | SLL                  | N.A. <sup>3</sup>                       | 0.03                   | 1.8%                 | 0%                  |
| "A" Granulator fan amps                                | SFA                  | amps                                    | 0.66                   | 1.7%                 | 1.1%                |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sup>2</sup> | +2.6 amps <sup>5</sup> | +5 amps <sup>6</sup> | 0 amps <sup>6</sup> |
| Liquor temperature in "A" granulator scrubber          | SLT                  | o <sub>F</sub>                          | 0.35                   | 0.3%                 | 0.8%                |
| Liquor feed rate to "A" granulator scrubber            | ISLF                 | gpm                                     | 0                      | 0                    | 0                   |
| "A" Granulator exit air temperature                    | AOS                  | o <sub>F</sub>                          | 0.48                   | 0.5%                 | 0.7%                |
| Production rate of urea synthesis process <sup>4</sup> | NH <sub>3</sub> feed | ton/day of 99.5% urea solution          | 4.24                   | 0.3%                 | 0.8%                |

(continued)

TABLE 23 (continued). TEST DESIGNATION F-3-1 DATE 10/11/78 TIME 1325 TO 1515

| Process parameter                     | Appendix code | Units   | Standard deviation                   | Maximum value                        | Minimum value                       |
|---------------------------------------|---------------|---|--------------------------------------|--------------------------------------|-------------------------------------|
|                                       |               |   | % deviation                          | % deviation                          | % deviation                         |
| Urea 70% solution tank level          | TK-101        | % of tank   | 1.14                                 | 14.6%                                | 11.6%                               |
| Additive feed rate                    | AFR           | lb/hr   | 5.14                                 | 0.6%                                 | 1.7%                                |
| Urea melt temperature                 | URT           | $\Delta^{\circ}\text{F}$ from design value <sup>1</sup>   | -4.1 $^{\circ}\text{F}$ <sup>5</sup> | -7 $^{\circ}\text{F}$ <sup>6</sup>   | -30 $^{\circ}\text{F}$ <sup>6</sup> |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 0.26                                 | 1.1%                                 | 1.3%                                |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | 0.33                                 | 1.1%                                 | 1.3%                                |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 0.31                                 | 1.1%                                 | 1.6%                                |
| "A" Granulator inlet air temperature  | AI GT         | $\Delta^{\circ}\text{F}$ from standard value <sup>2</sup> | +7.8 $^{\circ}\text{F}$ <sup>5</sup> | +9.5 $^{\circ}\text{F}$ <sup>6</sup> | +5 $^{\circ}\text{F}$ <sup>6</sup>  |
| "A" Granulator outlet air temperature | AO GT         | $\Delta^{\circ}\text{F}$ from standard value <sup>2</sup> | +8.5 $^{\circ}\text{F}$ <sup>5</sup> | +20 $^{\circ}\text{F}$ <sup>6</sup>  | 0 $^{\circ}\text{F}$ <sup>6</sup>   |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | Standard deviation     | Maximum value        | Minimum value        |
|--|----------------------|---|------------------------|----------------------|----------------------|
|  |                      |   | % deviation            | % deviation          | % deviation          |
| Liquid level in "A" granulator scrubber                | SLI                  | N.A. <sup>3</sup>                       | 0.09                   | 3.5%                 | 1.8%                 |
| "A" Granulator scrubber fan amps                       | SFA                  | amps                                    | 1.07                   | 2.0%                 | 2.3%                 |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sup>2</sup> | +3.2 amps <sup>5</sup> | +5 amps <sup>6</sup> | +1 amps <sup>6</sup> |
| Liquor temperature in "A" granulator scrubber          | SLT                  | °F                                      | 0.39                   | 0.8%                 | 0.9%                 |
| Liquor feed rate to "A" granulator scrubber            | ISLF                 | g pm                                    | 0.94                   | 5.8%                 | 3.8%                 |
| "A" Granulator scrubber exit air temperature           | AOS                  | °F                                      | 0.50                   | 0.6%                 | 0.6%                 |
| Production rate of urea synthesis process <sup>4</sup> | NH <sub>3</sub> feed | ton/day of 99.5% urea solution          | 8.00                   | 1.7%                 | 1.1%                 |

(continued)

TABLE 23 (continued) . TEST DESIGNATION F-3-0 DATE 10/11/78 TIME 1345 TO 1515

| Process parameter                     | Appendix code | Units   | Standard deviation       | Maximum value            | Minimum value          |
|---------------------------------------|---------------|---|--------------------------|--------------------------|------------------------|
|                                       |               |   | % deviation              | % deviation              | % deviation            |
| Urea 70% solution tank level          | TK-101        | % of tank                                     | 0.84                     | 10.8%                    | 9.3%                   |
| Additive feed rate                    | AFR           | lb/hr   | 5.50                     | 0.6%                     | 1.7%                   |
| Urea melt temperature                 | UMT           | $\Delta^0 F$ from design value <sub>1</sub>   | -4.2 $^0 F$ <sup>5</sup> | -7 $^0 F$ <sup>6</sup>   | -3 $^0 F$ <sup>6</sup> |
| "A" Granulator spray nozzle pressure  | CSP-A         | psig  | 0.28                     | 1.1%                     | 1.4%                   |
| "B" Granulator spray nozzle pressure  | CSP-B         | psig  | 0.37                     | 1.1%                     | 1.3%                   |
| "C" Granulator spray nozzle pressure  | CSP-C         | psig  | 0.33                     | 0.8%                     | 1.6%                   |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^0 F$ from standard value <sub>2</sub> | +8.1 $^0 F$ <sup>5</sup> | +9.5 $^0 F$ <sup>6</sup> | +6 $^0 F$ <sup>6</sup> |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^0 F$ from standard value <sub>2</sub> | +7.2 $^0 F$ <sup>5</sup> | +20 $^0 F$ <sup>6</sup>  | 0 $^0 F$ <sup>6</sup>  |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | N.A. <sup>3</sup> | Standard deviation     | Maximum value        | Minimum value        |
|--|----------------------|---|-------------------|------------------------|----------------------|----------------------|
|  |                      |   |                   | % deviation            | % deviation          | % deviation          |
| Liquid level in "A" granulator scrubber                | SLL                  |   | 0.09              | 3.5%                   | 3.5%                 | 1.8%                 |
| "A" Granulator scrubber SFA fan amps                   |                      | amps                                    |                   | 1.17                   | 2.0%                 | 2.3%                 |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sub>2</sub> |                   | +3.4 amps <sup>5</sup> | +5 amps <sup>6</sup> | +1 amps <sup>6</sup> |
| Liquor temperature in "A" granulator scrubber          | SLT                  | °F                                      |                   | 0.28                   | 0.6%                 | 0.4%                 |
| Liquor feed rate to "A" granulator scrubber            | ISLF                 | gpm                                     |                   | 0.94                   | 4.8%                 | 4.8%                 |
| "A" Granulator scrubber exit air temperature           | AOS                  | °F                                      |                   | 0.50                   | 0.7%                 | 0.4%                 |
| Production rate of urea synthesis process <sup>4</sup> | NH <sub>3</sub> feed | ton/day of 99.5% urea solution          |                   | 5.19                   | 0.8%                 | 0.9%                 |

(continued)

TABLE 23 (continued). TEST DESIGNATION PS-1-I DATE 10/12/78 TIME 0915 TO 0932

| Process parameter                     | Appendix code | Units   | Standard deviation                   | Maximum value                        | Minimum value                       |
|---------------------------------------|---------------|---|--------------------------------------|--------------------------------------|-------------------------------------|
| Urea 70% solution tank level          | TK-101        | % of tank   | 0.25                                 | 1.5%                                 | 1.5%                                |
| Additive feed rate                    | AFR           | lb/hr   | 40.0                                 | 6.6%                                 | 6.6%                                |
| Urea melt temperature                 | UMT           | $\Delta^{\circ}\text{F}$ from design value <sup>1</sup>   | -4.0 $^{\circ}\text{F}$ <sup>5</sup> | -50 $^{\circ}\text{F}$ <sup>6</sup>  | -30 $^{\circ}\text{F}$ <sup>6</sup> |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 0                                    | 0                                    | 0                                   |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | (Not operating during this test)     |                                      |                                     |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 0                                    | 0                                    | 0                                   |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^{\circ}\text{F}$ from standard value <sup>2</sup> | +8.0 $^{\circ}\text{F}$ <sup>5</sup> | +90 $^{\circ}\text{F}$ <sup>6</sup>  | +70 $^{\circ}\text{F}$ <sup>6</sup> |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^{\circ}\text{F}$ from standard value <sup>2</sup> | +8.5 $^{\circ}\text{F}$ <sup>5</sup> | +160 $^{\circ}\text{F}$ <sup>6</sup> | +10 $^{\circ}\text{F}$ <sup>6</sup> |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | Standard deviation   | Maximum value        | Minimum value        |
|--|----------------------|---|----------------------|----------------------|----------------------|
|  |                      |   | % deviation          | % deviation          | % deviation          |
| Liquid level in "A" granulator scrubber                | SLL                  | N.A. <sup>3</sup>                       | 0                    | 0                    | 0                    |
| "A" Granulator scrubber fan amps                       | SFA                  | amps                                    | 0.5                  | 0.7%                 | 0.7%                 |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sup>2</sup> | +2 amps <sup>5</sup> | +2 amps <sup>6</sup> | +2 amps <sup>6</sup> |
| Liquor temperature in "A" granulator scrubber          | SLT                  | °F                                      | 0                    | 0                    | 0                    |
| Liquor feed rate to "A" granulator scrubber            | ISLF                 | gpm                                     | 0                    | 0                    | 0                    |
| "A" Granulator scrubber exit air temperature           | AOS                  | °F                                      | 0                    | 0                    | 0                    |
| Production rate of urea synthesis process <sup>4</sup> | NH <sub>3</sub> feed | ton/day of 99.5% urea solution          | 0                    | 0                    | 0                    |

(continued)

TABLE 23 (continued). TEST DESIGNATION PS-1-0 DATE 10/12/78 TIME 1059 TO 1500

| Process parameter                     | Appendix code | Units   | Standard deviation         | Maximum value                    | Minimum value              | % deviation | % deviation |
|---------------------------------------|---------------|---|----------------------------|----------------------------------|----------------------------|-------------|-------------|
| Urea 70% solution tank level          | TK-101        | % of tank   | N.V.*                      | N.V.*                            | N.V.*                      |             |             |
| Additive feed rate                    | AFR           | lb/hr   | 58.21                      | 13.6%                            |                            | 27.4%       |             |
| Urea melt temperature                 | UMT           | $\Delta^{\circ}\text{F}$ from design value <sub>1</sub>   | -5.3 $^{\circ}\text{F}$ 5  | -12 $^{\circ}\text{F}$ 6         | -3 $^{\circ}\text{F}$ 6    |             |             |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 1.71                       | 5.0%                             |                            | 7.8%        |             |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  |                            | (Not operating during this test) |                            |             |             |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 1.70                       | 4.7%                             |                            | 8.2%        |             |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +12.4 $^{\circ}\text{F}$ 5 | +15.5 $^{\circ}\text{F}$ 6       | +10.5 $^{\circ}\text{F}$ 6 |             |             |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +7.9 $^{\circ}\text{F}$ 5  | +18 $^{\circ}\text{F}$ 6         | +3 $^{\circ}\text{F}$ 6    |             |             |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | Standard deviation     | Maximum value          | Minimum value         |
|--|----------------------|---|------------------------|------------------------|-----------------------|
|  |                      |   | % deviation            | % deviation            | % deviation           |
| Liquid level in "A" granulator scrubber                | SLL                  | N.A. <sup>3</sup>                       | 0                      | 0                      | 0                     |
| "A" Granulator fan amps                                | SFA                  | amps                                    | 1.23                   | 2.6%                   | 3.2%                  |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sub>2</sub> | +4.0 amps <sup>5</sup> | +4.6 amps <sup>6</sup> | + 1 amps <sup>6</sup> |
| Liquor temperature in "A" granulator scrubber          | SLT                  | °F                                      | 0.67                   | 0.8%                   | 1.3%                  |
| Liquor feed rate to "A" granulator scrubber            | ISLF                 | gpm                                     | 1.40                   | 18.8%                  | 5.9%                  |
| "A" Granulator scrubber AOS exit air temperature       | OF                   |   | 1.05                   | 1.8%                   | 1.4%                  |
| Production rate of urea synthesis process <sub>4</sub> | NH <sub>3</sub> feed | ton/day of 99.5% urea solution          | 0                      | 0                      | 0                     |

