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COMMONWEALTH OF VIRGINIA
Department of Air Pollution Control

INTRA-AGENCY MEMORANDUM

TO: File

FROM: R. O. Goetz

SUBJECT: DUPONT, WAYNESBORO, REGISTRATION #20517

DATE: October 12, 1989

RE: STACK TEST 10-6-89 NEW DMF SCRUBBERS ON ORLON FIBER DRYERS

A witnessed stack test was performed 10-6-89 on one of two (#2 scrubber, near river) new large scrubbers for DMF (VOC) scrubbing efficiency, inlet and outlet concentrations and pounds per hour emission rates. The scrubber tested was fully loaded (one-half scrubbing capacity) by full production on one-half (3) of the six orlon fiber dryers.

Opacity was always zero (0%) except for a short moisture plume - VEE's attached.

See the attached stack test monitoring sheet and proposed test protocol by ETS for more details.

- "Preliminary" results indicate (average of three one hour test runs):
- 96.6% scrubbing efficiency
- Inlet DMF: 332 ppm, 175.5 lb/hr
- Outlet DMF: 11.6 ppm, 5.9 lb/hr
- DMF concentration in scrubber water reservoir: 2.6% Plant hopes to increase this to 9 or 10% for more efficient recovery.
- Inlet feed water: 4000 lbs/hr -- 8 gal/min.
- Ran about six days before test day.

PERSONNEL PRESENT

duPont
Ron Shifflett - Environmental
Paul Dana - Orlon Process Engineer - Scrubber "father"
Merv Meckley - Wilmington, Del. - "Scrubber designer"
Jim Harris - Orlon manufacturing supervisor
Ed Mongan - Environmental
George Aylor - Retired duPont - construction coordinator
David Hopkins - Fiber Dryers

ETS
Ed Handel
Tony Underwood
Lori Graves

DAPC
Ray Goetz
Sam Barnard (VEE)

R. O. Goetz

Raymond O. Goetz
Engineer, Region II

ROG/bh

Attachments

cc: Division of Technical Evaluation
S. K. Barnard

STACK TEST MONITORING

10-6-89

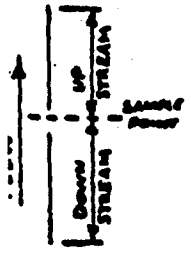
SOURCE NAME: DuPont #20517
LOCATION: Waynesboro — new DMF fiber dryers scrubber no2 near river
DATE: 10-6-89 OBSERVER'S NAME: R. O. GOETZ

PURPOSE OF TEST: COMPLIANCE - DMF reduction
TESTING DONE BY: ETS of ROANOKE — TED HANDEE, TORY UNDERWOOD, LORI GRAVES.
LAB ANALYSIS DONE BY: SAME - ON SITE

COMPANY CONTACT: RON SHIFFLETT TELEPHONE:
CONTROL EQUIPMENT OPERATING: — #2 DMF SCRUBBER (NEW RIVER) = 8 FT PACKED BED
OPACITY READING MADE: YES — NO — S.V. BARNARD - R.O. GOETZ SPOT CHECKING 0% OVG #2 BUBBLE CAP TRAYS.
UNIT/PROCESS NAME: ORLON FIBER DRYERS — #2 NEW DMF SCRUBBER-SEE ABOVE
RATED CAPACITY: CONFIDENTIAL
TYPE FUEL USED: — NONE —

APPROX. PROCESS RATE: CONFIDENTIAL (33% ABOVE NORMAL SHIFT MAX THRUPUT)
METHOD OF DETERMINING PROCESS RATE: PLANT DATA (WEIGH PRODUCT OUTPUT)
STACK HEIGHT: 93 FT
INDIVIDUAL STACK — COMMON STACK —
DIAMETER: (IF ROUND) 9 ft (IF RECTANGULAR) WIDTH — LENGTH —

I. SAMPLING POINT LOCATION



A. DISTANCE DOWNSTREAM FROM ANY FLOW DISTURBANCE:
NATURE OF DISTURBANCE ~ 3 ft
(BEND, CONTRACTION, EXPANSION, FAN, BAFFLES, ETC.)

B. DISTANCE UPSTREAM FROM ANY FLOW DISTRUBANCE:
NATURE OF DISTRUBANCE ~ 3 ft
(BEND, STACK EXIT, CONTRACTION, FAN, BAFFLES, EXPANSION, ETC.)

C. NUMBER OF PORTS IN STACK: 1

D. NUMBER OF POINTS SAMPLED PER PORT: 1 middle of stack (gas-vol-only)

II. STACK GAS

A. STACK TEMPERATURE: ~ 100 °F

B. ORSAT ANALYSIS: Only H_2O % per iced impingers
GRAB — CONTINUOUS — NUMBER OF INTEGRATED SAMPLES —

C. PRELIMINARY ΔP: MIN. NA MAX. NA

III. PARTICULATE TEST — method = OSHA DMF method = stack gas thru 2 iced D.I. water impingers + silica gel — then GC/FID on site analysis of each impinger & cond probe wash & scrubber water.

A. SAMPLING TRAIN:
1. MANUFACTURER Special test train MODEL —
2. TEST METHOD: EPA METHOD 5 — ASME PTC 21/27 —
OTHER (DESCRIBE)
3. TYPE FILTERS non-heated probe, ice/glass water impingers, silica gel, pump.
4. PROBE: LENGTH ~ 6 ft MATERIAL SS
5. PROBE HEATER SETTING —
6. HEATER BOX SETTING —
7. METER CALIBRATION FACTOR —
8. METER ΔH @ FACTOR —
9. DATE OF LAST CALIBRATION CHECK — flowmeter calibrated between test runs.
a. ORIFICE METER b. DRY GAS METER
c. TEMPERATURE DEVICES d. PITOT TUBE
e. NOZZLE DIAMETER f. OTHER

Test DMF specific VOC scrubber inlet w/ 6pm velocity traverse in breeching, scrubber outlet DMF conc, moisture inlet & outlet scrubber water DMF conc (large water recirculation) - inlet, 6pm & 8pm

DuPont #2 DMF scrubber on Orlon fiber dryers

B. NOMOGRAPH SETTINGS:
1. C FACTOR —
2. ASSUMED MOISTURE CONTENT —
3. INDICATED NOZZLE SIZE —

C. SAMPLING PROCEDURE
1. LEAK CHECK DONE: BEFORE ✓ AFTER ✓
2. PITOT TUBE: TYPE S ✓ for check reading TYPE P —
3. NOZZLE SIZE USED: —
4. TIME AT EACH PT.: 1 hr
5. TOTAL TIME OF TEST: 1 hr per run — 3 runs
6. TEST INTERRUPTED: NO ✓ YES(EXPLAIN) —

D. SAMPLE CLEAN-UP
1. FILTER HANDLED CAREFULLY: YES n.a. NO —
2. FILTER HOLDER WASHED OUT: YES n.a. NO —
3. PROBE WASHED OUT: YES ✓ NO —
4. CYCLONE WASHED OUT : YES n.a. NO —
5. WASHINGS SAVED: YES — NO —
MATERIAL USED: d.I. water

IV. ADDITIONAL COMMENTS:

10-6-89
Run #2 1126–1226 – witnessed – OSHA –
" #3 1347–1447 – witnessed – OSHA –
" #4 1717–1817 – not witnessed
C Run #1 was aborted due to test equipment power failure during test

All 3 runs =

① 1 of 2 scrubbers on line, all cfm on scrubber to fully load it, 3 of 6 dryers on at max throughput of about 33% more than normal plant throughput per 8 hr shift for dryers that are on line = 33% above normal dryer capacity = more than fully loaded scrubber tested.

② Tested: inlet DMF conc, water, cfm : outlet DMF conc, water ; DMF conc in big scrubber water reservoir.

③ Scrubber operating conditions:
~117 amps to 800 hp fan this scrubber. ~29 to 40 "pressure below" scrubber inlet.
~69,000 acfm, ~56,500 scfm 68°F, 29.92 "Hg. (wet)
~37,000 scfm control room gas flow meter (needs calibrating)
~9600 gpm recirculating water thru bottom = 8' hi packed bed.
~46°C
~400 pph (8 gpm) inlet make up water (~~filtered~~ softened fresh Cowner Spring water), 15C inputs to top of 12 bubble cap trays above packed bed.
~0 to 6,000 pph (0 to 12 gpm) — avg ~1500 pph (3 gpm) outlet blowoff take off water to DMF recovery. Outlet water flow varies widely due to level control of big reservoir in bottom of scrubber = avg ~3 gpm — takes days for much change in DMF conc in water.
~0.2" drop across fiber bed screen in breeching.
~-0.75" static press in breeching upstream of venturi.

→ DTE

Dnr
10-13-89

COMPLIANCE TEST REPORT

OCT 12 1989

FOR

#20517

E. I. DUPONT DMF SCRUBBER NO. 2

LOCATED IN

WAYNESBORO, VA

OCTOBER, 1989

ETS, INC.

A subsidiary of ETS International, Inc.

SUITE C-103

3140 CHAPARRAL DRIVE

ROANOKE, VA 24018

Pollution Control Consultants
Specializing In
Toxic Emission Measurement and Control

ETS CONTRACT NO. 89-210-T

OCT 13 1989

REPORT CERTIFICATION

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

DATE: October 10, 1989

SIGNATURE: E 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: October 10, 1989

SIGNATURE: James E Wright

James E. Wright
Manager/Technical Services
ETS, Inc.

