

CONFIDENTIAL

EMISSIONS TEST REPORT

NEW REACTOR D-700

CONOCO CHEMICALS COMPANY

P. O. BOX 91

ABERDEEN, MISSISSIPPI 39730

JUNE 21, 1982

CONFIDENTIALEMISSIONS TEST

NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

I. INTRODUCTION

- A. Source Information
- B. Testing Personnel
- C. Process Description

II. SAMPLING RESULTS SUMMARY

- A. Emissions Test Results
- B. PVC Production During Tests

III. SAMPLING PROCEDURES

- A. Sampling Apparatus
- B. Sampling Procedures

IV. ANALYTICAL PROCEDURES

- A. Analytical Apparatus
 - 1. Slurry Samples
 - 2. Reactor Opening Loss Samples
 - 3. Reagents
- B. Analytical Procedures
 - 1. Slurry Samples
 - 2. Reactor Opening Loss Samples

V. CHAIN OF CUSTODY

VI. APPENDIX

Operating Data Sheets
Sampling Data Sheets
Analytical Calculations
Analytical Data
Standard Gas Certifications
Reactor Opening Loss Calculations

A

CONFIDENTIAL

EMISSIONS TEST
NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

I. INTRODUCTION

In accordance with 40 CFR 61, Section 61.67 an emissions test was conducted on June 8 and 10, 1982 for the newly constructed PVC polymerization reactor D-700. The purpose of the test was to demonstrate compliance with 40 CFR 61, Sections 61.64 (a)(2) and 61.64 (e) (1)(ii).

A. Source Information

The emissions test was conducted at:

Conoco Chemicals
Highway 25
Aberdeen, Mississippi 39730

Questions concerning the testing or this report should be directed to:

R. A. Frohreich, Chief Process Engineer
Telephone Number (601) 369-8111, extension 2239

Dave Mahler, Environmental Engineer
Telephone Number (601) 369-8111, extension 2285

B. Testing Personnel

The testing and analytical work was conducted by Conoco Chemicals Company personnel at the Aberdeen facility. Personnel involved in sampling and sample analysis were:

Jimmy Grace - Quality Control Technician
Dave Mahler - Environmental Engineer
Wayne Myers - Engineering Co-Op Student

C. Process Description

Vinyl chloride monomer is received at the plant site in railroad tank cars and is transferred by compressors to the VCM storage sphere. The VCM is then pumped to fresh VCM batch tanks which are located in each of the two reactor areas.

EMISSIONS TEST
NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

CONFIDENTIAL

I. INTRODUCTION (CONTINUED)

C. Process Description (Continued)

PVC is produced batchwise from the vinyl chloride monomer. The plant produces only one type of resin; that by the suspension polymerization process. The reactor is first evacuated to remove air and inert gases and is then charged with carefully measured quantities of VCM, water and a suspension agent. The reaction is initiated with a peroxide-type catalyst. Following completion of polymerization, the reaction is killed and the unreacted VCM is removed from the reactor in the recovery sequence.

The recovery system is designed to recover unreacted VCM for recycle to subsequent batches. During the pressure recovery phase, VCM vapors are pulled from the reactor and compressed, condensed and liquified. The liquified VCM is routed to the recovered monomer tanks. As a part of the recovery operation, steam is injected into the slurry to aid in the removal of residual VCM in the slurry. When the reactor pressure approaches atmospheric pressure, vacuum pumps are started and the reactor pressure is reduced to a vacuum. These VCM vapors are also compressed and liquified and the liquified VCM is routed to the recovered monomer tanks. By this time the slurry has been brought to a boiling condition by action of the steam injection and the vacuum. The steam blows through the slurry and purges the vapor space above the slurry before being pulled out of the reactor by the recovery system. Cooling water is then put on the reactor in order to cool the slurry down prior to dumping the reactor.

When the recovery step is complete, the PVC/water slurry is dumped through a slurry screen which removes oversize material. The slurry is then pumped to blend tanks. After product pumpout, the reactor is rinsed to the blend tanks, which completes the reaction batch cycle.

The PVC/water slurry is pumped from the blend tanks to a centrifuge in the drying area, and the extracted water is discharged to the process sewer. Wet cake is fed to a dryer in which the resin is dried. The dried resin flows through a product sifter where oversize material is again separated and collected. Resin is then pneumatically conveyed to storage silos and eventually shipped from the plant in trucks or hopper cars.

EMISSIONS TEST
NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

CONFIDENTIAL

II. SAMPLING RESULTS SUMMARY

A. Emissions Test Results

The following is a summary of results from sampling and analyzing the reactor D-700 vapor space and dumped slurry for each of three batches.

<u>D-700</u> <u>Batch</u>	<u>Date</u>	<u>Resin</u> <u>Grade</u>	<u>Vinyl Chloride Emissions</u> <u>(Lbs. VCM/MM Lbs. PVC)</u>		
			<u>Vapor Space</u>	<u>Slurry</u>	<u>Total</u>
D-700-223	6/8/82	5385	32	82	114
D-700-224	6/8/82	5385	31	57	88
D-700-229	6/10/82	5305	96	107	203

The process employed at this plant utilizes the reactor for polymerization and stripping. Because VCM flashes out of the slurry and into the vapor space while the reactor is being dumped, it is impossible to accurately segregate the slurry residual and the reactor opening loss. The standard specifies maximum emissions of 400 lbs. VCM/MM lbs. PVC from the slurry and 20 lbs. VCM/MM lbs. PVC from the reactor vapor space. Thus, a maximum emission of 420 lbs. VCM/MM lbs. PVC from the entire reactor is applicable to reactor D-700.

The results of the emission tests summarized above demonstrate that reactor D-700 was in compliance with Sections 61.64 (a) (2) and 61.64 (e) (1) (ii) for each of the three reactor batches tested. The total emissions from reactor D-700 did not exceed 420 lbs. VCM/MM lbs. PVC produced.

B. PVC Production During Tests

The Aberdeen plant had a nominal PVC production capacity of 450 MM lbs. PVC/year at the time of the D-700 emission test. This corresponds to a daily average of 1,233 M lbs. PVC/day. During the two days of emission testing the plant produced 1,007 M lbs./day and 712 M lbs./day respectively, which is less than the nominal plant capacity. The maximum production rate can only be achieved in the winter time when colder cooling water temperatures are available which allow for higher catalyst loadings. Furthermore, the maximum production rate requires perfect reactor scheduling without any reactor or dryer downtime due to maintenance or other delays.

EMISSIONS TEST
NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

CONFIDENTIAL

II. SAMPLING RESULTS SUMMARY

B. PVC Production During Tests (Continued)

Since the plant utilizes a batch polymerization process, the plant's production rate is increased or decreased by processing more or less reactor batches per unit time. The size of the reactor batches varies only slightly over long periods of time for any specific grade of resin produced. During the emission testing, reactor D-700 was charged with quantities of VCM which are representative of the current batch size for reactor D-700.

EMISSIONS TEST
NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

CONFIDENTIAL

III. SAMPLING PROCEDURES

A. Sampling Apparatus

Reactor opening loss samples were taken with the apparatus shown on Figure 1. The components of the apparatus are described as follows:

1. New, unused teflon tubing; 6.4 mm OD.
2. Stainless steel plug valves with teflon plugs, stainless steel hose connectors and interconnecting tubing.
3. 15 liter Calibrated Instruments multilayer gas bags.
4. Rigid container, sealed airtight with RTV gel sealant.
5. Gast vacuum pump.
6. Calibrated MSA sampling pump rotameter.
7. Needle valve.
8. $\frac{1}{4}$ " Tygon tubing.

B. Sampling Procedures

The reactor slurry samples were obtained from a sample tap on the reactor dump line. The sample line was kept open throughout the dump period, which purged the line between samples. The three slurry samples were grabbed 5, 10 and 15 minutes into the dump period. 2-ounce samples bottles with screwed on covers were used to collect the samples. Immediately following sampling, the sample bottles were taped shut with electrical tape and labeled with the date and reactor batch number. The samples were then stored under refrigeration and analyzed within 24 hours of collection.

Reactor vapor space samples were taken immediately after the slurry dump and before the rinse. The sampling train was tightly assembled, using a sample bag which had been leak checked overnight. The sample bag was evacuated until the rotameter indicated zero flow, and the valve was shut. Reactor D-700's top manway was then opened, and the sampling line was lowered into the top of the reactor. The sampling line was purged at a rate of 1 to 2 liters per minute for approximately 1 minute. Immediately following this purge, the sample was taken. The top of the reactor, the middle of the reactor and the bottom of the reactor (within 6 inches of the bottom) were each sampled for 5 minutes, in that order. Flow through the

EMISSIONS TEST
NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

CONFIDENTIAL

III. SAMPLING PROCEDURES

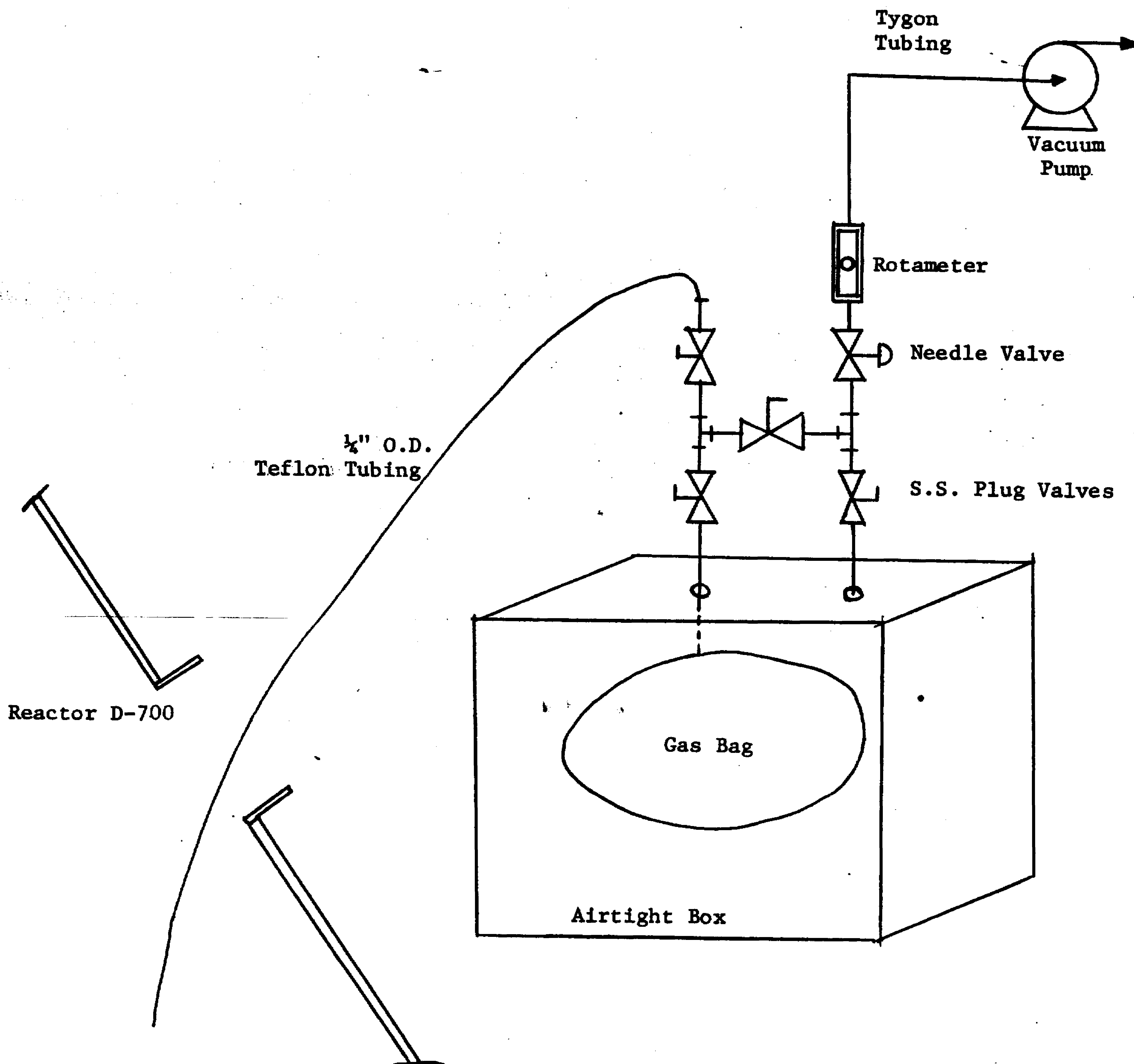
A. Sampling Apparatus (Continued)

sampling train was maintained at 0.5 liters per minute throughout the sampling period. At the end of sampling, the valve above the sample bag was shut, the pump turned off and the sampling apparatus disassembled. The sample bag was transferred to the plant's laboratory and analyzed within 24 hours of sample collection.

CONFIDENTIAL

FIGURE 1

REACTOR OPENING LOSS SAMPLING APPARATUS



EMISSIONS TEST
NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

CONFIDENTIAL

IV. ANALYTICAL PROCEDURES

A. Analytical Apparatus

1. Slurry Samples

The following apparatus was used to analyze slurry samples for residual VCM:

- a) Vials, unused with Septa and aluminum seals.
- b) Analytical balance, with minimum sensitivity of 0.001 gram.
- c) Syringe, 100 μ l, Hamilton #710.
- d) Vial Sealer, P-E No. 105-0106
- e) Filter Flask, 250 ml capacity.
- f) Buchner funnel, 7 cm.
- g) Filter paper, Whatman No. 4
- h) Pans, disposable aluminum
- i) Cork borer
- j) Oven, Blue-M natural draft
- k) Gas chromatograph, P-E F-42 Headspace Analyzer
- l) Chromatographic column, stainless steel 2 m X 3.2 mm, containing 0.4% Carbowax 1500 on Carbopak C.
- m) Thermometer, minimum range 0-100°C, minimum sensitivity 0.1 °C.
- n) Sample Tray Thermostat System, Haake Model N3B.
- o) Integrating Computer, Spectra Physics, SP 4000.
- p) Barometer, sensitivity 0.1 mm Hg.
- q) Thermometer, accurate to 1°C, near GC.
- r) Recorder, Houston Instruments potentiometric strip chart recorder set on 1 mm full-scale response and 1 cm per minute chart speed.