(continued)

TABLE 23 (continued). TEST DESIGNATION PS-2-1 DATE 10/13/78 TIME 1300 TO 1325

| Process parameter                     | Appendix code | Units   | Standard deviation         | Maximum value             | Minimum value           | % deviation               | % deviation             |
|---------------------------------------|---------------|---|----------------------------|---------------------------|-------------------------|---------------------------|-------------------------|
| Urea 70% solution tank level          | TK-101        | % of tank   | 0                          | 0                         | 0                       | 0                         | 0                       |
| Additive feed rate                    | AFR           | lb/hr   | 2.5                        | 0.4%                      | 0.4%                    | 0.4%                      | 0.4%                    |
| Urea melt temperature                 | UMT           | $\Delta^{\circ}\text{F}$ from design value <sub>1</sub>   | -3 $^{\circ}\text{F}^5$    | -3 $^{\circ}\text{F}^6$   | -3 $^{\circ}\text{F}^6$ | -3 $^{\circ}\text{F}^6$   | -3 $^{\circ}\text{F}^6$ |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 0.25                       | 0.7%                      | 0.7%                    | 0.7%                      | 0.7%                    |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | 0.25                       | 0.8%                      | 0.8%                    | 0.8%                      | 0.8%                    |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 0.25                       | 0.7%                      | 0.7%                    | 0.7%                      | 0.7%                    |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +0.25 $^{\circ}\text{F}^5$ | +0.5 $^{\circ}\text{F}^6$ | 0 $^{\circ}\text{F}^6$  | +0.5 $^{\circ}\text{F}^6$ | 0 $^{\circ}\text{F}^6$  |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | -3 $^{\circ}\text{F}^5$    | -4 $^{\circ}\text{F}^6$   | -2 $^{\circ}\text{F}^6$ | -4 $^{\circ}\text{F}^6$   | -2 $^{\circ}\text{F}^6$ |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | Standard deviation   | Maximum value        | Minimum value        |
|--|----------------------|---|----------------------|----------------------|----------------------|
|  |                      |   | % deviation          | % deviation          | % deviation          |
| Liquid level in "A" granulator scrubber                | SL                   | N.A. <sup>3</sup>                       | 0                    | 0                    | 0                    |
| "A" Granulator scrubber fan amps                       | SFA                  | amps                                    | 0                    | 0                    | 0                    |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sup>2</sup> | +3 amps <sup>5</sup> | +4 amps <sup>6</sup> | +2 amps <sup>6</sup> |
| Liquid temperature in "A" granulator scrubber          | SLT                  | °F                                      | 0                    | 0                    | 0                    |
| Liquor feed rate to "A" granulator scrubber            | ISLF                 | gpm                                     | 0                    | 0                    | 0                    |
| "A" Granulator scrubber exit air temperature           | AOS                  | °F                                      | 0.5                  | 0.6%                 | 0.6%                 |
| Production rate of urea synthesis process <sup>4</sup> | NH <sub>3</sub> feed | ton/day of 99.5% urea solution          | 3.0                  | 0.3%                 | 0.3%                 |

(continued)

TABLE 23 (continued). TEST DESIGNATION PS-2-0 DATE 10/12/78 TIME 1630 TO 2030

| Process parameter                     | Appendix code | Units   | Standard deviation                    | Maximum value                       | Minimum value                        |
|---------------------------------------|---------------|---|---------------------------------------|-------------------------------------|--------------------------------------|
|                                       |               |   | % deviation                           | % deviation                         | % deviation                          |
| Urea 70% solution tank level          | TK-101        | % of tank   | 1.36                                  | 7.2%                                | 11.0%                                |
| Additive feed rate                    | AFR           | lb/hr   | 34.57                                 | 8.3%                                | 15.6%                                |
| Urea melt temperature                 | UMT           | $\Delta^{\circ}\text{F}$ from design value <sub>1</sub>   | -4.4 $^{\circ}\text{F}$ <sup>5</sup>  | -5 $^{\circ}\text{F}$ <sup>6</sup>  | -4 $^{\circ}\text{F}$ <sup>6</sup>   |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 1.58                                  | 7.3%                                | 5.3%                                 |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | 2.74                                  | 12.2%                               | 10.3%                                |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 2.18                                  | 9.9%                                | 8.0%                                 |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +10.6 $^{\circ}\text{F}$ <sup>5</sup> | +13 $^{\circ}\text{F}$ <sup>6</sup> | +8.0 $^{\circ}\text{F}$ <sup>6</sup> |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +5.1 $^{\circ}\text{F}$ <sup>5</sup>  | +9 $^{\circ}\text{F}$ <sup>6</sup>  | +2 $^{\circ}\text{F}$ <sup>6</sup>   |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | Standard deviation     | Maximum value        | Minimum value        |
|--|----------------------|---|------------------------|----------------------|----------------------|
|  |                      |   | % deviation            | % deviation          | % deviation          |
| Liquid level in "A" granulator scrubber                | SLL                  | N.A. <sup>3</sup>                       | 0                      | 0                    | 0                    |
| "A" Granulator scrubber fan amps                       | SFA                  | amps                                    | 0.96                   | 2.3%                 | 2.3%                 |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sub>2</sub> | +3.2 amps <sup>5</sup> | +5 amps <sup>6</sup> | +1 amps <sup>6</sup> |
| Liquor temperature in "A" granulator scrubber          | SLT                  | °F                                      | 0.31                   | 0.1%                 | 1.0%                 |
| Liquor feed rate to "A" granulator scrubber            | ISI.F                | gpm                                     | 0                      | 0                    | 0                    |
| "A" Granulator scrubber exit air temperature           | AOS                  | °F                                      | 0.31                   | 0.1%                 | 1.0%                 |
| Production rate of urea synthesis process <sup>4</sup> | NH <sub>3</sub> feed | ton/day of 99.5% urea solution          | 10.84                  | 1.2%                 | 1.7%                 |

(continued)

TABLE 23 (continued). TEST DESIGNATION PS-3-1 DATE 10/13/78 TIME 1500 TO 1515

| Process parameter                     | Appendix code | Units   | Standard deviation       | Maximum value         | Minimum value           | Maximum % deviation       | Minimum % deviation       |
|---------------------------------------|---------------|---|--------------------------|-----------------------|-------------------------|---------------------------|---------------------------|
| Urea 70% solution tank level          | TK-101        | % of tank   | 0                        | 0                     | 0                       | 0                         | 0                         |
| Additive feed rate                    | AFR           | lb/hr   | 0                        | 0                     | 0                       | 0                         | 0                         |
| Urea melt temperature                 | UMT           | $\Delta^{\circ}\text{F}$ from design value <sub>1</sub>   | -4.5 $^{\circ}\text{F}$  | -6 $^{\circ}\text{F}$ | -3 $^{\circ}\text{F}$   | -6 $^{\circ}\text{F}^6$   | -3 $^{\circ}\text{F}^6$   |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 0.25                     | 0.25                  | 0.25                    | 0.7%                      | 0.7%                      |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | 0.25                     | 0.25                  | 0.25                    | 0.8%                      | 0.8%                      |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 0                        | 0                     | 0                       | 0                         | 0                         |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +2.25 $^{\circ}\text{F}$ | +3 $^{\circ}\text{F}$ | +1.5 $^{\circ}\text{F}$ | +1.5 $^{\circ}\text{F}^6$ | +1.5 $^{\circ}\text{F}^6$ |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | -0.5 $^{\circ}\text{F}$  | -1 $^{\circ}\text{F}$ | -0.5 $^{\circ}\text{F}$ | -1 $^{\circ}\text{F}$     | -1 $^{\circ}\text{F}$     |

(continued)

TABLE 23 (continued).

| Process parameter  | Appendix code        | Units                                   | Standard deviation   | Maximum value        | Minimum value        |
|--|----------------------|---|----------------------|----------------------|----------------------|
|  |                      |   | % deviation          | % deviation          | % deviation          |
| Liquid level in "A" granulator scrubber                  | SI.L                 | N.A. 3                                  | 0                    | 0                    | 0                    |
| "A" Granulator scrubber fan amps                         | SPA                  | amps                                    | 0                    | 0                    | 0                    |
| "A" Granulator drive motor amps                          | GMA                  | Δ amps from standard value <sup>2</sup> | +3 amps <sup>5</sup> | +4 amps <sup>6</sup> | +2 amps <sup>6</sup> |
| Liquor temperature in "A" granulator scrubber            | SLT                  | °F                                      | 0.5                  | 0.6%                 | 0.6%                 |
| Liquor feed rate to "A" granulator scrubber              | ISLF                 | gpm                                     | 0                    | 0                    | 0                    |
| "A" Granulator scrubber exit air temperature             | AOS                  | °F                                      | 0.5                  | 0.6%                 | 0.6%                 |
| Production rate of urea synthesis processes <sub>4</sub> | NH <sub>3</sub> feed | ton/day of 99.5% urea solution          | 3.0                  | 0.3%                 | 0.3%                 |

(continued)

TABLE 23 (continued). TEST DESIGNATION PS-3-0 DATE 10/13/78 TIME 0915 TO 1300

| Process parameter                     | Appendix code | Units   | Standard deviation                   | Maximum value                        | Minimum value                       |
|---------------------------------------|---------------|---|--------------------------------------|--------------------------------------|-------------------------------------|
|                                       |               |   | % deviation                          | % deviation                          | % deviation                         |
| Urea 70% solution tank level          | TK-101        | % of tank   | 0.50                                 | 6.4%                                 | 2.7%                                |
| Additive feed rate                    | AFR           | lb/hr   | 14.53                                | 4.2%                                 | 3.9%                                |
| Urea melt temperature                 | UNT           | $\Delta^{\circ}\text{F}$ from design value <sub>1</sub>   | -4.1 $^{\circ}\text{F}$ <sup>5</sup> | -8 $^{\circ}\text{F}$ <sup>6</sup>   | -30 $^{\circ}\text{F}$ <sup>6</sup> |
| "A" Granulator spray nozzle pressure  | GSP-A         | psig  | 0.33                                 | 0.5%                                 | 2.2%                                |
| "B" Granulator spray nozzle pressure  | GSP-B         | psig  | 0.67                                 | 2.1%                                 | 3.9%                                |
| "C" Granulator spray nozzle pressure  | GSP-C         | psig  | 0.48                                 | 3.7%                                 | 3.4%                                |
| "A" Granulator inlet air temperature  | AIGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | +1.1 $^{\circ}\text{F}$ <sup>5</sup> | +2.5 $^{\circ}\text{F}$ <sup>6</sup> | 0 $^{\circ}\text{F}$ <sup>6</sup>   |
| "A" Granulator outlet air temperature | AOGT          | $\Delta^{\circ}\text{F}$ from standard value <sub>2</sub> | -3.2 $^{\circ}\text{F}$ <sup>5</sup> | -7 $^{\circ}\text{F}$ <sup>6</sup>   | +2 $^{\circ}\text{F}$ <sup>6</sup>  |

(continued)

TABLE 23 (continued).

| Process parameter                                      | Appendix code        | Units                                   | Standard deviation     | Maximum value        | Minimum value        |
|--|----------------------|---|------------------------|----------------------|----------------------|
|  |                      |   | % deviation            | % deviation          | % deviation          |
| Liquid level in "A" granulator scrubber                | SLL                  | N.A. <sup>3</sup>                       | 0                      | 0                    | 0                    |
| "A" Granulator scrubber fan amps                       | SFA                  | amps                                    | 1.20                   | 3.0%                 | 2.7%                 |
| "A" Granulator drive motor amps                        | GMA                  | Δ amps from standard value <sub>2</sub> | +3.1 amps <sup>5</sup> | +5 amps <sup>6</sup> | +1 amps <sup>6</sup> |
| Liquor temperature in "A" granulator scrubber          | SLT                  | °F                                      | 0.42                   | 0.9%                 | 0.2%                 |
| Liquor feed rate to "A" granulator scrubber            | ISLF                 | gpm                                     | 0                      | 0                    | 0                    |
| "A" Granulator scrubber exit air temperature           | AOS                  | °F                                      | 0.57                   | 1.1%                 | 1.3%                 |
| Production rate of urea synthesis process <sup>4</sup> | NH <sub>3</sub> feed | tcn/day of 99.5% urea solution          | 6.18                   | 0.4%                 | 1.2%                 |

(continued)

