E.I DUPONT DE NEMOURS & CO.

DEC 18 1989

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REPORT CERTIFICATION

The sampling and analysis performed for this report were carried out under my direction and supervision.

DATE: 12-11-89

SIGNATURE: Edward D. Handel

Edward D. Handel, PhD
Senior Chemist
ETS, Inc.

I have reviewed all testing details and results in this report and hereby certify that the test report is authentic and accurate.

DATE: 12-11-89

SIGNATURE: Joseph F. Szalay
Joseph F. Szalay

P.E.: E-015225

STATE OF: Ohio

STATE OF OHIO
JOSEPH F. SZALAY
15225
REGISTERED
PROFESSIONAL ENGINEER

1.0 INTRODUCTION

A compliance test program was performed on the new wet scrubber system installed on the Orlon process at the duPont Waynesboro Plant. The testing program was designed to determine the level of abatement of N,N-Dimethylformamide (DMF) emissions. Compliance testing was performed on only one scrubber (designated Scrubber No. 1) at this time. Scrubber No. 2 was tested previously on October 6, 1989. The current tests were performed by T. Jeter, T. Underwood and T. Handel of ETS, Inc. on Tuesday, October 31, 1989. The testing was coordinated with P. Dana and R. Schifflett of duPont. All operations data was collected by duPont personnel and has been included in this report as Appendix E. The test program was witnessed by R. Goetz and P. Hunter of the Roanoke office of the State Air Pollution Control Board.

The test program consisted of three replicate series of measurements. All testing was conducted while the Orlon process was operating normally. Plant operations data were collected for each of the testing periods. Each series of tests consists of the following measurements:

1) DMF gas concentration at the inlet duct of the dual scrubber system.
2) DMF gas concentration at the outlet of Scrubber No. 1.
3) Total gas volumetric flowrate at the inlet duct of the scrubber system.

4) Moisture content of the inlet duct of the scrubber system.

5) Moisture content of the outlet duct of the scrubber system.

6) DMF concentration in the scrubber system wastewater effluent.

7) Orlon process operational data.

All inlet/outlet testing was performed simultaneously in order to evaluate real time scrubber efficiency.

Figure 1 illustrates the overall gas flow details for the DMF scrubber system. The scrubber system is designed with two wet scrubber units, each designed to handle up to 75,000 actual cubic feet per minute (acfm) of exhaust gases. With both scrubbers in operation, the combined exhaust gases from the six Orlon dryers, the Pidler exhaust and the waste exhaust unit can be treated simultaneously. Scrubber No. 2 was isolated from the system and was not involved in the current series of tests.

Figure 2 shows the details of the scrubber construction. Each unit is equipped with an 800 HP blower to force the inlet gas through each scrubber. In the current test program, only the blower to scrubber No. 1 was operated.

2.0 TEST PROCEDURES

The rectangular inlet duct is 96 inches wide and 80 inches

tall, with an effective diameter of 87 inches (7.3 feet). Three inlet sampling ports were located 56 feet downstream from the particulate screen and 14 feet upstream from the beginning of the right angle bend of the duct. Each port was three inches in diameter. Outlet sampling ports are located at the top of each scrubber, as shown in Figure 2. The diameter of the scrubber outlet stack is 8 feet.

2.1 DMF Gas Concentration

Determination of the DMF concentrations in the scrubber inlet and outlet gas streams was determined in general accordance with NIOSH Method 2004, which incorporates a solid sorbent tube (silica gel) for DMF absorption. Due to the high moisture content of the gas streams, the standard method was modified by placing a series of chilled impingers before the solid sorbent tube. The first two impingers each contained 15 mL of deionized water. A third impinger was empty. Due to the high solubility of DMF in water, it was anticipated that the DMF would be absorbed in the impinger solutions. Figure 3 illustrates the complete sample train employed in this test program.

Each sampling run was conducted for a period of 60 minutes at a nominal sampling rate of 1.5 liters per minute. Both inlet and outlet sampling was performed using the center of the respective ducts as the point of sample withdrawal.

The impinger solutions and the sorbent tube samples were analyzed using GC/FID in accordance with the general conditions outlined in NIOSH Method 2004. The impinger solution volumes were measured and recorded. Impinger 2 and 3 contents were combined and analyzed. Impinger 1 was analyzed separately. The probe and connecting lines were rinsed with deionized water. The probe wash volume was measured and the solution was analyzed for DMF.

2.2 Moisture Content

Moisture content of the inlet and outlet gas streams was determined by measuring the volume of water gain in the three impingers in the sampling train.

2.3 Total Gas Flowrate

The total volumetric flowrate of the inlet gases was measured using velocity traverses in accordance with EPA Methods 1 through 3 (40 CFR, Part 60, Appendix A). Four traverse points were used in each of the three inlet ports, giving a total of 12 traverse points (see Figure 4). The traverses were conducted before and after each of the DMF sampling runs. The traverses were performed using a Type "S" pitot tube, a type "K" thermocouple, and a digital manometer. Field data sheets for the stack velocity measurements are contained in Appendix B.

2.4 Wastewater Analysis

A sample of the scrubber wastewater was withdrawn during

each run. These samples were analyzed for DMF concentration by the ETS Mobile Laboratory utilizing the same procedures used to analyze the impinger solutions. The Analytical Method is contained in Appendix C and the results of the analytical determination of DMF are contained in Appendix D.

2.5 Process Data

Data collected by duPont plant personnel regarding the operation of the Orlon process and associated scrubber system is included in this report as Appendix E.

3.0 SAMPLING AND TEST METHODOLOGY

All sampling was conducted using sampling pumps calibrated onsite before and after each run. Each pump was calibrated with the sampling train in place using an inverted bubble flowmeter. Calibration data is contained in Appendix B. Each sampling train was the leak checked preceding sampling interval by plugging the tip of the sampling probe and observing the complete cessation of bubbling in each of the impingers. The rotameter was also observed to insure that no leaks were present in the sampling train.

Sampling was coordinated using radio communication to ensure that inlet and outlet sampling was conducted simultaneously.

3.1 DMF Analysis

DMF analysis was performed onsite in the ETS Mobile Laboratory located at the foot of the DMF scrubbers. Analysis was begun immediately following each sampling period. The analysis was performed on a Shimadzu GC-14A gas chromatograph equipped with a flame ionization detector (FID). The analysis was performed using the following conditions:

Injection: 0.5 uL direct on-column

Injector Temperature: 250°C

Detector: FID

Detector Temperature: 220°C

Column: 10-meter, 0.53mm i.d. FSOT (Alltech SuperoxII)

Carrier: Nitrogen (3.0 mL/min)

Column Temperature: 100°C Isothermal

Samples were analyzed together with standards, spikes and duplicates.

4.0 RESULTS

The compliance data consists of three consecutive tests (runs 1, 2 and 3) performed on Tuesday, October 31, 1989. All outlet data contained in this report is for Scrubber No. 1. The test log for the compliance testing is contained in Appendix A of this report. Duct flow measurements ranged between 59,970 and 62,290 acfm during the testing. The results of the DMF Compliance Tests for the duPont DMF Scrubber No. 1 are contained

in Table 1.

Each of the three compliance tests demonstrated an efficiency of greater than 90 percent abatement of DMF emissions. The scrubber was operated at full capacity during each of the tests. The specific plant operations data were provided by duPont personnel and are contained in Appendix E of this report. The inlet concentrations of DMF ranged between 213 and 265 ppm_{dv} during the compliance testing. The outlet concentrations ranged from 8.1 to 4.4 ppm_{dv}. The average scrubber efficiency measured for the three tests was 97.3%.

The concentration of DMF in the wastewater in the bottom of the stack averaged 3.0% during the testing.

Table 1. Results of Efficiency Testing on Scrubber No. 1.

