Report No. IITRI-C6030-9
(Progress Report)

BACKSTREAMING FROM OIL DIFFUSION PUMPS

National Aeronautics
and Space Administration

IIT RESEARCH INSTITUTE

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I. INTRODUCTION

The present program is concerned with evaluating the backstreaming characteristics obtained with oil diffusion pumps in modern, well baffled systems. In addition, the backstreaming characteristics of turbo-molecular pumps are also being evaluated.

The oil diffusion pumps are arranged in five test stations and employ fractionating and non-fractionating pumps, one bounce right angle elbow baffle, three types of commercial baffles, and the oils: DC 705, Convalex 10 and OS 124. It has been found that under optimum conditions an oil deposit equivalent to a monolayer is formed. The amount of this deposit is invariant with long or short runs. This low backstreaming value has been obtained with both pumps and both oils, but with only the two baffles, right angle elbow baffle and the chevron baffle.

The present effort has been confined to completing as much unfinished business as possible since experimental effort will terminate on March 31. The major effort was expended on developing an analytical method for turbo-molecular pump oil using gas chromatography.

II. EXPERIMENTAL WORK - OIL DIFFUSION PUMPS

The effect of leaks in the fine and rough side was evaluated. Table 1 shows that the admission of oxygen into the fine vacuum side of a station employing Convalex 10 had no effect on the amount of backstreaming oil deposit, the value being 0.7×10^{-4} mg/cm^2 over a run period of 290 hr. Table 2 shows that a small leak of air into the fore line (50 μ) had no effect but a large fore line leak (fore line pressure of 400 μ) caused pulsed pressure readings in the fine vacuum and an oil deposit of 19×10^{-4} mg/cm^2 over a run of 146 hr.

The evaluation of the HN-6 trap (Table 3) shows that it gives a backstreaming deposit of 7 to 14×10^{-4} mg/cm^2 compared to the standard value of 1×10^{-4} mg/cm^2 (Run 210). The larger values for this HN-6 trap with cooled sides are greater than those obtained with a BC-61 trap having room temperature walls (Run 205).

Table 4 shows the evaluation of the Cryo-Sorb trap. The cooling temperatures were varied from room temperature to liquid nitrogen temperature. It can be seen that the values varied from 8 to 55×10^{-4} mg/cm^2 whereas the BC-61 baffles gave 2 to 3.5×10^{-4} mg/cm^2. The latter values are the same order but significantly higher than the value of 1 mg/cm^2

TABLE 1

Backstreaming Measurements - Effect of Leaks in Fine Side

Run	Station	Oil	Bakeout Temp. °F	Run Hours	Trap Temp. °F	Fore Pressure μ	System Pressure, torr		Deposit Weight, mg/cm^2		Net Backstreaming,	
							Room Temp.	LN_2	Total	Analytical Blank	Deposit mg/cm^2	Rate mg/cm^2-min.
224[1)]	1	Convalex 10	212	126	-75	8		6.0×10^{-6}	1.35×10^{-4}	0.0	1.35×10^{-4}	$.0179 \times 10^{-6}$
166[2)]	1	Convalex 10	212	290	-75	8	3×10^{-7}	6×10^{-8}	0.75×10^{-4}	$.05 \times 10^{-4}$	0.70×10^{-4}	$.0041 \times 10^{-6}$
202[3)]	1	Convalex 10	212	306	-75	8	5×10^{-7}	1×10^{-9}	1.02×10^{-4}	0.13×10^{-4}	0.89×10^{-4}	$.0048 \times 10^{-6}$
180[3)]	1	Convalex 10	212	75	-75	8	1×10^{-7}	1×10^{-9}	1.02×10^{-4}	0.0	1.02×10^{-4}	$.0227 \times 10^{-6}$

1) With oxygen in the fine vacuum side $P_{LN_2} = 6 \times 10^{-6}$ torr. After 30 min., no leak, $P_{LN_2} = 1.6 \times 10^{-8}$ torr.

2) With air in the fine vacuum side $P_{LN_2} = 6 \times 10^{-8}$ torr. After 30 min., no leak, $P_{LN_2} = 2.1 \times 10^{-9}$ torr.

3) These runs represent the average low backstreaming values.