TABLE 23 (continued). TEST DESIGNATION ST-1-0 DATE 10/13/78 TIME 0921 TO 0945

| Process parameter   | Appendix code        | Units           | Standard deviation | Maximum value | Minimum value | % deviation |
|---|----------------------|-----------------|--------------------|---------------|---------------|-------------|
| CO <sub>2</sub> feed rate to urea synthesis process                         |                      | SCFH            | 2,000              | 0.3%          | 0.3%          | 0.3%        |
| "Dirty" steam exhaust flow rate from urea synthesis process                 | DSEF                 | lb/hr           | 0                  | 0             | 0             | 0           |
| "Dirty" steam exhaust valve actuator and % valve open                       | DSEP/valve cond      | psig/% open     | 0/2.5              | 0/9.1%        | 0/9.1%        | 0           |
| NH <sub>3</sub> feed rate to urea synthesis process                         | NH <sub>3</sub> feed | ton/day of urea | 3.0                | 0.3%          | 0.3%          | 0           |
| Vacuum on No. 1 evaporator  | EV-1                 | mm Hg           | 2.5                | 0.7%          | 0.7%          | 0           |
| Vacuum on No. 2 evaporator  | EV-2                 | mm Hg           | 5.0                | 0.8%          | 0.8%          | 0           |
| Temperature of circulating H <sub>2</sub> O to top of low pressure scrubber | TI 16                | °F              | 0                  | 0             | 0             | 0           |

(continued)

TABLE 23 (continued). TEST DESIGNATION ST-2-0 DATE 10/13/78 TIME 0955 TO 1057

| Process parameter   | Appendix code          | Units           | Standard deviation | Maximum value | Minimum value |
|---|------------------------|-----------------|--------------------|---------------|---------------|
|   |                        |                 | % deviation        | % deviation   | % deviation   |
| CO <sub>2</sub> feed rate to urea synthesis process                         | CO <sub>2</sub> feed   | SCFH            | 0                  | 0             | 0             |
| "Dirty" steam exhaust flow rate from urea synthesis process                 | DSEF                   | lb/hr           | 0                  | 0             | 0             |
| "Dirty" steam exhaust valve actuator and cond % valve open                  | DSEP/valve psig/% open | 0/2.5           | 0/9.1%             | 0/9.1%        | 0/9.1%        |
| NH <sub>3</sub> feed rate to urea synthesis process                         | NH <sub>3</sub> feed   | ton/day of urea | 0                  | 0             | 0             |
| Vacuum on No. 1 evaporator  | EV-1                   | mm Hg           | 2.5                | 0.7%          | 0.7%          |
| Vacuum on No. 2 evaporator  | EV-2                   | mm Hg           | 0                  | 0             | 0             |
| Temperature of circulating H <sub>2</sub> O to top of low pressure scrubber | TR 16                  | °F              | 1°F                | 0.9%          | 0.9%          |

(continued)

TABLE 23 (continued). TEST DESIGNATION ST-3-0 DATE 10/13/78 TIME 1125 TO 1143

| Process parameter   | Appendix code        | Units             |        | Standard deviation | Maximum value | Minimum value | % deviation |
|---|----------------------|-------------------|--------|--------------------|---------------|---------------|-------------|
| CO <sub>2</sub> feed rate to urea synthesis process                         | CO <sub>2</sub> feed | SCFH              | 0      | 0                  | 0             | 0             | 0           |
| "Dirty" steam exhaust flow rate from urea synthesis process                 | DSEF                 | lb/hr             | 0      | 0                  | 0             | 0             | 0           |
| "Dirty" steam exhaust valve actuator and % valve open                       | DSEP                 | psi/g/% open cond | 0.25/0 | 0.6%/0             | 0.6%/0        | 0.6%/0        | 0.6%/0      |
| NH <sub>3</sub> feed rate to urea synthesis process                         | NH <sub>3</sub> feed | ton/day of urea   | 0      | 0                  | 0             | 0             | 0           |
| Vacuum on No. 1 evaporator  | EV-1                 | mm Hg             | 2.5    | 0.7%               | 0.7%          | 0.7%          | 0.7%        |
| Vacuum on No. 2 evaporator  | EV-2                 | mm Hg             | 2.5    | 0.4%               | 0.4%          | 0.4%          | 0.4%        |
| Temperature of circulating H <sub>2</sub> O to top of low pressure scrubber | T1 16                | °F                | 0      | 0                  | 0             | 0             | 0           |

(continued)

TABLE 23 (concluded).

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<sup>1</sup>N.V. = Not Valid. Because only two granulators were operating, level in this tank increased from 20.6 to 28.5 during this test.

NOTES:

<sup>1</sup>Design value considered confidential.

<sup>2</sup>Standard value arbitrarily chosen by Agrico to protect confidentiality of this parameter.

<sup>3</sup>This parameter used by plant personnel as indicator of scrubber status. Translation to specific units was not available.

<sup>4</sup>Control board instrument reading multiplied by 12.4 equals production rate of synthesis process as percent of design capacity. Percent of design capacity multiplied by 1000 ton/day equals ton/day production rate.

<sup>5</sup>Average deviation from design or standard value.

<sup>6</sup>Values presented are maximum and minimum deviations from design or standard value observed during test.

TABLE 24. PARAMETER DEVIATIONS BETWEEN TESTS AT AGRICO  
CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Process<br>Parameter                             | Test Designation |        |        |                  |        | PS-3-0 |
|--|------------------|--------|--------|------------------|--------|--------|
|  | PS-1-1           | PS-2-1 | PS-3-1 | PS-1-0           | PS-2-0 |        |
| Additive Feed Rate                               | +7.4%            | 0%     | -1.5%  | -4.7%            | 0%     | -1.1%  |
| "A" Granulator Spray Nozzle<br>Pressure          | +5.0%            | -4.8%  | -3.5%  | +2.6%            | +4.0%  | -3.4%  |
| "B" Granulator Spray Nozzle<br>Pressure          | not<br>operating | -5.6%  | -2.7%  | not<br>operating | +10.9% | -2.6%  |
| "C" Granulator Spray Nozzle<br>Pressure          | +8.0%            | -7.5%  | -5.5%  | +4.4%            | +5.6%  | -5.0%  |
| Liquid Level in "A" Granulator<br>Scrubber       | 0%               | 0%     | 0%     | 0%               | 0%     | 0%     |
| "A" Granulator Scrubber Fan<br>Amps              | +1.4%            | -0.7%  | +2.1%  | -1.8%            | -1.6%  | +0.6%  |
| Liquor Temperature in "A"<br>Granulator Scrubber | +0.2%            | -3.1%  | -1.4%  | +3.7%            | +3.4%  | -2.9%  |
| Liquor Feed Rate to "A"<br>Granulator Scrubber   | +19.8%           | -5.2%  | -5.2%  | +0.8%            | -5.2%  | -5.2%  |
| "A" Granulator Scrubber Exit<br>Air Temperature  | +0.7%            | -3.3%  | -2.1%  | +4.4%            | +4.0%  | -3.7%  |
| Production Rate of Urea<br>Synthesis Process     | +0.6%            | +1.4%  | -2.0%  | +0.6%            | -1.7%  | +1.2%  |

(continued)

TABLE 24 (continued). UREA PARTICULATE TEST RUNS

| Process Parameter                             | Test Designation |        |        |        |        | UP-3-0 |
|---|------------------|--------|--------|--------|--------|--------|
|   | UP-1-1           | UP-2-1 | UP-3-1 | UP-1-0 | UP-2-0 |        |
| Additive Feed Rate                            | +2.3%            | +0.9%  | -3.2%  | +2.0%  | +1.1%  | -3.2%  |
| "A" Granulator Spray Nozzle Pressure          | +4.2%            | +3.6%  | -7.5%  | +3.8%  | +3.4%  | -7.5%  |
| "B" Granulator Spray Nozzle Pressure          | +8.1%            | +6.5%  | -13.9% | +7.4%  | +5.9%  | -13.9% |
| "C" Granulator Spray Nozzle Pressure          | +6.5%            | +5.1%  | -11.1% | +6.0%  | +4.6%  | -11.1% |
| Liquid Level in "A" Granulator Scrubber       | -1.2%            | +0.6%  | +0.6%  | -1.2%  | +0.6%  | +0.6%  |
| "A" Granulator Scrubber Fan Amps              | -0.3%            | -0.3%  | +0.2%  | -0.2%  | -0.2%  | +0.2%  |
| Liquor Temperature in "A" Granulator Scrubber | -0.8%            | -1.6%  | +2.3%  | -0.7%  | -1.6%  | +2.3%  |
| Liquor Feed Rate to "A" Granulator Scrubber   | +2.5%            | 0%     | -2.1%  | +3.0%  | -1.1%  | -2.1%  |
| "A" Granulator Scrubber Exit Air Temperature  | -0.8%            | -1.6%  | +2.4%  | -0.7%  | -1.7%  | +2.4%  |
| Production Rate of Urea Synthesis Process     | N.R.             | N.R.   | N.R.   | N.R.   | N.R.   | N.R.   |

TABLE 24 (continued). FORMALDEHYDE TEST RUNS

| Process Parameter                             | Test Designation |       |       |       |       | F-3-0 |
|---|------------------|-------|-------|-------|-------|-------|
|   | F-1-1            | F-2-1 | F-3-1 | F-1-0 | F-2-0 |       |
| Additive Feed Rate                            | +0.3%            | -0.3% | 0%    | +0.3% | -0.2% | 0%    |
| "A" Granulator Spray Nozzle Pressure          | -0.9%            | -0.5% | +1.5% | -0.9% | -0.6% | +1.5% |
| "B" Granulator Spray Nozzle Pressure          | -1.5%            | -0.7% | +2.3% | -1.5% | -0.9% | +2.3% |
| "C" Granulator Spray Nozzle Pressure          | +1.1%            | +1.1% | -2.1% | +1.1% | +0.9% | -2.1% |
| Liquid Level in "A" Granulator Scrubber       | 0%               | 0%    | 0%    | 0%    | 0%    | 0%    |
| "A" Granulator Scrubber Fan Amps              | +1.3%            | -0.3% | -0.8% | +1.3% | -0.6% | -0.8% |
| Liquor Temperature in "A" Granulator Scrubber | -2.0%            | 0%    | +1.9% | -2.0% | +0.1% | +2.0% |
| Liquor Feed Rate to "A" Granulator Scrubber   | +1.5%            | -2.9% | +1.0% | +1.5% | -2.9% | +1.9% |
| "A" Granulator Scrubber Exit Air Temperature  | -2.7%            | +0.3% | +2.4% | -2.7% | +0.3% | +2.3% |
| Production Rate of Urea Synthesis Process     | +0.9%            | 0%    | -0.8% | +0.9% | 0%    | -1.1% |

TABLE 24 (continued). SYNTHESIS TOWER TEST RUNS

| Process Parameter   | Test Designation |             |             |
|---|------------------|-------------|-------------|
|   | ST-1-0           | ST-2-0      | ST-3-0      |
| CO <sub>2</sub> Feed Rate to Urea Synthesis Process                         | -0.2%            | +0.1%       | +0.1%       |
| "Dirty" Steam Exhaust Flow Rate from Urea Synthesis Process                 | 0%               | 0%          | 0%          |
| "Dirty" Steam Exhaust Valve Actuator and % valve open                       | -0.2%/-2.9%      | -0.2%/-2.9% | +0.4%/+5.9% |
| NH <sub>3</sub> Feed Rate to Urea Synthesis Process                         | -0.2%            | +0.1%       | +0.1%       |
| Vacuum on No. 1 Evaporator  | +2.4%            | -0.5%       | -1.9%       |
| Vacuum on No. 2 Evaporator  | -0.6%            | +0.1%       | +0.5%       |
| Temperature of Circulating H <sub>2</sub> O to top of Low Pressure Scrubber | -2.0%            | -2.0%       | +4.0%       |