2. Reactor Opening Loss Samples

The following apparatus was used to analyze gas bags for VCM:

- a) Gas chromatograph, HP model 5711 A with a 1.0 ml heated sampling loop in a manual sample valve and flame ionization detector.
- b) Recorder, Perkin Elmer potentiometric strip chart recorder set on 1 mv full-scale response and 1 cm per minute chart speed.

EMISSIONS TEST
NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

CONFIDENTIAL

IV. ANALYTICAL PROCEDURES (CONTINUED)

A. Analytical Apparatus (Continued)

2. Reactor Opening Loss Samples (Continued)

- c) Chromatographic column, stainless steel, 2.0 m X 3.2 mm, containing 80/100 mesh chromosorb 102.
- d) Flow meter, rotameter type with 0-200 ml/minute capacity, with control valve.
- e) 6 gas regulators, dual stage brass.
- f) Thermometer, mercury type, accurate to 1°C, mounted near the sample loop.
- g) Barometer, accurate to 0.1 mm Hg.
- h) Pump, ITT diaphragm.
- i) Tubing, teflon, 6.4 mm O.D.
- j) Sample bags, Calibrated Instruments multilayer bag of aluminized Mylar.
- k) Data Processor, Spectra Physics SP4000.

3. Reagents

The following reagents were used to analyze slurry samples and gas bags for VCM:

- a) Nitrogen Gas, zero grade.
- b) Hydrogen Gas, zero grade.
- c) Compressed Air, zero grade.
- d) Calibration Gas, 496 ppm VCM in nitrogen, certified.
- e) Calibration Gas, 2188 ppm VCM in nitrogen, certified.
- f) Calibration Gas, 4015 ppm VCM in nitrogen, certified.

B. Analytical Procedures

1. Slurry Samples

The following procedure was used to analyze slurry residual VCM:

a) Sample Storage

Slurry samples must be stored under refrigeration. Analyses will be performed after sample has cooled to at least 30°C and within 24 hours of collection.

EMISSIONS TEST
NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

CONFIDENTIAL

IV. ANALYTICAL PROCEDURES (CONTINUED)

B. Analytical Procedures (Continued)

1. Slurry Samples (Continued)

b) Slurry Samples

Tare a vial with septum and seal and record weight. Tare and record the weight of an aluminum pan (label pan). Set up vacuum aspirator and filter flask assembly. Pour slurry into funnel and start vacuum until steady stream of water stops (15 sec.). Remove vacuum. Use the cork borer transfer about 1.5 g. to the vial and seal it. Take another plug for solids and transfer to an aluminum pan. Quickly weigh and record the pan and save this sample for determination of total solids. Weigh and record the vial weight.

c) Preparation of Gas Chromatograph

Flame should be lit at least one hour prior to analyses. Nitrogen pressure of 1.4 kg/cm² gives 32 cc/minute. Set hydrogen at 2.1 bar and air at 2.8 bar. The oven should be set at 65°C, the needle at 150°C, and the injector and detector at 150°C.

d) Programming the Chromatograph

Set "I" at 3 seconds, "A" at 07, "BF" at 09 and "S" at 2 minutes. Set "Analysis + Backflush Times" toggle switch at "X0.1" position. Both range and attenuation should be at a value of "1".

e) Preparation of Sample Turntable

The bath should have been on at least 4 hours prior to use. Check the temperature in the turntable to insure it is at 90 ± 0.5°C. Insert vials in turntable as follows: Positions 1 and 2 are old standards in the vials from a previous run; positions 3-6 are the calibration standards; positions 7-30 may be samples of any type. Condition all samples including standards at least one hour (not to exceed five hours) before analyzing.

After conditioning, start the chromatograph program and recorder.

EMISSIONS TEST
NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

CONFIDENTIAL

IV. ANALYTICAL PROCEDURES (CONTINUED)

B. Analytical Procedures (Continued)

1. Slurry Samples (Continued)

f) Determination of Total Solids

Place the label aluminum pans in the oven. The oven should be at $110 \pm 5^{\circ}\text{C}$. Allow the samples to dry for 45 minutes. Remove and cool to room temperatures. Record the weight and calculate the fraction of total solids (dry weight divided by wet weight). Record this value on the data sheet.

g) Calibration Standards

Calibration standards are prepared by filling the vials (at least two sets per series of samples) with the vinyl chloride/nitrogen standards, rapidly seating the septum and sealing with the aluminum seal. Use only stainless steel lines from the cylinder to the vial. The sample line from the cylinder must be purged into the hood prior to filling vials. After line purging, place the end of the tubing into the vial near the bottom and allow purging of the vial for one minute at 1000 cc/minute. (Hold the vial with finger tips only to minimize temperature affect of hand). Slowly remove tubing and quickly cap the vial with the septum and seal. Inject 100 ul of deionized water into vial.

2. Reactor Opening Loss Samples

The following procedure was used to analyze reactor opening loss samples for VCM:

a) Sample Storage

Samples must be kept out of direct sunlight during storage. Analysis will be performed after the bag has equilibrated to laboratory temperature and within 24 hours of sample collection.

b) Sample Recovery

Connect the sample bag to the GC sample valve inlet direct with Teflon ferrules. Prepare the equipment such that the sample gas will pass through the bag, through the sample loop, through the pump, rotameter, and flow control valve at a rate of 100 ml/minute.

VAB.0001115681

EMISSIONS TEST
NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

CONFIDENTIAL

IV. ANALYTICAL PROCEDURES (CONTINUED)

B. Analytical Procedures (Continued)

2. Reactor Opening Loss Samples (Continued)

c) Analysis

The GC should be previously prepared such that the column oven and sample loop are equilibrated at 130°C and 100°C respectively. The hydrogen flame should be ignited at least one hour prior to analysis using constant and previously optimized flow rates for both hydrogen and air. Carrier flow at 30 ml/minute should also be established at least one hour prior to analysis. (Leave instrument on overnight during routine use.) Observe the baseline to determine both noise level and drift at the 1×10^2 sensitivity are acceptable.

With the recorder on and an attenuator setting of 1×10^2 , purge the sample loop with the sample gas at least 30 seconds at 100 ml/minute. Stop the pump, and when the rotameter just indicates no flow, activate the sample valve to inject the sample gas. Mark the injection point on the chart and read the loop temperature and laboratory pressure and temperature. Record all conditions on the chart. Start the SP4000.

With the laboratory pressure and temperature, determine the water content of the sample at analysis from a water saturation vapor pressure table (assume 100% relative humidity in the sample).

d) Instrument Calibration

Fill evacuated Calibrated Instruments bags with the standard gases, always using the sample bag for the same concentration, daily. Use only stainless steel or the appropriate Teflon tubing connectors. Allow bags to equilibrate to laboratory temperature.

Measure each standard gas in duplicate each day using the same GC conditions and procedures described in the above analysis section. Record all conditions as described earlier for each analysis.

EMISSIONS TEST
NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

CONFIDENTIAL

V. CHAIN OF CUSTODY

Slurry samples and gas bags were handled as follows:

Slurry Samples

<u>Sample Run No.</u>	<u>Taken By</u>	<u>Transported By</u>	<u>Analyzed By</u>
1	Wayna Myers	Wayne Myers	Jimmy Grace
2	Wayne Myers	Wayne Myers	Jimmy Grace
3	Wayne Myers	Wayne Myers	Jimmy Grace

Reactor Opening Loss Gas Bags

<u>Sample Run No.</u>	<u>Taken By</u>	<u>Transported By</u>	<u>Analyzed By</u>
1	Wayne Myers	Wayne Myers	Jimmy Grace
2	Wayne Myers	Wayne Myers	Jimmy Grace
3	Waybe Myers	Wayne Myers	Jimmy Grace

EMISSIONS TEST
NEW REACTOR D-700
CONOCO CHEMICALS COMPANY
ABERDEEN, MISSISSIPPI

CONFIDENTIAL

VI. APPENDIX

The Appendix of this report contains the raw data and calculations used to obtain the sampling results. This information is presented in the following order:

Operating data sheets for reactor D-700 during the three batch sampling period.

Sampling data sheets.

Reactor opening loss and slurry residual analytical calculations

Integrator printout data.

Standard gas certifications.

Reactor opening loss calculations.

LARGE REACTOR BATCH SHEET

CONFIDENTIAL

REACTOR D200-0223

TYPE 5385

DATE 6-7-82

FORMULA

DURA SEAL

Water Tk. 75 % 1102 Counts

Oil Level Full Inches

Colloid 230 Gal. 42 Inches

Oil Pressure 240 Psig

VC Tank 75 % 998 Counts FVCM 20% RVCN

Seal Water 3 GPM

Catalyst 12 Gal. 223 Type

Seal Water Press. 180 Psig

Pre-charge Amps 40 A

PVA IN.

Running Temp. 138 °F Pressure 120 psig

7 Before PVA 100
49 After Kill Pot
42 Net OK

TIME	INITIAL
0240	OC
0230	OC
0240	OC
0240	OC
0250	OC
0250	OC
0255	OC
2400	Rec.
0815	87
1015	87
1015	87
1030	87
1125	87
1155	97
1155	87
1215	87

Reactor & Material O.K. For Charge

Evacuation Start

Standard 10 Minutes

Evacuation Stop 28.5 Inches Hg.

Actual Dev

Charge Start

Standard 11 Minutes

Charge Complete

Actual Dev

Catalyst Start

Time Reaction
Temperature Reached

Standard 3 Minutes

Catalyst Complete

Actual Dev

Turn Around Time

2 Hrs. 55 Mi.

Recovery Start Run Time

4 Hrs. 25 Mi.

Standard 30 Minutes

Recovery Complete Amt. Recovered To 18 "

Actual Dev

Dump Start To Tank 747 Bldg. No. 49403

Standard 25 Minutes

Dump Stop

Actual Dev

Chem Wash Start

Total
Stripping
Steam

Standard 25 Minutes

Chem Wash Finish

Actual Dev

Rinse Start

215

Standard 10 Minutes

Rinse Complete

Time Reaction
Killed

Actual Dev

Swirl Start

0720

Total Std. Dev

Swirl Complete

Comments: _____

VAB.0001115685

DEF/lnh

10



LARGE REACTOR BATCH SHEET CONFIDENTIAL[^]

REACTOR D-700-0224 TYPE 5385 DATE 6-8-82

FORMULA

DURA SEAL

Water Tk. 25 % 1102 Counts
 Colloid 330 Gal. 42 Inches
 VC Tank 25 % 998 Counts FVCM 209 Counts RVCM
 Catalyst 12 Gal. 223 Type
 Pre-charge Amps 40 A
 Running Temp. 138 °F Pressure 120 psig

Oil Level Full Inches
 Oil Pressure 240 Psig
 Seal Water 3 GPM
 Seal Water Press. 240 Psig
 PVA IN.

7 Before Pot 350
49 After Level 34
42 Net Pum 2500
Pum 1500

TIME	INITIAL
1230	87
1220	87
1230	87
1230	87
1240	87
1240	87
1245	87
0720	Rec
1645	87
1840	87
1840	87
1910	87
1940	87
2000	87
2000	87
2000	87
2000	87

Reactor & Material O.K. For Charge

Evacuation Start

Evacuation Stop 28⁵ Inches Hg.

Charge Start

Charge Complete

Catalyst Start

Catalyst Complete

Turn Around Time 5 Hrs. 25 Mi.

Recovery Start Run Time 4 Hrs. 0 Mi.

Recovery Complete Amt. Recovered To 10"

