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BACKSTREAMING FROM OIL DIFFUSION PUMPS

UNPUBLISHED PRELIMINARY DATA

Report No. IITRI-C6030-4
(Summary Report)

December 1964

National Aeronautics
and Space Administration
Washington, D. C.

(Prepared under Task Order No. NASr 65(08)
by the IIT Research Institute, Chicago, Illinois
W. M. Langdon, author)

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December 1, 1963, through November 30, 1964

Task Order No. NASr 65(08)
IITRI Project C6030

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FOREWORD

This is Report No. IITRI-C6030-4 (Summary Report) of IITRI Project C6030, Task Order No. NASr 65(08), entitled "Backstreaming from Oil Diffusion Pumps." The report covers the period of December 1, 1963, through November 30, 1964.

Personnel who contributed to this project include E. G. Fochtman, W. M. Langdon, R. H. Snow, D. R. Howard, V. R. Ivanuski, J. Frerichs, and H. J. O'Neill.

Data are recorded in IITRI Logbooks C14863, C14864, C14877, and C15234.

Respectfully submitted,

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BACKSTREAMING FROM OIL DIFFUSION PUMPS

20755

ABSTRACT

Previous work on backstreaming has been continued with more emphasis being placed on the mechanisms involved. The experimental variables were increased to include: pumps - fractionating and non-fractionating; baffles - chevron type, two bounce; one bounce, right angle elbow; oils - DC 705, Convalex 10, OS 124; operational procedures for fractionating effects; and mass spectrometer studies of mechanisms.

All vacuum stations which operated with -75°F baffles compared to a previous 40°F, gave high and varied backstreaming values and also high system pressures. Additionally, a pronounced pressure pulsing developed in systems with -75°F chevrons and not with -75°F elbow or with the room temperature chevron trap. Duplication of previous values (using -75°F cooling) were obtained with DC 705 (chevron) and Convalex 10 (elbow) after operation with room temperature cooling. Two stations with Convalex 10 (chevron type baffles) have not as yet been able to recover the low backstreaming values first obtained in the present runs. Widely different phenomena which cannot be completely isolated in our present test stands have been qualitatively characterized.

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I. INTRODUCTION

Space exploration requires extensive vacuum facilities to evaluate components and systems. All modern large-scale high vacuum facilities depend upon oil diffusion pumps. The critical nature of many component and systems studies requires very clean test facilities. This need for extreme cleanliness has directed attention to all details of the system and particularly to the contamination arising from oil backstreaming from the pump. It is the purpose of this program to evaluate the amounts and mechanisms of oil backstreaming.

Although many investigators have reported values for backstreaming from unbaffled systems very few published data are available for baffled systems. The most complete set of data for baffles systems was reported by Langdon and Fochtman in 1963.[13,14] They reported results on three oils and two trap configurations in terms of weight per unit area of the punp inlet and the development of a very sensitive analytical procedure. Typical results indicated backstreaming rates of 0.02 to 0.5×10^{-6} mg/(cm^2)(min). Operating pressures were generally 10^{-8} torr or lower.

The above work was limited as to conditions of operation and, while it defined backstreaming rates for a given set of conditions, it did not investigate the many variables which influence the rate. Parameters such as start-up and shutdown procedures, trap temperatures, oil type, baffle design, fore pressure, and fine pressure were not thoroughly investigated and the actual mechanism of backstreaming remain undefined.

This report summarizes the research of the past seven months* on the study of the mechanism of backstreaming. The report is organized so that the equipment features are thoroughly explained before presentation of results. The results are presented according to original planning on the program which indicated the need to investigate a number of mechanical arrangements and operational procedures. These results are briefly discussed.

II. EXPERIMENTAL

A. Equipment

The basic vacuum system consists of a 15 cfm mechanical fore pump, 6 inch oil diffusion pump, trap, and test dome. The original five stations used NRC-HS6-1500 pumps with water-cooled cold caps. Three stations had CVC-BC-61 chevron baffles, two had optically tight right-angle elbows. During this study we added a CVC-PMC-1440A, 6 inch non-fractionating diffusion pump with a "Dri-Cap" to one station. Station 3 (Figure 1) was modified by placing a 2 inch NRC H-2-P water-cooled oil diffusion pump between the 6 inch pump and the mechanical pump and replacing the chevron baffle with an NRC-HN6 baffle. A general view of the test facility is shown in Figure 2 and baffle and test head for Station 5 in Figure 3.

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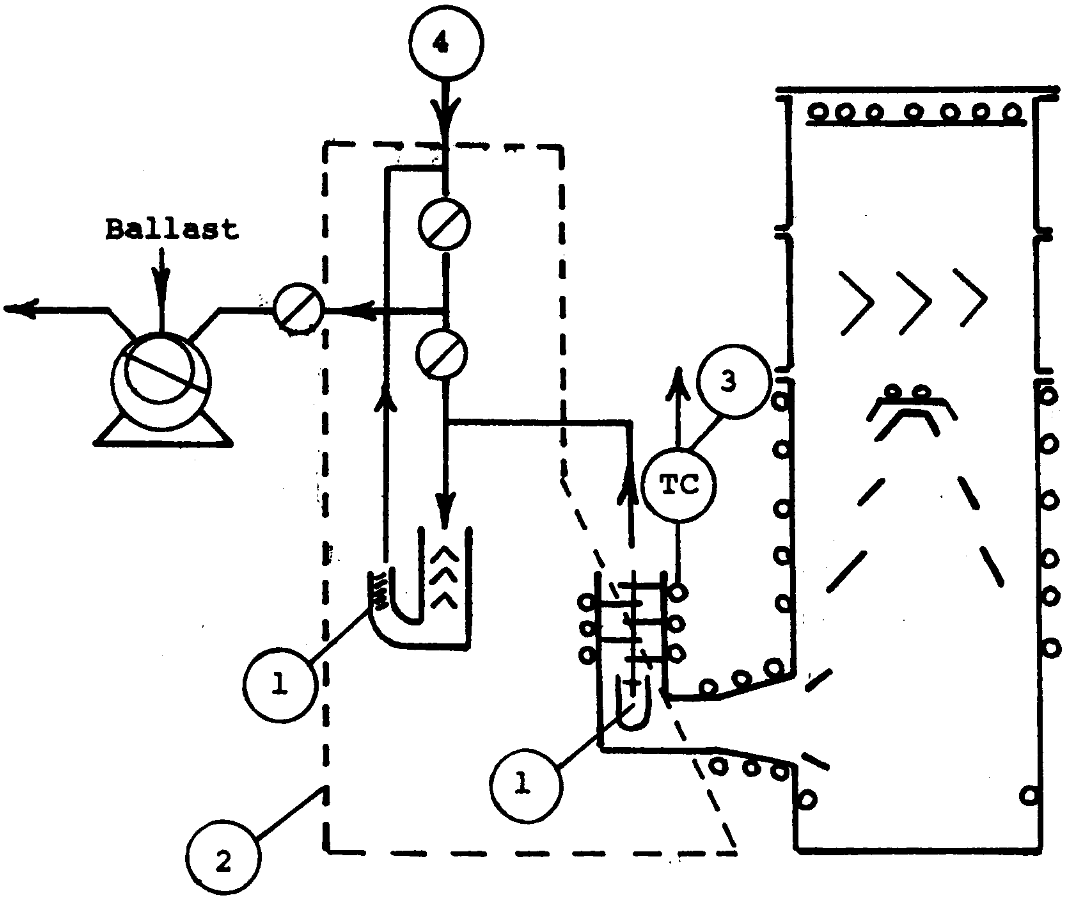
* All experimental work on this program was conducted during the last seven months of the twelve-month program.



1 Holdup Alembic

2 Dotted section Station 3 only

3 Exit temperature control on Station 1 only

4 Air leak, guard vacuum, quench coils

See Table 1 for schedule of pump, baffle and oil

Figure 1

SCHEMATIC OF STATION NO. 3

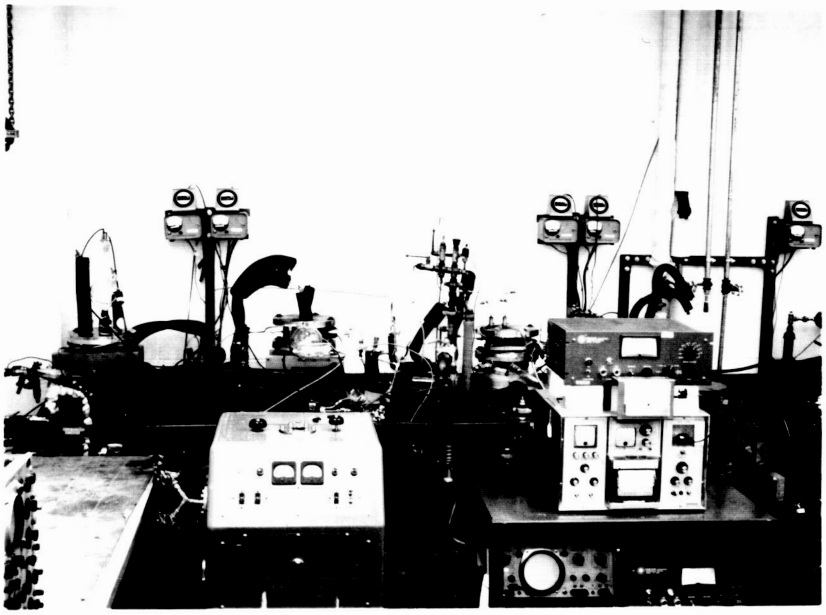


Figure 2

GENERAL VIEW OF TEST FACILITY

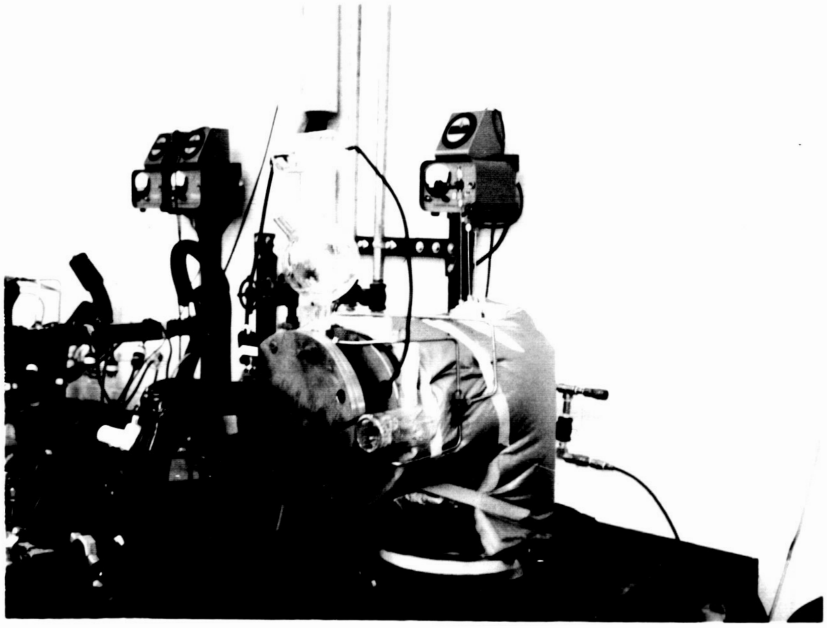


Figure 3

STATION NO. 5 RIGHT ANGLE ELBOW BAFFLE

Previously used test domes consisted of an oil collection plate set at 45° to the vertical. Experience has shown that, even under the most adverse conditions, very little oil is deposited on the collection plate and no drops large enough to drain from the plate are formed. In this work we have replaced these test domes with simple spools 4 inches high and equipped with two 1-inch diameter radial openings for the ionization gauge and the partial pressure analyzer (Figure 5). A typical internal, cooled collection plate is shown in Figure 4.

Variations of this equipment are desirable to evaluate certain parameters, therefore, the equipment for each specific study is outlined at the start of the pertinent discussion.

Ionization gauges were of the CVC-GlC-017 type; gauge controls were Varian 971-0003.

The partial pressure analyzer used for this work was the General Electric Partial Pressure Analyzer Model 22PC110, F&L-71. Permanent magnets of 2.5 and 5.0 Kilogauss were used. Complete experimental details concerned with this analyzer are given in Appendix B.

Oils used were DC 705 and Convalex 10. In some cases these oils had operated for 2 years, in other cases fresh oil was installed at the start of the study.

Summary of the experimental equipment is given in Table 1.

In order to improve leak testing sensitivity a ball valve was installed in the fore line so that all vent gas went through the CEC type 24-110 Helium Leak Detector.

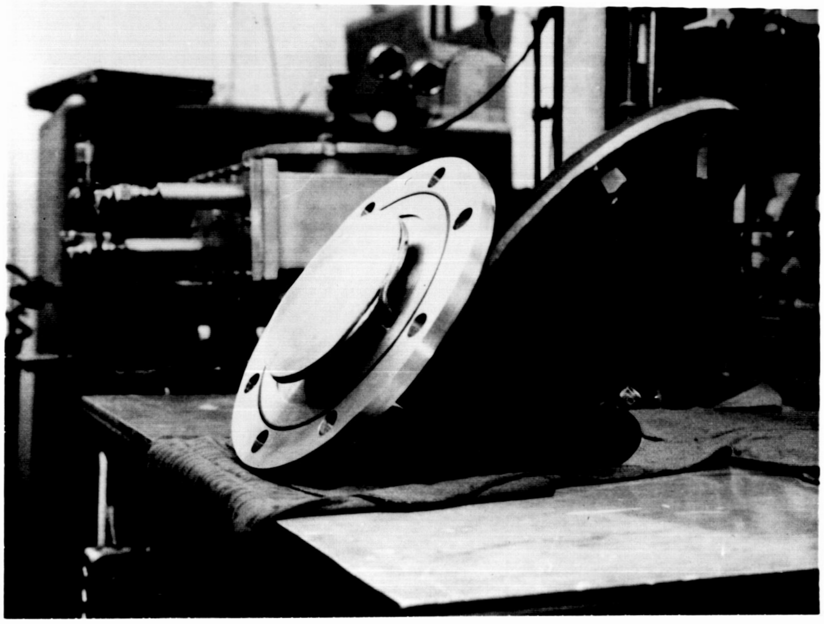


Figure 4

INTERNAL COLLECTION PLATE

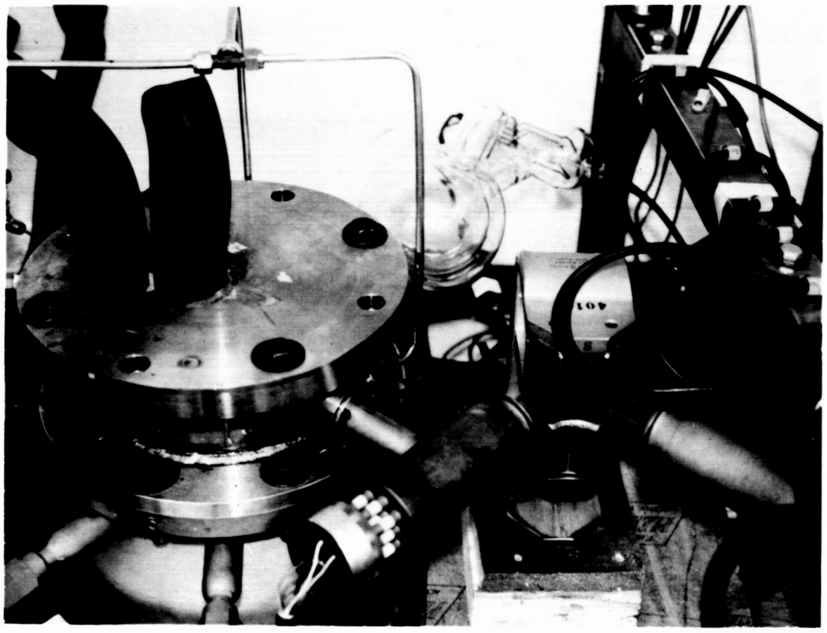


Figure 5

STATION NO. 3, TEST HEAD SHOWING TRAPPED GAUGE
AND MASS SPECTROMETER ANALYZER TUBE

Table 1

SCHEDULE OF EXPERIMENTAL EQUIPMENT

Station	Diffusion Pump	Trap	Oil
1	PMC-1440A	Chevron	Convalex 10, fresh
2	HS6-1500	Chevron	DC 705, 2 years old
3a	HS6-1500	Chevron	Convalex 10, 2 years old
3b	HS6-1500	Cryo	Convalex 10, 2 years old
3c	H2P HS6-1500	 Cryo	Convalex 10, fresh Convalex 10, 2 years old
4	HS6-1500	None	Station inactive
5	HS6-1500	Elbow	Convalex 10, fresh
6	HS6-1500	Chevron	OS-124, fresh

A mechanical refrigerator, which could provide cooled methanol at any temperature down to -100°F, was used to cool the traps and collection plates.

B. Analytical Procedures

The analytical procedure involves rinsing the oil deposits from the collection plate with methanol and determining the amount of oil from the ultraviolet absorption spectra. The amount of oil deposited can be calculated from:

$$W = V\ m\ A.U. \tag{1}$$

where

W = total weight of oil on collection plate, mg

V = total volume of rinse sample collected, ml

m = slope of calibration curve, mg of oil/ml/absorbance unit; 0.39 for DC 705 and 0.063 for Convalex 10

A.U. = absorbance unit determined in the Cary model 14 spectrophotometer at 258- and 278-mμ wavelength for DC 705 and Convalex 10, respectively.

The calibration data are given in Table 2. These new values of 0.39 and 0.063 compare very well with the previous values of 0.36 and 0.060.[13] Note that the original absorbance of 268 mμ for Convalex 10 should have been reported as 278 mμ. This discrepancy does not result in any error since the reading is always made from the correct peak by visual selection. The over-all accuracy of the analytical method is shown in Table 3. The values

Table 2

CALIBRATION DATA
FOR OIL CONCENTRATION VERSUS ULTRAVIOLET ABSORBANCE

Oil	Absorbance Band, mμ	Concentration, mg/ml	Absorbance Units	Slope
DC 705	258	0.0405	0.104	0.389
		0.101	0.258	0.391
		0.202	0.521	0.388
		0.304	0.768	0.396
			Avg.	0.391
Convalex 10	278	0.0412	0.662	0.0625
		0.0206	0.328	0.0628
		0.0103	0.163	0.0632
		0.0248	0.391	0.0634
			Avg.	0.0629

Table 3

ACCURACY OF ANALYTICAL METHOD

Oil	Wt. by Seeding, mg	Rinse Volume, ml	Absorbance	mg/ml[1]	Collection Wt., mg	Error	
						mg	%
DC 705	1.013	25	0.104	0.0407	1.017	+0.004	0.4
	0.912	25	0.099	0.0387	0.967	0.055	5
	0.678	25	0.06	0.0235	0.588	-0.110	-16
	0.304	25	0.04	0.015	0.390	0.086	14
Convalex 10	1.032	50	0.325	0.0205	1.037	+0.005	0.5
	0.775	25	0.475	0.0300	0.750	-0.025	-3
	0.310	25	0.190	0.012	0.300	-0.010	-3
	0.103	25	0.075	0.0047	0.117	0.014	9

[1] From Equation 1.

were obtained from a collection plate which was originally contaminated (seeded) with a known amount of oil in a very dilute solution of methanol.

Representative curves of a standard, an unknown, and a concentrated unknown are given in Figures 6, 7, and 8. The values of 0.286 and 0.694 A.U. (Figures 7 and 8) both correspond to 0.4×10^{-6} mg/(cm^2)(min) of backstreaming.

III. RESULTS

A. Backstreaming Measurements

1. Summary

A summary of backstreaming data are detailed in Table 4. Individual selected runs are abstracted in the sections below in order to assist in describing and analyzing their significance.

Initial operation of the test stations was to duplicate the results of previous work,[13,14] however, some conditions were changed to conform with the requirements of the present program. The major difference was that the baffle and the collection plate were maintained at -75°F instead of 40°F. Minor changes involved differences in the test head and collection plate.

In general the system pressures were higher than previously ($>1 \times 10^{-8}$ as compared to 4×10^{-9} torr previously). Initial samples were removed without warming the collection plate or trap.