TABLE 2

Backstreaming Measurements - Effect of Leaks in Fore Line

Run	Station	Oil	Bakeout Temp. °F	Run Hours	Trap Temp. °F	Fore Pressure μ	System Pressure, torr		Deposit Weight, mg/cm^2		Net Backstreaming,	
							Room Temp.	LN_2	Total	Analytical Blank	Deposit mg/cm^2	Rate mg/cm^2-min.
222 1)	5	OS 124	212	146	-75	400	$(5 \rightarrow 20 \times 10^{-7})$		19.2×10^{-4}	0.15×10^{-4}	19.05×10^{-4}	0.214×10^{-6}
175 2)	5	OS 124	212	313	-75	50	5×10^{-9}	4×10^{-9}	1.02×10^{-4}	0.0	1.02×10^{-4}	0.0059×10^{-6}
193 3)	5	OS 124	212	292	-74	5	6×10^{-8}	5×10^{-8}	1.36×10^{-4}	0.46×10^{-4}	0.90×10^{-4}	0.0052×10^{-6}
163 3)	5	OS 124	212	148.5	-76	4	5×10^{-9}	4×10^{-9}	0.92×10^{-4}	0.25×10^{-4}	0.63×10^{-4}	0.0075×10^{-6}

1) Fore line pressure 400 μ by TIC gauge, pulsing of pressure in fine vacuum.

2) Fore line pressure 50 μ by TIC gauge.

3) Two runs to compare deposit and time with no leak in fore line.

TABLE 3

Backstreaming Measurements - Evaluation of HN-6 Baffle

Run	Station	Oil	Bakeout Temp. °F	Run Hours	Trap Temp. °F	System Pressure, torr: Room Temp.	System Pressure, torr: LN_2	Deposit Weight, mg/cm^2: Total	Deposit Weight, mg/cm^2: Analytical Blank	Net Backstreaming, Deposit mg/cm^2	Net Backstreaming, Rate mg/cm^2-min.
216	3	Convalex 10	212	293	-75	3×10^{-9}	2×10^{-9}	12.0×10^{-4}	$.15 \times 10^{-4}$	11.85×10^{-4}	0.067×10^{-6}
223	3	Convalex 10	212	146	-75	5×10^{-9}	2×10^{-9}	7.41×10^{-4}	$.25 \times 10^{-4}$	7.16×10^{-4}	0.0815×10^{-6}
228	3	Convalex 10	212	249.5	-25 1)	5×10^{-9}	2×10^{-9}	13.7×10^{-4}	$.15 \times 10^{-4}$	13.5×10^{-4}	0.090×10^{-6}
233	3	Convalex 10	212	150	-75	4×10^{-9}	2×10^{-9}	9.5×10^{-4}	$.23 \times 10^{-4}$	9.3×10^{-4}	0.102×10^{-6}
235	3	Convalex 10	212	77	-75	8×10^{-9}	2×10^{-9}	7.5×10^{-4}	$.13 \times 10^{-4}$	7.4×10^{-4}	0.158×10^{-6}
238	3	Convalex 10	212	67	-75	8×10^{-9}	2×10^{-9}	7.5×10^{-4}	$.28 \times 10^{-4}$	7.2×10^{-4}	0.180×10^{-6}
243	3	Convalex 10	212	167	-75 2)	9×10^{-9}	4×10^{-9}	(9.9×10^{-4}) 2)	$.26 \times 10^{-4}$	(9.6×10^{-4}) 2)	(0.096×10^{-6}) 2)
205	3	Convalex 10	212	411	-44 3)	2×10^{-8}	3×10^{-9}	5.4×10^{-4}	$.17 \times 10^{-4}$	5.2×10^{-4}	0.0211×10^{-6}
210	3	Convalex 10	212	148	-68/-10 4)	2×10^{-8}	2×10^{-9}	0.92×10^{-4}	0.0	0.92×10^{-4}	0.0103×10^{-6}

1) Using HN-6 Baffle at a -25°F.

2) Using HN-6 Baffle with warm sides, trap itself at -75°F.

3) Chevron Baffle with warm sides. Refrigeration system was not cooling properly.

4) Chevron Baffle with cold sides. Refrigeration system still not cooling [illegible]