	Run 1	Run 2	Run 3	
Inlet Temperature (°F):	164.8	166.5	156.6	
Inlet Pressure (Static) (in.H_2O):	-1.04	-0.983	-1.06	
Duct Velocity (ft/s):	19.3	19.5	18.7	
Duct Flowrate (acfm):	61870	62290	59970	
(scfm):	49990	50200	49080	
(dscfm):	48420	43630	40960	
Inlet Moisture (%):	6.3	9.9	11.9	
Outlet Moisture (%):	3.1	13.1	16.5	
Inlet DMF Concentration (ppm_{dv}):	265	255	213	
Outlet DMF Concentration (ppm_{dv}):	4.4	7.0	8.1	9.3
Inlet Emission Rate (lbs/hr):	146.0	126.5	99.1	123.9
Outlet Emission Rate (lbs/hr):	2.4	3.5	3.8	3.2
Scrubber No. 1 Efficiency (%):	98.4	97.3	96.2	
Scrubber No. 1 Wastewater Analysis:				
Concentration (%(w/v)) before run:	2.71	2.84	3.26	
Concentration (%(w/v)) after run:	2.76	3.13	3.44	

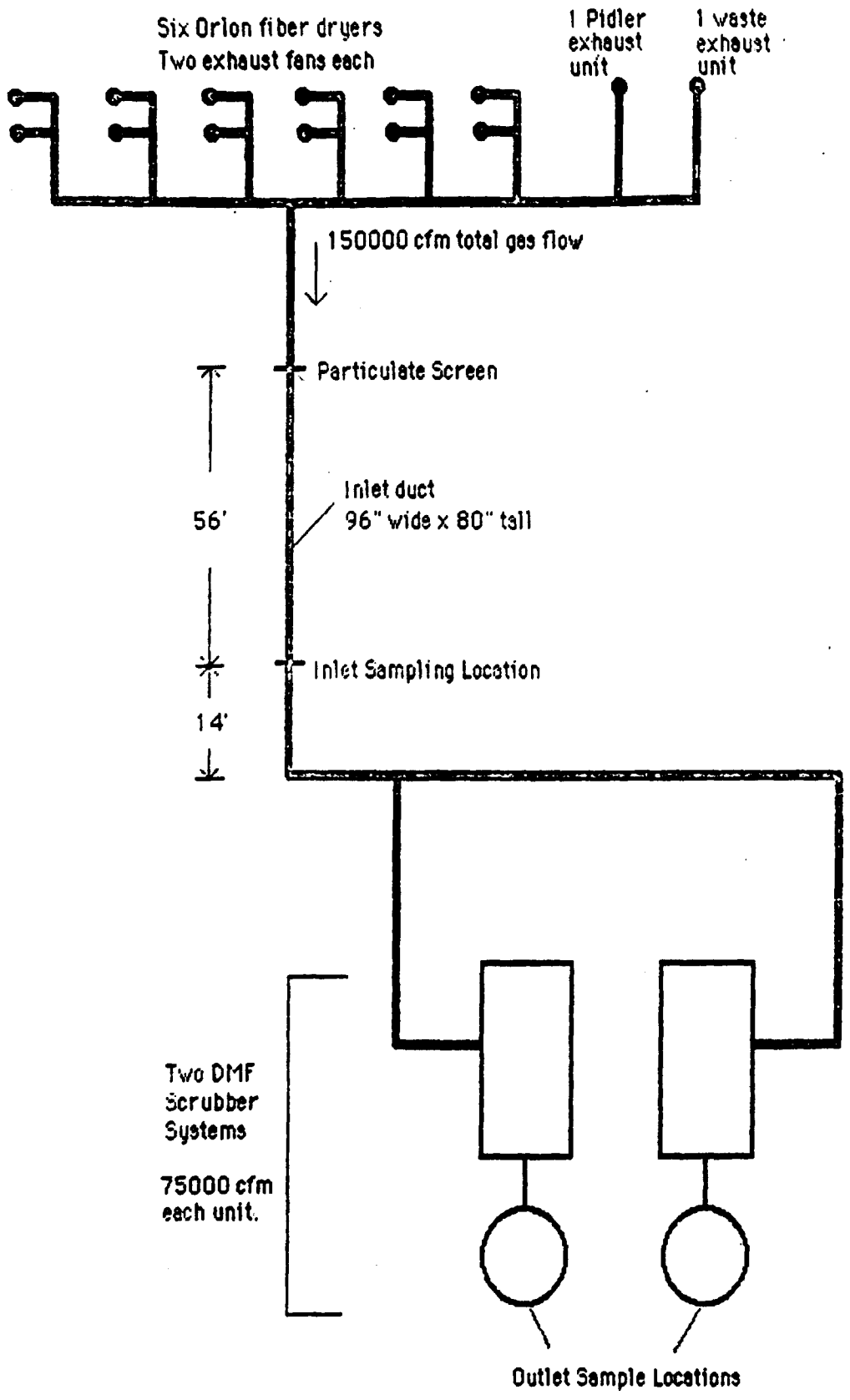


Figure 1 - Scrubber System

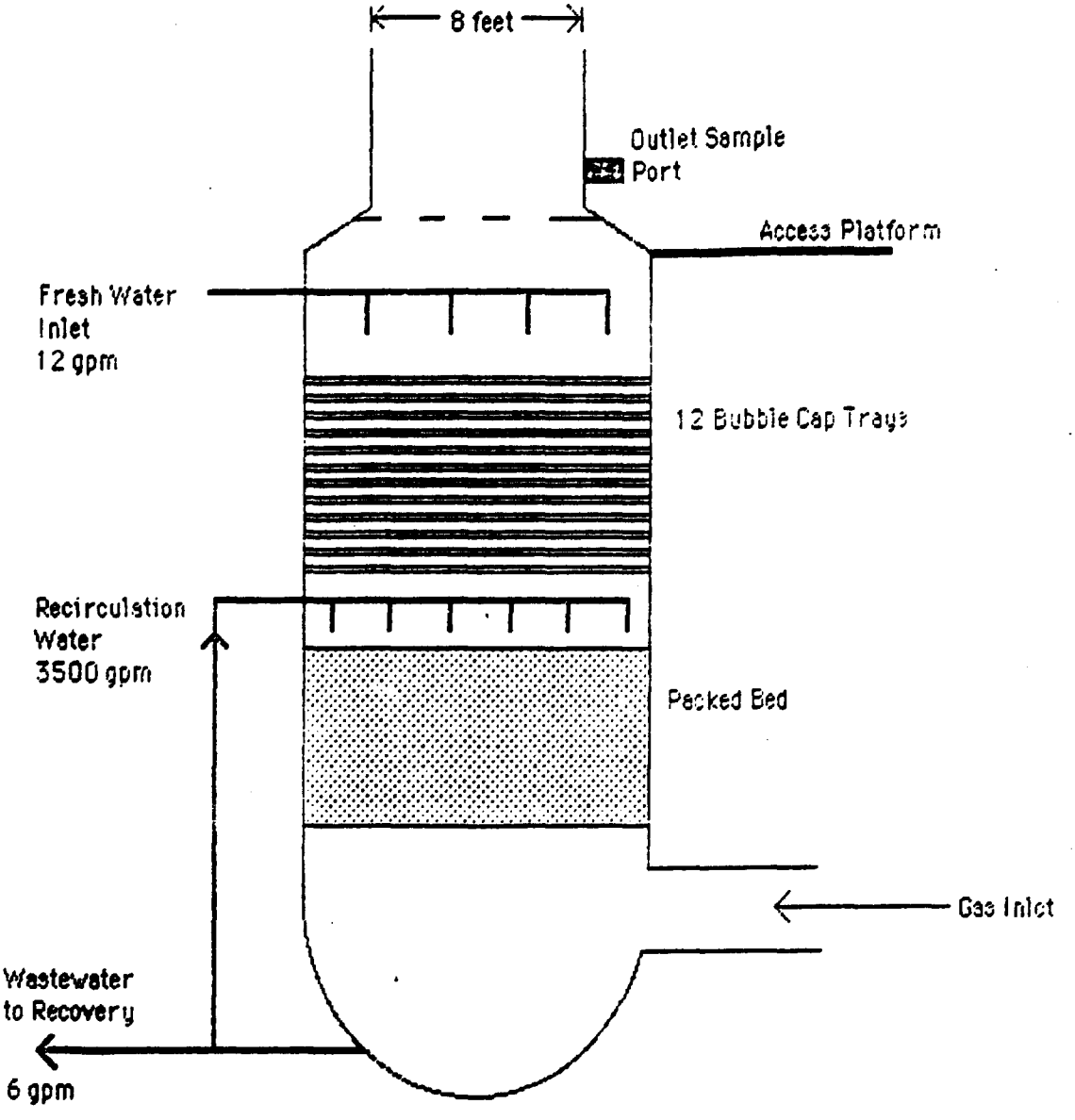


Figure 2 - Scrubber Schemetic

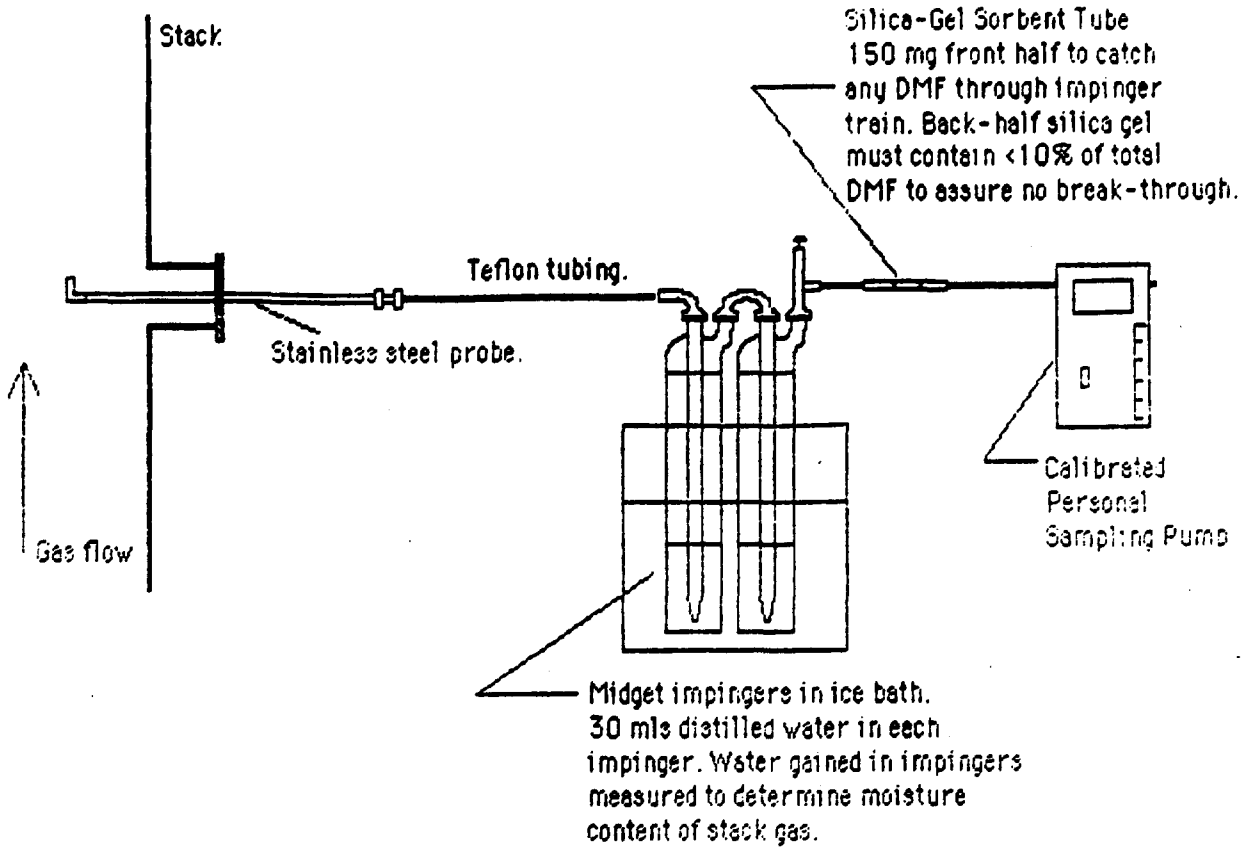


Figure 3 - Sampling Train

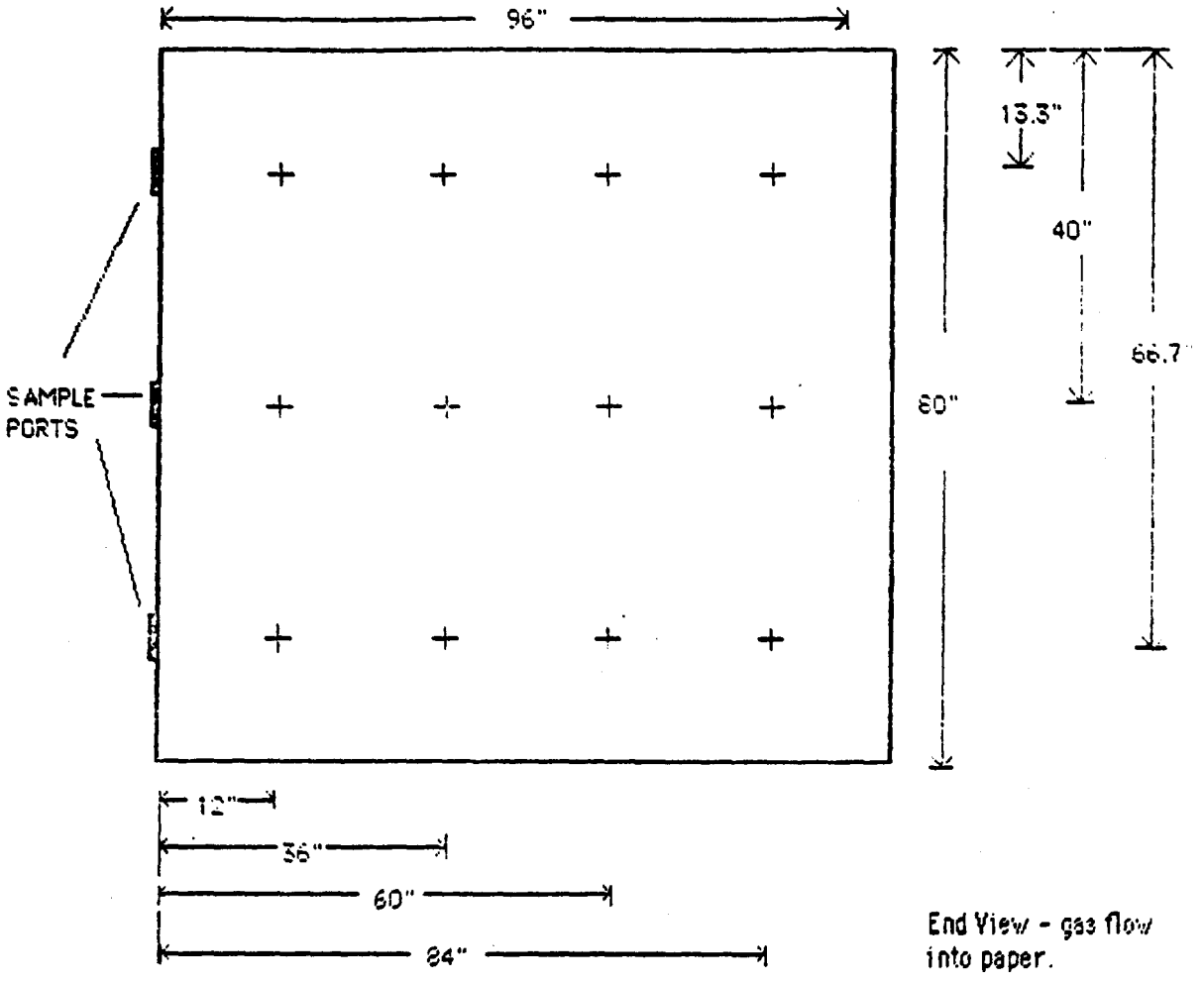


Figure 4 - Traverse Point Locations for Inlet Duct

APPENDIX A

COMPLIANCE TEST LOG

Compliance Test Log - 10/31/89

Run 1	-	Start Time:	09:48
		End Time:	10:48
Run 2	-	Start Time:	12:58
		End Time:	13:58
Run 3	-	Start Time:	14:54
		End Time:	15:54

APPENDIX B

STACK VELOCITY DATA FIELD SHEETS AND CALIBRATION DATA FOR SAMPLING PUMPS

FIELD DATA SHEET

Project Dupont
