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**THEORETICAL AND EXPERIMENTAL STUDY
TO DETERMINE OUTGASSING CHARACTERISTICS
OF VARIOUS MATERIALS**

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By

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March 1964

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I FOREWORD

This report was prepared by the National Research Corporation, Physics Department, Cambridge, Massachusetts, and covers the experimental studies conducted under Contract AF 40(600)-999, dated 18 June 1962. The objective of the work was to obtain outgassing characteristics of various candidate materials for use in the construction of space environmental facilities.

This research was administered by the Space Systems Office, Arnold Engineering Development Center, Arnold Air Force Station, Tennessee.

II ABSTRACT

The report presents the results of experimental studies in outgassing characteristics of materials including both total outgassing rates and analysis of constituents. Two types of experiments were performed: (1) outgassing of structural materials using metal bell jars made of the test material, and (2) outgassing of small test samples installed in a glass vacuum system. ←

Extensive outgassing data were obtained on stainless steel 304 ELC and aluminum alloy 5083-0 in the bell jar tests at several temperature increments between 100°C and LN₂ temperature (78°K). The effects on outgassing were evaluated on both materials with two surface finishes, "as received" mill finish and No. 4 polished finish.

The following materials were evaluated as typical examples of a wide range of candidate materials for use in construction of environmental facilities:

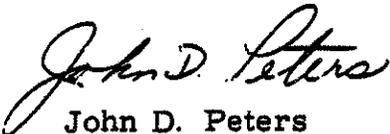
Metal:	Aluminum Alloy 5083-0
Ceramics:	Al-Si-Mag 614 Mycalex 400 Ceramic Coated Copper Wire
Elastomers:	Teflon No. 7 ← Butyl 035 ← Silastic 1602 Silastic 916 Vibrathane 5004
Paint:	Cat-a-lac Flat Black (463-1-8)

The aluminum alloy 5083-0 showed that this is an adequate material for vacuum chambers where either no bakeout or temperatures only up to +150°C to +200°C are applied. From the group of ceramics, Al-Si-Mag 614 and Mycalex 400 showed excellent properties for construction materials in vacuum chambers. Very low outgassing rates could be also measured on ceramic coated copper wire. However, the relative high amount of released hydrogen might be an undesirable property. From the five elastomers investigated, Silastic 1602 and Vibrathane 5004 cannot be recommended due to their high outgassing rates. Teflon No. 7 has low outgassing rates where it is preheated by a soft bakeout. It can be recommended under those conditions. Silastic 916 and Butyl 035 can be used as construction material in vacuum systems. A heat pretreatment is necessary to lower their outgassing rates to the required level. Representing the group

of paints, Cat-a-lac flat black (463-1-8) was studied. The materials can be recommended when an adequate curing time is kept.

PUBLICATION REVIEW

This report has been reviewed and publication is approved.



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1.0 INTRODUCTION

The scope of this program was to conduct a theoretical and experimental study to determine the outgassing characteristics of various materials to be used in construction of a large, high vacuum system. ✓

A previous program was conducted at the National Research Corporation under Contract No. AF 40(600)-923* on outgassing behavior of 304 stainless steel, 6061-T6 aluminum alloy, and 1020 mild carbon steel. This was an outgrowth of the ultimate goal of building large space simulation facilities. The limiting factor in the attainment of ultrahigh vacuum within a vacuum chamber is the gas load imposed on the pumping system from the surfaces of the various components inside the chamber and in particular the chamber wall itself. A realistic program must, therefore, attempt to determine the outgassing behavior not only of the chamber walls but also of other materials required, which of necessity have to be inside the chambers because of the nature of experiments being carried out.

The initial program completed in the previous contract concerned itself only with the materials for the chamber walls. The experiments were performed to determine the outgassing characteristics of the alloys mentioned above, at room temperatures and elevated bakeout temperatures, as well as the effect of the bakeout cycles on the subsequent room temperature behavior. Further experiments were carried out to determine the effects of the venting gas on a well outgassed system.

The experimental arrangement in the above program was novel, in that the sample material was a part of the ultrahigh vacuum enclosure. A bell jar constructed out of the test alloy was attached to an ultrahigh vacuum pumping system. A nude ionization gauge located inside was used to measure the pressure within the chamber. The pumping was done through an aperture on a plate attached to the bottom of the bell jar. The outgassing rates were calculated from the pressure rise data when the aperture was closed isolating the test chamber from the rest of the system. The pumping was thus interrupted for a short time periodically during the entire pumpdown cycle to obtain the outgassing vs. time curve.

Since the conclusion of the first outgassing program, there have been certain changes in the design concepts for the very large space chamber. First, for reasons of compatibility with nuclear fission reactions within the space chamber an aluminum alloy has been given preference over the stainless steel. Secondly, from consideration of the structural strength of aluminum, it has been decided that only a comparatively low

* AEDC-TDR-62-19

temperature bakeout of the shell is permissible if a bakeout is permitted at all. It is known, and was convincingly demonstrated during the first outgassing program, that during a pumpdown, if the temperature of the test chamber is lowered (from bakeout temperature to a lower temperature for instance), the pressure as well as the outgassing rate drop drastically. Thus, if a chamber is pumped down to a fairly low pressure at room temperature and then cooled below ambient, decrease in both pressure and outgassing rate should result.

The direction for the present outgassing study programs evolved from the foregoing concepts and reasoning. It consists of two parts: 1) vacuum enclosure alloys to be tested by the static pressure rise technique using bell jars constructed of the alloys and 2) outgassing studies of materials such as plastics, elastomers, insulating materials, etc. to be tested as small samples in a bakeable glass apparatus where the pressure drop across a known conductance is used as the dynamic measure of outgassing. The experimental procedure for the metal chambers were to include room temperature pumpdown, followed by a temperature cycling which would take the chamber down to liquid nitrogen temperature in a number of steps, followed by short bakeout at 100°C (boiling water temperature), and down to room temperature for the conclusion of the experiment. Chambers were also to be studied in their as-received surface condition and another one with a No. 4 surface finish. Provisions were to be made for determining the chemical composition of the evolved gases wherever possible.

2.0 EXPERIMENTAL PROGRAM

The experimental program will be discussed in two parts according to the experimental systems used.

In the first section, studies conducted in the 12 inch metal bell jars will be discussed. In the second section, measurement of outgassing from small samples in the glass system will be reported.

3.0 OUTGASSING STUDIES IN METAL BELL JAR SYSTEMS

3.1 DESCRIPTION OF BELL JAR TYPE OUTGASSING APPARATUS

3.1.1 Test Chamber and Vacuum System

The bell jar apparatus used for the outgassing measurements can be divided into three major parts; 1) the 14-inch standard vacuum pumping system, 2) the exchangeable test bell jars, (Figs. 1 and 3) the gas analysis system, (Fig. 2), including mass spectrometer and pump unit.

The 14-inch standard vacuum chamber is an L-shaped stainless steel system (Fig. 3), with one double O-ring seal which is water cooled. It is flanged on one end to a 6-inch diffusion pump, which is backed by a 2-inch diffusion pump and a mechanical pump. The foreline can be connected to a leak detector. Between the 6-inch diffusion pump and experimental chamber are four baffles to prevent diffusion pump oil from backstreaming. One water-cooled baffle is arranged directly on top of the pump followed by a liquid nitrogen trap and an optical baffle. The fourth baffle is again a liquid nitrogen trap. The other end of the L-shaped part is flanged with a water-cooled double O-ring seal to an 18-inch/14-inch T-piece. The top end of the T-section (Fig. 1) is covered by a lid carrying the plunger mechanism. The bottom end is facing the exchangeable test chambers. All flange connections are double O-ring seals with water cooling. All flanges are kept together by C-clamps. The T-piece is additionally provided with an ion gauge of the Bayard-Alpert type and some electrical feedthroughs.

The test bell jars fabricated from the material which was to be investigated consisted of the actual dome, one-foot diameter, one-foot length and one-quarter-inch wall thickness for the bell jars and one-half-inch wall thickness for the head plate, and a transition section. Both were welded together on a so-called middle flange which is a solid disc out of the same material as the dome. The outgassing area, therefore, was 4370 cm^2 . The free