Dump Start To Tank 247 Bldg. No. 49408

Dump Stop

Chem Wash Start

Chem Wash Finish

Rinse Start

Rinse Complete

Swirl Start

Swirl Complete

Time Reaction
Temperature Reached

Total
Stripping
Steam

2150

Time Reaction
Killed

1645

Standard 10 Minutes

Actual _____ Dev _____

Standard 11 Minutes

Actual _____ Dev _____

Standard 3 Minutes

Actual _____ Dev _____

Standard 30 Minutes

Actual _____ Dev _____

Standard 25 Minutes

Actual _____ Dev _____

Standard 25 Minutes

Actual _____ Dev _____

Standard 10 Minutes

Actual _____ Dev _____

Total Std. _____ Dev _____

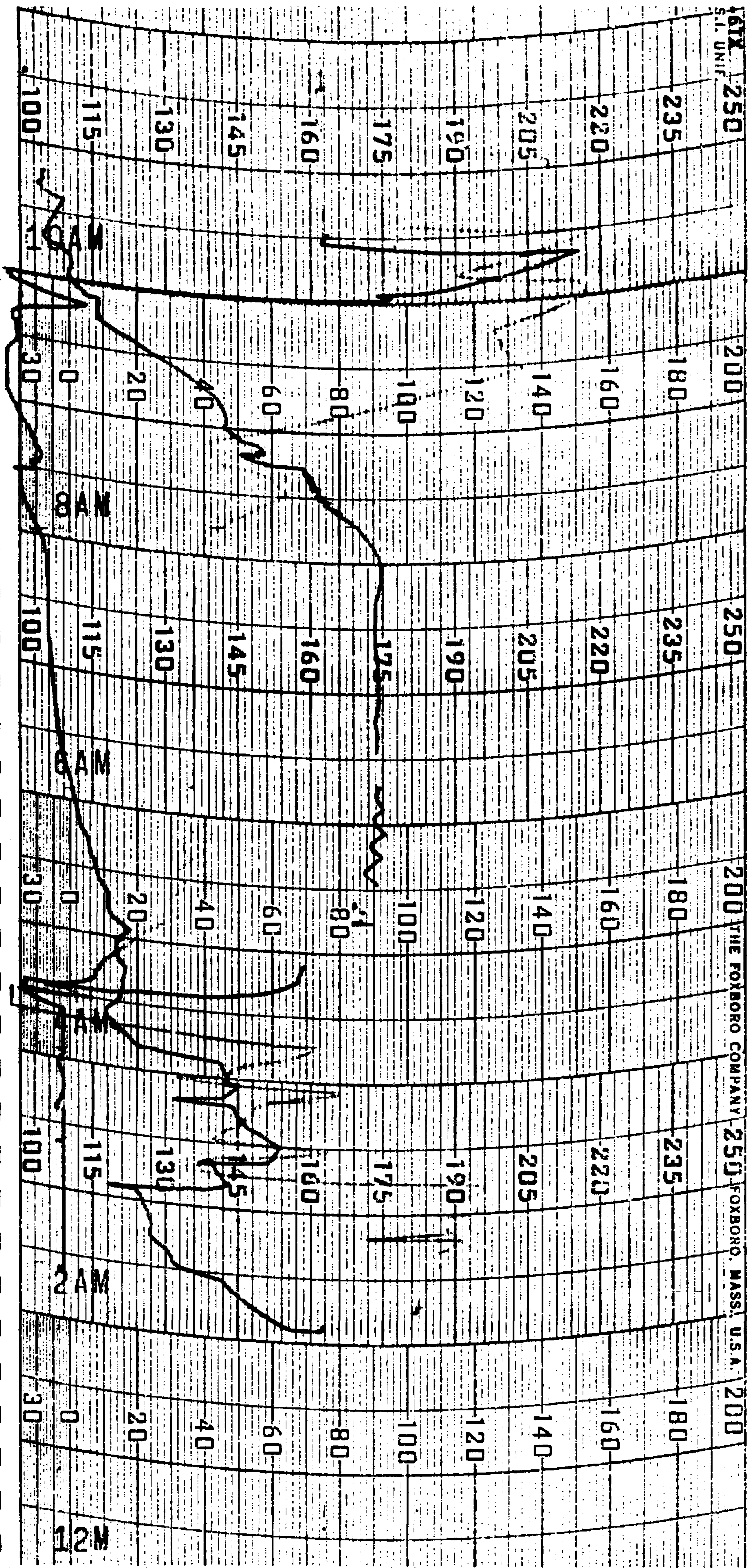
Comments: _____

VAB.0001115687

DEF/ich

CONFIDENTIAL

D-700-224



LARGE REACTOR BATCH SHEET

CONFIDENTIAL

REACTOR 0700 0229

TYPE S305

DATE 6 9 82

FORMULA

DURA SEAL

Water Tk. 75% 1178 Counts

Oil Level Full Inches

Colloid 345 Gal. 43 1/2 Inches

Oil Pressure 240 Psig

VC Tank 75% 1022 Counts FVCM 0 Counts RVCM

Seal Water 3 GPM

Catalyst 11 Gal. LS4 Type

Seal Water Press. 180 Psig

Pre-charge Amps 40 A

PVA IN.

Running Temp. 143 °F Pressure _____ psig
35 gal. CTA

7 Before

502 After

43 1/2 Net

1200
1000
350
3/4

TIME INITIAL

0615	RM
0555	RM
0615	RM
0615	RM
0630	RM
0630	RM
0635	RM
0315	ROR
1145	BK
1350	BK
1350	BK
1430	BK
1505	BK
1535	
1535	BK
1605	BK

Reactor & Material O.K. For Charge

Evacuation Start

Standard 10 Minutes

Evacuation Stop 25.5 Inches Hg.

Actual _____ Dev _____

Charge Start

Standard 11 Minutes

Charge Complete

Actual _____ Dev _____

Catalyst Start

Time Reaction
Temperature Reached

Standard 3 Minutes

Catalyst Complete

Actual _____ Dev _____

Turn Around Time 4 Hrs. 20 Mi.

Recovery Start Run Time 5 Hrs. 10 Mi.

Standard 30 Minutes

Recovery Complete Amt. Recovered To 10"

Actual _____ Dev _____

Dump Start To Tank 1646-7 Bldg. No. 49431

Standard 25 Minutes

Dump Stop

Actual _____ Dev _____

Chem Wash Start

Total
Stripping
Steam

Standard 25 Minutes

Chem Wash Finish

215

Actual _____ Dev _____

Rinse Start

Standard 10 Minutes

Rinse Complete

Time Reaction
Killed

Actual _____ Dev _____

Swirl Start

Total Std. _____ Dev _____

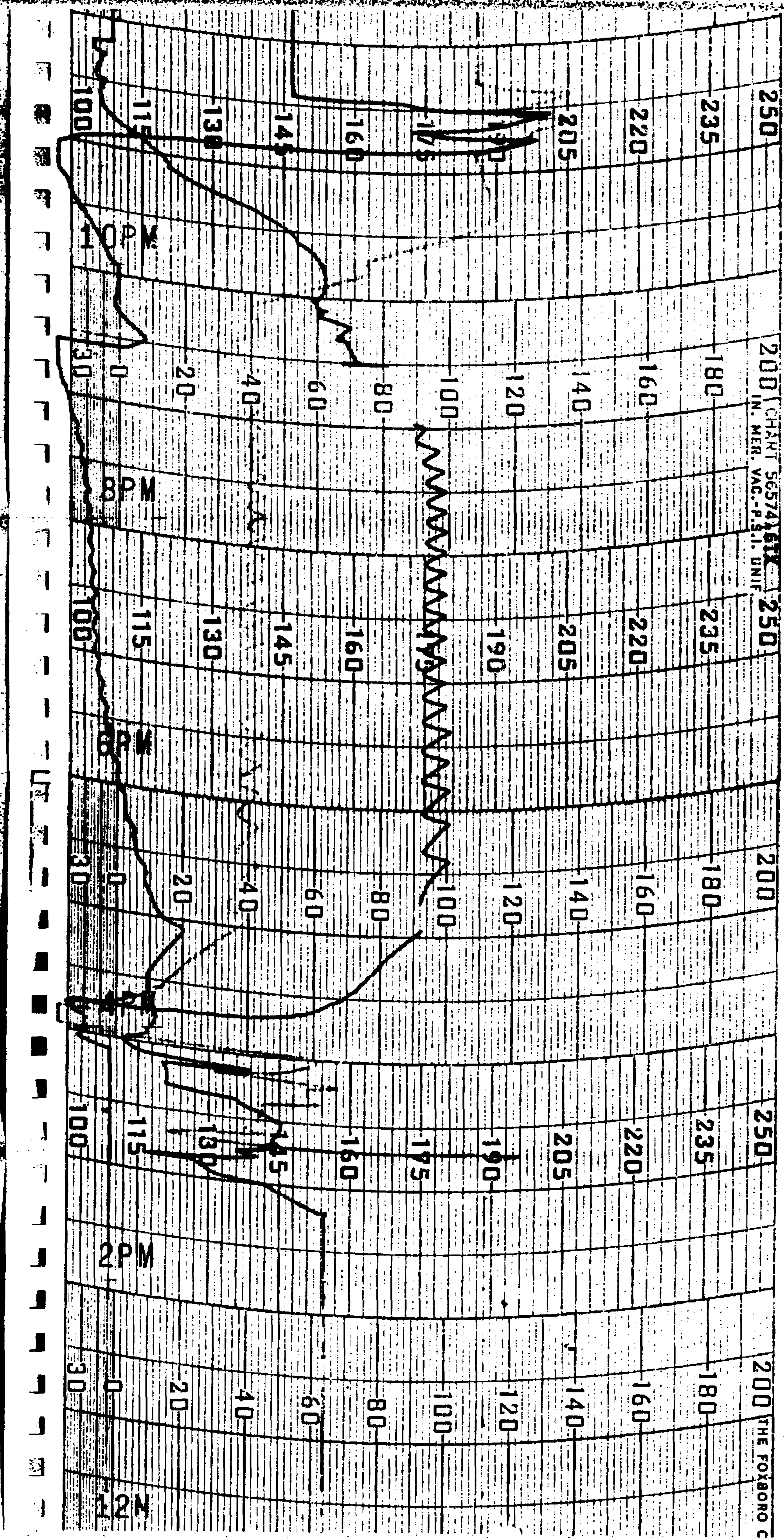
Swirl Complete

Comments:

VAB.0001115689

CONFIDENTIAL

D-700-229



CONFIDENTIAL

REACTOR D-700 COMPLIANCE TEST

DATE: 6/8/82

BLEND NO.: 49403

RESIN: 5385

SLURRY SAMPLES

DUMP STARTED: 10:10 AM

<u>Sample</u>	<u>Time</u>	<u>Taken By</u>	<u>Transported By</u>	<u>Analyzed By</u>
1	10:15 AM	Wayne Myers	Wayne Myers	Timothy Grace
2	10:20 AM	Wayne Myers	Wayne Myers	Timothy Grace
3	10:25 AM	Wayne Myers	Wayne Myers	Timothy Grace

REACTOR OPENING LOSS

<u>Time Started</u>	<u>Taken By</u>	<u>Transported By</u>	<u>Analyzed By</u>
10:45 AM	Wayne Myers	Wayne Myers	Timothy Grace

CONFIDENTIAL

REACTOR D-700 COMPLIANCE TEST

DATE: 6/8/82

BLEND NO.: 49408

RESIN: 5385

SLURRY SAMPLES

DUMP STARTED: 6:37 PM

<u>Sample</u>	<u>Time</u>	<u>Taken By</u>	<u>Transported By</u>	<u>Analyzed By</u>
1	6:42 AM	Wayne Myers	Wayne Myers	Timothy Grace
2	6:47 AM	Wayne Myers	Wayne Myers	Timothy Grace
3	6:52 AM	Wayne Myers	Wayne Myers	Timothy Grace

REACTOR OPENING LOSS

<u>Time Started</u>	<u>Taken By</u>	<u>Transported By</u>	<u>Analyzed By</u>
7:23 AM	Wayne Myers	Wayne Myers	Timothy Grace

CONFIDENTIAL

REACTOR D-700 COMPLIANCE TEST

DATE: 6/10/82

BLEND NO.: 49431

RESIN: 5305

SLURRY SAMPLES

DUMP STARTED: 1:55 PM

<u>Sample</u>	<u>Time</u>	<u>Taken By</u>	<u>Transported By</u>	<u>Analyzed By</u>
1	2:00 PM	Wayne Myers	Wayne Myers	Timmy Grace
2	2:05 PM	Wayne Myers	Wayne Myers	Timmy Grace
3	2:10 PM	Wayne Myers	Wayne Myers	Timmy Grace

REACTOR OPENING LOSS

<u>Time Started</u>	<u>Taken By</u>	<u>Transported By</u>	<u>Analyzed By</u>
2:45 PM	Wayne Myers	Wayne Myers	Timmy Grace

CONFIDENTIAL

PPM, VCM IN AIR BAG

SAMPLE ID: ROL Sample #1
DATE: 6-8-82

Sample Loop Temperature for Standard (Tr) = 100
Sample Loop Temperature for Sample (Ts) = 100
Barometric Pressure for Standard (Pr) = 761.0
Barometric Pressure for Sample (Ps) = 761.0
Water Vapor Content of Bag (B) = .025
Response Factor (Rf) = $\frac{\text{Area Standard}}{\text{PPM, VCM in Standard}}$ = 16.46
Area of Sample (As) = 147812
 $C_{vcm} = \frac{As}{Rf} \times \frac{Pr}{Ps} \times \frac{Ts}{Tr} \times \frac{1}{1-B}$ = 9210

Bag Temperature	(B)
20°C	.022
<u>21</u>	<u>.025</u>
22	.026
23	.028
24	.029
25	.031
26	.033
27	.035
28	.037

CONFIDENTIAL

ANALYSIS OF PVC SLURRY AND WET CAKE FOR PPM, VCM

SAMPLE TYPE: 5385
DATE: 6-9-82

REACTOR: 700
LOT #: 1A

Response Factor (RF)	=	<u>2727</u>
Area of Sample (As)	=	<u>288745</u>
Whole Sample Weight (SW)	=	<u>1.950</u>
Dry Weight ÷ wet weight (TS)	=	<u>0.62</u>
SW x TS = Resin Weight (RW)	=	<u>1.209</u>
RW ÷ 1.4 = Resin Volume (RV)	=	<u>.864</u>
SW - RW = Water Weight (WW)	=	<u>.741</u>
RV + WW = Sample Volume (SV)	=	<u>1.605</u>
23.0 - SV = Vial Volume (VV)	=	<u>21.395</u>
Lab Barometric Pressure at the Time the Standards were Prepared (P)	=	<u>758.8</u>
Lab Temperature at Time Standards were Prepared in °K = °C + 273 = (T)	=	<u>295</u>
1 - TS = Water Fraction (TW)	=	<u>0.38</u>

$$RVCM = \left[\left(\frac{VV \times 10^{-3}}{RW} \right) + (TS \times 2.36 \times 10^{-3}) + (TW \times 1.82 \times 10^{-3}) \right] \frac{As}{RF} \frac{P}{T}$$

RVCM of Sample Dry Basis = 54.1

A

CONFIDENTIAL

ANALYSIS OF PVC SLURRY AND WET CAKE FOR PPM, VCM

SAMPLE TYPE: 5381
 DATE: 6-9-82

REACTOR: -700
 LOT #: 1 B

Response Factor (RF)	=	<u>2727</u>
Area of Sample (As)	=	<u>200331</u>
Whole Sample Weight (SW)	=	<u>1.514</u>
Dry Weight ÷ wet weight (TS)	=	<u>.62</u>
SW x TS = Resin Weight (RW)	=	<u>.939</u>
RW ÷ 1.4 = Resin Volume (RV)	=	<u>.671</u>
SW - RW = Water Weight (WW)	=	<u>.575</u>
RV + WW = Sample Volume (SV)	=	<u>1.246</u>
23.0 - SV = Vial Volume (VV)	=	<u>21.754</u>
Lab Barometric Pressure at the Time the Standards were Prepared (P)	=	<u>758.8</u>
Lab Temperature at Time Standards were Prepared in °K = °C + 273 = (T)	=	<u>295</u>
1 - TS = Water Fraction (TW)	=	<u>0.38</u>

$$RVCM = \left[\left(\frac{VV \times 10^{-3}}{RW} \right) + (TS \times 2.36 \times 10^{-3}) + (TW \times 1.82 \times 10^{-3}) \right] \frac{As}{RF} \frac{P}{T}$$

