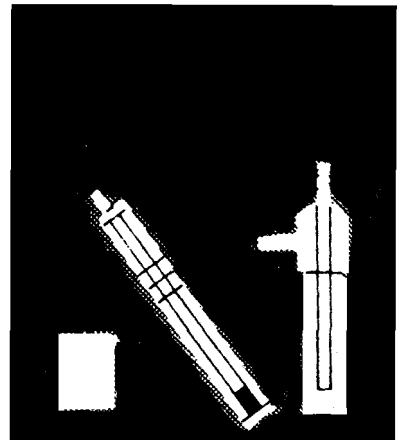

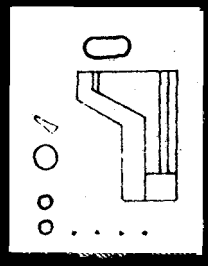

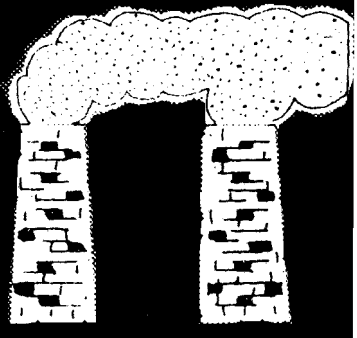

AIR POLLUTION EMISSION TEST

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Air and Waste Management
Office of Air Quality Planning and Standards
Emission Measurement Branch
Research Triangle Park, North Carolina

SOURCE TEST TRICHLOROETHYLENE DEGREASER ADSORBER

at

The Vic Manufacturing Plant
Minneapolis, Minnesota

by

George W. Scheil
Midwest Research Institute

EPA Project Report No. 76-DEG-1

FINAL REPORT

EPA Contract No. 68-02-1403, Task No. 16
MRI Project No. 3927-C(16)

For

Environmental Protection Agency
Research Triangle Park
North Carolina 27711

Attn: Mr. William Grimley

PREFACE

This work reported herein was conducted by Midwest Research Institute (MRI) under the Environmental Protection Agency (EPA) Contract No. 68-02-1403, Task No. 16.

The project was under the technical supervision of Mr. Paul C. Constant, Jr., Head, Environmental Measurements Section of the Physical Sciences Division of MRI. Dr. George W. Scheil served as project leader and was assisted by Messrs. R. G. Cobb and Bruce DaRos. Messrs. William Grimley and John Bollinger of EPA assisted with the field activities. Mr. Grimley, EPA Project Officer, coordinated activities and Mr. Bollinger, EPA Project Engineer, collected process data.

MIDWEST RESEARCH INSTITUTE

Paul C. Constant, Jr.
Program Manager

Approved:

L. J. Shannon, Assistant Director
Physical Sciences Division

February 11, 1976

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I. INTRODUCTION

This report discusses testing of a carbon adsorption unit used to control vapor emissions from an open top vapor degreaser.* The purpose of this test was to measure the adsorber bed efficiency, defined as the percentage of solvent that is adsorbed from the inlet gas stream. The unit tested was a Model 572, automatic, double-bed, carbon adsorption system made by Vic Manufacturing Company of Minneapolis, Minnesota. The adsorber is installed at the Vic Manufacturing plant and has been used to control trichloroethylene emissions from a Baron-Blakeslee Model D95P Degreaser for the past 8 years. Vapor emissions from the degreaser pit are collected by pulling air through lip vents around the top edge of the pit, filtering out particulates in a dust bag and then adsorbing the trichloroethylene vapors on activated charcoal beds. The solvent is then periodically steam-stripped from the beds and reused.

Tests were performed by Midwest Research Institute from September 9 to 11, 1975. The tests included one full cycle of the system from regeneration until breakthrough occurred on September 10. A shorter cycle was then tested on September 11. Semicontinuous measurements for total hydrocarbons were made with a flame ionization detector (FID) on the inlet and outlet ducts of the carbon adsorber. Several integrated gas-bag samples were also obtained at both the inlet and outlet ducts. These samples were analyzed for trichloroethylene by a gas chromatograph equipped with an FID. The identity of the trichloroethylene peak was confirmed by mass spectral analysis and a tentative identification made of two minor components in the gas stream.

II. SUMMARY AND DISCUSSION OF RESULTS

The inlet concentration of trichloroethylene to the carbon bed varied over two orders of magnitude during the test. The outlet concentration showed a very smooth gradual increase which appears to be related only to the total amount of trichloroethylene adsorbed on the bed. Bed saturation occurred at approximately 15 hr operating time--roughly twice the usual interval between regenerations at this plant. During the first 8 hr of operation after regeneration, the average bed efficiency met the design limit of 95% removal of trichloroethylene.

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* These data are intended to be used for the development of a standard of performance for new solvent degreaser operations.

Equipment setup was completed on September 8, 1975. Approximately 1 hr before the end of the shift, both adsorption beds were back on line after being recycled. At 0820 on September 9, 1 hr after degreaser startup, and two running hours after bed desorption, the total hydrocarbon (THC) flame ionization analyzer was brought on-line; therefore, the first data points occur at 2 hr from the start of the cycle. By 0914, the velocity traverse was completed and the first set of integrated gas sampling on both inlet and outlet was started. On the first day the calibration gases were not made until the afternoon so the THC analyzer was calibrated only at the end of the day. On the following days the THC analyzer was calibrated in the morning and in the afternoon. The gas chromatograph calibration was always made immediately before or in the middle of the sample analyses.

Table I shows the data from the gas chromatography analyses. Areas were measured by multiplying the height times the peak width at one-half the height. Due to the instability of the gas chromatograph under field conditions, only the area measurements were used in calculating concentrations although the peak heights are also reported. Figure 1 shows a sample chromatogram. The peak at retention time (T_r) = 3.7 min is the trichloroethylene peak. Copies of the chromatograms are in Appendix A, and the results of a mass spectra (MS) analysis of an inlet sample, which was brought back to MRI after testing, appears in Appendix B. The identification of the trichloroethylene peak was confirmed with MS with no detectable interferences. The peak at T_r = 3.0 min was identified as 1,1,1 trichloroethane and the peak at T_r = 6.4 min was 1,1,2,2 tetrachloroethane. The peak at T_r = 1.3 min is the air peak and most of the response probably comes from methane, although no confirmation of this was possible. The other peaks were too small to identify.

The peak areas were corrected back to a common sample loop temperature of 50° C, since the gas chromatograph responds to the mass of the sample and not the concentration. Sample concentrations were obtained from a linear least square fit of each day's calibration runs.

Table II shows the results of the THC analysis and Figure 2 shows the THC results in graph form. Figure 3 shows the THC data reported as bed efficiency. Due to the large quantity of data generated by the THC analyzer only the 1/2-hr averages are reported here. The morning and evening calibrations showed about a 10% drift over 7 to 8 hr. All calibration points for each day were used to generate a linear least square fit, and the results of this calculation were used to determine the concentrations at the inlet and outlet. The sharp decrease at 15 hr corresponds to the regeneration of the carbon beds from 1300 to 1500 on September 10.

TABLE I

GAS CHROMATOGRAPHY ANALYSIS DATA

Date	Sample Standard	Sample Loop Temperature (°C)	Peak Height	Peak Height[a/] (Corrected)	Peak Area	Peak Area[a/] (Corrected)	Trichloroethylene Measured (ppm)
9/9	46 ppm	62	800	830	3,440	3,570	-
	46 ppm	58	863	880	3,800	3,890	-
	46 ppm	55	902	920	4,060	4,120	-
	46 ppm	53	968	980	4,260	4,300	-
	9.0 ppm	54	196	198	920	930	-
	9.0 ppm	54	210	212	880	890	-
	9.0 ppm	52	202	203	910	915	-
	9.0 ppm	51	195	195	940	940	-
	4.6 ppm	51	121	121	545	545	-
	4.6 ppm	51	124	124	570	570	-
	4.6 ppm	51	134	134	535	535	-
	4.6 ppm	51	125	125	525	525	-
	Inlet 2	51	1,660	1,660	7,450	7,450	90
	Inlet 2	51	1,710	1,710	7,360	7,360	89
	Outlet 3	51	19	19	78	78	0.7
	Outlet 3	51	18	18	77	77	0.7
	Inlet 1	51	1,820	1,820	8,010	8,010	97
	Inlet 1	51	1,880	1,880	8,460	8,460	102
9/10	Outlet 3	50	14.5	14.5	55	55	0.8
	Outlet 3	50	15.0	15.0	52	52	0.8
	Inlet 4	51	1,700	1,700	7,140	7,140	64
	Inlet 4	51	1,760	1,760	7,200	7,200	65
	Outlet 5	51	148	148	620	620	5.9
	Outlet 5	51	152	152	610	610	5.8
	4.6 ppm	51	125	125	475	475	-
	4.6 ppm	51	110	110	440	440	-
	4.6 ppm	51	112	112	448	448	-
	8.9 ppm	51	234	234	980	980	-
	8.9 ppm	51	228	228	1,010	1,010	-
	43 ppm	50	1,070	1,070	4,800	4,800	-
	43 ppm	50	1,060	1,060	4,750	4,750	-
	Outlet 6	51	294	294	1,260	1,260	11.6
	Outlet 6	51	313	313	1,410	1,410	12.9
	Outlet 6	51	312	312	1,340	1,340	12.3
	Outlet 9	53	678	685	2,850	2,880	26.1
	Outlet 9	53	677	684	2,840	2,870	26.0
	Outlet 8	53	670	677	3,015	3,040	27.5
	Outlet 8	53	675	683	3,040	3,070	27.7
	Inlet 7	51	2,380	2,380	10,900	10,900	98
	Inlet 7	51	2,400	2,400	11,000	11,000	99
	Inlet 7	50	2,435	2,435	9,980	9,980	89
9/11	4.8 ppm	46	120	119	600	590	-
	4.8 ppm	46	124	123	620	610	-
	45 ppm	46	1,380	1,370	5,790	5,720	-
	45 ppm	46	1,370	1,360	5,750	5,680	-
	9.4 ppm	46	286	282	1,170	1,160	-
	9.4 ppm	46	270	266	1,160	1,150	-
	9.4 ppm	46	278	274	1,140	1,130	-
	Inlet 10	48	1,640	1,630	6,560	6,520	51
	Inlet 10	48	1,570	1,560	6,420	6,380	50
	Inlet 10	48	1,650	1,640	6,590	6,550	52
	Outlet 11	48	81	80	331	329	2.9
	Outlet 11	48	81	80	332	330	2.9
	Outlet 11	48	81	80	341	339	2.9
	Outlet 12	48	80	79	344	342	3.0
	Outlet 12	48	78	77	329	327	2.8
	Outlet 12	48	91	90	365	363	3.1

a/ Peak height/area corrected to sample loop temperature of 50 C.

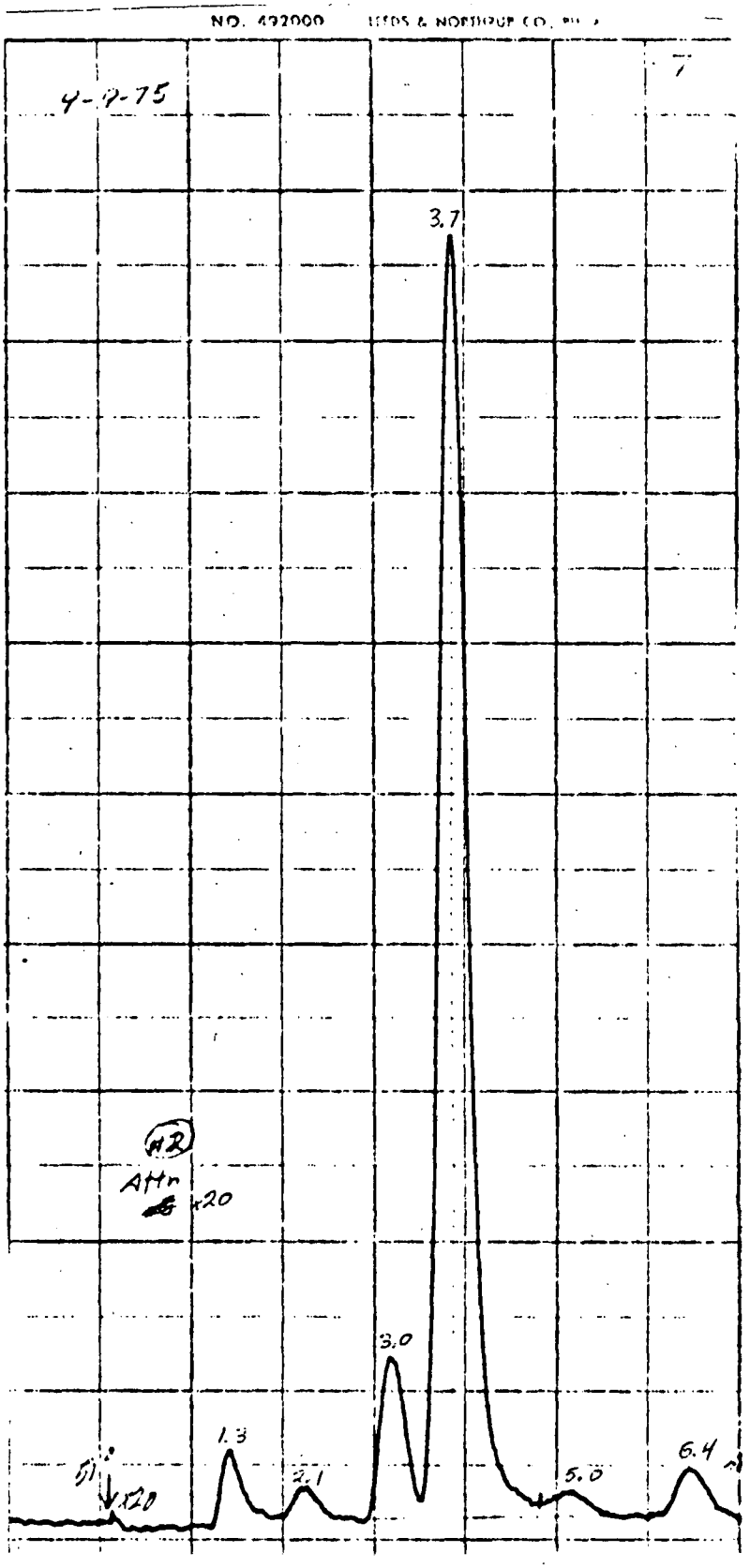


Figure 1 - Sample Chromatogram From an Inlet Sample

TABLE II

HALF-HOUR AVERAGES OF THC ANALYZER DATA

Date	Time	Total Hydrocarbons as Trichloroethylene (ppm) Inlet	Outlet
9/9	0820-0900	83	4.5
	0900-0930	192	4.6
	0930-1000	89	3.9
	1000-1030	144	4.0
	1030-1100	140	4.1
	1100-1130	106	3.6
	1130-1200	99	3.0
	1200-1230	70	2.8
	1230-1300	40	2.9
	1300-1330	133	4.4
	1330-1400	55	4.2
	1400-1430	58	5.7
	1430-1500	115	6.2
	1500-1528	100	8.0
9/10	0739-0815	37	6.7
	0839-0900	117	11.6
	0900-0930	120	14.2
	0930-1000	88	17.8
	1000-1030	111	22.2
	1030-1100	54	27.6
	1100-1130	95	34
	1130-1200	134	41
	1200-1230	109	49
	1230-1300	124	55
	1300-1330	127	86
	1330-1400	156	123
	1400-1430	128	63
	1430-1500	110	6.9
	1500-1527	212	12.2
9/11	0732-0800	56	7.0
	0820-0900	136	7.9
	0900-0930	78	7.8
	0930-1000	46	7.6
	1000-1030	54	7.5
	1030-1100	39	7.3
	1100-1130	62	7.5
	1130-1200	40	7.6
	1200-1230	46	7.3
	1230-1310	51	7.8

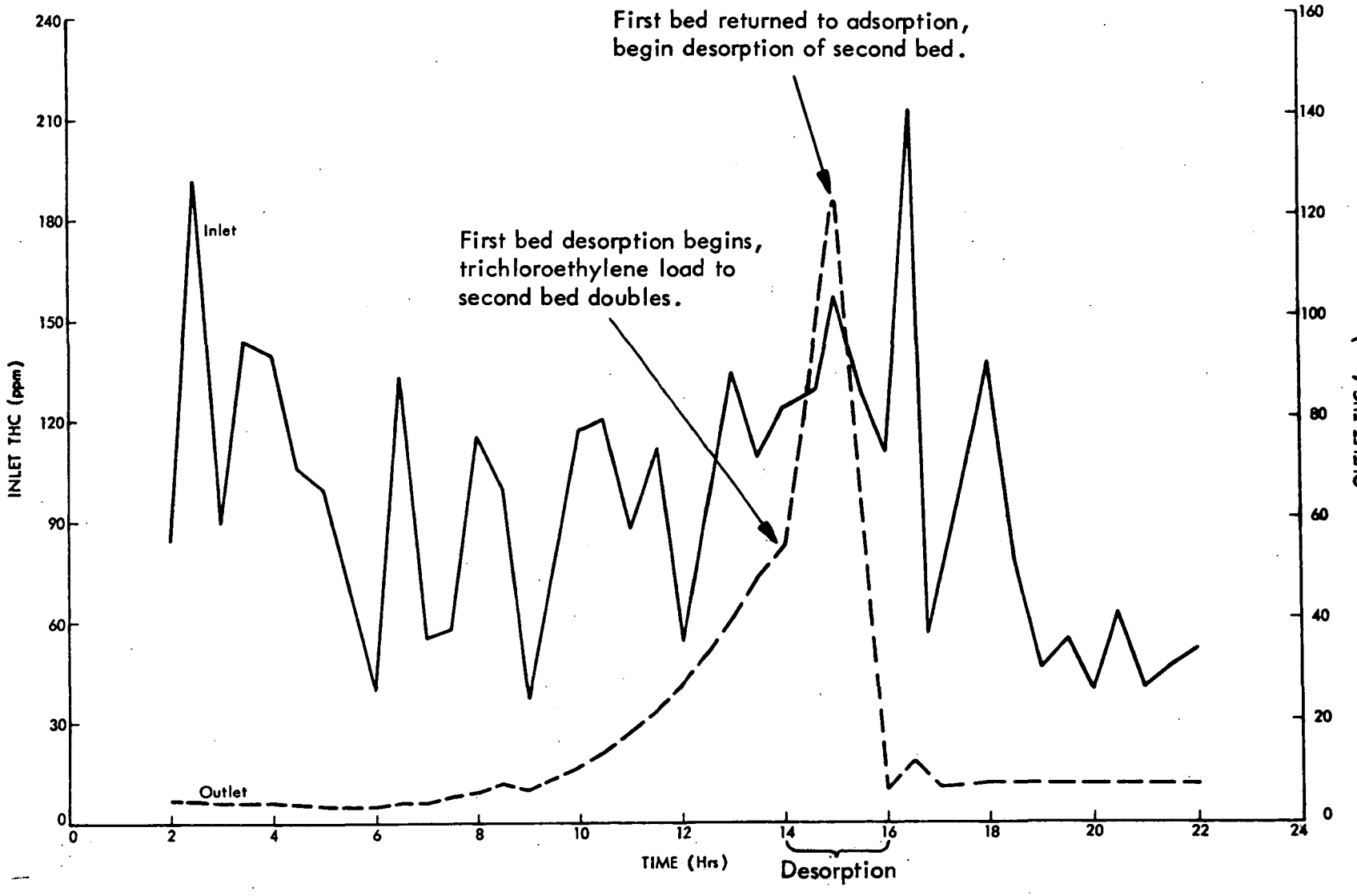


Figure 2 - Graph of Inlet and Outlet THC Concentrations During Testing

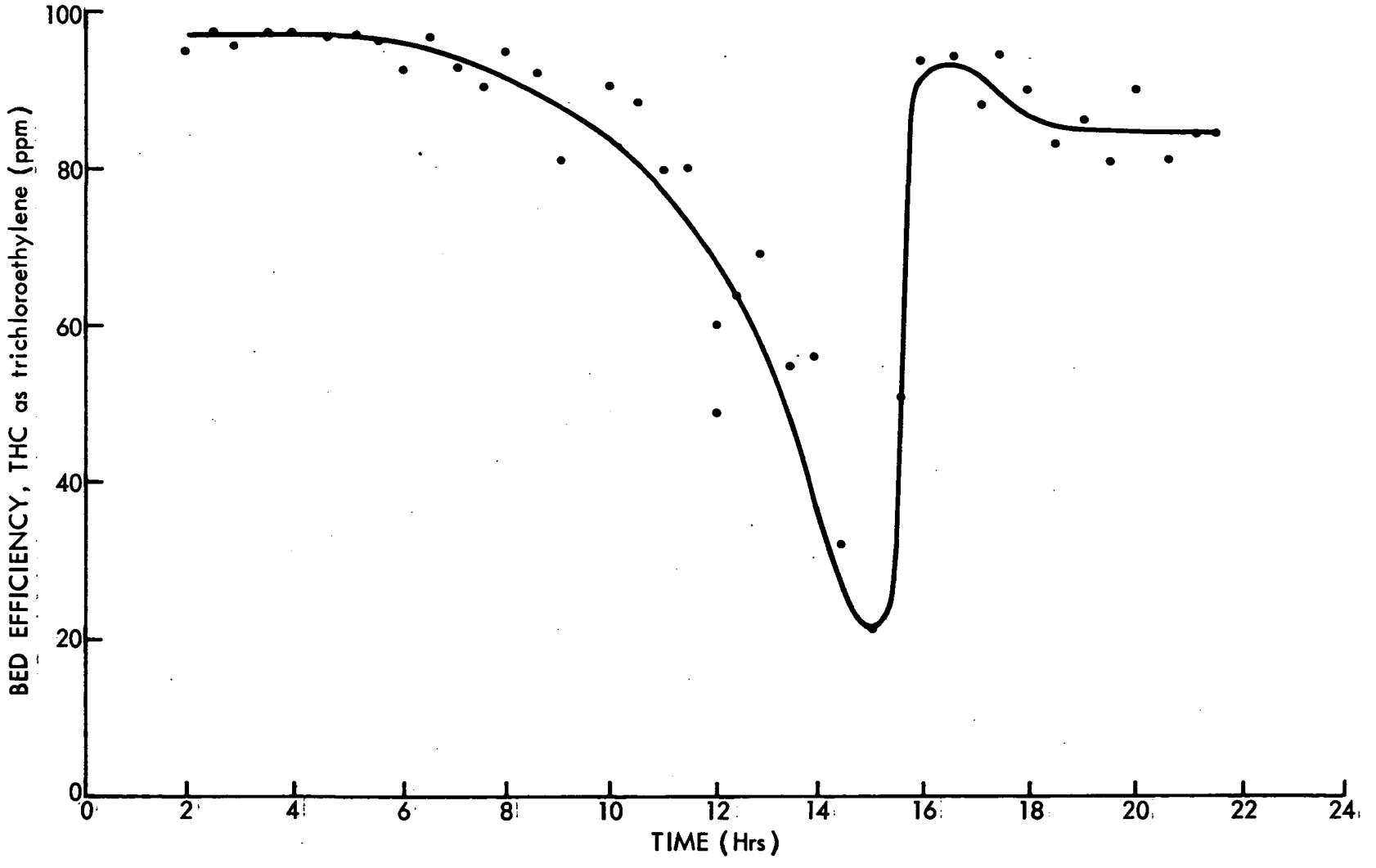


Figure 3 - Graph of Carbon Adsorption Bed Efficiency

Table III summarizes the integrated gas sample results and compares the approximate THC response found on the integrated samples to the average level measured by the THC analyzer. The THC response of the chromatograms was determined by measuring the total area of the chromatogram, multiplying it by the trichloroethylene concentration, and then dividing the result by the area of the trichloroethylene peak. The agreement of the two methods is excellent considering instrument drift and the approximation for the THC of the integrated samples.

The data from the daily pitot traverses are given in Table IV. Although the pitot readings were not made at the proper points prescribed in EPA Method 2,[1/] the sample point was more than 20 diameters from the nearest flow disturbance, which allows a reasonably accurate velocity determination since the velocity profile was nearly ideal in the duct. Details of the calculations are given in Appendix C.

Assuming an average flow of 5,100 scfm, time to breakthrough of 15 hr, and a density of 5.85 g/liter for trichloroethylene vapor, a total of 80 kg or 176 lb of solvent vapor went into the absorber. At the end of the first cycle, 267 lb were recovered from the holding tank. An explanation of this discrepancy is given in Section III.

A value for the bed efficiency of this unit is nearly impossible to determine due to the large inlet level variations which depend very strongly on the amount of degreasing being done, the speed of raising work out of the pit and other variables involved in the degreasing operation. Since the average inlet level for the first cycle was 104 ppm, an average bed efficiency of 95 to 97% was observed for the first 8 hr of operation. For the second cycle, the efficiency was lower, probably due to incomplete stripping after the bed reached saturation.