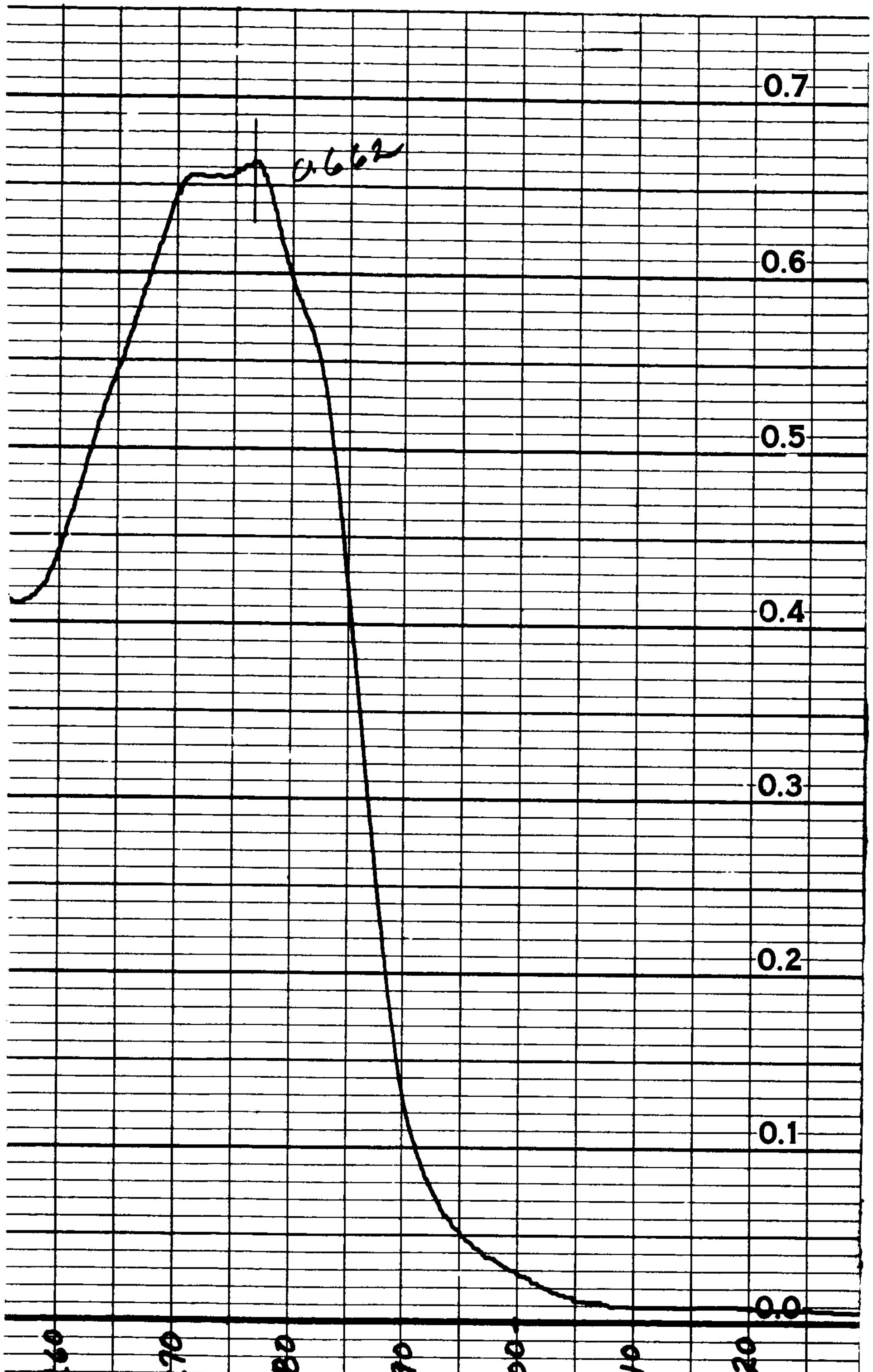


Figure 6

CALIBRATION CURVE FOR CONVALEX 10
(0.0412 mg/ml)

A.U. = .662
m = .0412/.662 = .0625

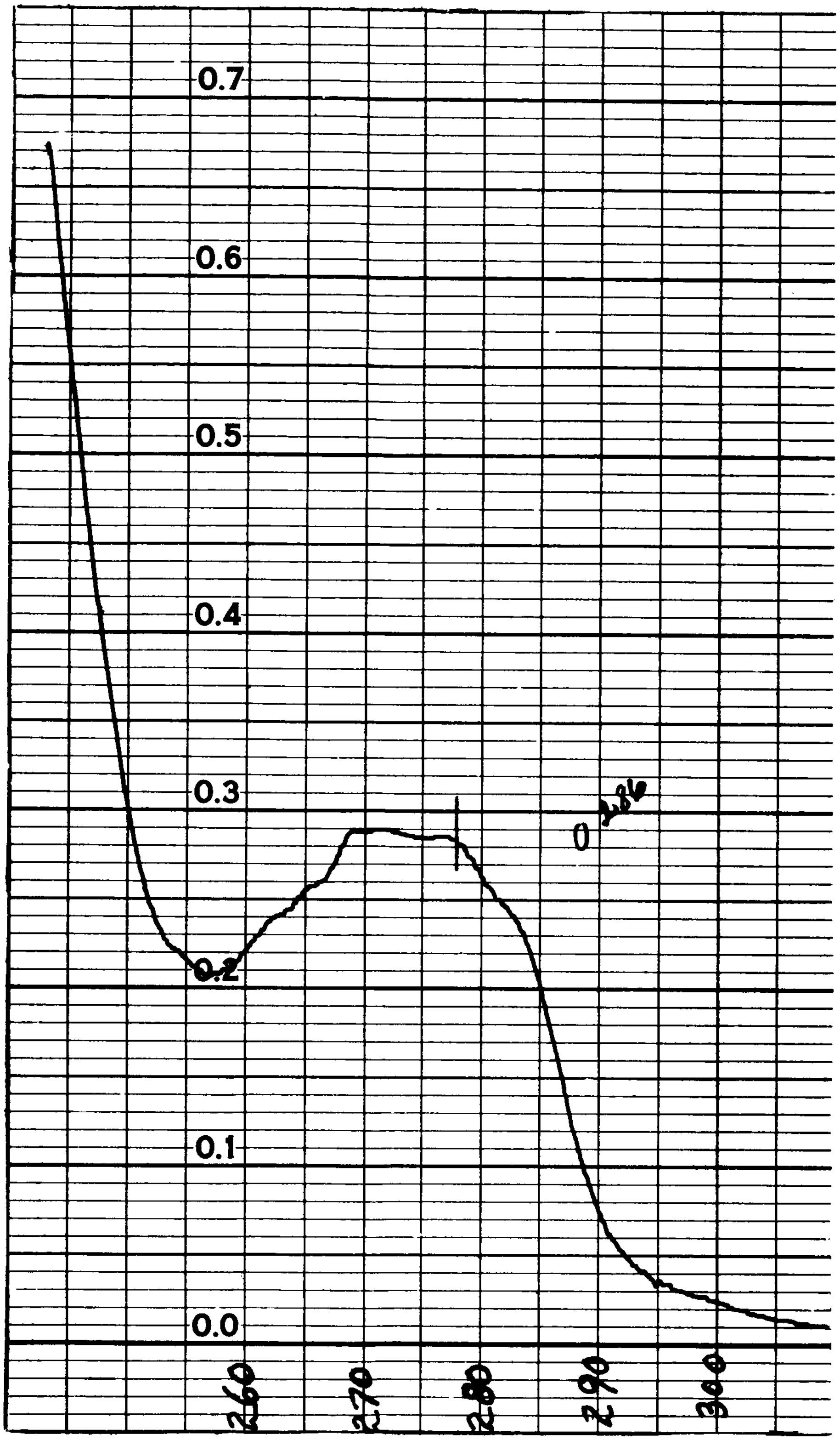


Figure 7

STATION NO. 3 - RUN 27, 163 HOURS AS TAKEN (50 ml)

A.U. = 0.286

W = (50)(.286)(.063) = .90 mg

B.S. = $(.9)/\left[(163)(60)(\pi/4)(6.5^2)(2.54^2)\right]$

= $.43 \times 10^{-6}$ mg/(cm^2)(min)

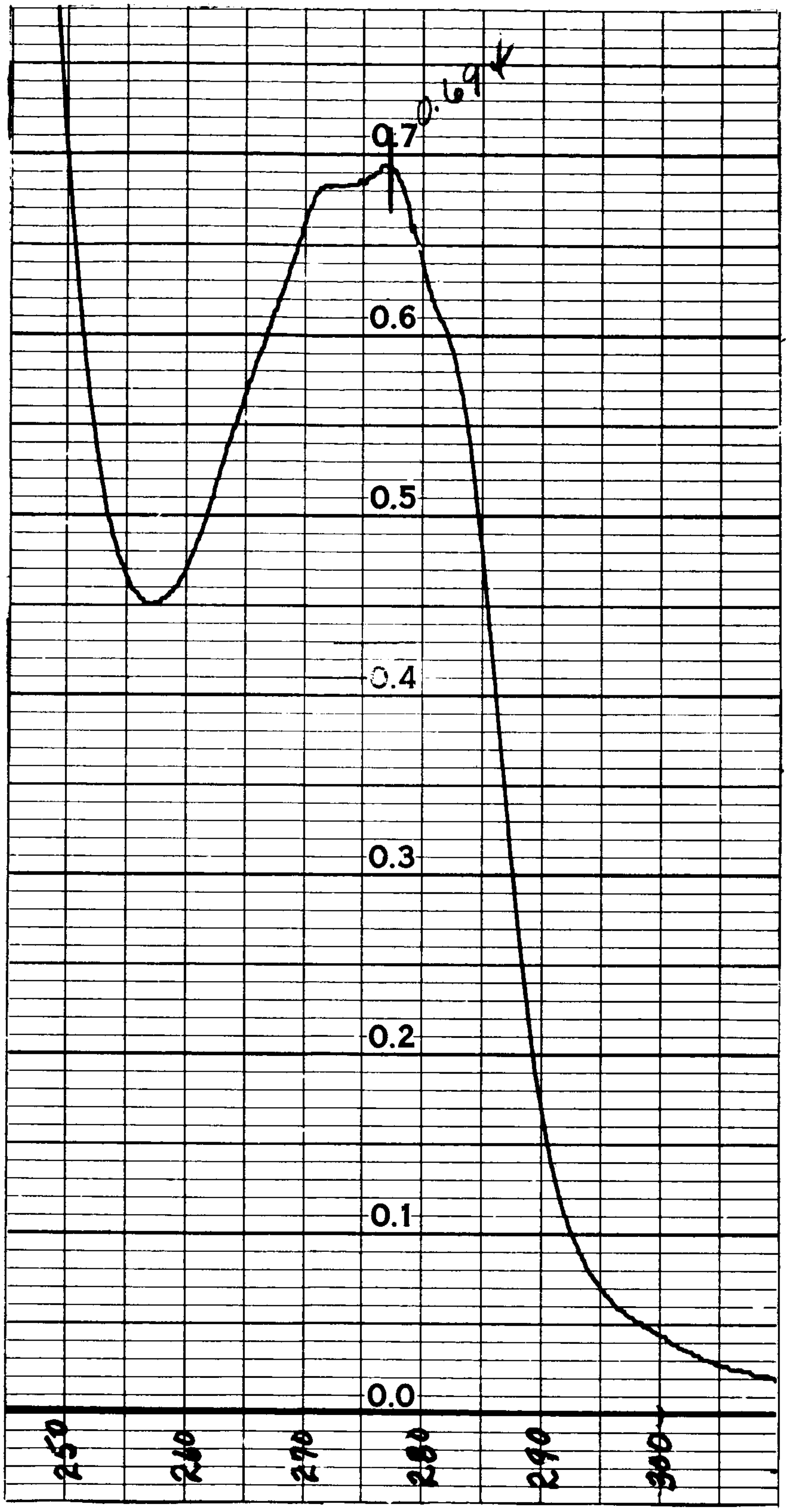


Figure 8

STATION NO. 3 - RUN 27, CONCENTRATED 2.5X

A.U. = .694
W = (50/2.5)(.694)(.063) = .88 mg
B.S. = Same as Figure 7

Table 4

SUMMARY OF BACKSTREAMING DATA

Run No.	Run, hrs	Fore Pressure, torr	System Pressure, torr		Cooling Temp., °F		Backstreaming, mg/(cm^2)(min)	Remarks
			Room Temp.	LN$_2$	Baffle	Plate		
Station No. 1, PMC 1440A, BC-61, Fresh Convalex 10								
26	162	2 x 10^{-2}	2 x 10^{-7}		-75	-75	.2 x 10^{-6}	
31	360	2 x 10^{-2}	5 x 10^{-8}		-75	-75	.3 x 10^{-6}	
51	86	0	Glass cover plate.		RT	RT	.5 x 10^{-6}	No pressure reading.
52	21	8 x 10^{-3}	4 x 10^{-7}		-100	-100	.4 x 10^{-6}	Pulsing first noticed.
55	92	0	4 x 10^{-6}		RT/200	-75	(7 x 10^{-6})	11.5 x 10^{-6} by wt.
59	121	4 x 10^{-3}	2 x 10^{-6}		RT/200	-75	(1 x 10^{-6})	
9/24	Refrigeration off - no pulses at room temperature, immediate and sharp pulses with cooling.							
9/29	163	9 x 10^{-3}	2 x 10^{-6}	8 x 10^{-9}	RT, -75	RT, -75		
9/30	Exit water thermostated to 190°F for all subsequent runs. Pulsing here and subsequently.							
65	23	2 x 10^{-2}	5 x 10^{-7}		-75	-75	.4 x 10^{-6}	
67	17	4 x 10^{-2}	2 x 10^{-5}		RT	-75	(4 x 10^{-6})	Attempt to purify oil.
69	21	2 x 10^{-2}	7 x 10^{-6}	2 x 10^{-8}	RT	-75	(2 x 10^{-6})	Attempt to purify oil.
71	67	4 x 10^{-2}	5 x 10^{-6}	7 x 10^{-8}	RT	-75	(3 x 10^{-6})	Attempt to purify oil.
74	23	5 x 10^{-2}	4 x 10^{-6}	7 x 10^{-8}	RT	-75	(.9 x 10^{-6})	Attempt to purify oil.
76	22	5 x 10^{-2}	3 x 10^{-6}	2 x 10^{-8}	RT	-75	(1 x 10^{-6})	Attempt to purify oil.
10/23	Fore pressure reduced to below 1 x 10^{-3}.							
81	115	0	7 x 10^{-7}	5 x 10^{-7}	RT	-75	(1 x 10^{-6})	
84	24	0	9 x 10^{-7}	5 x 10^{-7}	RT	-75	(1.2 x 10^{-6})	
11/5	160	9 x 10^{-4}	5 x 10^{-7}	4 x 10^{-9}	-75	-75		
Station No. 2, HS6-1500, BC-61, Used DC 705								
14	144	1 x 10^{-2}	5 x 10^{-8}		-75	-75	2 x 10^{-6}	
17	206	2 x 10^{-2}	4 x 10^{-8}		-75	-75		
21	71	2 x 10^{-2}	1 x 10^{-7}		-75	-75	100 x 10^{-6}	Emergency shutdown.
27	162	2 x 10^{-2}	1 x 10^{-6}		-75	-75	2 x 10^{-6}	
32	362	2 x 10^{-2}	1 x 10^{-6}		-75	-75	2 x 10^{-6}	
36	120	2 x 10^{-2}	6 x 10^{-7}		-75	-75	6 x 10^{-6}	
40	74	2 x 10^{-2}	9 x 10^{-8}		-75	-75	7 x 10^{-6}	
43	138	2 x 10^{-2}	4 x 10^{-8}		-75	-75	1 x 10^{-6}	
46	139	2 x 10^{-2}	5 x 10^{-8}		-99	-99	6 x 10^{-6}	Pulsing here and preceding.
8/28-9/2	166	2 x 10^{-2}	3 x 10^{-8}		RT	RT		No pulsing.
49	91	2 x 10^{-2}	4 x 10^{-8}		-100	-100	.3 x 10^{-6}	Very infrequent pulsing.

Table 4 (cont'd)

Run No.	Run, hrs	Fore Pressure, torr	System Pressure, torr		Cooling Temp., °F		Backstreaming, mg/(cm^2) (min)	Remarks
			Room Temp.	LN$_2$	Baffle	Plate		
53	27	2 x 10^{-2}	3 x 10^{-7}		-100	-100	1 x 10^{-6}	Spectrum NG.
56	92	2 x 10^{-2}	2 x 10^{-7}		RT	212/-75	(20 x 10^{-6})	20 x 10^{-6} by wt.
60	121	2 x 10^{-2}	9 x 10^{-8}		-75	-75	.7 x 10^{-6}	
63	49	2 x 10^{-2}	7 x 10^{-8}		RT	RT	1 x 10^{-6}	
72	69	2 x 10^{-2}	7 x 10^{-8}		-75	-75	.3 x 10^{-6}	
77	49	2 x 10^{-2}	3 x 10^{-8}		RT	-75	(3 x 10^{-6})	
79	20	2 x 10^{-2}	1 x 10^{-7}		RT	-75	(4 x 10^{-6})	
82	90	2 x 10^{-2}	1 x 10^{-7}	1 x 10^{-7}	-75	RT	.7 x 10^{-6}	
11/3	188	2 x 10^{-2}	8 x 10^{-8}	5 x 10^{-8}	-75	RT		No detectable pulsing.
11/11	379	2 x 10^{-2}	7 x 10^{-8}	4 x 10^{-8}	-75	RT		No detectable pulsing.
11/18	526	2 x 10^{-2}	8 x 10^{-8}	4 x 10^{-8}	-75	RT		No detectable pulsing.
Station No. 3, HS6-1500, BC-61, Used Convalex 10								
15	96	2 x 10^{-2}	3 x 10^{-7}		-75	-75	.5 x 10^{-6}	
18	209	3 x 10^{-2}	8 x 10^{-7}		-75	-75		
22	74	2 x 10^{-2}	1 x 10^{-7}		-75	-75	100 x 10^{-6}	Emergency shutdown.
28	163	2 x 10^{-2}	3 x 10^{-7}		-75	-75	.4 x 10^{-6}	
33	361	1 x 10^{-2}	1 x 10^{-7}		-75	-75	.2 x 10^{-6}	
HS6-1500, HN-6, Used Convalex 10								
37	136	2 x 10^{-2}	3 x 10^{-8}		-75	-75	.3 x 10^{-6}	
41	140	1 x 10^{-2}	3 x 10^{-8}		-75	-75	.4 x 10^{-6}	
44	138	1 x 10^{-2}	2 x 10^{-8}		-75	-75	.7 x 10^{-6}	
47	104	1 x 10^{-2}	3 x 10^{-8}		-97	-97	1 x 10^{-6}	Pulses first observed
50	41	2 x 10^{-2}	2 x 10^{-8}		-100	-100	.8 x 10^{-6}	here and in all sub-
54	24	2 x 10^{-2}	3 x 10^{-8}		-100	-100	.8 x 10^{-6}	sequent runs.
57	91	2 x 10^{-2}	2 x 10^{-6}		RT	212/-100	(28 x 10^{-6})	29 x 10^{-6} by wt.
61	120	2 x 10^{-2}	2 x 10^{-6}		-75	-75	3 x 10^{-6}	
62	47	2 x 10^{-2}	2 x 10^{-7}		-75	-75	2 x 10^{-6}	
64	60	2 x 10^{-2}	1 x 10^{-7}		RT	RT	(.9 x 10^{-6})	
66	27	2 x 10^{-2}	1 x 10^{-7}	5 x 10^{-8}	RT	-75	(4 x 10^{-6})	Attempt to purify oil.
68	17	2 x 10^{-2}	1 x 10^{-7}		RT	-75	(.7 x 10^{-6})	Attempt to purify oil.
70	22	2 x 10^{-2}	1 x 10^{-7}	6 x 10^{-8}	RT	-75	(4 x 10^{-6})	Attempt to purify oil.
73	66	2 x 10^{-2}	5 x 10^{-6}	3 x 10^{-8}	RT	-75	(.9 x 10^{-6})	Attempt to purify oil.
75	23	2 x 10^{-2}	1 x 10^{-7}		RT	-75	(3 x 10^{-6})	Attempt to purify oil.
78	20	2 x 10^{-2}	7 x 10^{-8}	2 x 10^{-8}	RT	RT	(1 x 10^{-6})	
80	20	2 x 10^{-2}	9 x 10^{-8}	5 x 10^{-8}	-75	RT	(1 x 10^{-6})	
83	9	2 x 10^{-2}	6 x 10^{-8}	3 x 10^{-8}	-75	RT	(.7 x 10^{-6})	
85	24	2 x 10^{-2}	8 x 10^{-8}	4 x 10^{-8}	-75	RT	1.2 x 10^{-6}	
Added H-2-P Pump to Fore Line								
10/30	17	8 x 10^{-4}	2 x 10^{-7}	6 x 10^{-8}	RT	RT		
11/2	91	8 x 10^{-4}	1 x 10^{-7}	3 x 10^{-8}	RT	RT		
Added Mass Spectrometer to System								
11/10	283	8 x 10^{-4}	2 x 10^{-7}	5 x 10^{-8}	RT	RT		Found leak in HN-6

Table 4 (cont'd)

Run No.	Run, hrs	Fore Pressure, torr	System Pressure, torr Room Temp.		Cooling Temp., °F Baffle	Plate	Backstreaming, mg/(cm^2)(min)	Remarks
				LN$_2$				
Station No. 4, HS6-1500, Right Angle Elbow, Used DC 705								
16	120	$.9 \times 10^{-2}$	5×10^{-8}		-75	-75	3×10^{-6}	Poor spectrum.
19	209		4×10^{-8}		-75	-75	4×10^{-6}	Poor spectrum.
23	115	1×10^{-2}	6×10^{-8}		-75	-75	$.8 \times 10^{-6}$	Poor spectrum.
29	62	1×10^{-2}	4×10^{-8}		-75	-75	4×10^{-6}	Poor spectrum.
34	36	1×10^{-2}	6×10^{-8}		-75	-75	$.8 \times 10^{-6}$	Good spectrum.
Station No. 5, HS6-1500, Right Angle Elbow, Fresh Convalex 10								
20	45	8×10^{-3}	4×10^{-8}		-75	-75		Emergency shutdown.
24	141	3×10^{-3}	1×10^{-8}		-75	-75		
25	255				-75	-75	$.2 \times 10^{-6}$	
30	146	3×10^{-3}	4×10^{-8}		-75	-75	$.2 \times 10^{-6}$	
86	165	2×10^{-3}	9×10^{-8}	8×10^{-8}	-75	-75	$.1 \times 10^{-6}$	Start up run.
88	141	2×10^{-3}	7×10^{-8}	6×10^{-8}	-75	-75	$.05 \times 10^{-6}$	Extremely infrequent pulsing.
89	17	2×10^{-3}	2×10^{-8}	4×10^{-9}	-75	-75		Shows pressure improvement.
		Mass Spectrometer Added to System						
38	382	5×10^{-3}	4×10^{-8}		-75	-75	$.3 \times 10^{-6}$	
45	138	3×10^{-3}	2×10^{-7}		-75	-75	1×10^{-6}	
56	95	3×10^{-3}	8×10^{-9}		RT/-75	212/-75	6×10^{-6}	8×10^{-6} (by wt).
Station No. 6, HS6-1500, BC-61 (Room Temperature), Fresh OS 124								
9/30	1421	4×10^{-2}	6×10^{-9}		+45	RT		

In general backstreaming values were high and showed fairly large variations. In Station 1 and Station 3 with baffle HN-6, the values appeared to increase with each subsequent run. In addition a new phenomenon, pulsating of the system pressure, was noted in Stations 1, 2, and 3 (chevron or cryo baffled systems). No pulsations were noted on Station 5 (elbow baffle) or on Station 6 with room temperature chevron baffle. Investigation revealed that the pulses, in the pressure range of 5 x 10^{-7} torr, were a result of moisture contamination of the system. The moisture contamination was eliminated by flooding the system with dry nitrogen and allowing both trap and collection plate to warm up before opening the system. Pulsing of the pressure continued in the 1 x 10^{-8} torr pressure range.