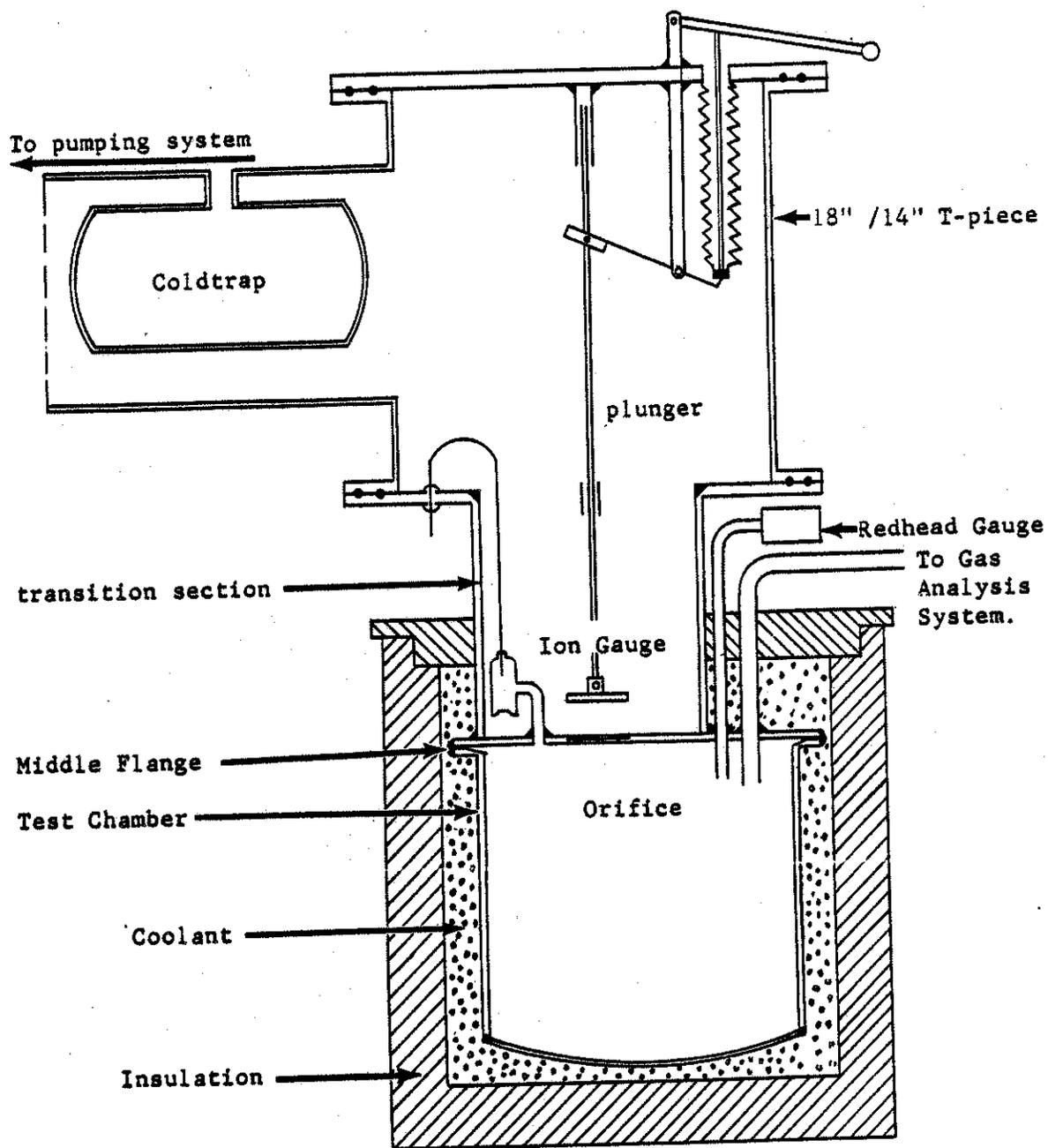


FIGURE 1

EXPERIMENTAL ARRANGEMENT FOR OUTGASSING MEASUREMENTS
OF STAINLESS STEEL 304 ELC AND ALUMINUM ALLOY 5083-0

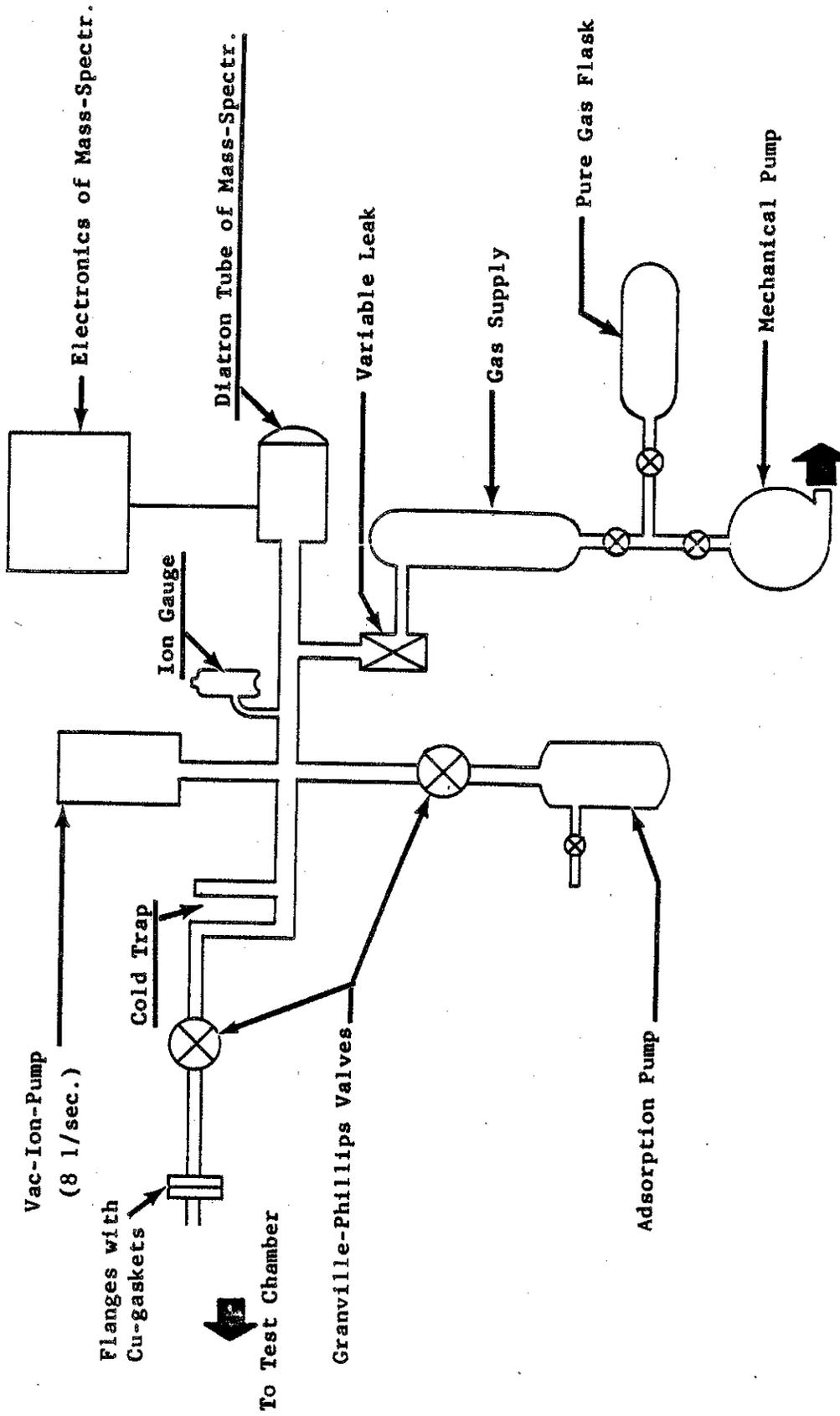


FIGURE 2 - Schematic Diagram of the Gas Analysis System

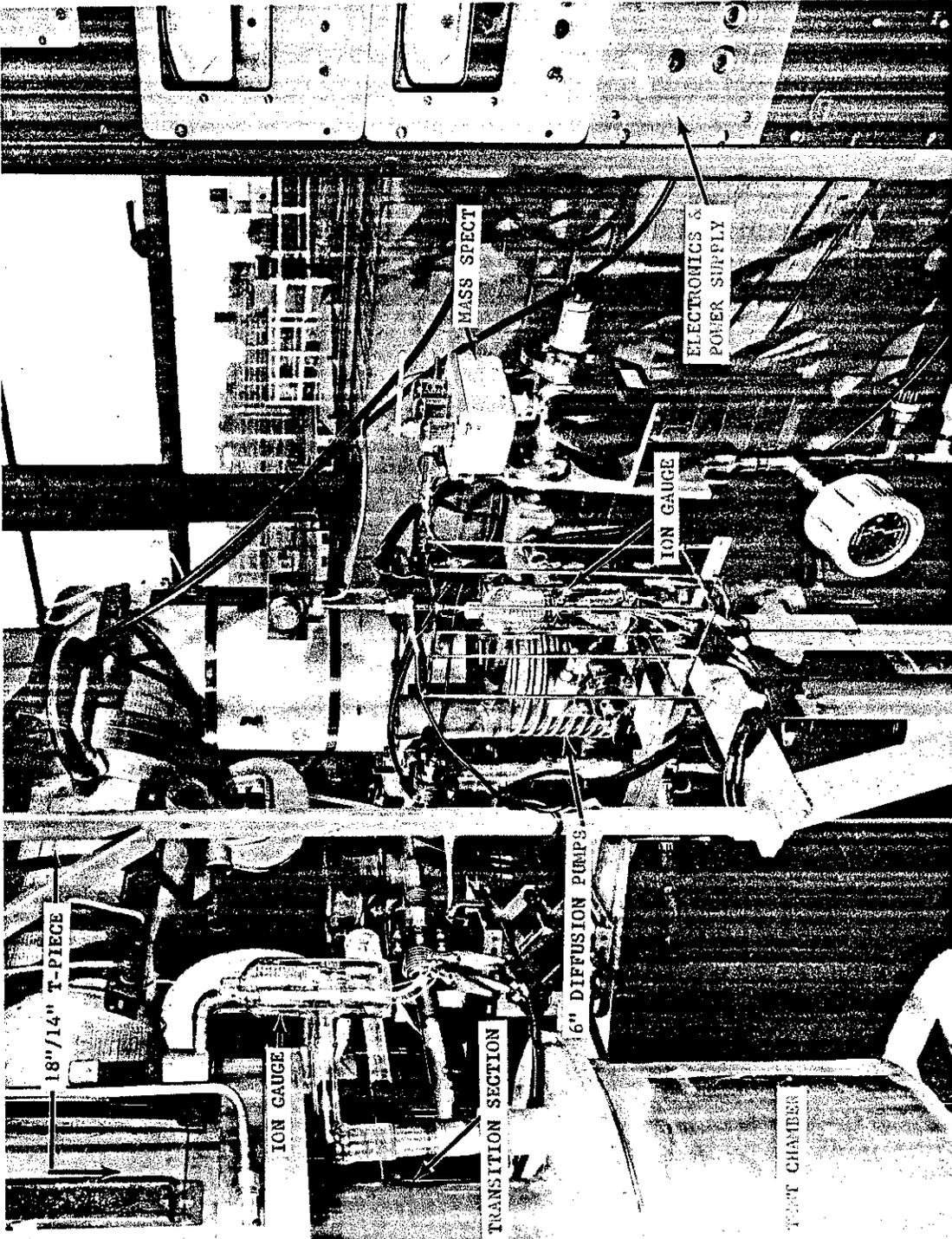


FIGURE 3 - PHOTOGRAPH OF ENTIRE METAL BELL JAR SYSTEM

end of the transition section was welded on an 18-inch flange which fitted on the 18-inch diameter T-piece of the standard system. The transition section was necessary in order to have only metal areas outgassing. The middle flange had an opening of two-inches diameter which determined the pumping speed to be about 223 l/sec. assuming an average mass of 30. This opening could be closed by a mechanical plunger mechanism operating through a bellows in order to measure pressure rise readings. The middle flange also carried an ion gauge to read the pressure in the chamber and to follow the pressure rise during the shut off period. Two further openings in the middle flange were provided to connect the test chamber with a Redhead gauge and with the gas analysis system.

The transition section consisted of a pipe welded on one end to the middle flange and with the other end to the flange facing the 18"/14" T-piece. The diameter of the pipe was kept smaller in order to cool the middle flange from the top as much as possible where the test chamber was immersed into the coolant which will be described later in the experimental procedure.

3.1.2 Gas Analysis System

This system (Fig. 2) is connected by a stainless steel flange, with a copper gasket over a Granville-Philips valve to the test chamber. It forms a high vacuum system for itself. A charcoal pump was used as forepump. After sufficient low pressure (10^{-4} torr) was reached, the adsorption pump could be valved off by a Granville-Philips valve. An 8 liter/sec. vacuum pump pumped the system down to the required pressure. This pump was arranged parallel with a Bayard-Alpert ion gauge leak valve and a mass spectrometer, type CEC 21-612. The stainless steel diatron tube was flanged directly to the system by using a Teflon gasket. The leak valve allowed gas inlet for calibrating the mass spectrometer.

3.1.3 Temperature Control

Outgassing measurements were made with bell jar temperatures controlled sequentially at nominal temperatures of 25°C, 0°C, -30°C -78°C -196°C and +100°C. A technique of using fixed temperature systems was adopted after an initial attempt to cool by using chilled gases. The room temperature point, of course, needed no control. Ice-water mixture was used for 0°C, dry ice powder

for -78°C and liquid nitrogen for -196°C . For control at about -30°C , an ethylene glycol-water immersion system was used. Boiling water maintained by use of immersion heaters provided the 100°C control. The cooling or heating medium was contained in a copper tank fitted into a styrofoam jacket. Uniform temperature was obtained at each of the temperature points on the entire surface of the chamber except for the orifice closure disc. Calculations of hypothetical outgassing rates as a function of temperatures (Table XVII) indicate that the outgassing from the warmer plunger would not introduce any measurable errors at any of the temperatures where valid measurements of outgassing could be made.