RVCM of Sample Dry Basis = 47.9

CONFIDENTIAL

ANALYSIS OF PVC SLURRY AND WET CAKE FOR PPM, VCM

SAMPLE TYPE: 5385

REACTOR: 700

DATE: 6-9-82

LOT #: 1C

Response Factor (RF) = 2727

Area of Sample (As) = 224701

Whole Sample Weight (SW) = 1.664

Dry Weight $\div$ wet weight (TS) = 0.62

SW x TS = Resin Weight (RW) = 1.032

RW $\div$ 1.4 = Resin Volume (RV) = .737

SW - RW = Water Weight (WW) = .632

RV + WW = Sample Volume (SV) = 1.369

23.0 - SV = Vial Volume (VV) = 21.631

Lab Barometric Pressure at the Time
the Standards were Prepared (P) = 758.8

Lab Temperature at Time Standards were
Prepared in $^{\circ}\text{K} = ^{\circ}\text{C} + 273 = (\text{T})$ = 295

1 - TS = Water Fraction (TW) = 0.38

$$\text{RVCM (dry)} = \left[\left(\frac{\text{VV} \times 10^{-3}}{\text{RW}} \right) + (\text{TS} \times 2.36 \times 10^{-3}) + (\text{TW} \times 1.82 \times 10^{-3}) \right] \frac{\text{As}}{\text{RF}} \frac{\text{P}}{\text{T}}$$

RVCM of Sample Dry Basis = 49.0

CONFIDENTIAL

PPM, VCM IN AIR BAG

SAMPLE ID: ROL Sample #2
DATE: 6/9/82

Sample Loop Temperature for Standard (Tr) = 100
Sample Loop Temperature for Sample (Ts) = 100
Barometric Pressure for Standard (Pr) = 758.8
Barometric Pressure for Sample (Ps) = 758.8
Water Vapor Content of Bag (B) = .026
Response Factor (Rf) = $\frac{\text{Area Standard}}{\text{PPM, VCM in Standard}}$ = 15.46
Area of Sample (As) = 143569
$$C_{vcm} = \frac{As}{Rf} \times \frac{Pr}{Ps} \times \frac{Ts}{Tr} \times \frac{1}{1-B}$$
 = 9534

Bag Temperature (B)

20°C	.022
21	.025
<u>22</u>	<u>.026</u>
23	.028
24	.029
25	.031
26	.033
27	.035
28	.037

A

CONFIDENTIAL

ANALYSIS OF PVC SLURRY AND WET CAKE FOR PPM, VCM

SAMPLE TYPE: 5385

REACTOR: 700

DATE: 6-9-82

LOT #: 2A

Response Factor (RF) = 2727

Area of Sample (As) = 174222

Whole Sample Weight (SW) = 1.818

Dry Weight ÷ wet weight (TS) = 0.62

SW x TS = Resin Weight (RW) = 1.127

RW ÷ 1.4 = Resin Volume (RV) = .805

SW - RW = Water Weight (WW) = .691

RV + WW = Sample Volume (SV) = 1.496

23.0 - SV = Vial Volume (VV) = 21.504

Lab Barometric Pressure at the Time
the Standards were Prepared (P) = 758.8

Lab Temperature at Time Standards were
Prepared in °K = °C + 273 = (T) = 295

1 - TS = Water Fraction (TW) = 0.38

$$RVCM = \left[\left(\frac{VV \times 10^{-3}}{RW} \right) + (TS \times 2.36 \times 10^{-3}) + (TW \times 1.82 \times 10^{-3}) \right] \frac{As}{RF} \frac{P}{T}$$

RVCM of Sample Dry Basis = 34.9

A

CONFIDENTIAL

ANALYSIS OF PVC SLURRY AND WET CAKE FOR PPM, VCM

SAMPLE TYPE: 5385
 DATE: 6-9-82

REACTOR: 700
 LOT #: 2B

Response Factor (RF)	=	<u>2727</u>
Area of Sample (As)	=	<u>148921</u>
Whole Sample Weight (SW)	=	<u>1.579</u>
Dry Weight ÷ wet weight (TS)	=	<u>0.62</u>
SW x TS = Resin Weight (RW)	=	<u>.979</u>
RW ÷ 1.4 = Resin Volume (RV)	=	<u>.699</u>
SW - RW = Water Weight (WW)	=	<u>.600</u>
RV + WW = Sample Volume (SV)	=	<u>1.299</u>
23.0 - SV = Vial Volume (VV)	=	<u>21.701</u>
Lab Barometric Pressure at the Time the Standards were Prepared (P)	=	<u>758.8</u>
Lab Temperature at Time Standards were Prepared in °K = °C + 273 = (T)	=	<u>295</u>
1 - TS = Water Fraction (TW)	=	<u>0.38</u>

$$\begin{aligned}
 \text{RVCM (dry)} &= \left[\left(\frac{VV \times 10^{-3}}{RW} \right) + (TS \times 2.36 \times 10^{-3}) + (TW \times 1.82 \times 10^{-3}) \right] \frac{As}{Rf} \frac{P}{T} \\
 \text{RVCM of Sample Dry Basis} &= \underline{34.2}
 \end{aligned}$$

CONFIDENTIAL

ANALYSIS OF PVC SLURRY AND WET CAKE FOR PPM, VCM

SAMPLE TYPE: 5-385

REACTOR: 700

DATE: 6-9-82

LOT #: 2C

Response Factor (RF) = 2727

Area of Sample (As) = 162935

Whole Sample Weight (SW) = 1.616

Dry Weight $\div$ wet weight (TS) = 0.63

SW x TS = Resin Weight (RW) = 1.018

RW $\div$ 1.4 = Resin Volume (RV) = .727

SW - RW = Water Weight (WW) = .598

RV + WW = Sample Volume (SV) = 1.325

23.0 - SV = Vial Volume (VV) = 21.675

Lab Barometric Pressure at the Time
the Standards were Prepared (P) = 758.8

Lab Temperature at Time Standards were
Prepared in $^{\circ}\text{K} = ^{\circ}\text{C} + 273 = (T)$ = 295

1 - TS = Water Fraction (TW) = 0.37

$$\text{RVCM (dry)} = \left[\left(\frac{\text{VV} \times 10^{-3}}{\text{RW}} \right) + (\text{TS} \times 2.36 \times 10^{-3}) + (\text{TW} \times 1.82 \times 10^{-3}) \right] \frac{\text{As}}{\text{RF}} \frac{\text{P}}{\text{T}}$$

RVCM of Sample Dry Basis = 36.0

A

CONFIDENTIAL

PPM, VCM IN AIR BAG

SAMPLE ID: ROL Sample #3
 DATE: 6-10-82

Sample Loop Temperature for Standard (Tr)	=	<u>100</u>
Sample Loop Temperature for Sample (Ts)	=	<u>100</u>
Barometric Pressure for Standard (Pr)	=	<u>760.6</u>
Barometric Pressure for Sample (Ps)	=	<u>759.4</u>
Water Vapor Content of Bag (B)	=	<u>.029</u>
Response Factor (Rf) = $\frac{\text{Area Standard}}{\text{PPM, VCM in Standard}}$	=	<u>16.10</u>
Area of Sample (As)	=	<u>414913</u>
$C_{vcm} = \frac{As}{Rf} \times \frac{Pr}{Ps} \times \frac{Ts}{Tr} \times \frac{1}{1-B}$	=	<u>26,540</u>

Bag Temperature	(B)
20°C	.022
21	.025
22	.026
23	.028
<u>24</u>	<u>.029</u>
25	.031
26	.033
27	.035
28	.037

CONFIDENTIAL

ANALYSIS OF PVC SLURRY AND WET CAKE FOR PPM, VCM

SAMPLE TYPE: 5305
 DATE: 6-11-82

REACTOR: 700
 LOT #: 3A

Response Factor (RF)	=	<u>2774</u>
Area of Sample (As)	=	<u>273802</u>
Whole Sample Weight (SW)	=	<u>1.592</u>
Dry Weight ÷ wet weight (TS)	=	<u>0.61</u>
SW x TS = Resin Weight (RW)	=	<u>.971</u>
RW ÷ 1.4 = Resin Volume (RV)	=	<u>.694</u>
SW - RW = Water Weight (WW)	=	<u>.621</u>
RV + WW = Sample Volume (SV)	=	<u>1.315</u>
23.0 - SV = Vial Volume (VV)	=	<u>21.685</u>
Lab Barometric Pressure at the Time the Standards were Prepared (P)	=	<u>760.6</u>
Lab Temperature at Time Standards were Prepared in °K = °C + 273 = (T)	=	<u>295</u>
1 - TS = Water Fraction (TW)	=	<u>0.39</u>

$$RVCM_{(dry)} = \left[\left(\frac{VV \times 10^{-3}}{RW} \right) + (TS \times 2.36 \times 10^{-3}) + (TW \times 1.82 \times 10^{-3}) \right] \frac{As}{RF} \frac{P}{T}$$

RVCM of Sample Dry Basis = 62.3

CONFIDENTIAL

ANALYSIS OF PVC SLURRY AND WET CAKE FOR PPM, VCM

SAMPLE TYPE: 5305

REACTOR: 700

DATE: 6/11/82

LOT #: 3B

Response Factor (RF) = 2774

Area of Sample (As) = 340061

Whole Sample Weight (SW) = 1.834

Dry Weight $\div$ wet weight (TS) = 0.64

SW x TS = Resin Weight (RW) = 1.174

RW $\div$ 1.4 = Resin Volume (RV) = .839

SW - RW = Water Weight (WW) = .660

RV + WW = Sample Volume (SV) = 1.499

23.0 - SV = Vial Volume (VV) = 21.501

Lab Barometric Pressure at the Time
the Standards were Prepared (P) = 760.6

Lab Temperature at Time Standards were
Prepared in $^{\circ}\text{K} = ^{\circ}\text{C} + 273 = (T)$ = 295

1 - TS = Water Fraction (TW) = 0.36

$$\text{RVCM (dry)} = \left[\left(\frac{\text{VV} \times 10^{-3}}{\text{RW}} \right) + (\text{TS} \times 2.36 \times 10^{-3}) + (\text{TW} \times 1.82 \times 10^{-3}) \right] \frac{\text{As}}{\text{RF}} \frac{\text{P}}{\text{T}}$$

RVCM of Sample Dry Basis = 64.7

CONFIDENTIAL

ANALYSIS OF PVC SLURRY AND WET CAKE FOR PPM, VCM

SAMPLE TYPE: 5305

REACTOR: 700

DATE: 6/11/82

LOT #: 3C

Response Factor (RF) = 2774

Area of Sample (As) = 340185

Whole Sample Weight (SW) = 1.732

Dry Weight ÷ wet weight (TS) = 0.63

SW x TS = Resin Weight (RW) = 1.091

RW ÷ 1.4 = Resin Volume (RV) = .779

SW - RW = Water Weight (WW) = .641

RV + WW = Sample Volume (SV) = 1.420

23.0 - SV = Vial Volume (VV) = 21.580

Lab Barometric Pressure at the Time
the Standards were Prepared (P) = 760.6

Lab Temperature at Time Standards were
Prepared in °K = °C + 273 = (T) = 295

1 - TS = Water Fraction (TW) = 0.37

$$RVCM_{(dry)} = \left[\left(\frac{VV \times 10^{-3}}{RW} \right) + (TS \times 2.36 \times 10^{-3}) + (TW \times 1.82 \times 10^{-3}) \right] \frac{As}{RF} \frac{P}{T}$$

RVCM of Sample Dry Basis = 69.4

SAMPLE 4052 Standard
PRESSURE 761.0
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 10
CHART SPEED 1 cm/min

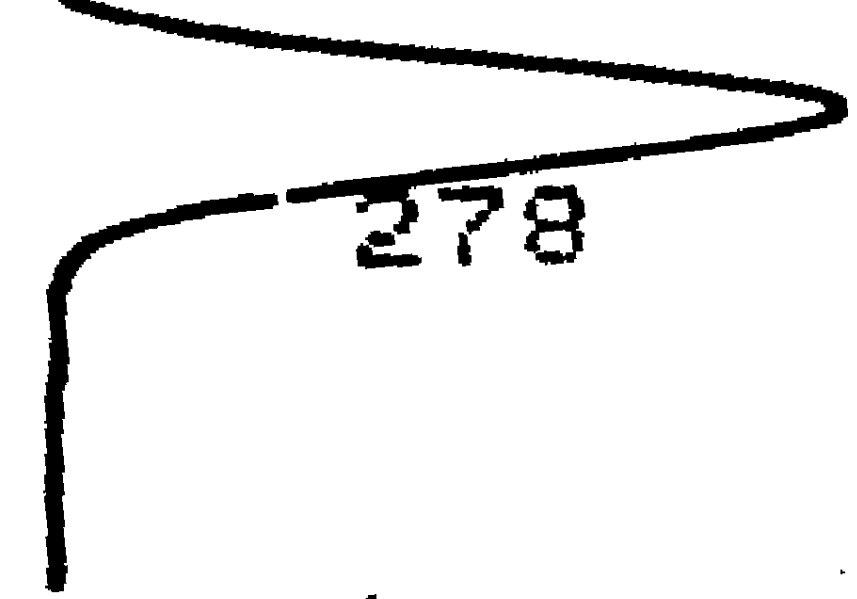
278

CONFIDENTIAL

ROL VCM
CHANNEL 1 RUN 81 1982 MAY 39 06.20:55
INDEX 1 CALIBRATION FILE 5 METHOD 5
NAME CONC RT AREA KE
VCM 4052 278 69102 17.05
TOTALS 4052 69102