Stations 1, 2, 3 and 5 were then operated 1 week with room temperature baffles and collection plates. Pulsing phenomena continued with Stations 1 and 3 but stopped in Station 2. The system pressure of both Stations 2 and 5 showed improvement. Other variations shown in the tables and discussed in other sections were resorted to without improving Stations 1 and 3.

2. Duplication of Previous Results

Specific run data made with both baffle and collection plate at -75°F and which duplicate previous work are given in Table 5.

Table 5

DUPLICATION OF PREVIOUS BACKSTREAMING TESTS

Run No.	System Pressure Room Temp.	Backstreaming, mg/(cm^2)(min)	Ratio Present/Previous	Remarks
Station No. 2, HS6-1500, BC-61, DC 705 (used 2 years) Ran baffles at room temperature for 1 week then both baffle and collection plate at -75 or -100°F				
49	4×10^{-8}	$.3 \times 10^{-6}$	.8	
53	3×10^{-7}	1.1×10^{-6}	(2.2)	Poor spectrum
60	9×10^{-8}	$.6 \times 10^{-6}$	1.3	
72	7×10^{-8}	$.3 \times 10^{-6}$	.5	
			1.2 Ave.	
Station No. 3, HS6-1500, BC-61, Convalex 10 (used 2 years)				
15	3×10^{-7}	$.6 \times 10^{-6}$	6	
28	3×10^{-7}	$.4 \times 10^{-6}$	4	
33	1×10^{-7}	$.2 \times 10^{-6}$	2	
			4 Ave.	
Station No. 4, HS6-1500, Right Angle Elbow, DC 705 (used 2 years)				
34	6×10^{-9}	$.8 \times 10^{-6}$	8	
Station No. 5, HS6-1500, Right Angle Elbow, Convalex 10 (using new Convalex 10)				
25		$.2 \times 10^{-6}$	9	
30	4×10^{-8}	$.2 \times 10^{-6}$	9	
86	9×10^{-8}	$.1 \times 10^{-6}$	5	
88	7×10^{-8}	$.05 \times 10^{-6}$	3	

Station 1 is not included since it is a non-fractionating* pump without an ejector stage which is different from the previous work. Station 1 is discussed in Section D.

When Station 2 was then operated with a -75°F baffle and collection plate no pulses were noted at 10^{-7} - 10^{-8} torr and the backstreaming results duplicated those obtained previously.

In Station No. 3, with BC-61 baffle, backstreaming rates averaged 4X those obtained previously, however, the system pressure was only 10^{-7} torr. The decreasing values indicate the system is gradually cleaning up. The last value (run 33) of $.2 \times 10^{-6}$ mg/(cm^2)(min) was considered a sufficient check and the system was changed over to the HN-6 baffle.

Station 5 could not be tested until recently due to the mass spectrometer tests. The recent values, runs 86 and 88 are .1 and $.05 \times 10^{-6}$ mg/(cm^2)(min), respectively. The latter value is more valid because of experimental conditions and is a close check to $.02 \times 10^{-6}$ mg/(cm^2)(min) obtained previously.

A very recent investigation[5] has just been concluded with very similar equipment, procedures and oils as Station 2 and 3. The main differences involved the use of liquid nitrogen cooling throughout and the removal of samples while the test system was still cold. The average backstreaming values referred to an 11 in. I.D. baffle chamber were $.7 \times 10^{-6}$ and 6×10^{-6} mg/(cm^2)(min) for Convalex 10 and DC 705, respectively. No mention of pulsing phenomena is noted. These values when referred to a 7 in. pump

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* Conventionally fractionating refers to a compartmented vaporizing chamber.

flange, are >10X the previous and present values considered representative here. Similar measurements using an HS32-50,000 pump with DC 705 and a newly designed chevron baffle gave values of .07 to .7 x 10^{-6} mg/(cm^2)(min). The average value, 0.5 x 10^{-7} mg/(cm^2)(min) is the value obtained in this work with Station 2. The values, however, appear to be decreasing significantly with subsequent runs and may be due to conditioning or light end removal in the samples.

It is of interest to compare the present backstreaming values with those given in a recent theoretical study.[10] Since the latter values are several orders of magnitude lower than any obtained here only a very quantitative comparison is warranted. Taking their worst example of case A, the oil transfer through a single bounce right angle elbow baffle, a backstreaming value of 0.1 x 10^{-6} mg/(cm^2)(min) (equivalent to their value 1 x 10^{-6} g/sec in a 36-inch elbow) would require a sticking coefficient of $<$.9 for a system oil pressure of 10^{-7} torr. While there is considerable doubt about the true value of this coefficient, it is usually considered to be close to unity. Considering the factor of two bounce chevron trap alone which affects the penetration probability by several orders of magnitude, this type of mechanism does not account for the present values.

B. Backstreaming Rates for Various Trap Types

Backstreaming data for the chevron and right angle elbow appear valid; however, results with the cryo baffle were not valid due to a minute leak in the baffle. This leak could only be detected with the partial pressure analyzer at system pressures of 3-5 x 10^{-8} torr and when the baffle was cold. The limited tests with Station 5 show the same marked superiority of the 1 bounce elbow over the two bounce chevron trap. The variation in values appears to be related to the pressure pulses in the system, since this was not experienced previously and when it was eliminated in Station 2 during this work the results duplicated the previous results.

C. Backstreaming as a Function of Distance

Results of backstreaming were too variable to attempt these measurements.

D. Backstreaming from Non-Fractionating Pump

Considerable difficulty has been experienced in bringing this pump to optimum operating conditions. Initial operation with both the baffle and the collection plate at -75°F gave pressures of about 1-10 x 10^{-7} torr. Even after 360 hours of operation the pressure only reached 5 x 10^{-8} torr measured with a liquid nitrogen trapped gauge. Various means of reducing the pressure were sought. The heat input was varied from 75% to 125% of the rated amount with no apparent effect. The temperature of the exit water was regulated to 190°F and a series of light end fractions stripped from the oil. No significant decrease in pressure or in the pulsing phenomena was observed. Next the fore pressure was reduced to less than a micron, while still maintaining the exit water at 190°F. This reduced the system pressure to 5 x 10^{-7} torr with an untrapped gauge and 4 x 10^{-9} torr with a liquid nitrogen trapped gauge.

Backstreaming during early operations was 0.2 x 10^{-6} mg/(cm^2)(min) which is the same as the initial runs of this investigation with the fractionating pump.

It was determined that the oil in this pump contained considerably more light ends than any other station using Convalex 10. This is discussed more fully in the section on oil stability.

E. Side Stream Purification

Although present diffusion pump oils are very stable, a limited amount of degradation always occurs.[2] The successful application of the modern oils to produce ultrahigh vacuums with only moderate or no cooling of the trap requires that the pump remove these degradation products adequately during continuous operation. In many cases a fresh pump oil requires a break-in period of a week or more before it will start to produce below 10^{-7} torr with a room temperature baffle. The effect of light ends on ultimate pressure is not the main problem since this can always be eliminated by cooled baffles. The more important problem is their influence on the amount of backstreaming. It was planned to study the magnitude of this problem by the use of a continuous refining system on a selected station. However, the unexpected phenomena of high and varied backstreaming values accompanied by pulsed gauge readings, required a more rapid and general approach. Consequently, massive batch stripping of light ends was effected on Stations 1, 2, 3, and 5. Light ends were fractionated out of the pump oil by operating with a room temperature baffle, 200°F test head and -75° collection plate. Additional light ends were subsequently removed from Stations 1, 2, and 3 with baffle and head at room temperature and the collection plate still at -75°F. The results of this fractionating procedure are given in Table 6. These data, while discussed below, are also vitally concerned with oil degradation and consequently are also discussed in that section. The major objective of this fractionating procedure was to see if the removal of light ends would eliminate the pressure pulsing and improve the system pressure readings at room temperature for Stations 1 and 3 (Station 2 was giving check results and Station 5 was not measurable due to mass spectrometer tests). It is seen that neither of these objectives were accomplished.

It appears that in the case of Station 1 with the non-fractionating pump the side stream stripping was ineffectual in improving the pump performance with regards to pulsing and system pressure. It does appear that this stripping operation after numerous fractions were removed decreased the amount of light end to a constant value equivalent to 1 x 10^{-6} mg/(cm^2)(min) in terms of backstreaming values. This value can also be considered as an effective vapor pressure value (Section G). It can be seen in Table 6 that the removal of light ends made no noticeable improvement in system pressure, measured at room temperature or with a liquid nitrogen gauge. The system pressure with the liquid nitrogen trapped gauge showed considerable improvement in the last

Table 6

LIGHT END REMOVAL

Station	Run	Fraction Removed, mg/(cm^2)(min) UV	Fraction Removed, mg/(cm^2)(min) wt	Light Ends by Non-Polar Gas Chromatography, %
Baffle at room temperature; head at 200°F; collection plate at -75°F				
1	55	7×10^{-6}	11.5×10^{-6}	60
1	59	1×10^{-6}		
2	56	20×10^{-6}	20×10^{-6}	
3	57	28×10^{-6}	29×10^{-6}	10
5	58	6×10^{-6}	8×10^{-6}	22
Baffle and head at room temperature; collection plate at -75°F				
1	67	4×10^{-6}		
1	69	2×10^{-6}		
1	71	3×10^{-6}		
1	74	$.9 \times 10^{-6}$		
1	76	1×10^{-6}		
1	81	1×10^{-6}		
2	49	3×10^{-6}		
2	79	4×10^{-6}		
3	64	$.9 \times 10^{-6}$		
3	66	4×10^{-6}		
3	68	$.7 \times 10^{-6}$		
3	70	4×10^{-6}		
3	73	$.9 \times 10^{-6}$		
3	75	3×10^{-6}		

run (Table 4, run 11/5), but this is presumably connected with the use of lower fore pressure. However, no logical explanation is presently available as to why the presence of light ends should interfere with the pumping action on non-condensible gases, i.e. the system pressure measured by the liquid-nitrogen trapped gauge.

No conclusions can be drawn from the results with Station No. 3, since it was subsequently found that this baffle had a very small leak in it. It is to be noted that the large periodical variation in the sequential values of the fractions stripped off should not be due to experimental error. It will be seen that a series of measurements with warm or cold baffle and warm collection plate (Table 4, runs 78, 80, 83, 85) gave a constant value of 1 x 10^{-6} mg/(cm^2)(min).

F. Oil Stability

1. Introduction

The question of oil stability is usually the controlling factor in the selection of the pump oil to be used in any ultra-high vacuum system. In the usual case, the greatest worry is system contamination although price, ultimate pressure versus baffle cooling temperature, and equipment performance cannot be neglected. Many people still hesitate to use the latest two oils which are being tested here and which have been known for over 2 years. DC 705 is now receiving considerable application, but Convalex 10 and OS 124 are receiving extremely minor usage.

The following work attempts to elucidate the most important factors involved in this problem.

2. Long-Term Use

In the present work both DC 705 and Convalex 10 have been in continuous use for over 2 years and OS 124 for a period of 1 year. This use involved fail safe operation so that the fluids have never been subjected to severe temperature or pressure conditions. The pump oils have been in contact with cylinder nitrogen or ambient air, which has permeated the elastomeric O-ring seals from the guard vacuum system. The systems all employed HS6-1500 pumps with baffles at 40°F to room temperature. Untrapped system pressures were in the 10^{-9} torr range after several weeks of continuous operation.

There is no measurable gross degradation in any case. The entire charge of 400 ml was apparently recovered. No visual change was apparent with DC 705, but the polyphenyl ethers yellowed within a few weeks and then remained essentially the same light tint with time. Almost the same color change occurred in samples exposed to light in glass containers at room temperature.

The appearance of the jet stacks only in the case of Convalex 10 (and presumably OS 124 which was not checked) showed very obvious and pronounced dark deposits (Figures 9, 10, 11). Since this deposit is external to all jets it is presumably due to oxidative degradation. The amount is so small that it would have no appreciable effect during several additional years of operation.

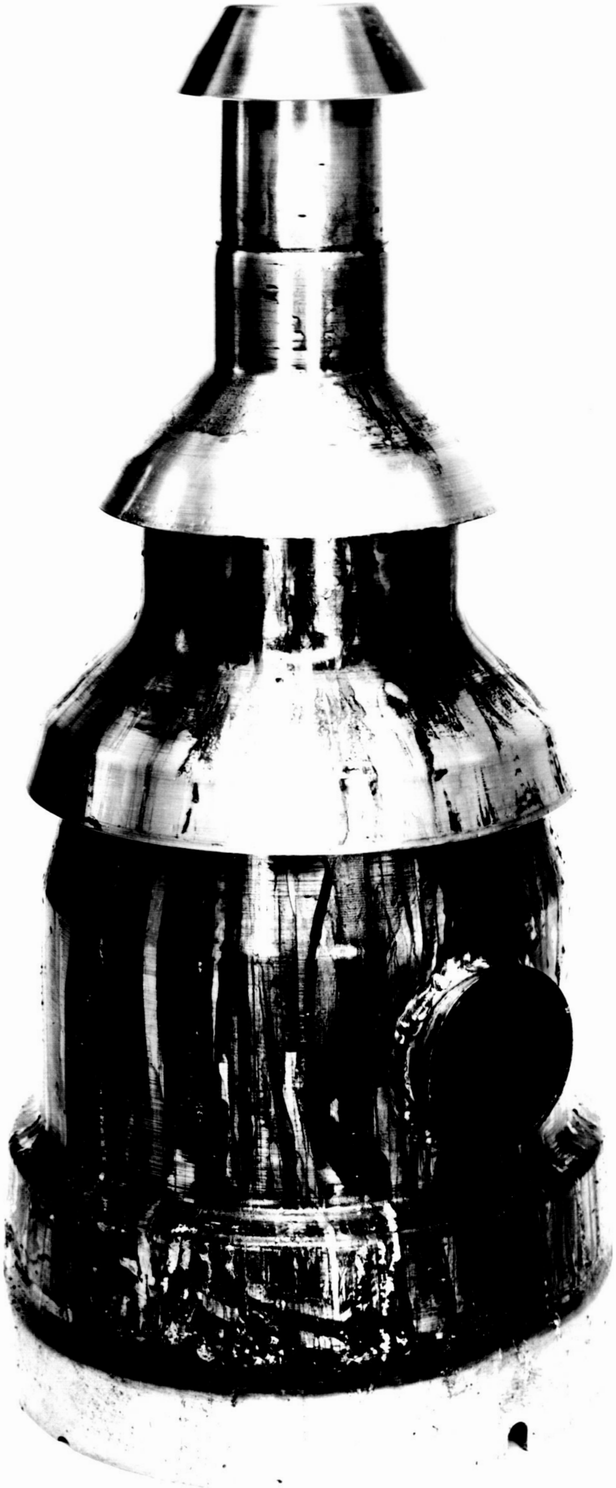

Figure 9

EXTERNAL JET SURFACES OF STATION NO. 5
Convalex 10 after 1 year

Figure 10

INNER AND OUTER JET SURFACES OF STATION NO. 5
Convalex 10 after 1 year

Figure 11

INNER JET SURFACES OF STATION NO. 5
Convalex 10 after 1 year

The presence of light ends is indicated by the samples fractionated off as discussed in the previous section (Table 6). It will be seen that the fractions from the old oils amounted to 28 and 38 mg/(cm^2)(min) for DC 705 and Convalex 10 in Stations 2 and 3, respectively, by ultraviolet analyses whereas the new oils in Stations 1 and 5 were 8 and 7 mg/(cm^2)(min), respectively. The figures by weight and chromatography analyses indicate light end hydrocarbons which have no effect on the spectra at the wavelength we are concerned with. The different values are a complex result of purity of the original oil, length of use, type of pump and method of operation, so that definitive conclusions are not possible. It is known that the original Convalex 10 contains two trace amounts of light ends (Figure 12). The first trace is probably a light end polyphenyl ether and is essentially absent in any fractionated sample (Figures 14, 15, 16). The second (smallest) peak is a light end non-polyphenyl ether. The areas of these bands relative to the original main peaks is 60 and 22% for runs 55 and 58 both using fresh Convalex and could be attributed to this original impurity. The variations of 60 and 22% are presumably due to the poorer self purging ability of the non-fractionating pump.

The larger amounts of oil by ultraviolet analyses in the used oil samples, 56 and 57, with equal values determined by weight correlates with their being light end degradation products from the longer use and have similar structure to parent compound. These light ends apparently have no noticeable effect on the

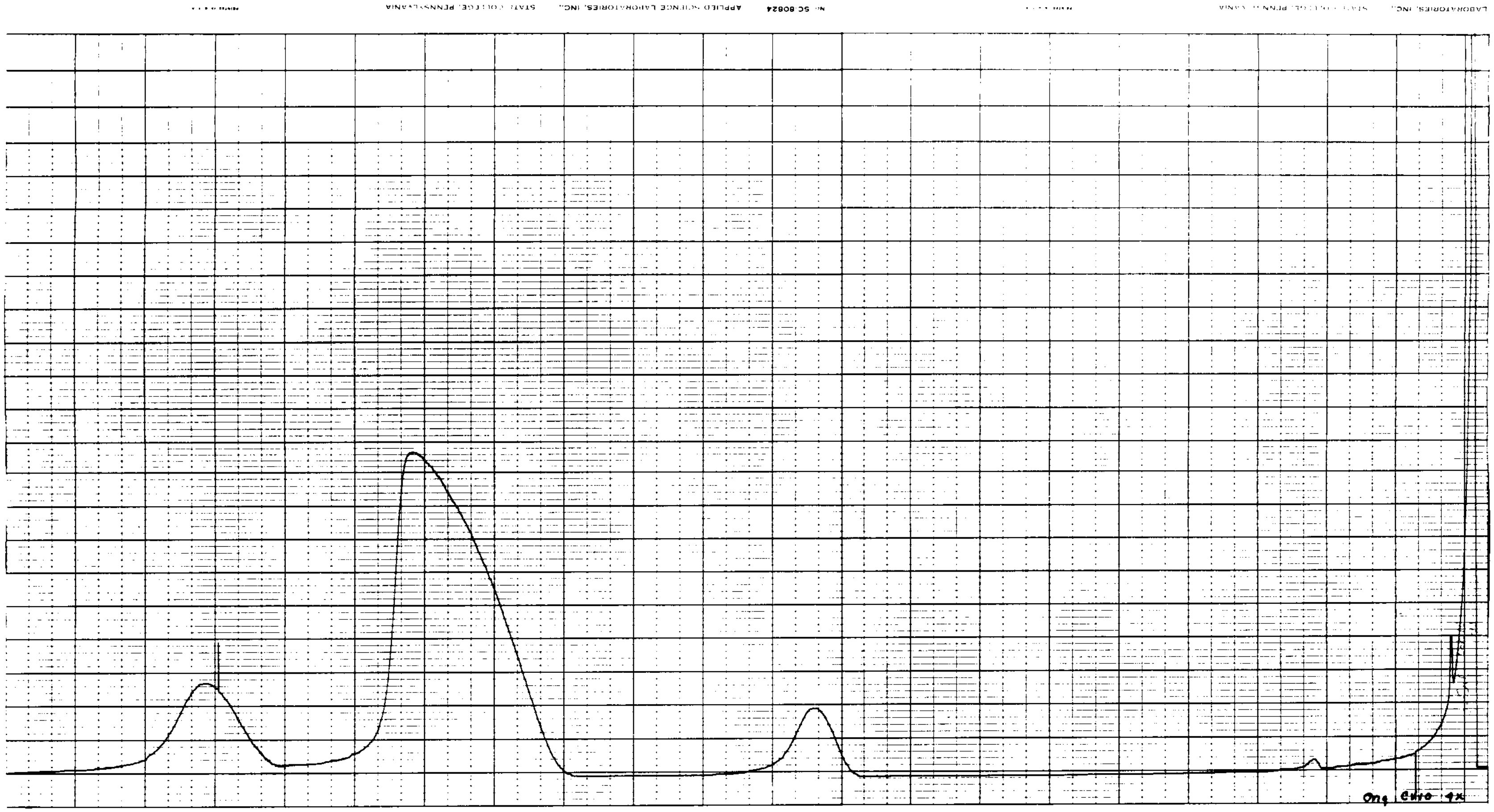

Figure 12

GAS CHROMATOGRAPH OF ORIGINAL CONVALEX 10 OIL

All peaks same 4X scale factor
Third peak - light end polyphenyl ether
Fourth peak - light end non-polyphenyl ether
Volatility decreases toward left end of chart