From the behavior of the bed during these tests, the outlet concentration is independent of the inlet concentration, but instead is a function of the quantity of solvent present in the bed. The outlet level rises on a smooth curve independent of changes in the inlet levels. Once the entire bed reaches saturation, the outlet level increases very rapidly. During the first 50% of the time to breakthrough, the outlet level is at a low level and nearly constant.

While trichloroethylene may represent 80% of the inlet THC response, it was sometimes only 20% on the outlet. Thus, the use of a THC analyzer may give inflated readings if used to measure trichloroethylene in such a unit.

1/ Federal Register, Vol. 36, No. 247, December 23, 1971.

TABLE III

SUMMARY OF ANALYTICAL RESULTS

Sample No.	Date/Time	Trichloroethylene Found (ppm)	Approximate THC Content of Integrated Bag Sample (ppm)	Time Average of THC Analyzer During Bag Sample Period (ppm)
Inlet 1	9-9/0914-1214	100	125	120
Inlet 2	9-9/0914-1214	90	110	120
Outlet 3	9-9/0914-1214	0.7	3.5	3.7
Inlet 4	9-10/0800-1000	64	80	90
Outlet 5	9-10/0800-1000	5.8	10	12.5
Outlet 6	9-10/0949-1049	12	20	22
Inlet 7	9-10/1100-1300	98	120	115
Outlet 8	9-10/1100-1300	28	45	45
Outlet 9[a/]	9-10/1100-1300	26	45	45
Inlet 10	9-11/0825-1125	51	65	70
Outlet 11	9-11/0825-1125	2.9	6.0	7.6
Outlet 12	9-11/0825-1125	3.0	6.0	7.6

a/ Leak in the outer container on this sample--very little actual flow until 1215.

TABLE IV

PITOT DATA AND CALCULATED STACK FLOW RATES AT INLET

Pitot Traverse and Static Pressure in Inches of Water

Date	Test No.	Inches from Port 3	6	9[a]	12	15	Static Pressure	Barometric Pressure (mm Hg)	Temperature (°C)	Percent H_2O (Assumed)	Stack Velocity (ft/min)	Flow Rate (ft^3/min)	Flow Rate Standard Conditions (ft^3/min)
9/9	1	1.00	0.80	0.83	0.81	0.82	6.9	746	21	1	3,180	5,520	5,370
9/10	2	0.70	0.69	0.81	0.78	0.76	6.5	740	21	1	2,900	5,120	4,860
9/11	3	0.65	0.70	0.82	0.84	0.79	6.6	742	21	1	2,880	5,090	4,840

a/ Sampling location.

The major problem encountered during testing was serious leaks in the outer rigid container on the integrated gas trains. After functioning properly on the first day, two of the four containers developed leaks on the second day. Three partial sets of bag samples were collected on the second day while repairs were being made. On the third day, no sampling problems were encountered. Since the rotameter measures only the air going out of the container, it cannot show if air is being pulled into the outer case instead of sample gas going into the bag.

III. PROCESS DESCRIPTION AND OPERATION

Figure 4 is a diagram of the degreaser and the carbon adsorption unit at the plant. The trichloroethylene solvent is boiled at the bottom of the pit. The vapors then condense on the metal components being cleaned. Large objects are lowered into the pit by hoist. Smaller objects are loaded onto a pallet or metal cage which is then lowered into the pit. The work being cleaned should be left in the pit until condensation ceases to be formed on the work. Most of the solvent vapor that escapes beyond the cooling coils is then pulled into the lip vents around the top of the pit. The gases go underground to a dust bag behind the degreaser and through an overhead duct to the carbon adsorption unit. After passing through the carbon beds, the airstream is vented to the roof.

The plant normally operates on a 0730-1630 weekday schedule. When the degreaser is not in use, the adsorption unit and draft fan are shut down and a lid is put on the degreaser pit. The adsorption unit operates with a nominal flow of 5,000 cfm and both beds in parallel. In normal operation for this plant, the beds are recycled once per 8-hr day.[1/] The first bed is usually steam-stripped for about 1 hr, then the second bed is similarly regenerated. With one bed on recycle, the air flow is cut to 3,000 cfm. The recovered trichloroethylene is condensed out, separated from the water condensate, and then stored in a holding tank for later return to the degreaser. The unit design adsorption rate is 225 lb/hr of trichloroethylene.

1/ However, to develop breakthrough data, the beds were not desorbed until 14 operating hours had elapsed.

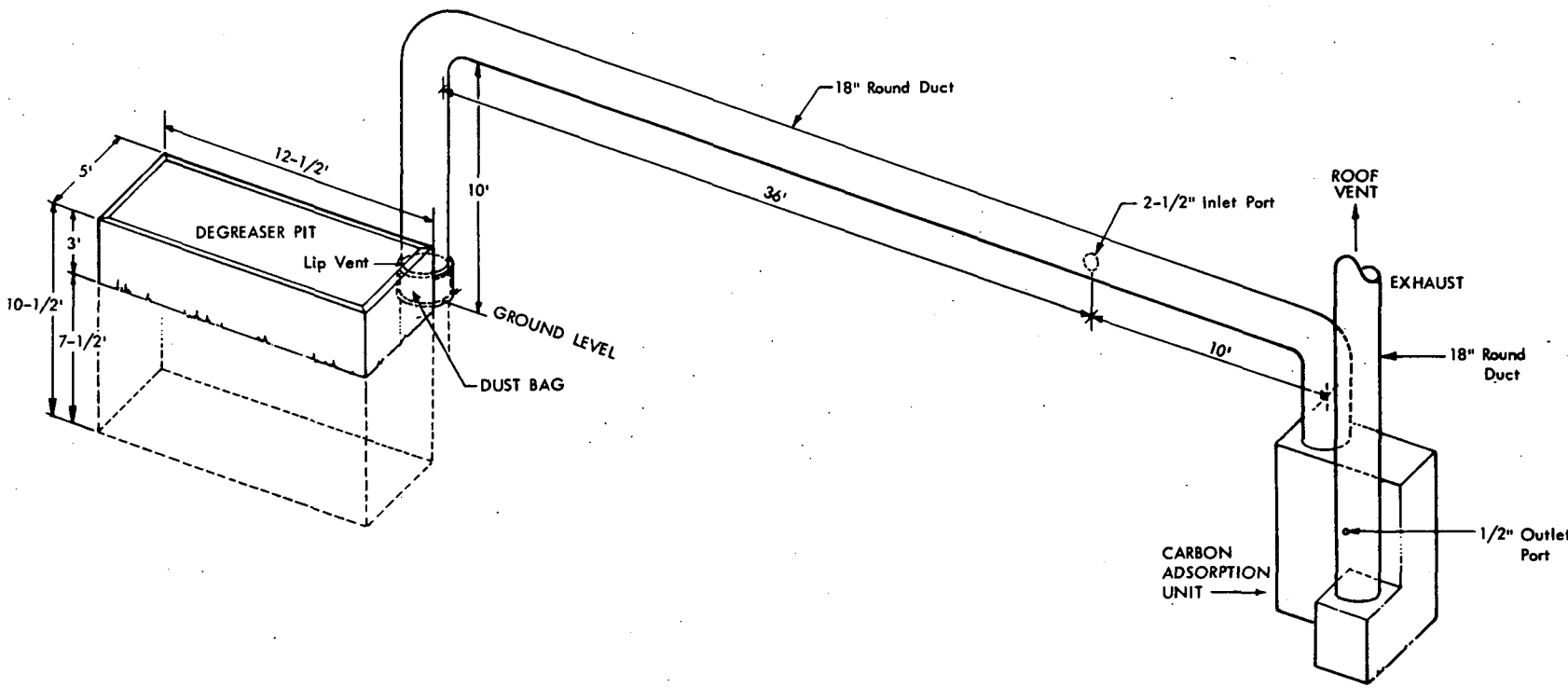


Figure 4 - Diagram of Degreaser, Carbon Adsorber, Ductwork, and Sample Points

The equipment specifications for the carbon bed and degreaser are summarized below:

Equipment Specifications

For Carbon Adsorption System

Beds: Two
Depth: 20-21 in. carbon
1,500 lb carbon per bed
Diameter - 72 in.
Type activated carbon: Union Carbide JXC4 by 10
Age carbon: installed 1968--about 7 years old

Model: No. 572 Vic
Fan: 20 h.p.
Duct diameter: 18 in.
Steam pressure: 2-4 psig
Scheduling: Regenerated each bed once per 8-hr day usually 12:30 to 1:30 and 1:45 to 2:45 p.m.
Fan: 7:15 a.m. to 3:30 p.m.

For Degreaser

Type: Open top vapor degreaser
Model: Baron Blakeslee Model No. D95P
Opening: 5 ft by 12 ft 6 in. - freeboard
4 ft by 12 ft - vapor chamber
Freeboard height: 34 in. when at equilibrium (i.e., solvent vapor line to lip)
Heating: steam at about 12 psig
Cooling coils: seven levels; condensing on second to bottom coil; uses tap water
Solvent: Trichloroethylene

Table V lists the workload of the degreaser during the test as recorded by the EPA project engineer.

Solvent Recovery Data

The data of the solvent recovered from the carbon adsorber are too inaccurate to use. A malfunction of the carbon adsorber condenser and water separator caused the inaccuracy. The purpose of the data was to provide a check for the concentration measurements.

TABLE V

WORKLOAD DATA

12/9/75	Object Cleaned	Unit Weight (lb)	Times Cleaned	Total Weight (lb)
Tuesday morning	Large rack load	1,025	4	4,100
	Smaller rack load	965	5	4,825
Tuesday afternoon	Square tank	250	2	500
	Rack load	965	2	1,930
Wednesday morning[a/]	Rack load	965	3	7,720
	Cylinder wrap	900	1	900
	Heads	1,500	2	3,000
	Long cylinder	1,000	1	1,000
			Total	23,975
				≈ 12 tons

a/ Workload data was recorded until desorption began. That is time 200 to 1400.

The solvent recovered was measured as 267 lb or 24 gal., but this is based upon the incorrect assumption that the solvent in the water separator was all boiled away. One to 3 days before the test began a malfunction occurred when the cooling water from the condenser cut off. Consequently, the steam passed directly into the water separator and boiled off most of the solvent. Vic personnel made no mention of this upset until John Bollinger inquired as to why the recovery system failed to produce solvent after the September 10 desorption. Although the condenser cooling system had been corrected, most of the solvent had been boiled out of the water separator, thus upsetting the measurements. The water separator normally contains a constant volume of solvent, so that the runoff after each desorption equals the solvent recovered. An undetermined volume in the separator was boiled off during the malfunction, so that the amount of desorbed solvent could not be quantified. An approximation of the solvent desorbed comes to 176 lb as derived from average flow and concentration data. The value of 267 is unreasonably high in comparison, and thus the assumption that the water separator was empty before the measured desorption is probably incorrect.

IV. SAMPLING LOCATIONS

The sampling points are shown in Figure 4. Since the adsorption unit is sealed (no flow can be added or lost between inlet and outlet), pitot traverses were run only on the inlet duct. After the traverse was run each morning, the pitot was fixed at the center of the duct and both inlet and outlet integrated samples were drawn proportional to this pitot reading. At both the inlet and the outlet a 1/4-in. O.D. stainless steel probe was mounted with the end of the tube at the center of the duct. A glass wool plug in the end of the probe kept out particulates. The 1/4-in. O.D. stainless steel sample lines were wrapped with heating tape and heated slightly above ambient temperature to prevent condensation. The inlet and outlet lines were brought to the THC analyzer which was located on a table between the sample points. At the analyzer, each sample stream was then split with lines going into the analyzer and to the integrated gas bags.

V. SAMPLING AND ANALYTICAL PROCEDURES

Integrated gas sampling and analysis was done according to the July 18, 1975 draft procedure for Method 106 - Determination of Vinyl Chloride from Stationary Sources. A copy of the procedure is given in Appendix D. For this test the following changes and modifications were made in the procedure:

1. The Teflon tubing used to connect the Tedlar bags to the various other parts of the apparatus was not changed for each sample due to the low reactivity of trichloroethylene.

2. Since an automatic sample valve was not available, a manually operated valve with a 5-ml stainless steel loop was used. The sample loop temperature was approximately 50°C.

3. The column used was a 3 m x 1/8 in. O.D. stainless steel column packed with 20% SP-2100/0.1% Carbowax 1500 on 100/120 mesh Supelcoport. The column was operated at a flow rate of 20 ml/min and 120°C.

4. The calibration procedure was modified since trichloroethylene boils above room temperature. A septum vial containing a small amount of liquid trichloroethylene was immersed in a water bath at room temperature. A gas tight syringe was then flushed twice from the vial headspace and then filled to maximum. After briefly flaming the needle to expel any trace of liquid, the plunger was set to the proper volume and gas was injected into the Tedlar bag in the normal manner.

The primary danger in using this method is that traces of liquid might be injected into the bag with the saturated vapor. If care is taken to ensure that the needle does not contact the liquid phase, the only source of liquid is the thin film of liquid on the inside surface of the septum. By briefly heating the needle before setting the plunger to the proper index mark, any liquid within the needle or on its outer surface will be boiled off. (Since the syringe is already filled with vapor, the vapor from the boiling trichloroethylene will be expelled out the end of the needle.)

The temperature of the water bath was measured with an accurate thermometer and the barometric pressure was also measured. With this information the vapor pressure of trichloroethylene can be calculated:[1/]

$$\log 10\ P_T = 7.02808 - \frac{1315.04}{230 + T\ (^{\circ}C)}$$

The final trichloroethylene concentration (C) is then:

$$C = \frac{P_T \cdot Vi}{P \cdot V \cdot 1{,}000}$$

1/ Lange, Handbook of Chemistry, 10th Edition, p. 1438 (1961).

P is the barometric pressure in mm Hg, Vi is the volume in milliliters of gas injected, and V is the volume in liters of nitrogen in the bag. The calibration data for this test are given in Table VI.

5. The flow rate into the Tedlar bags was not always 0.5 liters/min, but varied according to the sampling duration of each run. On the 9th the flow rate was initially 0.25 liters/min, on the 10th it was 0.5 liters/min, and on the 11th the flow rate was 0.4 liters/min. During all runs the pitot reading did not change enough to require a change in the sampling rate initially established.

6. When analyzing the calibration gases the bag was connected directly to the sample valve and compressed gently for 10 sec to purge the valve. Then the valve was actuated immediately, and a shut-off valve located on the sample inlet was closed until the next sample.

Total hydrocarbons were measured with a modified Beckman Model 6800 THC Analyzer. The instrument was modified so that the two sample streams could be alternately injected directly into the flame ionization detector. A 10-port pneumatic sample valve inside the instrument was connected as shown in Figure 5. The sample loops had 1-ml volumes. The carrier gas used was air. The vent connections went to a small vacuum pump via flow restrictors so that each sample line was being pulled through the valve by the pump. At a flow rate of ~ 100 ml/min the sample loops remain at ambient pressure. The instrument was calibrated each day using the same gas mixtures used to calibrate the gas chromatograph. The internal valve control timing was set to activate the valve to sample the first gas stream, then, 30 sec later, the valve deactivated to sample the second stream. At 60 sec the master timer is reset and begins a new cycle. Thus the recorder trace shows peaks every 30 sec with every other peak being on the same sample line. A sample of the chart is shown in Figure 6. The series of peaks which slowly increase from right to left represent the outlet concentration. The highly variable peaks in between are for the inlet. The analyzer also contains provisions for differing scale factors for the two peaks. In the example the inlet is operating at a x10 attenuation factor and the outlet at x1. The sudden increases in the inlet readings are caused by work being lowered or raised into the degreaser pit, which disturbs the air flow temporarily. Also, if the work is raised rapidly, some solvent evaporates after passing the cooling zone and the vapor is then drawn into the lip vent.

TABLE VI

CALIBRATION GAS DATA

Date	Nominal Concentration (ppm)	Rotameter Reading (mm)	Flow Rate (liter/min)	Time (min)	Volume Injected (ml)	P_T (mm Hg)	P (mm Hg)	N_2 Volume (liter)	Concentration (ppm)
9/9	5	141	.806	12.0	.5	66	746	9.672	4.6
	10	141	.806	10.0	.8	68	746	8.06	9.0
	50	141	.806	10.0	4.0	69	746	8.06	46
9/10	5	140	.800	12.0	.5	66	740	9.60	4.6
	10	140	.800	10.0	.8	66	740	8.00	8.9
	50	140	.800	10.33	4.0	66	740	8.26	43
9/11	5	139	.794	12.25	.5	69	742	9.73	4.8
	10	139	.794	10.0	.8	69	742	7.94	9.4
	50	139	.794	10.5	4.0	69	742	8.34	45

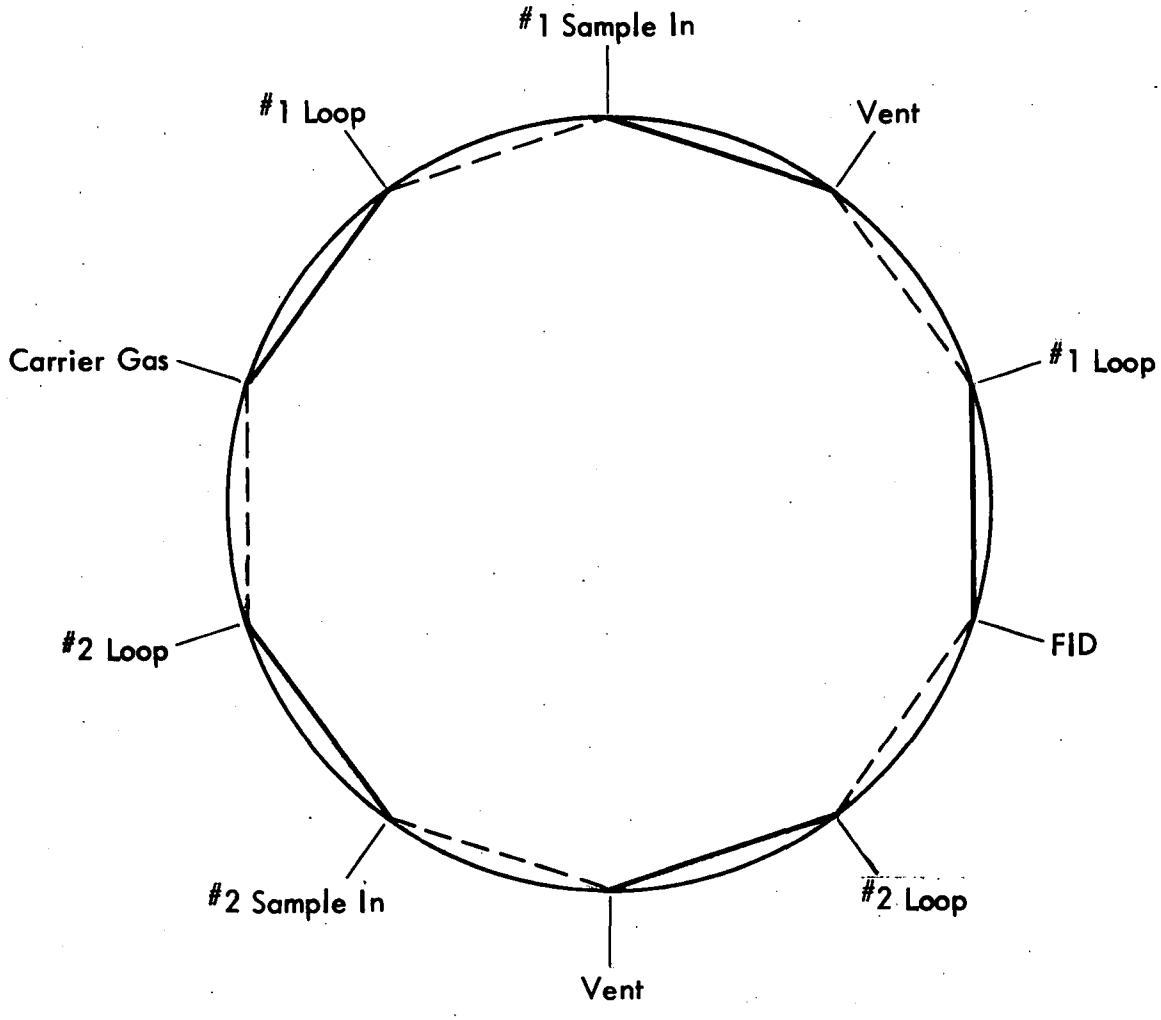


Figure 5 - Diagram of THC Valve Porting

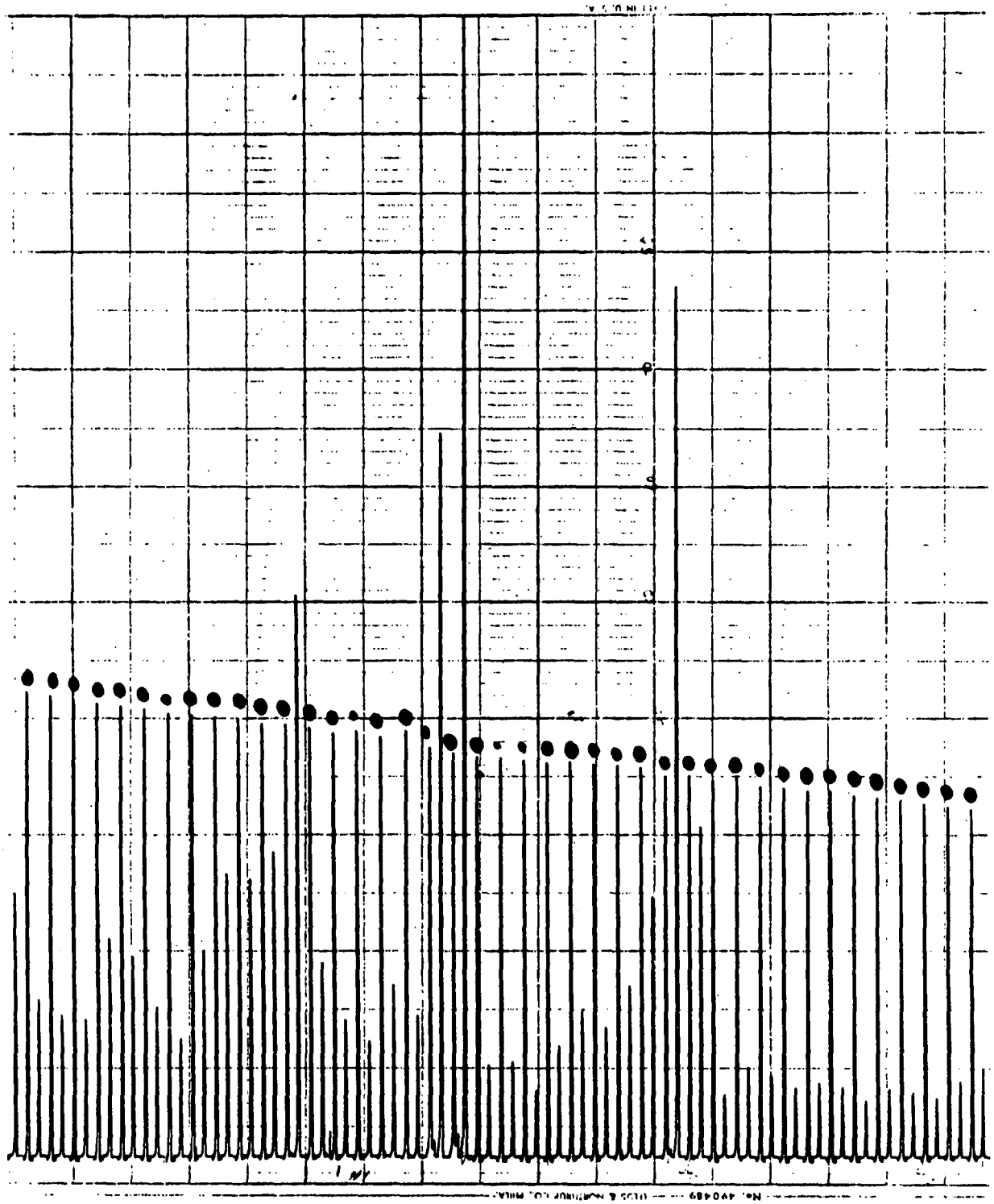
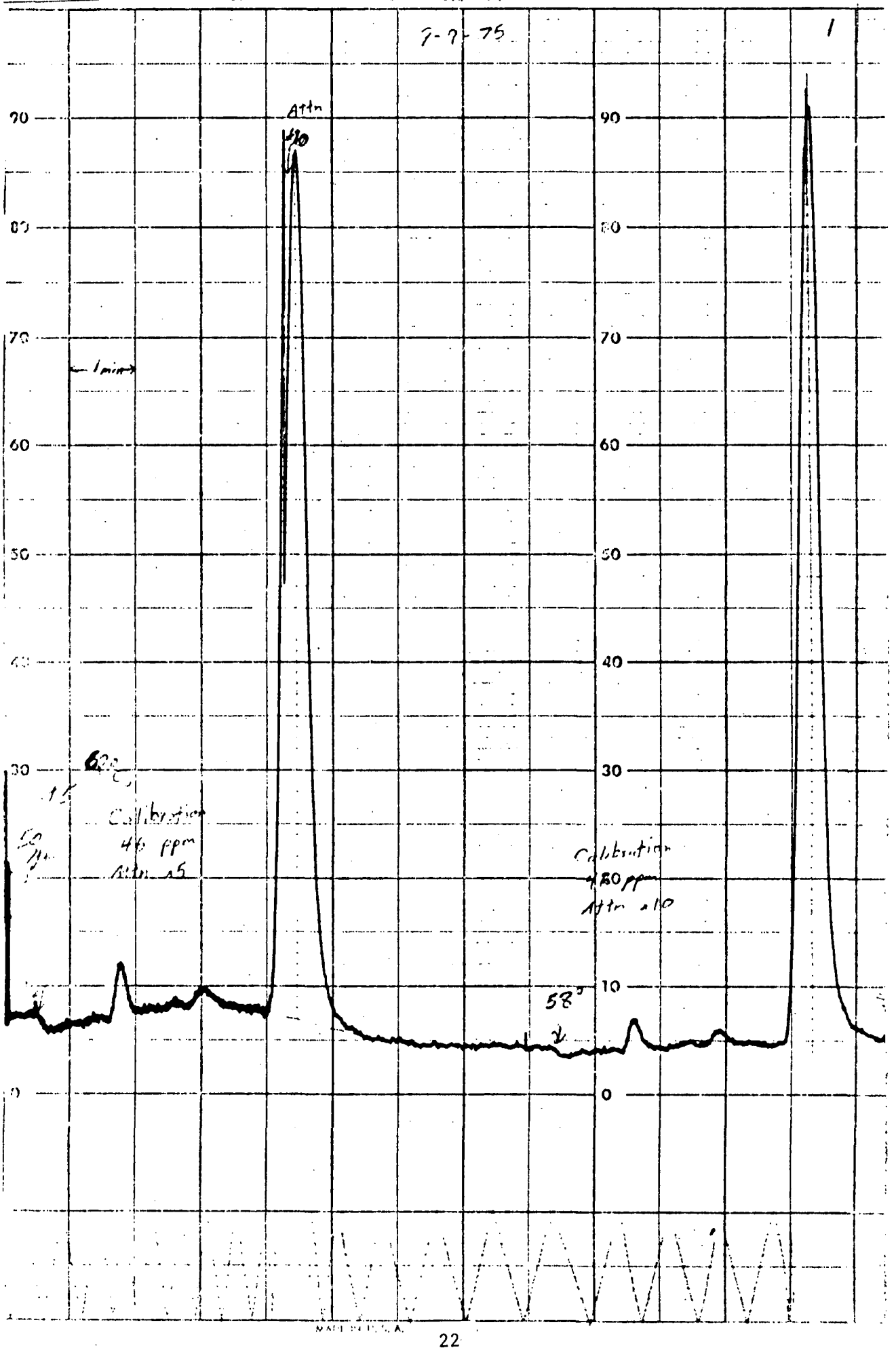