Location Waynesboro
Date 10-31-89 TK

RUN NUMBER 1 Inlet
START TIME 0947
END TIME 1046

Traverse Data

PRE-TEST

Time	0925	
POINT	ΔP STACK In. W.C.	T STACK Deg. F
A1	.086	146
2	.097	155
3	.098	158
4	.097	152
B1	.091	148
2	.102	159
3	.105	157
4	.100	161
C1	.075	145
2	.081	148
3	.089	150
4	.082	153

Static -1.04

POST-TEST

Time		
POINT	ΔP STACK In. W.C.	T STACK Deg. F
A1	[illegible]	[illegible]
2	.094	[illegible]
3	[illegible]	176
4	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
2	[illegible]	[illegible]
[illegible]	.103	[illegible]
[illegible]	.16[illegible]	164
C1	[illegible]	[illegible]
2	.089	[illegible]
3	.094	[illegible]
4	.089	178
	0.3084	164.75

Temp 1 - 18
2 - 16
Press - 30 mls

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - lpm
Pre-Test	32, 32, 32	750	1.41
Post-Test	32, 32, 32	750	1.41
		AVERAGE	1.41

Notes

Initial Imp Exit T° - 68F
Final 62°

FIELD DATA SHEET

Project DuPont 99-210

Location Outlet

Date 10/31/99 TJ

RUN NUMBER 1

START TIME 09:48

END TIME 10:48

Traverse Data

PRE-TEST

Time		
POINT	ΔP STACK In. W.C.	T STACK Deg. F

POST-TEST

Time		
POINT	ΔP STACK In. W.C.	T STACK Deg. F

Imp 1 - 18
Imp 2 - 14
P.W. - 69

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - 1pm
Pre-Test	30s	[illegible]	1.42
Post-Test	30s	[illegible]	1.42
		AVERAGE	1.42

Notes

Set point 5.0
Impinger exit 58°F
[illegible]