NEW FILE
NAME RT KE
VCM 278 17.05

SAMPLE 4052 Standard
PRESSURE 761.0
LOOP TEMP. 180
COLUMN TEMP. 113
FLOW RATE 30
ATTENUATOR 10
CHART SPEED 1 cm/min

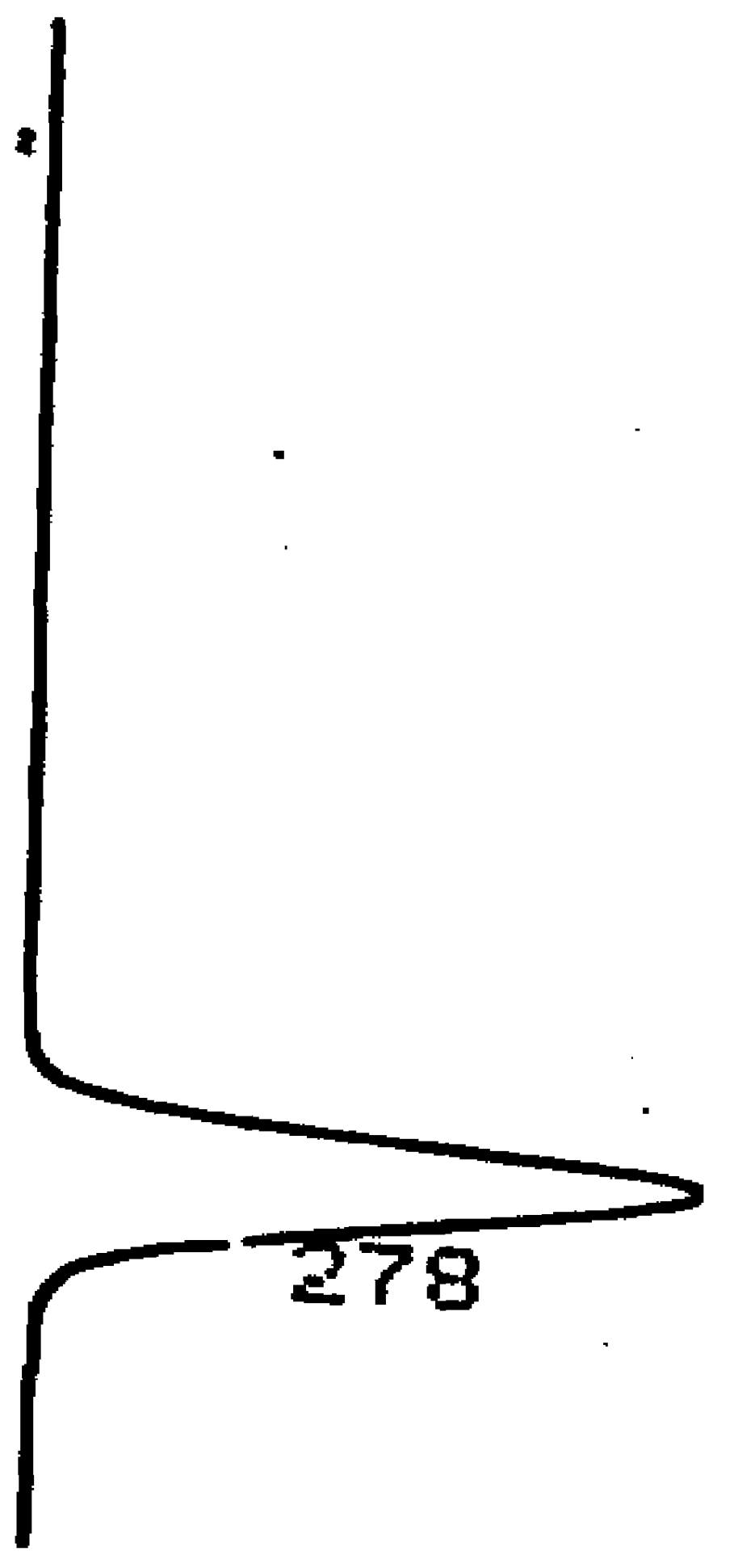


CONFIDENTIAL

ROL VCM 1982 MAY 30 05:28:42
CHANNEL 1 RUN 82 FILE 5 METHOD 5
INDEX 2 CALIBRATION
NAME CONC RT AREA KF
VCM 4052 278 64176 15.84
TOTALS 4052 64176

NEW FILE
NAME RT KF
VCM 278 15.44

SAMPLE 4052 Standard
 PRESSURE 261.0
 LOOP TEMP. 100
 COLUMN TEMP. 10.3
 FLOW RATE 30
 ATTENUATOR 10
 CHART SPEED 1 cm/min



CONFIDENTIAL

ROL VCM
 CHANNEL 1 RUN 93 FILE 5 METHOD 5
 INDEX 3 CALIBRATION
 NAME CONC RT AREA KF
 VCM 4052 278 65847 16.5
 TOTALS 4052 65847

NEW FILE
 NAME RT KF
 VCM 278 16.46

SAMPLE 53 Standard
 PRESSURE 261.0
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 1
 CHART SPEED 1 cm/min

47

278

CONFIDENTIAL

ROL VCM 1982 MAY 39 06:49:20
 CHANNEL 1 RUN 84 FILE 5 METHOD 5
 INDEX 1
 NAME CONC RT AREA KF
 VCM¹ 51.28 47 54 15.46
 278 844
 TOTALS 51.28 992

47

SAMPLE 53 Standard
 PRESSURE 761.0
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 1
 CHART SPEED 1 cm/min

278

CONFIDENTIAL

ROL VCM

1982 MAY 30 07:17:13

CHANNEL 1 RUN 87

FILE 5 METHOD 5

INDEX 2

NAME	CONC	RT	AREA	KE
VCM ¹	47.57	47 278	44 783	15.46
TOTALS	47.57		827	

SAMPLE 53 Standard
 PRESSURE 241.0
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 1
 CHART SPEED 1 cm/min

47

278

CONFIDENTIAL

ROL VCM

1982 MAY 39 07:25:52

CHANNEL 1 RUN 88 FILE 5 METHOD 5

INDEX 3

NAME	CONC	RT	AREA	KF
VCM ¹	44.78	47 278	37 737	15.46
TOTALS	44.78		774	

SAMPLE 496 Standard
 PRESSURE 261.0
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 5
 CHART SPEED 1 cm/min

278

CONFIDENTIAL

ROL VCM

1982 MAY 30 07:40:53

CHANNEL 1 RUN 89

FILE 5 METHOD 5

INDEX 1

NAME

CONC

RT

AREA

VF

VCM

508

278

8362

16.16

TOTALS

508

8362

SAMPLE 496 Standard
 PRESSURE 761.0
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 5
 CHART SPEED 1 cm/min

278

CONFIDENTIAL

ROL VCM 1982 MAY 39 07:49:09
 CHANNEL 1 RUN 90 FILE 5 METHOD 5
 INDEX 2
 NAME CONC RT AREA KF
 VCM 466 278 7670 15.46
 TOTALS 466 7670

Spectra-Physics

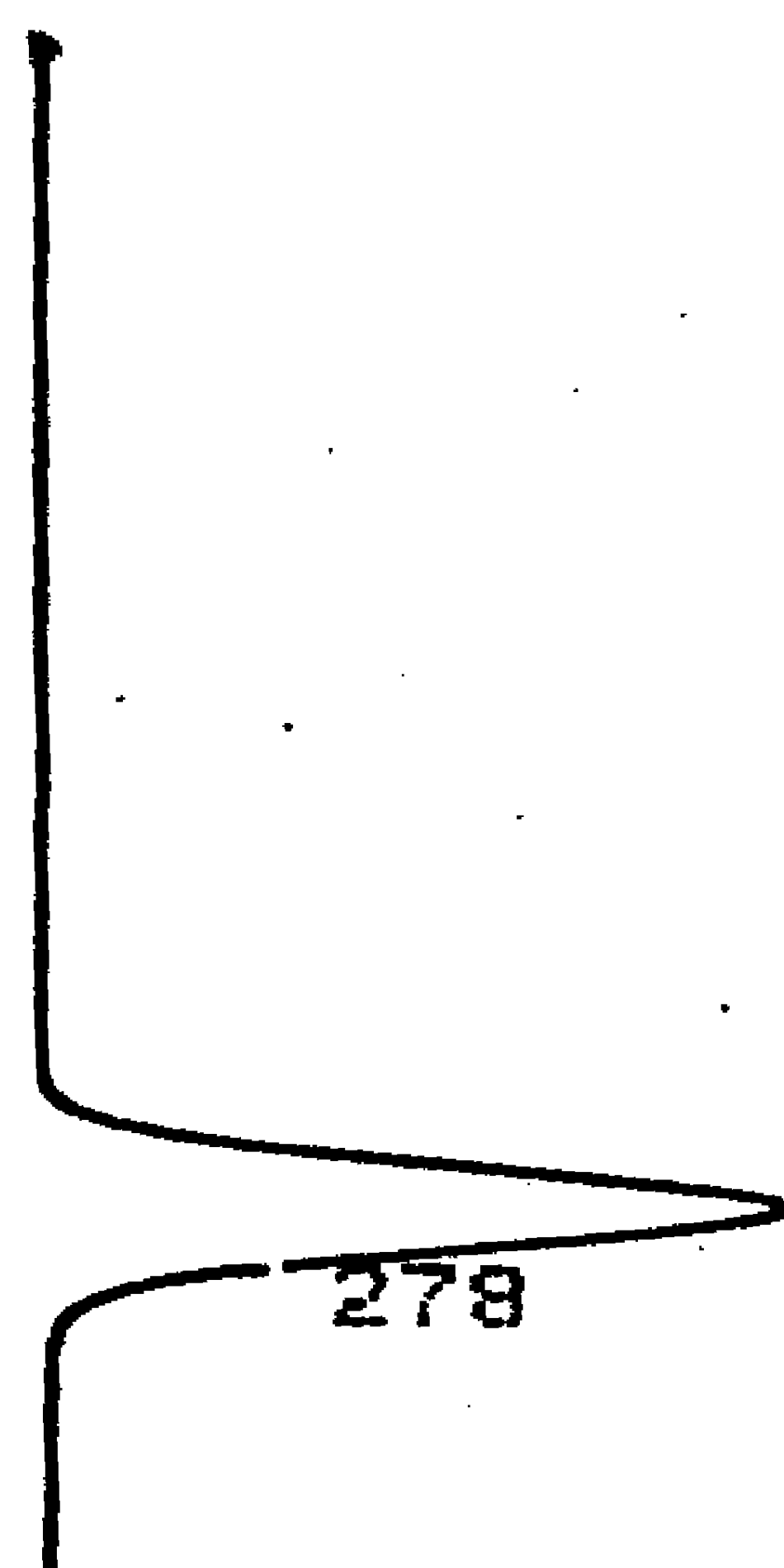
SAMPLE 496 Standard
 PRESSURE 761.0
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 5
 CHART SPEED 1 cm/min

278

CONFIDENTIAL

ROL VCM 1982 MAY 30 07:57:16
 CHANNEL 1 RUN 91 FILE 5 METHOD 5
 INDEX 3
 NAME CONC RT AREA KF
 VCM 484.2 278 7969 16.45
 TOTALS 484.2 7969

SAMPLE 2188 Standard
 PRESSURE 761.0
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 5
 CHART SPEED 1 cm/min



CONFIDENTIAL

ROL VCM

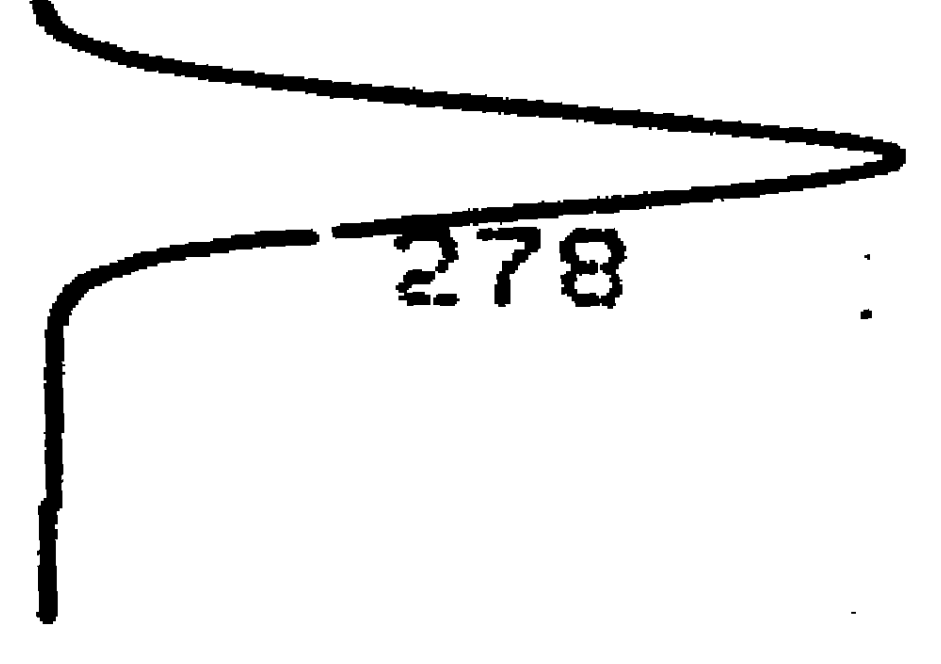
1982 MAY 39 08:08:57

CHANNEL 1 RUN 92 FILE 5 METHOD 5

INDEX 4

NAME	CONC	RT	AREA	KF
VCM	2171	278	35732	16.46
TOTALS	2171		35732	

SAMPLE 2188 standard
PRESSURE 761.0
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 5
CHART SPEED 1 cm/min



CONFIDENTIAL

ROL VCM

1982 MAY 30 08:25:07

CHANNEL 1 RUN 94

FILE 5 METHOD 5

INDEX 6

NAME	CONC	RT	AREA	KF
VCM	2075	278	34148	16.46
TOTALS	2075		34148	

start

29

182

279

SAMPLE ROL Sample 41
PRESSURE 761.0
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 50
CHART SPEED 1 cm/min

111

CONFIDENTIAL

ROL VCM

1982 MAY 39 11:25:50

CHANNEL 1 RUN 96 FILE 5 METHOD 5

INDEX 6

NAME	CONC	RT	AREA	KE
------	------	----	------	----

VCM ¹	9140	182 278	226 150442	15.46
------------------	------	------------	---------------	-------

TOTALS	9140		150668	
--------	------	--	--------	--

A
112

SAMPLE ROL Sample #1
PRESSURE 761.0
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 50
CHART SPEED 1 cm/min

start

182

278

CONFIDENTIAL

ROL VCM

1982 MAY 30 11:19:55

CHANNEL 1

RUN 97

FILE 5

METHOD 5

INDEX 7

NAME

CONC

RT

AREA

KF

VCM¹

9864

182
278

212
145890

16.46

TOTALS

9863

146103

Spectra-Physics

VAB.0001115718

start

A
113

SAMPLE ROL Sample #1
PRESSURE 261.0
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 50
CHART SPEED 1 cm/min