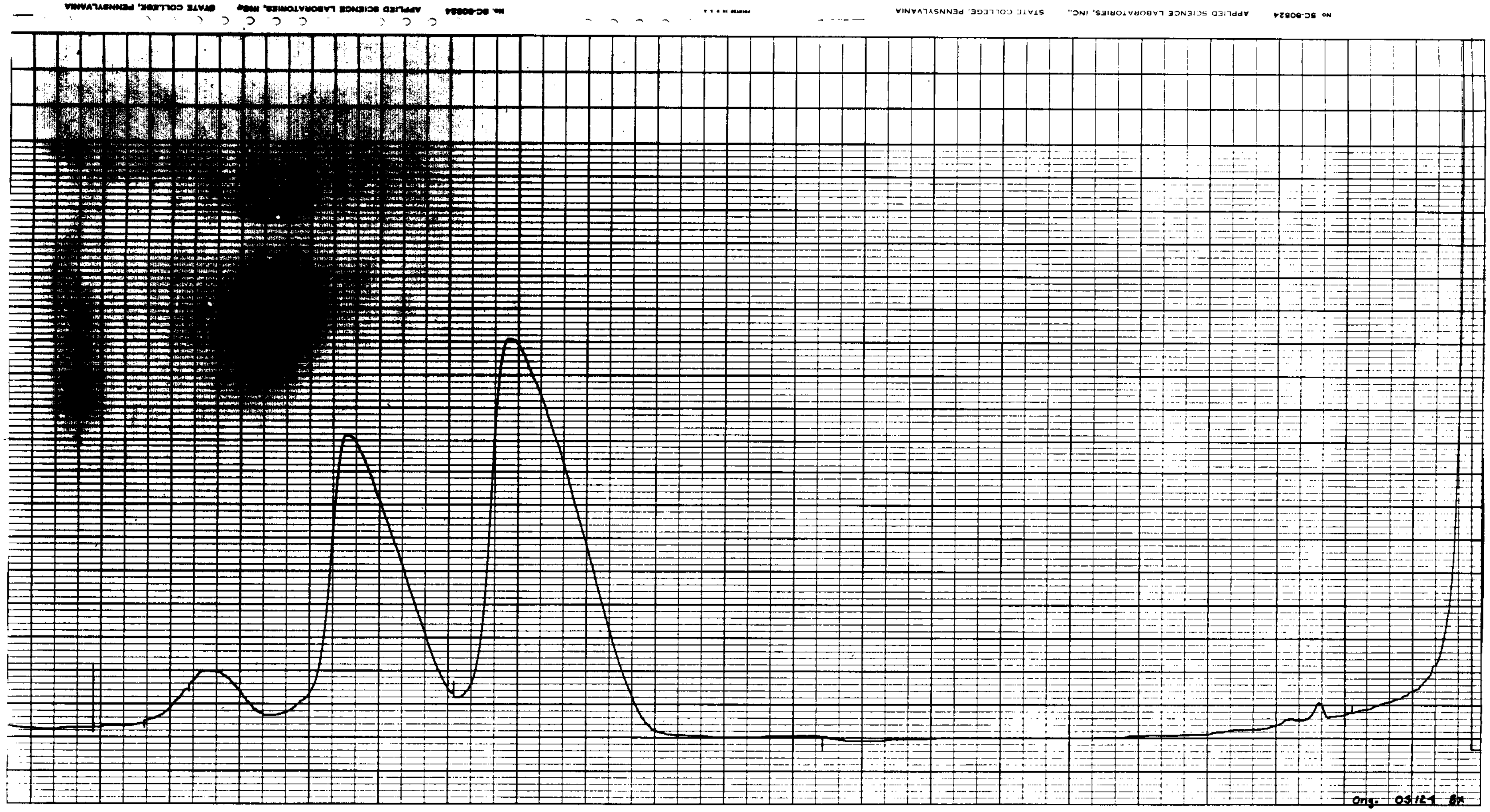

Figure 13

GAS CHROMATOGRAPH OF ORIGINAL OS 124 OIL

All peaks same scale 8X
First peak - pmp isomers
Second peak - mpm isomers
Third peak - mmm isomers

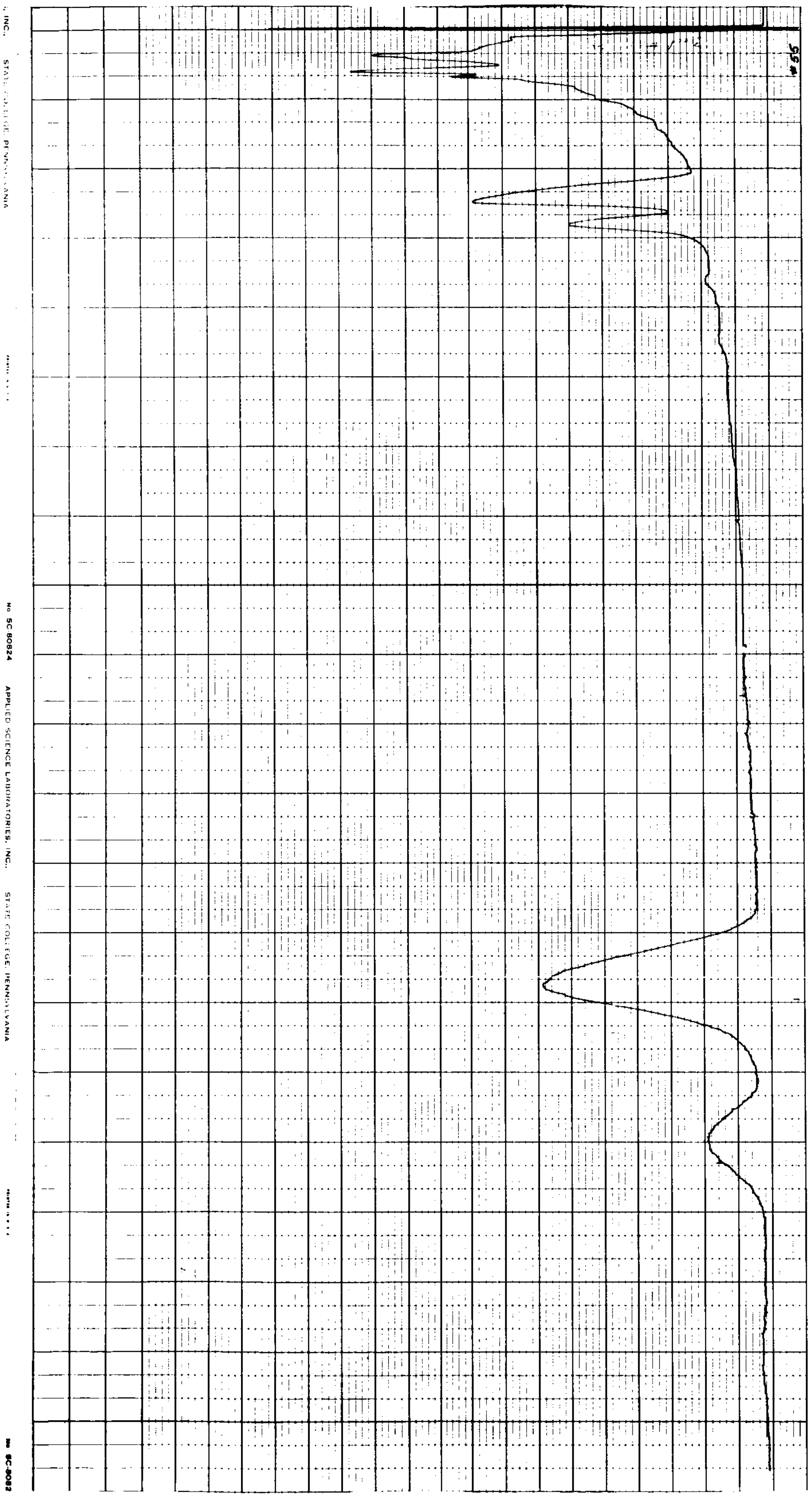

Figure 14

GAS CHROMATOGRAPH FROM STATION NO. 1 RUN 55

First two peaks 2X, remainder 8X scale factor
First peak has shifted relative to first peak Figure 12
Light end non-polyphenyl ether degradation products

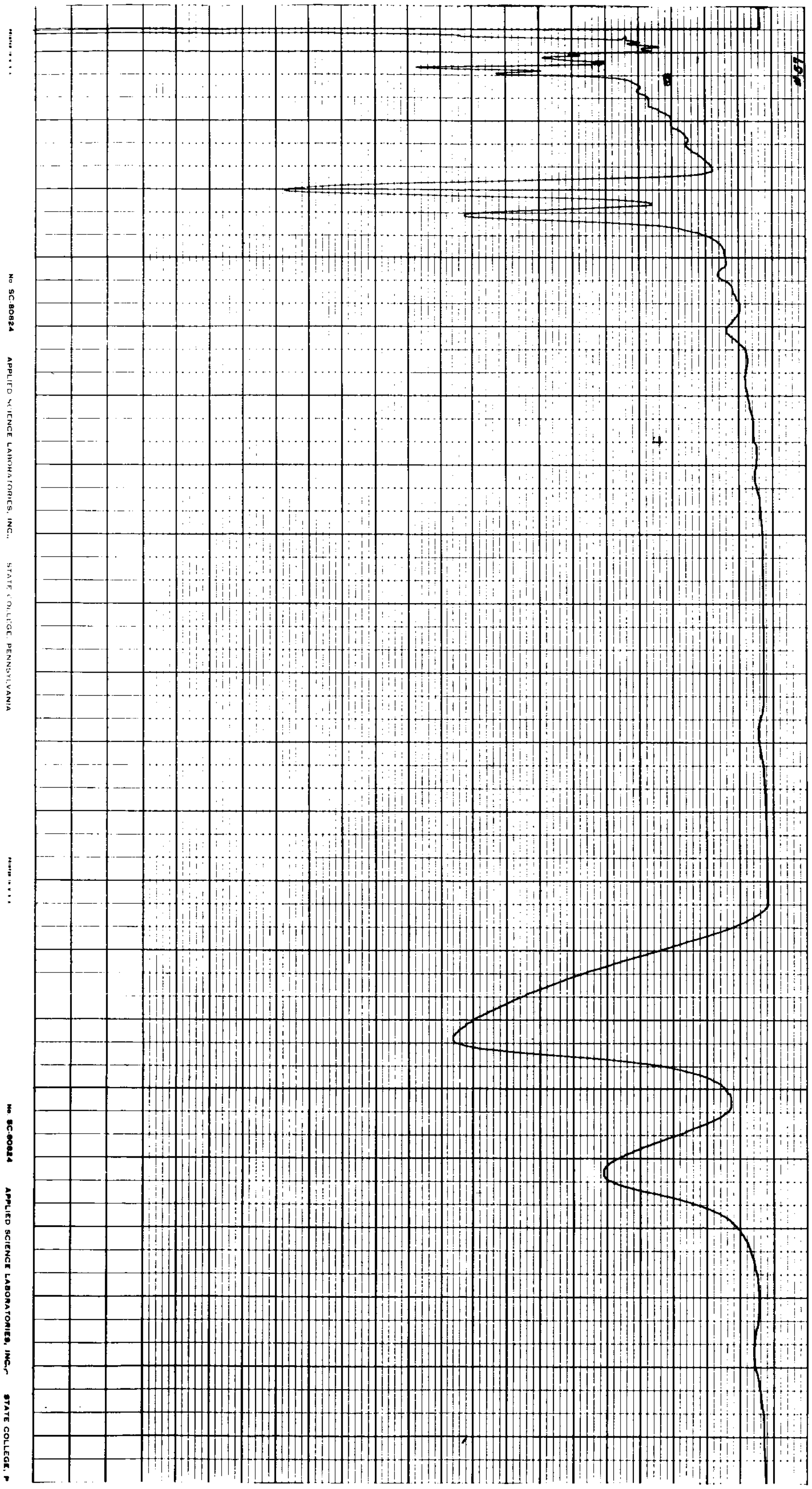

Figure 15

GAS CHROMATOGRAPH FROM STATION NO. 3, RUN 57

All peaks 4X scale factor
Light ends non-polyphenyl ether degradation products

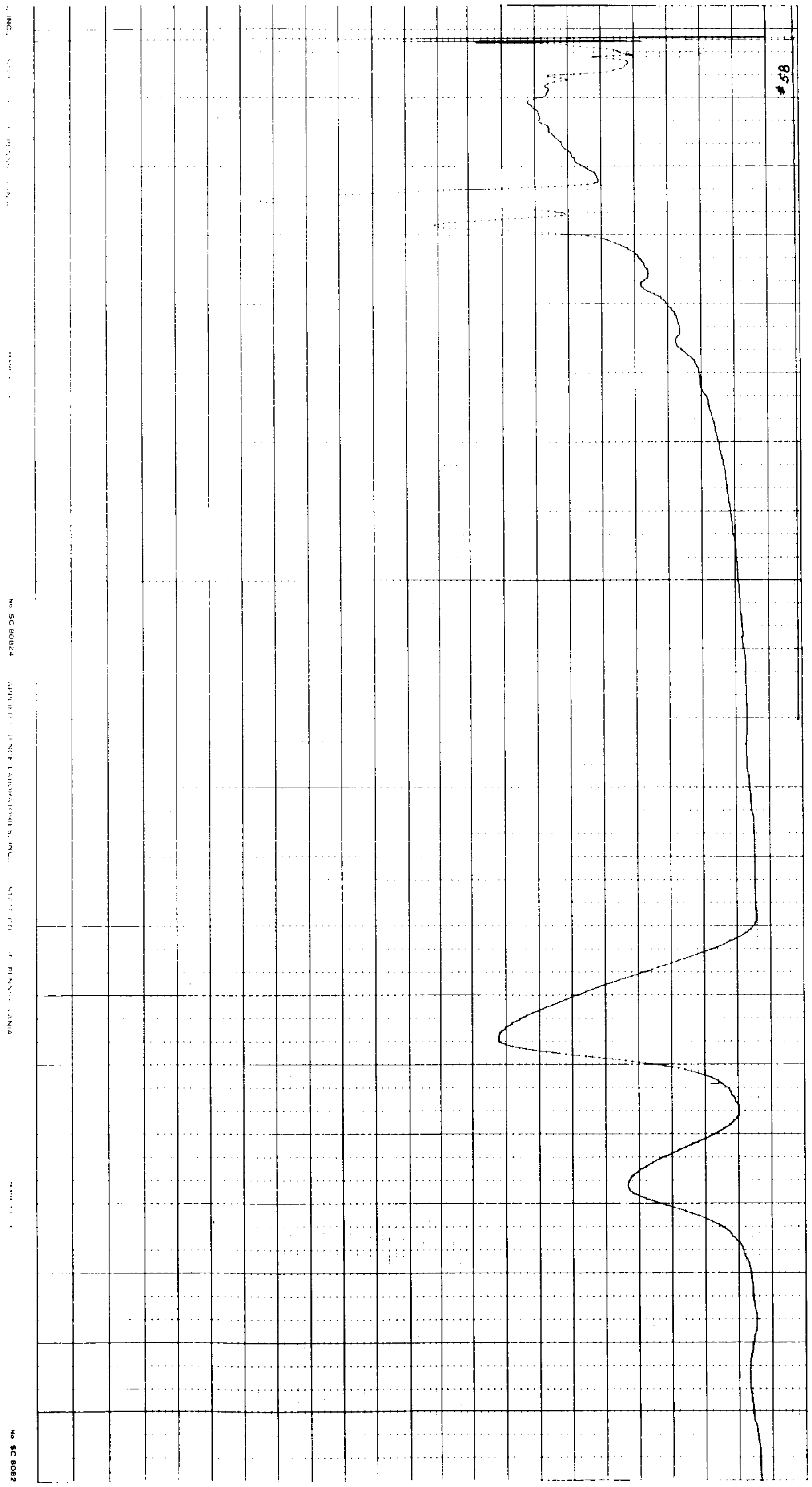

Figure 16

GAS CHROMATOGRAPH FROM STATION NO. 5, RUN 58

All peaks 4X scale factor

Light end non-polyphenyl ether degradation products performance of Station 2 since, prior to their removal, original backstreaming values were obtained (run 49). The baffle leak in Station 3 prevents any valid conclusions for this station.

3. Catalytic Degradation

Various substances,[11,16] including metals and decomposition products, have been mentioned as possible causes of excessive degradation rates for pump oils. In the present study only Convalex 10 in Station 3 shows evidence of catalytic degradation. Mass spectrometer studies, Figures 17 and 18, have found only mass 2 and 3 pulsing synchronously* with ion gauge pulses. While the entire mass spectrum to 150 has been scanned with extreme care, it is still possible to miss the pulsing of isolated peaks. The pulsing of mass three in the 10^{-11} amp range, and generally in 10^{-12} amp range, Figure 18, demonstrates that the recorder time constant is sufficient to indicate pulses for the higher mass numbers in this ampere range. The decreased sensitivity of the higher masses leaves the possibility of a pulsing heavy mass in the 10^{-10} torr range. A complete mass spectrum is shown for Station 3 in Figure 19. The system was operated with the baffle evacuated to $< 30\ \mu$ to eliminate the major effects of the leak. The spectrum shows that hydrogen is the main component. The hydrogen pulses vary in height from 2X to 100X as recorded

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*The charts chosen to illustrate pulse variations show notations of no Bayard-Alpert pulse in some places. Later more careful observation showed 100% synchronism between mass spectrometer and gauge.

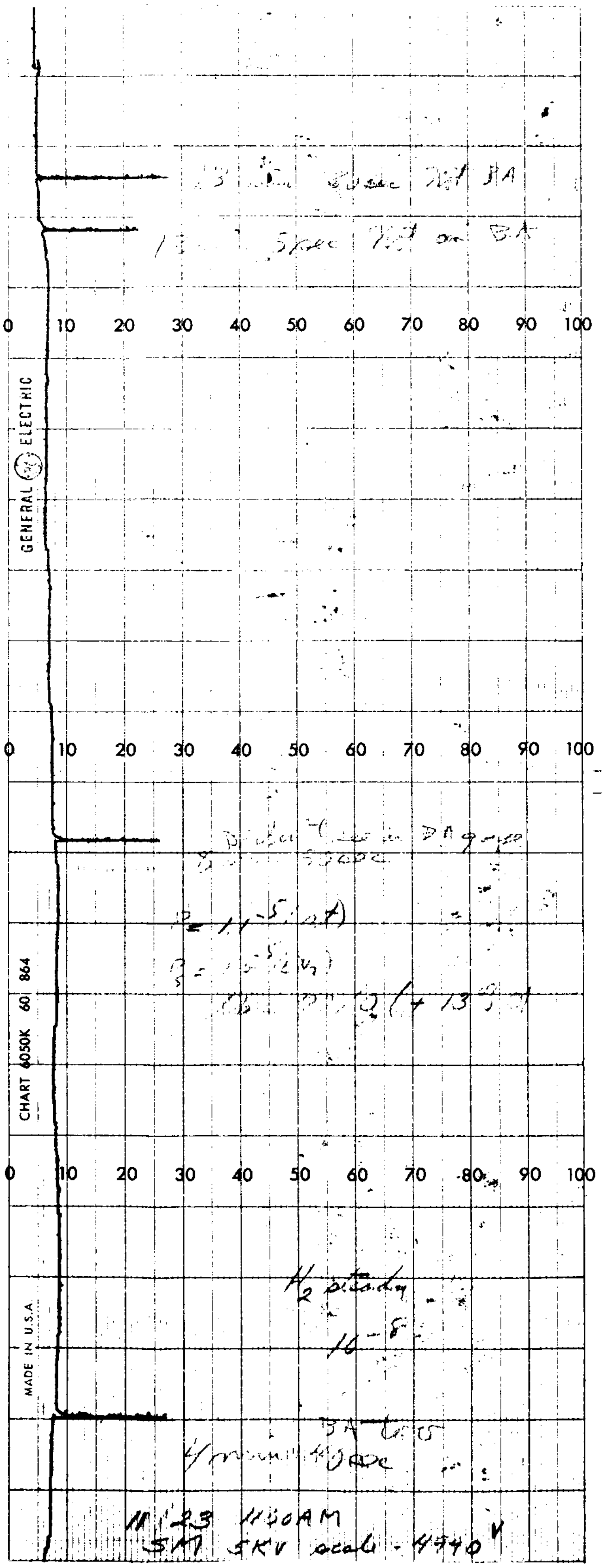


Figure 17

MASS 2 PULSE RATE

Station No. 3, Convalex 10 (used)
Baffle HN-6 (with leak) at
room temperature and less
than 30 μ

Acceleration volts = 4990 S.M.

Chart Notes

1) 2 div = 1 min

2) Scale 10^{-8} amp

Pulse a fraction of a second so amplitude not significant.