Figure 6 - Section of THC Recorder Chart
(chart reads from right to left)

APPENDIX A

GAS CHROMATOGRAMS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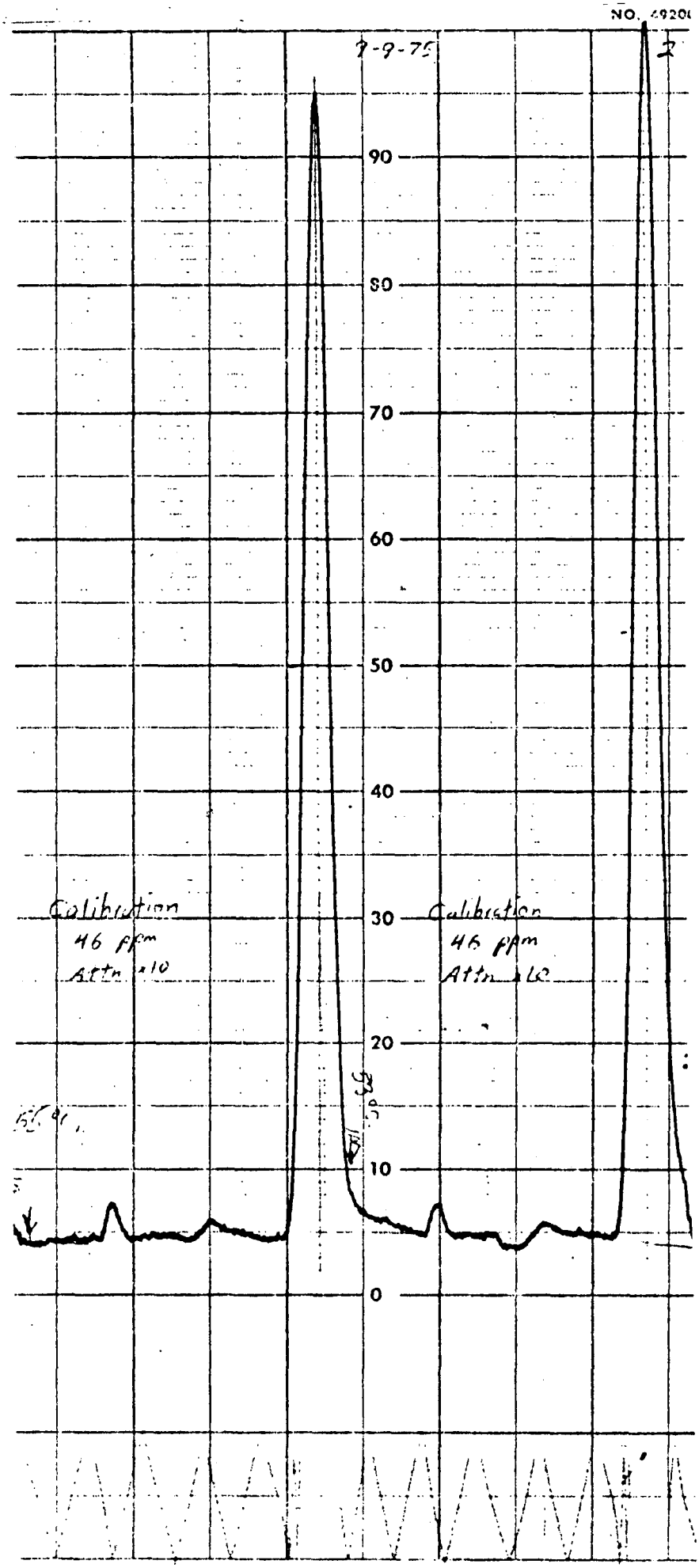
7-7-75
1
Attn
1/10
1 min
90
80
70
60
50
40
30
10
0
Calibration
Attn x5
Calibration
Attn x10
58°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80
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60
50
40
30
20
10
0
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46 ppm
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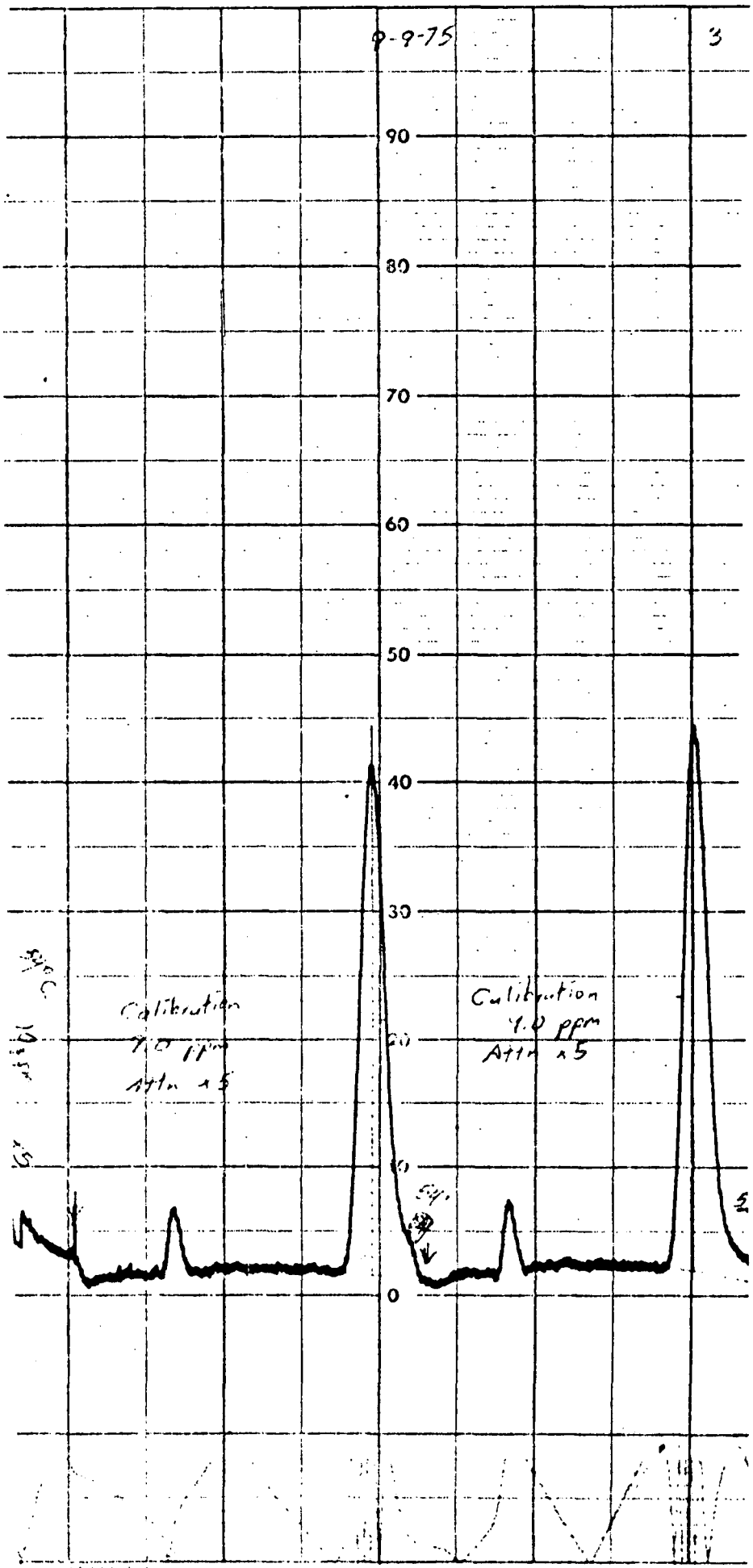
Calibration
46 ppm
Attn x10


9-9-75
3
90
80
70
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50
40
30
20
10
0
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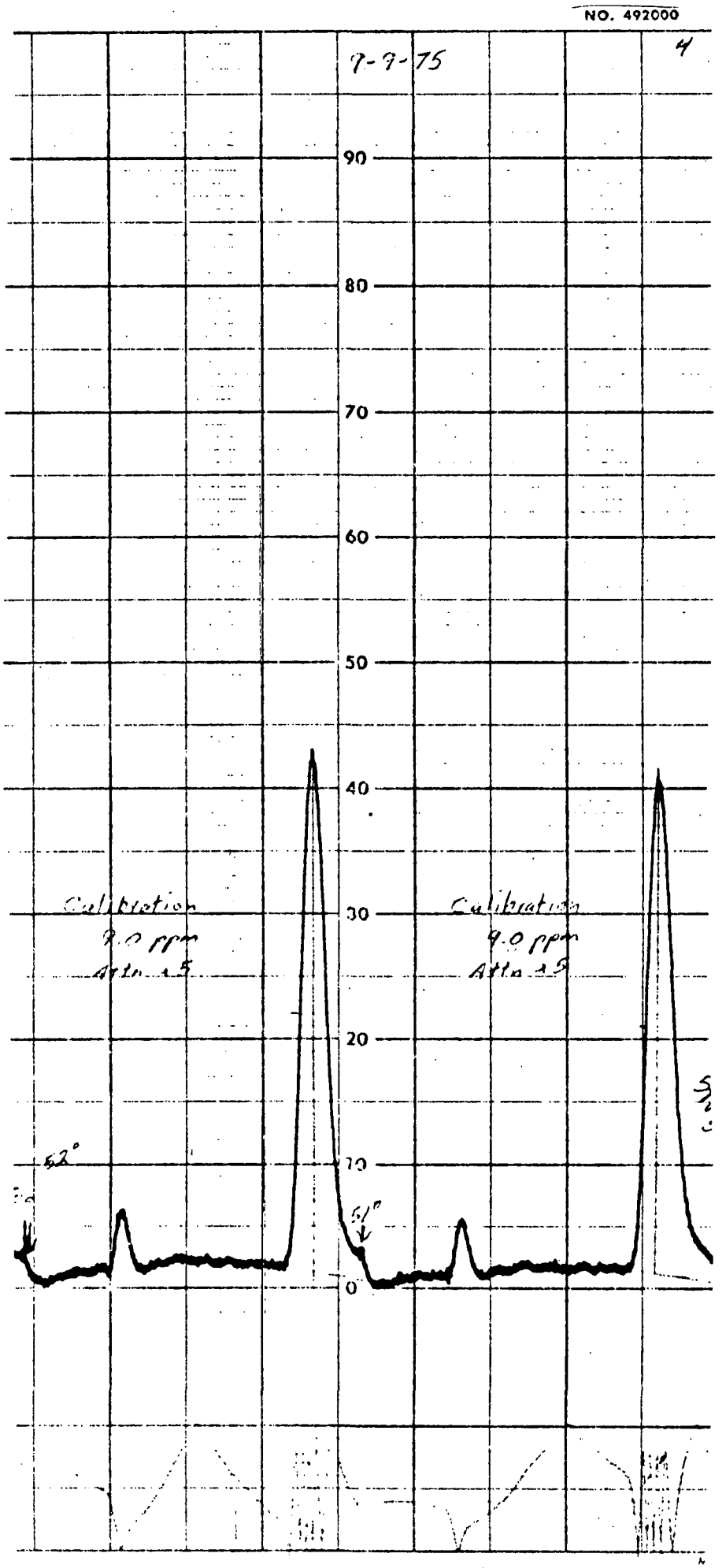
Attn x5
Calibration
1.0 ppm
Attn x5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7-9-75
4
90
80
70
60
50
40
30
20
10
0
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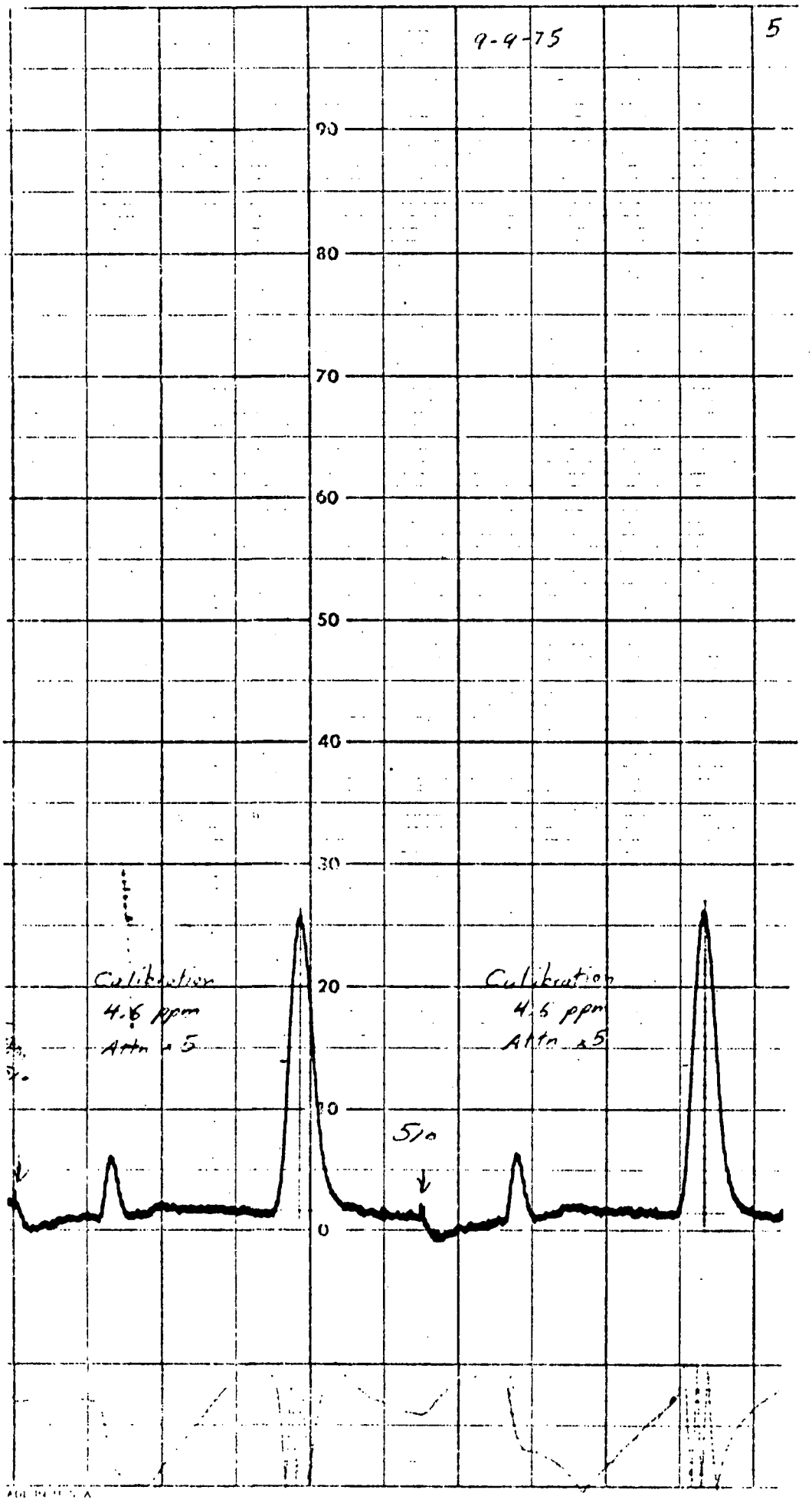
Atten 5
Calibration
9.0 ppm
Atten 5
52°
51°