TABLE 24 (concluded). PRODUCTION RATES DURING TESTING - AGRICO

| Test type                 | Test designation | Date   | Time period         |              | Production |         |
|---------------------------|------------------|--------|---------------------|--------------|------------|---------|
|                           |                  |        | of rate calculation | Tons         | Ton/minute | Ton/day |
| Urea particulate<br>Run 1 | Inlet            | UP-1-I | 10/10/78            | 1130 to 1410 | 44.07      | 0.2754  |
|                           | Outlet           | UP-1-0 | 10/10/78            | 1130 to 1340 | 35.40      | 0.2723  |
| Urea particulate<br>Run 2 | Inlet            | UP-2-I | 10/10/78            | 1500 to 1700 | 32.22      | 0.2685  |
|                           | Outlet           | UP-2-0 | 10/10/78            | 1530 to 1730 | 32.59      | 0.2716  |
| Urea particulate<br>Run 3 | Inlet            | UP-3-I | 10/11/78            | 1620 to 1820 | 29.18      | 0.2431  |
|                           | Outlet           | UP-3-0 | 10/11/78            | 1620 to 1820 | 29.18      | 0.2431  |
| Formaldehyde<br>Run 1     | Inlet            | F-1-I  | 10/11/78            | 0920 to 1030 | 19.29      | 0.2756  |
|                           | Outlet           | F-1-0  | 10/11/78            | 0920 to 1030 | 19.29      | 0.2756  |
| Formaldehyde<br>Run 2     | Inlet            | F-2-I  | 10/11/78            | 1120 to 1240 | 22.20      | 0.2775  |
|                           | Outlet           | F-2-0  | 10/11/78            | 1130 to 1240 | 19.43      | 0.2776  |
| Formaldehyde<br>Run 3     | Inlet            | F-3-I  | 10/11/78            | 1325 to 1515 | 31.98      | 0.2908  |
|                           | Outlet           | F-3-0  | 10/11/78            | 1345 to 1515 | 26.08      | 0.2898  |
| Particle size<br>Run 1    | Inlet            | PS-1-I | 10/12/78            | 0915 to 0932 | 4.92       | 0.2892  |
|                           | Outlet           | PS-1-0 | 10/12/78            | 1059 to 1500 | 68.04      | 0.2835  |
| Particle size<br>Run 2    | Inlet            | PS-2-I | 10/13/78            | 1300 to 1325 | 6.32       | 0.2529  |
|                           | Outlet           | PS-2-0 | 10/12/78            | 1630 to 2030 | 65.75      | 0.2740  |
| Particle size<br>Run 3    | Inlet            | PS-3-I | 10/13/78            | 1500 to 1515 | 3.84       | 0.2560  |
|                           | Outlet           | PS-3-0 | 10/13/78            | 0915 to 1300 | 55.49      | 0.2466  |

The plant personnel indicated that the factor which had the greatest influence on quantities of "dust" entering the scrubber was the additive to urea ratio. The data indicates some variance in this ratio and therefore the dust loading into the scrubber is expected to be somewhat higher when the ratio is smaller.

## II. CONTROL EQUIPMENT PARAMETERS MONITORED

Of the 30 parameters monitored, items 10, 11 and 21 through 23 in Table 21 were recorded specifically as indicators of the granulator scrubber operation during testing.

In addition to the instrument data recorded by GCA, TRC collected samples from various portions of the scrubber solution, whose analysis should aid in characterizing the process operation. These samples include: composite samples of the scrubber solution entering and leaving the scrubber for analysis of urea, ammonia and formaldehyde content and the pressure drop across the scrubber.

As was the case with the granulator, the process parameters listed in Table 21 for the granulator scrubber showed little variation during or between runs. The only significant change was the rate of liquor fed to the scrubber during test PS-1-I. As requested in Table 16, the scrubber pressure drop did vary considerably during each test. This was probably due to variation in scrubber liquid level, however, this could not be confirmed by the recorded process data.

## III. SOLUTION PROCESS

Items 24 through 30 in Table 21 were monitored during testing of the solution process vent as indicators of process stability and rate.

No significant changes in the parameters during or between tests is discernable from the data.

#### IV. PRODUCTION RATE DETERMINATION

This plant is typical of the industry in that it does not determine "instantaneous" production rates. A calculation of daily production is made each day by applying conversion factors to the quantity of ammonia consumed by the urea synthesis plant for a 24-hour period. All solid urea is weighed as it leaves the plant. Thus, a rough comparison between what should have been produced and what was actually shipped can be obtained. This comparison is sufficiently accurate to satisfy the company's requirements.

Plant operators monitor three major parameters which give them an indication of overall plant rate. These parameters are ammonia feed rate, 70 percent urea tank level, and the spray pressure of the granulator nozzles.

Other instrumentation which is available to monitor production rates at this plant consists solely of weigh belts. Weigh belts monitor the amount of unscreened solids leaving each granulator separately, the amount of screened product leaving each of the three lines separately, and the combined total amount of screened product from all three granulators being conveyed to the warehouse.

With the exception of the weigh belt measuring total combined product to the warehouse, the weigh belt rates were indicated on totalizers as well as strip charts. The total product weigh belt rate was recorded only on a strip chart. Production rates recorded during testing are presented in Table 25.

Plant personnel were quick to state that the accuracy of any production rate calculated from a weigh belt indication could not be assured. The inaccuracy results from the inherent design of the weigh belt and accumulation of urea solids which fall off the belt onto the device. Plant personnel cleaned the accumulated solids off the "A" Granulator weigh belt devices in anticipation of testing.

TABLE 25. PRODUCTION RATES DURING TESTING AT AGRICO  
CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

| Test type                 | Test designation | Date   | Time period of rate calculation |              | Production |         |
|---------------------------|------------------|--------|---------------------------------|--------------|------------|---------|
|                           |                  |        | Tons                            | Ton/minute   | Ton/day    | Ton/day |
| Urea particulate<br>Run 1 | Inlet            | UP-1-I | 10/10/78                        | 1130 to 1410 | 44.07      | 397     |
|                           | Outlet           | UP-1-0 | 10/10/78                        | 1130 to 1340 | 35.40      | 392     |
| Urea particulate<br>Run 2 | Inlet            | UP-2-I | 10/10/78                        | 1500 to 1700 | 32.22      | 387     |
|                           | Outlet           | UP-2-0 | 10/10/78                        | 1530 to 1730 | 32.59      | 391     |
| Urea particulate<br>Run 3 | Inlet            | UP-3-I | 10/11/78                        | 1620 to 1820 | 29.18      | 350     |
|                           | Outlet           | UP-3-0 | 10/11/78                        | 1620 to 1820 | 29.18      | 350     |
| Formaldehyde<br>Run 1     | Inlet            | F-1-I  | 10/11/78                        | 0920 to 1030 | 19.29      | 397     |
|                           | Outlet           | F-1-0  | 10/11/78                        | 0920 to 1030 | 19.29      | 397     |
| Formaldehyde<br>Run 2     | Inlet            | F-2-I  | 10/11/78                        | 1120 to 1240 | 22.20      | 400     |
|                           | Outlet           | F-2-0  | 10/11/78                        | 1130 to 1240 | 19.43      | 400     |
| Formaldehyde<br>Run 3     | Inlet            | F-3-I  | 10/11/78                        | 1325 to 1515 | 31.98      | 419     |
|                           | Outlet           | F-3-0  | 10/11/78                        | 1345 to 1515 | 26.08      | 417     |
| Particle size<br>Run 1    | Inlet            | PS-1-I | 10/12/78                        | 0915 to 0932 | 4.92       | 0.2892  |
|                           | Outlet           | PS-1-0 | 10/12/78                        | 1059 to 1500 | 68.04      | 0.2898  |
| Particle size<br>Run 2    | Inlet            | PS-2-I | 10/13/78                        | 1300 to 1325 | 6.32       | 0.2529  |
|                           | Outlet           | PS-2-0 | 10/12/78                        | 1630 to 2030 | 65.75      | 0.2740  |
| Particle size<br>Run 3    | Inlet            | PS-3-I | 10/13/78                        | 1500 to 1515 | 3.84       | 0.2560  |
|                           | Outlet           | PS-3-0 | 10/13/78                        | 0915 to 1300 | 55.49      | 0.2466  |

On Wednesday, 11 October 1978, a 1-1/2 hour production rate test was conducted in an effort to determine the accuracy of the various production rate measuring methods. This test was conducted between 1300 and 1430. At 1300 all three granulators were placed on total recycle. The readings on each of the three totalizers recording screened product from each line to the warehouse were recorded at that time. When the strip chart for the weigh belt recording total combined screened product to the warehouse reached zero, the granulators were returned to normal operation. The reading of zero indicated a blank space on the product belt.

When the blank space reached the warehouse, an operator diverted the belt from the warehouse floors such that all subsequent product coming down the belt was loaded directly into a tared railcar. Until approximately 1430, all product leaving the granulators was loaded directly into this railcar. When the railcar approached capacity, the granulators were again placed on total recycle, totalizer readings recorded, and a blank space on total product belt generated. When the blank space reached the warehouse, an operator diverted the product back to the warehouse floor and the railcar was sealed and weighed. The third test run for formaldehyde on the scrubber inlet and outlet was ongoing during part of this production test, but testing was suspended while the granulators were being placed on total recycle and returned to normal operation.

The weight of urea granules produced during the production rate check was found to be 78 tons from the railcar weighings. Thus, the plant's production rate was 0.8667 ton/minute or 1248 ton/day. During the test the "A" line screened product totalizer indicated 27.6 tons produced or a rate of 0.3067 ton/minute (441.6 ton/day).

Fluid mechanics principles indicate that flow through a nozzle is proportionate to the square root of the pressure drop across the nozzle. Thus, if all the nozzles in the three granulators were operating and the pressures were all equal, the flow rate into each granulator would theoretically be equal. Also, if the pressures at each granulator were not equal then the production rate of a particular granulator could be determined by multiplying the total production by the ratio of the square root of the pressure to all three granulators. This method of apportioning the production assumes that all the nozzles have identical configurations which may not be the case because of differing wear rates.

Based on the above discussion the production rate of the "A" Granulator should be given by the following equation;

$$TP \times \frac{\sqrt{GSP-A}}{\sqrt{GSP-A} + \sqrt{GSP-B} + \sqrt{GSP-C}} = PA$$

where; TP = total production of all three granulators during the given time.

GSP-A = the average nozzle pressure for the subject granulator during the given time.

PA = production of the "A" Granulator during the given time.

The accuracy of the above equation is influenced by the standard deviation of the nozzle pressures during the averaging time.

Thus, using the weight produced as determined by the railcar (78 tons) for TP and the average nozzle pressure for the A, B and C Granulators (41.0, 41.6, 41.7 psig, respectively) for the period from 1300 to 1430, a value for PA can be calculated;

$$78 \times \frac{\sqrt{41.0}}{\sqrt{41.0} + \sqrt{41.6} + \sqrt{41.7}} = PA$$

PA = 25.85 tons

However, the production of the "A" Granulator was indicated to be 27.6 tons by the weigh belt totalizer. Assuming that the above calculation more accurately determines the actual production rate, a factor (C.F.) can be calculated which can be used to correct the "A" Granulator totalizer readings for any time period;

$$27.6 \text{ (C.F.)} = 25.85$$
$$\text{C.F.} = 0.9366$$

The actual production of the "A" Granulator for any time period can now be determined from the totalizer readings by the equation:

$$PA_T \times 0.9366 = PA_A$$

where;  $PA_T$  = the production in tons for a given time period as indicated by the "A" Granulator product weigh belt totalizer.

$PA_A$  = the actual production in tons of the "A" Granulator for the given time period.

$PA_T$  is determined by calculating the difference of the totalizer readings at the beginning and end of the time period and multiplying this difference by 0.05 to convert to tons.

A sample calculation using data for urea particulate, run 1, outlet on 10 October 1978 is presented below.

Example: (Refer to Table 3-2 and Appendix J)

Test started - 1131  
Test ended - 1336  
Totalizer reading at 1130\* = 3976  
Totalizer reading at 1340\* = 4732  
Time between initial and final  
totalizer readings = 130 minutes

$$PA_T = (4732 - 3976) \times 0.05 = 37.8 \text{ tons}$$

$$37.8 \times 0.9366 = PA_A = 35.4 \text{ tons}$$

---

\* Data from column designated PWTA in Appendix J.

$$\frac{35.4 \text{ tons}}{130 \text{ minutes}} = 0.272 \text{ ton/minute average production rate}$$

$$35.4 \text{ tons} \times \frac{1440 \text{ minutes/day}}{130 \text{ minutes}} = 392 \text{ ton/day}$$

Production rates for all test runs are presented in Table 25.