3) P_s $(LN_2) = 1.0 \times 10^{-5}$ air leak

4) Chart selected to show three equal and one short pulse

5) Comments on 2, 3, and 4 pulse not significant. Later with practice always on Bayard-Alpert

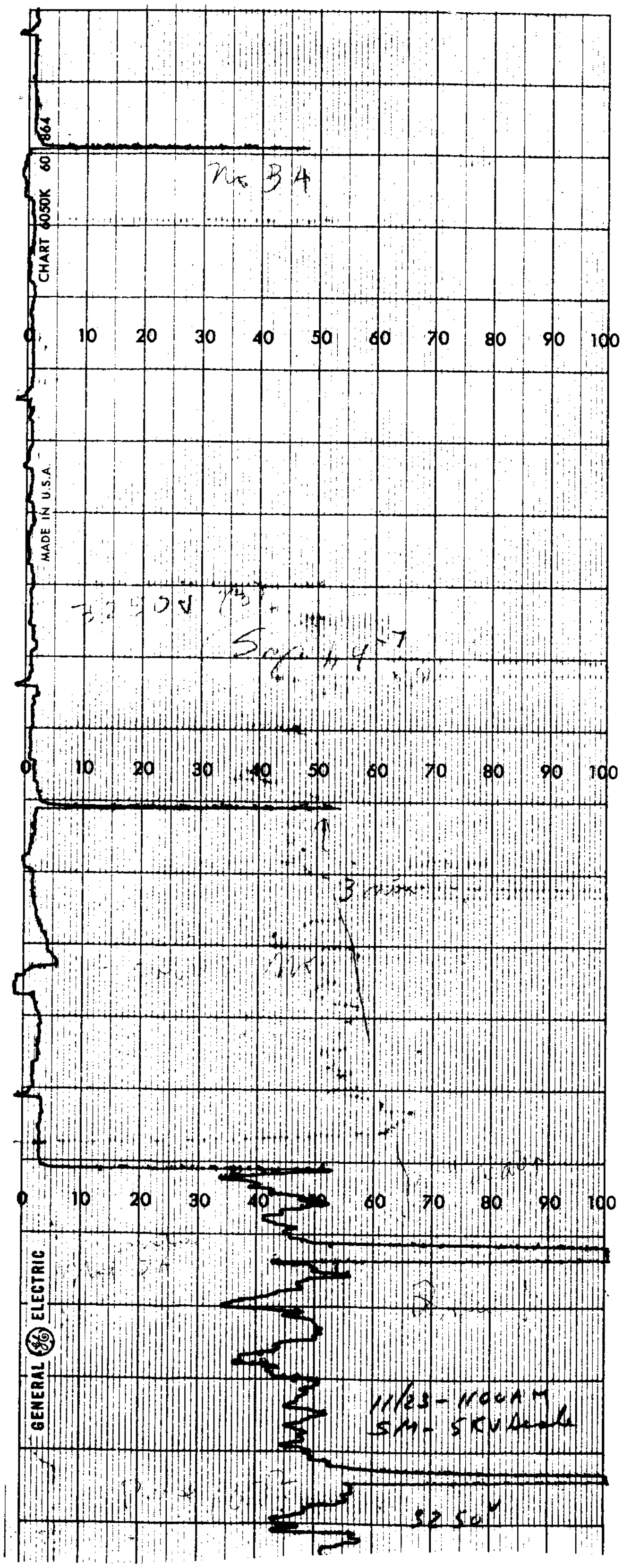


Figure 18

MASS 3 PULSE RATE

Station No. 3, Convalex 10 (used)
HN-6 baffle, room temperature
with leak less than 30 μ

Acceleration volts = 3250 S.M.

Chart Notes

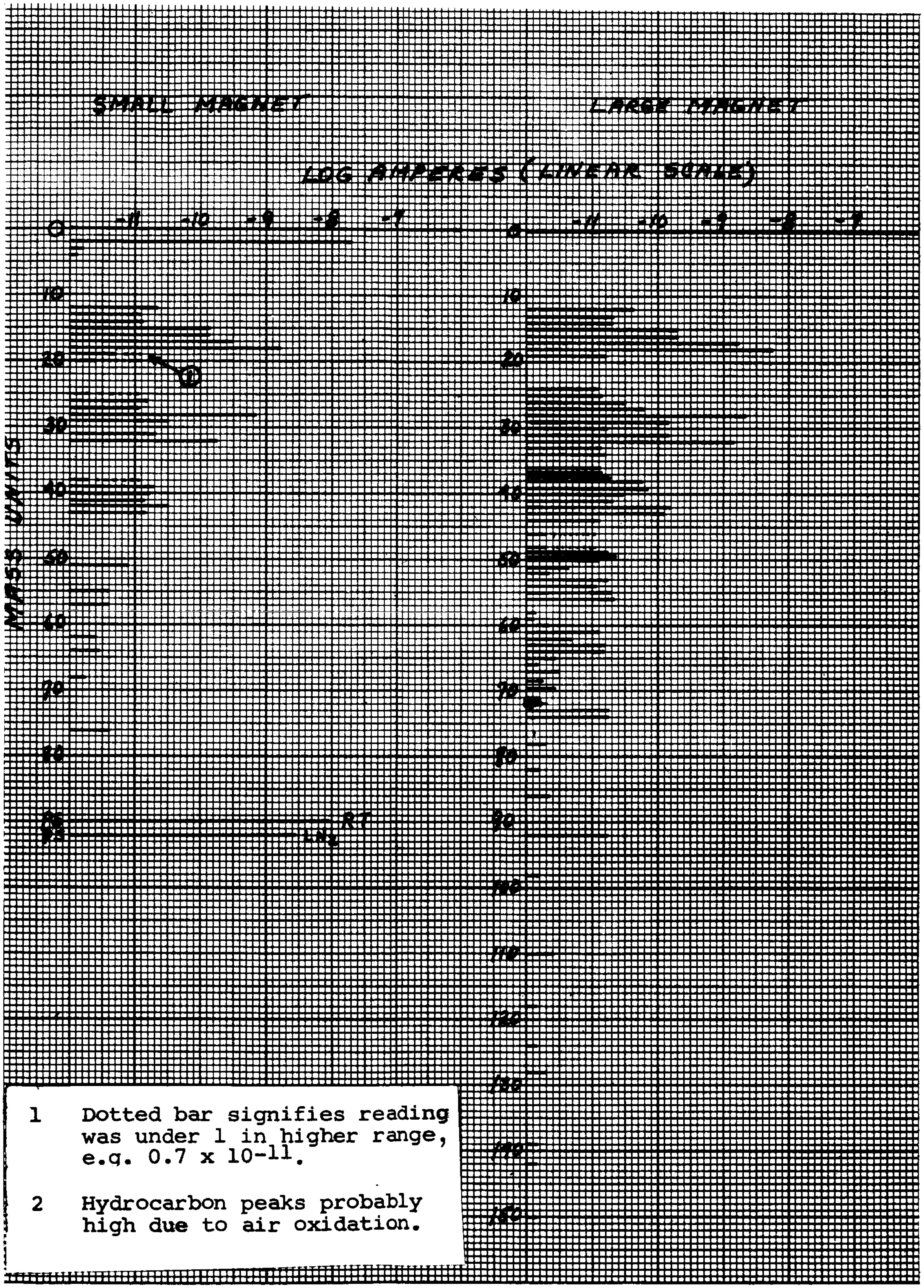
1) 2 div = 1 min

2) Scale: pulse 1 and 2 10^{-12}A
pulse 3 and 4 10^{-11}A

3) P_s (LN_2) 1.4 x 10^{-7}

4) See preceding chart comments on Bayard-Alpert

5) Leak 77/0 air and 13 mol % benzene



1 Dotted bar signifies reading was under 1 in higher range, e.g. 0.7 x 10-11.

2 Hydrocarbon peaks probably high due to air oxidation.

Figure 19

STATION No. 3 MASS SPECTRUM SURVEY
HS6-1500; Fore pump H-2-P (10^{-5}t, r.t.)
HN-6, room temperature and 30 μ internal pressure
Convalex 10 (used 2 years)
November 13, 1964

visually on the oscilloscope. These pulses have been observed when the baffle was at room temperature, -75°F with MeOH, cooled with liquid nitrogen, open to the air, and evacuated. Initially, with room temperature MeOH in the baffle, the hydrogen pulses were extremely steady at 180 ± 10 sec over a period of days (MeOH peaks, etc., pulsing 1 per second). With -75°F MeOH the pulse frequency increased immediately to 105 seconds and remained there. After vacuum was applied to the trap to counteract the leak and measurement made several days later the period had become irregular and varied from several to 15 minutes. A replacement baffle has been recently installed. It is planned to verify the phenomena. Catalytic effects can then be demonstrated by runs with new oil in a clean pump, seeding with the old oil, etc.

The above evidence seems to indicate a catalytic phenomenon, although variations in pulsing occur with changes in baffle temperature and leak rates. The catalytic agent could be various substances, either extraneous materials or autodegradation products. The age of this oil, 2 years, could well lead to the latter. It is to be noted that Station 1 with fresh oil and Station 2 with used DC705 have shown this pulsing phenomenon, but they were not checked with the mass spectrometer. One source of extraneous catalytic material has recently been brought to our attention. This is the nickel plating on the baffle of Station 3 which is observed to be sloughing off. Further speculation does not seem profitable since future experimental studies are expected to clear up these points.

4. Effect of Air Leakage on Stability

An exploratory study of the effects of air leakage was made with the mass spectrometer. It was not expected that quantitative results would be forthcoming, since the stability of the oils would appear to preclude this. Rather it was hoped that patterns of the mass spectra and their changes with varying leakage conditions could be used to monitor these effects, since their rate of formation and disappearance should constitute an indication of their importance.

A series of mass spectra for Station 5 are shown in Figure 20, A through G. Blanks in the mass numbers do not necessarily indicate missing peaks but are subject to the attention given to particular masses. There is usually considerable noise in the lower 10^{-12} ampere range. Consequently only mass series, lower and on both sides of one or more peak in this region, are fairly conclusive evidence of the presence of higher masses in this low amperage range.

Prior to these particular tests the station had been subjected to varied regimes which cannot be well defined. The particular regimes are indicated in Table 4. Of pertinence to air degradation, Station 5 has been subjected to undefined air leakage for periods of over 2 months. The variation of system pressure between 10^{-7} to 10^{-8} torr can serve as a rough guide. The periods for which the mass spectra are shown involve periods of 3 days where ambient air was leaked into the test heads of each station. The magnitude of any leak can be readily determined

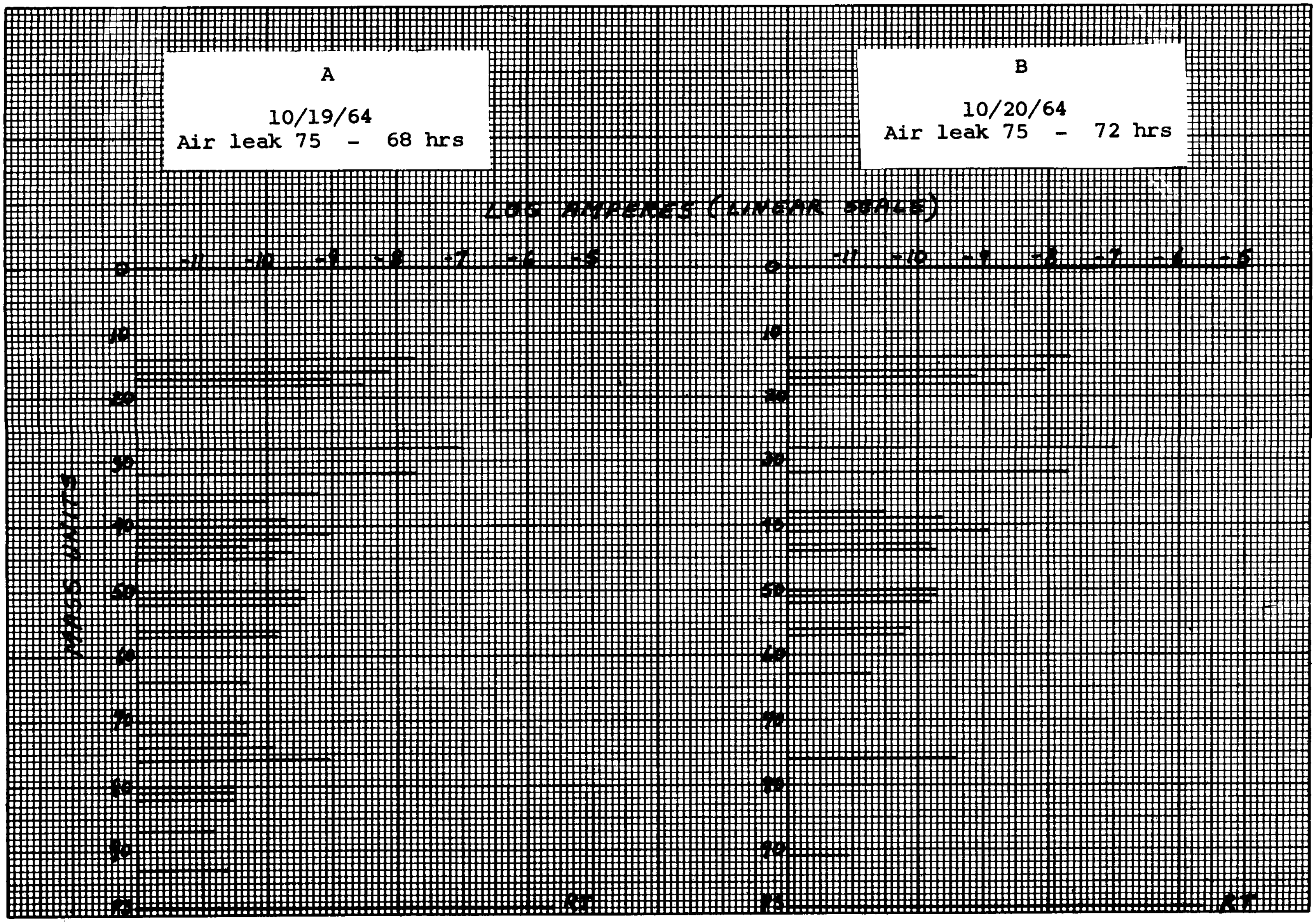


Figure 20 (A and B)

STATION No. 5 MASS SPECTRA SURVEY

HS6-1500
Right angle elbow at room temperature
Convalex 10 (new oil)

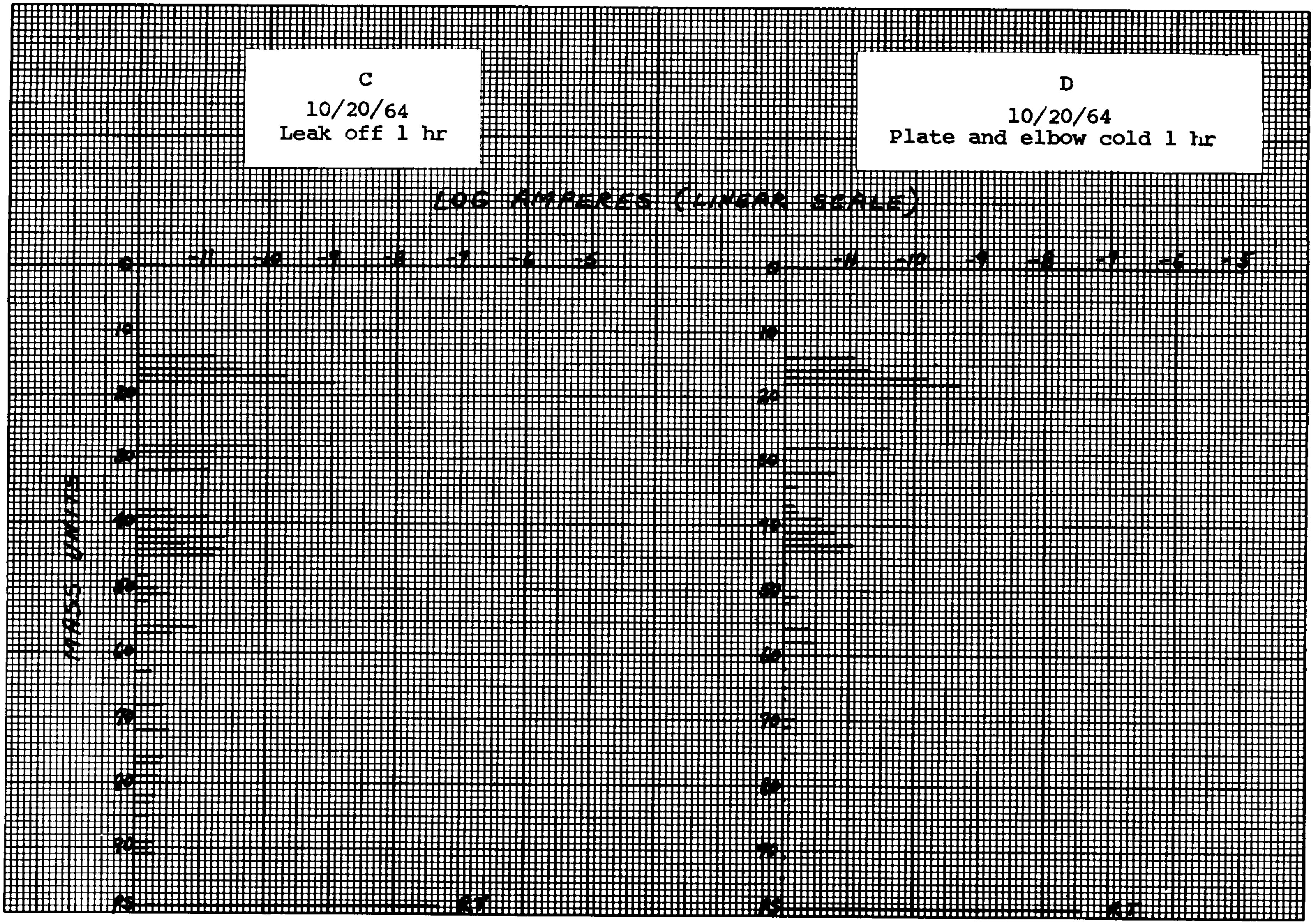
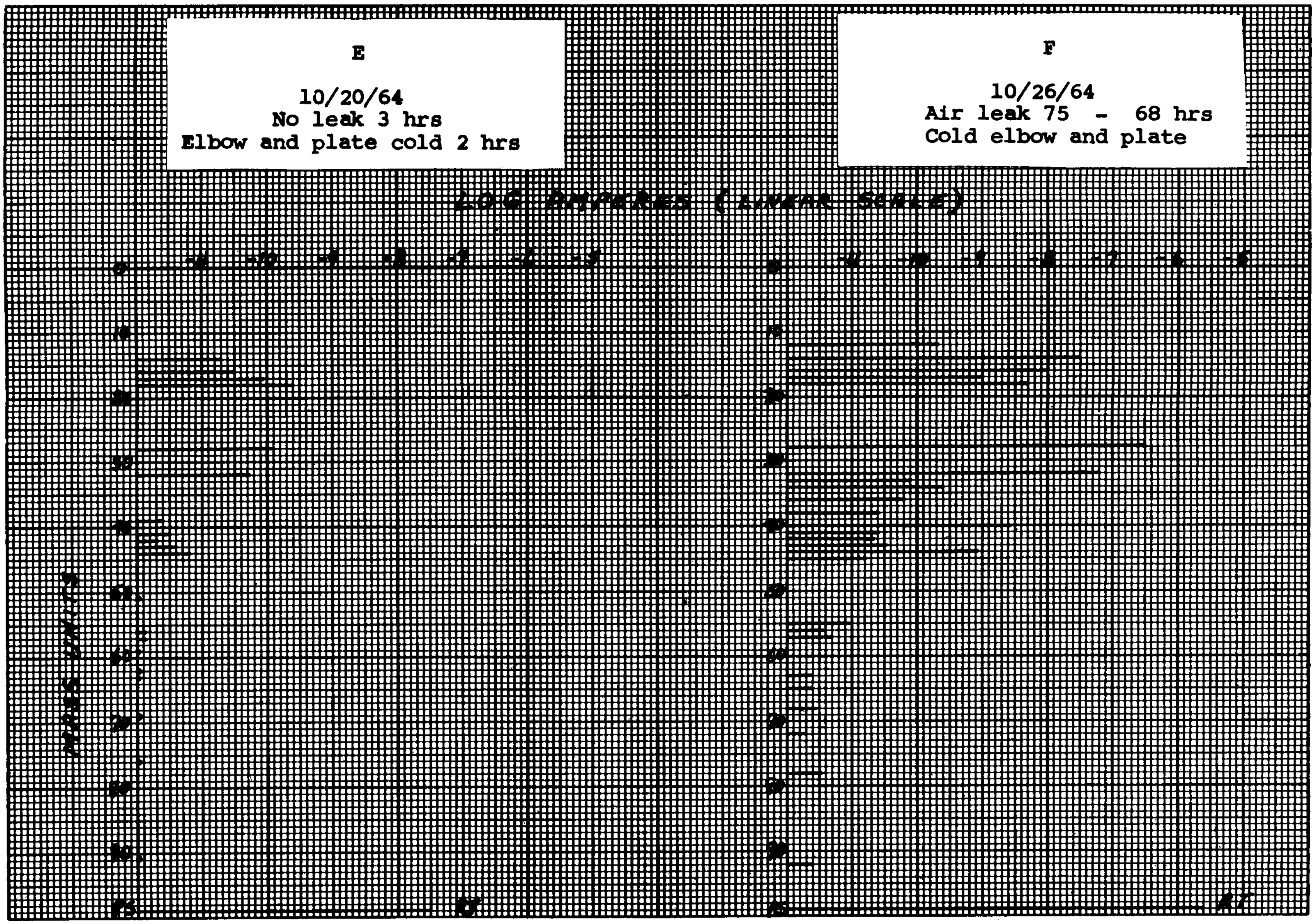