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

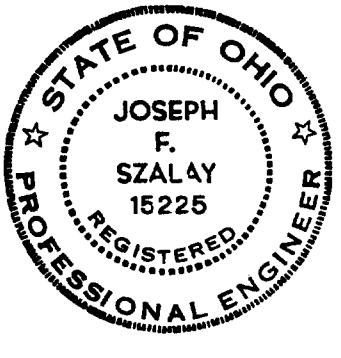
DATE: OCTOBER 10, 1989

SIGNATURE: Joseph F. Szalay

Joseph F. Szalay

P.E.: E-015225

STATE OF: Ohio



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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. 2) at this time. Scrubber No. 1 will be tested on a subsequent date. Testing was performed by L. Graves, T. Underwood and T. Handel of ETS, Inc. on Friday, October 6, 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 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. This information is included as Appendix E. 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. 2.
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. Due to a lack of demand for Orlon product, only one scrubber was conditioned for testing at this time. Scrubber No. 1 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. 2 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

Test run 1 was aborted during the test due to a power failure. The compliance data consists of three consecutive tests (runs 2, 3 and 4) performed on Friday October 6, 1989. All outlet data contained in this report is for Scrubber No. 2. The test log for the compliance testing is contained in Appendix A of this report. Duct flow measurements ranged between 61,000 and

68,000 acfm during the testing. The results of the DMF Compliance Tests for the duPont DMF Scrubber No. 2 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 286 and 397 ppm_{dv} during the compliance testing. The outlet concentrations ranged from 17.1 to 8.9 ppm_{dv}. The average scrubber efficiency measured for the three tests was 96.3%.

The concentration of DMF in the wastewater in the bottom of the stack remained relatively constant at 2.5% throughout the testing.

Table 1. Results of Efficiency Testing on Scrubber No. 2.

	Run 2	Run 3	Run 4
Inlet Temperature (°F):	156.8	153.4	153.3
Inlet Pressure (Static) (in.H_2O):	-0.999	-0.972	-0.980
Duct Velocity (ft/s):	21.5	20.2	19.0
Duct Flowrate (acfm):	68760	64790	60950
(scfm):	56080	53130	49820
(dscfm):	51090	44460	43160
Inlet Moisture (%):	8.9	16.3	13.4
Outlet Moisture (%):	10.4	10.4	8.9
Inlet DMF Concentration (ppm_{dv}):	397	286	312
Outlet DMF Concentration (ppm_{dv}):	8.9	17.1	12.6
Inlet Emission Rate (lbs/hr):	230.9	144.7	153.2
Outlet Emission Rate (lbs/hr):	3.0	8.6	6.2
Scrubber No. 2 Efficiency (%):	98.8	94.0	96.0
Scrubber No. 2 Wastewater Analysis:			
Concentration (%(w/v)) before run:	2.19	2.59	2.55
Concentration (%(w/v)) after run:	2.59	2.55	2.55

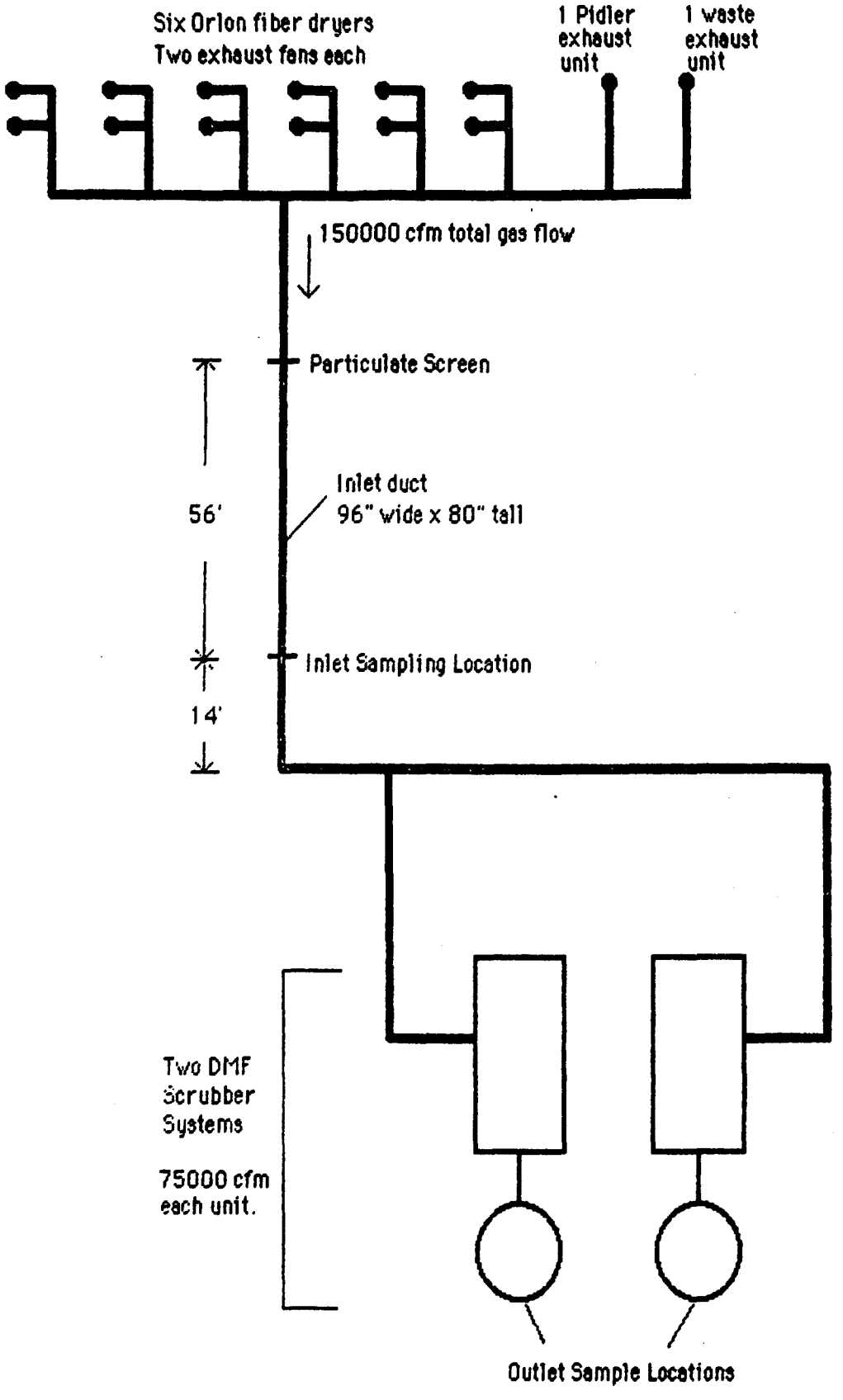
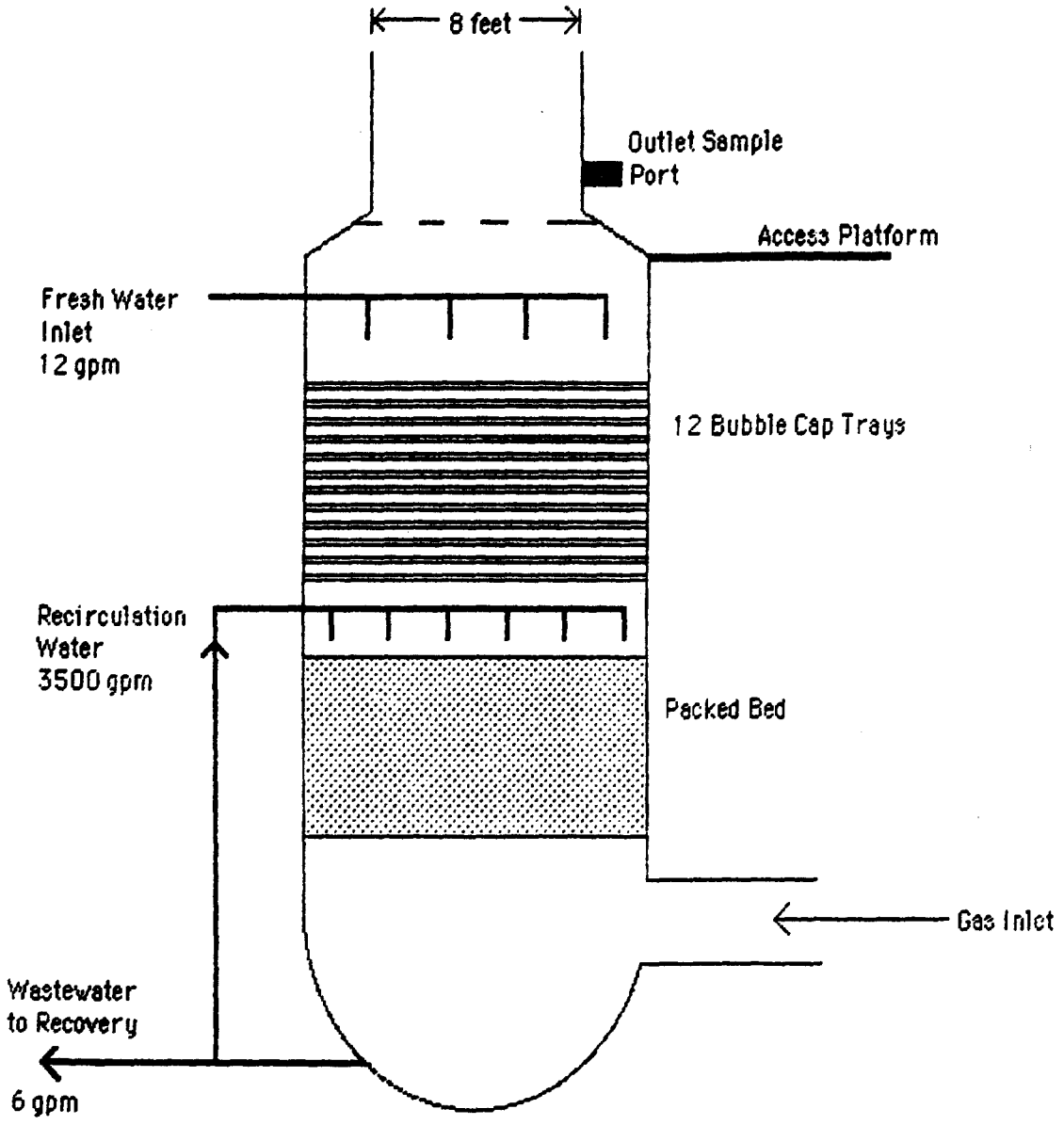