TABLE 4

Backstreaming Measurements - Evaluation of Cryo-Sorb Baffle

Run	Station	Oil	Bakeout Temp. °F	Run Hours	Trap Temp. °F	System Pressure, torr: Room Temp.	System Pressure, torr: LN_2	Deposit Weight, mg/cm^2: Total	Deposit Weight, mg/cm^2: Analytical Blank	Net Backstreaming: Deposit mg/cm^2	Net Backstreaming: Rate mg/cm^2-min.
155	4	DC 705	RT	55.5	LN_2 4)	9×10^{-9}	6×10^{-9}	17.8×10^{-4}	4.3×10^{-4}	13.5×10^{-4}	0.408×10^{-6}
159	4	DC 705	RT	51	LN_2		6×10^{-9}	21.5×10^{-4}	10.8×10^{-4}	10.7×10^{-4}	0.349×10^{-6}
163	4	DC 705	RT	95	LN_2	6×10^{-9}	4×10^{-9}	20.7×10^{-4}	4.8×10^{-4}	15.9×10^{-4}	0.293×10^{-6}
168	4	DC 705	RT	240	RT 1)	2×10^{-8}	4×10^{-9}	211.0×10^{-4}	6.5×10^{-4}	204.5×10^{-4}	1.424×10^{-6}
176	4	DC 705	RT	290	-76 2)	7×10^{-8}	2×10^{-8}	61.0×10^{-4}	6.5×10^{-4}	54.5×10^{-4}	0.314×10^{-6}
187	4	DC 705	212	200	-74	6×10^{-8}	2×10^{-8}	32.6×10^{-4}	7.4×10^{-4}	25.2×10^{-4}	0.211×10^{-6}
230	4	DC 705	212	150	-75	5×10^{-8}	1×10^{-8}	24.6×10^{-4}	6.5×10^{-4}	19.1×10^{-4}	0.199×10^{-6}
231	4	DC 705	212	79.5	-75 5)	4×10^{-8}	3×10^{-9}	11.2×10^{-4}	2.8×10^{-4}	8.4×10^{-4}	0.174×10^{-6}
236	4	DC 705	212	126	-75 5)	4×10^{-8}	4×10^{-9}	16.1×10^{-4}	3.6×10^{-4}	12.5×10^{-4}	0.166×10^{-6}
237	4	DC 705	212	67	-75 3)	2×10^{-7}	2×10^{-9}	3.48×10^{-4}	0.79×10^{-4}	2.69×10^{-4}	0.066×10^{-6}
240	4	DC 705	212	71	-75 3)	3×10^{-7}	1×10^{-9}	3.31×10^{-4}	1.26×10^{-4}	2.05×10^{-4}	0.048×10^{-6}
244	4	DC 705	212	167	-75 3)	7×10^{-7}	2×10^{-9}	3.49×10^{-4}	0.0	3.49×10^{-4}	0.035×10^{-6}

1) A light end removal run with a room temperature plate.

2) Alcohol is used as the coolant instead of liquid nitrogen.

3) Chevron Baffle used in these runs.

4) Cryo-Sorb Baffle used in runs 155 through 236.

5) New Cryo-Sorb trap was installed.

usually obtained under optimum conditions. However, the light end values of 205 x 10^{-4} mg/cm^2 (Run 168) show that the oil of Station 4 contains a large amount of light end components and this is probably responsible for the higher BC-61 values. The Cryo-Sorb baffle was examined after the run, at which time it was first appreciated that the outer surfaces of the two plates covering the center baffle must operate close to room temperature. Consequently, the oil deposit on the bottom plate could build up at this edge and see the upper edge with the result that some warm oil will see the vacuum space.

The effect of added light end components was evaluated using DC 705 containing 1% DC 704 (Table 5). It is seen (Runs 232, 239, 245) that the DC 704 had no effect on increasing the backstreaming. Gas chromatographic techniques, which evolved along with the work below, showed that the deposits contained 10.5, 10.6, and 6.8 wt. % DC 704. A value of 10% is to be expected from an equilibrium vaporization process.

III. EXPERIMENTAL WORK - TURBO-MOLECULAR PUMP

The major effort of this work period was devoted to developing gas chromatographic techniques for analyzing the Turbo-Molecular pump oil. The detailed data will be presented in the final report. A summary of comparison values is given in Table 6.