FIELD DATA SHEET

Project Dupont
Location Waynesboro
Date 10-31-89 TW

RUN NUMBER 2 Inlet
START TIME 1255
END TIME 1358

Use Pretest from Run 1 Post test

Traverse Data

Post ~~PRE~~-TEST

Time	1436	
POINT	ΔP STACK In. W.C.	T STACK Deg. F
A1	.086	146
2	.088	158
3	.095	160
4	.097	160
B1	.082	152
2	.098	158
3	.096	157
4	.092	161
C1	.066	149
2	.073	151
3	.085	154
4	.084	153
	0.3041	166.47

Static -.983

POST-TEST

Time		
POINT	ΔP STACK In. W.C.	T STACK Deg. F

Imp 1 - 20
Imp 2 - 17
P.W. - 17

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - lpm
Pre-Test	29,29,29	750	1.55
Post-Test	28,28,29	750	1.59
		AVERAGE	1.57

Notes

Initial Imp. Exit T - 63° F
Final 66° F

FIELD DATA SHEET

Project Dupont 89-210
Location outlet
Date 10-31-89TJ

RUN NUMBER 2
START TIME 12:58
END TIME 13:58

Traverse Data

PRE-TEST

Time		
POINT	ΔP STACK In. W.C.	T STACK Deg. F

POST-TEST

Time		
POINT	ΔP STACK In. W.C.	T STACK Deg. F

Imp 1 - 23
Imp 2 - 16
P.W - 38

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - lpm
Pre-Test	30s	770	1.54
Post-Test	30s	760	1.52
		AVERAGE	1.53

Notes

temp - 60°F

FIELD DATA SHEET

Project Dupont
Location Waynesboro, VA
Date 10-31-89 TU

RUN NUMBER 3 Inlet
START TIME 1454
END TIME 1554

Traverse Data

PRE-TEST

Time	1628	
POINT	ΔP STACK In. W.C.	T STACK Deg. F
A1	.064	
2	.068	
3	.067	
4	.075	
B1		
2		
3		
4		
C1		
2		
3		
4		

POST-TEST

Time	1632	
POINT	ΔP STACK In. W.C.	T STACK Deg. F
A1	.085	153
2	.088	161
3	.108	164
4	.100	159
B1	.082	153
2	.093	157
3	.098	157
4	.092	159
C1	.063	158
2	.075	162
3	.075	160
4	.068	157

Static 1.062

~~16~~ 156.62

Imp 1-23
Imp 2-15
P.W-16

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - lpm
Pre-Test	30,30,30	750	1.50
Post-Test	30,30,29	730	1.50
		AVERAGE	1.50

Notes

Initial Imp T -	650
Final	680

FIELD DATA SHEET

Project Dupont 89-210
Location outlet
Date 10-31-89 TJ

RUN NUMBER 3
START TIME 14:54
END TIME 15:54

Traverse Data

PRE-TEST

Time		
POINT	ΔP STACK In. W.C.	T STACK Deg. F

POST-TEST

Time		
POINT	ΔP STACK In. W.C.	T STACK Deg. F

Imp 1-25
Imp 2-16
P.W. 35

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - lpm
Pre-Test	30s	730	1.46
Post-Test	30s	750	1.50
		AVERAGE	1.48

Notes

temp - 65°F

APPENDIX C

ANALYTICAL METHODS

(1) DIMETHYLACETAMIDE and (2) DIMETHYLFORMAMIDE

FORMULA: (1) $CH_3CON(CH_3)_2$; C_4H_9NO
(2) $O{=}CHN(CH_3)_2$; C_3H_7NO
M.W.: (1) 87.12; (2) 73.10

METHOD: 2004
ISSUED: 5/15/85

OSHA: 10 ppm (skin) [(1) and (2)]
NIOSH: no standard
ACGIH: 10 ppm (skin) [(1) and (2)]
[1 ppm = 3.56 mg/m³ @ NTP (1)]; [1 ppm = 2.99 mg/m³ @ NTP (2)]

PROPERTIES: Table 1

SYNONYMS: (1): N,N-dimethylacetamide; CAS #127-19-5.
(2): N,N-dimethylformamide; DMF; CAS #68-12-2.

SAMPLING

SAMPLER: SOLID SORBENT TUBE
(silica gel, 150 mg/75 mg)

FLOW RATE: 0.01 to 1 L/min

VOL-MIN: 15 L @ 30 mg/m³
-MAX: 80 L

SHIPMENT: routine

SAMPLE STABILITY: stable ≥ 5 days @ 25 °C [1]

FIELD BLANKS: 10% (≥2) of samples

ACCURACY

RANGE STUDIED: (1): 18 to 105 mg/m³ [1];
(2): 11 to 61 mg/m³ [1]
(45-L samples)

BIAS: not known [1]

OVERALL PRECISION (s_r): (1): 0.067 [1];
(2): 0.056 [1]

MEASUREMENT

TECHNIQUE: GAS CHROMATOGRAPHY, FID

ANALYTE: dimethylacetamide or dimethylformamide

DESORPTION: 1 mL methanol; 1 hr in ultrasonic bath

INJECTION VOLUME: 5 µL

TEMPERATURE-INJECTION: 240 °C
-DETECTOR: 320 °C
-COLUMN: 140 °C

CARRIER GASES: N_2, 50 mL/min

COLUMN: 1.5 m x 6 mm OD glass; 10% UCON 50-HB-5100, 2% KOH on 100/120 mesh Chromosorb WHP

CALIBRATION: analyte in methanol

RANGE: 0.5 to 4 mg per sample [1,2]

ESTIMATED LOD: 0.05 mg per sample [2]

PRECISION (s_r): (1): 0.032 [1];
(2): 0.037 [1]

APPLICABILITY: The working range is 10 to 80 mg/m³ of either analyte for a 50-L air sample. The lower limit is determined by the desorption efficiency which must be determined over the range used. Silica gel has a high affinity for water; high relative humidity will adversely affect the efficiency of analyte adsorption.

INTERFERENCES: None identified. Separation conditions (column, temperature, etc.) may be changed to circumvent problems. Alternate columns include: 60/80 mesh Chromosorb P coated with 20% UCON LB 550X and 2% KOH; 100/120 mesh Chromosorb WHP with 10% Carbowax 20M and 2% KOH; 100/120 mesh Chromosorb WHP with 10% SP-2250; and 5% FFAP.

OTHER METHODS: This combines and replaces Methods S254 and S255 [2].

REAGENTS:

1. Methanol, reagent grade.*
2. Analyte: dimethylacetamide, reagent grade, or dimethylformamide, reagent grade.*
3. Acetone, reagent grade.*
4. Desorption efficiency (DE) stock solution, 0.05 mg/mL. Prepare solutions of dimethylacetamide or dimethylformamide fresh daily in acetone.
5. Hydrogen, prepurified.
6. Air, filtered, compressed.
7. Nitrogen, purified.

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6 mm OD, 4 mm ID, flame-sealed, with plastic caps, containing two sections of 20/40 mesh silica gel (front = 150 mg; back = 75 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section. The pressure drop across the tube must be less than 3.4 kPa (2.5 cm Hg) at an airflow of 1 L/min. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 1 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator, and column (page 2004-1).
4. Vials, 2-mL, PTFE-lined crimp cap, or automatic sampler vials.
5. Microliter syringes, 10-µL and other convenient sizes for making standards.
6. Pipets, 1.0 mL, delivery.
7. Volumetric flasks, 10-mL.
8. Ultrasonic bath.

SPECIAL PRECAUTIONS: Acetone and methanol are flammable and a dangerous fire and explosion risk. They are moderately toxic by ingestion and inhalation.

Dimethylacetamide and dimethylformamide are strong irritants to skin and tissue and moderate fire risks.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.01 and 1 L/min for a total sample size of 15 to 80 L.
4. Cap the samplers. Pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials. Discard the glass wool and foam plugs.
6. Add 1.0 mL methanol to each vial. Attach crimp cap to each vial.
7. Agitate for 60 min in an ultrasonic bath.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least five working standards over the ranges 0.05 to 4 mg dimethylacetamide or dimethylformamide per sample.
   a. Add known amounts of analyte (neat or diluted with methanol) to methanol in 10-mL volumetric flasks and dilute to the mark.
   b. Analyze together with samples and blanks (steps 11 and 12).
   c. Prepare calibration graph (peak area vs. mg analyte).