LEEDS & NORTHRUP CO., PHILA.
9-9-75
5
90
80
70
60
50
40
30
20
10
0
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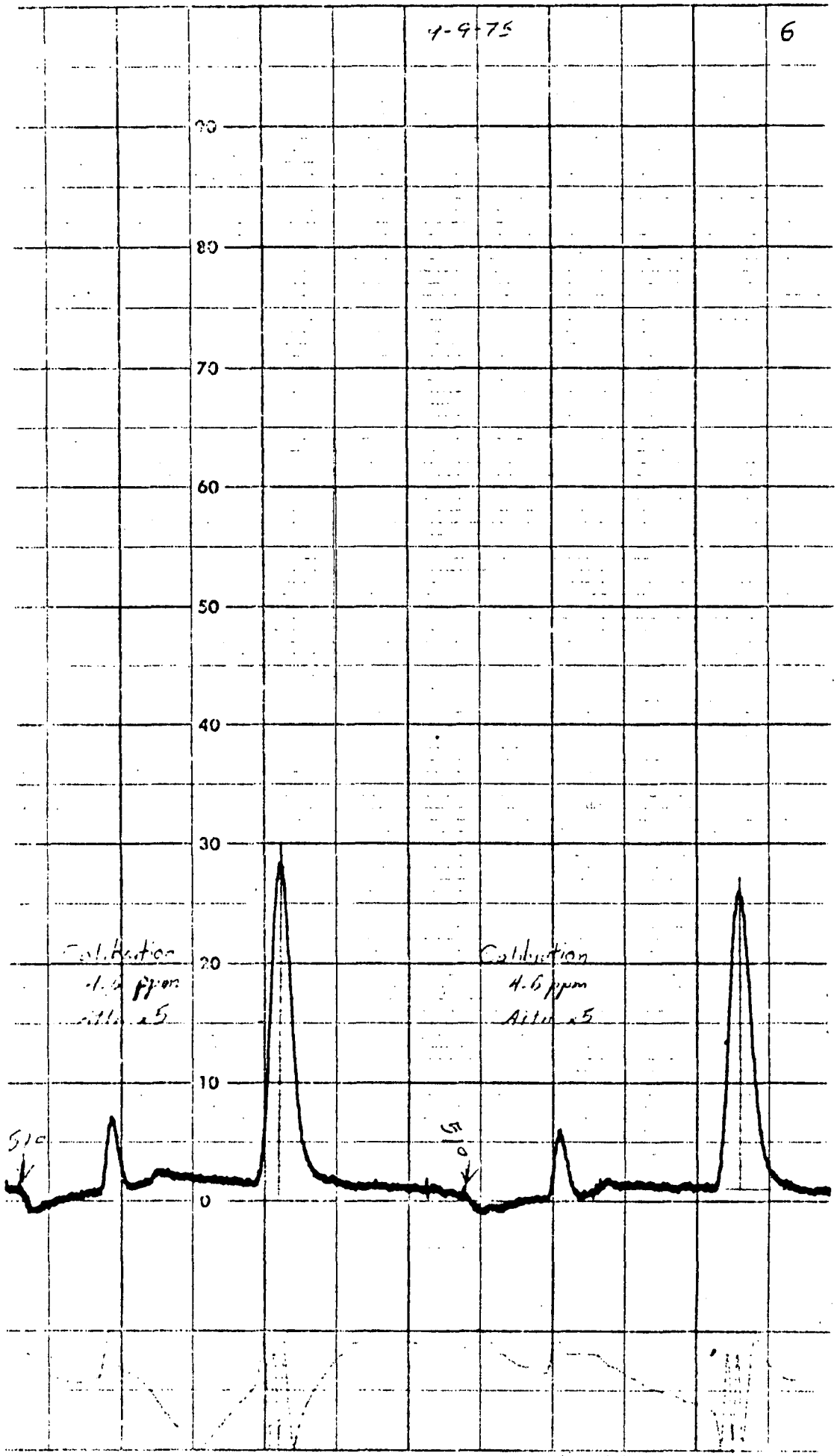
Attn = 5
5/0
Calibration
4.6 ppm
Attn = 5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80
70
60
50
40
30
20
10
0
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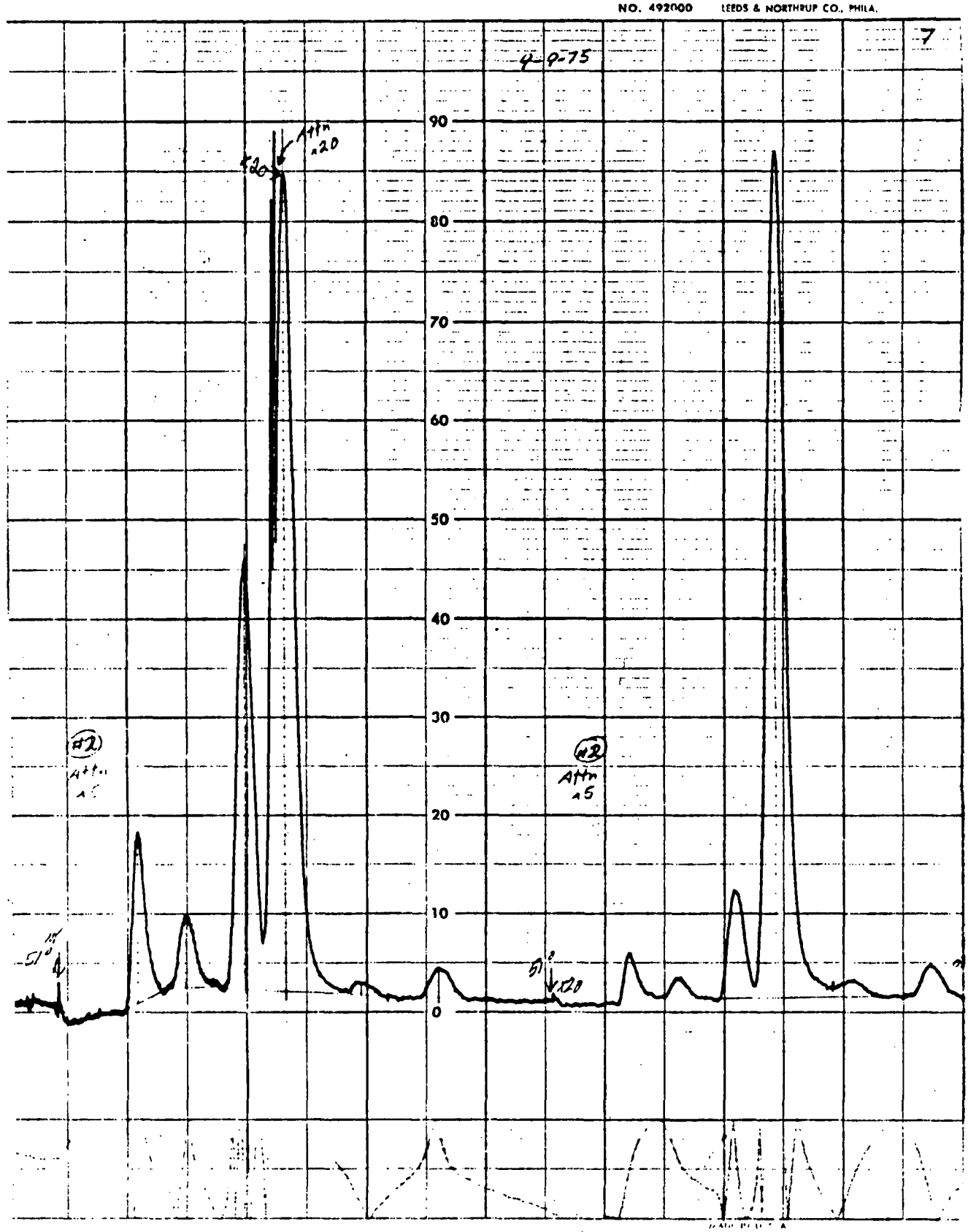
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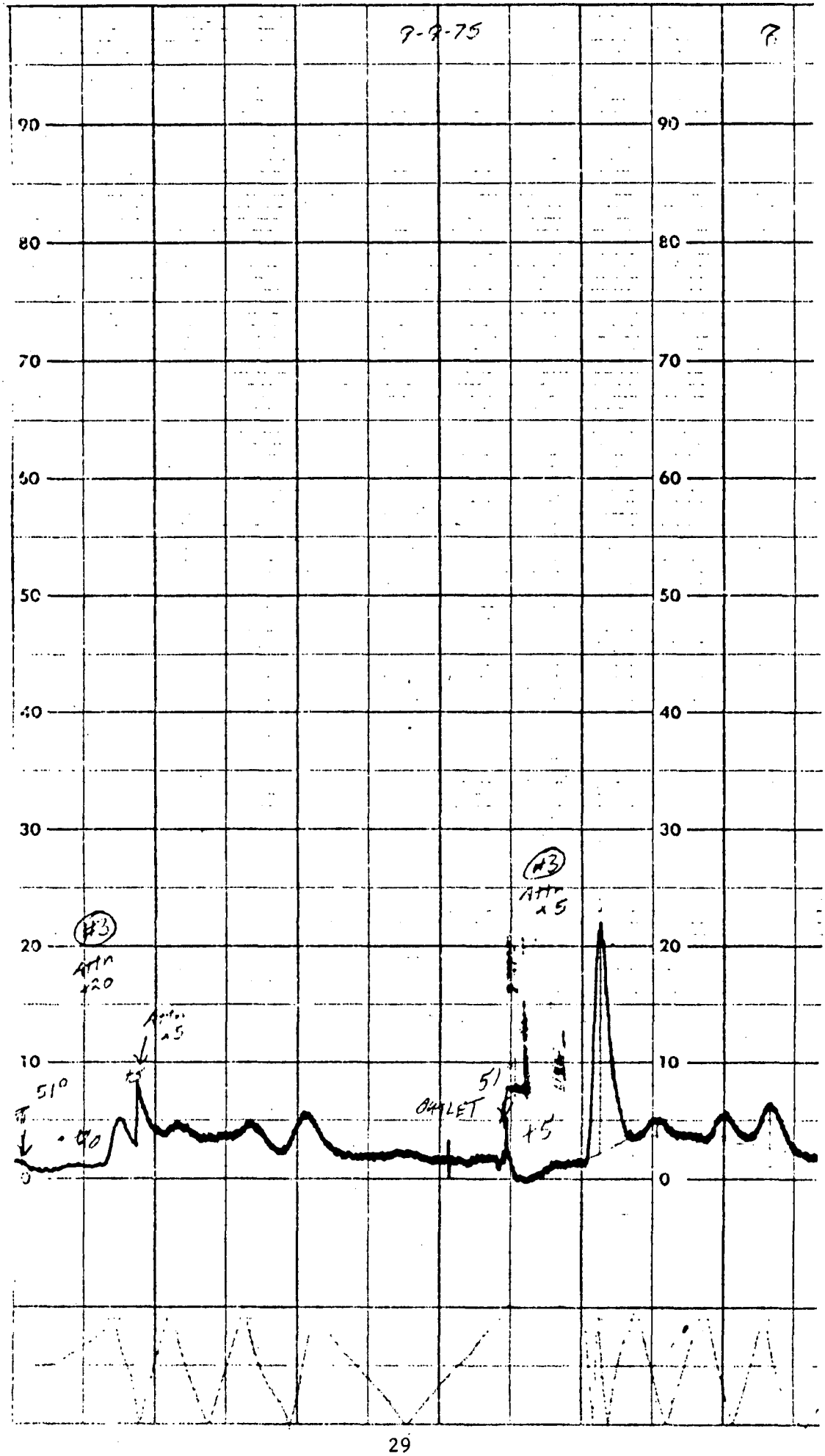
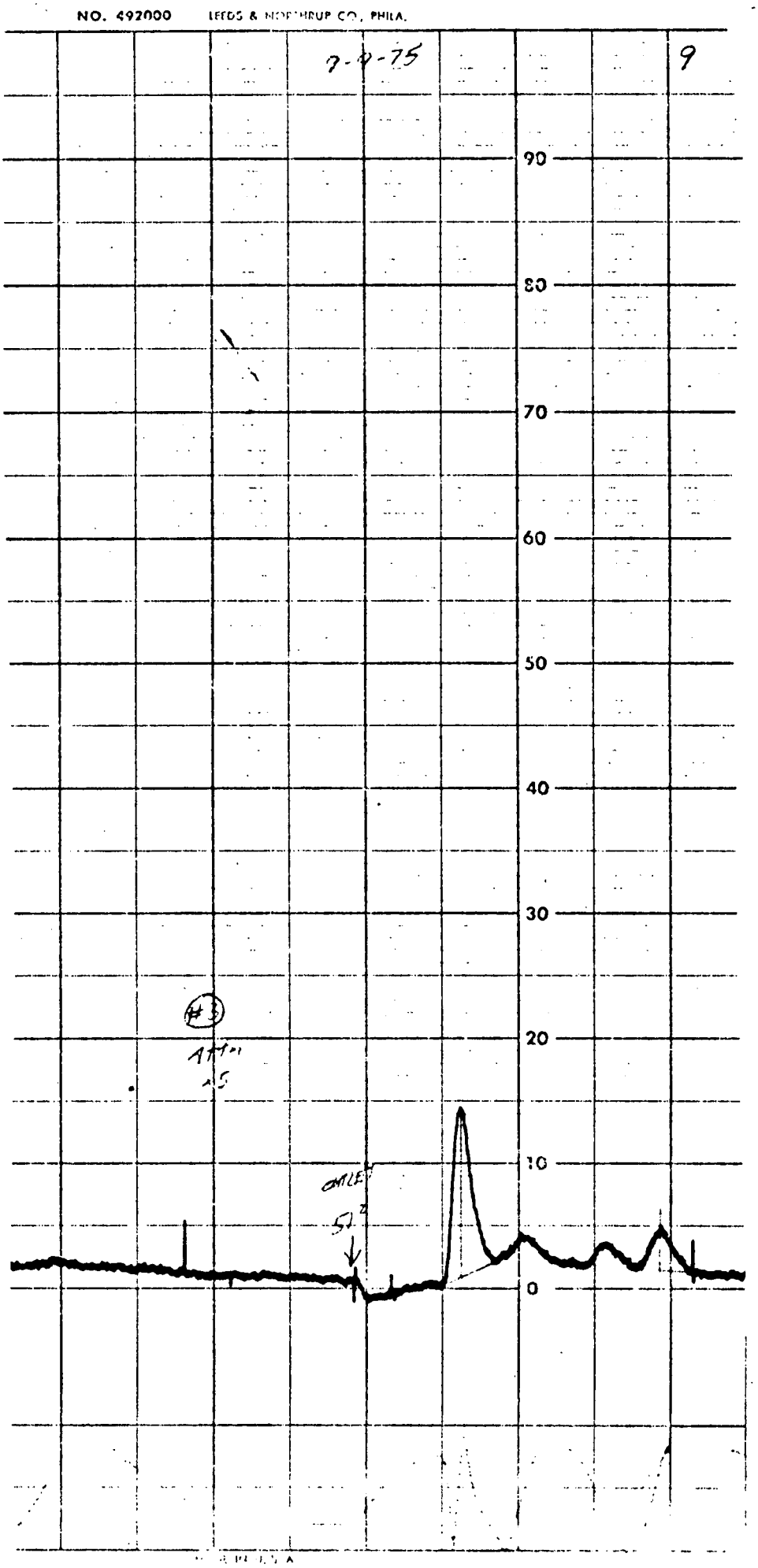
4-9-75
7
90
80
70
60
50
40
30
20
10
0
#2
#2
Attn
Attn
51°
51°


7-7-75
90
80
70
60
50
40
30
20
10
0
#3
ATTN x20
ATTN x5
51°
51
OUTLET
#3
ATTN x5
+5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60
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20
10
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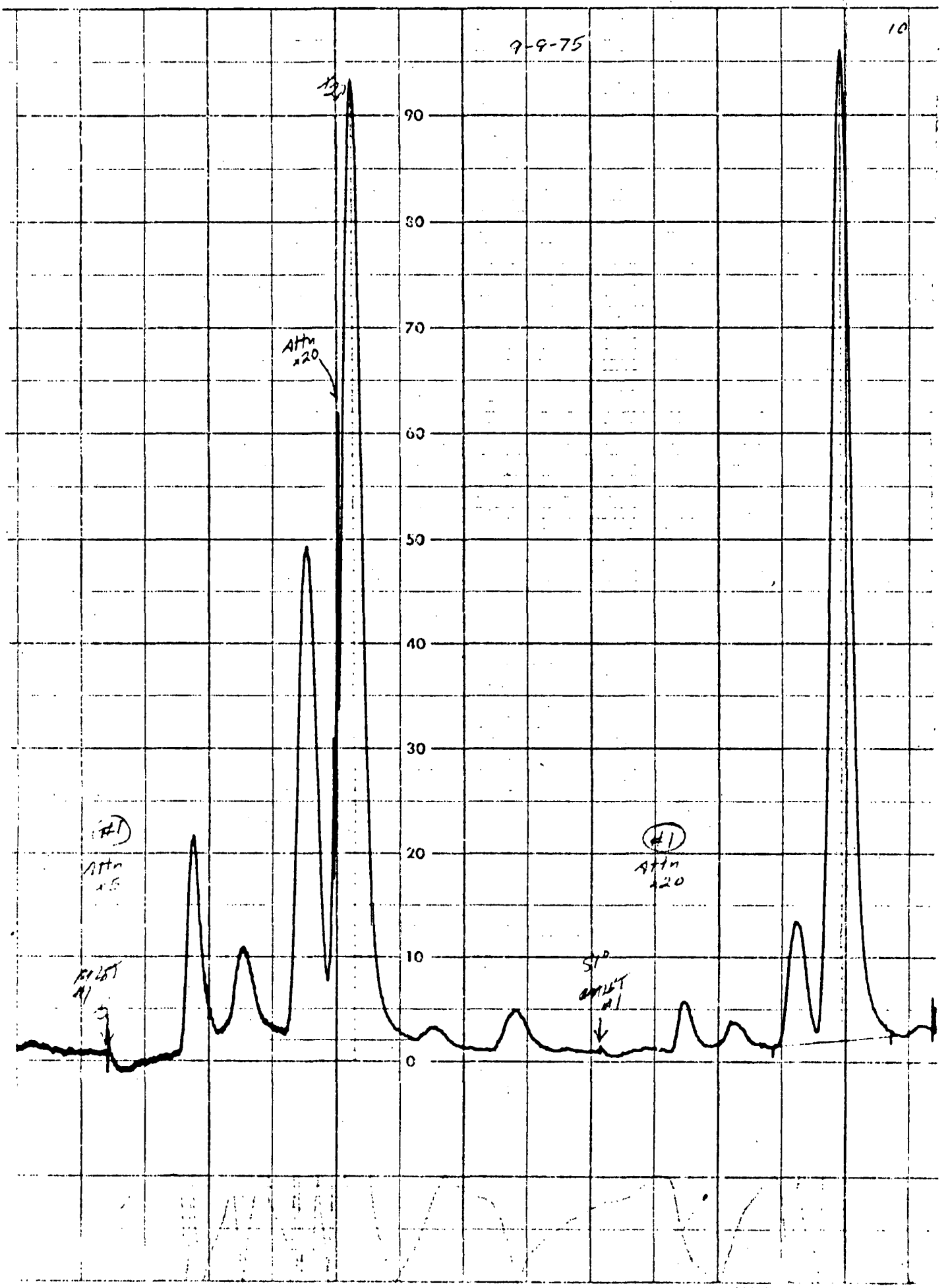
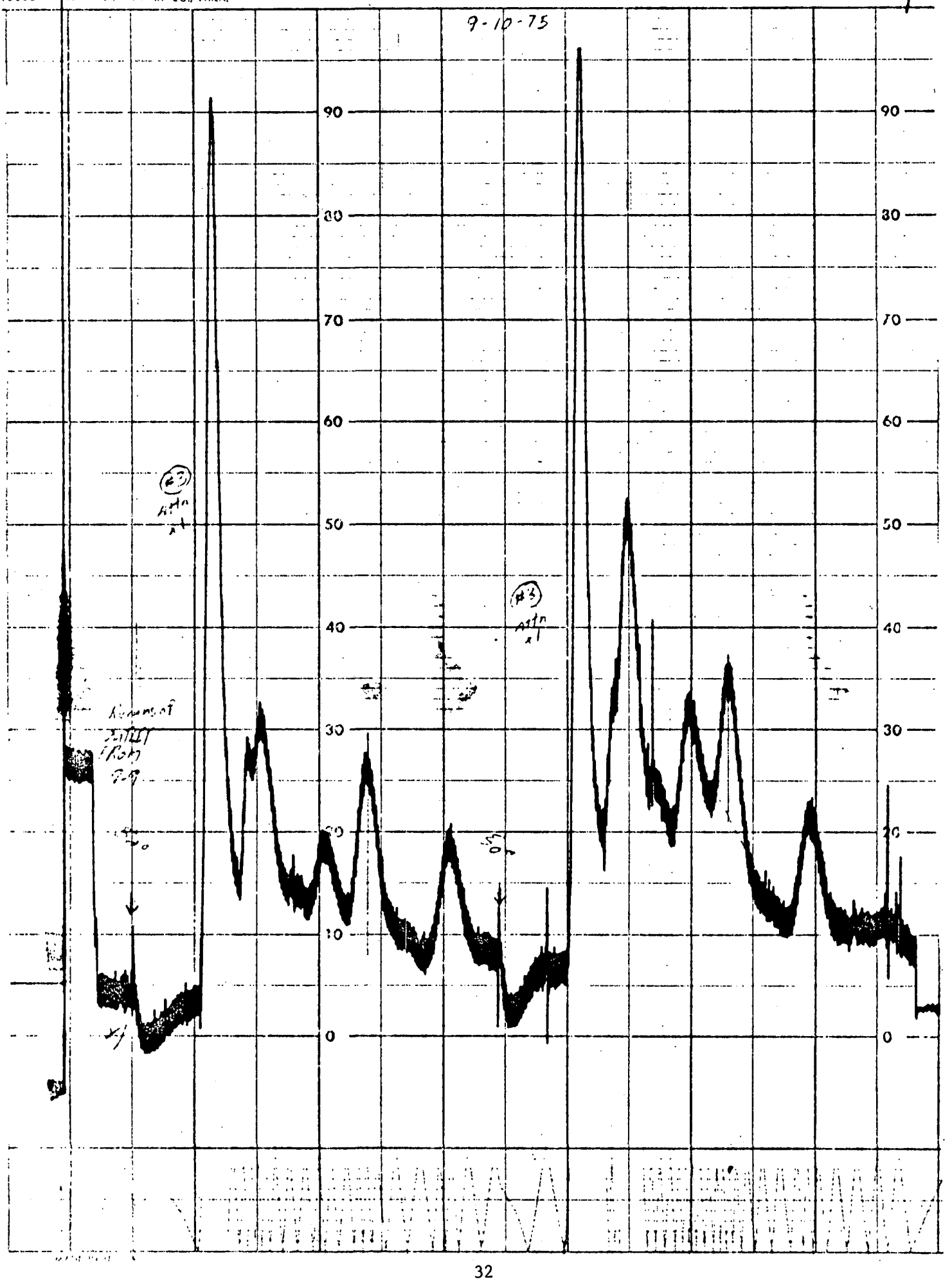
#3
AMPL
.5
CAL
5


7-9-75
10
90
80
70
60
50
40
30
20
10
0
Attn x20
#1
Attn x5
#1
Attn x20
Stop
Outlet #1