## SECTION 4

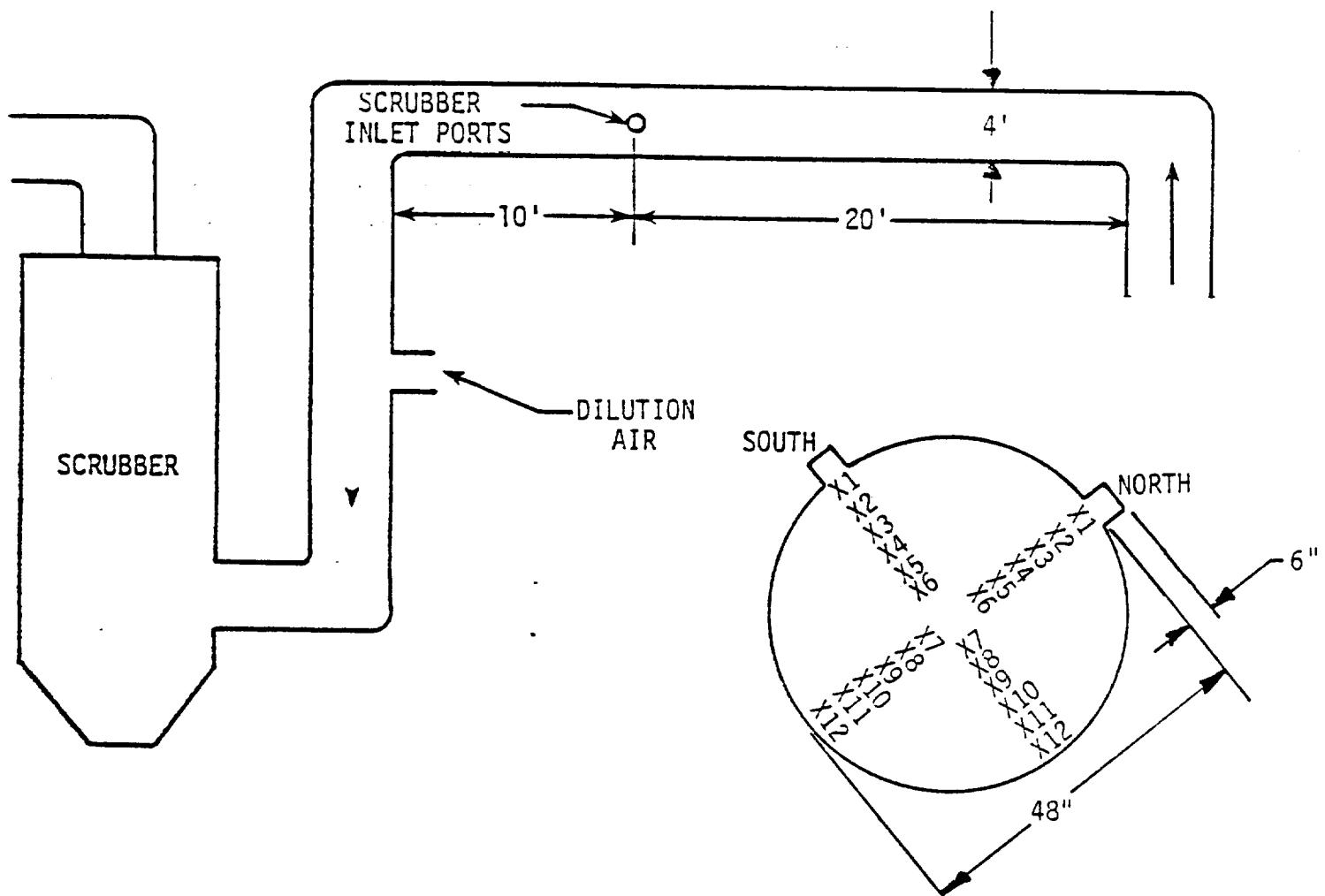
### LOCATION OF SAMPLING POINTS

Testing was conducted on the "A" Granulator and the Synthesis Tower Vent at the Agrico Chemical Company in Blytheville, Arkansas. This section presents the detailed descriptions of the sampling locations for the urea, ammonia, formaldehyde, particle sizing and the opacity measurements.

#### SCRUBBER INLET, TP-I

The scrubber inlet sampling site was located in a 48-inch I.D. horizontal section of a steel duct. A schematic of the sampling site including the traverse point sampling locations and duct dimensions is presented in Figure 5. Two 4-inch I.D. pipe-flange sampling ports positioned 90° apart were located 20 feet (5 stack diameters) upstream of a short-radius, right angle bend. The distance from the ports to the nearest downstream disturbance (another right-angle bend) was 10 feet (2.5 stack diameters).

The inlet sampling location did not meet the "eight and two diameters" criteria as outlined in EPA Method 1; consequently, 12 sampling points were chosen for each axis traverse for a total of 24 sampling points (as specified by the method). These points were located as shown in Figure 5. This figure shows the cross-sectional view of the duct at the sampling location and lists the exact distance each traverse point is located from the outside flange edge. The identifying number sequence and point locations were the same for all testing conducted at the inlet location.



1 - LOCATION OF TEST PORTS

| TRaverse Point No. | Traverse Point Distance From Outside Edge of Nipple (Inches) |
|--------------------|--|
| 1                  | 7  |
| 2                  | 9 1/4  |
| 3                  | 11 5/8   |
| 4                  | 14 1/2   |
| 5                  | 18   |
| 6                  | 23 1/3   |
| 7                  | 36 7/8   |
| 8                  | 42   |
| 9                  | 45 1/2   |
| 10                 | 48 3/8   |
| 11                 | 50 3/4   |
| 12                 | 53   |

FIGURE 5: LOCATIONS OF "A" GRANULATOR SCRUBBER INLET TEST PORTS & POINTS AT AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

#### SCRUBBER OUTLET, TP-2

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.

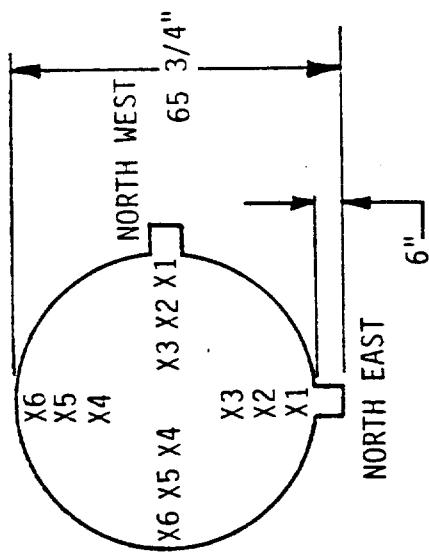
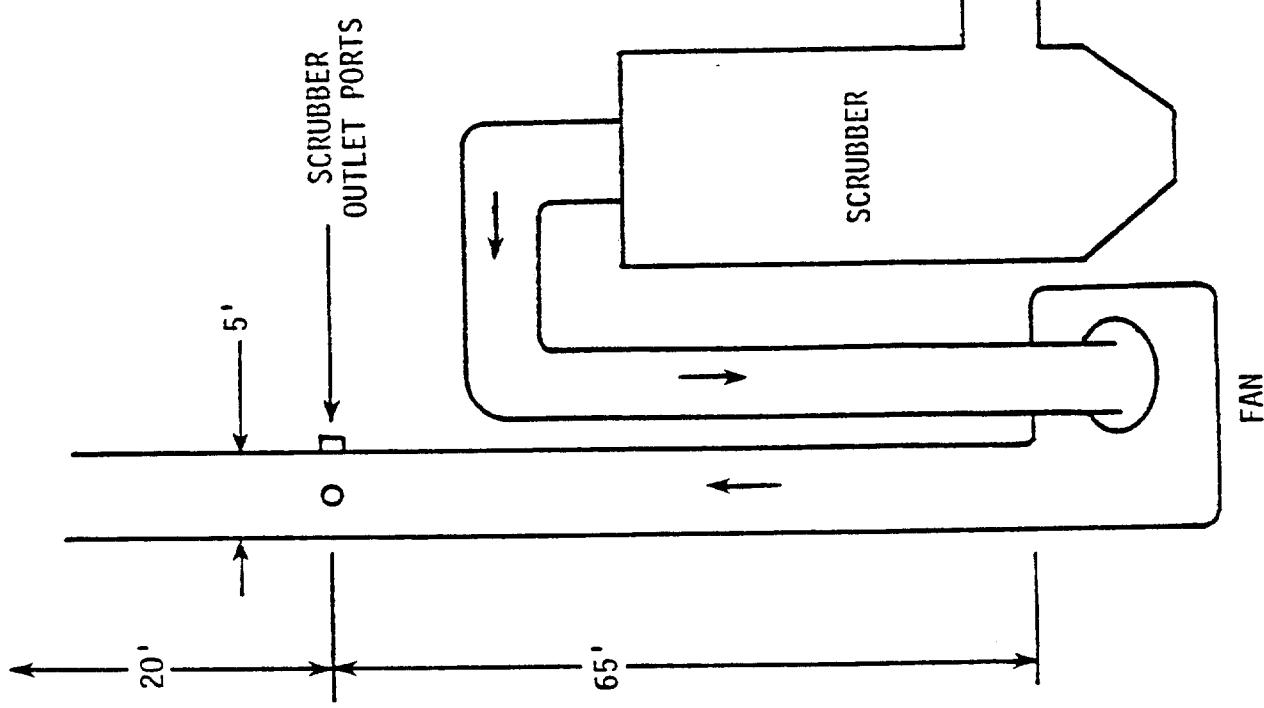
The "A" scrubber 60-inch I.D. outlet stack was fitted with two 4-inch I.D. pipe-flanged sampling ports positioned 90° apart in a horizontal plane. The two ports were located 65-feet (13 stack diameters) downstream of the fan outlet, and 20 feet (4 stack diameters) upstream of the stack discharge; See Figure 6. The port locations met the "eight and two diameters" criteria as delineated in EPA Method 1. Consequently, six sampling points were calculated for each axis traverse for a total of twelve sampling points (as specified in the method).

#### INLET PARTICLE SIZING SAMPLING LOCATION, TP-1

Particle sizing tests were performed in the "A" Granulator Scrubber inlet gas stream by sampling with an in-stack cascade impactor positioned in the duct through the test ports used for the emissions tests shown in Figure 5. The impactor nozzle was located at the centroid of the duct for each impactor run.

#### OUTLET PARTICLE SIZING SAMPLING LOCATION, TP-2

The size distribution of the particulate in the "A" Granulator Scrubber outlet stream was measured with an in-stack cascade impactor positioned in the stack through the test ports used for the emissions tests (see Figure 6). The impactor nozzle was located at the centroid of the stack for each impactor run.



| TRaverse Point No. | Traverse Point Distance From Outside Edge of Nipple (Inches) |
|--------------------|--|
| 1                  | 8 5/8"   |
| 2                  | 14 3/4"  |
| 3                  | 23 3/4"  |
| 4                  | 48 1/8"  |
| 5                  | 57"  |
| 6                  | 53 1/8"  |

FIGURE 6: LOCATIONS OF "A" GRANULATOR SCRUBBER OUTLET TEST PORTS & POINTS AT FAN  
AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

#### VISIBLE EMISSIONS OBSERVATION LOCATIONS

Figure 7 indicates the relative position of the two different observation sites employed to observe the white scrubber stack plume. These locations were chosen in order to conform to EPA Method 9 guidelines and specifications. The stack discharge was approximately 100 feet above ground level. The observer was positioned at distances of 100 to 150 feet from the stack. Heights of the observation sites ranged from ground level to about 100 feet above grade. Depending upon the sun position and the cloud cover, the observer either positioned himself on the synthesis tower and used the trees as a dark background or he stood in a field and used the synthesis tower for a dark background.

#### SCRUBBER PRESSURE DROP PRESSURE TAPS LOCATIONS

Pressure drop across the "A" Granulator Scrubber was measured with a vertical U tube water manometer which was connected to pressure taps at the scrubber inlet and outlet. The inlet pressure tap consisted of a stainless steel tube inserted into the middle of the duct through the dilution air damper. The tubing was fixed in place so that the open end was perpendicular to the flow stream. The outlet pressure tap consisted of a hole drilled through the section of ducting between the scrubber outlet and the inlet of the fan.

#### SCRUBBER LIQUID COLLECTION LOCATIONS

Scrubber solution samples were collected from the streams entering and leaving the "A" Granulator Scrubber (see Figure 8). The inlet sample was tapped from the line immediately before entering the scrubber. The outlet sample was tapped from the pump discharge.