Figure 1 - Scrubber System



OCT 12 1989

Figure 2 - Scrubber Schemetic

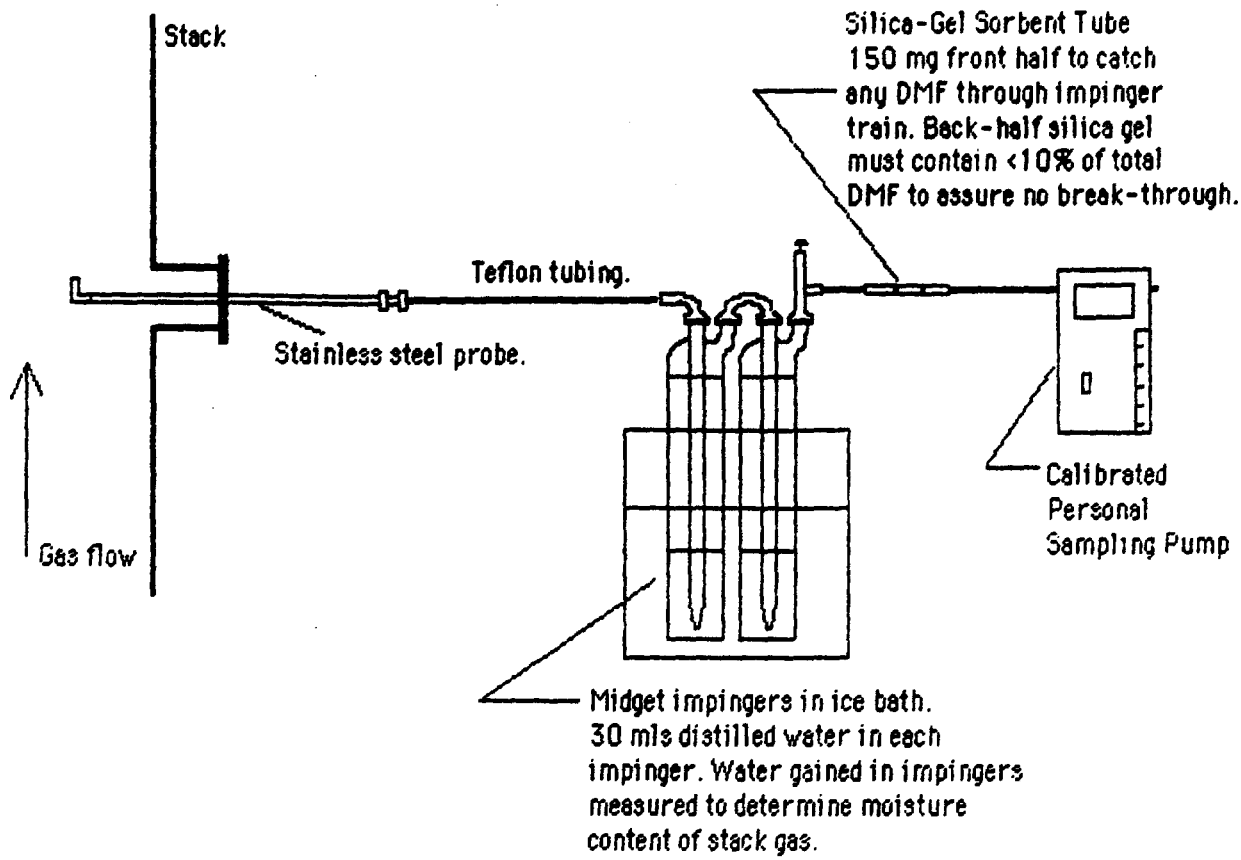


Figure 3 - Sampling Train

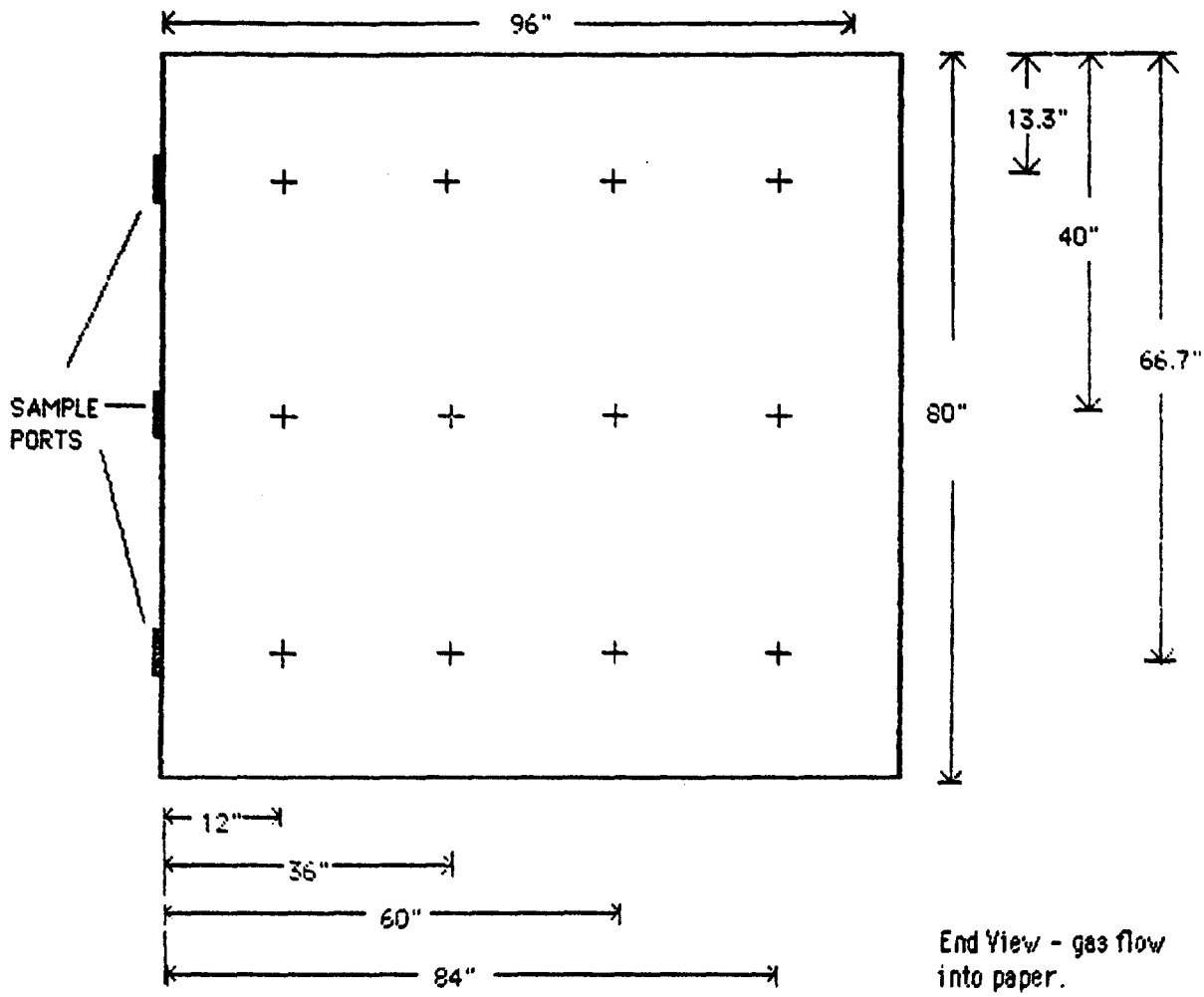


Figure 4 - Traverse Point Locations for Inlet Duct

APPENDIX A

Compliance Test Log

Run 1	-	Aborted due to power failure during test.	
Run 2	-	Start Time:	11:26
		End Time:	12:26
Run 3	-	Start Time:	13:37
		End Time:	14:47
Run 4	-	Start Time:	17:14
		End Time:	18:14

APPENDIX B

STACK VELOCITY DATA FIELD SHEETS

AND CALIBRATION DATA FOR

SAMPLING PUMPS

FIELD DATA SHEET

Project Dupont
Location Waynesboro, VA
Date October 6, 1989

RUN NUMBER 1 Inlet
START TIME 1013
END TIME

JOINED Power loss @ 10Hz

Traverse Data

PRE-TEST

Time	0755	
POINT	ΔP STACK In. W.C.	T STACK Deg. F
A 1	.048	82
2	.052	84
3	.052	85
4	.058	84
B 1	.050	82
2	.055	84
3	.053	84
4	.052	83
C 1	.049	81
2	.039	81
3	.040	81
4	.027	80
	.218	82.6

Static -.604

POST-TEST

Time	1000	
POINT	ΔP STACK In. W.C.	T STACK Deg. F
A 1	.126	155
2	.122	158
3	.133	161
4	.137	161
B 1	.114	159
2	.119	160
3	.127	162
4	.135	163
C 1	.102	152
2	.093	152
3	.100	152
4	.068	146
	.338	156.75

Static -.978

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - lpm
Pre-Test	30/30/30	.730	1.50
Post-Test	—		
		AVERAGE	

Notes

Initial Imp T° - 64°F

FIELD DATA SHEET

Project DUPONT 89-210
Location WAYNESBORO, VA
Date 10-6-89

RUN NUMBER #1 OUTLET
START TIME 1013
END TIME 1044

POWER OUT @ INLET 1044-1055

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

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - lpm
Pre-Test	19.6	500ml	1.56
Post-Test		500ml	
		AVERAGE	