9-10-75
90
80
70
60
50
40
30
20
10
0


NO. 492000 LEEDS & NORTHRUP CO., PHILA.
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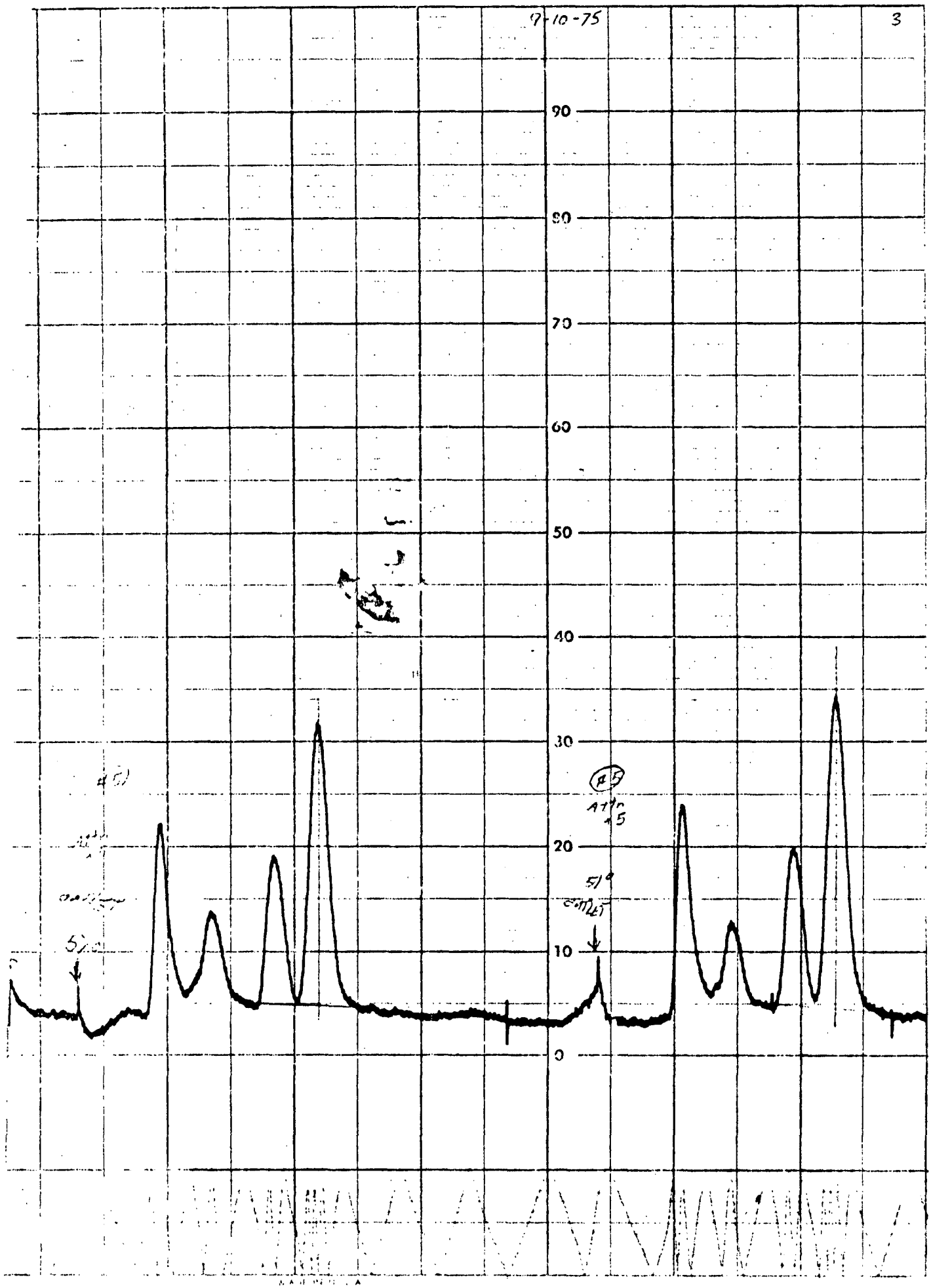
90
80
70
60
50
40
30
20
10
0
ATTn
20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90
80
70
60
50
40
30
20
10
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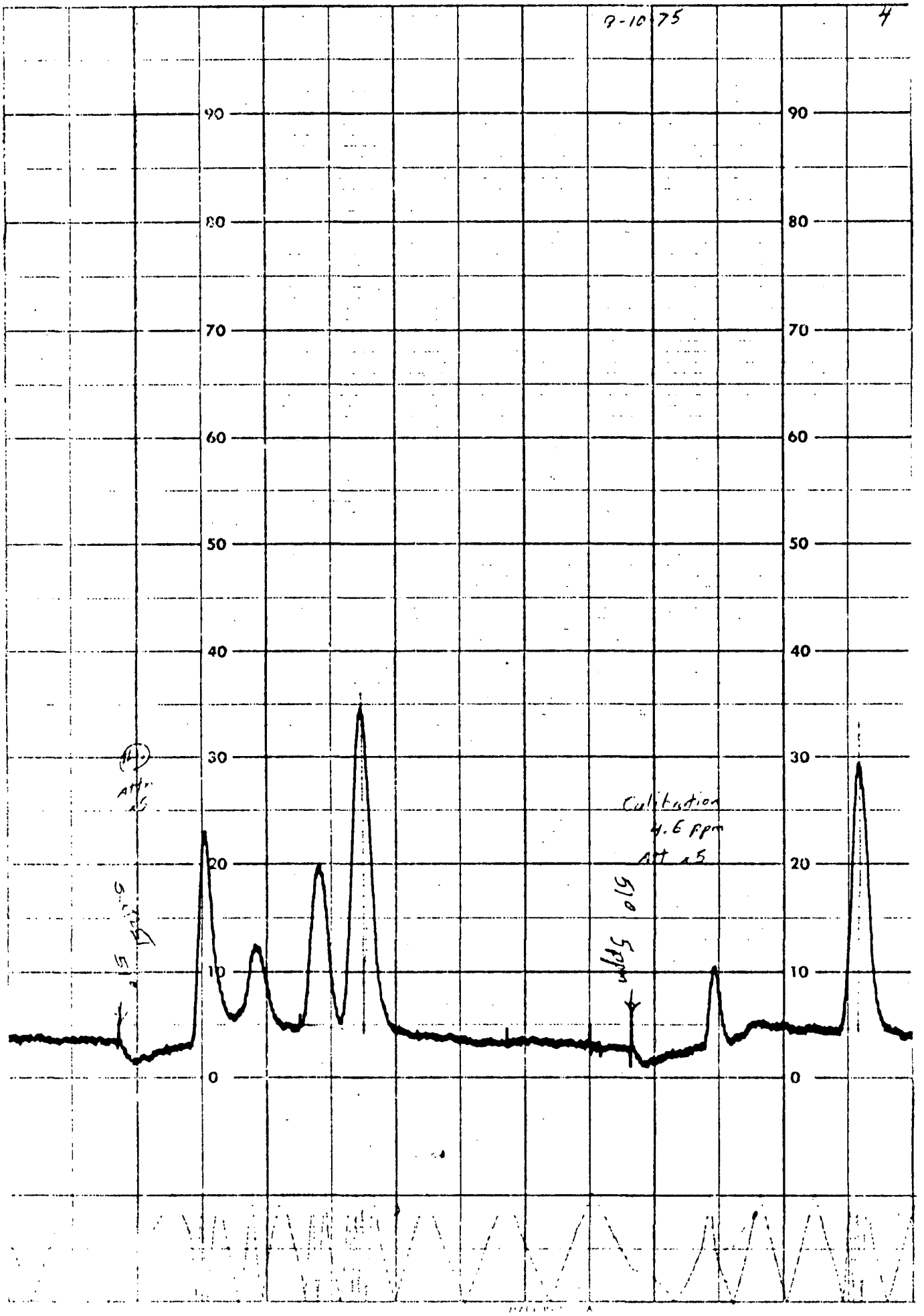
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90
80
70
60
50
40
30
20
10
0
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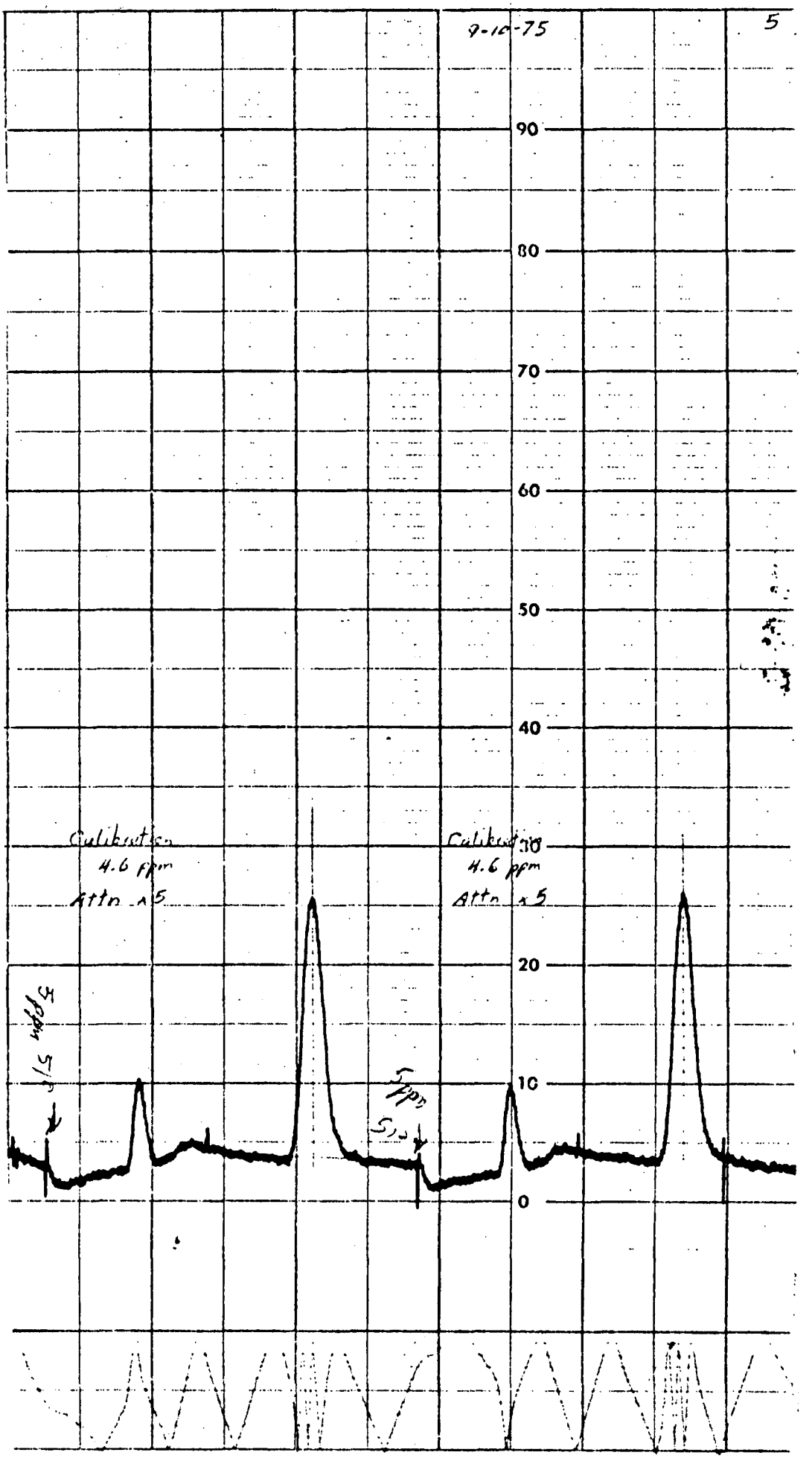
5 ppm
5 ppm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50
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30
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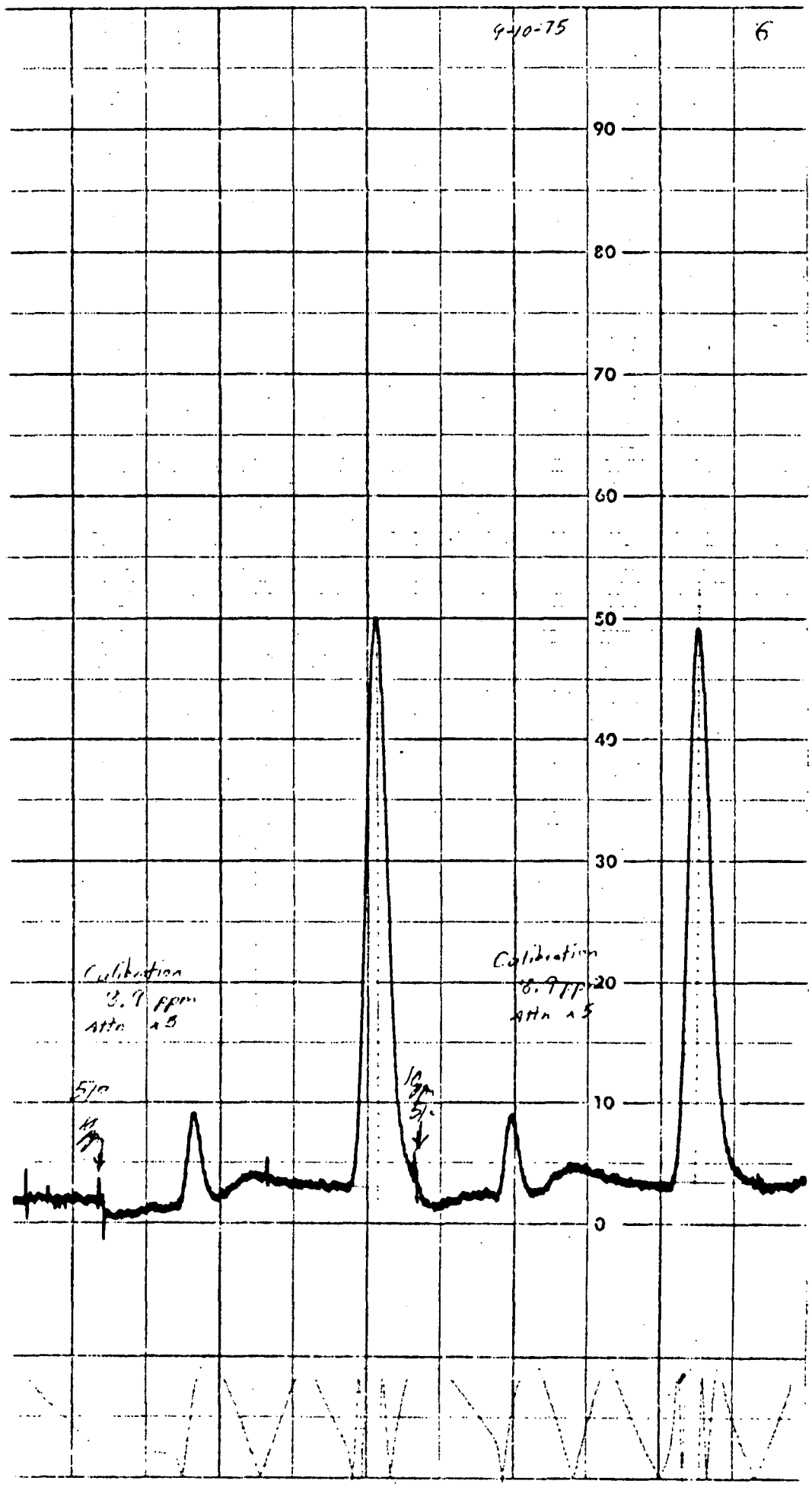
Calibration
4.6 ppm
Attn x 5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80
70
60
50
40
30
20
10
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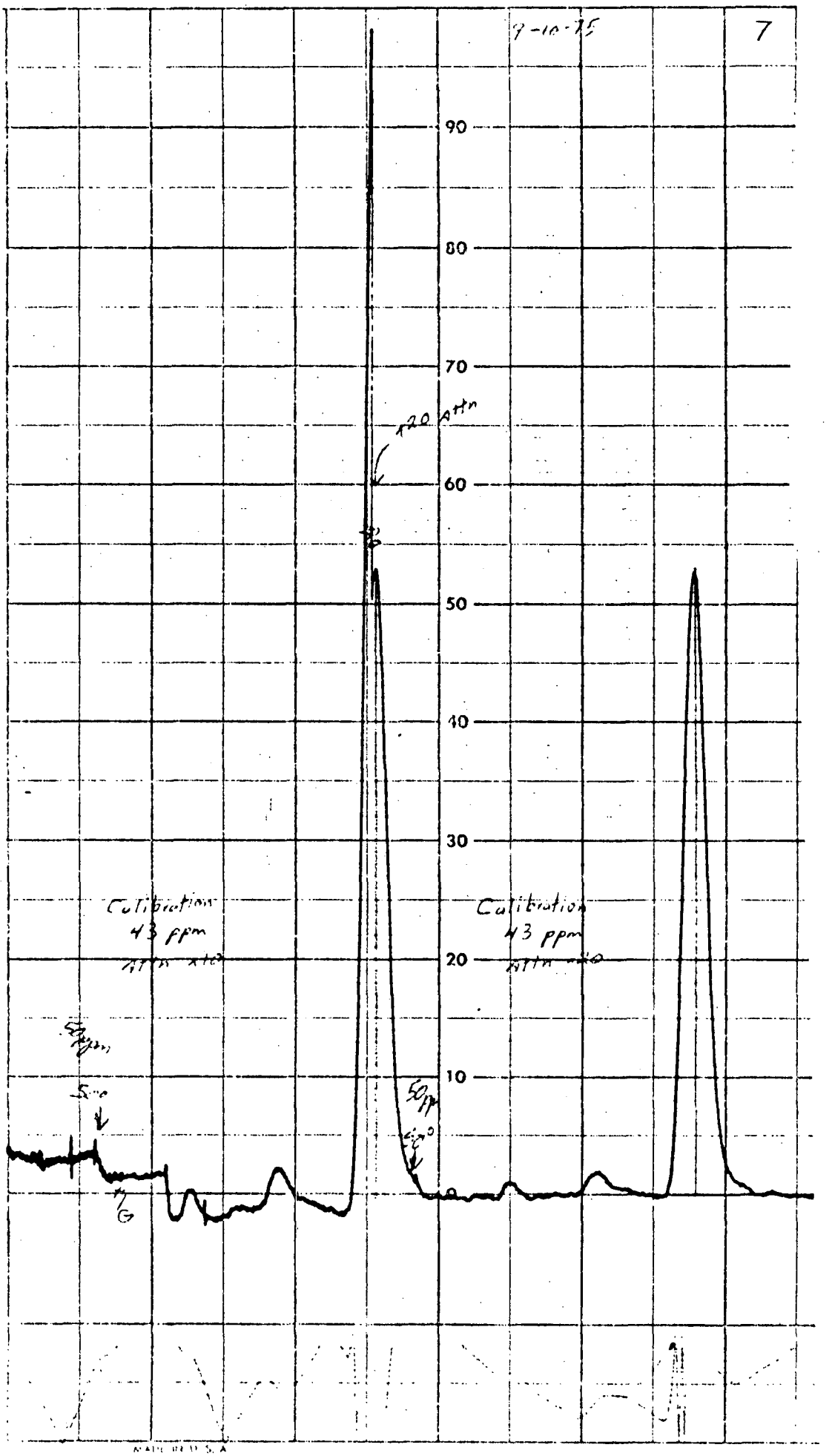
Atten x 5
Calibration
8.9 ppm
Atten x 5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90
80
70
x20 Attn
60
50
40
30
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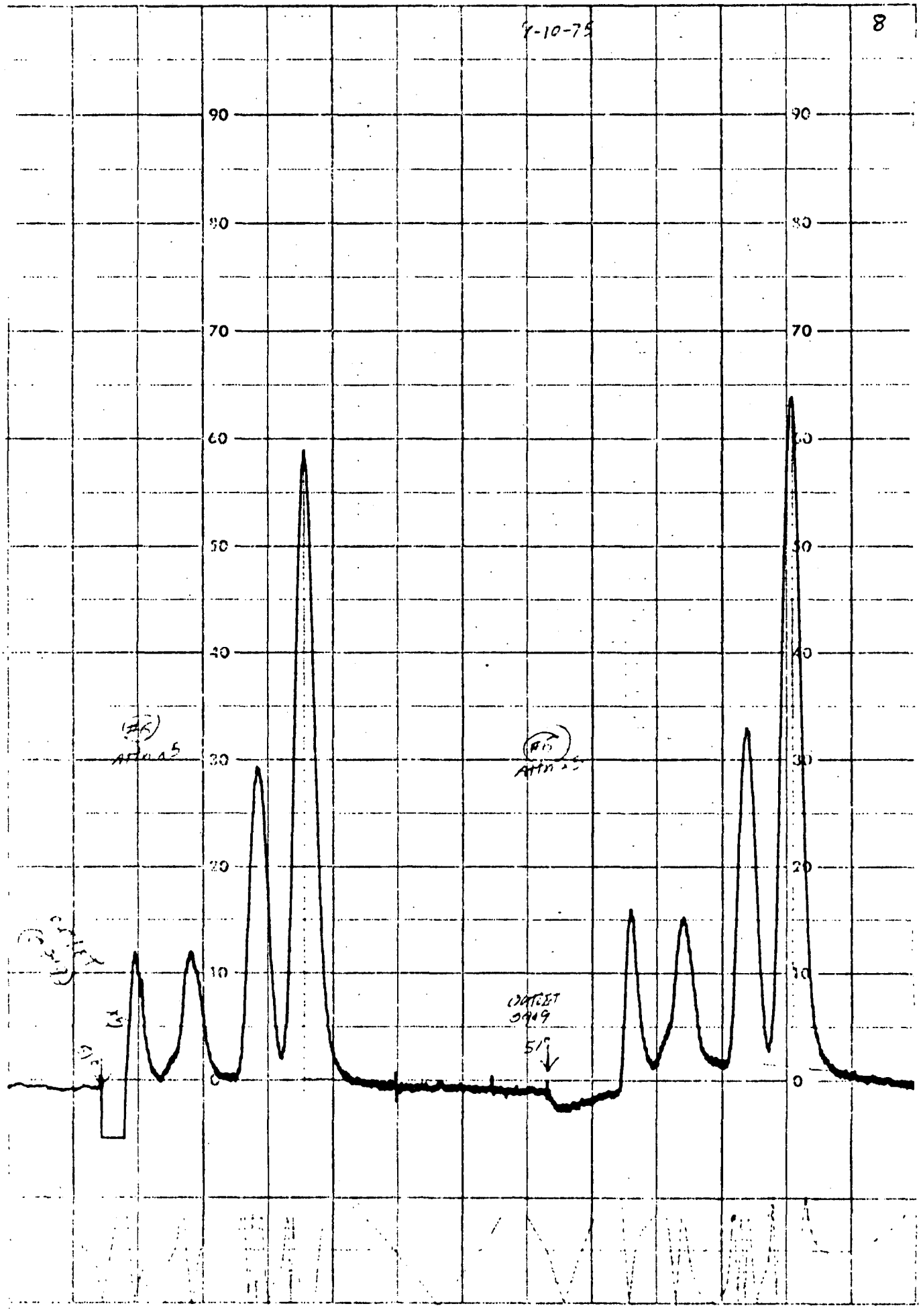
20
Calibration
43 ppm
10
MADE IN U.S.A.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80
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60
50
40
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20
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9-10-75
9
90
80
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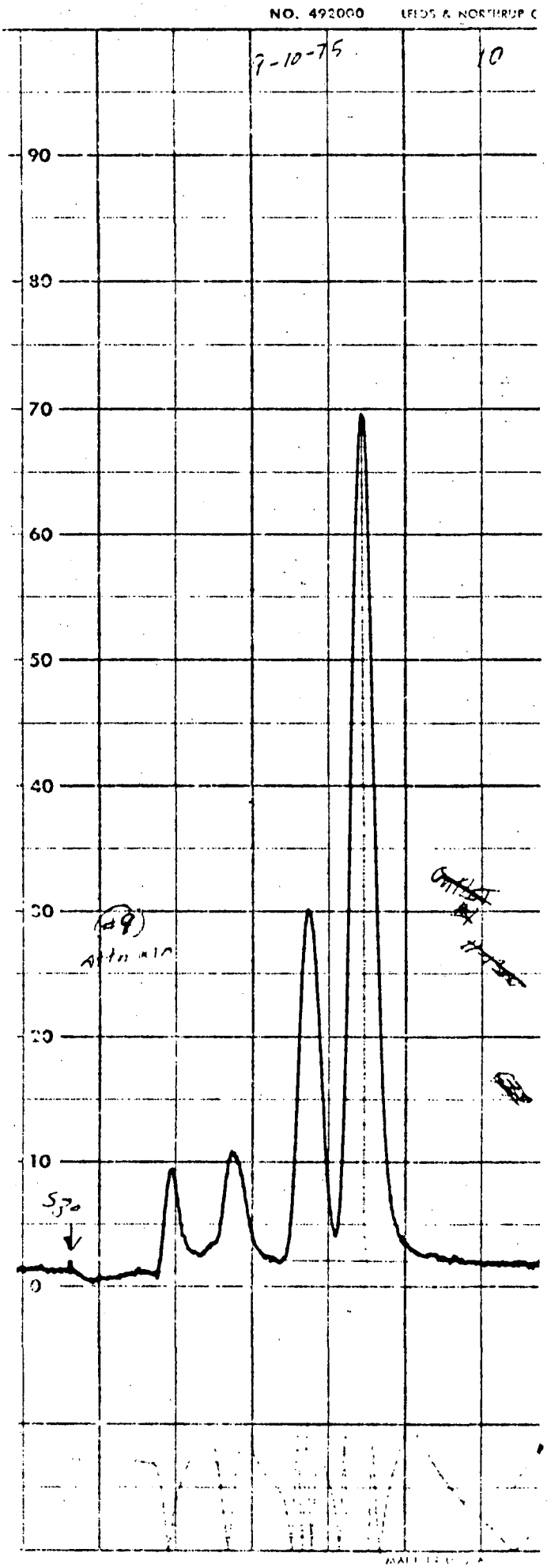
60
50
40
30
20
10
Attn x10
Attn x5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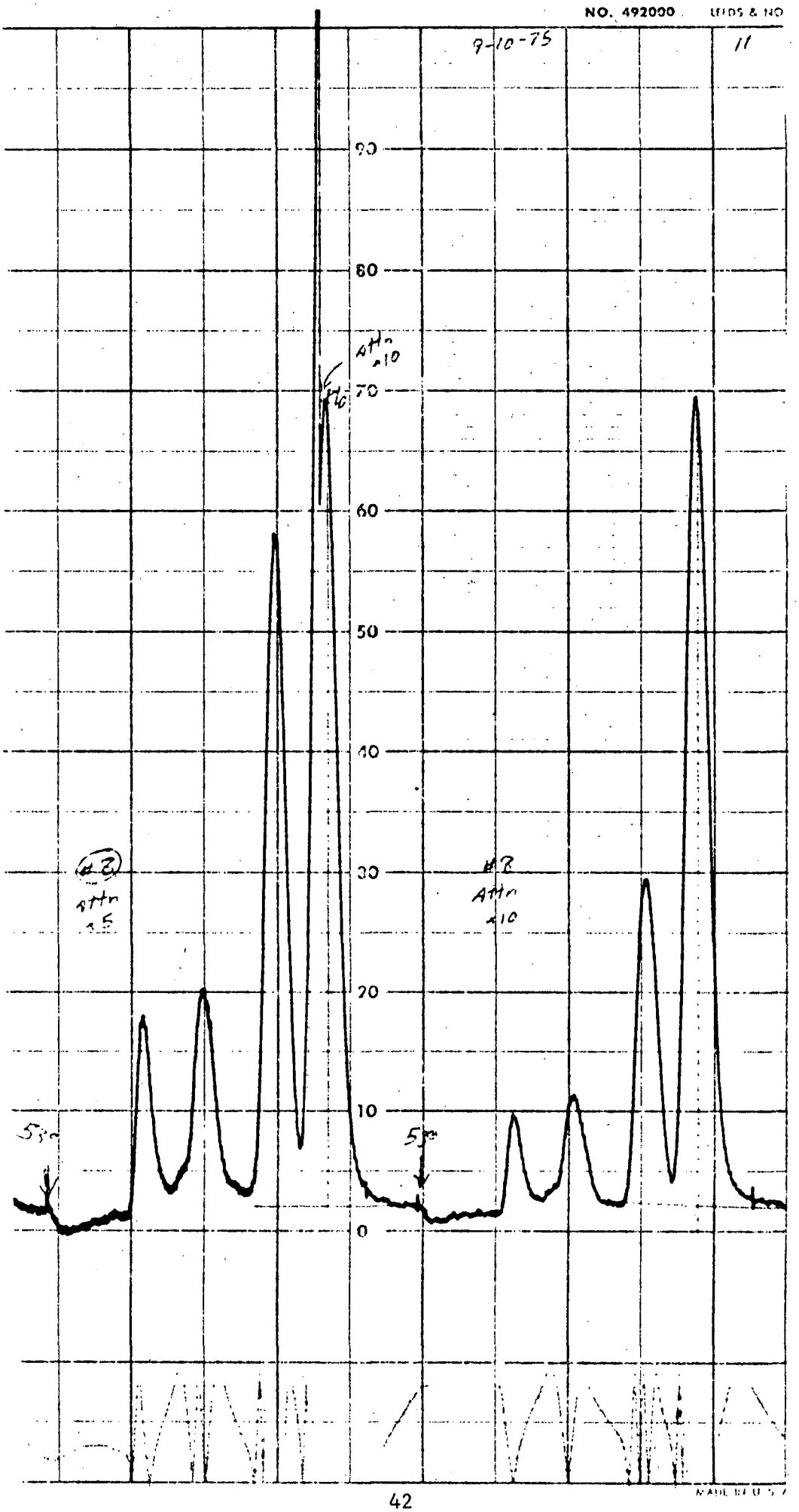
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9-10-75
11
90
80
70
60
50
40
30
20
10
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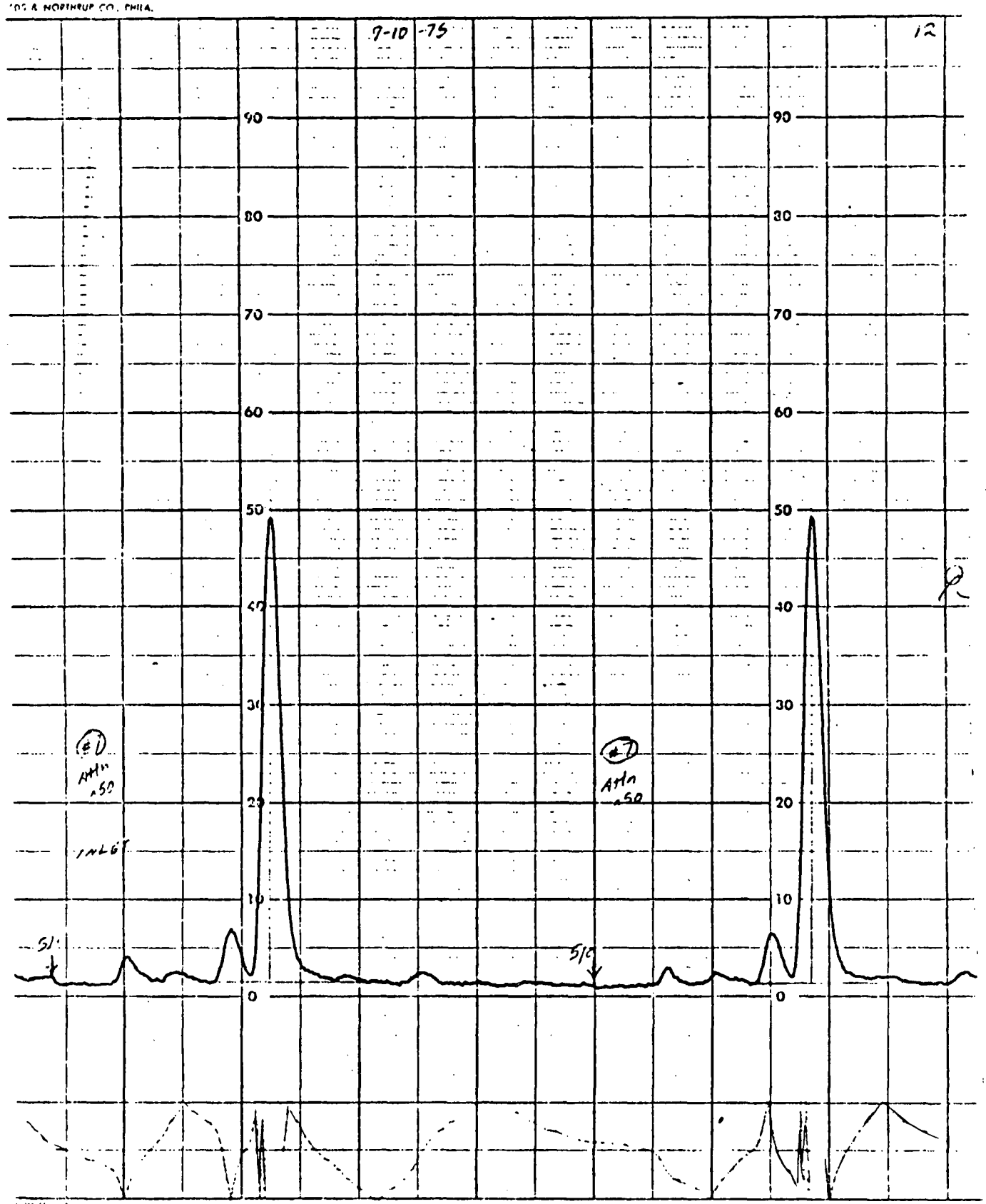
#2
ΔHn
÷5
#8
ΔHn
÷10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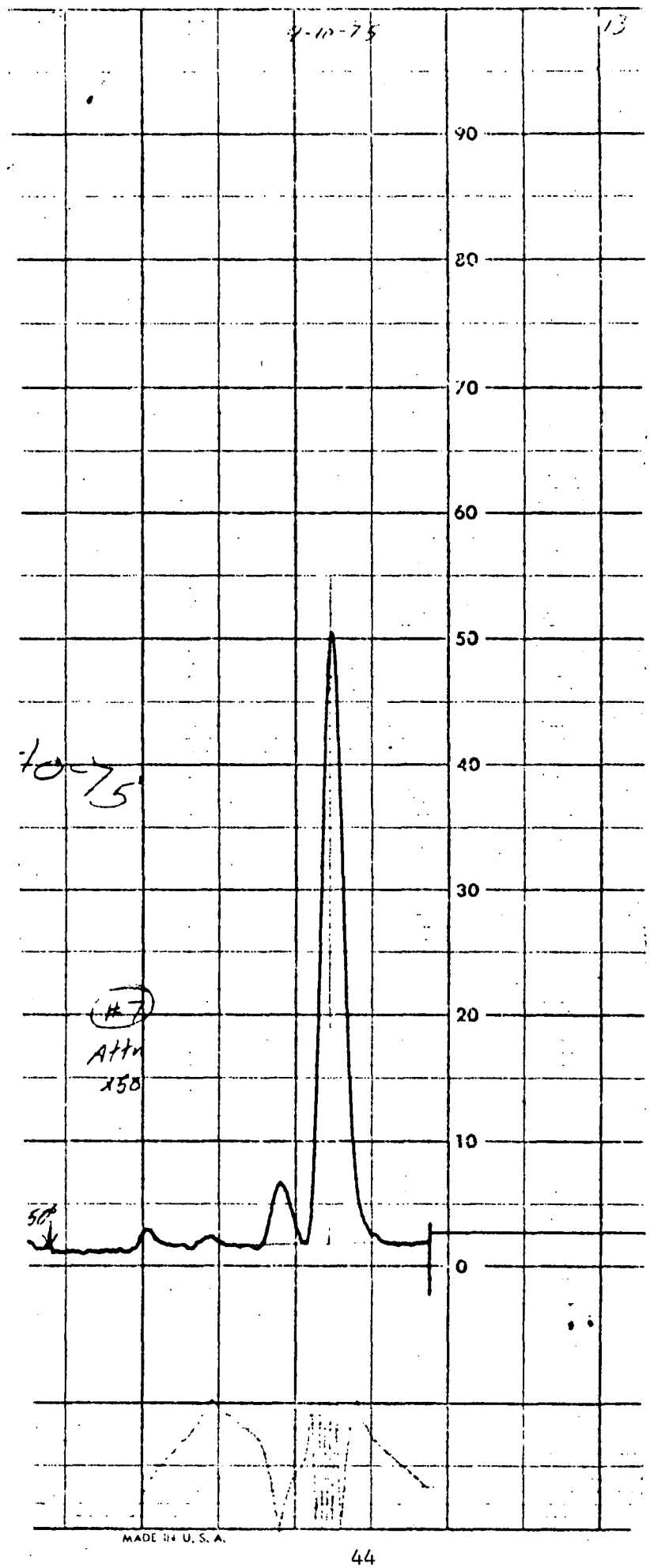
90
80
70
60
50
40
30
20
10
0
#1
#7
5/C
5/C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LEEDS & NORTHRUP CO., PHILA.
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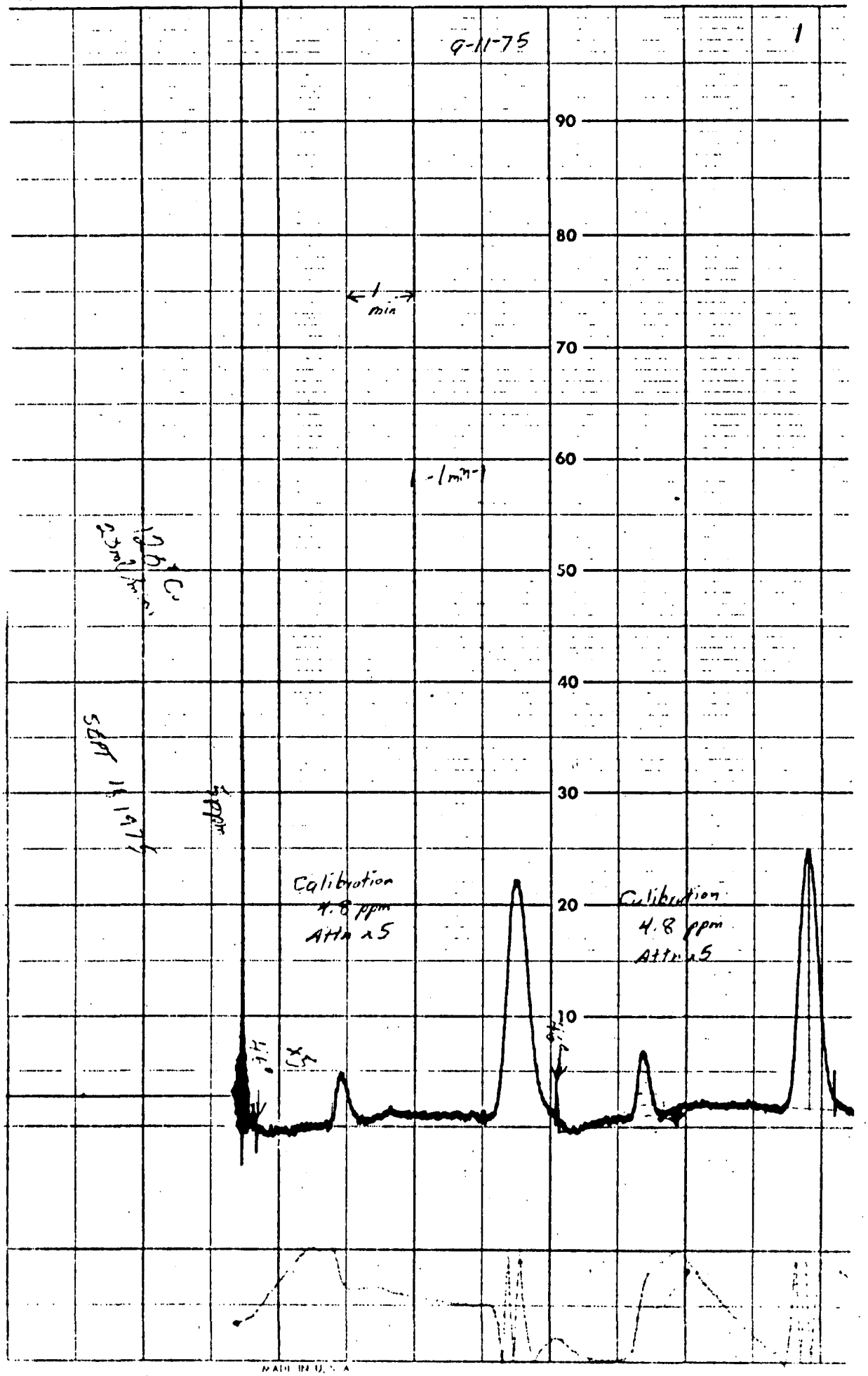
13
90
80
70
60
50
40
30
20
10
0
10-75
#7
Attn
x50
50
MADE IN U.S.A.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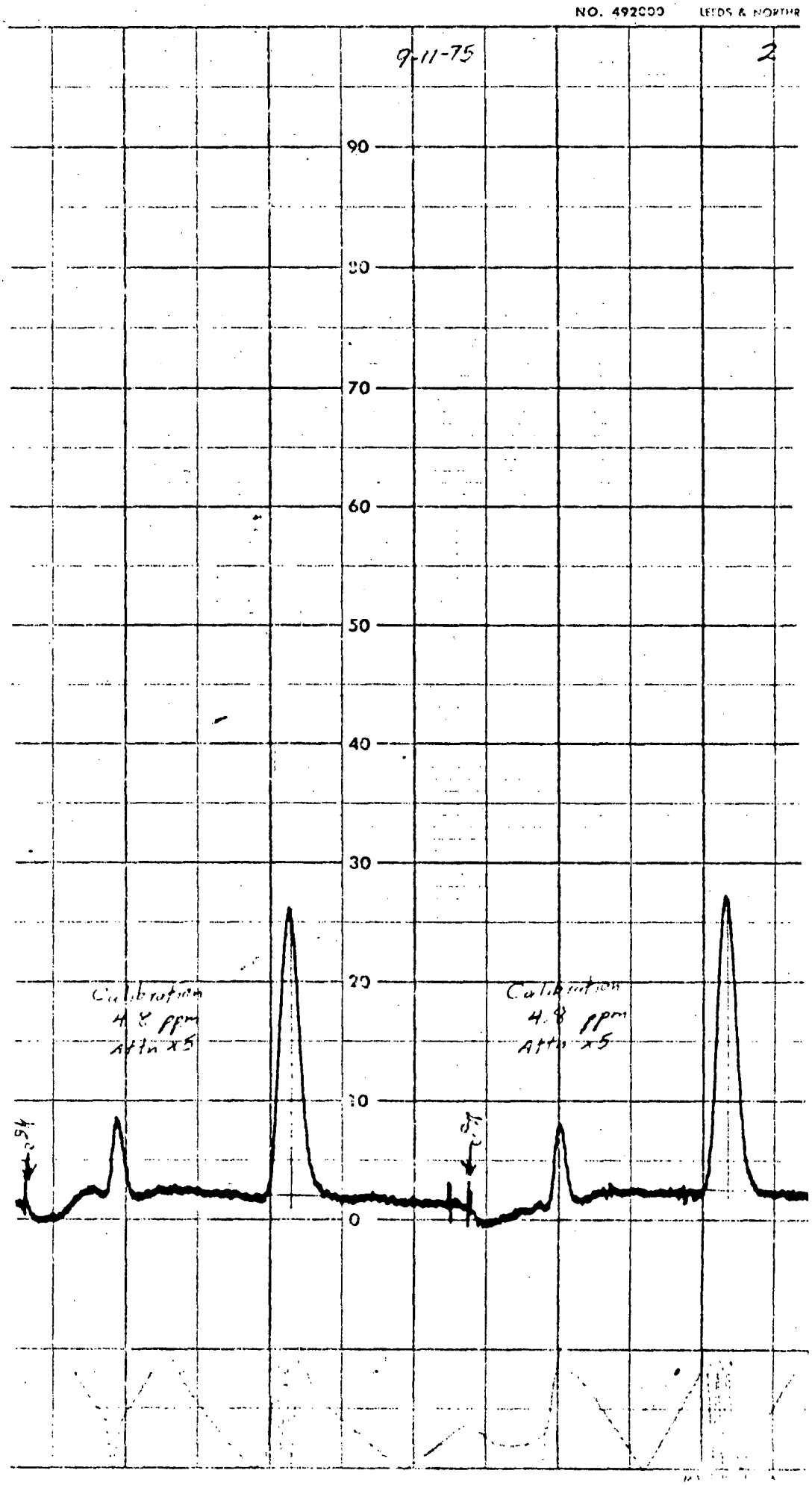
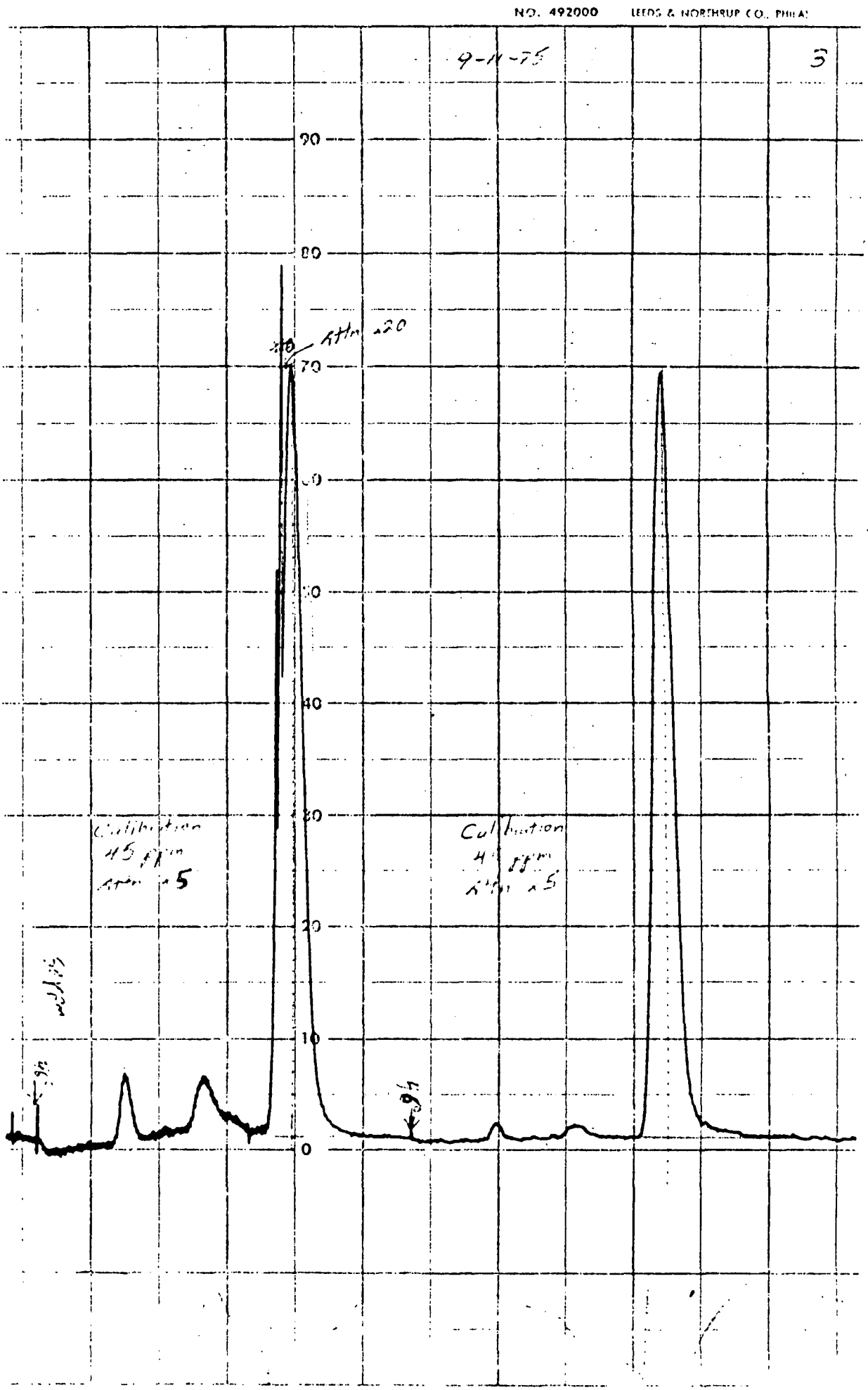
9-11-75
1
90
80
1 min
70
60
50
40
30
20
10
Calibration
4.8 ppm
Atten x5
Calibration
4.8 ppm
Atten x5
MADE IN U.S.A.