3.2 DESCRIPTION OF EXPERIMENTAL PROCEDURE

3.2.1. Outgassing Experiments

Altogether four metal chambers were tested, two constructed of 304 ELC stainless steel and two of 5083-0 aluminum alloy. One each of stainless steel and the aluminum were in as-received condition and the others were given a No. 4 finish, which is the surface finish obtained by grinding the surface with 150 grit abrasive paper. Grinding was performed on the bell jars after fabrication and welding. Just prior to the final weld between the bell jar and transition section, all interior surfaces were wiped with alcohol to remove fingermarks and other dirt picked up during fabrication. After the assembled test chamber was connected to the pumping system and before the actual testing began, the sample chamber was pumped down to a low enough pressure (10^{-6} torr range) to leak check it with a helium probe and a mass spectrometer leak detector. When it was definitely established to be leak free, down to this pressure of 10^{-6} torr, the chamber was vented with room air and allowed to remain at atmospheric pressure for at least 48 hours. After the leak checking and the conditioning with room air, the actual outgassing experiment began. The pumps were started, and as soon as the pressure was low enough (about 10^{-5} torr) the ionization gauge was turned on. Within six or seven hours the pressure-time curve became quite flat. The low temperature excursion began at this time, which took the sample chamber through the temperature points of 0°C , -30°C , -78°C and -196°C and back to room temperature. In some runs, temperature was carried to $+100^{\circ}\text{C}$ and then back to room temperature. Throughout the experiment, measurements were made to obtain the outgassing rates, and mass spectrometer data were recorded to determine the partial pressures of various components. The outgassing measurements were achieved by closing the aperture between the test chamber and the pumping system and noting the pressure rise with time. The initial slope of the pressure-rise time curve is the outgassing

rate in terms of torr liters/cm² sec. The numerical value of the outgassing was calculated from the pressure rise data and the known geometry of the test chamber. The mass spectrometer data were obtained by first making a scan of the background and then making a second scan after allowing a gas sample from the test chamber into the gas analysis system through the high vacuum valve. When the pressure in the test chamber was quite high compared to the gas analysis system pressure, the flow was achieved by simply opening the valve. This was not possible when the test chamber pressure was also low. The difficulty was overcome by isolating the test chamber from the pumps and allowing the pressure to build up sufficiently and then opening the high vacuum valve between the test chamber and the subsystem.

3.2.2 Mass Spectrometer

The CEC mass spectrometer 21-612 system was pumped down with a charcoal pump through a high vacuum valve. As soon as the pressure reached a low value, the vac-ion pump was turned on. When the vac-ion pump took over, the charcoal pump was valved off. The gas analysis system including the mass spectrometer tube was thoroughly baked out and a pressure of around 5×10^{-9} torr was reached as indicated by the UHV gauge located in the gas analysis system. On turning on the filament in the diatron tube, the pressure would rise with a minimum obtainable of 1×10^{-8} torr. This was deemed satisfactory. A calibration of the mass spectrometer was carried out, using pure sample gases, N₂, CO, H₂ and CO₂, introduced through the Granville-Philips microleak valve. The calibration partial pressure was not allowed to exceed 10^{-7} torr. The following parameters were determined, 1) peak position, 2) various mass fractions, and 3) peak height vs. partial pressure.

3.3 DISCUSSION OF RESULTS

The results of the experiments are presented in both tabulated and graphical form in Tables I through IV, Tables I Unabridged through IVA (see Appendix), and Figs. 4 through 11; further details are presented below.

3.3.1 Stainless Steel 304 ELC Bell Jar Tests

Two experiments were conducted using stainless steel ELC vessels. Both were fabricated from the same sheet stock and differed only in the surface finish. One vessel was used in the "as received" condition and the other was given a No. 4 finish.

The outgassing procedure planned for the "as received" finish on 304 ELC included a temperature excursion from room temperature to 0°C, to -30°C, to -78°C and -196°C. The +100°C point was deliberately omitted from this run in order not to

TABLE I

Pressure Rise on Closing Aperture in 304 ELC
Stainless Steel Test Chamber with As-Received Surface

Elapsed Time (hrs)	Pressure Rise dp/dt (torr/sec) x 10 ⁷	Outgassing Rate (Tl/sec cm ²) x 10 ⁹	Temperature
0.75	11.0	5.86	
2.5	4.8	2.55	
3.5	3.4	1.81	Room
4.5	3.21	1.73	Temp.
5.5	2.0	1.06	
-	Liquid nitrogen added to the cold trap		
7.0	1.1	0.586	
8.0	0.94	0.50	
9.5	0.22	0.117	
10.0	0.22	0.117	
11.25	0.14	0.0745	0°C
13.5	0.13	0.0692	
15.2	0.081	0.043	
16.5	0.076	0.041	-30°C
20.0	0.046	0.0008	
23.5			
24.5	0.046	0.024	-78°C
25.5	0.043	0.023	

After the dry ice run, liquid nitrogen was started. The pressure rose rather than dropped. Removed liquid nitrogen and located a large leak at the glass-metal seal of the Redhead gauge. The experiment was, therefore, abandoned at this point.

TABLE II

Pressure Rise on Closing Aperture in 304 ELC
Stainless Steel Test Chamber with No. 4 Surface Finish

Elapsed Time (hrs)	Pressure Rise $\frac{dp}{dt}$ (torr/sec) $\times 10^7$	Outgassing Rate (Tl/sec cm^2) $\times 10^9$	Temperature
0.5	7.2	3.83	
1.75	2.6	1.38	Room
2.75	1.4	0.74	Temp.
4.5	1.0	0.554	
6.0	0.28	0.149	
7.0	0.26	0.138	0°C
10.0	0.12	0.064	
13.0	0.076	0.040	-27°C
14.0	0.060	0.032	
15.25	0.054	0.0287	
16.25	0.054	0.0287	-78°C
18.25	0.054	0.0287	
20.0	0.014	0.00745	
21.0	0.014	0.00745	-196°C
31.0	Liquid nitrogen cooling maintained up to this time		
32.8	0.36	0.192	Room
35.3	0.306	0.163	Temp.
37.0	22.6	12.8	
38.0	8.2	4.36	
39.2	6.2	3.30	100°C
40.2	2.2	1.17	
41.0	Hot water removed		
43.5	0.095	0.0505	Room
44.7	0.031	0.0165	Temp.
52.0	0.032	0.017	-196°C
52.75	0.017	0.0089	

TABLE III

Pressure Rise on Closing Aperture in
5083-0 Al Alloy Test Chamber with As-Received Surface

Elapsed Time (hrs)	Pressure Rise dp/dt (torr/sec) x 10 ⁷	Outgassing Rate (Tl/sec cm ²) x 10 ⁹	Temperature
0.8	100.0	53.2	
2.0	30.0	15.9	
3.8	14.2	7.55	
5.2	7.0	3.71	Room Temp.
5.75	6.8	3.61	
7.3	4.5	2.39	
10.3	4.1	2.18	
11.3	0.25	0.133	
12.5	0.24	0.128	0°C
13.7	0.18	0.0957	
14.5	0.18	0.0957	
15.7			
16.5	---	---	-30°C
17.3			
18.6	---	---	-78°C
22.3			
25.0	---	---	-196°C
26.0	3.7	1.97	Room Temp.
27.3	2.3	1.22	
30.0	2.0	1.06	

TABLE IV

Pressure Rise on Closing Aperture in
5083-0 Al Alloy Test Chamber with No. 4 Finish

Elapsed Time (hrs)	Pressure Rise dp/dt (torr/sec) x 10 ⁷	Outgassing Rate (Tl/sec cm ²) x 10 ⁹	Temperature
0	284.0	151.0	
1	224.0	119.0	
2	164.0	87.2	Room Temp.
3.25	Found and sealed a leak		
3.26	2.2	1.17	
5.0	0.98	0.521	
6.0	0.54	0.287	
7.0	0.189	0.10	
8.0	0.189	0.10	0°C
9.0	0.092	0.0489	
12.0	0.168	0.0895	
13.25	0.084	0.0446	-30°C
14.5	0.072	0.0382	
16.0	0.062	0.0329	
17.5	0.046	0.0244	-78°C
18.5	0.046	0.0244	
19.5	0.0244	0.0129	-196°C
20.5	0.0244	0.0129	
24.0	0.18	0.0956	Room Temp.
25.0	51.2	27.4	
26.0	15.4	8.2	100°C
27.0	6.56	3.48	
28.0	0.066	0.035	Room Temp.
29.0	0.064	0.034	Temp.