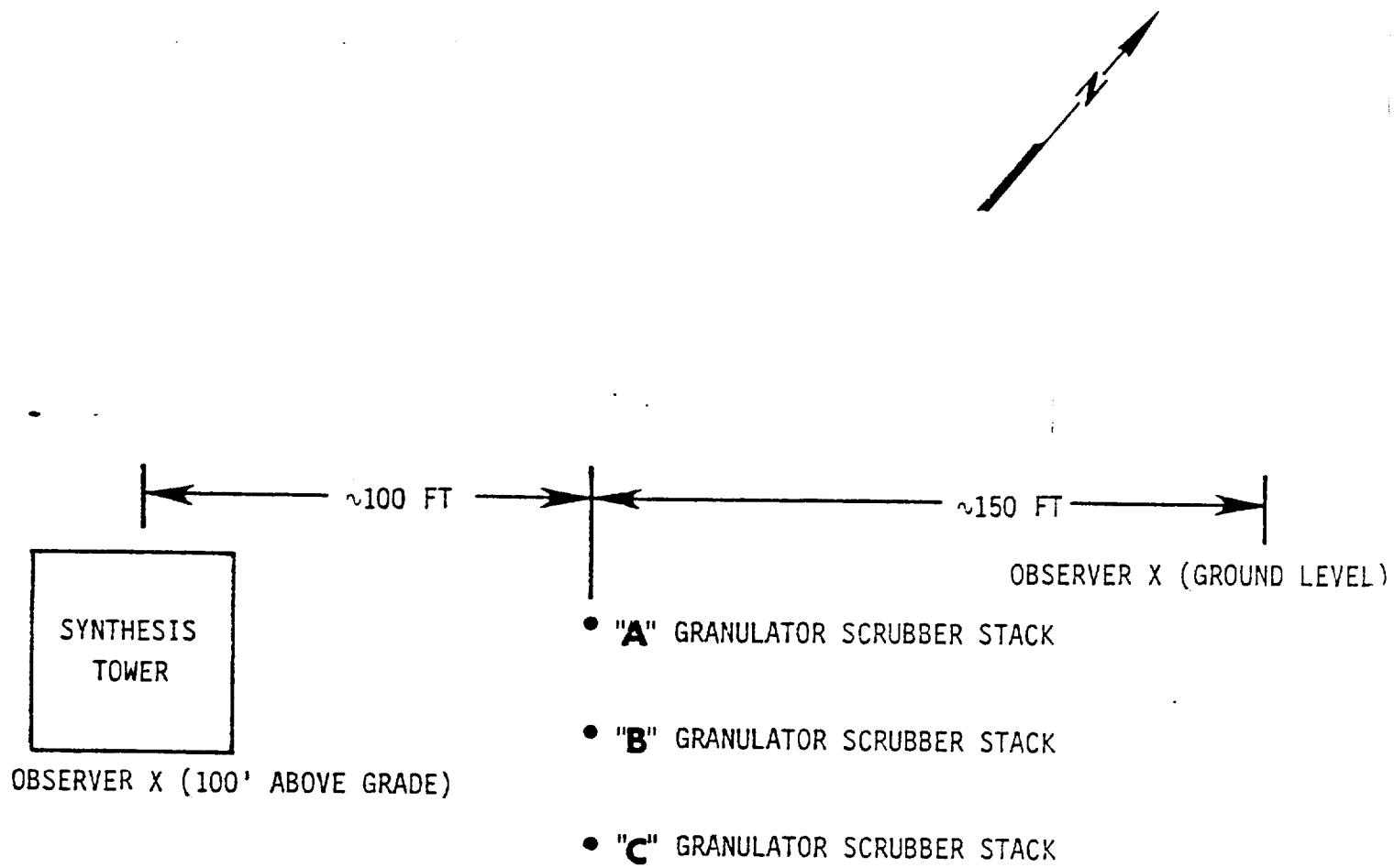


FIGURE 7: LOCATIONS OF SMOKE OBSERVER FOR OCTOBER 10 - 12, 1978  
OPACITY READINGS ON "A" GRANULATOR SCRUBBER STACK AT  
AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

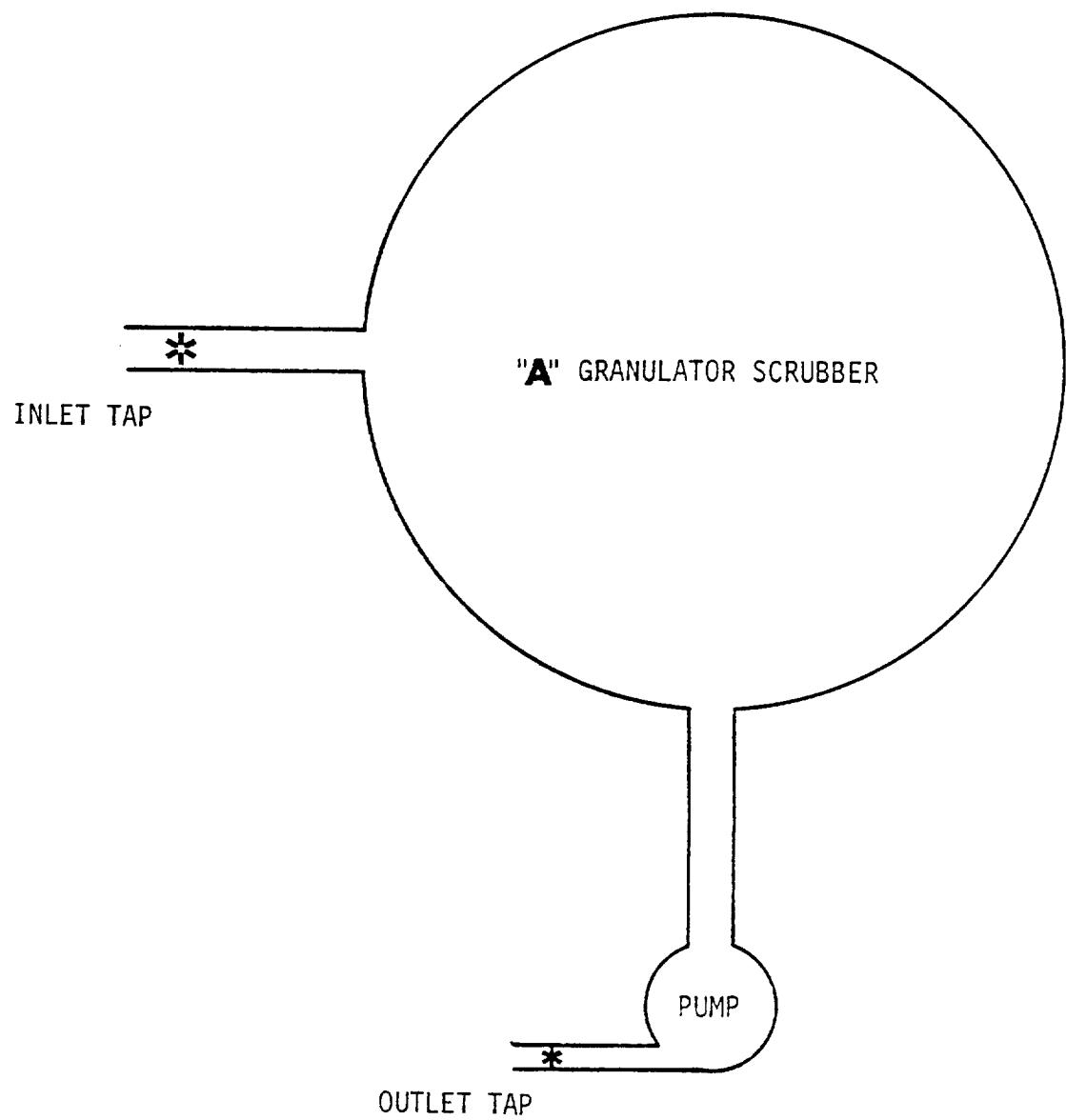


FIGURE 8: LOCATION OF SCRUBBER LIQUID COLLECTION TAPS FOR OCTOBER 10 & 11, 1978. TESTS ON "A" GRANULATOR AT AGRICO CHEMICAL COMPANY IN BLYTHEVILLE, ARKANSAS

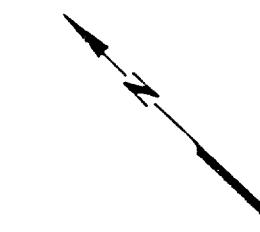
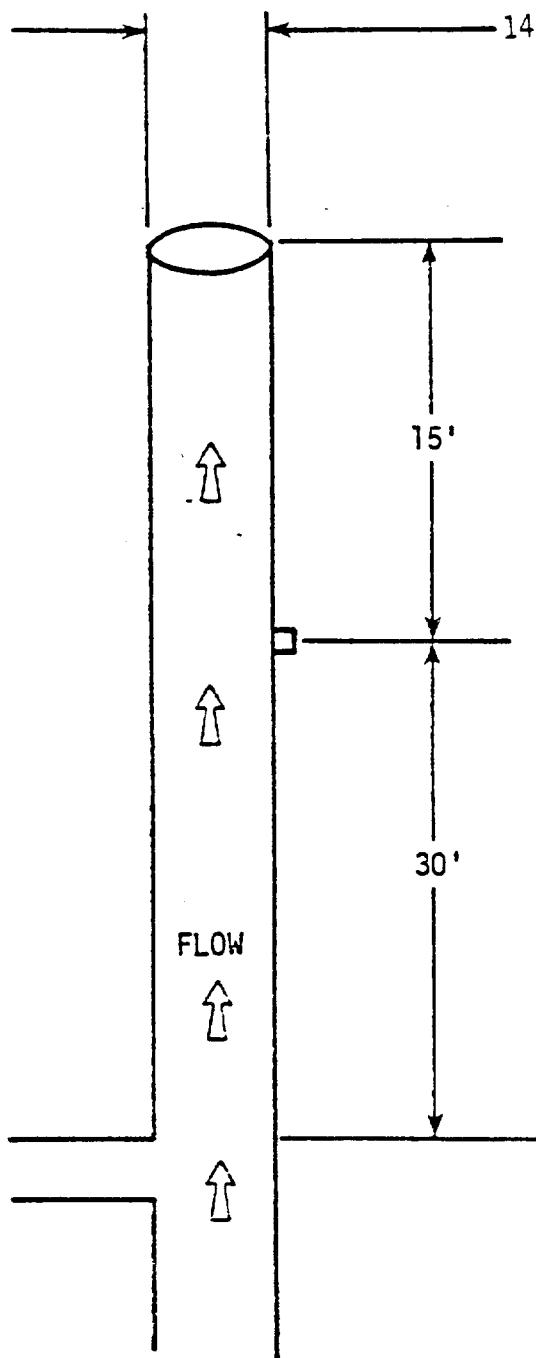
## PROCESS SAMPLES COLLECTION LOCATIONS

Throughout the testing program, various process samples were collected directly from their applicable process units/operations. Included were samples of urea melt, a formaldehyde-based additive, and the granulator un-screened and screened products.

### UREA SYNTHESIS TOWER VENT LOCATION

The urea synthesis tower sampling site was located in a 14-inch I.D. vertical section of a heavy gauge stainless steel pipe. A schematic of the sampling site including the velocity traverse points and duct dimensions is presented in Figure 9. One 6-inch I.D. pipe-flange sampling port was positioned approximately 30 feet (25 stack diameters) upstream of a connecting exhaust vent. The distance between the sampling port and the stack vertex was approximately 15 feet (12 stack diameters). The port location met the EPA Method 1 criteria. However, only a single sampling point located 12 inches inside the stack from outside the flange (the centroid of the duct) was used during the five test runs. This was requested by the Technical Manager, due to the following problems encountered during the testing periods.

1. Excessive moisture content (80 percent).
2. High ammonia concentrations.  
(1,300 lb/hour)
3. Extremely high stack gas velocities.  
(12,000 feet/minute)
4. Hostile working environment
  - a. Cold temperature (0°F windchill)
  - b. High winds (40 to 50 miles per hour)
  - c. Raining
5. Plugging of pitot and orifice pressure lines with water.



| VELOCITY<br>TRAVERSE POINT NO. | TRAVERSE POINT DISTANCE<br>FROM OUTSIDE EDGE OF NIPPLE<br>(INCHES) |
|--------------------------------|--|
| 1                              | 7.0  |
| 2                              | 9.5  |
| 3                              | 16.5   |
| 4                              | 19.0   |

FIGURE 9: LOCATION OF SYNTHESIS TOWER SOLUTION VENT  
SAMPLING PORT AND VELOCITY TRAVERSE POINTS  
AT AGRICO CHEMICAL COMPANY IN  
BLYTHEVILLE, ARKANSAS

SECTION 5  
SAMPLING AND ANALYSIS METHODOLOGIES

This section presents the descriptions of the sampling and analysis methodologies employed at the Agrico Chemical Company facility in Blytheville, Arkansas during October 9-13, 1978.

The EPA designated methods are contained in the respective Appendices. This section presents general descriptions and deviations from the methods in the Appendices.