NO. 492000 LEEDS & NORTHR
9-11-75
2
90
80
70
60
50
40
30
20
10
0
Calibration
4.8 ppm
Attn x5
Calibration
4.8 ppm
Attn x5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9-1-75
3
90
80
70
60
50
40
30
20
10
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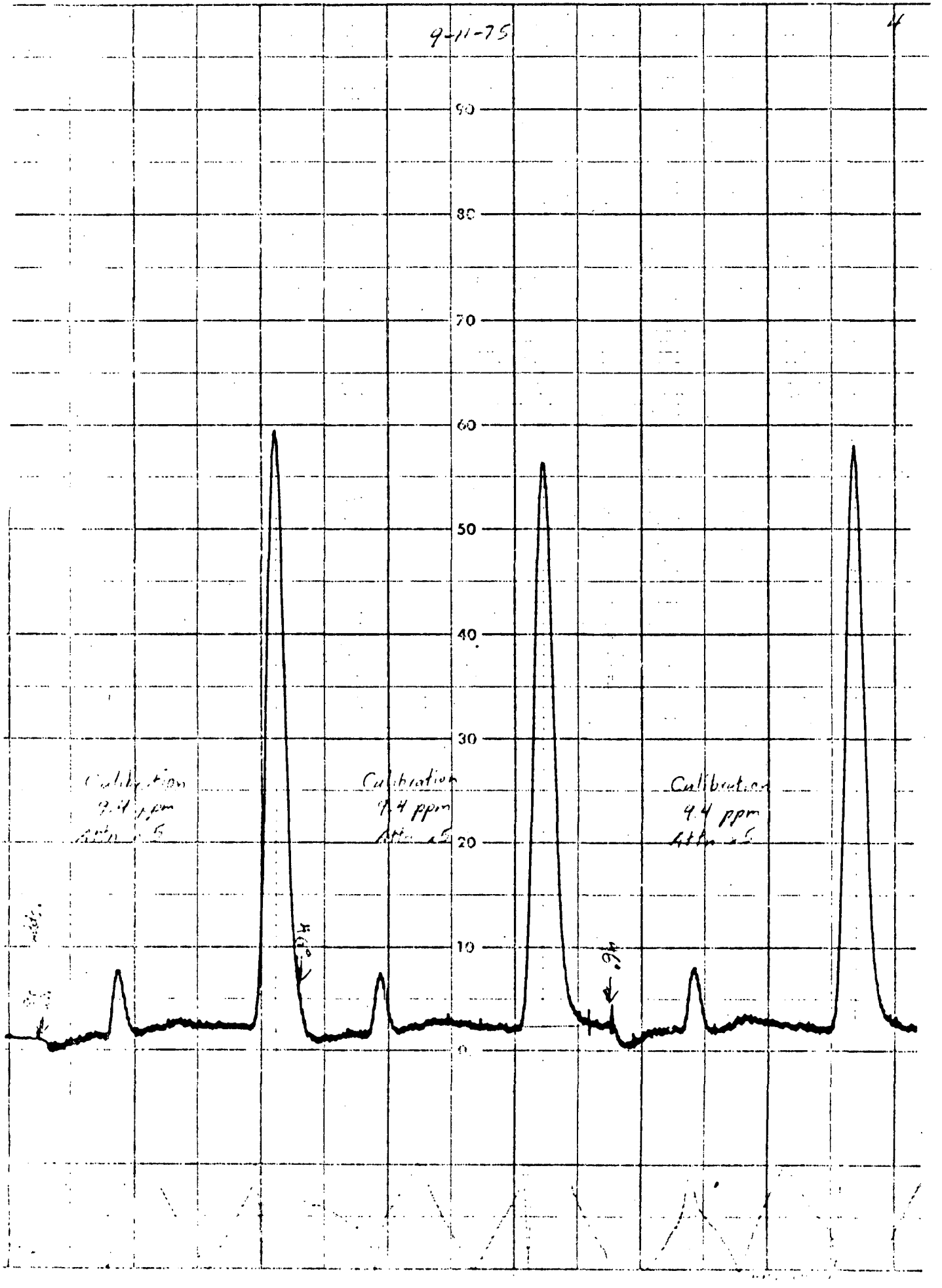
Calibration
45 ppm
Atm ~5
Calibration
45 ppm
Atm ~5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Calibration
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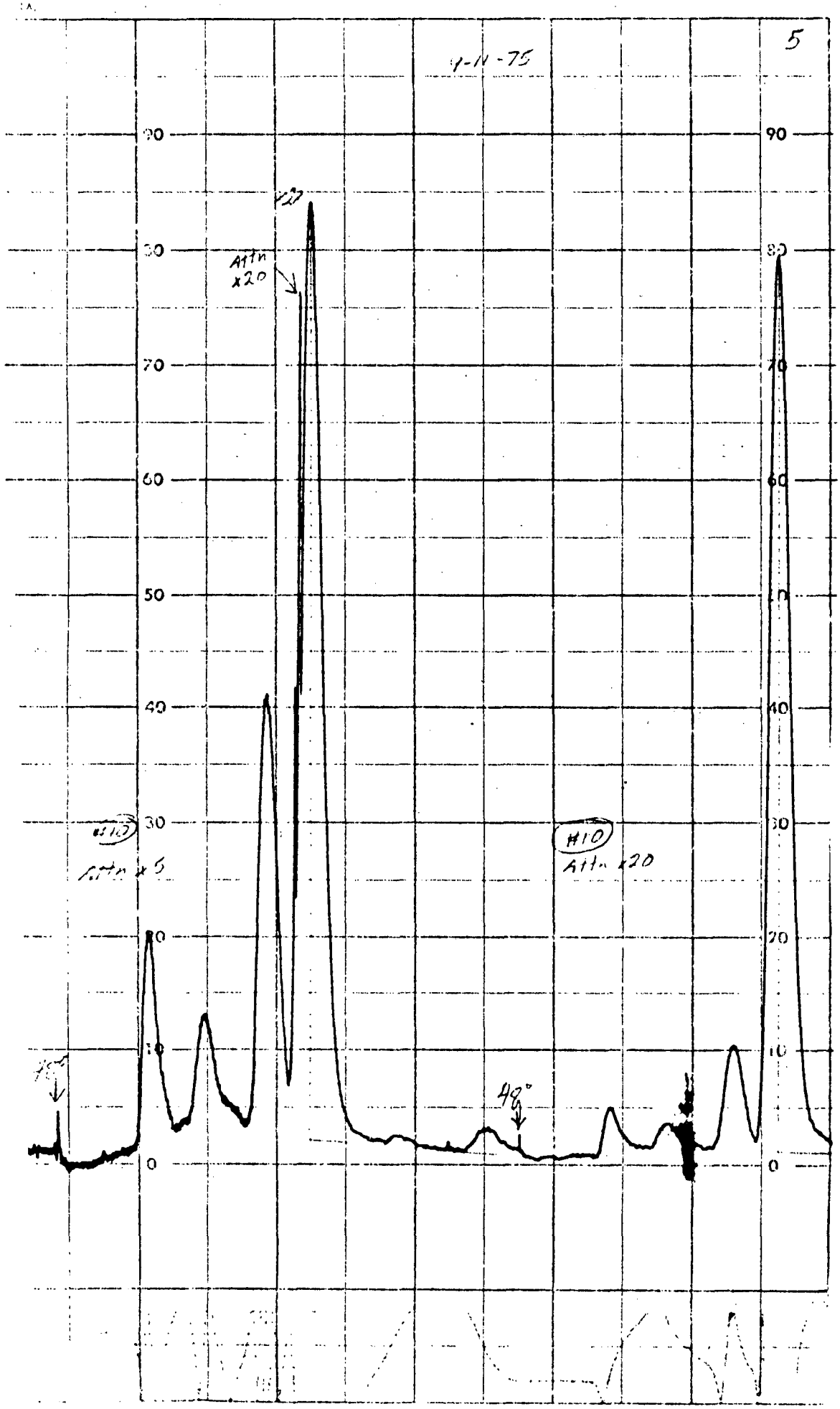
Calibration
9.4 ppm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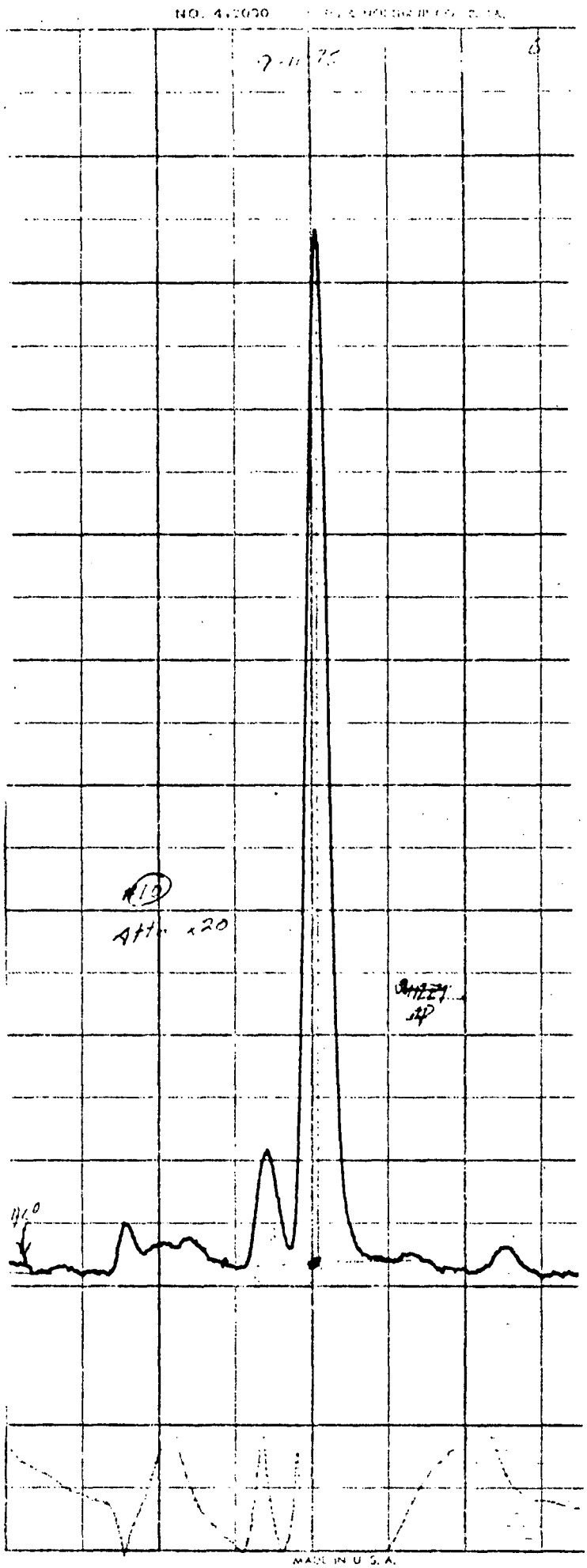
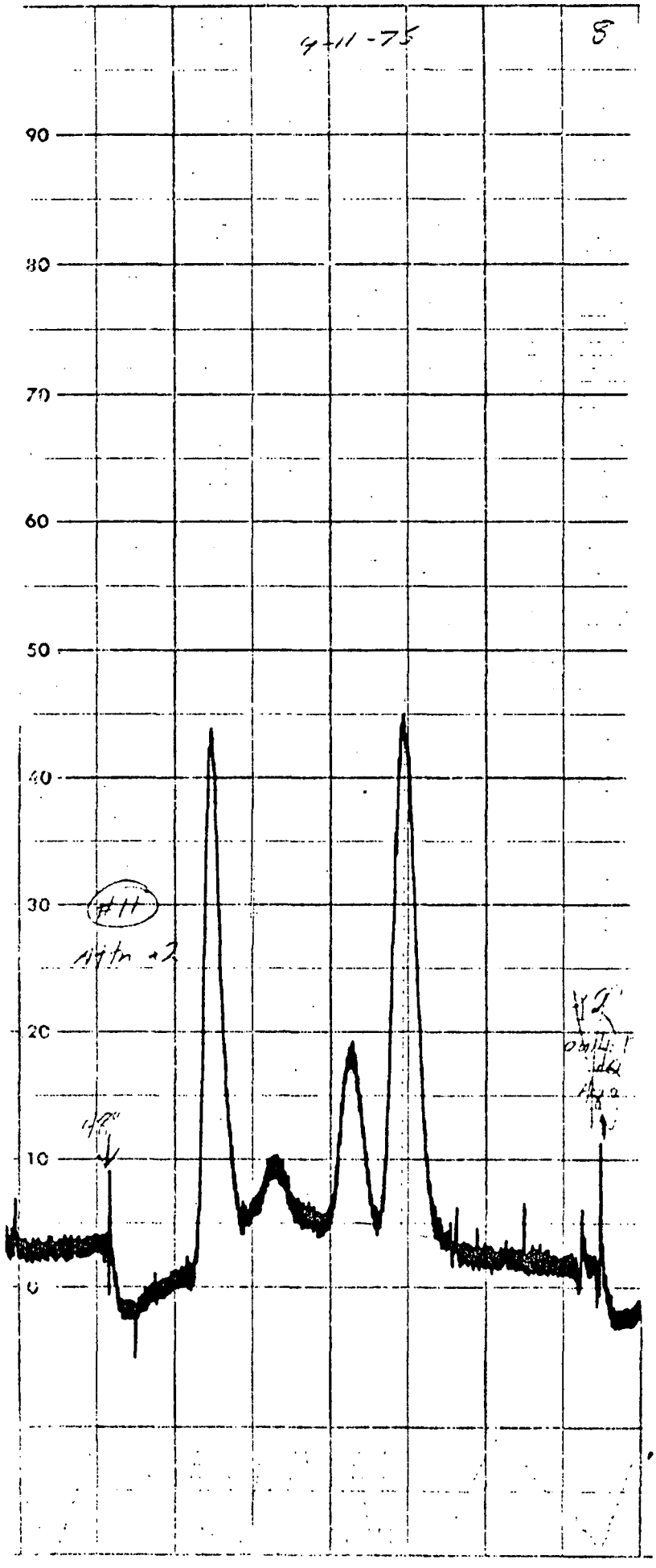
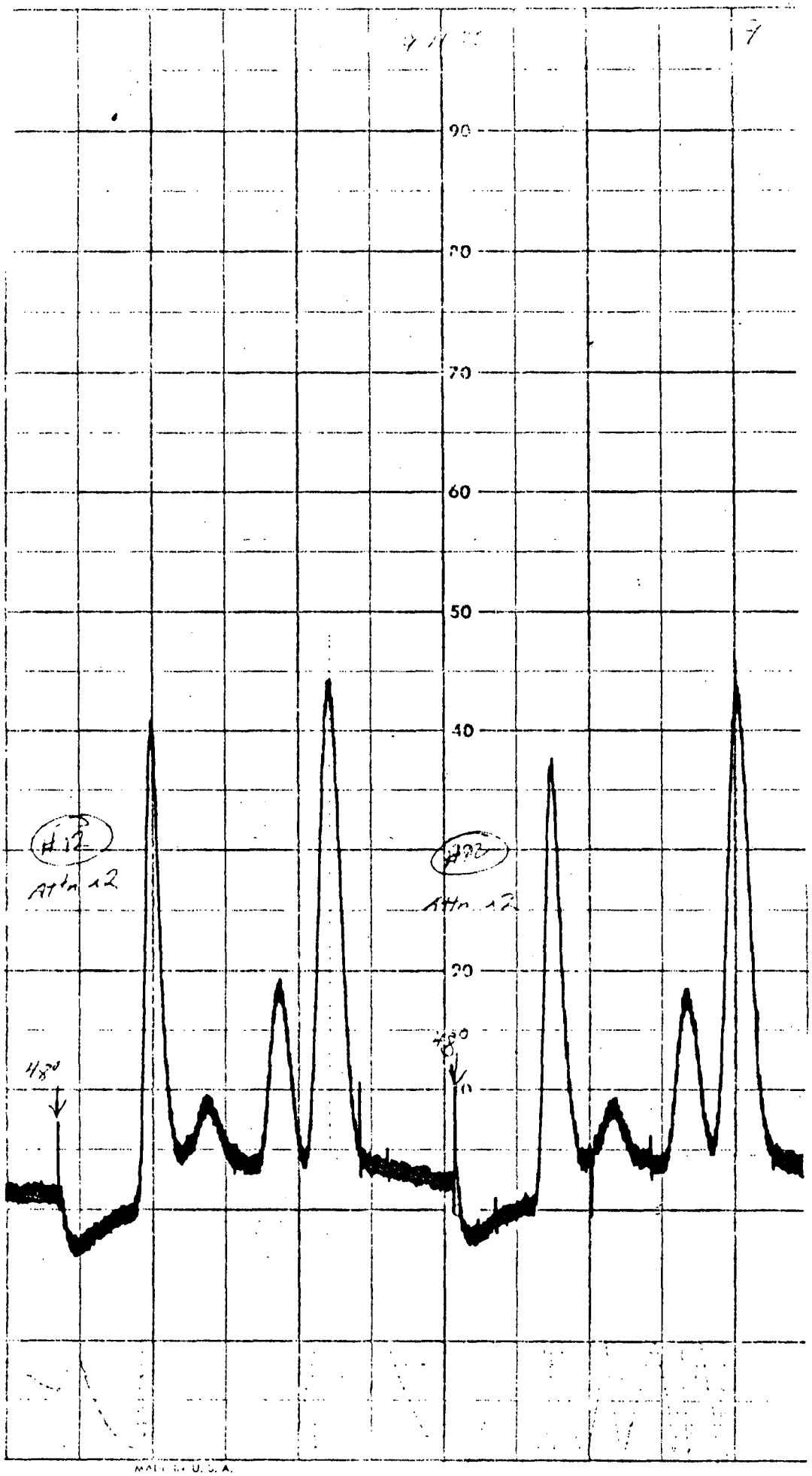
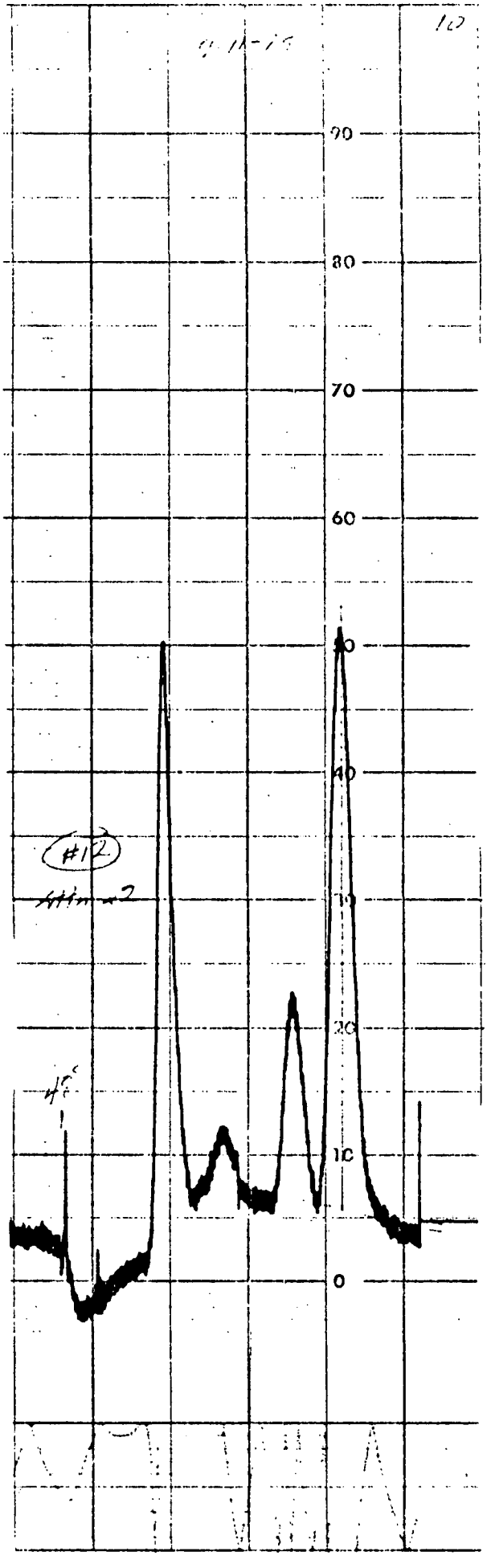
5
90
80
70
60
50
40
30
20
10
0
Attn x20
H10
Attn x20
49°