CONFIDENTIAL

ROL VCM
CHANNEL 1 RUN 98 FILE 5 METHOD 5
INDEX 8

1982 MAY 39 13 02:36

NAME	CONC	RT	PRER	KE
VCM ¹	8938	182 278	209 147104	15.46
TOTALS	8937		147313	

stand

SAMPLE ROL Sample #2
PRESSURE 258
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 1
CHART SPEED 1 cm/min

A11

182

278

CONFIDENTIAL

ROL VCM

1982 MAY 40 07:07:57

CHANNEL 1 RUN 99 FILE 5 METHOD 5

INDEX 1

NAME	CONC	RT	AREA	KF
1		182	205	
VCM	8770	278	144356	15.46
TOTALS	8770		144561	

start

SAMPLE ROL Sample #2
PRESSURE 2582
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 1
CHART SPEED 1 cm/min

A 11

192

278

CONFIDENTIAL

ROL VCM

1982 MAY 40 07:23.11

CHANNEL 1 RUN 101 FILE 5 METHOD 5

INDEX 3

NAME	CONC	RT	AREA	KE
VCM ¹	8452	182 278	200 139109	15.45
TOTALS	8451		139309	

start

SAMPLE ROL Sample #2
PRESSURE 758.8
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 1
CHART SPEED 1 cm/min

118

A

CONFIDENTIAL

1992 MAY 40 07:37:21

EL 1 RUN 102 FILE 5 METHOD 5

4

CONC

RT

AREA

KF

8946

182
275

218
147242

10.46

8945

147461

Spectra-Physics

VAB.0001115722

SAMPLE 4052 Standard
PRESSURE 758.6
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 10
CHART SPEED 1cm/min

284

CONFIDENTIAL

ROL VCM

1982 MAY 40 13:24:41

CHANNEL 1 RUN 107 FILE 5 METHOD 5

INDEX 9 CALIBRATION

NAME	CONC	RT	AREA	KF
VCM	4052	284	61060	15.07
TOTALS	4052		61060	

NEW FILE

NAME	RT	KF
VCM	281	15.3

SAMPLE 4052 Standard
PRESSURE 258.8
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 10
CHART SPEED 1 cm/min

284

CONFIDENTIAL

ROL VCM

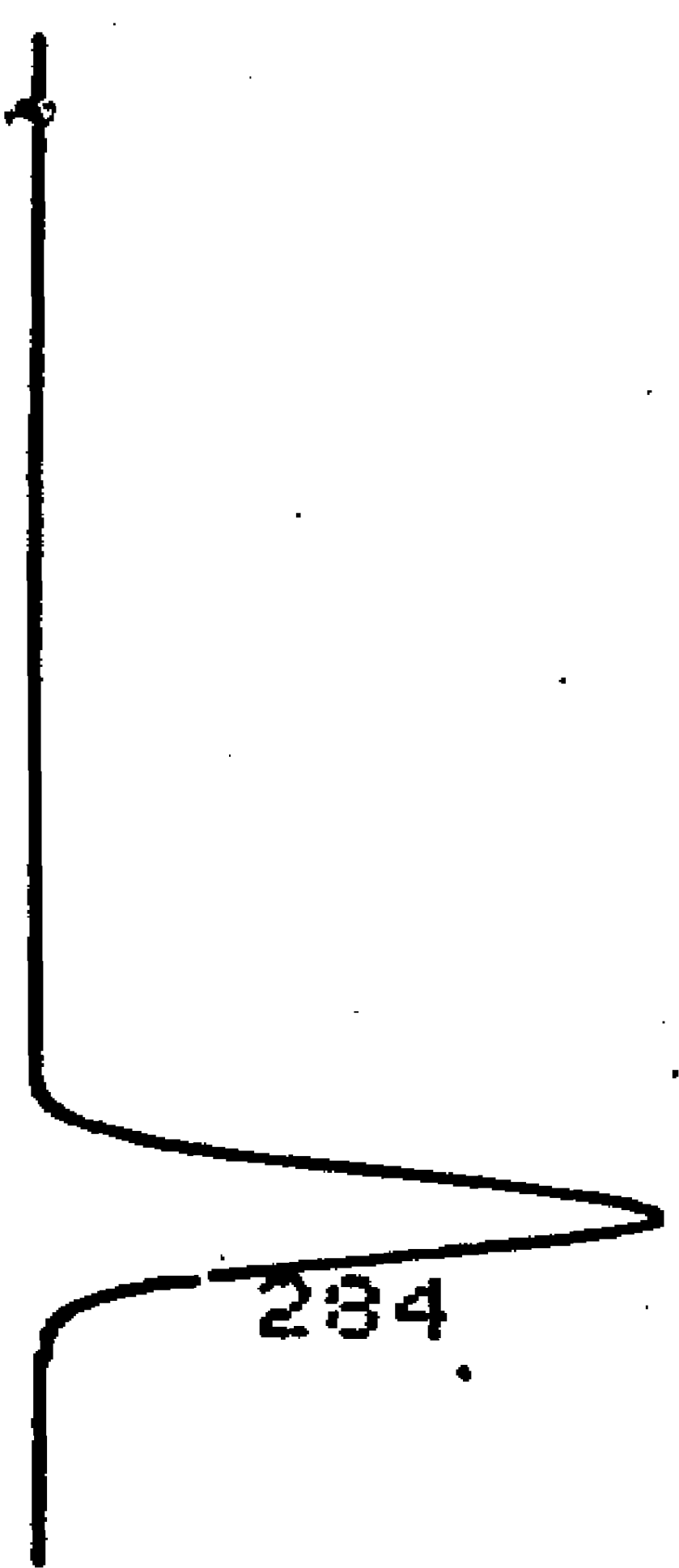
1982 MAY 40 13:32:57

CHANNEL	1	RUN	108	FILE	5	METHOD	5
INDEX	10	CALIBRATION					
NAME		CONC	RT	AREA		KF	
VCM		4052	284	64160		15.93	
TOTALS		4052		64160			

NEW FILE

NAME	RT	KF
VCM	282	15.25

SAMPLE 4052 standard
 PRESSURE 796.8
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 10
 CHART SPEED 1 cm/min



CONFIDENTIAL

ROL VCM 1982 MAY 40 13:15:29
 CHANNEL 1 RUN 109 FILE 5 METHOD 5

INDEX 11 CALIBRATION

NAME	CONC	RT	AREA	KF
VCM	4052	284	62677	15.47
TOTALS	4052		62677	

NEW FILE

NAME	RT	KF
VCM	283	16.18

SAMPLE 5.3 Standard
PRESSURE 728.2
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 1
CHART SPEED 1 cm/min

47

284

CONFIDENTIAL

ROL VCM

1982 MAY 40 13:59:57

CHANNEL 1 RUN 110 FILE 5 METHOD 5

INDEX 12

NAME	CONC	RT	AREA	KF
VCM ¹	50.38	47 284	52 815	16.18
TOTALS	50.38		867	

SAMPLE 53 Standard
PRESSURE 758.8
LOOP TEMP. 100
COLUMN TEMP. 113
FLOW RATE 30
ATTENUATOR 1
CHART SPEED 1 cm/min

CONFIDENTIAL

1982 JUN 09 14:20:21

1 RUN 112 FILE 5 METHOD 5

CONC	RT	AREA	KF
48.65	284	787	16.18
48.65		787	

SAMPLE 5-3 Standard
PRESSURE 758.8
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 1
CHART SPEED 1 cm/min

CONFIDENTIAL

1992 JUN 09 14:27:47

1 RUN 113 FILE 5 METHOD 5

CONC	RT	AREA	KF
50.75	47 284	47 821	16.18
50.75		863	

Spectra-Physics

VAB.0001115728

SAMPLE 496 Standard
PRESSURE 758.8
LOOP TEMP. 100
COLUMN TEMP. 10.3
FLOW RATE 30
ATTENUATOR 5
CHART SPEED 1 cm/min

130

305

CONFIDENTIAL

ROL VCM

1982 JUN 09 14:54:24

CHANNEL 1 RUN 114 FILE 5 METHOD 5

INDEX 16

NAME	CONC	RT	AREA	KF
VCM	445.1	305	7200	15.18
TOTALS	445.1		7200	

132

SAMPLE 496 Standard
 PRESSURE 758.8
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 3
 CHART SPEED 1cm/min

305

CONFIDENTIAL

ROL VCM

1982 JUN 09 15:09:14

CHANNEL 1 RUN 116 FILE 5 METHOD 5

INDEX 18

NAME	CONC	RT	AREA	KF
VCM	428.6	305	6933	15.19
TOTALS	428.6		6933	

Spectra-Physics

VAB.000111500

SAMPLE 496 Standard
PRESSURE 758.6
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 5
CHART SPEED 1 cm/min

305

CONFIDENTIAL

ROL VCM

1992 JUN 09 15:16:47

CHANNEL 1 RUN 117 FILE 5 METHOD 5

INDEX 19

NAME	CONC	RT	PRER	KE
VCM	423.3	305	5848	16.18
TOTALS	423.3		5848	

SAMPLE 2188 Standard
PRESSURE 758.8
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 5
CHART SPEED 1 cm/min

302

CONFIDENTIAL

ROL VCM

1982 JUN 09 15:25:29

CHANNEL 1

RUN 118

FILE 5

METHOD 5

INDEX 20

NAME	CONC	RT	AREA	KF
VCM	1948	302	31506	15.18
TOTALS	1948		31506	

SAMPLE 2188 Standard
PRESSURE 758.1
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 5
CHART SPEED 1 cm/min

13

302

CONFIDENTIAL

ROL VCM

1982 JUN 09 15:22:29

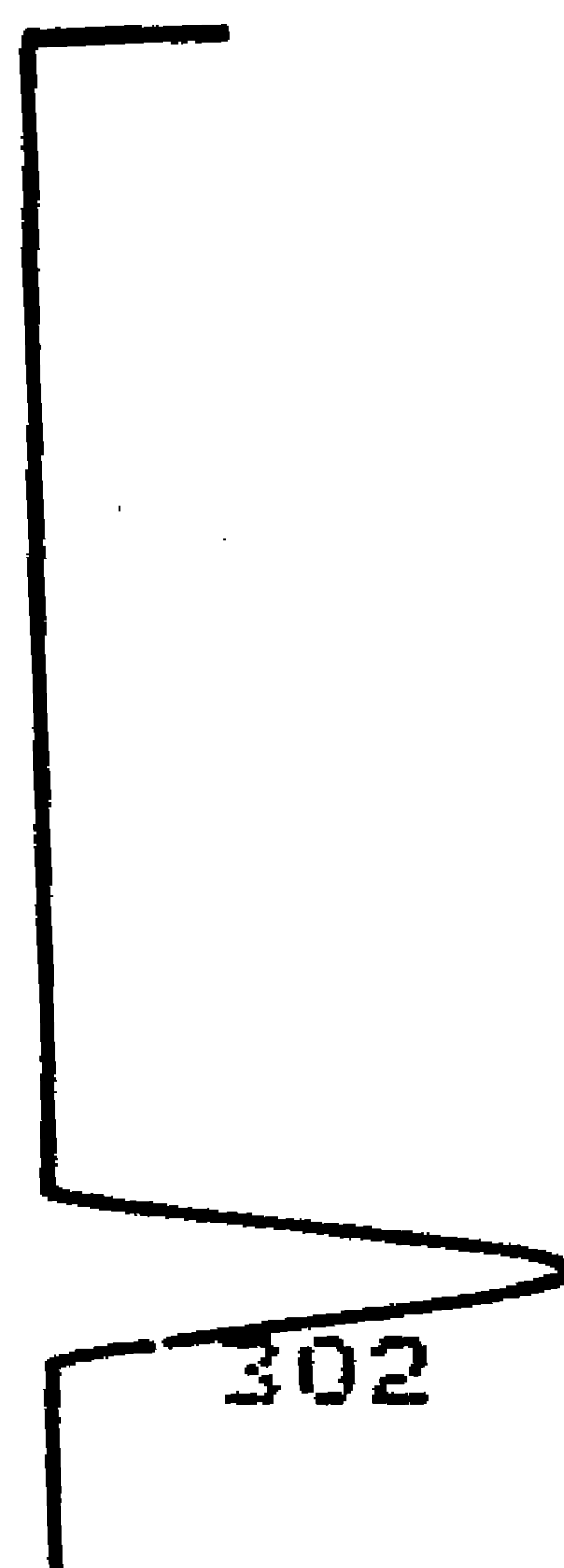
CHANNEL 1 RUN 119 FILE 5 METHOD 5

INDEX 21

NAME	CONC	RT	AREA	KE
VCM	1948	302	31519	15.18
TOTALS	1948		31519	

136

SAMPLE 2188 Standard
 PRESSURE 758.6
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 5
 CHART SPEED 1cm/min