UREA AND AMMONIA "A" GRANULATOR SCRUBBER INLET AND OUTLET

Urea and ammonia in the "A" Granulator Scrubber inlet and outlet gas streams were sampled at points identified by EPA Method 1 in accordance with the relationship of the sampling ports to upstream and downstream diameters. The velocity of the duct gas was measured using a calibrated, S-type pitot tube in accordance with EPA Method 2. Construction and calibration of the S-type pitot tubes was consistent with EPA Method 2. The complete method of sampling and analysis for urea and ammonia is contained in Appendix M.

The sampling train is shown schematically in Figure 10 and consists of a nozzle, probe, filter, Teflon line connector, five impingers, vacuum pump, dry gas meter, and an orifice flow meter. The nozzle (1) is stainless steel and is of a buttonhook shape. It was connected to a 5/8" stainless steel glass lined probe (2) that is wrapped with nichrome heating wire and jacketed. The probe temperature was maintained at 160° F to prevent condensation of the sampled gas. Following the probe, the gas stream impacted on a 4 1/2" glass filter supported on a coarse fritted glass disc in a glass filter holder (3). Reeve Angel Type 934 AH filter paper

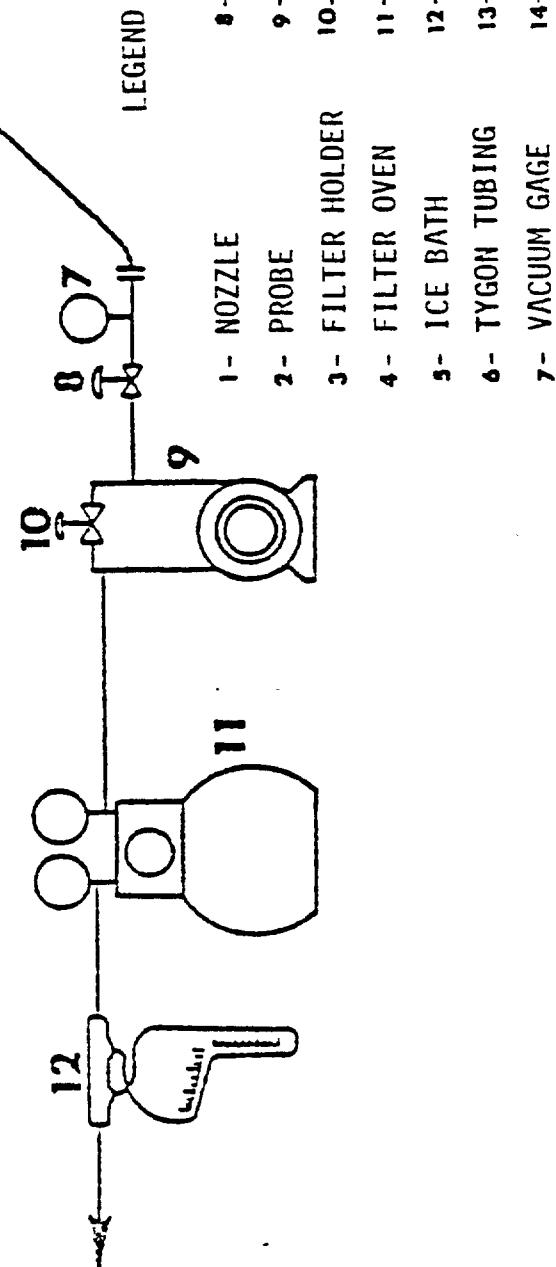
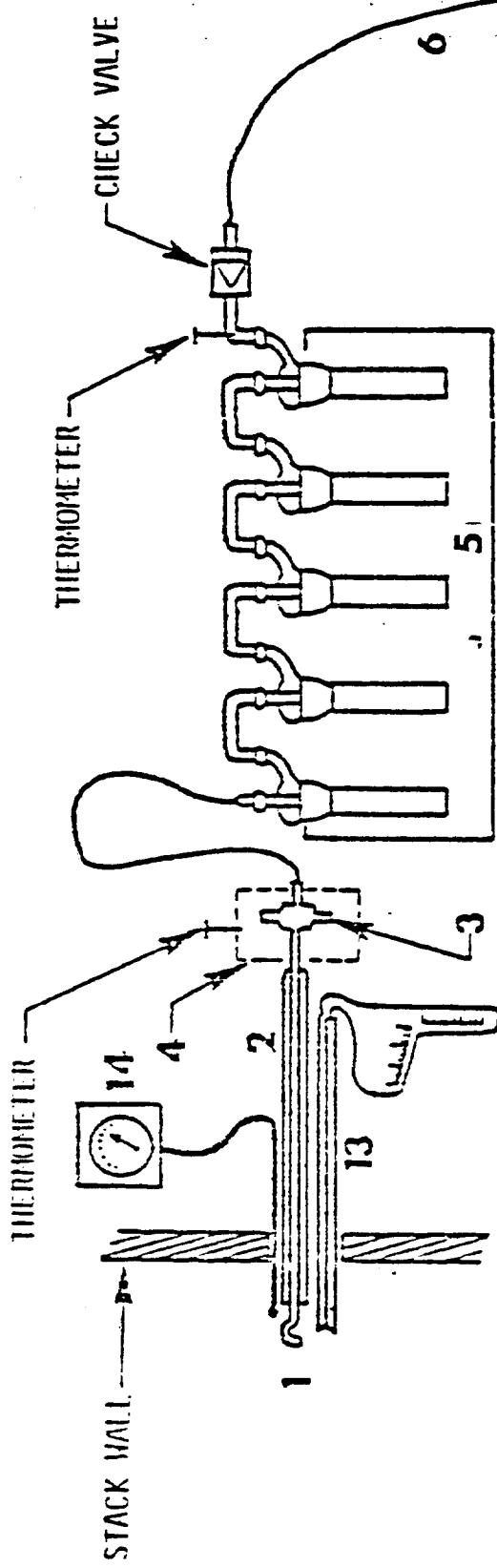


FIGURE 10: MODIFIED EPA PARTICULATE SAMPLING TRAIN  
AUGUST 18, 1977, FEDERAL REGISTER

was used. Enclosing the whole filter assembly was a heated box (4) to maintain temperature of the gas leaving the filter at 160° F. EPA Reference Method specifies a temperature of 248° F ± 25° F, however, urea melts and readily decomposes to ammonia at temperatures above 200°. The filter and heated box were not used for outlet run 3 (test #7). An ice bath containing five impingers(5) was attached to the back end of the filter via a section of Teflon tubing. The first impinger contained deionized distilled water (100 ml), the second and third contained 100 ml of 1N H<sub>2</sub>SO<sub>4</sub> each, the fourth was dry and the fifth contained silica gel (200 grams). Leaving the fifth impinger, the dry sample stream flowed through flexible tubing (6), a vacuum gauge (7), needle valve (8), pump (9), and a dry gas meter (11). A calibrated orifice and inclined manometer (12) complete the sampling train. The stack velocity pressure was measured using a pitot tube (13) and inclined manometer. Stack temperature was monitored by a thermocouple attached to the probe and connected to a potentiometer (14). A nomograph determined the orifice pressure drop required for any pitot velocity pressure and stack temperature in order to maintain isokinetic sampling conditions.

Test data recorded included test time, sampling duration of each traverse point, pitot pressure, stack temperature, meter volume, meter inlet-outlet temperature and orifice pressure drop. At completion of each run the probe was removed from the stack and was purged for 15-20 minutes with ambient air. This was done so that any remaining ammonia in the train would be collected in the impingers. A final leak check was then performed.

The nozzle and probe were then washed and brushed three times into a glass sample jar which was sealed with a Teflon-lined cap. The probe washes were performed on site near the stack. The Teflon line between the filter and first impinger was rinsed with distilled, deionized water three times into a second glass sample jar and sealed. The sample jars and impingers were returned to the sample train prep and clean-up room.

The contents of impingers were transferred to tare weighted sample jars at the clean-up area. The sample containers were used as follows:

Container #1 - Filter

Container #2 - Deionized distilled water wash of nozzle, probe and all glassware up to the filter.

Container #3 - Silica gel from the fifth impinger.

Container #4 - Contents of first impinger and deionized distilled water wash of first impinger, Teflon line and glassware between filter and first impinger.

Container #5 - Contents of second, third, and fourth impingers and deionized distilled water wash of impingers plus connecting glassware.

Aliquots of some of the samples were analyzed for urea and ammonia by Agrico chemists. The remainder of the samples were transported to TRC's laboratory and analyzed for urea and ammonia. The urea concentration was measured by adding urea color reagent (see Appendix M) to the samples and measuring the absorbance of the solutions with a spectrophotometer at 420 nm. The ammonia concentration was measured by adding Nessler reagent to the samples and measuring the absorbance of the solutions with a spectrophotometer at 405 nm. Calibration curves were drawn from the corresponding absorbances of standard urea and ammonia solutions. The calibration curves were used to determine the urea and ammonia concentrations of the samples.

The sample concentration was multiplied by the sample volume to determine the total urea and ammonia. It should be noted that a aliquot was removed for analysis by Agrico chemists, however, this aliquot volume was included in the sample volume for calculation of total urea, ammonia and formaldehyde collected.

#### FORMALDEHYDE "A" GRANULATOR SCRUBBER

The complete method for sampling and analysis for formaldehyde is contained in Appendix M. Formaldehyde in the "A" Granulator Scrubber inlet and outlet gas streams was sampled using the methodology and equipment described in the subsection entitled Urea and Ammonia "A" Granulator Scrubber with the following modifications:

1. There were only four impingers used. Impingers one and two contained 100 ml each of distilled deionized water, impinger three was dry and impinger four contained 200 grams of silica gel (See Figure 11).
2. The filter and heated box were not used for the outlet tests.

The contents of the impingers were transferred to tare weighted sample jars at the clean-up area. The sample containers were used as follows:

Container #1 - Filter (inlet only)

Container #2 - Deionized distilled water wash of nozzle, probe, Teflon line, filter holder (inlet only), impingers 1, 2 and 3 and their connecting glassware, plus the contents of impingers 1, 2, and 3.