9. Determine desorption efficiency (DE) at least once for each lot of silica gel used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
   a. Remove and discard back sorbent section of a media blank sampler.
   b. Inject a known amount (1 to 20 µL) of pure analyte or DE stock solution directly onto front sorbent section with a microliter syringe.
   c. Cap the tube. Allow to stand overnight.
   d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
   e. Prepare a graph of DE vs. mg analyte recovered.
10. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 2004-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
    NOTE: If peak area is above the linear range of the working standards, dilute with methanol, reanalyze, and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of analyte found in the sample front (W_f) and back (W_b) sorbent sections, and in the average media blank front (B_f) and back (B_b) sorbent sections.
    NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
14. Calculate concentration, C, of analyte in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b) \cdot 10^3}{V} \cdot \text{mg/m}^3.$$

EVALUATION OF METHOD:

Method S254 for dimethylacetamide was evaluated over the range of 18 to 105 mg/m³ at an atmospheric temperature and pressure of 24 °C and 760 mm Hg using a 45-L sample [1]. Breakthrough occurred when sampling a test atmosphere containing 105.6 mg/m³ of dimethylacetamide at 0.876 L/min for 240 min. The front section of the silica gel tube was found to hold 22.2 mg dimethylacetamide under these conditions. The collection efficiency test conducted at a concentration of 105.6 mg/m³ was determined to be 1.00. Desorption efficiency at 0.943, 1.886, and 3.77 mg per silica gel tube was 88.8, 93.8, and 94.9%, respectively. A storage study for five days at 1.866 mg per silica gel tube gave a recovery of 93.6%. Overall sampling and measurement precision, s_r, was 0.067.

Method S255 for dimethylformamide was evaluated over the range of 11 to 61 mg/m³ at an atmospheric temperature and pressure of 23 °C and 761 mm Hg using a 45-L sample [1]. Breakthrough occurred when a test atmosphere containing 119.5 mg/m³ of dimethylformamide was sampled at 0.859 L/min for 146 min. The front section of the silica gel tube was found to hold 15 mg of dimethylformamide under these conditions. The collection efficiency test conducted at a concentration of 61.1 mg/m³ was determined to be 1.00. Desorption efficiency at 0.759, 1.518, and 3.04 mg per silica gel tube was 88.7, 90.4, and 92.2%, respectively. A storage study for five days at 1.5 mg dimethylformamide per silica gel tube gave a recovery of 91.7%. Overall sampling and measurement precision, s_r, was 0.056.

REFERENCES:
[1] Documentation of the NIOSH Validation Tests, S254 (Dimethylacetamide) and S255 (Dimethylformamide), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as GPO Stock #017-033-00231-2 from Superintendent of Documents, Washington, DC 20402.
[2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, S254 and S255, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).

METHOD REVISED BY: C. Neumeister, NIOSH/DPSE; S254 and S255 originally validated under NIOSH Contract CDC-99-74-45.

Table 1. Properties.

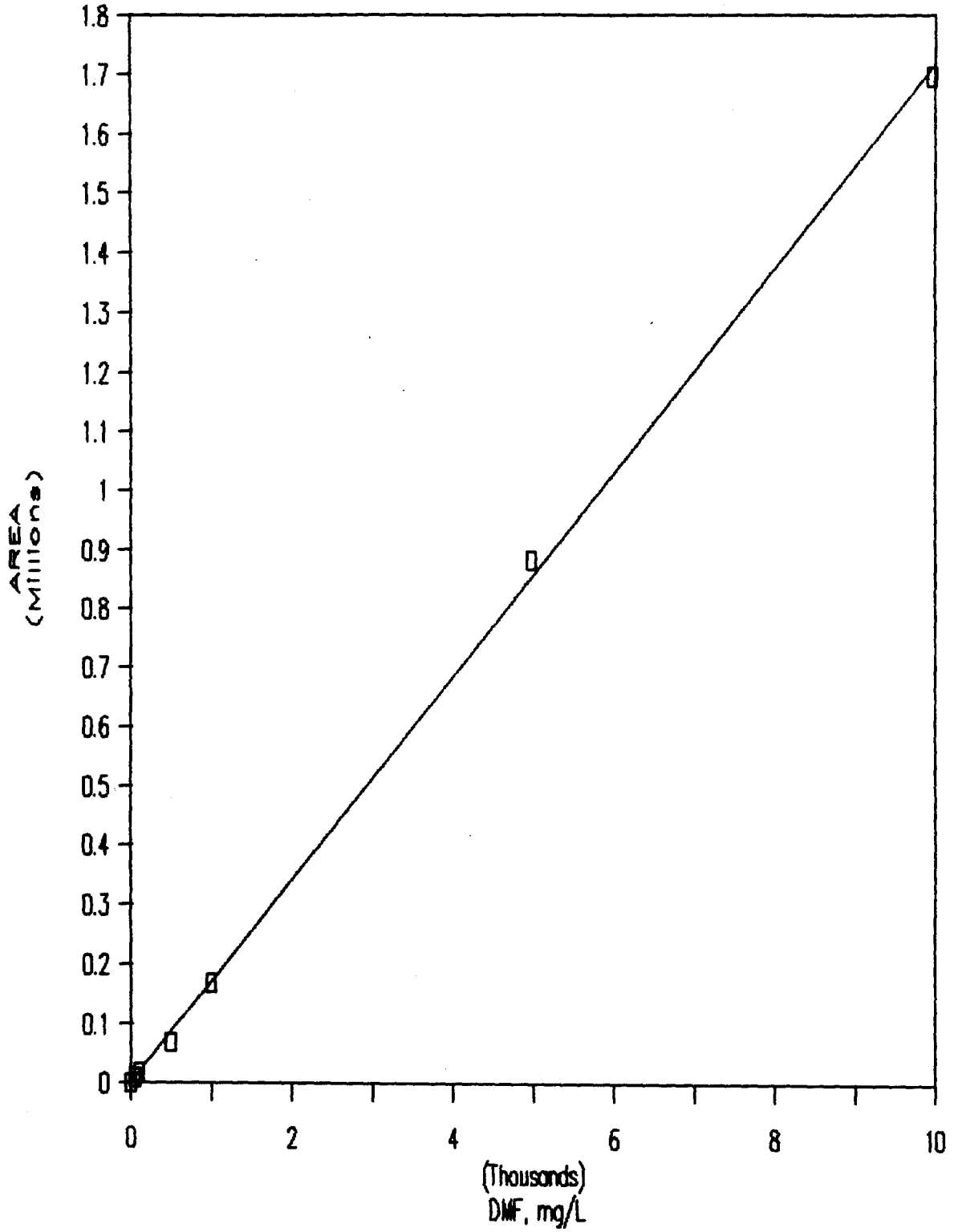
Analyte	Liquid density, g/mL @ 25 °C	BP (°C)	MP (°C)	VP @ 25 °C Pa	VP @ 25 °C mm Hg	VP @ 25 °C ppm	Explosive range in air, % v/v
Dimethylacetamide	0.9366	166	-20	170	1.3	1700	1.8 to 11.5
Dimethylformamide	0.9440	153	-60	490	3.7	4900	2.2 to 15.2

APPENDIX D

ANALYTICAL DATA

E.I. du Pont - 89-210

DMF STUDY - SCRUBBER 1



Standards

Analyte: DMF

Solvent: DI H_2O

Analyst: LG Date: 10/30/8

Client: Dupont

Standard Concentrations	Procedure
9960 mg/L	498 mg DMF diluted to 50 mls DI H_2O
4980 mg/L	5 mls 9960 mg/L stnd + 5 mls DI H_2O
996 mg/L	2 mls 4980 mg/L stnd + 8 mls DI H_2O
498 mg/L	1 ml 4980 mg/L stnd + 9 mls DI H_2O
100 mg/L	1 ml 498 mg/L stnd + 4 mls DI H_2O
50 mg/L	1 ml 498 mg/L stnd + 9 mls DI H_2O

DuPont Scrubber #1
Compliance Test 8-4-216

Analysis for: DMF (N,N-Dimethylformamide) Analyst: TH Date: 6/3/97

Method: 2000 Reference: NIOSH

Sample Collection:

X Impinger: Volume: 15 ml Solution: DIW

X Tube: Type: Silica Gel

Desorption: Volume: 1.0 ml Solvent: MeCl2

GC Conditions:

Injector Temp: 225 °C Injector volume: 0.5 ul

Detector Temp: 225 °C Detector (FID) Range: 10

Column Type: 0.53mm ID, Supelcowax 10, 15m

Column Temp: Initial: 100 °C Time: 1.0 min

Ramp: °C/min

Final: °C Time: min

Carrier: Gas: N2 Flowrate: 40 ml/min

Sample ID	Collection Time (min)	Final Sample Volume (ml)	Retention Time	Area	
9960 ppm DMF			3.066	1643182	
9960 ppm DMF			3.102	1755149	
4980 ppm DMF			3.094	975683	
996 ppm DMF			3.061	167527	
198 ppm DMF			3.157	68784	
100 ppm DMF			3.131	16891	
50 ppm DMF			3.062	8491	

DuPont Scrubber # 1
Compliance Test 89-210

Analysis for: DMF (N,N-Dimethylformamide) Analyst: T.H. Date: 10/31/89

Method: 2004 Reference: NICSH

Sample Collection:

X Impinger: Volume: 15 ml Solution: DIW

X Tube: Type: Silica Gel

Desorption: Volume: 1.0 ml Solvent: MeOH

GC Conditions:

Injector Temp: 225 °C Injector volume: 0.5 ul

Detector Temp: 225 °C Detector (FID) Range: 10

Column Type: 0.53mm ID Supercox II, 10m

Column Temp: Initial: 100 °C Time: 1 min

Ramp: °C/min

Final: °C Time: min

Carrier: Gas: N_2 Flowrate: 4.0 ml/min

Sample ID	Collection Time (min)	Final Sample Volume (ml)	Retention Time	Area	
9960 ppm DMF			3.066	1643182	
9960 ppm DMF			3.102	1755149	
4980 ppm DMF			3.094	975683	
996 ppm DMF			3.061	167527	
498 ppm DMF			3.157	68784	
100 ppm DMF			3.131	16891	
50 ppm DMF			3.062	8491	

21-210 Scrubber #1

Laboratory Analysis DMF Continuation page 2

	Sample ID	Collection Time (min)	Final Sample Volume (ml)	Retention Time	Area	
	Blank				0	
09:40	Scrubber Bottom A	dil 1:11		3.104	423637	2.72%
10:50	Scrubber Bottom B	dil 1:11		3.103	431419	2.76%
	Run 1 Inlet Probe Wash		30	3.198	215614	
	Run 1 Inlet Imp 1		18	3.166	285567	
	Run 1 Inlet Imp 2		16	3.09	6438	
	Run 1 Outlet Probe Wash		69	3.08	2116	
	Run 1 Outlet Imp 1		18	3.183	2229	
	Run 1 Outlet Imp 2		14	3.102	529	
	1/19 Scrubber DMF			3.127	791508	
12:52	Scrubber Bottom C	dil 1:11		3.095	443139	[illegible]
[illegible]	Scrubber Bottom D	dil 1:11		3.149	481303	[illegible]
	Scrubber D Dup	dil 1:11		3.212	413845	
	Run 2 Inlet Probe Wash		17	3.223	293682	
	Run 2 Inlet Imp 1		20	3.221	353938	
	Run 2 Inlet Imp 2		17	3.155	28042	
	Run 2 Outlet Probe Wash		38	3.183	3111	
	Run 2 Outlet Imp 1		23	3.221	8644	
	Run 2 Outlet Imp 2		16	3.144	1165	
15:00	Scrubber Bottom E	dil 1:11		3.185	509990	
	Run 3 Inlet Probe Wash		16	3.228	366769	
	Run 3 Inlet Imp 1		23	3.227	166433	
	Run 3 Inlet Imp 2		15	3.222	16964	

89-21C Scrubber A1

Laboratory Analysis DMF Continuation page 3

Sample ID	Collection Time (min)	Final Sample Volume (ml)	Retention Time	Area	
Run 3 Outlet Imp	1	24	3.17	10651	
Run 3 Outlet Imp	2	16	3.183	2560	
Run 3 Outlet Probe	Wash	55	3.183	2026	
Scrubber bottom F	1:11		3.144	53876	
498 ppm DMF			3.221	82274	

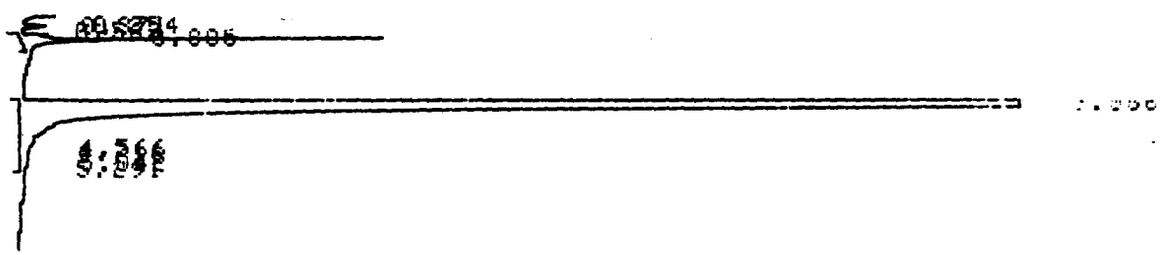
DUPONT 89-210 10/31/89
DMF STACK 1 COMPLIANCE TEST

SUPEROXII,10M,0.53MMID
COL 100C

ANALYSIS PARAMETER, FILE 0

WIDTH	5	SLOPE	282.24
DRIFT	0	MIN.AREA	1500
T.DBL	0	STOP.TM	8
ATTEN	4	SPEED	4
METHOD#	2021	FORMAT#	0
SPL.WT	100	IS.WT	1

5960PPM DMF



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 1

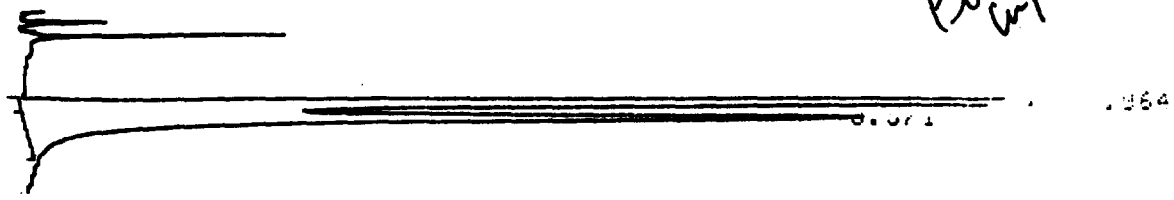
PKNO	TIME	AREA	MK	TYPE	CONC	PHASE
1	0.805	14994 3729nl	V		0.9042	
2	3.066	1643182 225985nl	S		99.0958	
	TOTAL	1658176			100	

025

9960PPM DMF
TIME PROGRAM FILE 0

0.1 LOCK ON
0.5 LOCK OFF

Bad run

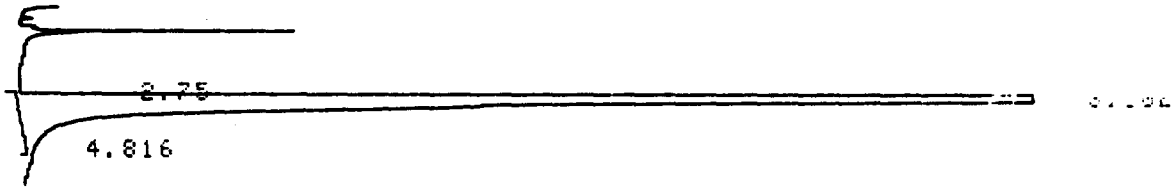


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 2

PKNO	TIME	AREA	MK	TYPE	CONC	NAME
1	3.064	1271627 198808n1			82.3783	
2	3.571	260854 13252n1	V		17.0217	
	TOTAL	1532480			100	

9960PPM DMF



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 3

PKNO	TIME	AREA	MK	TYPE	CONC	NAME
1	3.102	1755149 223487n1	S		100	
	TOTAL	1755149			100	

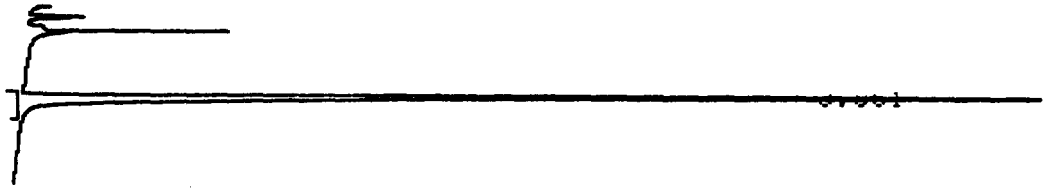
223-02037-01 904124A Shimadzu

CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 4

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.094	975683			100	
		126088ni				
	TOTAL	975683			100	

996PPM DMF

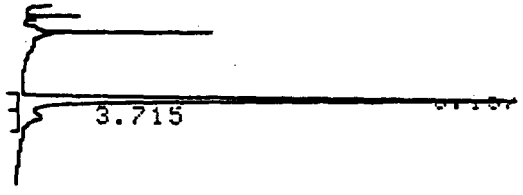


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 5

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.061	167527			100	
		24045ni				
	TOTAL	167527			100	

498PPM DMF

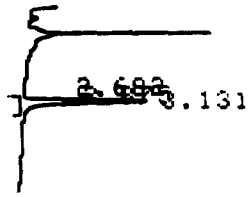


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 6

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.157	68784			90.4366	
		7866ni				
2	3.715	7279	V		9.5692	
		372ni				
	TOTAL	[illegible]			[illegible]	

026
223-02037-01
904124A
Shimadzu

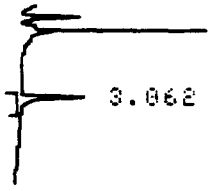


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 7

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.131	16691	V		100	
		197701				
	TOTAL	16691			100	