Notes

LEAK CHECK = — 6000
EXIT TEMP = —

FIELD DATA SHEET

Project Dupont
Location Waynesboro, VA
Date October 6, 1989

RUN NUMBER D-Inlet
START TIME 1126
END TIME 1226

Traverse Data

PRE-TEST

Time	1112	
POINT	ΔP STACK In. W.C.	T STACK Deg. F
A 1	.128	158
2	.133	157
3	.128	160
4	.136	161
B 1	.118	157
2	.120	159
3	.129	157
4	.133	156
C 1	.114	149
2	.094	148
3	.113	150
4	.075	147
	.343	154.92

static .035

POST-TEST

Time	1301	
POINT	ΔP STACK In. W.C.	T STACK Deg. F
A 1	.123	160
2	.131	163
3	.119	165
4	.134	166
B 1	.112	158
2	.123	160
3	.121	162
4	.125	163
C 1	.117	155
2	~~.080~~ .100	153
3	.106	153
4	.067	147
	2nd .015	
	.338	158.75

static .967

1336 Retraverse

.103	154
.104	155
.115	156
.122	155
.105	151
.103	153
.119	154
.110	155
.086	145
.088	146
.094	143
.060	149

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - lpm
Pre-Test	30/30/30	.750 l	1.500
Post-Test	30/30/29	.750 l	1.500
		AVERAGE	1.50

Notes

FIELD DATA SHEET

Project DUPONT 89-210
Location WAYNESBORO, VA
Date 10-6-89

RUN NUMBER #2 OUTLET
START TIME 1126 COMPLIANCE
END TIME 1226

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

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - lpm
Pre-Test	19.9 sec	500 ml	1.5
Post-Test	20.0 sec	500 ml	1.5
		AVERAGE	1.5

Notes

LEAK CHECK - EXCELLENT
EXIT TEMP - 68°C

FIELD DATA SHEET

Project Dupont
Location
Date 10-6-89

RUN NUMBER 3 Inlet
START TIME 1348
END TIME 1448

Traverse Data

PRE-TEST

Time	1336	
POINT	ΔP STACK In. W.C.	T STACK Deg. F
A 1	.108	154
2	104	155
3	.115	156
4	.122	155
B 1	.105	151
2	.103	153
3	.119	154
4	.110	155
C 1	.086	145
2	.088	146
3	.094	148
4	.060	149
	0.318	151.75

.964

POST-TEST

Time	1505	
POINT	ΔP STACK In. W.C.	T STACK Deg. F
A 1	.103	157
2	.108	159
3	.117	160
4	.112	161
B 1	.102	152
2	.110	154
3	.113	156
4	.118	158
C 1	.083	147
2	.088	151
3	.100	153
4	.054	153
	0.316	155.08

static
-.979

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - lpm
Pre-Test	30/30/29	.750 l	1.50
Post-Test	29/29/28	.750 l	1.51
		AVERAGE	1.50

Notes

Initial Imp T° - 67°
Final T° 74°

FIELD DATA SHEET

Project DUPONT 89-210
Location WAYNESBORO, Va
Date 10-6-89

RUN NUMBER #3 OUTLET
START TIME 1348 COMPLIANCE
END TIME 1448

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

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - lpm
Pre-Test	20.2	500ml	1.44
Post-Test	20.5	500ml	1.44
		AVERAGE	1.44

Notes

LEAK CHECK = EXCELLENT
EXIT TEMP - 68-72°F

FIELD DATA SHEET

Project Dupont
Location
Date 10-6-89

RUN NUMBER 4 Inlet
START TIME 1714
END TIME 1814

Traverse Data

PRE-TEST

Time	1700	
POINT	ΔP STACK In. W.C.	T STACK Deg. F
A 1	.100	150
2	.103	150
3	.108	151
4	.113	152
B 1	.099	149
2	.103	151
3	.103	152
4	.108	154
C 1	.072	151
2	.083	149
3	.093	148
4	.055	143
		150.42

static .917

POST-TEST

Time	1825	
POINT	ΔP STACK In. W.C.	T STACK Deg. F
A 1	.101	161
2	.103	161
3	.111	162
4	.119	162
B 1	.098	160
2	.107	153
3	.111	.155
4	.102	156
C 1	.077	148
2	.084	148
3	.095	150
4	053	152
	0.310	156.08

static 1.042

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - lpm
Pre-Test	30/30/30	750 ml's	1.50
Post-Test	29/29/29	750 ml's	1.51
		AVERAGE	1.50

Notes

Initial Imp T - 64
Final Imp T - 67

FIELD DATA SHEET

Project DUPONT 89-210
Location WAYNESBORO Va.
Date 10-6-89

RUN NUMBER #4 OUTLET
START TIME 1714
END TIME 1814

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

CALIBRATION DATA

	NET TIME	NET VOLUME	RATE - lpm
Pre-Test	19.1 sec	500ml	1.56
Post-Test	20.2	500ml	1.44
		AVERAGE	1.50

Notes

LEAK CHECK = EXCELLENT
EXIT TEMP = 65-70°F

APPENDIX C

ANALYTICAL METHODS

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

(1) DIMETHYLACETAMIDE and (2) DIMETHYLFORMAMIDE

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):

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

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.

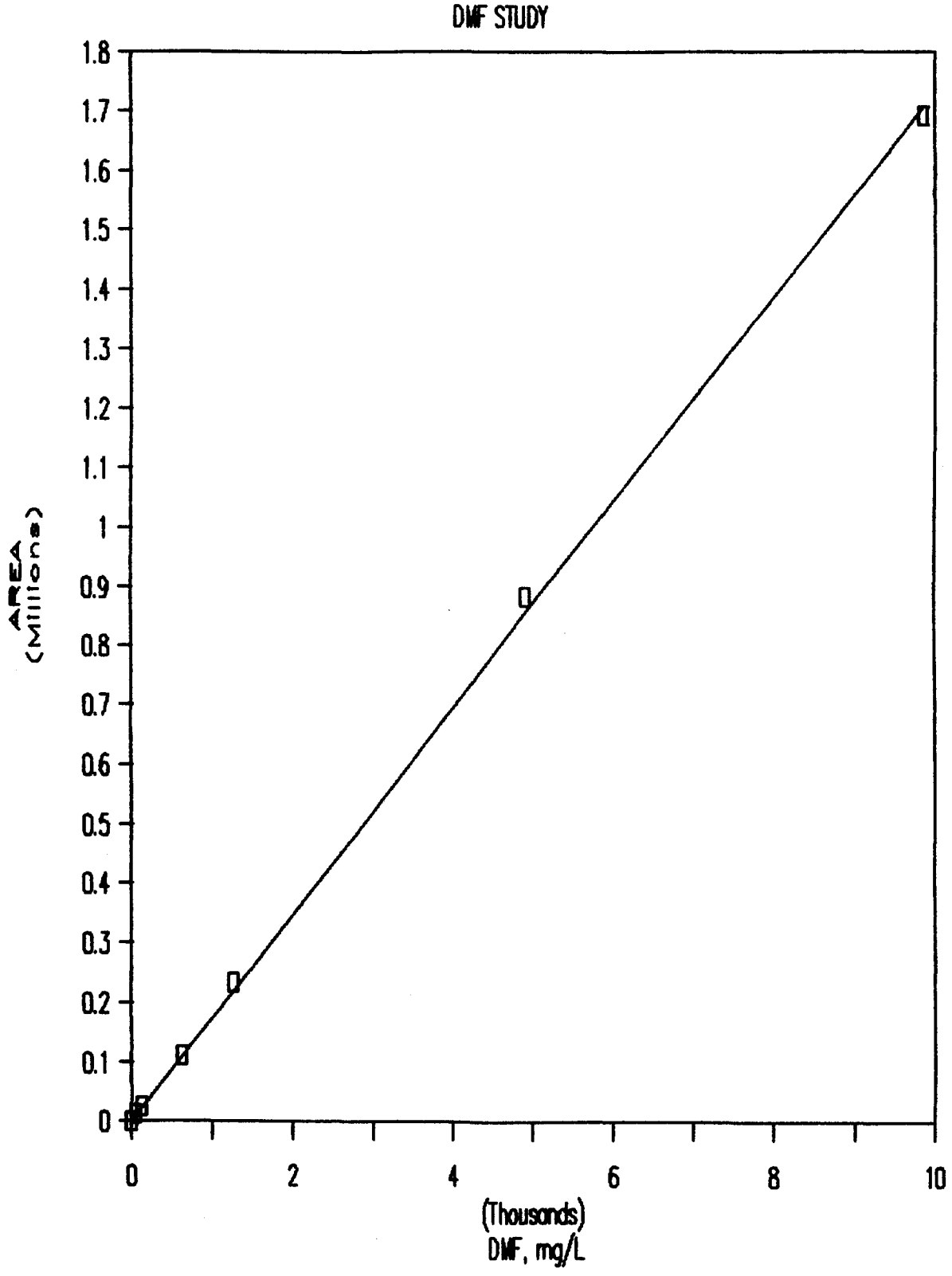
Table 1. Properties.