Container #3 - Silica gel from the fourth impinger

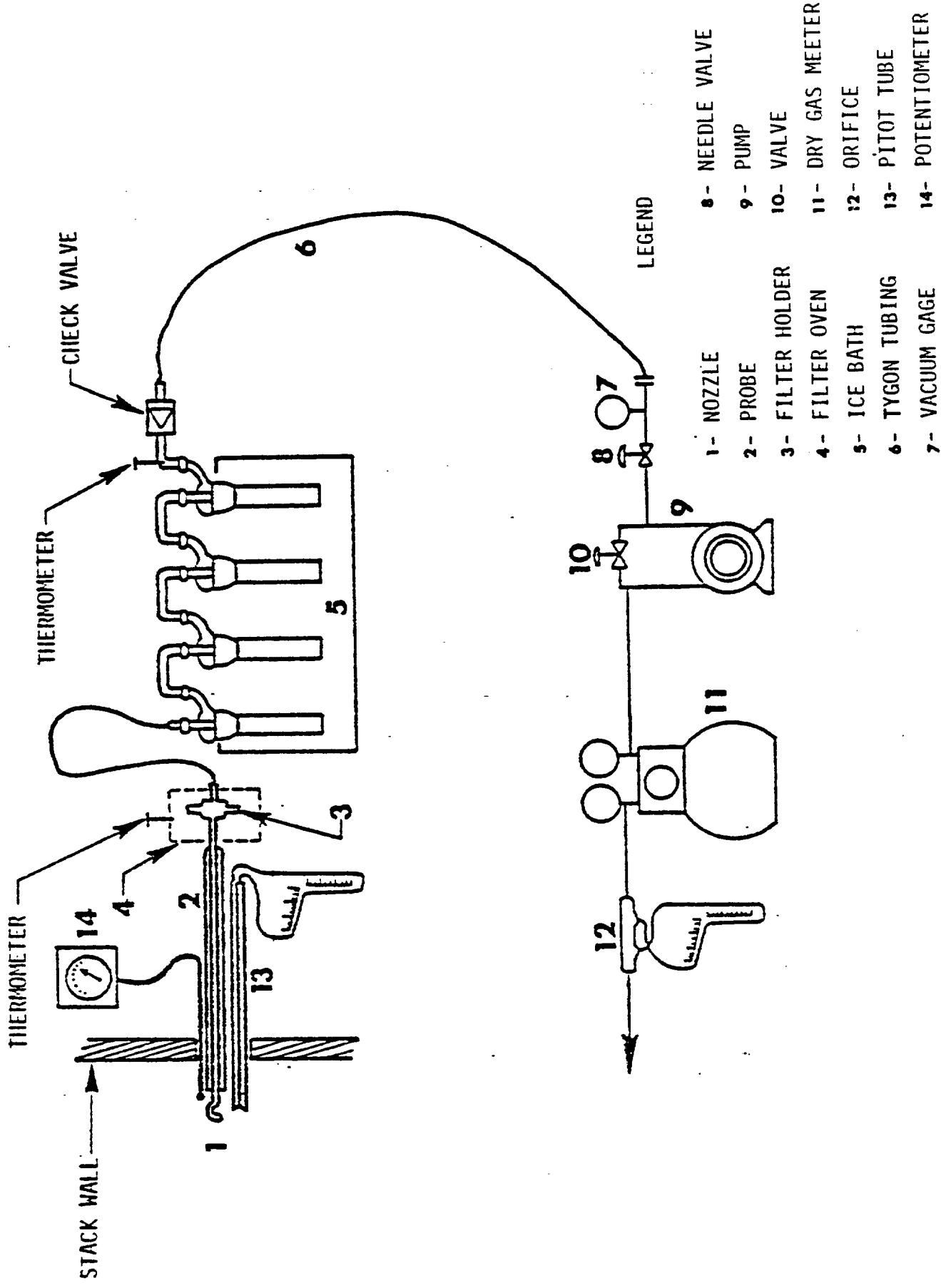


FIGURE 11 : MODIFIED EPA PARTICULATE SAMPLING TRAIN  
AUGUST 18, 1977, FEDERAL REGISTER

The samples were analyzed for urea and ammonia in addition to formaldehyde. The analyses for urea and ammonia were performed as described previously. The analysis for formaldehyde consisted of reacting an aliquot of the sample solution with chromatropic-sulfuric acid reagent to form a purple chromogen. This colored solution was analyzed colorimetrically using a spectrophotometer at 580 nm. The absorbance of the colored solution is proportional to the quantity of formaldehyde. Standard formaldehyde solutions were prepared to produce a calibration curve which was used to determine formaldehyde concentration of the individual sample. The sample concentration was multiplied by the sample volume to determine the total formaldehyde in the sample.

#### VISIBLE EMISSIONS "A" GRANULATOR

The visible emissions measurements were conducted by a certified visual emission evaluator in accordance with EPA Reference Method 9. The readings were taken at 15 second intervals. Since the plume was white, it was necessary to read the emissions against a dark background. The dark background was provided by either the synthesis tower or a row of trees.

#### PARTICLE SIZE "A" GRANULATOR

A Sierra Model 226 multi-stage cascade impactor was operated in its instack mode. Sampling was performed isokinetically from a single point at the center of the scrubber inlet and outlet ducts. Prior to the

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

The impactor was loaded before each run with preweighed glass fiber collection substrates. After sampling the impactor was removed to the clean up room and the substrates placed in plastic petri dishes and sealed. The cyclone was brushed and washed with distilled deionized water into a sample jar and sealed. These samples were returned to TRC and weighed on an analytical balances to 0.01 mg. The filters were dessicated for at least 24 hours before the initial weighing. The filters were then weighed until a constant weight was reached. If constant weight was not attained, the filters were then redessicated for at least six hours. The weighing room had an environment where the relative humidity was less than 50 percent. More information on particle sizing can be found in Appendix B.

#### SCRUBBER LIQUID "A" GRANULATOR

Samples (approximately 500 ml) of the scrubbing liquid streams entering and exiting the "A" Granulator Scrubber were collected at approximately 15 minute intervals during the urea and ammonia tests. The sample temperature was measured immediately upon collection.

The sample was then allowed to reach room temperature and then the pH was measured with a pH meter.

The individual samples were then composited into one inlet and one outlet sample for each associated run. The pH of the composite sample was also measured with the pH meter. The composited samples were returned to TRC's laboratory and analyzed for urea, ammonia, and formaldehyde concentration in accordance with Appendix M.

#### PROCESS SAMPLES "A" GRANULATOR

Grab samples of the urea melt, unscreened and screened product were collected at their respective locations in the process. A grab formaldehyde additive sample was also collected and retained for possible future analysis.

The urea melt, unscreened and screened product samples were prepared for analysis by dissolving a known weight (approximately 200 mg) into 100 ml of distilled deionized water. The solution was then analyzed for urea, ammonia and formaldehyde using the procedures in Appendix M and Appendix E.

#### UREA AND AMMONIA SYNTHESIS TOWER MAIN VENT

Urea and ammonia in the Synthesis Tower Main Vent Stack were sampled from a single point located in the geometric center of the duct. The velocity of the duct gas was measured using a S-type pitot tube in accordance with EPA Method 2. Construction of the S-type pitot tube was consistent with EPA Method 2. The velocity profile across the duct was found to be flat during the preliminary traverse.

The sampling train is shown schematically in Figure 12 and consists of a nozzle, instack orifice, probe, Teflon line, eight impingers, vacuum pump and a dry gas meter. The nozzle (1) is stainless steel and is of a buttonhook shape. An instack orifice (3) was used to permit isokinetic sampling of a stream with a moisture content greater than 50%. The instack orifice meters the sample at the stack conditions and, therefore, changes in moisture will not affect isokinetic sampling. The instack orifice was connected to a 5/8" stainless steel glass lined probe (2). An ice bath containing eight impingers (5) was attached to the probe via a section of Teflon tubing. Impingers one through four contained 100 ml each of distilled deionized water, impinger five was empty and numbers six and seven each contained 100 ml of 10N  $H_2SO_4$ . The 10N  $H_2SO_4$  was necessary because of the high ammonia concentration. The smell of ammonia was noticed in the exhaust of the train during tests 1 and 2 and the silica gel turned purple during these tests indicating incomplete ammonia collection in the impingers. The eighth impinger contained silica gel (200 grams). Leaving the eight impinger, the dry sample stream flowed through flexible tubing (6), a vacuum gauge (7), needle valve (8), pump (9), and a dry gas meter (11). A calibrated orifice and included manometer (12), complete the sampling train. The stack velocity pressure was measured using a pitot tube (13) and inclined manometer. Stack temperature was monitored by a thermocouple attached to the probe and connected to a potentiometer (14). A calculation was made to determine the instack orifice pressure drop required for any pitot velocity pressure in order to maintain isokinetic sampling conditions.

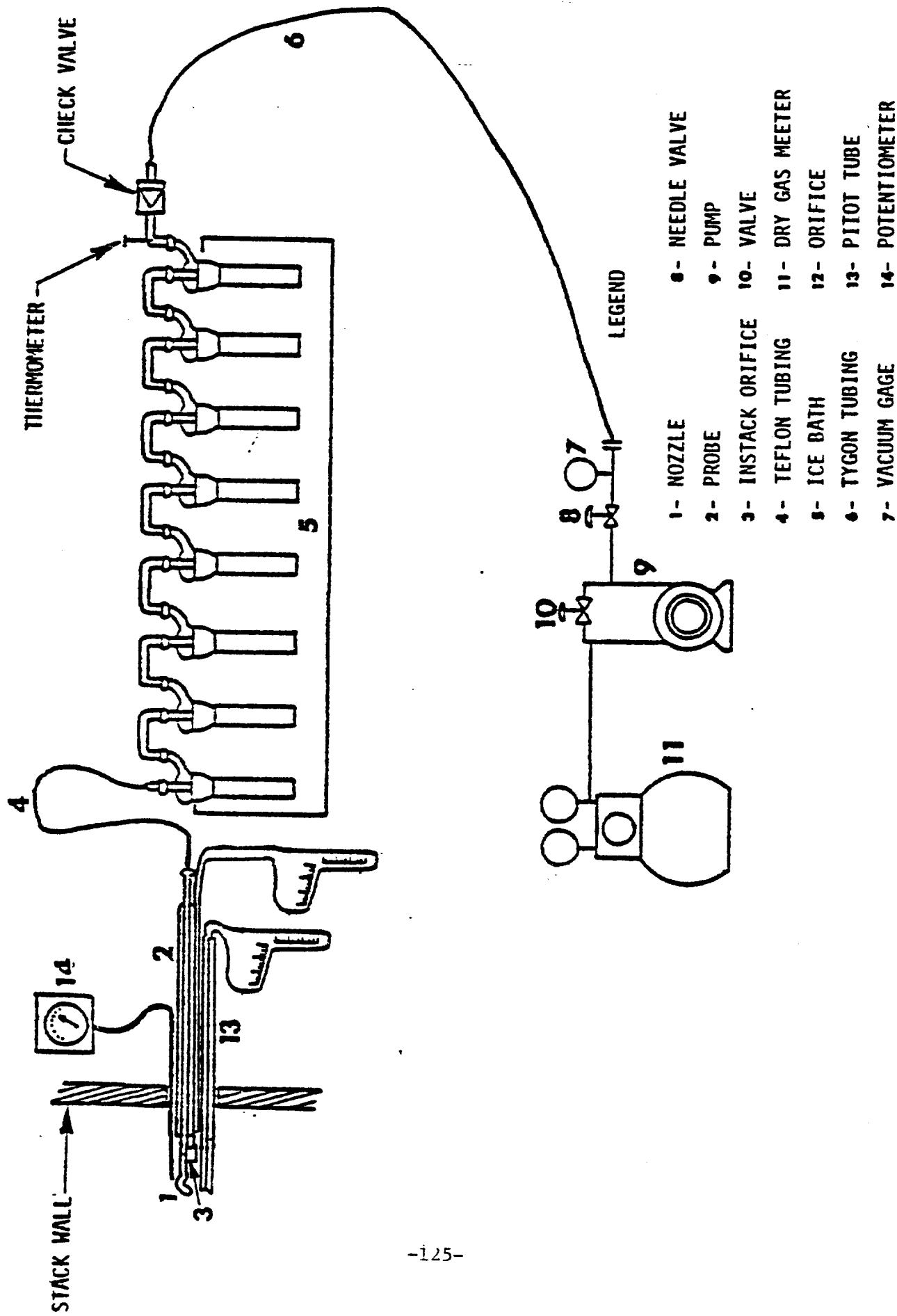


FIGURE 12: MODIFIED EPA PARTICULATE SAMPLING TRAIN WITH INSTACK ORIFICE  
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Test data recorded included test time, pitot pressure, stack temperature, meter volume, meter inlet-outlet temperature and orifice pressure drop. At completion of each run the train was leak checked, then purged for 15 minutes to assure complete collection of ammonia. The nozzle and probe were washed and brushed three times on site into a glass sample jar which was sealed with a Teflon-lined cap. The Teflon line between the probe and 1st impinger was rinsed three times with distilled, deionized water into a second glass sample jar and sealed. The sample jars and impingers were returned to the sample train prep and clean up room for storage and measurement of volume of liquid collected.

The contents of impingers were transferred to tare weighted sample jars at the clean up area. The sample containers were used as follows:

- Container #1 - Contents of first five impingers and deionized distilled water wash of impingers, Teflon line, nozzle and probe.
- Container #2 - Contents of impingers six and seven and deionized distilled water wash of impingers.
- Container #3 - Silica gel from eighth impinger.

The samples were transported to TRC's laboratory and analyzed for urea, ammonia and formaldehyde. The urea concentration was measured by adding urea color reagent (see Appendix M) to the samples and measuring the absorbance of the solutions with a spectrophotometer at 420 nm. The ammonia concentration was measured by adding Nessler reagent to the samples and measuring the absorbance of the solutions with a spectrophotometer at 405 nm. The formaldehyde concentration was measured by reacting an aliquot of the sample solution with chromatropic-sulfuric acid reagent.

to form a purple chromogen. This colored solution was analyzed colorimetrically using a spectrophotometer at 580 nm. Calibration curves were drawn from the corresponding absorbances of urea, NH<sub>3</sub>, and formaldehyde standard solutions. The calibration curves were used to determine the urea and ammonia concentrations of the samples. The sample concentration was multiplied by the sample volume to determine the total urea, ammonia and formaldehyde. Special calculations were performed to determine the volume of dry gas sampled, percent moisture and molecular weight of duct gas. The gaseous volume of ammonia collected in the impingers was added to the volume measured by the dry gas meter to equal the total dry sample volume. The gaseous equivalent volume of water collected in the impingers was divided by the total sample volume (H<sub>2</sub>O gaseous equivalent volume + NH<sub>3</sub> gaseous equivalent volume + dry gas volume measured by dry gas meter) to determine the percent moisture in the duct gas stream. The molecular weight of the duct gas stream was calculated from the percent moisture, percent ammonia and the percent CO<sub>2</sub>, O<sub>2</sub>, and N<sub>2</sub> of the dry gas volume based on measurements at a similar synthesis tower. Appendix G contains the complete calculations.

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