#13
Att. x20
H2O
MADE IN U.S.A.


NO. 492000 LEEDS & NORTHRUP CO., PHILA.
7-11-75
7
90
80
70
60
50
40
30
20
10
0
#11
Attn x2
#11
Attn x2


4-11-75
8
90
80
70
60
50
40
30
20
10
0
#11


90
80
70
60
50
40
30
20
10
H12
Attn 12
H8°
H23
Attn 12
H8°


90
80
70
60
50
40
30
20
10
0

APPENDIX B

MASS SPECTRA DATA

A mass spectra scan using conditions identical to those during the field test shows only three identifiable peaks. The air peak obscures the peaks with retention times of 1.3 and 2.1 min. The peak with retention time of 3.7 min is the trichloroethylene peak. Table B-I shows the major mass peaks and their intensities. All eight characteristic peaks are present in the scan (labeled 7416 by the mass spectrometer) and the correlation is excellent for this peak. No evidence of a co-eluting interference could be found in the MS data.

The scan labeled 7413 corresponds to the peak of retention time 3.0 min and the scan labeled 7427 corresponds to the peak of retention time 6.4 min. The mass spectral scans for these are of low intensity. Table B-II shows the eight major peaks of the probable compounds in order and indicates only the presence (+) or absence (-) in the scan. A (B) indicates the peak also appeared in the background scan. The probable major components of these peaks are 1,1,1 trichloroethane for 7413 and 1,1,2,2 tetrachloroethane for 7427. Copies of the computer scan for the three peaks and the background scan together with the ion-current response of the chromatogram follow Table B-II.

TABLE B-I

MAJOR PEAKS - TRICHLOROETHYLENE PEAK VERSUS TABLE OF MAJOR PEAKS DATA

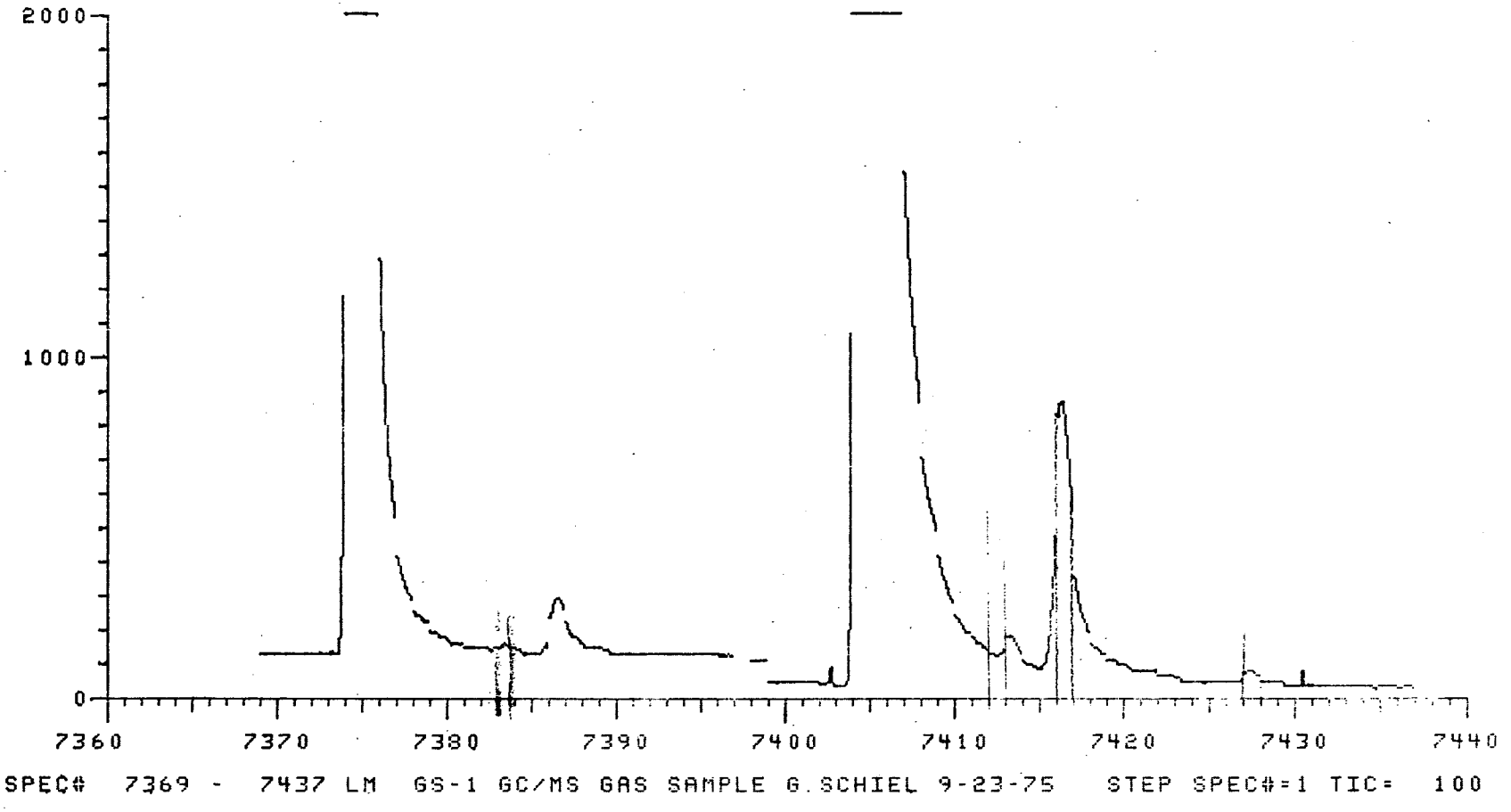
Trichloroethylene Major Peaks		Scan No. 7416	
Mass	Relative Intensity	Mass	Relative Intensity
130	100	130	100
132	97	132	80
95	87	95[a/]	69
97	57	97	60
134	32	60	32
60	27	134	28
99	9	62	9
62	9	99	6

a/ Also in background.

TABLE B-II

EIGHT MAJOR PEAKS OF TRICHLOROETHANE AND TETRACHLOROETHANE

1,1,1 Trichloroethane		1,1,2 Trichloroethane		1,1,2,2 Tetrachloroethane		1,1,1,2 Tetrachloroethane	
Mass	Scan 7413	Mass	Scan 7413	Mass	Scan 7427	Mass	Scan 7427
97	+	83	-	83	B	131	+
99	+	97	+	85	B	133	B
61	+	61	+	168	+	117	B
26	B	85	B	87	-	119	-
27	B	99	+	95	B	135	-
63	+	26	B	166	+	95	B
117	B	27	B	131	+	121	-
119	+	63	+	133	B	97	-


2000
1000
0
7360
7370
7380
7390
7400
7410
7420
7430
7440
SPEC# 7369 - 7437 LM GS-1 GC/MS GAS SAMPLE G.SCHIEL 9-23-75 STEP SPEC#=1 TIC= 100

09-22-75/SPEC# 7400/LM/ BACKGROUND SPECTRA

BASE	SUM
9762	40579

PEAK	I/BASE	MASS	TEST	PEAK	I/BASE	MASS	TEST
1	2.39%	14	-4	72	2.43%	341	2
2	5.44%	15	-3	73	2.60%	355	1
3	1.23%	16	-2	74	1.91%	356	1
4	10.03%	17	-1	75	0.74%	431	3
5	65.58%	18	-1				
6	7.30%	20	-0				
7	2.44%	26	0				
8	3.15%	27	0				
9	100.00%	28	0				
10	2.60%	29	0				
11	2.44%	29	0				
12	12.55%	32	0				
13	4.46%	36	0				
14	1.51%	38	-0				
15	2.89%	39	0				
16	4.35%	40	-1				
17	5.93%	41	-0				
18	1.72%	42	-0				
19	5.25%	43	-1				
20	19.04%	44	-1				
21	0.82%	48	0				
22	0.72%	50	-2				
23	1.14%	51	-3				
24	5.82%	55	-2				
25	2.05%	56	-2				
26	5.15%	57	-2				
27	0.87%	58	-2				
28	1.21%	64	-2				
29	1.84%	65	-1				
30	2.70%	67	-1				
31	2.11%	69	-1				
32	3.51%	70	-0				
33	2.98%	71	-0				
34	11.73%	73	-1				
35	1.31%	74	-1				
36	0.72%	75	-1				
37	1.30%	78	-0				
38	1.65%	79	-0				
39	2.73%	81	-0				
40	1.47%	82	0				
41	4.60%	83	0				
42	2.27%	85	0				
43	1.40%	87	-1				
44	0.90%	88	1				
45	3.25%	91	-0				
46	1.66%	93	-0				
47	2.00%	95	0				
48	4.55%	96	-1				
49	1.71%	109	-1				
50	1.41%	111	-1				
51	1.83%	117	-1				
52	0.99%	123	0				
53	3.52%	133	0				
54	0.91%	137	1				
55	1.63%	147	0				
56	1.61%	149	-0				
57	1.32%	150	-0				
58	0.76%	163	1				
59	1.51%	177	-1				
60	1.65%	191	-0				
61	1.38%	193	0				
62	32.17%	207	0				
63	4.93%	208	0				
64	3.47%	209	0				
65	1.92%	221	0				
66	1.19%	239	1				
67	1.03%	254	-1				
68	1.56%	267	0				
69	5.90%	281	1				
70	1.98%	282	1				
71	2.29%	283	0				

09-22-75/SPEC# 7416/LN/ GS-1 GC/MS G.SCHIEL 9-23-75

BASE 12604 SUM 99935

PEAK	I/BASE	MASS	TEST
1	3.85%	14	-3
2	5.30%	15	-2
3	2.23%	16	-1
4	1.54%	16	-1
5	15.00%	17	-1
6	61.51%	18	-0
7	3.04%	20	-0
8	1.26%	24	-0
9	5.31%	25	-0
10	1.44%	26	-1
11	3.71%	27	-0
12	100.00%	28	-0
13	2.06%	29	-0
14	1.27%	30	-0
15	19.12%	32	-0
16	3.54%	35	-0
17	10.33%	36	-0
18	1.76%	37	-0
19	2.64%	38	-0
20	0.65%	39	-0
21	2.46%	39	0
22	3.27%	40	-0
23	1.03%	40	0
24	4.00%	41	0
25	1.37%	42	0
26	5.32%	43	0
27	51.38%	44	0
28	1.00%	45	0
29	6.16%	47	-0
30	1.37%	48	-0
31	1.68%	49	-1
32	0.95%	51	-1
33	0.84%	53	-1
34	3.74%	55	-1
35	2.57%	56	-1
36	4.69%	57	-0
37	3.49%	59	-1
38	28.34%	60	-1
39	2.02%	61	-1
40	8.35%	62	-1
41	2.77%	65	-1
42	0.89%	65	-1
43	2.38%	66	-1
44	1.14%	67	-1
45	0.77%	68	-1
46	3.08%	69	-0
47	0.79%	70	-5
48	1.88%	70	-0
49	1.50%	71	-0
50	5.95%	73	-0
51	0.90%	74	-0
52	1.26%	75	-0
53	1.17%	77	-0
54	1.16%	81	0
55	1.36%	82	0
56	1.61%	83	0
57	1.10%	84	0
58	0.71%	85	-1
59	1.47%	85	0
60	2.37%	91	-0
61	0.71%	92	-0
62	5.59%	94	-1
63	62.13%	95	-2
64	5.53%	96	-2
65	54.17%	97	-2
66	1.48%	97	-0
67	2.30%	98	-2
68	5.67%	99	-3
69	0.76%	102	-1
70	1.13%	105	-0
71	0.96%	109	0
72	0.99%	111	0
73	1.02%	117	0
74	1.12%	122	0
75	0.85%	125	0
76	0.88%	126	0
77	0.77%	128	0
78	89.70%	130	-1
79	3.01%	131	-1
80	71.80%	132	-1
81	2.27%	133	-1
82	24.69%	134	-0
83	0.80%	135	1
84	2.47%	136	0
85	1.33%	145	0
86	1.49%	147	0
87	1.07%	149	0
88	0.80%	163	-1
89	0.53%	166	1
90	0.60%	171	-3
91	0.57%	184	0
92	1.02%	191	-0
93	0.81%	193	0
94	13.24%	207	1
95	2.30%	208	1
96	1.69%	209	1
97	1.36%	221	0
98	0.73%	241	0
99	1.27%	267	0
100	0.61%	268	1
101	0.80%	269	0
102	3.89%	281	0
103	1.32%	341	1
104	1.72%	355	1
105	0.57%	383	1

09-22-75/SPEC# 7413/LM/ 6S-1 GC/MS G.SZ.NIEL 9-23-75

BASE	SUM
18532	63393

PEAK	I/BASE	MASS	TEST
1	2.12%	14	-3
2	3.24%	15	-2
3	1.76%	16	-1
4	8.03%	17	-1
5	57.15%	18	-0
6	2.82%	20	-0
7	0.97%	22	-0
8	0.78%	25	-0
9	2.18%	26	-0
10	3.12%	27	-0
11	100.00%	28	-0
12	1.14%	29	0
13	1.40%	30	0
14	12.25%	32	0
15	4.79%	36	0
16	1.44%	38	0
17	1.82%	39	0
18	2.43%	40	0
19	2.55%	41	0
20	1.76%	42	0
21	3.94%	43	0
22	59.66%	44	0
23	1.00%	45	0
24	0.35%	51	-0
25	2.06%	55	0
26	1.68%	56	0
27	2.56%	57	0
28	2.34%	61	-0
29	0.67%	62	0
30	1.16%	63	-0
31	1.06%	64	-0
32	0.65%	67	0
33	0.38%	68	0
34	1.43%	69	-0
35	0.78%	70	-0
36	1.67%	71	-0
37	3.51%	73	-0
38	0.56%	75	-0
39	0.71%	77	0
40	0.52%	79	0
41	0.92%	81	0
42	0.94%	84	0
43	0.79%	85	0
44	0.42%	87	1
45	0.69%	89	-1
46	0.81%	91	-1
47	0.68%	94	-1
48	0.87%	95	-0
49	1.66%	96	-1
50	3.25%	97	-1
51	3.93%	99	-1
52	0.84%	111	0
53	0.38%	115	0
54	1.17%	117	-0
55	0.81%	119	-0
56	1.11%	133	0
57	0.96%	147	1
58	0.51%	151	0
59	0.45%	165	1
60	1.36%	191	-0
61	10.72%	207	0
62	1.29%	208	0
63	1.44%	209	0
64	0.57%	221	1
65	0.56%	268	1
66	2.46%	281	0
67	0.83%	282	0
68	0.87%	341	1
69	1.17%	355	-1
70	0.36%	369	-2