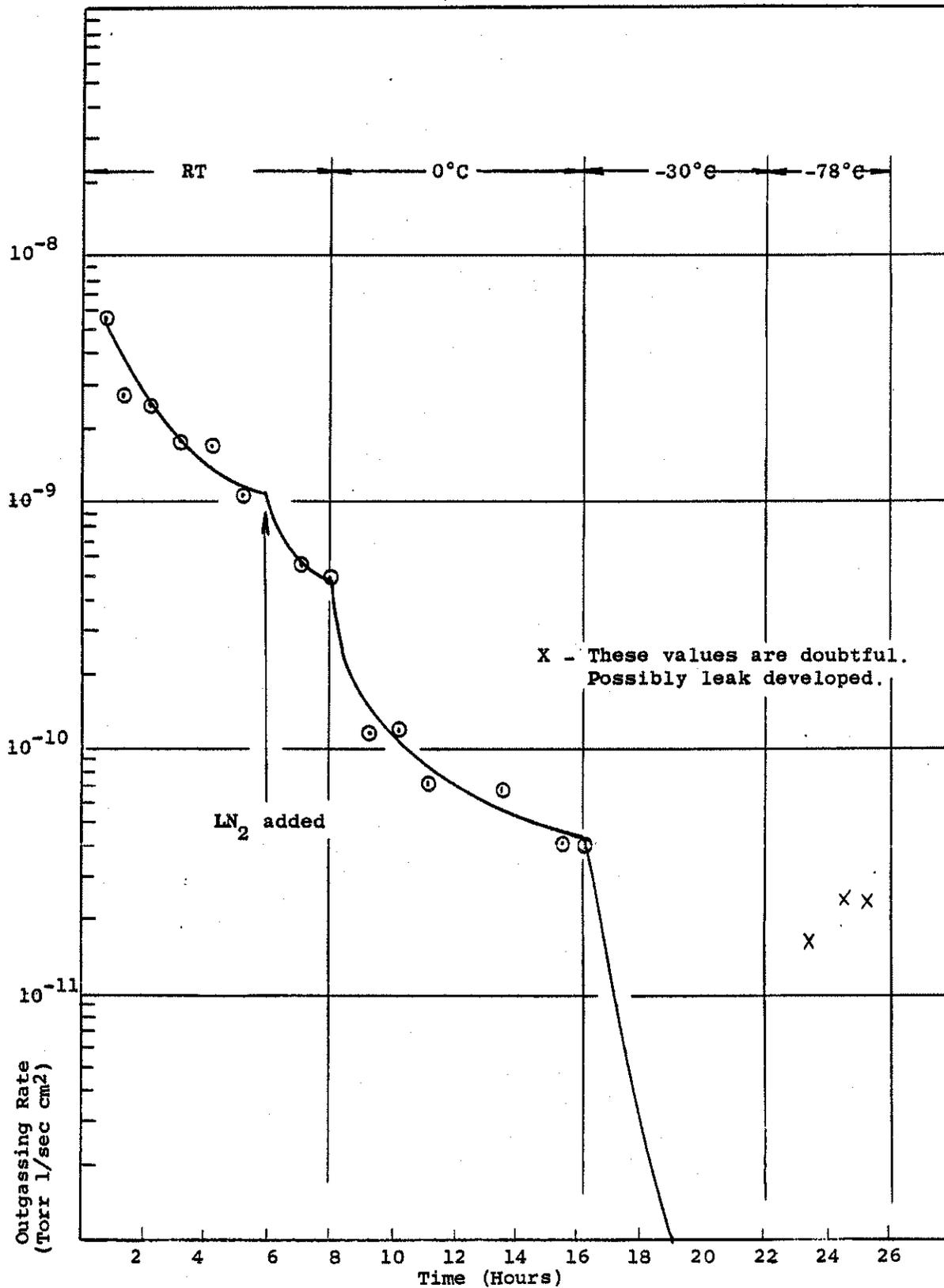


FIGURE 4 - OUTGASSING RATES, Stainless Steel ELC, Surface "As Received"