CONFIDENTIAL

ROL VCM 1982 JUN 09 15:39:52
 CHANNEL 1 RUN 120 FILE 5 METHOD 5
 INDEX 22

NAME	CONC	RT	AREA	KF
VCM	1796	302	29059	16.18
TOTALS	1796		29059	

56

197

293

CONFIDENTIAL

ROL VCM

1992 JUN 10 15:09:17

CHANNEL 1 RUN 129

FILE 5 METHOD 5

INDEX 31

NAME	CONC	RT	AREA	KE
1		56	65	
2		197	348	
VCM	26870	293	434624	L 16.19
TOTALS	26870		435037	

107

197

293

CONFIDENTIAL

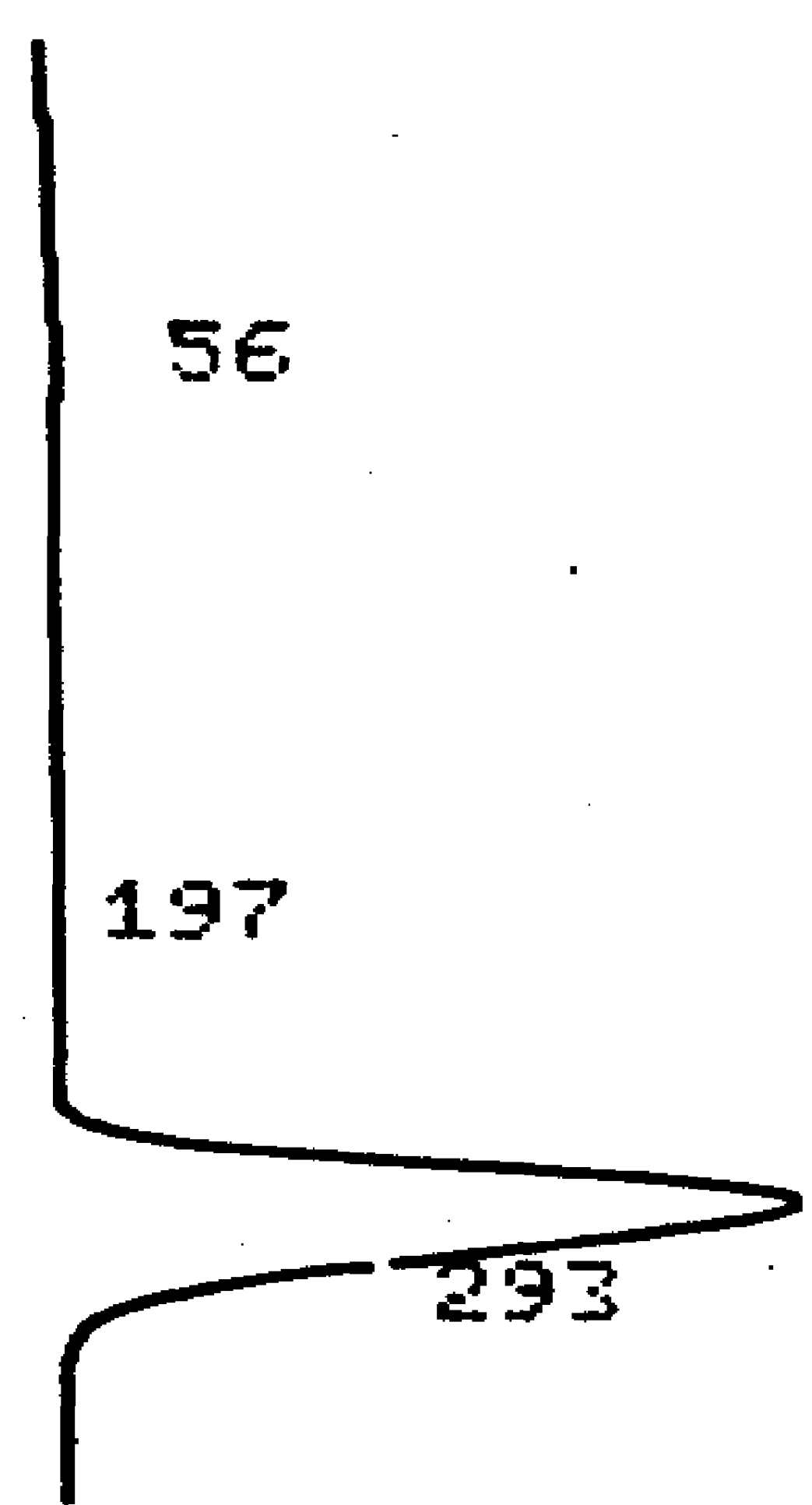
ROL VCM

1982 JUN 10 15:16:50

CHANNEL 1 RUN 130 FILE 5 METHOD 5

INDEX 32

NAME	CONC	RT	AREA	KE
1		107	6484	L
2		197	319	L
VCM	24640	293	398621	L 16.18
TOTALS	24640		405423	



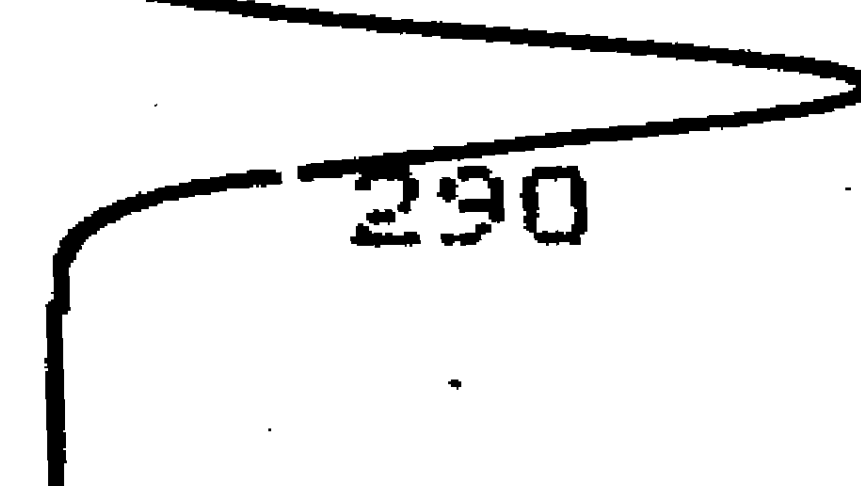
CONFIDENTIAL

```

ROL VCM                                1982 JUN 10 15:25:28
CHANNEL 1      RUN 131      FILE 5      METHOD 5
INDEX 33
NAME          CONC          RT      AREA          KF
  1          56          5615
  2          197          331
VCM          25440          293      411492      L      16.18
TOTALS          25440          417439
    
```


Start

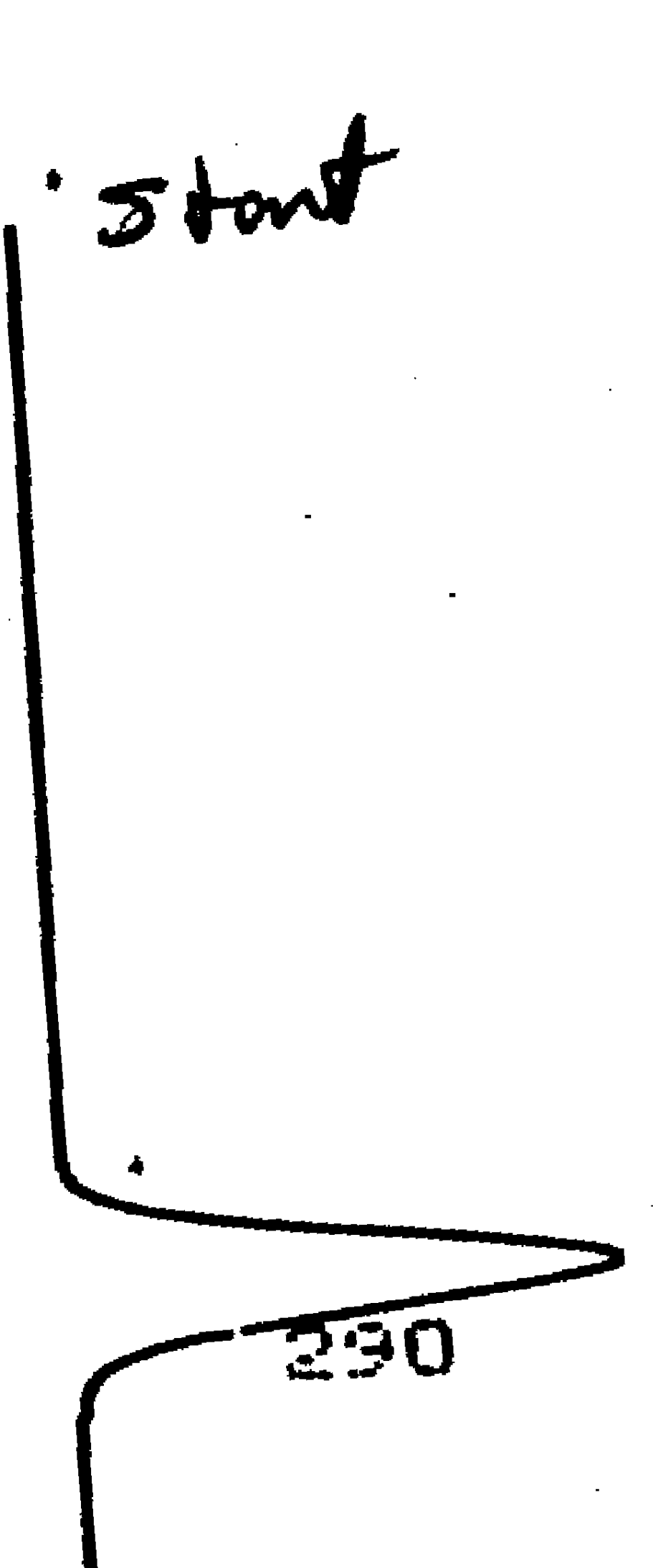
SAMPLE 4052 Standard
 PRESSURE 760.6
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 10
 CHART SPEED 1 cm/min



CONFIDENTIAL

ROL VCM 1982 JUN 11 09:41:40
 CHANNEL 1 RUN 134 FILE 5 METHOD 5
 INDEX 36 CALIBRATION
 NAME CONC RT AREA KF
 VCM 4052 290 68010 16.73
 TOTALS 4052 68010

NEW FILE
 NAME RT KF
 VCM 286 15.10



SAMPLE 4052 Standard
 PRESSURE 260.6
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 10
 CHART SPEED 1cm/min

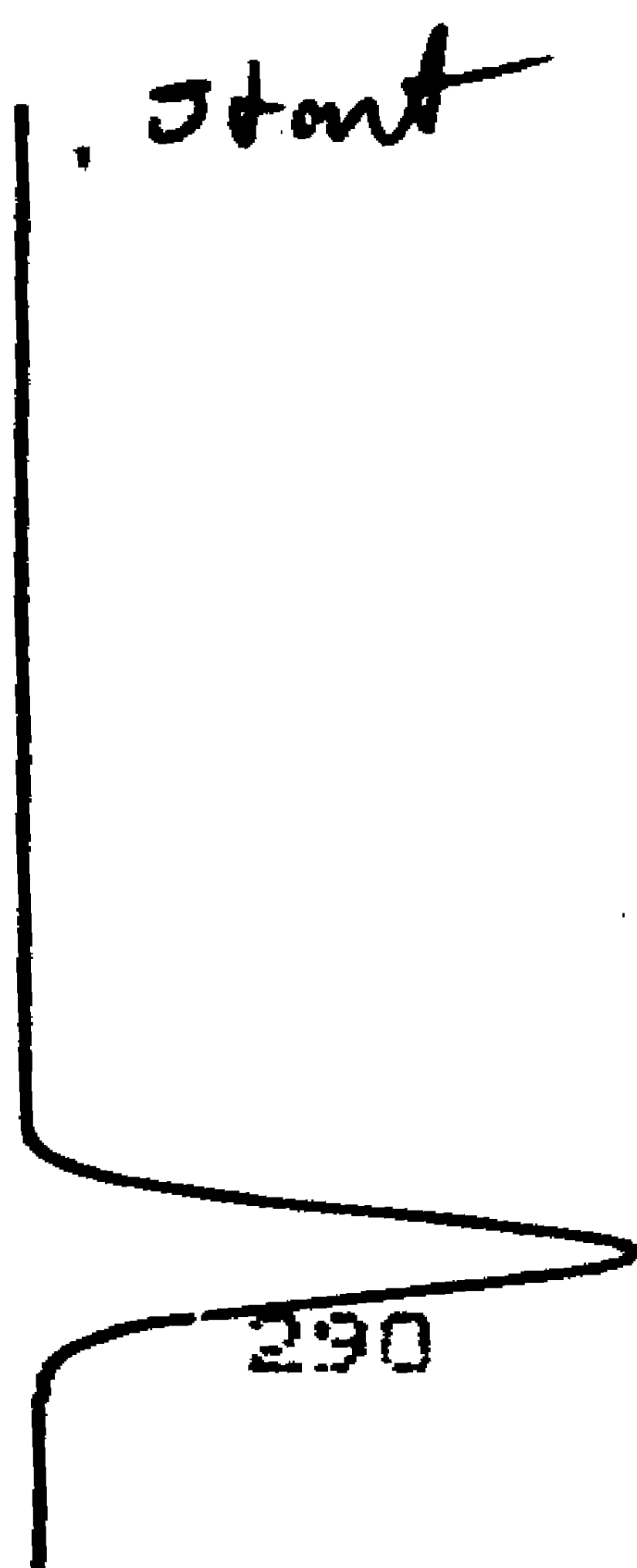
CONFIDENTIAL

1982 JUN 11 00:49:10

ROL VCM	CHANNEL	1	RUN 135	FILE	5	METHOD	5
INDEX	37	CALIBRATION					
NAME		CONC	RT	AREA	KF		
VCM		4052	290	53256	15.61		
TOTALS		4052		53256			

NEW FILE

NAME	RT	KF
VCM	299	16.17



SAMPLE 4052 Standard
PRESSURE 760.6
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 10
CHART SPEED 1 cm/min

154

CONFIDENTIAL

ROL VCM

1992 JUN 11 10:22:16

CHANNEL 1 RUN 136 FILE 5 METHOD 3

INDEX 38 CALIBRATION

NAME	CONC	RT	AREA	KF
VCM	4052	290	61431	15.0
TOTALS	4052		61433	

NEW FILE

NAME	RT	KF
VCM	289	15.10

Spectra-Physics

VAB.0001115710

50 start

SAMPLE 53 Standard
PRESSURE 760.6
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 1
CHART SPEED 100/min

156

290

CONFIDENTIAL

ROL VCM

1982 JUN 11 10:14:48

CHANNEL 1 RUN 139 FILE 5 METHOD 5

INDEX 39

NAME	CONC	RT	AREA	KF
VCM	47.46	290	767	16.15
TOTALS	47.46		767	

SAMPLE 53 Standard
PRESSURE 260.6
LOOP TEMP. 100
COLUMN TEMP. 10.3
FLOW RATE 30
ATTENUATOR 1
CHART SPEED 6 cm/min

47 start

287

CONFIDENTIAL

ROL VCM

1982 JUN 11 10:51:45

CHANNEL 1 RUN 140 FILE 5 METHOD 5

INDEX 40

NAME	CONC	RT	AREA	WF
VCM ¹	47.95	47 297	39 775	15.16
TOTALS	47.95		814	

SAMPLE 5.3 standard
 PRESSURE 260.6
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 1
 CHART SPEED 1 cm/min

50 *start*
 287

CONFIDENTIAL

ROL VCM 1992 JUN 11 11 32.32
 CHANNEL 1 RUN 141 FILE 5 METHOD F
 INDEX 41
 NAME CONC RT AREA HF
 1
 VCM 50.37 287 914 15.15
 TOTALS 50.37 962