09-22-75/SPEC# 7427/LM/ GS-1 GC/MS G.SCHIEL 9-23-75

BASE	SUM
11794	43906

PEAK	I/BASE	MASS	TEST
1	1.99%	14	-3
2	4.22%	15	-1
3	0.99%	16	-1
4	1.07%	16	-1
5	12.10%	17	-1
6	52.31%	18	-0
7	4.64%	20	-0
8	1.38%	26	-1
9	2.61%	27	-1
10	100.00%	28	-0
11	2.52%	29	-0
12	0.64%	30	-0
13	10.96%	32	-0
14	4.34%	36	-0
15	0.66%	37	0
16	1.92%	38	-0
17	2.68%	39	0
18	2.84%	40	-0
19	4.68%	41	0
20	1.20%	42	0
21	4.08%	43	0
22	0.62%	44	-1
23	35.18%	44	-0
24	1.33%	45	-0
25	1.12%	47	-1
26	1.88%	54	-1
27	4.28%	55	-1
28	3.51%	56	-0
29	3.32%	57	-0
30	0.73%	59	-1
31	1.11%	64	3
32	0.89%	66	-1
33	1.55%	67	-1
34	0.99%	68	-1
35	2.67%	69	-0
36	1.66%	70	-0
37	1.23%	71	-0
38	8.58%	73	-0
39	0.83%	74	-0
40	1.01%	75	-0
41	0.85%	79	0
42	0.88%	80	0
43	1.56%	81	0
44	1.89%	82	0
45	2.00%	83	0
46	0.89%	84	0
47	1.51%	85	0
48	0.58%	91	-3
49	2.01%	91	-1
50	0.95%	93	-0
51	1.92%	94	-2
52	1.61%	95	-1
53	0.99%	95	-0
54	1.31%	96	-1
55	0.94%	96	3
56	1.17%	105	0
57	1.21%	109	0
58	0.74%	116	-5
59	0.85%	117	0
60	0.62%	124	-0
61	1.62%	129	-1
62	1.44%	130	-1
63	3.34%	131	-1
64	1.72%	132	-0
65	2.34%	133	0
66	0.72%	141	2
67	1.97%	147	0
68	1.27%	149	0
69	1.04%	151	0
70	0.64%	155	1
71	0.72%	156	0
72	1.67%	164	-0
73	0.55%	164	2
74	4.25%	166	-0
75	1.58%	168	0
76	0.71%	169	1
77	1.07%	191	0
78	1.01%	193	0
79	1.06%	200	-4
80	12.94%	207	0
81	2.73%	208	0
82	2.75%	209	0
83	0.76%	219	1
84	0.91%	221	1
85	1.21%	267	0
86	4.15%	281	0
87	0.87%	335	3
88	1.00%	355	0
89	1.00%	415	0

APPENDIX C

PITOT CALCULATIONS

Since the pitot readings were not made at the centroids of equal areas, some method of estimating the proper readings must be made. By plotting the square roots of the pitot readings versus distance from the port (shown in Figure C-1) and reading the profiles at the normal equal area centroids, an average value of the stack velocity can still be calculated using Eq. 2-2 of EPA Method 2.[1/] The readings from this method of approximation are given in Table C-I. The readings from the second axis are assumed to be equivalent. Due to the nearly flat velocity profile, the resulting average flow velocity error is < 5%, even though the wrong points were measured. Copies of the original data sheets are at the end of this section. After making the approximation for $\sqrt{\Delta p_{ave.}}$, all calculations were made using EPA Method 2.

1/ Federal Register, Vol. 36, No. 247, December 23, 1971.

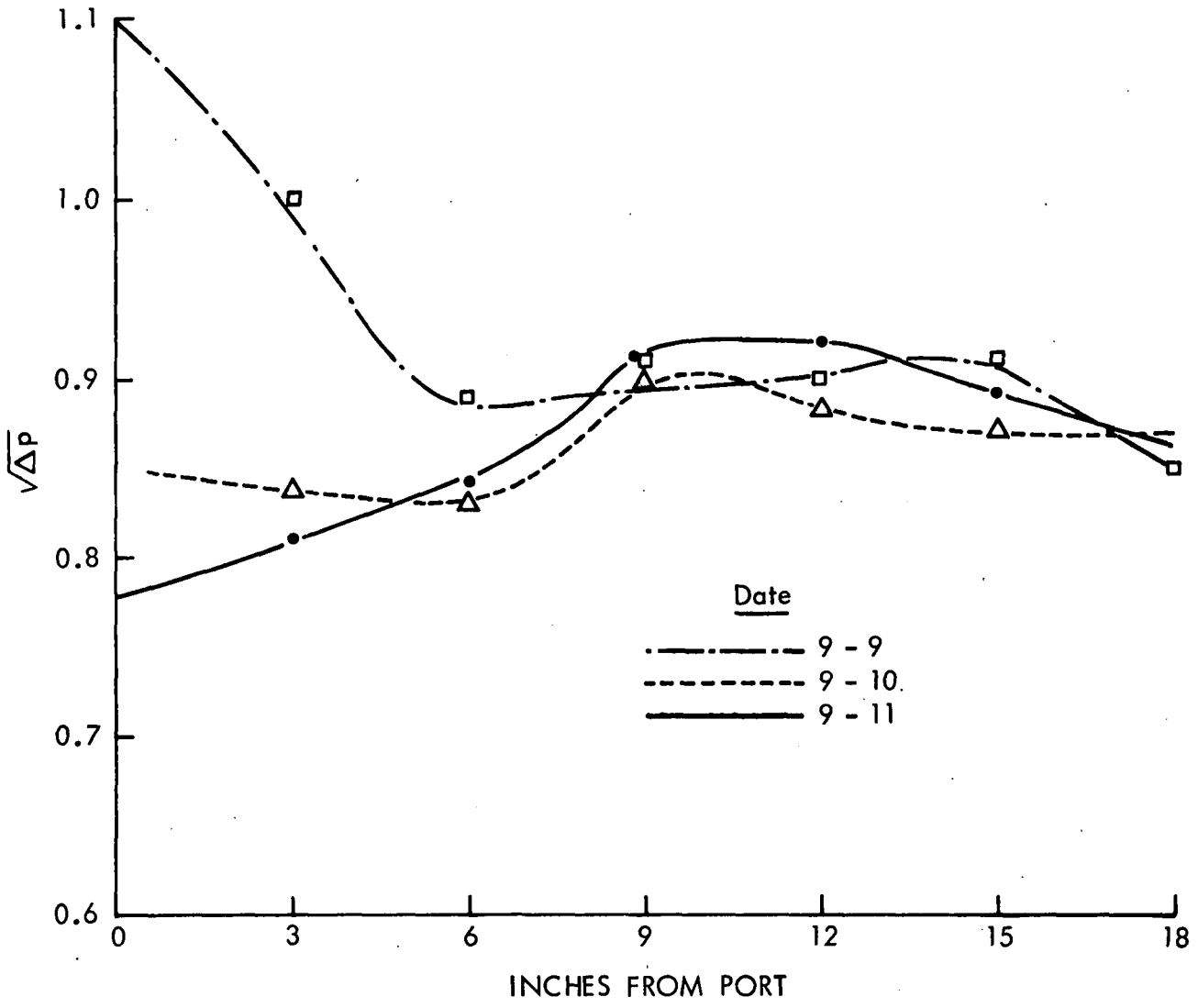


Figure C-1 - Approximate Velocity Profiles in Inlet Duct

TABLE C-I

$\sqrt{\Delta p}$ ESTIMATES FROM FIGURE C-1

Point No.	$\sqrt{\Delta p}$ from Figure C-1		
	9-9	9-10	9-11
1 (1.2 in.)	1.06	0.85	0.79
2 (4.5 in.)	0.92	0.83	0.83
3 (13.5 in.)	0.91	0.87	0.91
4 (16.8 in.)	0.87	0.87	0.87
$\sqrt{\Delta p_{ave.}}$	0.94	0.855	0.85

DAROS. B.C.

Project No. 3927-C Date of Work: 9-9-75 Work Performed by: COBB, G

Title or Purpose: TRAVERSE of INLET DUCT. Continued From:

Barometric Pressure = 30.20"

PITOT TRAVERSE

1.00	3"	INCHES
.80	6	
.83	9	
.81	12	
.82	15	
.72	18	

6.9" NEG PRESS

.88 INCHES H_2O

P_b = 29.5

Static Press. 6.9" H_2O

Gas Temp = AMBIENT 70° F

ASSUMPTIONS:

MW = AIR = 28.8

Project No. Date of Work: 9-10-75 Work Performed by:

Title or Purpose: Continued From:

PITOT TRAVERSE

~~.3~~	.70	3"
~~.79~~	.69	6"
~~.96~~	.81	9"
~~.79~~	.78	12"
~~.80~~	.76	15"

P = 29.95"

STATIC PRESSURE 6.5 in H_2O NEGATIVE

9-11-75

PITOT TRAVERSE

.65
.70
.82
.84
.79

6.6" H_2O NEGATIVE

Barometric 29.98

APPENDIX D

METHOD 106 - DETERMINATION OF VINYL CHLORIDE FROM STATIONARY SOURCES

METHOD 106--DETERMINATION OF VINYL CHLORIDE FROM STATIONARY SOURCES

INTRODUCTION

Performance of this method should not be attempted by persons unfamiliar with the operation of a gas chromatograph, nor by those who are unfamiliar with source sampling, as there are many details that are beyond the scope of this presentation. Care must be exercised to prevent exposure of sampling personnel to vinyl chloride, a carcinogen.

1. Principle and Applicability.

1.1 An integrated bag sample of stack gas containing vinyl chloride (chloroethene) is subjected to chromatographic analysis, using a flame ionization detector.

1.2 The method is applicable to the measurement of vinyl chloride in stack gases from both vinyl chloride and polyvinyl chloride manufacturing processes, except where the vinyl chloride is contained in particulate matter.

2. Range and Sensitivity.

The lower limit of detection will vary according to the chromatograph used. Values reported include 1×10^{-7} mg and 4×10^{-7} mg.

3. Interferences.

In the course of a study to identify the interference potential of several hydrocarbons associated with vinyl chloride, none were found to prevent resolution of the vinyl chloride peak with the Chromosorb 102[1] column. However, if resolution of the vinyl

[1]Mention of trade names on specific products does not constitute endorsement by the Environmental Protection Agency.

chloride peak is not satisfactory for a particular sample, then chromatograph parameters may be altered with prior approval of the Administrator.

4. Apparatus.

4.1 Sampling (Figure 1).

4.1.1 Probe--Stainless steel, Pyrex glass, or Teflon tubing according to stack temperature, each equipped with a glass wool plug to remove particulate matter.

4.1.2 Sample line--Teflon, 6.4 mm outside diameter, of sufficient length to connect probe to bag. A new unused piece is employed for each series of bag samples that constitutes an emission test.

4.1.3 Male (2) and female (2) stainless steel quick-connects, with ball checks (one pair without) located as shown in Figure 1.

4.1.4 Tedlar bags, 100 liter capacity--To contain sample.

4.1.5 Rigid leakproof containers for 4.1.4, with covering to protect contents from sunlight.

4.1.6 Needle valve--To adjust sample flow rate.

4.1.7 Pump--Leak-free. Minimum capacity 2 liters per minute.

4.1.8 Charcoal tube--To prevent admission of vinyl chloride to atmosphere in vicinity of samplers.

4.1.9 Flow meter--For observing sample flow rate; capable of measuring a flow range from 0.10 to 1.00 liters per minute.

4.1.10 Connecting tubing--Teflon, 6.4 mm outside diameter, to assemble sample train (Figure 1).

4.1.11 Pitot tube--Type S (or equivalent), attached to the probe so that the sampling flow rate can be regulated proportional to the stack gas velocity.

4.2 Sample recovery.

4.2.1 Tubing--Teflon, 6.4 mm outside diameter, to connect bag to gas chromatograph sample loop. A new unused piece is employed for each series of bag samples that constitutes an emission test, and is to be discarded upon conclusion of analysis of those bags.

4.3 Analysis.

4.3.1 Gas chromatograph--With flame ionization detector, potentiometric strip chart recorder and 1.0 to 5.0 ml heated sampling loop in automatic sample valve.

4.3.2 Chromatographic column--Stainless steel, 2.5 m x 6.4 mm, containing 80/100 mesh Chromosorb 102.

4.3.3 Flow meters (2)--Rotameter type, 0 to 100 ml/min capacity.

4.3.4 Gas regulators--For required gas cylinders.

4.3.5 Thermometer--Accurate to one degree centigrade, to measure temperature of heated sample loop at time of sample injection.

4.3.6 Barometer--Accurate to 5 mm Hg, to measure atmospheric pressure around gas chromatograph during sample analysis.

4.4 Calibration.

4.4.1 Tubing--Teflon, 6.4 mm outside diameter, separate pieces marked for each calibration concentration.

4.4.2 Tedlar bags--Sixteen-inch square size, separate bag marked for each calibration concentration.

4.4.3 Syringe--0.5 ml, gas tight.

4.4.4 Syringe--50 µl, gas tight.

4.4.5 Flow meter--Rotameter type, 0 to 1000 ml/min range accurate to ± 1%, to meter nitrogen in preparation of standard gas mixtures.

4.4.6 Stop watch--Of known accuracy, to time gas flow in preparation of standard gas mixtures.

5. Reagents. It is necessary that all reagents be of chromatographic grade.

5.1 Analysis.

5.1.1 Helium gas or nitrogen gas--Zero grade, for chromatographic carrier gas.

5.1.2 Hydrogen gas--Zero grade.

5.1.3 Oxygen gas--Zero grade.

5.2 Calibration.

5.2.1 Vinyl chloride, 99.9+%--For preparation of standard gas mixtures.

5.2.2 Calibration cylinders (3), optional--One each of 50, 10 and 5 ppm vinyl chloride in nitrogen with certified analysis.

5.2.3 Nitrogen gas--Zero grade, for preparation of standard gas mixtures.

6. Procedure.

6.1 Sampling. Assemble the sample train as in Figure 106-1. Perform a bag leak check according to Section 7.4. Observe that all connections between the bag and the probe are tight. Place the end of the probe at the centroid of the stack and start the pump with

the needle valve adjusted to yield a flow of 0.5 lpm. After a period of time sufficient to purge the line several times has elapsed, connect the vacuum line to the bag and evacuate the bag until the rotameter indicates no flow. Then reposition the sample and vacuum lines and begin the actual sampling, keeping the rate proportional to the stack velocity. Direct the gas exiting the rotameter away from sampling personnel. At the end of the sample period, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container. Protect the bag container from sunlight.

6.2 Sample Storage. Sample bags must be kept out of direct sunlight. When at all possible, analysis is to be performed within 24 hours of sample collection.

6.3 Sample recovery. With a new piece of Teflon tubing identified for that bag, connect a bag inlet valve to the gas chromatograph sample valve. Switch the valve to withdraw gas from the bag through the sample loop.

6.4 Analysis. Set the column temperature to 155°C, the detector temperature to 225°C, and the sample loop temperature to 70°C. When optimum hydrogen and oxygen flow rates have been determined, verify and maintain these flow rates during all chromatograph operations. Using zero helium or nitrogen as the carrier gas, establish a flow rate in the range consistent with the manufacturer's requirements for satisfactory detector operation. A flow rate of 40 ml/min has been shown to produce adequate separations. Observe the base line periodically and determine that the noise level has stabilized and that base line drift has ceased. Purge the sample loop for thirty

seconds at the rate of 100 ml/min, then activate the sample valve. Record the injection time (the position of the pen on the chart at the time of sample injection), the sample number, the sample loop temperature, the column temperature, carrier gas flow rate, chart speed and the attenuator setting. Record the laboratory pressure. From the chart, select the peak having the retention time corresponding to vinyl chloride, as determined in Section 7.2. Measure the peak area, A_m, by use of the automatic integrator. Record A_m and the retention time. Repeat the injection at least two times or until two consecutive vinyl chloride peaks do not vary in area more than 5%. The average value for these two areas will be used to compute the bag concentration.

7. Calibration and Standards.

7.1 Preparation of vinyl chloride standard gas mixtures. Evacuate a sixteen-inch square Tedlar bag that has passed a leak check (described in Section 7.4) and meter in 5.0 liters of nitrogen. While the bag is filling, use the 0.5 ml syringe to inject 250 µl of 99.9+% vinyl chloride through the wall of the bag. Upon withdrawing the syringe needle, immediately cover the resulting hole with a piece of adhesive tape. This gives a concentration of 50 ppm of vinyl chloride. In a like manner use the other syringe to prepare dilutions having 10 and 5 ppm vinyl chloride concentrations. Place each bag on a smooth surface and alternately depress opposite sides of the bag 50 times to further mix the gases.

7.2 Determination of vinyl chloride retention time. This section can be performed simultaneously with Section 7.3. Establish

chromatograph conditions identical with those in Section 6.3, above. Set attenuator to X 1 position. Flush the sampling loop with zero helium or nitrogen and activate the sample valve. Record the injection time, the sample loop temperature, the column temperature, the carrier gas flow rate, the chart speed and the attenuator setting. Record peaks and detector responses that occur in the absence of vinyl chloride. Maintain conditions. Flush the sample loop for 30 seconds at the rate of 100 ml/min with one of the vinyl chloride calibration mixtures and activate the sample valve. Record the injection time. Select the peak that corresponds to vinyl chloride. Measure the distance on the chart from the injection time to the time at which the peak maximum occurs. This quantity, divided by the chart speed, is defined as the retention time. Record.

7.3 Preparation of chromatograph calibration curve. Make a gas chromatographic measurement of each standard gas mixture (described in Section 7.1) using conditions identical with those listed in Section 6.3 above. Flush the sampling loop for 30 seconds at the rate of 100 ml/min with each standard gas mixture and activate the sample valve. Record C_c, the concentrations of vinyl chloride injected, the attenuator setting, chart speed, peak area, sample loop temperature, column temperature, carrier gas flow rate, and retention time. Record the laboratory pressure. Calculate A_c, the peak area multiplied by the attenuator setting. Repeat until two injection areas are within 5%, then plot those points vs C_c. When the other concentrations have been plotted, draw a smooth curve

through the points. Perform calibration daily, or before and after each set of bag samples, whichever is more frequent.

7.4 Tedlar bag leak checks. Before each use, make sure a bag is leak-free by checking it for leaks. To leak check, connect a water manometer and pressurize the bag to 5-10 cm H_2O (2-4 in. H_2O). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. (Note: An alternative leak check method is to pressurize the bag to 5-10 cm H_2O or 2-4 in. H_2O and allow to stand overnight. A deflated bag indicates a leak.)

8. Calculations.

8.1 Determine the sample peak area as follows:

$$A_c = A_m A_f \qquad \text{Equation 106-1}$$

where:

A_c = The sample peak area.

A_m = The measured peak area.

A_f = The attenuation factor.

8.2 Vinyl chloride concentrations. From the calibration curve described in Section 7.3, above, select the value of C_c that corresponds to A_c, the sample peak area. Calculate C_b as follows:

$$C_b = \frac{C_c P_r T_i}{P_i T_r} \qquad \text{Equation 106-2}$$

where:

C_b = The concentration of vinyl chloride in the bag sample in ppm.

C_c = The concentration of vinyl chloride indicated by the gas chromatograph, in ppm

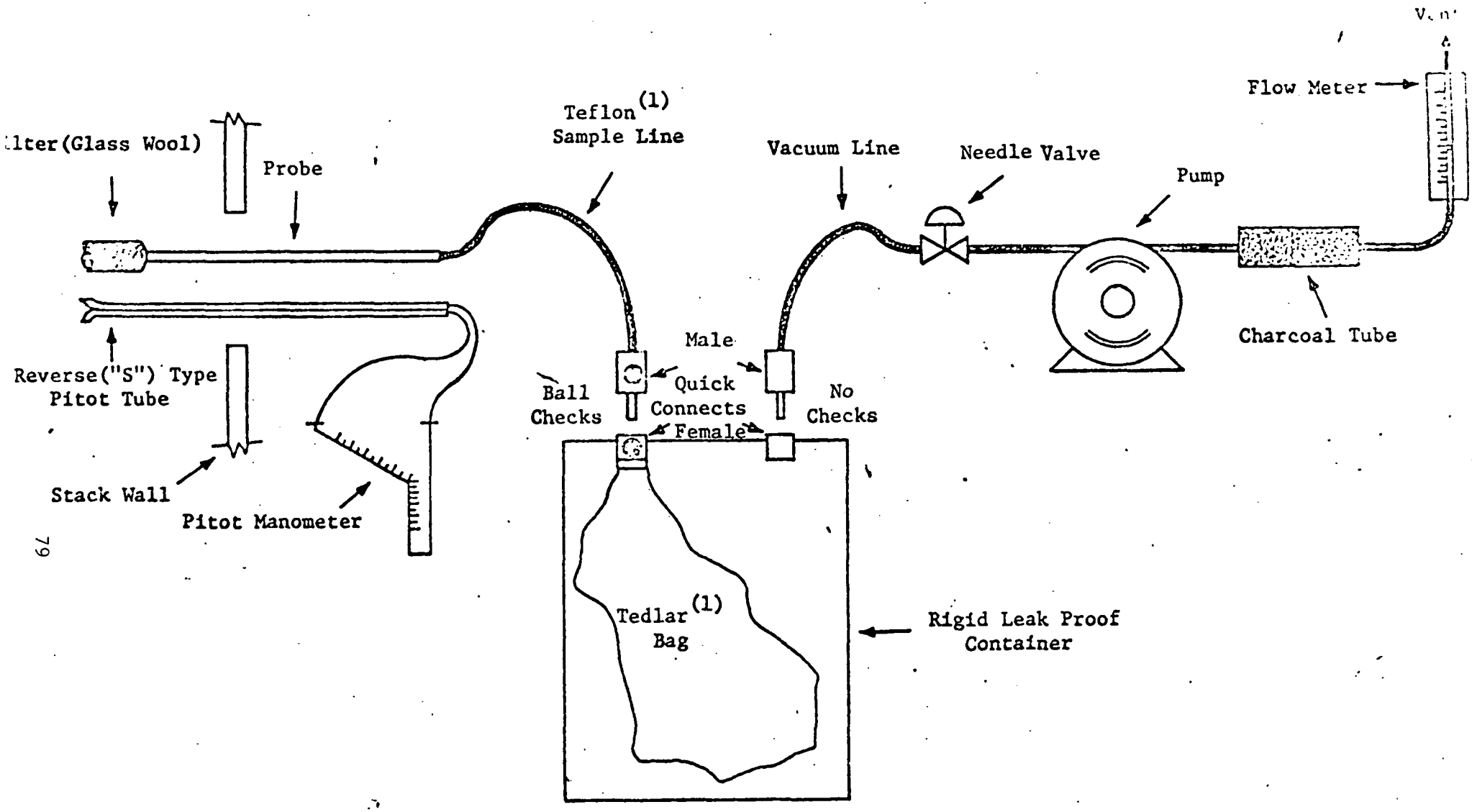


Figure 106-1. Integrated bag sampling train.

(1) Mention of trade names on specific products does not constitute endorsement by the Environmental Protection Agency.

P_r = The reference pressure, the laboratory pressure recorded during calibration, mm Hg.

T_f = The sample loop temperature on the absolute scale at the time of analysis, °K.

P_f = The laboratory pressure at time of analysis, mm Hg.

T_r = The reference temperature, the sample loop temperature recorded during calibration, °K.

9. References.

1. Brown, D. W., Loy, E. W. and Stephenson, M. H. "Vinyl Chloride Monitoring Near the B. F. Goodrich Chemical Company in Louisville, Kentucky." Region IV, U. S. Environmental Protection Agency, Surveillance and Analysis Division, Athens, Georgia, June 24, 1974.

2. "Evaluation of A Collection and Analytical Procedure for Vinyl Chloride in Air," by G. D. Clayton and Associates, December 13, 1974. EPA Contract No. 68-02-1408, Task Order No. 2, EPA Report No. 75-VCL-1.