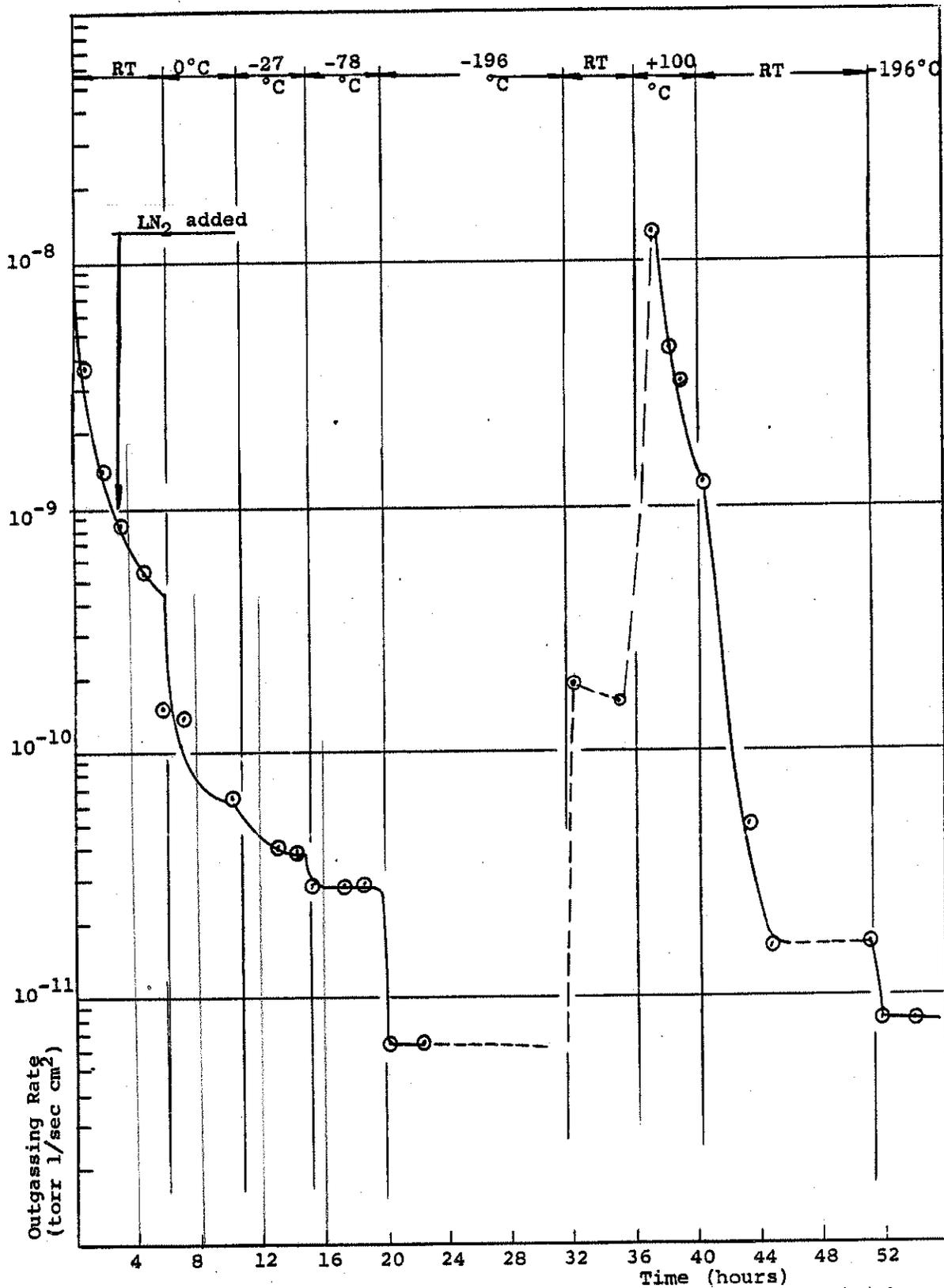


FIGURE 6-OUTGASSING RATES, Stainless Steel ELC, Surface No. 4 Finish

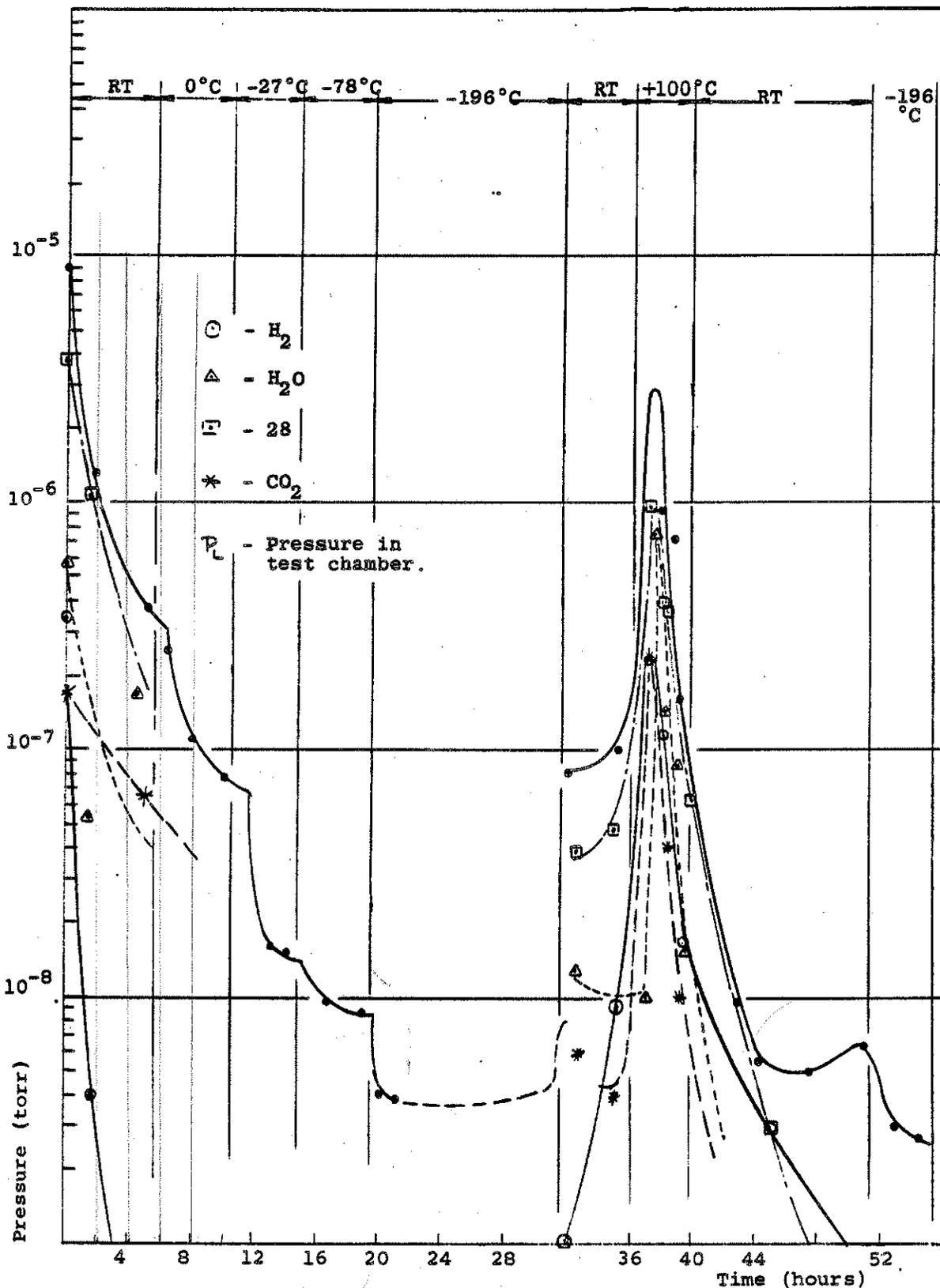


FIGURE 7 - MASS SPECTROMETER DATA, Stainless Steel ELC, Surface No. 4 finish

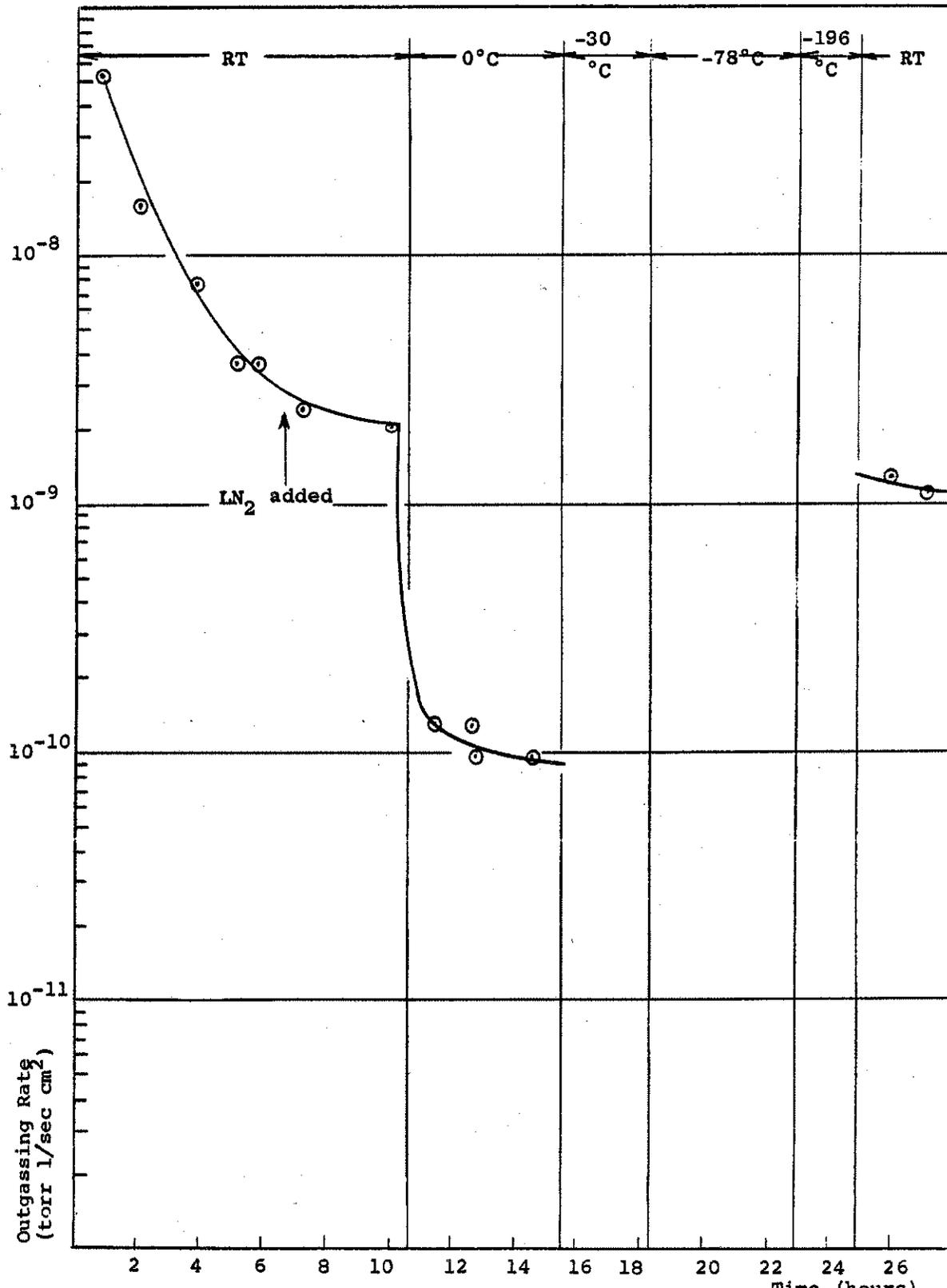


FIGURE 8 - OUTGASSING RATES, Aluminum 5083-0, Surface "as received"

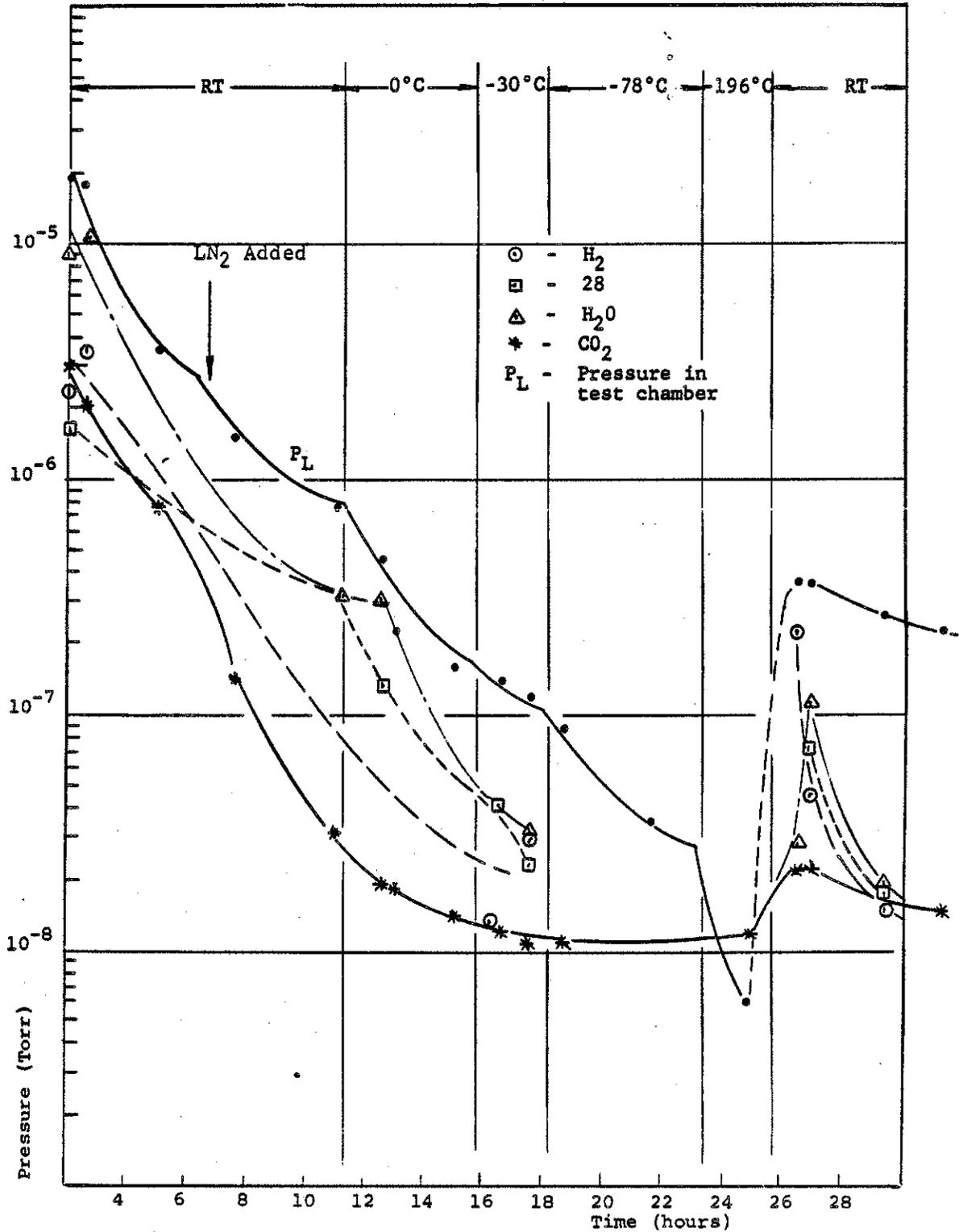


FIGURE 9 - MASS SPECTROMETER DATA Aluminum 5083-0 Surface "as received".