Figure 20 (C and D)

STATION NO. 5 MASS SPECTRA SURVEY

HS-6-1500; Right angle elbow
Convalex 10 (new oil)



E

10/20/64
No leak 3 hrs
Elbow and plate cold 2 hrs

F

10/26/64
Air leak 75 - 68 hrs
Cold elbow and plate

Figure 20 (E and F)

STATION NO. 5 MASS SPECTRA SURVEY

HS-6-1500; Right angle elbow
Convalex 10 (new oil)

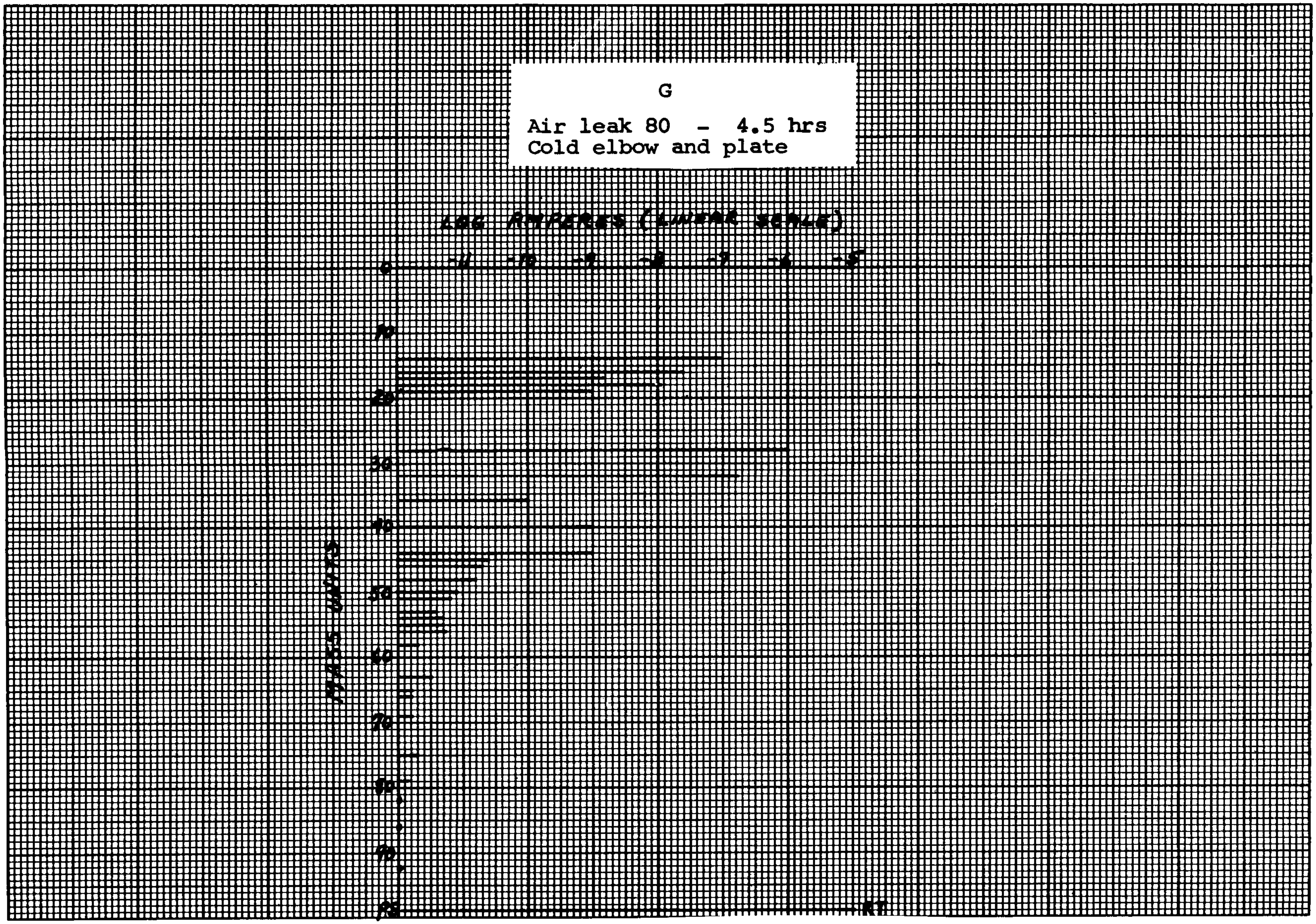


Figure 20 (G)

STATION NO. 5 MASS SPECTRUM SURVEY

HS-6-1500; Right angle elbow
Convalex 10 (new oil)

from any system pressure using the net pumping speed at the head for nitrogen of 500 L/sec. The following observations can be made:

a. The higher mass numbers are not measured precisely, e.g. values in Figure 20A for mass 76 could be mass 78.

b. The room temperature baffle with an air leak shows numerous high masses over 60, in the 10^{-10} amp range and one very predominate peak at 76 (or 78) of 10^{-10} amp. With -75°F baffle all peaks over 50, including 76, decrease to the low 10^{-12} amp range. With air off and the baffle at either room temperature or -75°F all heavy peaks decrease essentially to zero.

c. The rapid decrease of these peaks with the leak off indicates they are due to air degradation and such degradation is minor and self purging in nature.

d. Since the vapor pressure of benzene at -75°F is $\sim 10^{-4}$ torr, the effect of baffle temperature in decreasing the mass peaks indicates that these are degradation products of fairly high molecular weight.

e. The mass values obtained with Station 3 (Figure 19) using a room temperature baffle (baffle had a small leak but was pumped to 30 μ), are very similar to those of Station 5 with an air leak. It is probable that the air leak in Station 3 baffle, whose magnitude is indicated by 28 and 32 peaks, is responsible for this spectrum.

5. Relative Stability

A true comparison of the stability of DC 705 versus that of Convalex 10 is not possible with the data obtained to date. However, the evidence cited above generally favors the DC 705. This evidence includes the absence of jet deposits, clear liquid, immediate recovery of system pressure and low backstreaming values at operation with room temperature baffle, no evidence of excessive decomposition rates, and ease of self purging. It is further seen that a long run of 526 hours with a cold baffle (Table 4, 11/18) has not yet re-established the previous pulsing phenomena.

The absence of deleterious effects with DC 705 is not definitive since more attention has been paid to Convalex 10. It might behave in the same fashion if it had been in Stations 1 and 3. It is noted that Stations 5 and 6, with Convalex 10 and OS 124, are generally performing considerably better than Station 2 with regard to backstreaming, ultimate pressures, and pulsing phenomena.

G. Estimation of Vapor Pressure

The effective vapor pressure at room temperature of a diffusion pump oil and its change with use is an important measure of oil stability. It is of importance to backstreaming contamination since it is directly related to light ends which in turn can influence system pressure (measured at room temperature) and degree of backstreaming. The greatest importance is in UHV systems operating with moderately cooled baffles. The actual measurements are usually made at ~250°C and extrapolated ~10^6 times to room temperature values. The effective values when used in a pump are often in doubt by a factor of ten.

The vapor pressures of pump oils can be measured[15] at ambient temperatures by collecting the amount which effuses from a small hole in a relatively large chamber whose vapor space is in equilibrium with a pool of oil. Such measurements are complicated by the need of measuring the very small quantities of oil involved. These values are measurements of the vapor pressure of the gross body of oil. The effective vapor pressure which is of interest here is that due to the light ends and could be measured precisely by collecting sufficient and representative fractions to form a pool in an effusion chamber. The difficulty lies in collecting the representative fraction even though only milligram quantities are required. The nature of the fraction varies with innumerable factors and is best obtained under a given set of operating conditions. In any case the precision is quite variable and black body effusion measurements are not warranted. The present technique of collecting on a -75°F plate all of the oil passing a room temperature baffle appears to be an effective vapor pressure measurement of the light ends which are collected on the baffle surfaces under operating conditions. It should be appreciated that such measurements are probably 1/2 to 1/3 less than those which would be obtained by black body effusion. This is due to conductivity and directional effects through and above the baffle. The conversion formula,[13] usually attributed to Langmuir,[15] assuming the fractions have a molecular weight of 500 is

$$P_{torr} = \frac{W}{4.5 \times 10^3}$$

where W, in mg/(cm^2)(min), is the measured value of collected oil minus true backstreaming value obtained with both baffle and plate cooled. Since the latter is less than 20% of W it can be neglected. The values for these stripping runs are given in the second section of Table 5 and are reproduced in Table 7. The values for Convalex 10 in Station 1 have decreased to a constant value of 1 mg/(cm^2)(min) which corresponds to a pressure of 2×10^{-9} torr. DC 705 in Station 2 has a value of 3.5 mg/(cm^2)(min) corresponding to a pressure of 8×10^{-9} torr. The values for Station 3 which vary from .7 to 4 times that of Station 1 are not considered since they show periodic variations from 1 to 4×10^{-6} mg/(cm^2)(min).

These values are the same and 20X the recently published values[7,8] of 1.7×10^{-9} and 4×10^{-10} torr for Convalex 10 and DC 705, respectively. The literature value for Convalex 10 was estimated[7] from backstreaming values under essentially identical conditions except that system pressures were 10^{-9} torr (r.t.) compared to our 10^{-6} torr (r.t.).

Table 7

FRACTIONS WITH ROOM TEMPERATURE BAFFLE AND -75°F PLATE

Station 1		Station 2	
Run	mg/(cm^2)(min)	Run	mg/(cm^2)(min)
67	4	77	3
69	2	79	4
71	3		
74	.9		
76	1.		
81	1.0		

H. Pressure Pulsing

1. Introduction

This phenomenon is of extreme importance to backstreaming contamination since, de facto, it shows excessive unsteady state conditions which could result in liquid or gaseous matter being ejected backward through the pump and baffles into the work space. These pulses which are indicated by an ion gauge are generally different from those occurring in average boiling when metal pumps of 4 in. or over are being considered. These pulses have been attributed to many causes,[1,11] light end hydrocarbons, moisture, dissolved light gases, degradation heavy ends, eruptive boiling, leaks, hydrogen evolution and oil decomposition, degassing processes (particularly with water), etc. While there is essentially the same operating principal involved for both ion gauges and mass spectrometers, at least one case of gauge pulsing has been reported[6] where no individual peak pulses could be detected. Presumably the frequency and reproducibility of these pulses rules out false gauge readings. Undoubtedly any and all of these factors have been demonstrated to cause pulsing but their exact relationships and, more importantly, their prevention or cure have not been well established.

Pulsing phenomena have been reported with both oil and mercury diffusion pumps and also ion pumps.[6] One report[3] refers to an oil (DC 704) system employing a liquid nitrogen cooled zeolite trap which does not pulse and infers this is the only oil pumped system in their experience without evidence of pulsing.

It is to be noted that the preceding work at IIT Research Institute[13] on backstreaming using essentially identical systems, except the baffles were at 40°F instead of -75°F, gave no signs of pulsing in 5 stations operated for periods of two years.

It is obvious that the interplay among the many factors has required their consideration with interrelationships of pulsing. The following discussion will be mainly concerned with causes of pulsing itself and only minor emphasis will be given to its relationship to backstreaming. It is probable that the phenomena encountered here is not confined to any one cause. It is a fact of vacuum phenomena that almost every process, whether boiling, leaking, degassing, etc. will occur as a series of explosions. The phase change from absorbed gas or liquid to gas involves 10^6 X volume change and can therefore be quite violent.

It is just recently that a rapid response recorder has been connected to the Varion ion gauge output. This instrument shows numerous $\Delta 1 \times 10^{-8}$ pulses every 10 seconds. The latter being so rapid that there is only a quiver of the ion gauge indicator. Preliminary measurements on other stations with room temperature baffles show large $\Delta 10 \times 10^{-8}$ pulses at irregular periods of up to 1 hour and longer. It is concluded that all of the present stations are probably giving pressure bursts of this magnitude. It is seen that their presence can be easily missed on both the ion gauge and mass spectrometer unless special recording techniques are being employed. The small volume of our test chambers make the ion gauge readings very sensitive to such pulses.

2. Explosive Vaporization of Light Ends

The presence of light ends can account for much of the pulsing phenomena encountered in the present investigation, and can be used to explain many variations in the backstreaming measurement. As discussed under oil stability, mass spectrometer readings on Station 3 show that the pulses are confined to hydrogen. This evidence would seem to preclude revaporizing effects of light ends. However, the pulse period is influenced by the temperature and condition of the baffle which implies a relationship with light ends.

In the earliest use of oil diffusion pumps, the phenomena of "wet cap,"[4,17,19] drops of oil falling off the top cap and hitting the hot jet stack, was related to pressure pulses and high backstreaming values. These mixed component oils contained massive amounts of light ends and were not particularly stable. The use of a trough to lead drops off to the cold walls readily eliminated the problem. The use of modern, stable, single component diffusion pump oils has further minimized this phenomenon. Obviously, since any oil undergoes some degree of decomposition, the buildup of such light ends is a function of the ability of the pump to purge these light ends to the fore line. Thus the present variations in backstreaming encountered in Stations 1, 2, and 3 using cold caps could be due to build up of light ends which has taken place (Table 6). It is known from distillation theory that the lower baffle temperature will seriously interfere with the process of fractionating light ends to the fore line. The gradual increase of most initial backstreaming values in subsequent runs indicates a fairly regular process. The pulsing phenomenon and high backstreaming values were never observed in any large IIT Research Institute systems operated only with 40°F or room temperature baffles. Station 5 with a -75°F right angle baffle was not observed to pulse although the backstreaming values are higher than previous results. It is apparent that the nature of the latter baffle does not allow drops to impinge on a hot stack, and thereby produce explosive vaporization. This type of explosion requires a volatile drop since single component pump oil would not vaporize. Such volatility can result from light end fractions or even dissolved gases in a single component oil. This mechanism also provides a possible explanation for the difference between DC 705 and Convalex 10. The latter and its light ends are very viscous materials and would tend to freeze in place rather than run off. DC 705 is a very fluid material with an extremely low viscosity index, thus being more prone to "dripping." The recovery of low backstreaming values following operation with room temperature baffles in Station 2 containing DC 705 tends to substantiate this. This recovery was not effected as yet in Stations 1 and 3 containing Convalex 10. This failure has been discussed elsewhere.

3. Moisture and Pulsing

Moisture in the system, either as ice or absorbed liquid, was found to cause pulsing in our initial measurements and presumably could be responsible for the initial high values. It was observed that in these runs, during the first 24-48 hours very sharp and regular pulses with a period of a few seconds occurred in the 10^{-7} torr range. These started immediately and decreased in period and intensity with time. It was found that this type of pulsing could only be eliminated by allowing baffle to reach room temperature, flooding the system with dry nitrogen, and rapid sample removal and startup. Our previous method of flooding with nitrogen and removing samples while the baffles were cold was not able to prevent some internal ice formation. The pulsing discussed in 2 above is distinctly different since it occurs at a lower pressure and has a period on the order of minutes to 1/2 hour.

4. Eruptive Boiling

No direct study was made on this phenomenon, since attention was confined to the related phenomenon discussed above. Undoubtedly eruptive boiling is an important factor in pressure pulsing. In small 2 inch pumps audible boiling clicks can be correlated with pulses. These pulses penetrate unbelievable flow resistances and distances. Earlier in the present program these pulses were found to penetrate a 5/16 in. x 8 in. gauge tabulation with little attenuation. There appears to be no definitive correlation between the physical violence of the of the boiling process and the pulsing phenomena. Particular small pumps can be observed audibly and visually to boil more violently than others and give no sign of pulsing. It has been demonstrated that such non-pulsing pumps containing air in the fore line will often pulse with hydrogen in the fore line. The fore line hydrogen partial pressures varied from 10^{-1} torr to 10^{-8} torr (LN_2).

I. Mass Spectrometer Studies with Gas Injection

1. Introduction

The use of a mass spectrometer with gas injection is a relatively old technique for studying the operation of diffusion pumps. It has appeared to us that it should be possible to develop this technique sufficiently to permit rapid and sensitive measurements of backstreaming phenomena. "Backstreaming" is used here to signify the movement of any material, including hydrogen, backwards through the pump. It is more common to consider the effect with hydrogen as a measure of pump efficiency rather than backstreaming contamination. Our present objective is mainly concerned with hydrogen backstreaming as it will help us determine the mechanism involved in backstreaming of much heavier components. Of course, information on hydrogen backstreaming is of importance per se for obtaining improved pump performance (ultimate pressure).

There are two main mechanisms by which material may move backward through a diffusion pump. One involves direct flow through a jet and countercurrent to the issuing pump oil. The second involves solution in the condensing pump oil whereupon part of it reissues through the top jet (and also lower jets) together with the oil vapor. This material then curves around and up due to the random motion that exists in these jet streams. The classical form of fractionating diffusion pump employed a compartmented boiler so that the vaporizing oil furnishes vapor consecutively from lower to higher jets. Recently pumps are termed fractionating which subject the condensed oil to a hot wall section prior to their reentering the boiler, thus removing light ends before the oil reaches the boiler.

The modern oils with high stability and absence of appreciable light ends have minimized the importance of the fractionating effects. Pump design has been changed to get improved performance, which in some cases involved less fractionating design. However, all such designs are usually based on relatively short-term tests, and the same results are often reversed in long-term usage. The present tests with moderately cooled baffles (-75°F) is strong evidence that there is inadequate fractionation. It is most probably that with continuous liquid nitrogen cooling the lower temperature could delay the appearance of light ends by freezing them out throughout any extended test period. However, even here, startup and bakeout procedures and possible variations in cooling temperature are probable causes of contamination in UHV systems.

2. Change Rate Studies

The rate of appearance of gases in the test head as shown on the mass spectrometer and oscilloscope should be different depending upon the mechanism of backstreaming. Thus, gas diffusion through the top jet itself should be essentially instantaneous. However, backstreaming via solution in the oil and out with the vapor stream should involve appreciable time, probably many seconds. The problem is complicated by the nature of gases, different species having different degrees of oil solubilities, interplay of concentration streams due to the different fractionating processes in the pump and location of the leak.

The purpose of the initial experiments was to observe whether any significant differences could be observed when hydrogen, helium, or air was injected just below the top jet or in the bottom oil outside the jet stack. In all of these experiments the gas entered in pulses which were extremely constant for a given leak valve setting and a given gas. The usual phenomena consisted of a quiescent period of 3-10 seconds with a constant low gas (sometimes zero) concentration followed by a large pulse and then series of gradually diminishing pulses at fairly uniform periods. No visual differences could be detected in any series of experiments. The series of diminishing peaks indicates the absence of solubility effects which would not only change the period but also smooth them out.

These experiments were conducted at high leak rates so that they could be observed on the oscilloscope. Consequently, the effects we are concerned with at lower pressures could be completely masked by the magnitude of the leak. It appears that a more satisfactory scheme would involve the use of carrier gas containing several components of interest. The study of relative changes would eliminate the time element and permit differences to the detected at low partial pressures.

The values of the over-all compressibility factors for selected runs of helium and hydrogen are given in Table 8. Arbitrary judgement is involved in selecting the average value since most of the readings are pulsing to some extent. The values cited are presumably within 75% of the average value. The data are presently too limited for any definitive conclusions to be drawn. However, a plot (Figure 21) of the compressibility factors* versus the rates (abcissa changed to give rough equivalence in leak rates) show similar changes for both helium and hydrogen except for the initial value of the hydrogen at leak 3. This hydrogen value should be verified as well as additional determinations made at leak 4.

IV. SUMMARY OF RESULTS

A. Backstreaming Measurements

1. The initial measurements, using cold baffles, generally show higher and increasing values of backstreaming from those obtained with 40°F baffles. The phenomenon of pulsed pressure readings was present with the cold chevrons but not cold elbows or 40°F chevron traps.

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*Also called ultimate pressure ratio.