50PPM DMF

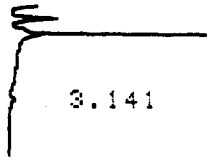


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 8

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.062	8491			100	
		108701				
	TOTAL	8491			100	

BLNK



CHROMATOGRAM 1 MEMORIZED
WARNING NO PEAK
SCRUBBER BOTTOM WATER - A

027

223-02037-01

904124A

Shimadzu

DIL 1:11

CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 9

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.104	423637 58793ni			90.6977	
2	3.569	43450 2043ni	SV		9.3023	
TOTAL		467087			100	

SCRUBBER BOTTOM WATER - B
DIL 1:11

CHROMATOGRAM 1 MEMORIZED

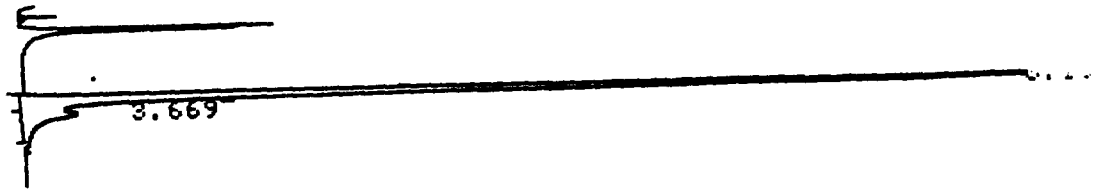
CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 10

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.173	431419 59275ni			83.9394	
2	3.76	82424 3203ni	SV		16.0406	
TOTAL		513843			100	

028

223-02037-01

904124A

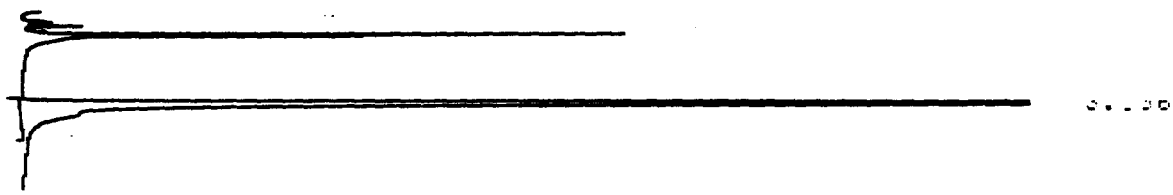


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 11

PKNO	TIME	AREA	MK	TYNO	CONC	NAME
1	3.198	215614			91.0909	
		24264hi				
2	3.669	19109	V		8.141	
		885hi				
	TOTAL	234723			100	

RUN1 INLET IMP1

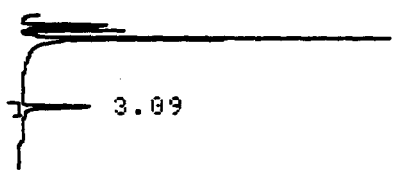


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 12

PKNO	TIME	AREA	MK	TYNO	CONC	NAME
1	3.106	285567			100	
		37696hi				
	TOTAL	285567			100	

RUN1 INLET IMP2



CHROMATOGRAM 1 MEMORIZED

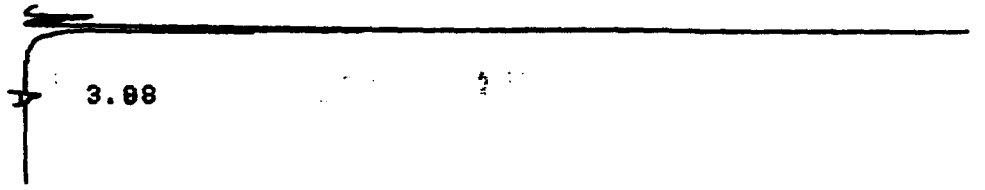
CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 13

PKNO	TIME	AREA	MK	TYNO	CONC	NAME
1	3.09	6438			100	
		1115hi				
	TOTAL	6438			100	

029

223-02037-01

90412



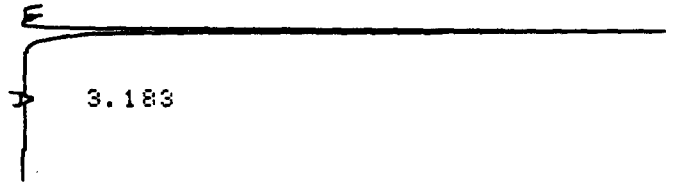
CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 14

DEC 18 1989

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.08	2116			100	
		297ni				
	TOTAL	2116			100	

RUN1 OUTLET IMP1

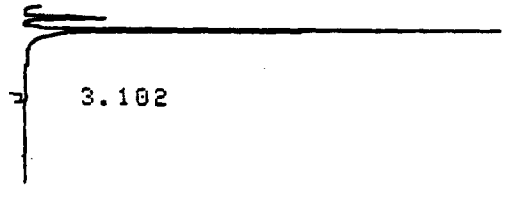


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 15

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.183	2229			100	
		223ni				
	TOTAL	2229			100	

RUN1 OUTLET IMP2



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 16

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.102	529			100	
		76ni				
	TOTAL	529			100	

4980PPM DMF

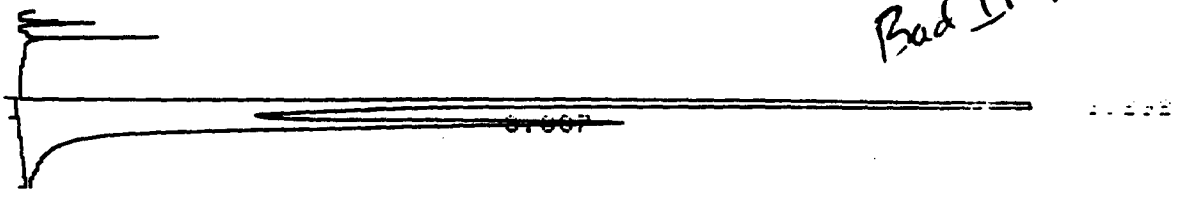
030

223-02037-01

9041

4980PPM DMF

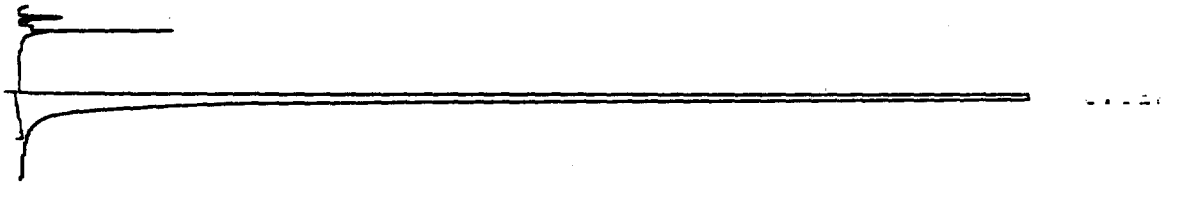
Bad I(2)



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 17

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.202	650937 70381hi			73.6362	
2	3.887	230469 9533hi	V		26.1479	
	TOTAL	881407			100	



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 18

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.127	791508 110956hi			100	
	TOTAL	791508			100	

9041724A

Shimadzu

SCRUBBER BOTTOM WALK
DIL 1:11

CHROMATOGRAM MEMORIZED
WARNING NO PEAK

CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 19

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.095	443139 64312n1			96.0641	
2	3.555	45955 2055n1	V		9.3959	
	TOTAL	489093			100	

SCRUBBER WATER - D
DIL 1:11

CHROMATOGRAM 1 MEMORIZED

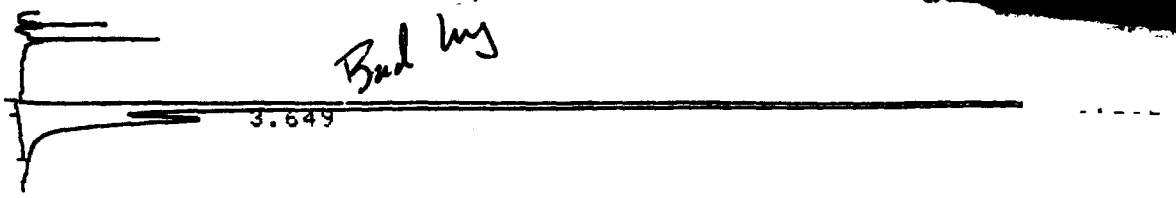
CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 20

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.149	489303 69582n1			100	
	TOTAL	489303			100	