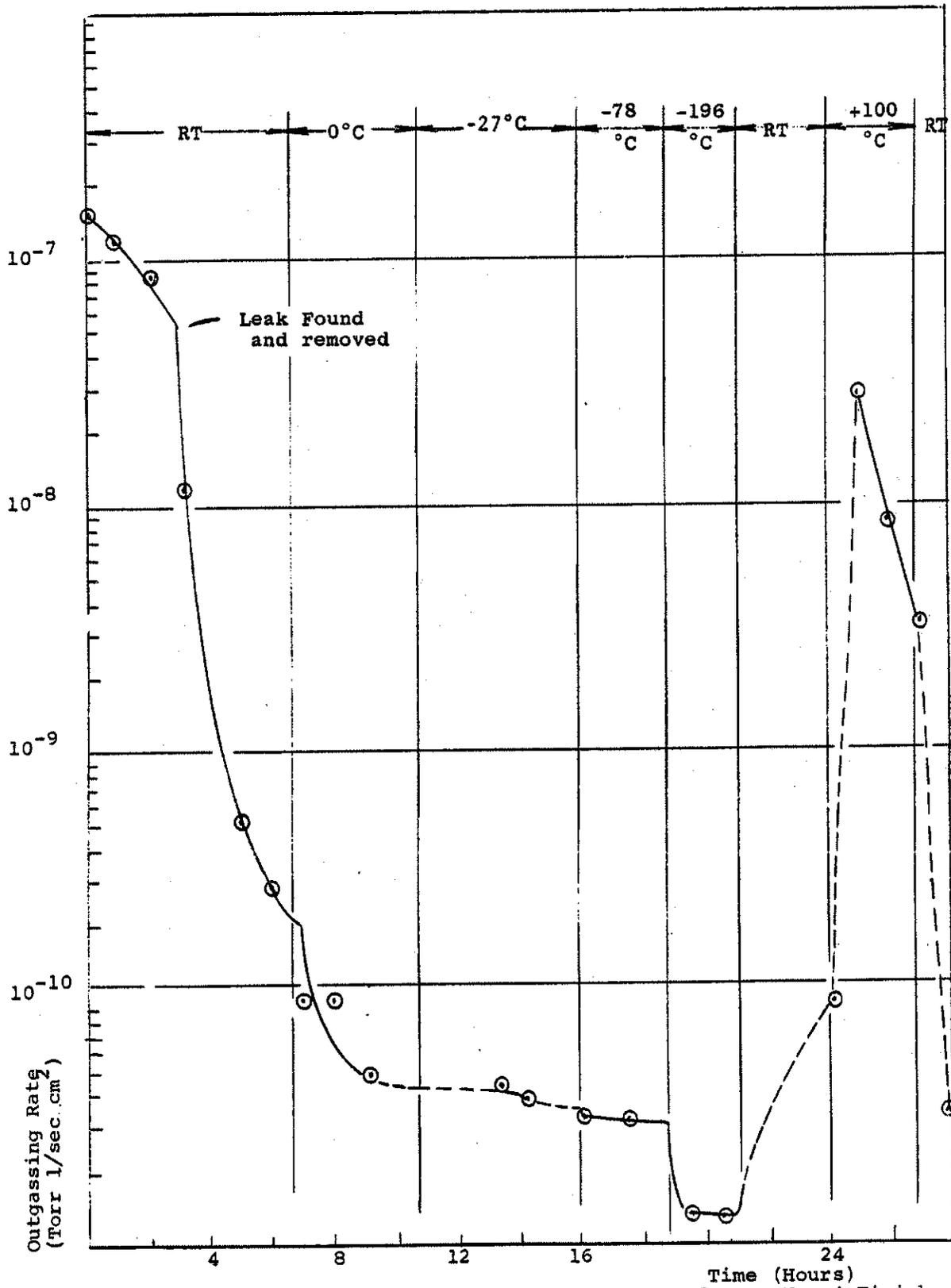


Figure 10 - OUTGASSING RATES, Aluminum 5083-0, Surface - No. 4 Finish

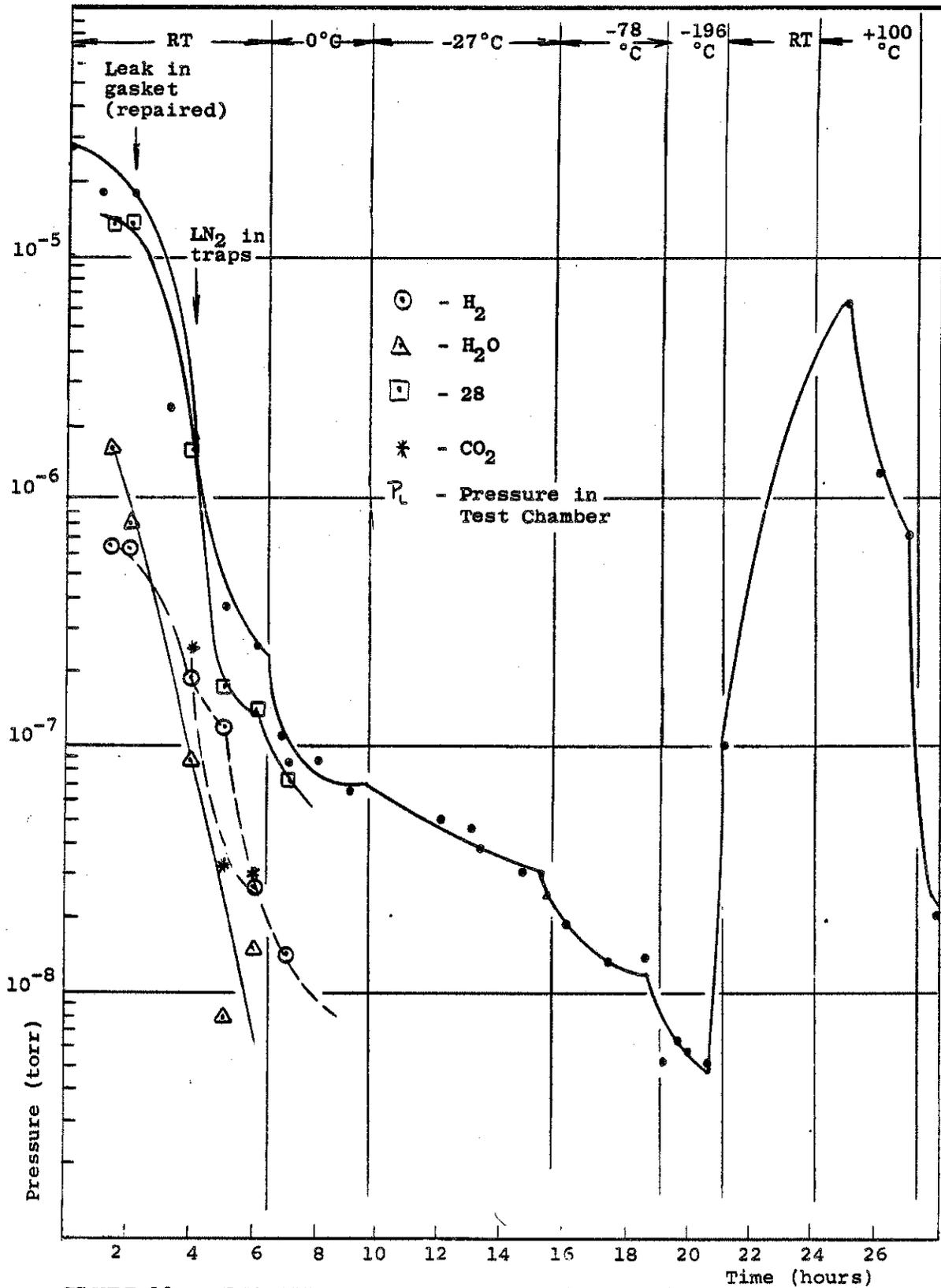


FIGURE 11 - MASS SPECTROMETER DATA, Aluminum 5083-0 Surface-Finish No.4

disturb the internal gas content of the wall since it was anticipated that the interior surface of this bell jar might be subsequently refinished and utilized to obtain additional data on some desired surface finish. Fig. 4 and Table I present the outgassing rate data and Fig. 5 and Table IA (see Appendix) present the total pressure and composition as a function of time during the run.

This run was not carried through the entire planned schedule since a leak developed at the glass-to-metal seal to the Redhead gauge, when the vessel was immersed in liquid nitrogen. In subsequent runs, the tubulation of the Redhead gauge was modified to bring the glass-to-metal transition above the low temperature level and this difficulty did not recur.

The experiment using a No. 4 finish stainless steel ELC bell jar is reported in Fig. 6 and Table II (outgassing data) and Fig. 7 and Table IIA (see Appendix), total pressure and gas composition data. This run was carried out successfully throughout the entire temperature cycle as follows:

Room temperature, 0°C, -27°C, -78°C, -196°C, room temperature, +100°C and finally room temperature again.

Comparing the two runs it may be observed that both the rate of pumpdown (Figs. 6 and 7) and the decrease in outgassing rate (Figs. 4 and 5) is more rapid for the No. 4 finish bell jar, a value of 5×10^{-10} torr l/cm² sec. being reached, in eight hours for the "as received" vessel and in slightly over one-half that time for the No. 4 finish vessel. At this point, a lowering of temperature to 0°C produced a comparable decrease in outgassing rate 4.3×10^{-11} torr l/cm² sec. for the "as received" finish and 6.4×10^{-11} torr l/cm² sec. for the No. 4 finish vessel. Reduction in temperature to -78°C produced a further decrease to 8×10^{-13} torr l/cm² sec. for the "as received" vessel. For the No. 4 finish vessel a value of 7.5×10^{-12} torr l/cm² sec. was obtained.

On return of the No. 4 finish stainless steel vessel to room temperature a value of 1.6×10^{-10} torr l/cm² sec. was obtained, somewhat lower than the room temperature value of 5.5×10^{-10} measured before the low temperature excursion and indicating that gas was being removed throughout the various low temperature runs.

The bell jar was then heated to +100°C to obtain an additional temperature data point. The initial value measured was 1.28×10^{-8} which fell rather rapidly until after three hours a value of 1.2×10^{-9} torr l/cm² sec. was recorded.

The bell jar was then cooled to room temperature and a value of 1.7×10^{-11} torr l/cm² sec. was measured.

These results indicate a definite effect of lower temperature on outgassing rate. However, the effect of very low temperatures is not as great as predicted. This is believed to be in part the result of experimental limitations which make it difficult to measure very low outgassing rates. The two limitations are very low pressures in the system and the outgassing of absorbed gases from gauges and tubulations which remain at room temperature. A rate of rise of 1×10^{-9} torr/sec. is equivalent to an outgassing rate of 5×10^{-12} torr/cm² sec. Since pressures no lower than the 10^{-9} torr range were obtained in these runs, this no doubt limits the ability to detect low rates of outgassing.

Mass spectrometer measurements made during the runs are not entirely comparable. Hydrogen was the predominating gas evolved from the "as received" surface of stainless steel; whereas, mass 28 (CO and/or N₂) was the most abundant gas from the No. 4 finish surface. Water vapor and CO₂ were observed in both runs as minor gases.

Mass spectrometer data were not obtainable at the lower pressures due to sensitivity limitations. Also during part of the run on No. 4 finish operating difficulties with the instrument prevented taking reading during part of the run.

3.3.2 Aluminum 5083-0 Bell Jar Tests

Outgassing data for the aluminum alloy bell jars are shown in Figs. 8 and 9 and Tables III and III Unabridged (see Appendix). Both aluminum vessels were fabricated from the same sheet stock and differed only in surface finish.

Referring to Figs. 8 and 9, it is observed that outgassing rate decreases more rapidly and reaches a lower value for the No. 4 finish surface than for the "as received" surface. In six hours a value of 2.9×10^{-10} torr/cm² sec. for the No. 4 finish was obtained as contrasted with 2.2×10^{-9} torr/cm² sec. after ten hours for the "as received" surface. Cooling to 0°C decreased outgassing rates to 4.9×10^{-11} and 9.6×10^{-11} for the No. 4 and "as received" surfaces respectively.

Below 0°C, useful data is available only for the No. 4 finish bell jar. For the "as received" bell jar, the expected pressure rise did not occur. When the valve was closed, a small pressure increase was noted within the first five seconds, followed by a decrease and leveling off. The experimental set-up for this run differed from the others only in that the Redhead gauge was not attached to the system because it was in process of repair, due to earlier seal difficulties. Under these circumstances it is difficult to assign any real values; based on ability to detect pressure increases, one would estimate the outgassing rate to be less than about 1×10^{-11} torr/cm² sec. This situation persisted at the -30°C, -78°C and -196°C levels.

Upon returning to room temperature, an outgassing rate of 1×10^{-9} torr $1/\text{cm}^2$ sec. was measured, which is in reasonable agreement with the room temperature value prior to start of the low temperature excursions. The No. 4 finish aluminum bell jar was taken through the entire cooling cycle with pressure rise data collected at every temperature level. Small decreases were noted with successive drops in temperature and even at -196°C an outgassing rate value of 1.3×10^{-11} torr $1/\text{cm}^2$ sec. was measured. As noted earlier in the other runs, this value is believed to represent outgassing from gauge tubulation and not from the cold vessel wall.

Of significance is the outgassing rate change occurring as result of an excursion to 100°C and then back to room temperature. A sharp increase as expected was experienced at 100°C , a value of 2.7×10^{-8} torr $1/\text{cm}^2$ sec. being observed. This dropped markedly to 3.5×10^{-9} during the three hours the system was held at 100°C . Then after return to room temperature a value of 3.5×10^{-11} torr $1/\text{cm}^2$ sec. was obtained which is equivalent to rate obtained at -30°C prior to the short "bakeout" at 100°C .

Partial pressure data for the aluminum bell jar runs are reported in Figs. 10 and 11 and Tables IV and IVA (see Appendix). Differences in behavior were noted between the two surface finishes. Water vapor was the predominant gas for the "as received" finish throughout the room temperature run; mass 28 (CO and/or N_2) and hydrogen were next in abundance. For the No. 4 finish bell jar water vapor, while present, was not the major peak. Mass 28 (CO or N_2) accounted for nearly all of the total pressure. During this latter run, the mass spectrometer broke down in the middle of the 0°C run and was inoperable throughout the rest of the run.

3.4 CONCLUSIONS

The following general conclusions can be drawn from these data:

- (1) From an outgassing point of view, there is no major difference between the behavior of stainless steel and aluminum.
- (2) Temperature decrease results in lowering of outgassing rate. The decrease is consistent with the actual predictions for the change from room temperature to 0°C . Experimental limitations prevent valid measurements of outgassing rates below about -30°C .
- (3) The effect of surface finish for both stainless steel and aluminum is measurable, the smoother surface giving the lower outgassing rate. The effect is more marked in the case of aluminum; the difference in gas

evolved from the "as received" versus No. 4 finish aluminum bell jars suggests that the no doubt heavier oxide coating on the mill finish metal may hold substantial amounts of water.

The advantage of polished surfaces vs. mill finish must be analyzed from an economic viewpoint, and in terms of operating cycle time to decide whether the reduced pumpdown cycles possible with polished surfaces offset the initial costs of providing the smoother finish.

- (4) Although all the outgassing experiments were conducted in vacuum tight systems, it was considerably more difficult to obtain sound leak free weldments in the case of the aluminum alloy than of stainless steel. However, whenever leaks did occur it was possible to repair defective weldments by rewelding those spots.