Table 8

DATA FROM GAS INJECTION STUDIES

Date	Gas	Leak		Fore Pressure, torr[3]		Pressure[4]		PPA, amps	Apparent CF[5]
		S/ΔP[1]	Location[2]	TC	McLeod	System	Gas		
7/29	O			3	5	1.9×10^{-7}	1.4×10^{-10} (S)	2×10^{-12}	
	He		4	11	14	1.8×10^{-7}	1.4×10^{-10} (S)	2×10^{-12}	
	He		4	41	70	1.8×10^{-7}	1.4×10^{-10} (S)	2×10^{-12}	
	He		4	40	63	1.8×10^{-7}	1.4×10^{-10} (S)	2×10^{-12}	
	He		4	53	83	1.7×10^{-7}	1.4×10^{-10} (S)	2×10^{-12}	6×10^{7}
7/30	O		4	7	8	1.7×10^{-7}	1.4×10^{-10} (S)	2×10^{-12}	
	He		4	93	150	1.65×10^{-7}	3.5×10^{-10} (S)	5×10^{-12}	4×10^{8}
	He		4	140	220		7×10^{-9} (S)	1×10^{-11}	
	He		4	200	325		5.6×10^{-8} (S)	8×10^{-11}	
	He		4	290	440	1.7×10^{-7}	2.7×10^{-7} (S)	3×10^{-9}	2×10^{6}
	He		4	350	530	2.05×10^{-7}	2.0×10^{-7} (P)	3.5×10^{-8}	3×10^{6}
	He		4	420	630	$.8\text{–}1.4 \times 10^{-6}$		$1 \to 1.5 \times 10^{-8}$	
7/31	O		-	8	10	1.75×10^{-7}	1.4×10^{-10} (S)	2×10^{-12}	
	He		4	310	480	1.9×10^{-7}	9×10^{-8} (S)	1.3×10^{-8}	
	He		4	380	530	2.3×10^{-7}	3.1×10^{-7} (P)	3.5×10^{-8}	2×10^{6}
9/23	O			3×10^{-3}		6.8×10^{-8}			
	He	65/48 (1.1)	2		(10×10^{-3})	7.0×10^{-7}	3.9×10^{-6} (P)	1×10^{-7}	2×10^{3}
	He	70/48 (9.7)	2		(10×10^{-3})	4.2×10^{-6}	2.3×10^{-5} (P)	8.3×10^{-7}	5×10^{2}
	He	74/48 (15)	2		(10×10^{-3})	9.5×10^{-6}	5.3×10^{-5} (P)	3×10^{-6}	2×10^{2}
9/24	O			3×10^{-3}		1.8×10^{-8}		6.8×10^{-10}	
	He	60/50 (.03)	3	3×10^{-3}	(10×10^{-3})	2×10^{-8}	1.7×10^{-9} (S)	7.0×10^{-10}	6×10^{6}
	He	65/50 (1.1)	3	4×10^{-3}	(10×10^{-3})	1.3×10^{-6}	2×10^{-6} (P)	6.5×10^{-7}	5×10^{3}
	He	70/50 (10)	3	7×10^{-3}	(10×10^{-3})	9.2×10^{-6}	5×10^{-5} (P)	7.5×10^{-6}	10^{3}
9/29	O			3×10^{-3}	(10×10^{-3})	1.8×10^{-8}		6.8×10^{-10}	
	H_2	60/50	2	3×10^{-3}	(10×10^{-3})	2×10^{-8}	1.7×10^{-9} (S)	7.0×10^{-10}	6×10^{6}
	H_2	65/50 (.3)	2	3×10^{-3}	(10×10^{-3})	1.3×10^{-6}	2×10^{-6} (P)	6.5×10^{-7}	5×10^{3}
	H_2	70/50 (1.7)	2	3×10^{-3}	(10×10^{-3})	9.2×10^{-6}	5×10^{-5} (P)	7.5×10^{-6}	2×10^{2}
9/29	O			3×10^{-3}	(10×10^{-3})	1.9×10^{-8}			
	H_2	60/50	3	3×10^{-3}	(10×10^{-3})	2.3×10^{-8}	1.6×10^{-8} (S)	6.5×10^{-10}	6×10^{5}
	H_2	65/50 (.3)	3	3×10^{-3}	(10×10^{-3})	1.8×10^{-7}	3×10^{-7} (P)	1×10^{-7}	3×10^{4}
	H_2	70/50 (1.7)	3	3×10^{-3}	(10×10^{-3})	3.3×10^{-6}	6×10^{-6} (P)	2.2×10^{-6}	2×10^{3}

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[1] Signifies an undefined leak. S/ΔP signifies valve setting/Inlet gas pressure. Setting is not reproducible and used only for comparison. Estimated rate given as (ml per min).

[2] Locations are: 1 - Test head, 2 - Between top and next lower jet, 3 - Liquid at bottom outside jet stack, 4 - Fore line ahead of TC gauge.

[3] Normally measured with thermocouple (TC) gauge. Absolute value of non-condensible measured with Lippincott-Fitting McLeod, lowest reading 1 mm = 1×10^{-3} torr.

[4] System pressure as read by ion gauge at r.t. Injection gas pressure determined by differences in ion gauged reading using Varian gauge constants (P) or by mass spectrometer sensitivities (S) from Table 16.

[5] Apparent compressibility factor. Fore pressure/head pressure.

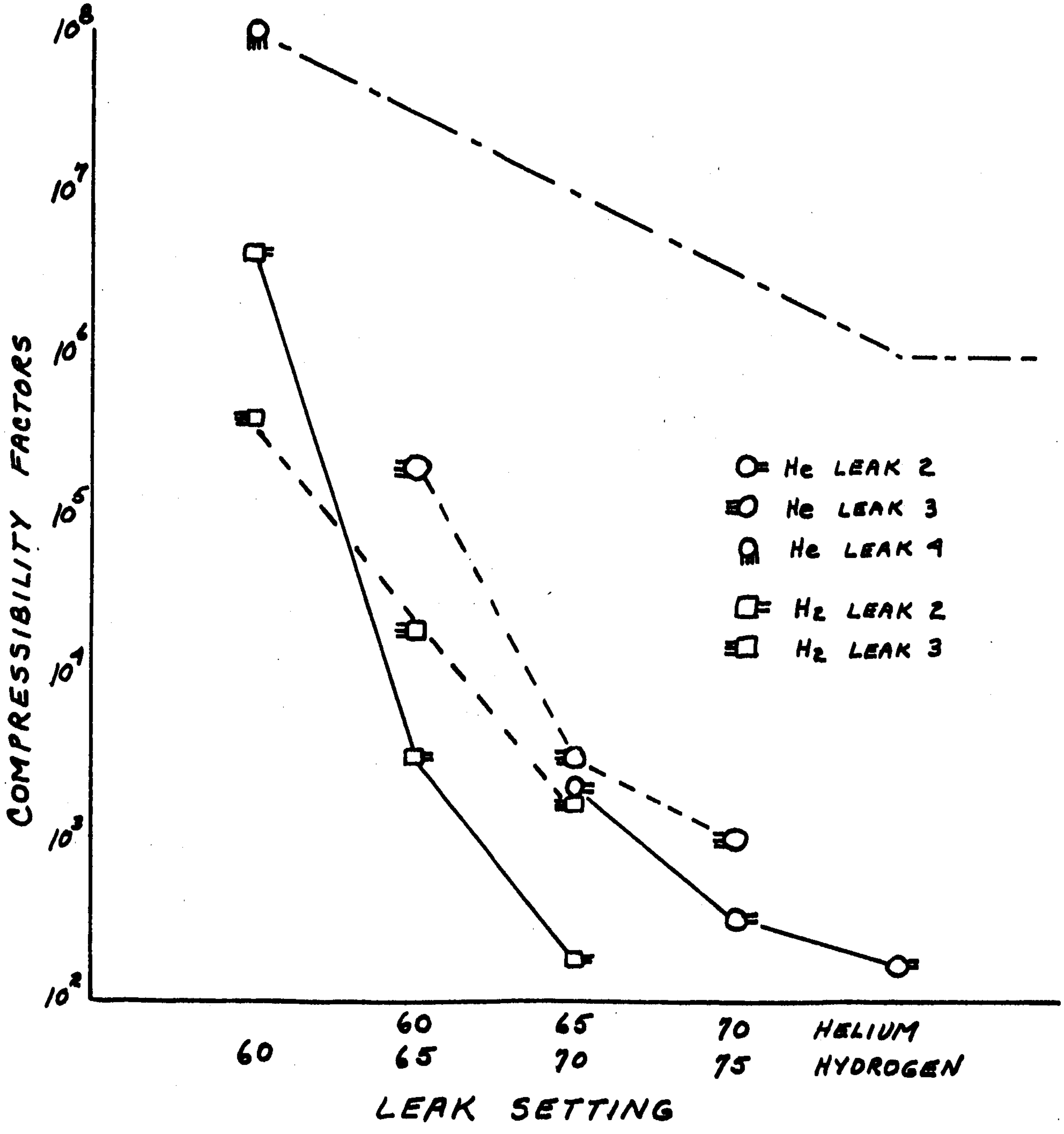

Figure 21

COMPRESSIBILITY FACTORS VERSUS LEAK RATE AND LEAK LOCATION

2. Essentially duplicate backstreaming values were obtained with -75°F baffles only in two cases after operating for various periods with room temperature baffles. These are Station 2 (HS-6-1500, old DC 705, BC-61) and Station 5 (HS-6-1500, fresh Convalex 10 and elbow). Station 3 (HS-6-1500, old Convalex 10, BC-61) was approaching the previous values at the time the baffle was changed to HN-6.

B. Backstreaming Measurements for Various Trap Types

1. The one bounce elbow baffle with Convalex 10 still gives the lowest backstreaming value.
2. A valid comparison between the chevron baffle (BC-61) and cryo baffle (HN-6) cannot be drawn due to the leak in the latter, and the variable nature of the values. However, the values in the initial runs indicate that any difference is probably minor.

C. Backstreaming as a Function of Distance

Results of backstreaming were too variable to attempt these measurements.

D. Backstreaming from Non-Fractionating Pump

Initial values of 0.2 mg/(cm^2)(min) are only twice as large and essentially the same as previous values. The values increased presumably due to light end build up. This situation has not been remediable as yet by high temperature exit water and lower fore line pressures.

E. Side Stream Purification

Side stream purification has only been effected by batch fractionating in situ of light ends out the top of the pump with warm baffle and cold collection plate.

This procedure decreased the light ends in Station 1 to a minimum constant value without any noticeable decrease in pulsing or system pressure (room temperature gauge). The system pressure (liquid nitrogen trapped gauge) decreased to 4×10^{-9} torr, which may also be due to operational changes.

No conclusion can be drawn for Station 3 with a defective baffle.

The purification, which was not needed to reduce backstreaming in Stations 2 and 5, was used to evaluate amount and nature of light ends.

F. Oil Stability

1. Long-Term Use

Both oils have been used for 2 years continuously with no apparent change in DC 705 and little apparent change in Convalex 10. The latter only showed very slight jet deposits that would not be appreciable in several more years of use and which are presumably due to oxidative degradation.

The amount of light ends (similar to parent compound) are 28 and 38 mg/(cm^2)(min) for DC 705 and Convalex 10 in oil used 2 years compared to 8 and 7 mg/(cm^2)(min), respectively, for fresh Convalex 10. The non-polyphenyl light ends are considerably greater in the newer oil and are presumably mainly a function of the self purging ability of the individual pumps.

2. Catalytic Degradation

Mass spectrometer studies of Station 3 with Convalex 10 show only mass 2 and 3 to pulse synchronously with the ion gauge. This implies catalytic degradation that could be due to heavy ends (old oil) or metal particles (sloughed nickel plating from defective baffle).

3. Effect of Air Leakage on Stability

Intentional air leaks lowering system pressures to 10^{-5} torr for several days cause Convalex 10 to show numerous mass peaks in 76 to 100 range in the 10^{-11} torr and lower range. These are not yet characterizable and essentially disappear within 3 hrs after cessation of leak. The magnitude and rate of disappearance indicate negligible effect of this massive leak.

4. Relative Stability

All present evidence indicates DC 705 is the more stable oil. The absence of deleterious effects is not definitive since more attention has been paid to polyphenyl ether and Station 5 is performing better than Station 3 with regards to backstreaming values and system pressures.

G. Estimation of Vapor Pressure

The effective vapor pressures were found to be approximately 2×10^{-9} torr and 8×10^{-9} torr for Convalex 10 and DC 705. These values are the same and 20 times recently reported values.

H. Pressure Pulsing

1. Explosive Vaporization of Light Ends

The explosive vaporization of light ends hitting the hot jet stack can explain much of the present pulsing phenomena and its relationship to differences in backstreaming values, cooled baffles, oils and operating procedures.

2. Moisture and Pulsing

Moisture was found to be partially responsible for the pulsing phenomena and was eliminated by additional care in opening the system for sampling.

3. Eruptive Boiling

No direct study was made of eruptive boiling. In small 2 inch pumps pressure pulses can often be correlated both audibly and visually to the eruptive boiling process. There is no general correlation since some small pumps boil more eruptively than others and show no indication of pulsing.

I. Mass Spectrometer Tests with Gas Injection

The tests to date involving gross time and concentration effects have not given any significant differences. They should be continued with dilute gases mixed in a carrier gas and the ratio changes used to measure gas transfer through the vapor jet or by solution mechanisms.

A partial pressure analysis is indispensible in characterizing, distinguishing, and separating the many different variables which influence backstreaming. Techniques have demonstrated recorder scan of pressure pulses in the low amperage ranges.

APPENDIX A

SYSTEM PRESSURE MEASUREMENTS

APPENDIX A

SYSTEM PRESSURE MEASUREMENTS

I. PREVIOUS TECHNIQUE

The previous work employed simple Bayard-Alpert gauges connected to the system by small tubulations, 5/16 in. I.D. x 4 in. long, without provision for trapping. It was demonstrated that the gauge readings, usually in the 10^{-9} torr range, were not in error due to gauge pumping phenomena by comparing two simultaneous gauge readings on one tubulation versus a single gauge reading. Furthermore, limited tests with liquid nitrogen in the baffles gave negligible change in pressure readings from those obtained with 40°F baffles. This indicated the absence of appreciable amounts of condensible components including light ends from the pump oil and outgassing from the baffle surfaces.

II. PRESENT TECHNIQUE

A. Description

The present work to October 1964 employed gauge tubulation 23 mm I.D. x 3 in. long with no provision for trapping. Since then all pertinent readings have been made with trappable gauges. The tubulation employs an H. S. Martin spherical trap (1 liter sphere with internal spherical reservoir, 1/2 in. clearance x 3 in. tubulation on each end). These traps are pictured in Figures 3 and 5. The pressure readings are differentiated by r.t. (room temperature) or LN_2 (liquid nitrogen cooled). This sytem has been found necessary in the present work. The light ends present due to operation with cooled baffles often result in room temperature readings of 10^{-6} torr versus 10^{-9} torr liquid nitrogen (Table 4, Station 1, Run 11/5). Also, valid readings cannot be obtained without liquid nitrogen on DC 705 systems (see below).

B. Experimental

No gauge calibrations have been made since we are concerned only with pressure ranges. No gauges, employing their nominal or calibration emission current, have been found to differ from any other by more than 20%. It was previously determined[13] by routine checks against a relatively unused standard gauge on the same tubulation that even a severely discolored gauge tube did not change its calibration in the worse case by more than a factor of 2.

It was recently observed that untrapped gauge readings on DC 705 systems were inconsistent. Our gauge readings are all made with at least 1/4 hour degassing by ion bombardment (Varian control) and a waiting period of at least 1 hour. Usually on alternate days a gauge is read in the morning after an 18-hour waiting period. Variations of consecutive readings became pronounced only with DC 705. Investigation* revealed that such untrapped gauges, after degassing and $\sim$1 hour wait, would read

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*Station 2 after operation with room temperature baffles and presumably with very small quantities of light end oil molecules using individually tubulated gauges.

1 decade lower than liquid nitrogen trapped pressure reading. The untrapped gauge reading would then start to rise and in a period, which varied in an uncharacterized manner from 1 to several hours, balance out 1 decade above the liquid nitrogen trapped pressure reading.

APPENDIX B

MASS SPECTROMETER INFORMATION

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MASS SPECTROMETER INFORMATION

I. EQUIPMENT SPECIFICATIONS

The general equipment and conditions used in the mass spectrometer tests are listed in Table 9. These conditions give maximum definition for 28 N_2^+ and are used in all tests unless stated otherwise. The mass spectrometer tube is inserted (Figure 5) radially into the test spool through a 1-inch opening. The latter is sealed by double silicone O-rings provided with a guard vacuum. The tube was baked out repeatedly for periods of 2 to 18 hours at temperatures of 250°C.

II. SENSITIVITY

A. Objectives

Knowledge of the sensitivity for different gases of the partial pressure analyzer is essential if actual partial pressures of various gases are to be determined in experiments designed to measure backstreaming, oil degradation, compressibilities, effectiveness of baffles, etc. Knowledge of reproducibility of these sensitivity values is needed so that the reliability of the results can be assessed. Determining the effect of system variables on sensitivity is part of this requirement. Gases such as nitrogen, oxygen, helium, and hydrogen are directly involved in these experiments. Benzene is of interest because it gives a strong peak at a high mass (78), and this can conveniently be

Table 9

SPECIFICATIONS OF MASS SPECTROMETER TEST EQUIPMENT

1. General Electric Partial Pressure Analyzer
    - a. Purchase Order 29257, 5/19/64
    - b. Electronic control, fast and slow scan with cable and recorder mounted cabinet.
      Model 22PC110- P&L-71, Serial No. 30055
    - c. Metal analyzer tube, straight tubulation
      1 in. diameter, 305 L, stainless steel
      Model 22PT110- P&L-71, Serial No. -62
      Electron multiplier numbers: Z6-8007, 6413, 183-6
    - d. 3KG magnet (2.85 kg)
      Model 22PM103- P&L-71, Serial No. 4016
    - e. 5KG magnet (4.8 kg)
      Model 22PM105- P&L-71, Serial No. 5056
    - f. Acceleration voltmeter after 11/20/64
      Assembly Product Instrument, super calibrated
      Taut Band, 50 μ A,
      Catalog No. 65F302 (Allied Radio Corporation)
2. Hewlett Packard Oscilloscope
    - a. Purchase Order 37163 (10/7/64)
    - b. Model 130 C, Serial 425-02474
    - c. Input impedance 1 meg ohm shunted by 45 pf
    - d. Maximum sensitivity 0.2 mv/cm
3. Standard operating conditions (set for maximum N_2 sensitivity)
    - a. Emission current, 1 ma (tungsten, pins 1 and 2)
    - b. Electron voltage, 70 v
    - c. Drawout, 4.6 units
    - d. Deflection 1, 4.7 units
    - e. Deflection 2, 5.0 units
    - f. Electron multiplier, 2000 volts (setting No. 4)
    - g. Scan rate: ammeter - 10 min; oscilloscope - 0.1 sec

used to determine the sensitivity at mass 78. Sensitivities for intermediate masses, such as are produced by degradation of oil, can then be estimated by interpolation. Furthermore benzene may be used to determine the accommodation coefficient of baffles, so its sensitivity will be needed for this purpose.

B. Calculation Methods

The sensitivity of the partial pressure analyzer for a particular gas is defined as the ratio of the response of the analyzer, in amperes, to the actual pressure of the particular gas in the vacuum system. There are three ways to determine the actual partial pressure. One is to leak into the system the gas to be measured, at such a rate that it is the main gas in the vacuum system, and to determine its pressure with a Bayard-Alpert gauge. This is called the method of direct comparison with the Bayard-Alpert gauge.

If the leak rate is not sufficient to raise the system pressure 100-fold over the pressure without a deliberate leak, then the gas in the system cannot be assumed to consist entirely of the leaked gas. It is desirable to correct the pressure for this effect. This can be done by subtracting the no-leak Bayard-Alpert reading from reading with leak. The result is then multiplied by the Bayard-Alpert gauge calibration factor for the leaked gas.

A second method is to introduce the gas as a controlled small per cent of a carrier gas such as air or nitrogen. This is an indirect method of comparison with the Bayard-Alpert gauge, and that of the minor constituent from

$$P_x = P_{carrier} X_x \sqrt{\frac{M_x}{M_{carrier}}}$$

where

P = pressure, torr

X = mole fraction

M = molecular weight

subscript x = minor gas constituent

The square root expression gives the theoretical effect of pumping speed on the pressure in the system of gases of different molecular weights.

A third method employs a calibrated leak, V, into the system. Then

$$P_x = 500\sqrt{\frac{M_x}{28}} \left[\frac{V}{(60)(1000)}\right]$$

where

V = leak rate, ml/min

500 = pump speed for nitrogen, liters/sec

28 = molecular weight of nitrogen.

This equation can also be used to calibrate the leak, by measuring the pressure with the Bayard-Alpert gauge. It was found that the present leak valve (Phillips-Granville Catalog No. 9101-S) cannot be set reproducibly enough to make this method of much use except to estimate absolute values.

C. Results

A series of sensitivity measurements for helium were made over a period of several months and the results are given in Table 10. The results are very reproducible on each day (within 20 per cent), except when the leak rate is so low that the system pressure differences do not give a reliable reading of the leak gas pressure. This confirms the validity of the method of correcting the Bayard-Alpert pressure for gases other than the leaked gas in the system. No reason is known for the change in sensitivity from day to day. In the later experiments the sensitivity is between 0.011 and 0.019 amp./torr.

Sensitivities were also determined for a number of other gases over a period of months, and for various experimental conditions. Results are given in Table 11. Sensitivities for nitrogen and oxygen were determined by both direct and indirect comparison with the Bayard-Alpert gauge. Values for water, methanol, and benzene were determined by indirect comparison. Sensitivities for nitrogen vary from 0.045 to 0.17, for oxygen from 0.008 to 0.13, and for water from 0.07 to 0.28. In general the values for the small magnet are lower than for the large magnet. Comparison of sensitivities measured on the same day show that changing the magnet changes sensitivity by a factor of two for masses 28 and 32. The effect is even larger for benzene (mass 78), for which the sensitivity is 0.00001 with the small magnet and is 0.003 to 0.022 with the large magnet.