Analyte	Liquid density, g/mL @ 25 °C	BP (°C)	MP (°C)	VP @ 25 °C			Explosive range in air, % v/v
				Pa	mm Hg	ppm	
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



Durant Waynesboro
DMF Scrubber #2
Compliance Testing

(DMF)

Analysis for: N,N-Dimethylformamide Analyst: T.H. Date: 10/6/89

Method: NIOSH Reference: 2004

Sample Collection:

X Impinger: Volume: 15 ml Solution: Deionized Water

X Tube: Type: Silica Gel

Desorption: Volume: 1.0 ml Solvent: Methanol

GC Conditions:

Injector Temp: 225°C Injector volume: 0.5 ul

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

Column Type: Supelcox II, 10m, 0.53mm ID

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

Ramp: °C/min

Final: °C Time: min

Carrier: Gas: N_2 Flowrate: 3.0 ml/min

Sample ID	Collection Time (min)	Final Sample Volume (ml)	Retention Time	Area	
Blank (water)			ND	ND	
Blank (water			ND	ND	
9860 ppm DMF	Std		3.945	1692744	
4930 ppm DMF	Std		3.908	881939	
Scrubber WW/50			3.874	101600	
Tray 1/50			3.88	27629 ~~234~~	
1260 ppm DMF	Std		3.905	234646	

Dupont
Waynesboro
DMF Scrubber #2

Laboratory Analysis

Continuation page 2

Sample ID	Collection Time (min)	Final Sample Volume (ml)	Retention Time	Area	
630 ppm DMF	Std		3.88	109807	
126 ppm DMF	Std		3.863	24181	
63 ppm DMF	Std		3.862	11852	
Tray 4			3.857	100811	
Tray 8			3.886	40506	
Tray 12			3.872	9056	
Tray 1/50			3.926	21072	
Bottom 11:20/50		—	3.979	75811	2.19%
R2-Inlet Imp 1		21 ml	4.097	776678	94.1 mg
R2-Inlet Imp 2		15	4.046	40364	3.5 mg
R2-Inlet Probe	Wash	13	4.03	147893	11.1 mg
R2-Outlet-Imp 1		22	3.969	9104	1.2 mg
R2-Outlet-Imp 2		15	ND	ND	0 mg
R2-Outlet-Probe	Wash	24	4.042	1685	0.2 mg
Bottom after R2/50		—	4.007	89807	2.59%
R3-Outlet-Imp 1		21	4.054	20915	49.7 mg
R3-Outlet-Imp 2		20	4.044	4413	3.2 mg
R3-Outlet-Probe	Wash	24	4.066	12623	25.4 mg
R3-Inlet-Imp 1		20	4.078	410081	2.4 mg
~~R3-Inlet-Imp 2~~			4.001	42300	
R3-Inlet-Probe	Wash	25	4.002	183382	4.7 mg
Bottom Water 17:00/50		—	3.983	88337	2.54%
R3-Inlet Imp 2		17	3.977	27561	0.4 mg

DuPont
Waynesboro
DMF Scrubber #2

Laboratory Analysis

Continuation page 3

Sample ID	Collection Time (min)	Final Sample Volume (ml)	Retention Time	Area		
R3-Inlet Imp 2 Dup		—	3.971	29608	—	4%RR
R3-Inlet Imp 2 1:1 w/ 4930		Spike	4.04	380274	—	86% Re
R4-Outlet Imp 1		~~20~~ 21	3.991	18578	~~58.3mg~~	2.3mg
R4-Outlet Imp 2		~~19~~ 15	4.008	2221	~~9.9mg~~	0.2mg
R4-Outlet Probe Wash		~~10~~ 26	3.973	6696	~~17.2mg~~	1.0mg
R4-Inlet Imp 1		20	3.995	504737	58.3mg	
R4-Inlet Imp 2		19	3.948	89958	9.9mg	
R4-Inlet Probe Wash		10	3.971	298783	17.2mg	
Bottom Water 19:00/50		—	3.961	~~88~~290	2.55%	

T.OBJ	0	S.O.T.#	0
ATTEN	4	SPEED	4
METHOD#	2021	FOR.MT#	0
SPL.WT	100	LOCK	1

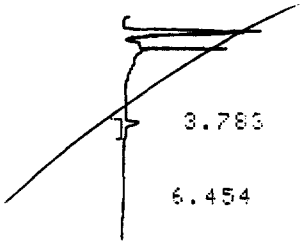
TIME PROGRAM FILE 0

0.01 LOCK ON
3.5 LOCK OFF

OCTOBER 6,1989
DUPONT DMF COMPLIANCE

10M; 0.53MM; SUPEROX II FSOT
ISOTHERMAL 100C
DET225; INJ225
RANGE 10

0.5UL BLANK(WATER)



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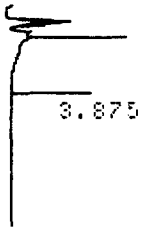
IR501 CHROMATOPAC
CHANNEL NO 1 FILE 6
SAMPLE NO 0 METHOD 2021
REPORT NO 60

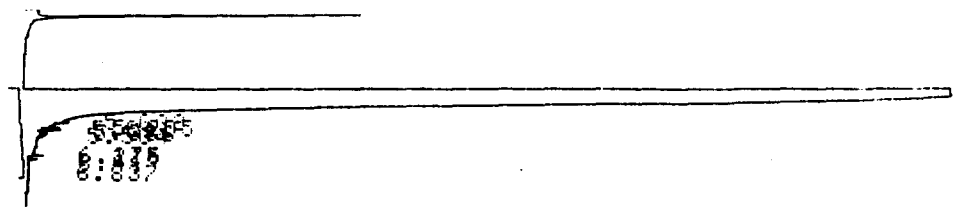
PKNO	TIME	AREA	MK	WIDTH	DOMN	NAME
1	3.783	2936			100	
		31341				
TOTAL		2936			100	

BLANK



CHROMATOGRAM 1 MEMORIZED
WARNING NO PEAK
BLANK

223-02037-01 904124A ⊕ Shimadzu

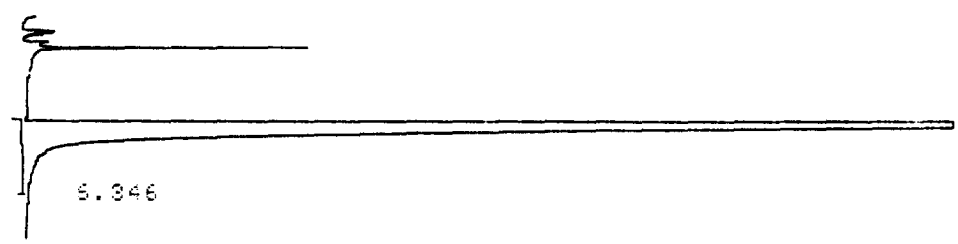


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD EDIT
REPORT NO 61

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.954	1692744	B		100	
		161712M1				
TOTAL		1692744			100	

4930PPM DMF

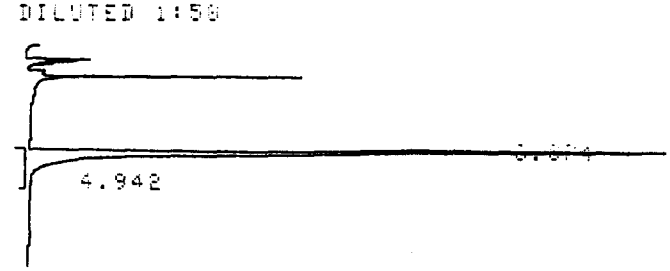


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD EDIT
REPORT NO 62

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.968	881999	B		100	
		88127M1				
TOTAL		881999			100	

BOTTOM DILUTED 1:50

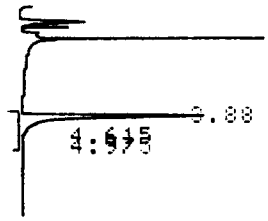


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD EDIT
REPORT NO 63

TOTAL 101600

TRAY1 DILUTED 1:50



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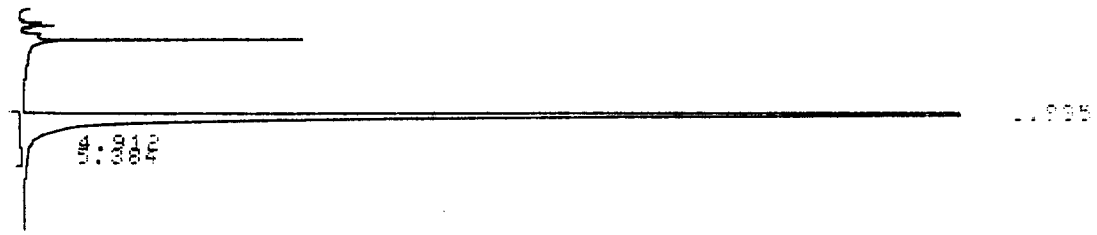
CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 64

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.88	27629	B		100	
		312mi				
TOTAL		27629			100	

1260PPM DMF



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 65

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.905	234646	B		100	
		21010mi				
TOTAL		234646			100	

630PPM

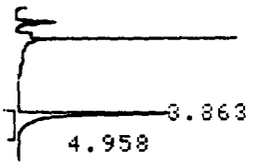
CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0

223-02037-01
904124A
Shimadzu

1	3.88	10980?	b	100	
		12432n1			
		---		---	
	TOTAL	109867		100	

126PPM DMF

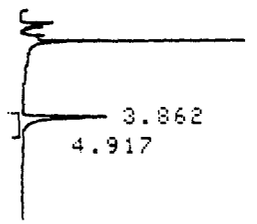


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 67

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.863	24181			100	
		2607n1				
		---			---	
	TOTAL	24181			100	

63PPM DMF

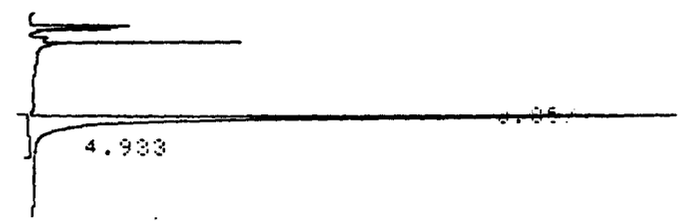


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 68

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.862	11852			100	
		1463n1				
		---			---	
	TOTAL	11852			100	