SAMPLE 496 Standard
PRESSURE 260.6
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 5
CHART SPEED 1 cm/min

287

CONFIDENTIAL

ROL VCM 1982 JUN 11 11:11:20
CHANNEL 1 RUN 142 FILE 5 METHOD F
INDEX 42

NAME	CONC	RT	AREA	KF
VCM	470	287	7595	16.15
TOTALS	469.9		7595	

50

50

287

SAMPLE 496 Standard
 PRESSURE 760.6
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 5
 CHART SPEED 1cm/min

CONFIDENTIAL

ROL VCM

1982 JUN 11 11:21:29

CHANNEL 1 RUN 143 FILE 5 METHOD 5

INDEX 43

NAME	CONC	RT	AREA	KF
VCM	491.7	287	7946	16.15
TOTALS	491.7		7946	

start

161

SAMPLE 496 Standard
PRESSURE 760.6
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 5
CHART SPEED 1cm/min

287

CONFIDENTIAL

ROL VCM

1992 JUN 11 11:20:10

CHANNEL 1 RUN 144 FILE 5 METHOD 5

INDEX 44

NAME	CONC	RT	AREA	KF
VCM	500.3	297	8085	15.15
TOTALS	500.3		8085	

Spectra-Physics

VAB.000111546

Start

SAMPLE 2188 Standard
PRESSURE 760.6
LOOP TEMP. 140
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 3
CHART SPEED 1 cm/min

163

287

CONFIDENTIAL

ROL VCM

1982 JUN 11 11.39 39

CHANNEL 1 RUN 145 FILE 5 METHOD 5

INDEX 45

NAME	CONC	RT	AREA	KE
VCM	2040	287	32968	15.16
TOTALS	2040		32968	

Spectra-Physics

Start

SAMPLE 2188 Standard
PRESSURE 760.6
LOOP TEMP. 100
COLUMN TEMP. 103
FLOW RATE 30
ATTENUATOR 5
CHART SPEED 1 cm/min

287

CONFIDENTIAL

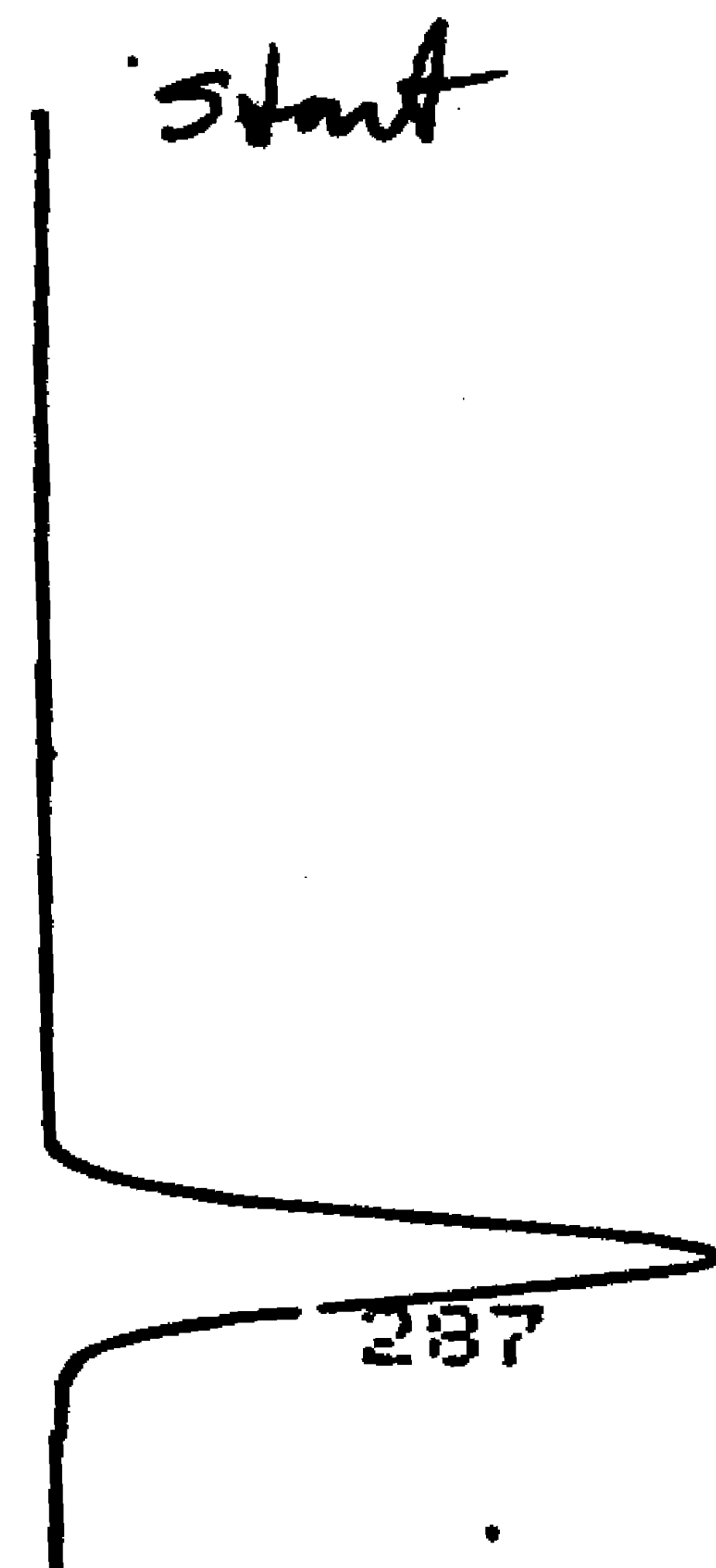
ROL VCM

1982 JUN 11 11.51.20

CHANNEL 1 RUN 146 FILE 5 METHOD 5

INDEX 46

NAME	CONC	RT	AREA	KF
VCM	2093	287	33824	15.15
TOTALS	2093		33824	



SAMPLE 2188 Standard
 PRESSURE 760.6
 LOOP TEMP. 100
 COLUMN TEMP. 103
 FLOW RATE 30
 ATTENUATOR 5
 CHART SPEED 1 cm/min

CONFIDENTIAL

ROL VCM

1992 JUN 11 11:52:27

CHANNEL 1 RUN 147 FILE 5 METHOD 5

INDEX 47

NAME	CONC	RT	AREA	MF
VCM	2169	287	35061	16.16
TOTALS	2169		35061	

CONFIDENTIAL

MA-03-10/77-A

Matheson
A DIVISION OF WILL ROSS, INC.

GAS ANALYSIS

CYL. NO. 24261T INVOICE NO. 12970

CONTROL NO. (69)

GAS	VOLUME PERCENT	
	REQUESTED	ANALYSIS
CARBON DIOXIDE		
OXYGEN		
HYDROGEN		
CARBON MONOXIDE		
NITROGEN	<u>bal.</u>	<u>bal.</u>
ARGON		
AIR	<u>checked 8/14/81</u>	
METHANE	<u>4015 ppm</u>	
HELIUM	<u>4000 ppm</u>	<u>4052 ppm</u>

COMPONENT MAY LIQUEFY AT _____ °F.

ANALYZED BY D. E. Ller

PRESSURE 2000

CONTENTS 209 CUBIC FEET.

READ OTHER SIDE BEFORE USE.

1-4-79

PRINTED IN U.S.A.

FORM MA-03-2-80

Matheson
DIV. SEARLE MEDICAL PRODUCTS USA, INC.

GAS ANALYSIS

CYL. NO. AH-015520 INVOICE NO. 104-48936

CONTROL NO. 3

GAS	VOLUME PERCENT	
	REQUESTED	ANALYSIS
CARBON DIOXIDE		
OXYGEN		
HYDROGEN		
CARBON MONOXIDE		
NITROGEN	<u>bal.</u>	<u>bal.</u>
ARGON		
AIR		
METHANE		
HELIUM	<u>500 ppm</u>	<u>496 ppm</u>

COMPONENT MAY LIQUEFY AT _____ °F.

ANALYZED BY J. E.

PRESSURE 2000#

CONTENTS 209 CUBIC FEET.

READ OTHER SIDE BEFORE USE.

8-27-81

CONFIDENTIAL

CONFIDENTIAL

PRINTED IN U.S.A.

FORM
MA-03-10/77-A

NBS
Traceable

Matheson

A DIVISION OF WILL ROSS, INC.

GAS ANALYSIS 10K

CYL. NO. 3705P INVOICE NO. 34972

CONTROL NO. 5

VOLUME PERCENT

GAS REQUESTED ANALYSIS
CARBON DIOXIDE *is of limited shelf life. Use is recommended within one year from date of manufacture.*
OXYGEN
HYDROGEN
CARBON MONOXIDE

NITROGEN *Bnl* *Bnl*

ARGON

AIR

METHANE

HELIUM

Chloride 2000ppm 2206ppm

COMPONENT MAY LIQUEFY AT 2F

ANALYZED BY *K6*

PRESSURE *200*

CONTENTS *.209* CUBIC FEET

READ OTHER SIDE BEFORE USE.

2-5-82

REACTOR OPENING LOSS CALCULATION

Data

Volume of Reactor, $V = 4,487 \text{ ft.}^3$
Barometric Pressure, $P = 29.95 \text{ in. Hg}$
Reactor Temperature, $T = 650^\circ\text{R}$
Molecular Weight of VCM, $M = 62.5 \frac{\text{lb.}}{\text{lb.-mole}}$
Gas Constant, $R = 21.9 \frac{(\text{in. Hg}) (\text{ft.}^3)}{(\text{lb.-mole } (^\circ\text{R}))}$
Density of VCM at reactor conditions, D
 $D = \frac{PM}{RT} = \frac{(29.95) (62.5)}{(21.9) (650)} = 0.131 \frac{\text{lb.}}{\text{ft.}^3}$
Fraction of water vapor in reactor gas, $W = 0.635$
PVC Production, $Z = 61,924 \text{ lbs. for 5385 resin}$
 $Z = 57,357 \text{ lbs. for 5305 resin}$
Concentration of vinyl chloride in reactor, dry basis, C
 $C = 9210 \text{ ppm for batch 700 - 223}$
 $= 8955 \text{ ppm for batch 700 - 224}$
 $= 25,650 \text{ ppm for batch 700-229}$

CONFIDENTIAL

Reactor Opening Loss Calculations

$$\begin{aligned} \text{ROL} &= \frac{(C) (1-W) (V) (D)}{(Z)} \\ &= \frac{(\text{ft.}^3 \text{ VCM})}{(\text{MM ft.}^3 \text{ dry gas})} \times \frac{(\text{ft.}^3 \text{ dry gas})}{(\text{ft.}^3 \text{ reactor gas})} \times (\text{ft.}^3 \text{ reactor gas}) \times \\ &\quad \frac{(\text{lb. VCM})}{(\text{ft. VCM})} \times \frac{(1)}{\text{lbs. PVC}} = \frac{\text{lbs. VCM}}{\text{MM Lbs. PVC}} \end{aligned}$$

Batch No. 700-223

$$\begin{aligned} \text{ROL} &= \frac{(9210) (1-0.635) (4,487) (0.131)}{61,924} \\ &= \frac{32 \text{ lbs. VCM}}{\text{MM Lbs. PVC}} \end{aligned}$$

Batch No. 700-224

$$\begin{aligned} \text{ROL} &= \frac{(8955) (1-0.635) (4,487) (0.131)}{(61,924)} \\ &= \frac{31 \text{ lbs. VCM}}{\text{MM Lbs. PVC}} \end{aligned}$$

conoco

Conoco Chemicals Company
Conoco Inc.
P. O. Box 91, New Highway 25
Aberdeen, Mississippi 39730
(601) 369-8111

April 7, 1982

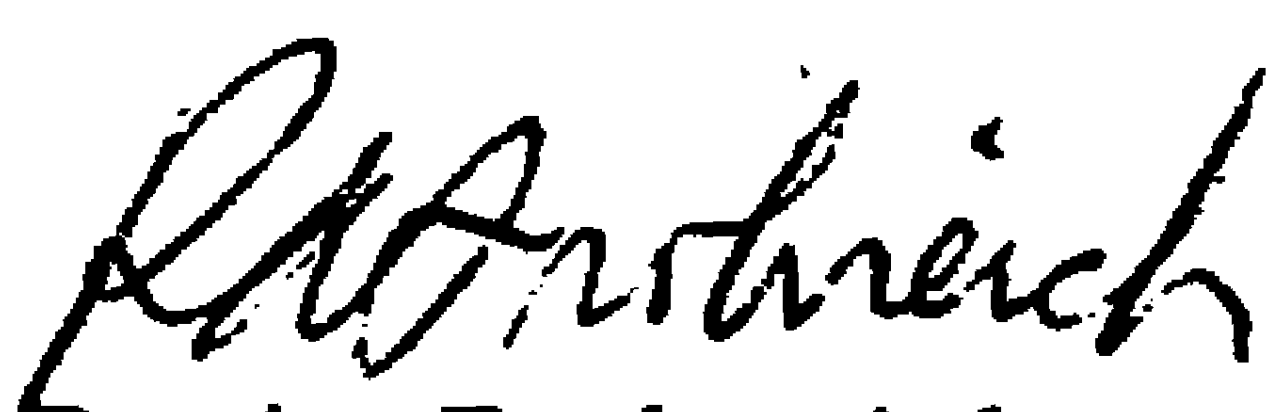
Mr. Jerry B. Banks
Mississippi Department of Natural Resources
Bureau of Pollution Control
P. O. Box 10385
Jackson, MS 39204

Dear Mr. Banks:

In accordance with Section 61.67 of the National Emission Standards for Hazardous Air Pollutants, Conoco Chemicals Company, a division of Conoco Inc. is submitting the attached Emission Test Report for the newly constructed polymerization reactor D-745, at its PVC production facility located in Aberdeen, Mississippi. The new reactor was brought on-line on December 18, 1981.

The emission test was conducted on March 9 and 10, at which time you observed one of the test runs.

Please note that we regard all information contained in this report confidential. If you have any questions concerning this report, please contact me or Dave Mahler at (601) 369-8111.


R. A. Frohreich
Chief Process Engineer

tap

Attachments

bcc: JF, CRM, SJV, PEM, DLM, DSC, JCL

VAB.0001115753