4.0 OUTGASSING STUDIES OF VARIOUS MATERIALS IN THE GLASS SYSTEM

4.1 INTRODUCTION

The second part of this program deals with the investigation of various materials which might be used as construction elements for space chambers or vehicles including their equipment.

Before the experiments were started, about fifty samples of different materials were screened and grouped together in five major categories. Out of each category one sample was selected as representative. The selection was made according to the properties and usability of the material for space simulation. The five major groups are:

- a) Metals
- b) Ceramics
- c) Elastomers
- d) Plastics
- e) Paints

All the materials belonging to this group are shown in Table V.

A total of ten samples had been studied during the contract period. For the metals, the aluminum alloy Al-5083-0 was selected in order to have a direct comparison with the bell jar experiments. The material was cut from the same plate of which the metal bell jars were made. For the ceramic group ground Al-Si-Mag 614 plates, Mycalex 400 and ceramic coated Copper wire were taken. Teflon No. 7, Silastic 1602, Silastic 916, Butyl 035 and

TABLE V

Samples of Materials Obtained for Outgassing Studies

Material	Manufacturer	Application or Chemical Identification
<u>I. Elastomers</u>		
1. Butyl	Greene Rubber Co. Cambridge	-70°F, airplane hydraulic system seals. Automotive inner tubes
2. Silicone Rubber	"	
3. 470 Gum Rubber	"	
4. Buna N	"	
5. Viton	"	
6. 4240 Neoprene	"	
7. Buna S	"	
8. Viton B	United States Rubber Company	Copolymer of vinylidene ride and hexafluoro propylene
9. Butyl HT 10-36	Wayne, New Jersey	Polyisobutylene
10. Butyl 035		Chlorobutyl
11. Vibrathane		Polyurethane
12. ERP 1026		Ethylene Propylene
13. Neoprene WRT		Polychloroprene
14. COHR-lastic 500		Silicone (All insulators)
15. Silastic No. 50	Dow Corning Corp.	Chemical Resistant, for
16. 80	Midland, Mich.	extruding pieces
17. 651		gaskets, electronic
18. 675		fittings, bellows
19. 916		-130°F +500°F, high
20. LS-53		tensile strength
21. Silastic 1601	Dow Corning Co.	Wire insulations. Cable in-
22. 1602	Midland, Mich.	ulations. Navy electrical equipments. Ignition wire insulation for combustion engines.
<u>II. Plastics</u>		
23. Teflon	Greene Rubber Co.	Insulator, gaskets, tubing
24. Marlex 2380	U. S. Rubber Co.	Polyethylene
25. Marvinol VR-10		Polyvinyl chloride (high molecular weight)
26. Marvinol VR-24	U.S. Rubber Co.	Polyvinyl chloride (low mole- cular weight)
27. Profax 6411		Polypropylene
28. Kralastic SS		ABS (acrylonitrile-butadiene- styrene)
29. Styrene 666		Polystyrene (all thermoplastics)

TABLE V - continued

Material	Manufacturer	Application or Chemical Identification
<u>II. Plastics - cont.</u>		
30. Polypropylene 702	Dow Chemical Co. Midland, Mich.	moldable, automotive parts, tools, industrial appliance Injection molding, general purpose polystyrene
31. Styron 666		
32. Polyethylene 710M		
33. Zerlon 150	Dow Chemical Co.	Styrene-methyl methacrylate copolymer. Heat resistant outdoor weatherability, clear material.
34. Tyril 767		copolymer of styrene and acrylonitrile. Terminal boxes battery cases, communication equipment components.
<u>III. Ceramics</u>		
35. Al-Si-Mg 614	American Lava Corp. Chattanooga, Tenn.	Injections molding, feed- throughs, dyes.
36. Mycalex 400	Mycalex Corp. of America, Clifton, N.J.	
37. Ceramic Body 209 (95% Alumina)	Centralab Milwaukee, Wisc.	Insulating feedthroughs, ter- minal supports, high-frequency transmitter panels. Application in electronic parts sockets, feedthroughs, supports.
<u>IV. Metals</u>		
38. Al-Alloy 5083-0		
39. SS 304 ELC		
40. OHFC Copper		
<u>V. Paints</u>		
41. Tile - Cote	Wilbur & Williams Co., Inc. Norwood, Mass.	Activated epoxy coating No. 1202 black
42. Cat-a-lac	Finch Paint and Chemical Co. Torrance, Calif.	Paint on epoxy base.

Vibrathane 5004 were investigated representing the elastomers. Cat-a-lac 463-1-8 flat black, a coating on epoxy base was taken to study the outgassing characteristics of paints.

4.2 DESCRIPTION OF THE GLASS SYSTEM

The glass outgassing system utilizes the principle of measuring gas flow rate by accurate measurement of pressure drop across an orifice of known conductance.

A schematic drawing of the apparatus is shown in Fig. 12. The apparatus comprises a sample container which was heated by means of a resistance heated mantle, a sample loading port, a pair of ionization gauges, an Omegatron mass spectrometer, an orifice plate, means for admittance of pure gas for calibration and venting, and a pumping system. As indicated in the figure, the entire system is bakeable except for the cold trap and diffusion pump. An overall picture of the experimental arrangement is shown on Figure 13.

The ion gauges were attached to the tubulation on either side of the orifice plate. These gauges were Veeco RG-75P gauges, with Platinum iridium filaments operating at reduced filament temperature ($\sim 1000^{\circ}\text{C}$). The use of low temperature filament gauges reduces error due to pumping by the gauges. They were compared against each other in a gauge calibration unit and were found to read identically within one percent down to the 10^{-10} torr range. The same control boxes were used throughout the calibration and operational runs.

An orifice plate with several openings of graduated dimension was provided. This was constructed by drilling holes of varying diameters in a stainless steel plate to give the following conductances, 2.5; 0.975, 0.387, 0.079 and 0.030 l/sec. These provided a wide range of conductance to accommodate samples of varying outgassing loads. The under side of the plate was machined with a recess around each orifice which permitted the plate to be firmly positioned over the ground edge of the glass pumping port. The orifice plate was moved by magnets positioned external to the glass tube. It was also possible to use the orifice plate to completely close off the pumping port and thus make pressure-rise measurements which could be compared with the orifice pressure-drop measurements.

The sample loading port was a stainless steel flanged cover connected to the glass system with stainless steel bellows and a glass-to-metal seal. Shear seal copper gaskets were used. Thermocouple leads were provided through the stainless steel end plate. A close up picture is shown on Fig. 14.