TRAY 4

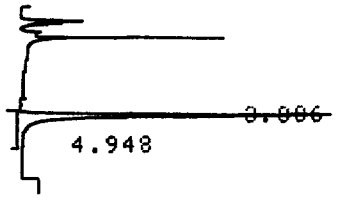


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC

	10933n1		
TOTAL	100811		100

TRAY 8

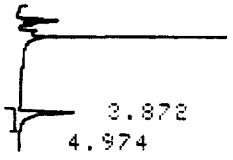


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2001
REPORT NO 70

PKNO	TIME	AREA	MK	TUNO	CONC	NAME
1	3.886	40506	S		100	
		5298n1				
TOTAL		40506			100	

TRAY 12

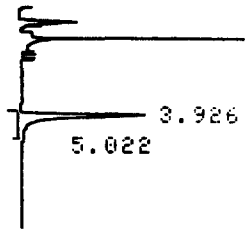


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2001
REPORT NO 71

PKNO	TIME	AREA	MK	TUNO	CONC	NAME
1	3.872	9056			100	
		1002n1				
TOTAL		9056			100	

TRAY 1 DILUTED 1:50

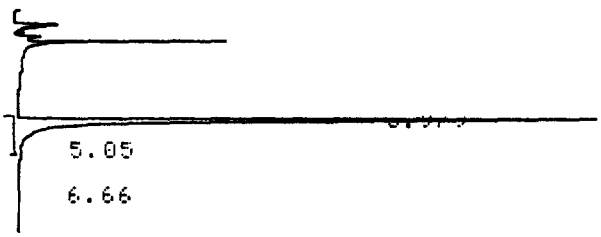


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0

1	3.926	21072 2119m1		100
	TOTAL	21072		100

BOTTOM 11:20
DILUTED 1:50

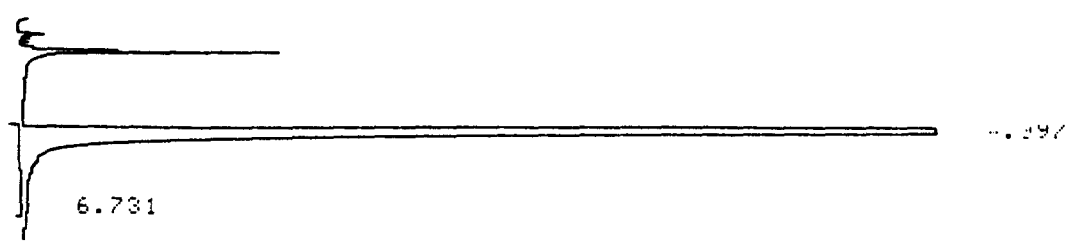


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 73

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.979	75811 9877m1	S		100	
	TOTAL	75811			100	

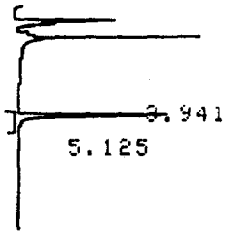
R2-INLET-IMP1



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 74

009
223-02037-01
904124A
Shimadzu



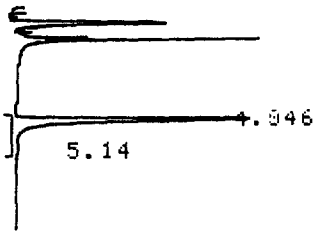
Bad Inj

CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD EDIT
REPORT NO 75

PKNO	TIME	AREA	MK	TYPE	CONC	NAME
1	3.941	14957			100	
		2539hi				
	TOTAL	14957			100	

R2-INLET-IMP2

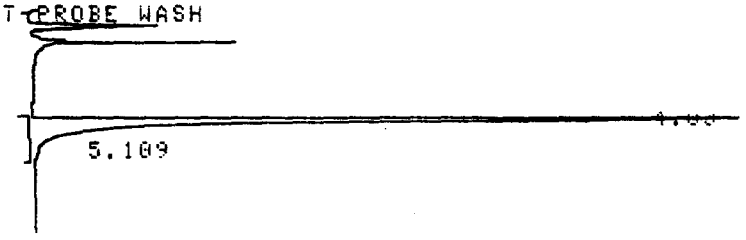


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD EDIT
REPORT NO 76

PKNO	TIME	AREA	MK	TYPE	CONC	NAME
1	4.046	40364	B		100	
		4048hi				
	TOTAL	40364			100	

R2-INLET-PROBE WASH



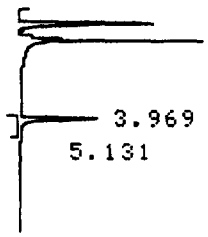
CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD EDIT
REPORT NO 77

010
223-02037-01
904124A
Shimadzu

TOTAL	147893	100

R2-OUTLET-IMP1

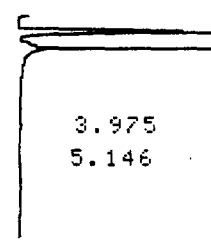


CHROMATOGRAM 1 MEMORIZED

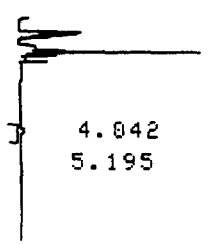
CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 78

PKNO	TIME	AREA	MK	AMNT	CONC	NAME
1	3.969	9104			100	
		1341n1				
TOTAL		9104			100	

R2-OUTLET-IMP2



CHROMATOGRAM 1 MEMORIZED
WARNING NO PEAK
R2-OUTLET-PROBE WASH

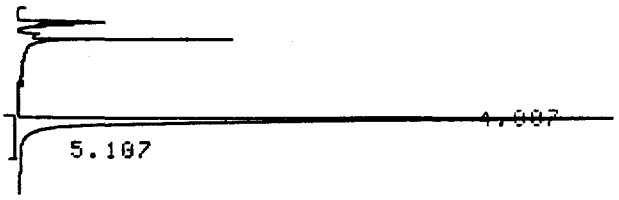


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 79

PKNO	TIME	AREA	MK	AMNT	CONC	NAME
1	4.042	1685			100	
		128n1				
TOTAL		1685			100	

BOTTOM AFTER R2 DILUTED 1:50

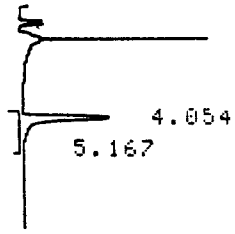


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 80

PKNO	TIME	AREA	MK	AMNO	CONC	NAME
1	4.007	89807	S		100	
		10003hi				
TOTAL		89807			100	

R3-OUTLET-IMP1

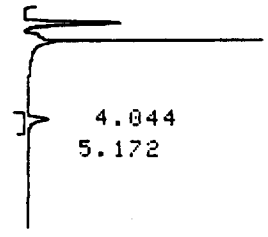


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 81

PKNO	TIME	AREA	MK	AMNO	CONC	NAME
1	4.054	20915	S		100	
		1491hi				
TOTAL		20915			100	

R3-OUTLET-IMP2

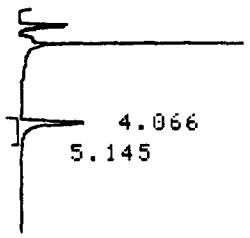


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE

1	4.044	4413		100	
		391hi			
		---		---	
	TOTAL	4413		100	

R3-OUTLET-PROBE WASH



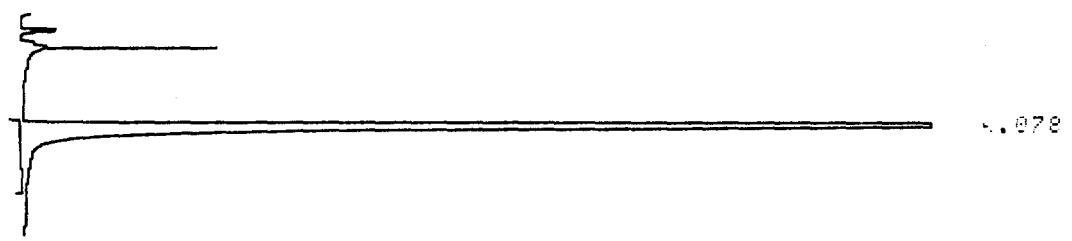
CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD EDC1
REPORT NO 83

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.066	12623			100	

		1122hi			
		---		---	
	TOTAL	12623		100	

R3-INLET-IMP1

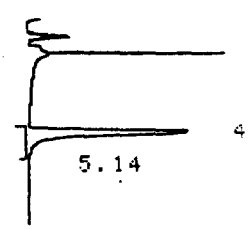


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD EDC1
REPORT NO 84

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.078	410081			100	
		33950hi				
		---		---		
	TOTAL	410081			100	

R3-INLET-IMP2



CHROMATOGRAM 1 MEMORIZED

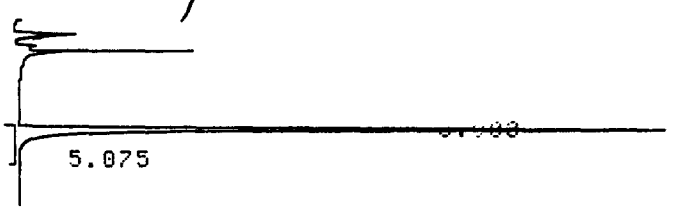
1	4.001	42300		100	
		2725n1			
TOTAL		42300		100	

CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 86

PKNO	TIME	AREA	MK	WIDTH	CONC	NAME
1	4.002	183382	b		100	
		18315n1				
TOTAL		183382			100	

BOTTOM WATER 17:10 150



CHROMATOGRAM 1 MEMORIZED

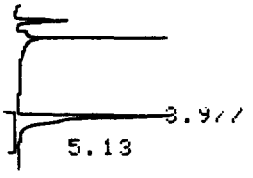
CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 87

PKNO	TIME	AREA	MK	WIDTH	CONC	NAME
1	3.983	88332	B		100	
		11014n1				

223-02037-01 904124A

Shimadzu

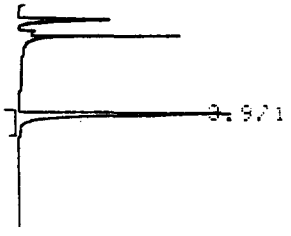
DUP R3-INLET-IMP2



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 88

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.977	27561	S		100	
		2602n1				
TOTAL		27561			100	

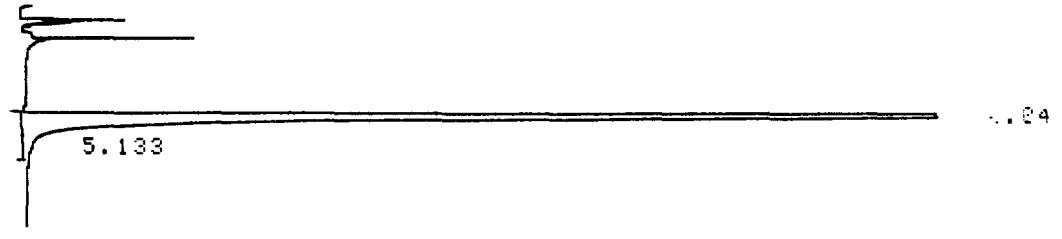


CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 89

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.971	29608			100	
		3620n1				
TOTAL		29608			100	