031
223-02037-01
904124A
Shimadzu

SCRUBBER D DUP

Bad Inj

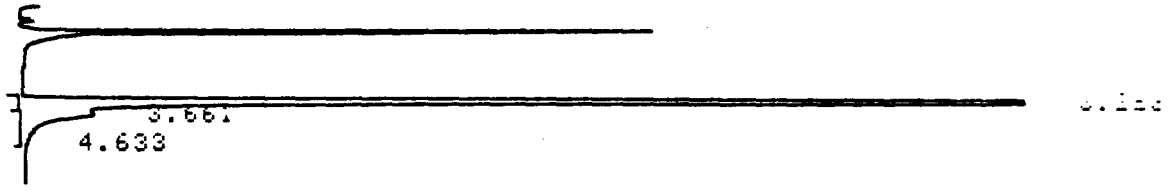


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 21

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.212	413840			86.4724	
		59477mi				
2	3.649	64741	V		13.5276	
		2845mi				
	TOTAL	478587			100	

RUN2 INLET PROBE WASH



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 22

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.223	293662			91.7623	
		37432mi				
2	3.661	26365	SV		8.2377	
		1176mi				
	TOTAL	320064			100	

032
223-02037 01
904124A

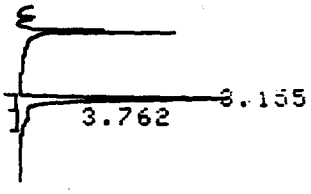
inlet imp1

CHROMATOGRAM 1 MEMORIZED

CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 23

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.221	353958	S		98.7541	
		33412hi				
2	3.658	4465	T		1.2458	
		346hi				
	TOTAL	358423			100	

RUN2 INLET IMP2

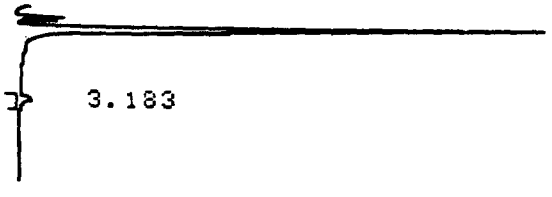


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 24

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.155	28642			89.3178	
		3279hi				
2	3.762	3354	V		10.6822	
		146hi				
	TOTAL	31395			100	

RUN2 OUTLET PROBE WASH



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 25

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.183	3111			100	
		252hi				
	TOTAL	[illegible]			[illegible]	

Run2

Shimadzu

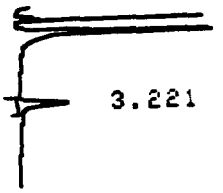
033

223-02037-01

904124A

Run2 Outlet Imp1

TOTAL	3111	100

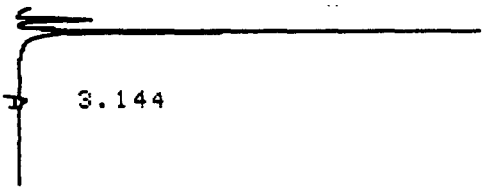


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 26

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.221	8644			100	
		807ml				
TOTAL		8644			100	

RUN2 OUTLET IMP2



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 27

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.144	1165			100	
		184ml				
TOTAL		1165			100	

SCRUBBER BOTTOM WATER - E
DIL 1:11

CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 28

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.185	509990	S		100	
		72740ml				

Shimadzu

904124A

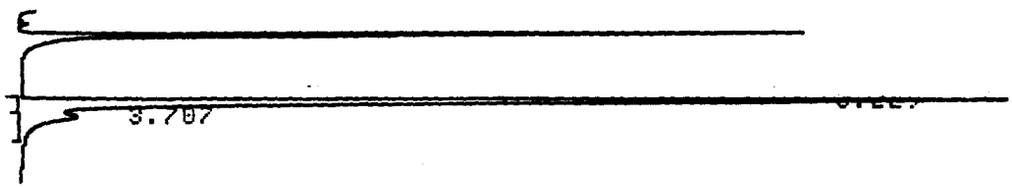
RUN3 INLET PROBE WASH

CHROMATOGRAM 1 MEMORIZED

CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 29

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.228	366709 44741hi			90.5159	
2	3.708	38423 1580hi	V		9.4841	
	TOTAL	405132			100	

RUN3 INLET IMP1

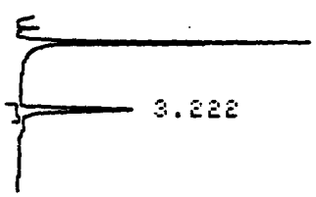


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 30

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.229	168433 17053hi			88.989	
2	3.707	20841 936hi	V		11.011	
	TOTAL	189275			100	

RUN3 INLET IMP2



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 31

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.222	16964			100	

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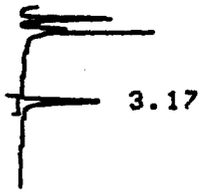
904124A

RUN3 INLET IMP1



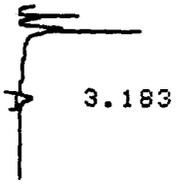
CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0

SAMPLE NO 0 METHOD 2021
REPORT NO 32

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.17	10631			100	
		1266hi				
	TOTAL	10631			100	

RUN3 OUTLET IMP2

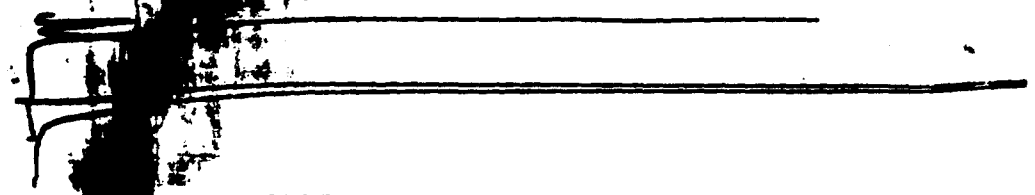


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 33

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.183	2560			100	
		242hi				
	TOTAL	2560			100	

DIL 1:11

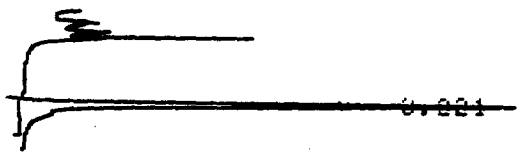


CHROMATOGR[illegible] 1 MEMORIZED

CR501 CH[illegible]OPAC
CHANNEL [illegible] FILE 0
SAMPLE [illegible] METHOD 2021
REPORT [illegible] 35

PKNO	[illegible]ME	AREA	MK	IDNO	CONC	NAME
1	[illegible].144	538761			100	
		61418hi				
	TOTAL	538761			100	

498PPM



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 36

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.221	80274			100	
		7825hi				
	TOTAL	80274			100	

223-02037-01

APPENDIX E

PLANT OPERATIONAL DATA

SCRUBBER NUMBER 1

TESTED 10/31/89

TEST NUMBER 1

MEASUREMENT	START	MID-TEST	END
TIME	09:48		10:48
DP ACROSS SCREEN, IN. H2O		0.18	
PRESSURE BELOW BED, IN. H2O	30	31	31
FAN MOTOR CURRENT, AMP.		120	
VENTURI FLOW, MSCFM*	39	40	39
WATER FEED, LB/HR	5350	5300	5350
WATER TAKE-OFF, LB/HR		ABOUT 3000 LB/HR	
RECIRCULATING FLOW, GPM		ABOUT 2860	
BOTTOMS TEMP., DEG. C		51	

* VENTURI FLOW HAS NOT BEEN CALIBRATED AND IS READING LOW. ETS,INC. READINGS SHOULD BE USED.

SCRUBBER NUMBER 1

TESTED 10/31/89

TEST NUMBER 2

MEASUREMENT	START	MID-TEST	END
TIME	12:58		13:58
DP ACROSS SCREEN, IN. H2O		0.18	
PRESSURE BELOW BED, IN. H2O	31	31	31
FAN MOTOR CURRENT, AMP.		119	
VENTURI FLOW, MSCFM*	39	40	40
WATER FEED, LB/HR	5200	5300	5200
WATER TAKE-OFF, LB/HR		ABOUT 3500 LB/HR	
RECIRCULATING FLOW, GPM		ABOUT 2860	
BOTTOMS TEMP., DEG. C		52	

* VENTURI FLOW HAS NOT BEEN CALIBRATED AND IS READING LOW. ETS,INC. READINGS SHOULD BE USED.

SCRUBBER NUMBER 1

TESTED 10/31/89

TEST NUMBER 3

MEASUREMENT	START	MID-TEST	END
TIME	14:54		15:54
DP ACROSS SCREEN, IN. H20		0.20	
PRESSURE BELOW BED, IN. H20	30	30	30
FAN MOTOR CURRENT, AMP.		119	
VENTURI FLOW, MSCFM*	39	37	39
WATER FEED, LB/HR	5300	5200	5200
WATER TAKE-OFF, LB/HR		ABOUT 3400 LB/HR	
RECIRCULATING FLOW, GPM		ABOUT 2860	
BOTTOMS TEMP., DEG. C		51	

* VENTURI FLOW HAS NOT BEEN CALIBRATED AND IS READING LOW. ETS, INC. READINGS SHOULD BE USED.