TABLE 5

Backstreaming Measurements - Effect of Added Light Ends

Run	Station	Oil	Bakeout Temp. °F	Run Hours	Trap Temp. °F	System Pressure, torr: Room Temp.	System Pressure, torr: LN_2	Deposit Weight, mg/cm^2: Total	Deposit Weight, mg/cm^2: Analytical Blank	Net Backstreaming: Deposit mg/cm^2	Net Backstreaming: Rate mg/cm^2-min.
181	2	DC 705	212	75	-76	7×10^{-9}	1×10^{-9}	1.57×10^{-4}	1.57×10^{-4}	0.0	0
203	2	DC 705	212	306	-76	3×10^{-9}	6×10^{-10}	7.60×10^{-4}	1.57×10^{-4}	6.03×10^{-4}	$.0327 \times 10^{-6}$
207	2	DC 705	212	512	-60	3×10^{-9}	7×10^{-10}	3.17×10^{-4}	0.0	3.17×10^{-4}	$.0103 \times 10^{-6}$
219	2	DC 705	212	486	-75	3×10^{-9}	7×10^{-10}	4.75×10^{-4}	2.40×10^{-4}	2.35×10^{-4}	$.0081 \times 10^{-6}$
225	2	DC 705	212	126	-75	5×10^{-9}	1×10^{-9}	2.85×10^{-4}	0.0	2.85×10^{-4}	$.038 \times 10^{-6}$
232	2	[1]	212	150	-75	5×10^{-9}	1×10^{-9}	2.40×10^{-4}	0.32×10^{-4}	2.08×10^{-4}	$.0228 \times 10^{-6}$
234	2	DC 705	212	77	RT[2]	1×10^{-8}	2×10^{-9}	51.7×10^{-4}	2.2×10^{-4}	49.5×10^{-4}	1.070×10^{-6}
239	2	DC 704	212	67	-75	6×10^{-9}	1×10^{-9}	1.89×10^{-4}	0.79×10^{-4}	1.10×10^{-4}	$.0275 \times 10^{-6}$
245	2		212	295[3]	-75	9×10^{-9}	3×10^{-9}	0.79×10^{-4}	0.0	0.79×10^{-4}	$.0045 \times 10^{-6}$

1) The side stream stripper was added to the system. A mixture of 99% DC 705 and 1% DC 704 was used.

2) A light end removal run was performed.

3) The side stream stripper ran for 123 of the 295 hours of the run.

TABLE 6

Comparison of Ultraviolet Spectrophotometric and Gas Chromatographic Analysis of Turbo-Molecular Oil - Sample Comparison

Run	UV Absorbance		Gas Chromatograph		Backstreaming Net Deposit	
	Total Weight mg/cm^2	Analytical Blank mg/cm^2	Total Weight mg/cm^2	Analytical Blank mg/cm^2	UV mg/cm^2	GC mg/cm^2
189	99.0×10^{-4}	21.9×10^{-4}	14.3×10^{-4}	0.34×10^{-4}	77.1×10^{-4}	14.0×10^{-4}
201	38.6×10^{-4}	40.4×10^{-4}	2.1×10^{-4}	1.6×10^{-4}	-1.8×10^{-4}	0.5×10^{-4}
214	12.3×10^{-4}	22.8×10^{-4}	0.27×10^{-4}	1.04×10^{-4}	-10.5×10^{-4}	-0.77×10^{-4}
215	13.3×10^{-4}	37.2×10^{-4}	0.84×10^{-4}	0.82×10^{-4}	-23.9×10^{-4}	0.02×10^{-4}

It was found possible to concentrate the sample obtained from the collection plate in a 60° cone, Teflon test tube and transfer the complete residue into the chromatograph by redissolving in benzene. The results shown in Table 6 were obtained considering only the areas for the C_{16} and C_{17} compounds. The gas chromatograph was calibrated with unused Turbo-Molecular pump oil.

The values for deposits, as determined by ultraviolet light absorbance, are completely inconsistent. The value of 99×10^{-4} mg/cm^2 (Run 189) is large enough to be a visible deposit yet no visible deposit was observed. No analytical blanks over 1×10^{-4} mg/cm^2 should be obtained since calibration studies with unused oil show solvent efficiencies (ability to wash off deposits with limited volume of isooctane) of $>$ 90% at this contamination level. The results indicate that the analytical blank was larger than the sample in three out of four runs; this is completely inconsistent and there is no obvious explanation of how this can happen. It is probable that unsaturated hydrocarbons which result from the lubrication process are responsible for the high and unrealistic ultraviolet absorbance values.

The gas chromatographic results are consistent in three out of four runs. The one inconsistent run (214) is probably due to variations in the present technique. Time did not permit development of tracer techniques and the mechanical treatment of samples can easily permit a 3X variation in the measured value. The gas chromatographic curves for runs 201, 214, and 215 show a strange peak which is at least partially responsible for the abnormal ultraviolet results. The unsaturated hydrocarbons should have very little effect on gas chromatography. There is no absolute demonstration that the contamination level is higher or lower than the measured value of $\sim 1 \times 10^{-4}$ mg/cm^2. However, the two peaks, whose detention time and relative response are the same as C_{16} and C_{17} in the original oil, tend to substantiate the validity of the values.

IV. FUTURE WORK

Future efforts will be devoted to the preparation of the final report.

Respectfully submitted,

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