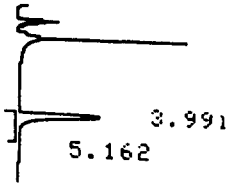
R3-INLET-IMP2 1:1 W/4930



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 90

R4-OUTLET-IMP1



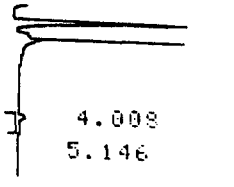
CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1
SAMPLE NO 0
REPORT NO 91

FILE 0
METHOD EDIT

PKNO	TIME	AREA	MK	AMNO	CONC	NAME
1	3.991	18578			100	
		1408n1				
	TOTAL	18578			100	

R4-OUTLET-IMP2



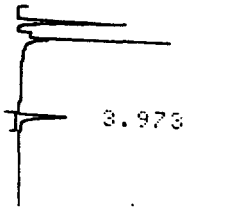
CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1
SAMPLE NO 0
REPORT NO 92

FILE 0
METHOD EDIT

PKNO	TIME	AREA	MK	AMNO	CONC	NAME
1	4.008	2221			100	
		183n1				
	TOTAL	2221			100	

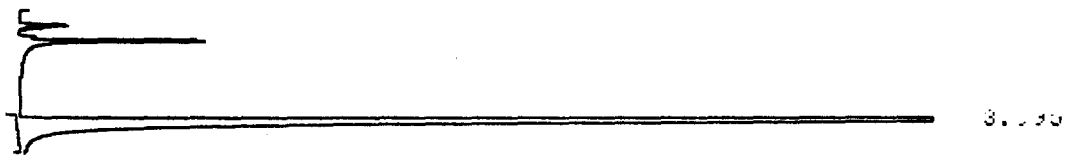
R4-OUTLET-PROBE WASH



CHROMATOGRAM 1 MEMORIZED

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.973	6696			100	
		856h1				
TOTAL		6696			100	

R4-INLET-IMP1



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 94

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.995	504737			100	
		6681h1				
TOTAL		504737			100	

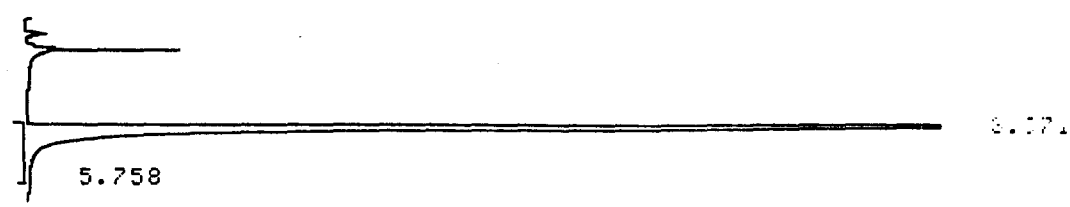
R4-INLET-IMP2

CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 95

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.948	89958			100	
		12106h1				
TOTAL		89958			100	

R4-INLET-PROBE WASH



CHROMATOGRAM 1 MEMORIZED

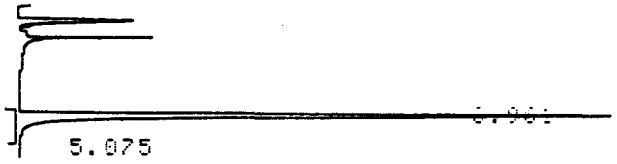
CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0

CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 96

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.971	298783 37573ni	S		100	
	TOTAL	298783			100	

BOTTOM 19:00



CHROMATOGRAM 1 MEMORIZED

CR501 CHROMATOPAC
CHANNEL NO 1 FILE 0
SAMPLE NO 0 METHOD 2021
REPORT NO 97

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.961	88290 9962ni			100	
	TOTAL	88290			100	

DuPont
Waynesboro
Scrubber #2 Compliance Test

Analysis for: DMF Analyst: LG Date: 10/9/89

Method: NIOSH Reference: 2004

Sample Collection:

___ Impinger: Volume: ___ ml Solution: ___

X Tube: Type: Silica Gel (back-up to impingers)

Desorption: Volume: 1.0 ml Solvent: methanol

GC Conditions:

Injector Temp: 225 °C Injector volume: 0.5 ul

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

Column Type: Superox II, 10 m, 0.53 mm id

Column Temp: Initial: 100 °C Time: isothermal ~~min~~

Ramp: ___ °C/min

Final: ___ °C Time: ___ min

Carrier: Gas: He Flowrate: ___ ml/min

Sample ID	Collection Time (min)	Final Sample Volume (ml)	Retention Time	Area	
Stnd. 630 ppm			4.397	131,175	
Stnd. 126 ppm			4.386	28,239	
Stnd. 63 ppm			4.448	15,777	
Dup: Stnd. 63 ppm			4.387	13,947	
Dup (2): Stnd 63 ppm			4.456	15,072	
Run 2, inlet, Front				N.D.	
Run 3, inlet, Front				N.D.	

Dupont DMF analysis
back-up tubes

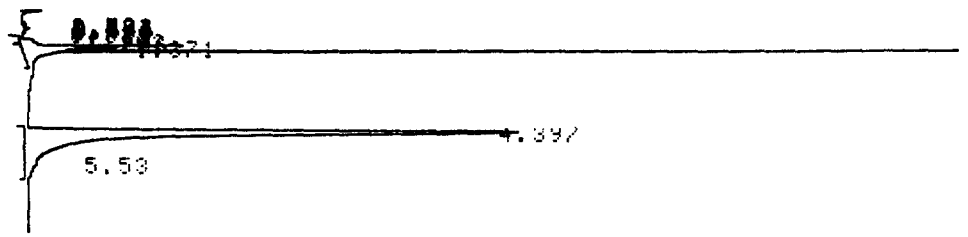
Laboratory Analysis DMF Continuation page 2

Sample ID	Collection Time (min)	Final Sample Volume (ml)	Retention Time	Area	
Run 4, inlet, front				N.D.	
SPIKE: 63 ppm stnd Run 2, inlet, front			4.329	4206	
SPIKE: 126 ppm stnd. Run 2, inlet, Front			4.340	10,181	
Run 2, outlet, Front				N.D.	
Run 3, outlet, Front				N.D.	
Run 4, outlet, Front				N.D.	

...PRINT OF PEAK
ANALYSIS PARAMETER FILE 0

WIDTH	5	SLOPE	100.00
DRIFT	0	MIN.AREA	1000
T.DBL	0	STOP.TM	0
ATTEN	4	SPEED	4
METHOD$	2021	FORMAT#	0
SPL.WT	100	IS.WT	1

10/9/89 DUPONT DMF ANALYSIS
SILICA GEL BACK-UP TUBES
DMF STND-630PPM

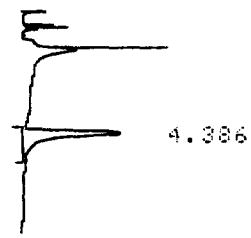


CHROMATOGRAM 1 MEMORIZED

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

PKNO	TIME	AREA	MK	WIDTH	HEIGHT	AREA
1	1.103	6121			1.01/0	
		253n1				
2	1.371	27793	V		17.69/1	
		1621n1				
3	4.397	131175	B		81.4/84	
		838/n1				
TOTAL		161088			100	

DMF STND-126PPM



CHROMATOGRAM 1 MEMORIZED

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

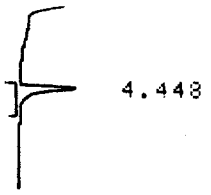
PKNO	TIME	AREA	MK	WIDTH	HEIGHT	AREA
1	4.386	63693			100	
		1603n1				

(12)

223 02037 01

904124A

Shimadzu

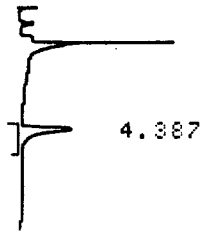


CHROMATOGRAM 1 MEMORIZED

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

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.448	15777			100	
		1027n1				
TOTAL		15777			100	

DUP: DMF STND-63PPM

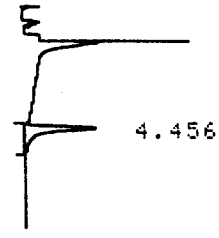


CHROMATOGRAM 1 MEMORIZED

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

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.387	13947			100	
		893n1				
TOTAL		13947			100	

DUP(#2)-DMF STND-63PPM



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

→DTE

COMMONWEALTH OF VIRGINIA
Department of Air Pollution Control

INTRA-AGENCY MEMORANDUM

TO: File

FROM: R. O. Goetz

SUBJECT: DUPONT, WAYNESBORO, REGISTRATION #20517

DATE: November 2, 1989

RE: STACK TEST [redacted]EW DMF SCRUBBERS ON ORLON FIBER DRYERS

A witnessed stack test was performed 10-31-89 on one of two (#1 scrubber, away from river) new large scrubbers for DMF (VOC) scrubbing efficiency, inlet and outlet concentrations and pounds per hour emission rates. The scrubber tested was fully loaded (one-half scrubbing capacity) by full production on one-half (3) of the six orlon fiber dryers.

Opacity appeared to be zero (0%). It was impossible to be sure this date due to fog the first two test runs and a cloudy background the third of three test runs.

See the attached stack test monitoring sheet and proposed test protocol by ETS for more details.

- "Preliminary" results indicate better than 95% DMF collection efficiency, less than 5 lbs/hr DMF emission per scrubber.
  - DMF concentration in scrubber water reservoir: 3%. Plant hopes to increase this to 9 or 10% for more efficient recovery.
  - Inlet feed water: 5300 lbs/hr -- 11 gal/min.
  - Ran about four days before test day.