Table 10

HELIUM SENSITIVITIES BY DIRECT COMPARISON WITH BAYARD-ALPERT GAUGE

Experiment	Leak Setting	B-A Gauge Helium Press., torr (N_2 equiv)	Sensitivity, amps/torr	Experiment	Leak Setting	B-A Gauge Helium Press., torr (N_2 equiv)	Sensitivity, amps/torr	Experiment	Leak Setting	B-A Gauge Helium Press., torr (N_2 equiv)	Sensitivity, amps/torr
8/12	57.5/0	3×10^{-8}	.11	9/17	64/0	3.3×10^{-6}	.054	10/6	65/52	9.0×10^{-7}	.067
8/13	57	5×10^{-8}	.046		65	4.1×10^{-6}	.060		67.5/52	3.6×10^{-6}	.011
	58	2.1×10^{-7}	.041		66	5.0×10^{-6}	.067		70/52	9.0×10^{-6}	.010
	60	8.9×10^{-7}	.024		67	5.6×10^{-6}	.080		75/52	3.4×10^{-5}	.012
	61	1.3×10^{-6}	.028		68	6.1×10^{-6}	.084		77.5/52	9.0×10^{-5}	.011
	62	2.2×10^{-6}	.026		69	7.8×10^{-6}	.093		80/52	1.5×10^{-4}	.013
8/14	58	1.2×10^{-7}	.067		70	1.1×10^{-5}	.086		70/52	1.2×10^{-5}	.013
	59	2.7×10^{-7}	.067		71	1.2×10^{-5}	.083		65/52	2.6×10^{-6}	.012
	60	5.6×10^{-7}	.061	9/23	60/67	3.4×10^{-6}	.012		65.5/52	8.0×10^{-7}	.010
	61	8.4×10^{-7}	.060		65/66	1.4×10^{-5}	.014		60/52	3.4×10^{-8}	.030
	62	1.2×10^{-6}	.072		70/63	1.2×10^{-4}	.021		72.5/52	1.6×10^{-5}	.013
	63	1.7×10^{-6}	.063		74/56	1.7×10^{-4}	.053	10/5	60/52	3.0×10^{-7}	.0007
	64	2.1×10^{-6}	.071		74/52	1.2×10^{-4}	.063		62.5/52	9.0×10^{-7}	.006
	65	3.0×10^{-6}	.074		65/48	3.9×10^{-6}	.026		65/52	2.1×10^{-6}	.012
	66	3.7×10^{-6}	.075		70/48	2.3×10^{-5}	.036		67.5/52	8.4×10^{-6}	.011
	67	5.0×10^{-6}	.077		74/48	5.3×10^{-5}	.057		70/52	1.4×10^{-5}	.014
	68	6.2×10^{-6}	.088	9/24	70/50	9.0×10^{-6}	.024		72.5/52	2.5×10^{-5}	.016
9/17	59	1.4×10^{-6}	.064		65/50	1.4×10^{-6}	.036		75/52	5.6×10^{-5}	.016
	60	1.6×10^{-6}	.065		60/50	5.6×10^{-8}	.009		77.5/52	1.2×10^{-4}	.017
	61	1.8×10^{-6}	.074		65/52	7.8×10^{-7}	.041		80/52	2.0×10^{-4}	.017
	62	2.2×10^{-6}	.073		70/52	1.0×10^{-5}	.035		82.5/52	3.4×10^{-4}	.019
	63	2.7×10^{-6}	.069		65/52	1.3×10^{-6}	.040				

Note: Dynode voltage setting 2000, electron voltage 70, emission current 1.0 ma. Based on helium pressure as determined by Bayard-Alpert Gauge.

Table 11

SENSITIVITIES** FOR NITROGEN, OXYGEN, WATER, METHANOL, AND BENZENE

Experiment	Leak Setting	Magnet	B-A Gauge Pressure, torr	Leaked Gas	Sensitivity, amp/torr				
					Nitrogen (28)	Oxygen (32)	Water (18)	Methanol (31)	Benzene (78)
7/22	-	S	2.1×10^{-5}	N_2	.048				
7/21	-	S	2.0×10^{-5}	N_2	.066				
7/22	-	S	2.1×10^{-5}	N_2	.062				
7/22	-	S	1.9×10^{-5}	N_2	.17				
7/23	-	S	3.1×10^{-5}	O_2		.0082			
7/22	-	S	3.7×10^{-5}	O_2		.011			
11/19	70/0	S	*3.2×10^{-7}	Air	.10	.057			
10/13	75/0	S	8.5×10^{-7}	Air	.055	.031	.22		
11/20	77/0	S	*8.0×10^{-6}	Air	.070	.060	.17	.36	
11/20	77/0	S	*1.0×10^{-5}	Air	.051	.062	.20		.000013
10/19	75/0	L	2.8×10^{-6}	Air	.045	.034	.11		
10/26	75/0	L	3.0×10^{-6}	Air	.17	.13	.17		
10/26	80/0	L	1.0×10^{-6}	Air	.11	.08	.08		
10/26	70/0	L	6.7×10^{-7}	Air	.093	.063	.16		
10/14	75/0	L	7.7×10^{-7}	Air	.057	.050	.066		
10/15	80/0	L	2.6×10^{-5}	Air	.097	.073	.13		.016
10/16	77.5/0	L	1.5×10^{-5}	Air	.083	.070	.23		.022
10/16	75/0	L	6.0×10^{-6}	Air	.068	.039	.28		.014
10/14	80/0	L	4.8×10^{-6}	Air	.13	.083	.11		.011
11/20	77/0	L	*1.0×10^{-5}	Air	.11	.10	.35		.003
11/20	77/0	L	*1.1×10^{-5}	Air	.092	.078			.007

*Bayard-Alpert gauge trapped with liquid N_2. This lowered the reading 10-20%.

**Dynode voltage 2000, electron voltage 70, emission current 1.0 ma.

Table 12 gives sensitivities for hydrogen. The values are between 0.28 and 0.55.

The sensitivity of the partial pressure analyzer is of course affected by the amplification of the signal in the electron multiplier. This effect is evaluated in Table 13* since special requirements may necessitate the use of different dynode voltages. For each gas an increase of 500 volts dynode voltage triples the sensitivity. In all subsequent measurements a dynode voltage of 2000 volts was used. Further measurements should be done at other conditions to determine whether a higher voltage is beneficial or if it causes excessive noise.

The position of the magnet affects both the sensitivity and the position of the mass peaks (in terms of acceleration voltage). The data in Table 14 was obtained to see if this factor could explain large variations in sensitivity on different days. A procedure for positioning the magnets at a certain location has been adopted, and only a slight effect is now noticeable.

The sensitivity differs for the two sizes of magnet used with the partial pressure analyzer. Table 15 shows this effect for several masses and the effect at lower system pressure. In general the large magnet gives about twice the sensitivity of the small magnet. However, the sensitivity of the small magnet falls off

---

*Note: The sensitivities for helium and hydrogen in this table are much lower than subsequent measurements in Tables 10 and 12. The reason may be that the hydrogen leak source was not purged of air, so that the actual partial pressure was lower than that used in Table 15. The relative effect of dynode voltage on sensitivity is still valid, however.

Table 12

SENSITIVITIES FOR HYDROGEN BY DIRECT COMPARISON WITH BAYARD-ALPERT GAUGE

Experiment	Leak Setting	B-A Gauge Hydrogen Press., torr (N_2 equiv)	Sensitivity, amps/torr
9/29	65/50	2.3×10^{-6}	.28
	67.5/50	7.0×10^{-6}	.39
	70/50	1.6×10^{-5}	.47
	65/50	1.4×10^{-5}	.25
	62.5/50	6.8×10^{-6}	.32
	65/50	2.9×10^{-7}	.35
	67.5/50	2.3×10^{-7}	.35
	70/50	5.9×10^{-6}	.37
	65/50	7.7×10^{-6}	.43
	62.5/50	9×10^{-8}	.55
11/10	0*	7.2×10^{-8}	.32

---

*The mass spectrum showed that hydrogen was the main component. Probably arising from degradation of the oil.

Table 13

EFFECT OF DYNODE VOLTAGE ON SENSITIVITY* FOR VARIOUS GASES

Experiment	System Pressure, torr (N_2 equiv)	Leaked Gas	Sensitivity at Dynode Voltage, amps/torr 1500 v	2000 v	2500 v
7/23	6.0×10^{-5}	H_2 (2)	.0013	.0033	.0092
7/22	3.4×10^{-5}	He (4)	.0011	.0041	.026
7/22	2.1×10^{-5}	N_2 (28)	.021	.062	.19
7/23	3.1×10^{-5}	O_2 (32)	.0025	.0082	.026

---

*Other conditions: small magnet, electron voltage 70, emission current 1 ma.

Table 14

EFFECT OF MAGNET POSITION ON SENSITIVITY VALUES FOR WATER

Magnet Position	$H_2O(18)$	m/e	Amps	% Deviation Δm	% Deviation ΔA
Normal	1650 Volts	18	9.0×10^{-9}		
1/4 in to right	1680 Volts	17.6	7.0×10^{-9}	2	20
1/4 in to left	1620 Volts	18.3	9.0×10^{-9}	2	-

Table 15

EFFECT OF MAGNET SIZE ON SENSITIVITY VALUES

	Small Magnet			Large Magnet			Ratio of Sensitivities, LM/SM
Mass	Peak, volts	amps	Sensitivities, amp/torr	Peak, volts	amps	Sensitivities, amp/torr	
12*	605	3.5×10^{-11}		2400	6.4×10^{-11}		2
18*	545	2.0×10^{-9}		1630	7.5×10^{-9}		3.7
18	275 x 2	9.0×10^{-9}	.16	1530	1.1×10^{-8}	.20	
18	275 x 2	1.1×10^{-8}	.20	1530	1.1×10^{-8}	.30	1.5
28*	360	8.2×10^{-10}		1050	3.3×10^{-9}		4
28	172 x 2	4.2×10^{-7}	.052	999	9.5×10^{-7}	.12	
28	177 x 2	4.1×10^{-7}	.051	990	1.0×10^{-6}	.13	
28	177 x 2	4.0×10^{-7}	.050	990	7.0×10^{-7}	.087	2
32*	320	2.4×10^{-10}		920	1.1×10^{-9}		5
				890	1.3×10^{-9}		
32	155 x 2	1.3×10^{-7}	.062	880	2.1×10^{-7}	.10	1.5
32				870	2.0×10^{-7}	.10	
40*	255	1.7×10^{-11}		705	5.2×10^{-11}		3
40	125 x 2	3.5×10^{-9}	.035	700	6.0×10^{-9}	.06	2
40	125 x 2	2.7×10^{-9}	.027				
44	114 x 2	6.0×10^{-10}		322 x 2	1×10^{-9}		2
44	113 x 2	5.0×10^{-10}					
44	113 x 2	5.0×10^{-10}					
44	113 x 2	5.0×10^{-10}					
44	113 x 2	5.0×10^{-10}					
78	64 x 2	8.0×10^{-11}	4.2×10^{-5}	355	6.0×10^{-9}	.0032	
78	64.5 x 2	11×10^{-11}	5.8×10^{-5}	356	6.0×10^{-9}	.0032	
78	64.5 x 2	15×10^{-11}	8.0×10^{-5}	356	9.0×10^{-9}	.0047	60

*Experiment 11/13 system No. 3 no leak. Ps = 9.3×10^{-8} torr.
Rest are experiment 11/20 system No. 3 leak 77/0 with benzene. Pressure 1.0×10^{-5} (LN_2 trapped B-A gauge).

at higher masses, so that the large one gives 60 times the sensitivity at mass 78. Presumably the same fall off occurs with the large magnet and effects the sensitivities for the higher masses including benzene.

Table 15 also indicates the reproducibility of the peak voltages and the sensitivities during a single experiment. The reproducibility of the peak voltage is not sufficient to identify higher masses with the voltmeter supplied with the partial pressure analyzer. We replaced this meter with a meter of the taut-band type (see Table 9) which is accurate to 1/2 per cent of full scale. This has helped to identify masses. The results in Table 15 were obtained with this meter. The scale factor relating peak voltage to mass still varies from experiment to experiment, especially when the magnet is moved. It appears to be essential to introduce a tracer near the highest mass to be identified. The calibration factor deduced from mass 32 is not sufficiently precise to identify masses near 80. Benzene can serve this purpose if it is present, or a rare gas such as xenon could be used. It would have the advantage of not introducing fragments of lower mass.

D. Summary of Sensitivity Values

A summary of the best values to December 1964 are listed in Table 16. These values are used to estimate partial pressures when no other sensitivity calibration points are available.

Table 16

SUMMARY OF SENSITIVITIES

Gas	Mass M	Sensitivities: Small Magnet amps/torr	Sensitivities: Small Magnet Relative/N_2	Sensitivities: Large Magnet amps/torr	Sensitivities: Large Magnet Relative/N_2	Sensitivity* of B-A Gauge Relative/N_2
H_2	2	.4	6.7			.5
He	4	.015	.25			.16
H_2O	18	.18	3.0	.25	2.8	1.1
N_2	28	.06	1.0	.09	1.0	1.0
O_2	32	.04	.67	.08	.9	.8
MeOH	30	.36	6.0			
Benzene	78	.00001	.0002	.003	.03	

*Data from Varian Company.

E. Special Effects

Hydrogen, mass 2 and 3, and helium, mass 4, exhibit the unexplained phenomena of requiring a period of three minutes before the peak can be read on either the oscilloscope or micro micro ammeter. Once this peak has developed, millisecond variations can be recorded. This phenomenon is related also to the variation in magnitude of sweep voltage. For example, if mass 2 is being scanned manually, the voltage can be moved from 4750^V to 3750^V and left at the latter for minutes. Upon return to 4750^V, mass 2 value is obtained immediately. However, if the voltage goes to 3500^V for a millisecond,the full 3 minutes is required to develop mass 2 at 4750^V. Somewhere around 4000^V and lower the peak magnitude on return to 4750^V suffers a gradual decrease. This lower value returns instantly but recovery to the correct value takes 3 minutes. The mass 2, 3, and 4 peaks, if essentially eliminated by decreasing the emissivity current, return immediately upon resumption of current. These effects can be precisely delineated by the oscilloscope display versus magnitude of the voltage sweep. Care must be taken when looking for these peaks that sufficient time is allowed for them to develop.

A similar but extremely small effect was observed with other peaks. Peak heights on the oscilloscope at 0.1 sec scan require a reasonably narrow scan in order to duplicate values on the recorder. No differences in peak heights have been observed using 10 or 30 minute scans as reported by another investigator.[12]

F. Cracking Patterns

1. Objectives

An objective was to determine the mass frequencies of the cracking patterns of some substances which are to be used in other parts of the program. Measurements were made introducing these materials into the vacuum system, and the results were compared with published cracking patterns. These patterns have been found useful even in the case of simple gases like hydrogen. In the latter case comparison of mass 2 and mass 3 on the oscilloscope and recorder, respectively, permits a direct evaluation of the time constant. The pulse measurement of mass 3 constitutes proof that no other recordable mass peak is pulsing synchronously with hydrogen.*

2. Procedures

The cracking patterns of individual gases were determined by leaking the gas into the vacuum system and measuring the mass spectrum with the partial pressure analyzer. Volatile liquids such as benzene and methanol were introduced by saturating an air stream with the liquid and injecting the air. In one experiment methanol without air leaked in through the defective baffle.

3. Results

Mass frequency patterns are given in Tables 17 to 22 for hydrogen, water, nitrogen, air, methanol, and benzene. In each

---

*The pulsing of isolated peaks with a frequency of minutes may be missed unless a recorder scan is made of the individual peaks over a fairly long period.

Table 17

HYDROGEN MASS FREQUENCY PATTERN

Mass	Relative Frequency This work	 MRI Report 2675-P	 API
1			2.1
2	100.	100.	100.
3	.1	.27	-

Table 18

WATER MASS FREQUENCY PATTERN

	Relative Frequency		
	This work		
Mass	LM	SM	Dow
18	100.	100.	100.
17	25.	25.	21.
16			.9
19			.5
20			.3

Table 19

NITROGEN MASS FREQUENCY PATTERN

	Relative Frequency	
Mass	This work SM	API
28	100.	100.
14	5.	5.2
29	-	.7

Table 20

AIR MASS FREQUENCY PATTERN

	Relative Frequency					
Mass	Air This Work, 11/18 SM	N_2 API	O_2 API	H_2O Dow	CO_2 API	Ar API
28	100.	100			8.2	
32	13.		100			
14	11.	5.2				
18	18.			100		
17	3.7			21		
16	4.0		5.1		9.4	
12	.2				6.7	
20	.2					13.
40	1.0					100
44	.3				100	

Table 21

METHANOL MASS FREQUENCY

	Relative Frequency		
Mass	From API	No air*** LM	With air SM
31	100	100	100
29	42	92	100
32	72	84	80*
33	1	16	30
30	8	8	10
2	-	-	74
15	-	150	10
13	-	-	9
12	-	-	6
27	-	-	8**
Conditions:			
Pressure		2.0×10^{-7} RT	1.2×10^{-5} RT

---

[*] After subtracting the contribution from oxygen in the air.

[**] After subtracting the contribution from nitrogen in the air.

[***] Composite of two experiments.

Table 22

BENZENE MASS FREQUENCY PATTERN
MOST PROMINENT PATTERN

Mass	API	Relative Frequency*
2		(64)
12		(9)
13		(3.4)
15		(57)
25	.5	2
26	3.4	14
27	3.0	11
29		(106)
30		(7)
31		(78)
37	4.1	7
37.5	1.3	-
38	5.8	11
39	14.2	36
49	2.5	5
50	15.7	32
51	18.6	36
52	19.4	36
53	8.4	-
63	2.8	5
73	1.5	-
74	4.6	8
75	1.6	-
76	6.0	-
77	14.0	-
78	100.0	100
79	6.0	-

( ) Values are not associated with benzene spectrum as such.
* All determined with large magnet except mass 2.

case data from another source[9,18] is given. The results agree satisfactorily.

Our results for air in Table 20 are compared with patterns for its constituents showing that nitrogen, oxygen, water, carbon dioxide, and argon are responsible for the observed peaks.

In the case of methanol we have observed important peaks at mass 2 and 15. The results at 15 should be redetermined because of poor reproducibility.

In the case of benzene a spectrum with many mass peaks was determined and is shown in Figure 22. The most prominent peaks are listed in Table 22. Prominent peaks due to air are masses 28, 32, 14, 16, 40, and 44. Those due to water are 18 and 17. Masses higher than 78 are probably due to oil degradation products. The same may be true for some of the less important peaks between masses 35 and 78.

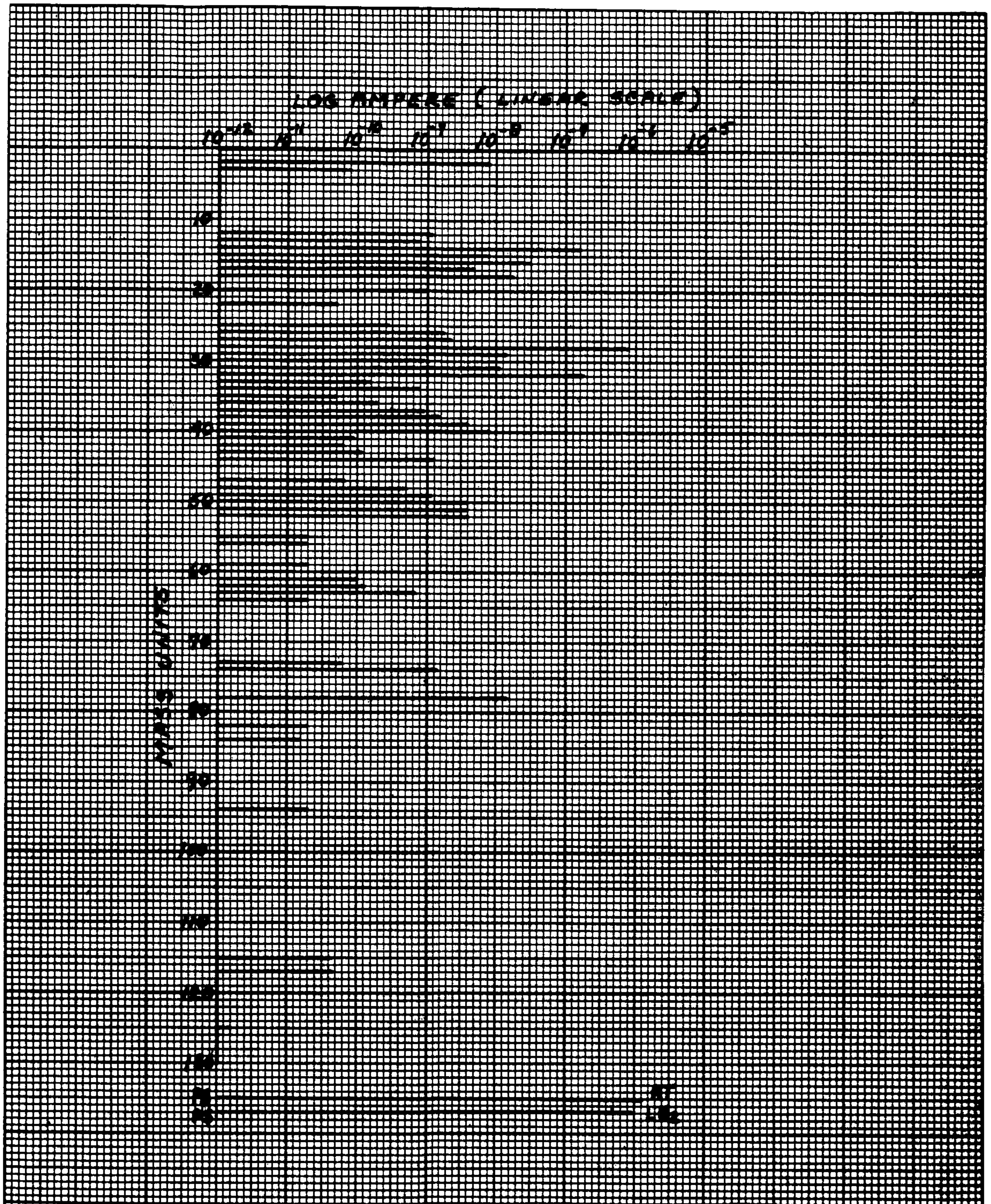

Figure 22

MASS SPECTRUM OF BENZENE SYSTEM

Air leak rate 77/0 over benzene at 80°F
Benzene = 13 mole percent

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