STACK TEST MONITORING [Refs; 10/6/89 other scrubber]

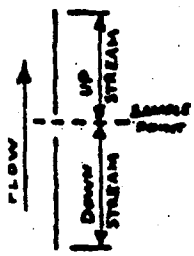
SOURCE NAME: DuPont – #20517 – Orlon Fiber Dryer Scrubber (DMF) VOC
LOCATION: Waynesboro
DATE: 10-31-89 OBSERVER'S NAME: R.D. Goff / P.H. Hunter

PURPOSE OF TEST: Compliance (DMF reduction)
TESTING DONE BY: ETS of Roanoke, Edward (Ted) Handel
LAB ANALYSIS DONE BY: same – on site

COMPANY CONTACT: Ron Shifflett TELEPHONE:
CONTROL EQUIPMENT OPERATING: #1 DMF Scrubber (away from river) = 8 ft packed bed + 12 bubble cap trays.
OPACITY READING MADE: YES see below NO —
Unofficial observation = 0% opacity, no the best background.
UNIT/PROCESS NAME: Orlon fiber dryer scrubber no 1 (of 2) for DMF collection.
RATED CAPACITY: confidential
TYPE FUEL USED: — none —

APPROX. PROCESS RATE: Confidential (at rated capacity of 3 = 1/3 of 6 dryers +
METHOD OF DETERMINING PROCESS RATE: product scales, noted 1/2 (1 of 3) scrubbers = fully loaded scrubber tested.
STACK HEIGHT: 93 ft ±
INDIVIDUAL STACK — COMMON STACK —
DIAMETER: (IF ROUND) 8 ft (IF RECTANGULAR) WIDTH — LENGTH —

I. SAMPLING POINT LOCATION



A. DISTANCE DOWNSTREAM FROM ANY FLOW DISTURBANCE:
NATURE OF DISTURBANCE ~ 4 ft
(BEND, CONTRACTION, EXPANSION, FAN, BAFFLES, ETC.)

B. DISTANCE UPSTREAM FROM ANY FLOW DISTRUBANCE:
NATURE OF DISTRUBANCE ~3 ft
(BEND, STACK EXIT, CONTRACTION, FAN, BAFFLES, EXPANSION, ETC.)

C. NUMBER OF PORTS IN STACK: 1

D. NUMBER OF POINTS SAMPLED PER PORT: 1 middle of stack (gas-VOC only)

II. STACK GAS

A. STACK TEMPERATURE: ~ 100°F

B. ORSAT ANALYSIS: Only H_2O per iced impingers
GRAB — CONTINUOUS — NUMBER OF INTEGRATED SAMPLES —

C. PRELIMINARY ΔP: MIN. N.A. MAX. N.A.

III. PARTICULATE TEST

DMF OSHA method = stack gas thru 2 iced impingers w/ D.I. water + silica gel — then GC/FID on-site analysis in mobile test lab – analyze each impinger & probe wash & scrubber water

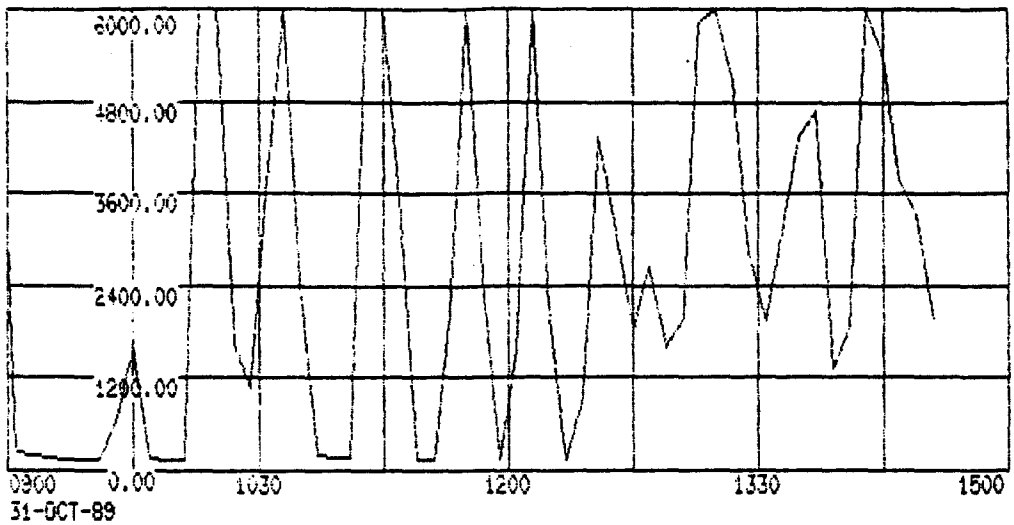
A. SAMPLING TRAIN:
1. MANUFACTURER Special test train – see above MODEL —
2. TEST METHOD: EPA METHOD 5 — ASME PTC 21/27 —
OTHER (DESCRIBE) see above
3. TYPE FILTERS Non-heated probe, iced glass D.I. water impingers, silica gel pump
4. PROBE: LENGTH ~ 6 ft MATERIAL SS (w/ teflon tube)
5. PROBE HEATER SETTING —
6. HEATER BOX SETTING —
7. METER CALIBRATION FACTOR —
8. METER Δ H @ FACTOR —
9. DATE OF LAST CALIBRATION CHECK Flow rate calibrated each test run.
a. ORIFICE METER b. DRY GAS METER
c. TEMPERATURE DEVICES d. PITOT TUBE
e. NOZZLE DIAMETER f. OTHER —

Test: DMF (VOC) is specific via GC/FID from cold water impingers water, probe wash, scrubber inlet & outlet ppm & ppb DMF & moisture; scrubber water DMF %.

[redacted] NEXT

31-OCT-89 14:38:20

SB#1 LIQUOR FLOW
6813 LB/HR



DMF Scrubber #1 liquid flow to solvent recovery during stack test 10/31/89.

COMMONWEALTH of VIRGINIA

Department of Air Pollution Control
VALLEY OF VIRGINIA REGIONAL OFFICE
SUITE D, EXECUTIVE OFFICE PARK
5338 PETERS CREEK ROAD
ROANOKE, VIRGINIA 24019
703 857-7328
FAX 703 857-7577
TDD 804 371-8471

October 16, 1989

Mr. Ron Shifflett
E. I. duPont de Nemours and
Company, Incorporated
Waynesboro, VA 22980

Dear Mr. Shifflett:

Our review of the [redacted] of DMF removal efficiency of the [redacted] and the test report you submitted has led us to conclude that [redacted] the capability of this [redacted] design to remove [redacted] of captured DMF. In conjunction with your earlier emissions inventorying and stack testing that indicated that approximately 77% of plant DMF emissions are exhausted by the stacks now controlled by these scrubbers, we also conclude that the 69% overall DMF reduction target specified in condition #15 of your July 3, 1989 permit has been met. You may therefore start-up the new Lycra spinning equipment described by that permit.

You are reminded that you [redacted] in order to comply with condition #5 and with the remainder of Condition #15 of th[redacted] and that conditions #6 and #16 must still be met by April 1, 1990.

Please feel free to call if you have any questions.

Sincerely,

Donald L. Shepherd

Donald L. Shepherd
Director, Region II

cc: [redacted]

→DTE

R-12003-WC

DU PONT
ESTABLISHED 1802

[redacted]
INCORPORATED

WAYNESBORO PLANT
WAYNESBORO, VIRGINIA 22980

TEXTILE FIBERS DEPARTMENT

OCT 12 1989

October 10, 1989

A20517

CONFIDENTIAL

Mr. Donald L. Shepherd
Director, Region II
Department of Air Pollution Control
Suite D, Executive Office Park
5338 Peters Creek Road
Roanoke, Virginia 24019

Dear Mr. Shepherd:

On Friday, [redacted] on our [redacted] which is located in the ORLON area of our plant. The compliance sampling was performed by ETS, Inc., of Roanoke, Virginia.

Prior to the start of this test, we were requested to provide information about certain measurements that were to be taken during the compliance testing. A copy of that requested information has been sent to your office. You also requested information about the ORLON production rates during the testing. This production information was not included in our recent submittal due to our opinion that it is [redacted] Below is the information that you requested.

Test No.	Start Time	Finish Time	Dryers Operating	Pounds of ORLON dried
2	11:26	12:26	#2, #3 & #5	15,928
3	13:47	14:47	#2, #3 & #5	14,845
4	17:17	18:17	#1, #3 & #5	14,981

I would request that you treat this information as confidential. If you have additional questions call me at (703) 949-2844.

Sincerely,

Ronald B. Shifflett

Ronald B. Shifflett
Safety, Health & Environmental Affairs

CONFIDENTIAL

→DTE

COMMONWEALTH OF VIRGINIA
Department of Air Pollution Control

INTRA-AGENCY MEMORANDUM

TO: File

FROM: R. O. Goetz

SUBJECT: DUPONT, WAYNESBORO, REG. NO. 20517

DATE: October 16, 1989

RE: STACK TEST NEW DMF SCRUBBERS (ORLON)

A stack test report has been received for the October 6, 1989 stack test of the new no. 2 (near river) DMF scrubber on orlon fiber drying. The test was witnessed by R. O. Goetz with S. K. Barnard observing opacity (always 0%).

The test report showed that this scrubber was "in compliance" with an efficiency exceeding 90%. It averaged 96.3% efficiency for three test runs.

The scrubber was fully loaded by all emissions from one-half (3) of the dryers at capacity venting through one-half the scrubbers (1 of 2).

This test is in reference to the July 3, 1989 permit for several areas of the plant and a related consent agreement.

There will be a similar test of the other scrubber in a few weeks.

For additional details, see the test report and 10-12-89 memo of test observations.

R. O. Goetz

Raymond O. Goetz
Engineer, Region II

ROG/bh

Attachments

cc: DTE
S. K. BARNARD