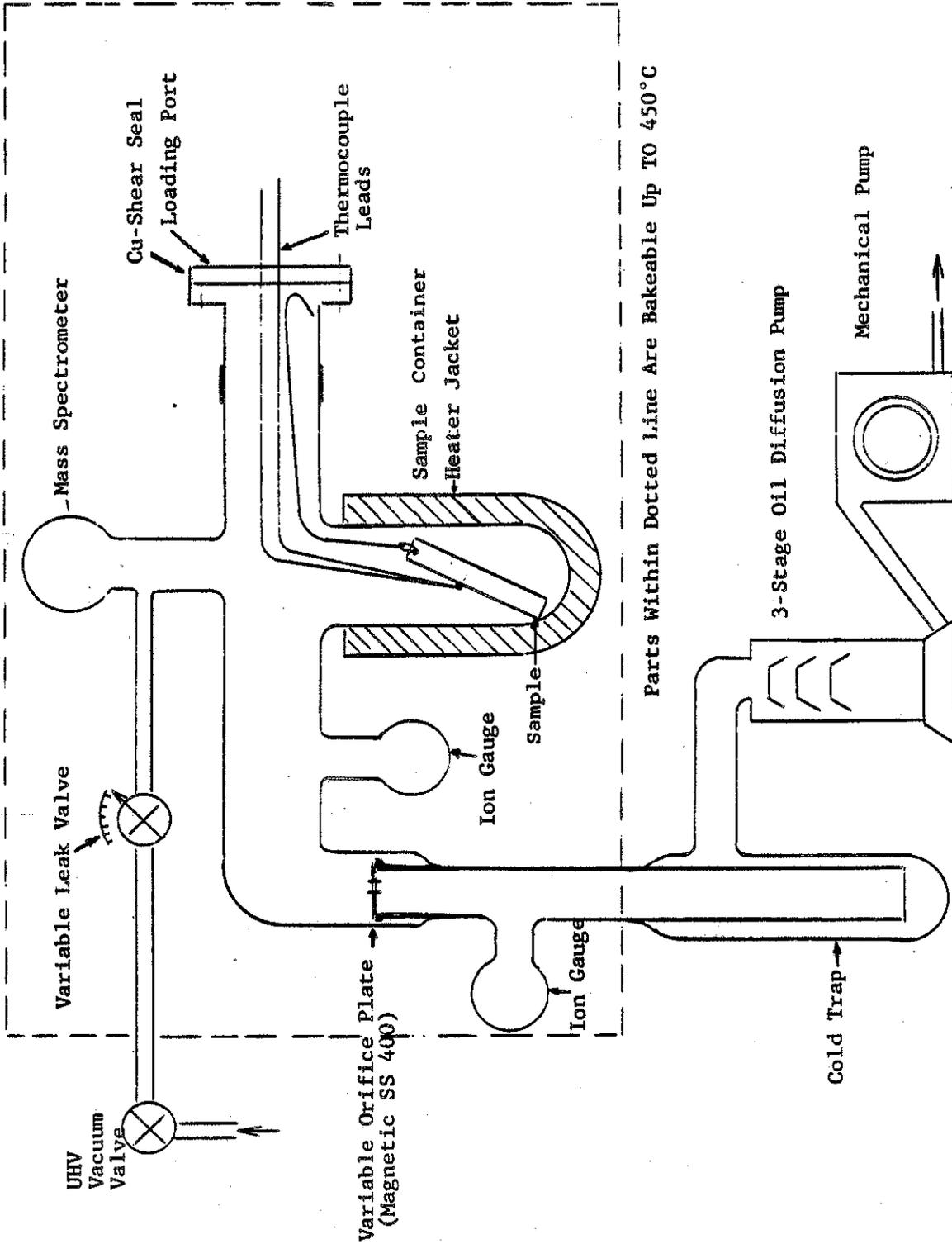


FIGURE 12 - Experimental Arrangement of the Glass System for Outgassing Studies

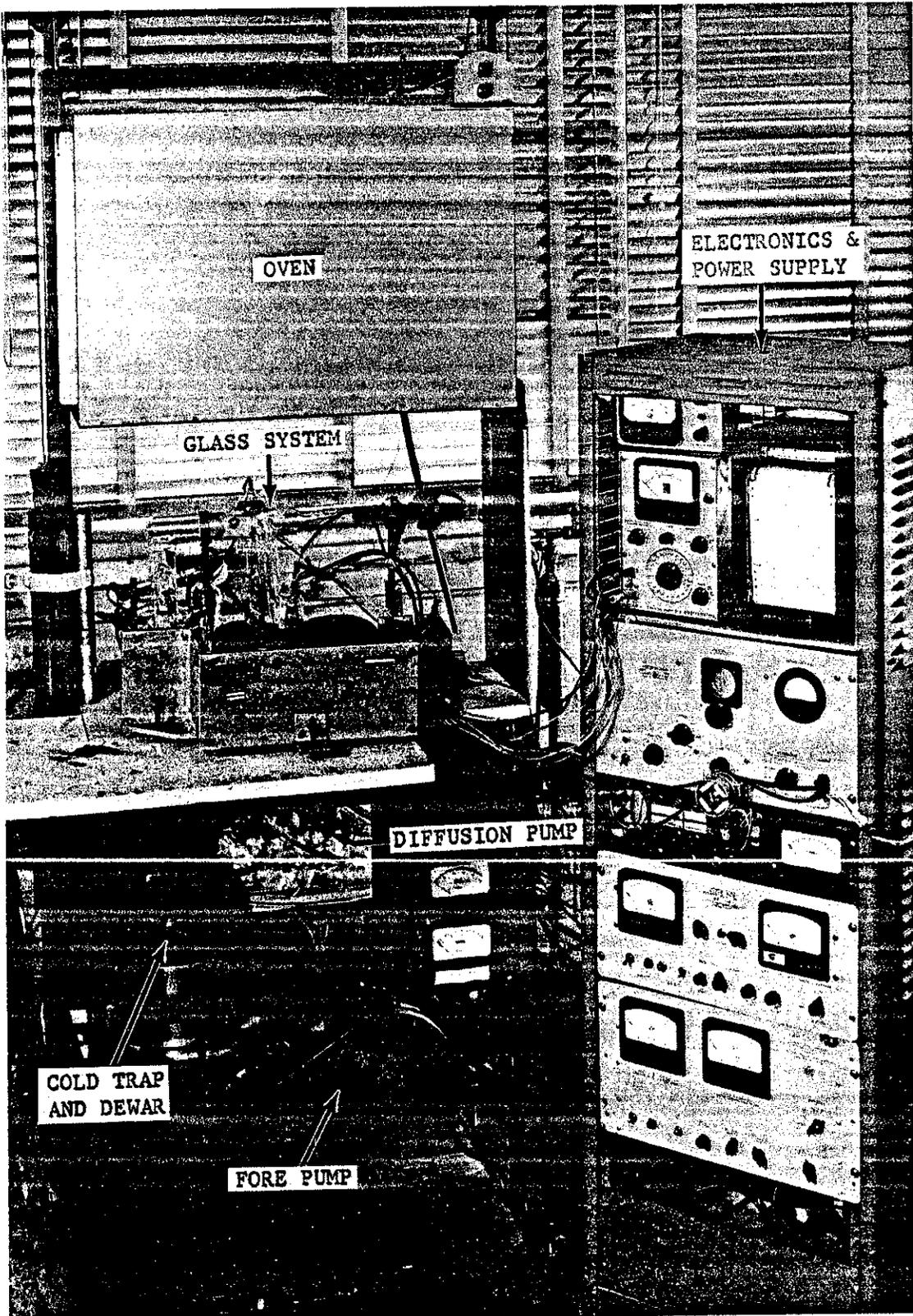


FIGURE 13 - GLASS SYSTEM AND AUXILIARY EQUIPMENT

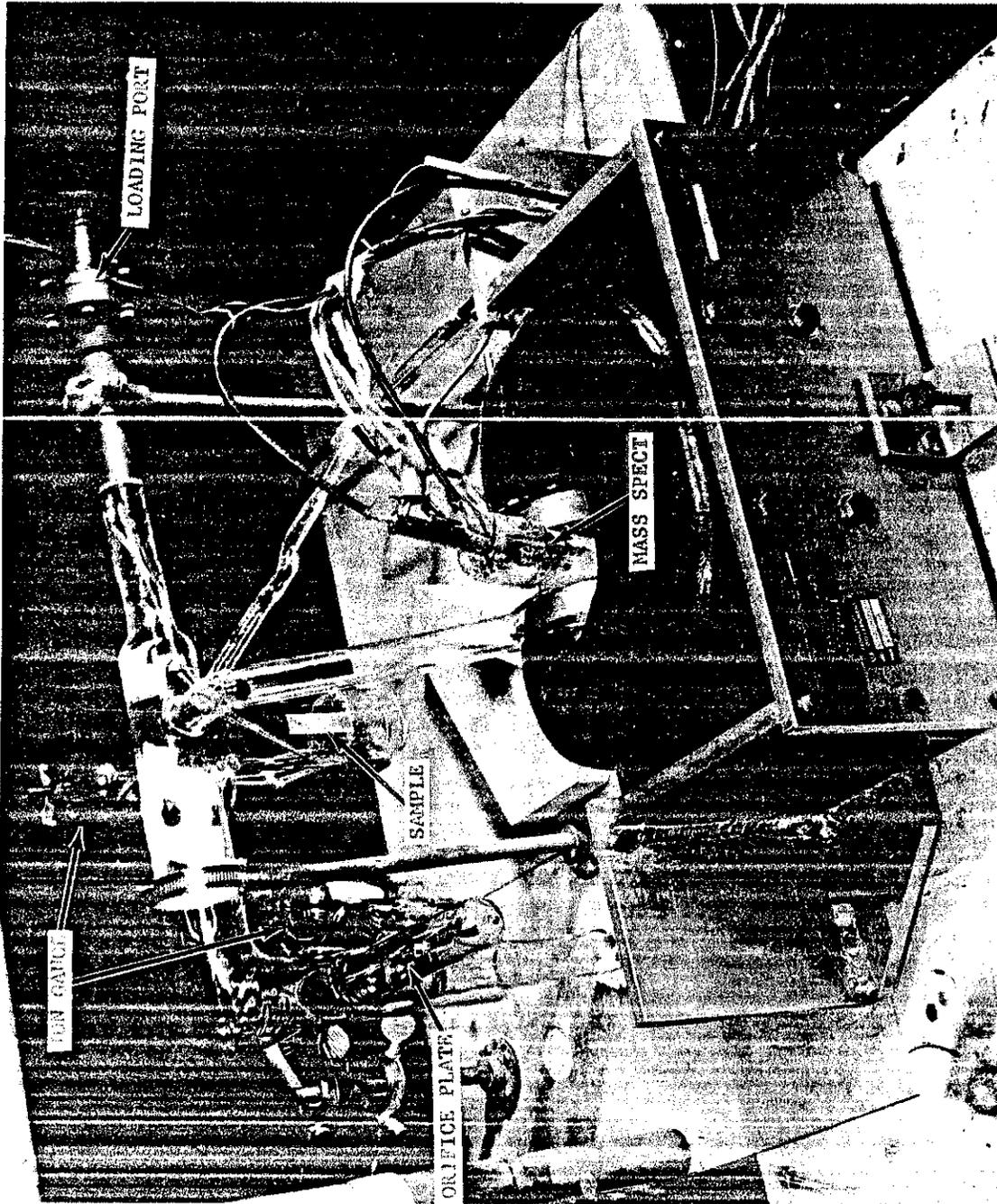


FIGURE 14 - GLASS SYSTEM WITH MASS SPECTROMETER

A controlled gas leak, provided by a Granville-Philips bakeable microleak valve in series with a second UHV valve (Type 2101B) provided means for admitting pure gases for calibration of the Omegatron and for venting the apparatus following a run.

The system was evacuated by a three-stage all glass oil diffusion pump (Type CEC-AF25) preceded by a liquid nitrogen cooled trap. The diffusion pump was backed by a two-stage mechanical pump.

4.3 EXPERIMENTAL PROCEDURES

4.3.1 System Background Test (or Blank Run)

The background outgassing from the glass system must be subtracted from the total outgassing measured during tests. An experiment to measure the outgassing from the empty system is termed hereafter a "blank" or "blank run". To reduce the outgassing from the glass system, the system was thoroughly baked out prior to installing the test specimen.

The system was first pumped down and baked out at about +400°C for eight hours after which it was cooled and a pressure in the middle of the 10⁻¹⁰ torr range was obtained. During the bakeout the ion gauges were outgassed and the mass spectrometer was maintained at a somewhat higher temperature than the rest of the system. An ultimate outgassing rate of 6 x 10⁻¹³ torr liters/sec. cm² for the cooled system was obtained under these conditions. After this, the system was vented with purified helium (research grade) up to one atmosphere. The pumps had been turned off at this time. The system was left in this condition for a time comparable to that which would be required later to load a sample, approximately ten minutes.

After this the system was pumped down without further bakeout to the lowest obtainable pressure which was in the 10⁻⁹ torr range. A temperature excursion was carried out in the same way as was planned for the samples. The different steps were +40°C, +100°C, +180°C, +240°C and back to room temperature. The final pressure obtained after this bakeout was only half a decade higher than before venting the system with helium.

At the same time, partial pressure, total pressure and outgassing data were recorded. Fig. 15 and Table VI show the outgassing rates in torr liter/cm²sec., as a function of elapsed time and temperature. Figure 16 and Table VIA (see Appendix) show total and partial pressures of the blank run as a function of elapsed time and temperature.

Next, a short bake-out of about two hours at 300°C brought the system down to the upper 10⁻¹⁰ torr range. The system was then ready for venting with helium and to be loaded with the first sample.

The blank proved to be negligibly small in comparison with the total gas loads encountered when the system was run with test samples.

4.3.2 Al-5083-0 Run ("as received" finish)

This aluminum alloy was selected as representative of the metal group because this metal is a candidate for use in large space chambers and also it was useful to check the data against the data obtained from the metal bell jars. The samples used in the glass system were six small metal plates with a total outgassing area of 205 cm². They were cut from the same sheet as the bell jar samples. The surface was not polished but only cleaned by wiping with alcohol. At the beginning of the run when the system was pumped down to 3 x 10⁻⁶ torr, the shear seal had not been tightened sufficiently and had to be retightened. This tiny leak caused the appearance of masses which could not be interpreted in connection with the sample. However, this will be discussed later. Figs. 17 and 18, Tables VII and VIIA (see Appendix) show the outgassing rates as well as the total and partial pressures.

Outgassing rates of this alloy were available from the results of the measurements taken with the metal bell jars and the data is shown in Fig. 8 of the first part. If one compares the slopes of the curves (Fig. 8) with the Fig. 17, agreement between the two is quite obvious.

Within the first seven hours the outgassing rate dropped rapidly about two orders of magnitude from 1 x 10⁻⁷ torr l/cm² sec. to about 1 x 10⁻⁹ torr l/cm² sec. which is almost the final stage for this material in the "as received" state without any heat treatment. Within the following fifteen hours the rate dropped only somewhat less than half a decade down to 6 or 7 x 10⁻¹⁰ torr l/cm² sec.

The following heat treatment up to +220°C within five hours increased the outgassing rate about an order of magnitude for every 50°C temperature increase. At +220°C an outgassing rate of 4 x 10⁻⁵ torr l/cm² sec. was measured. After

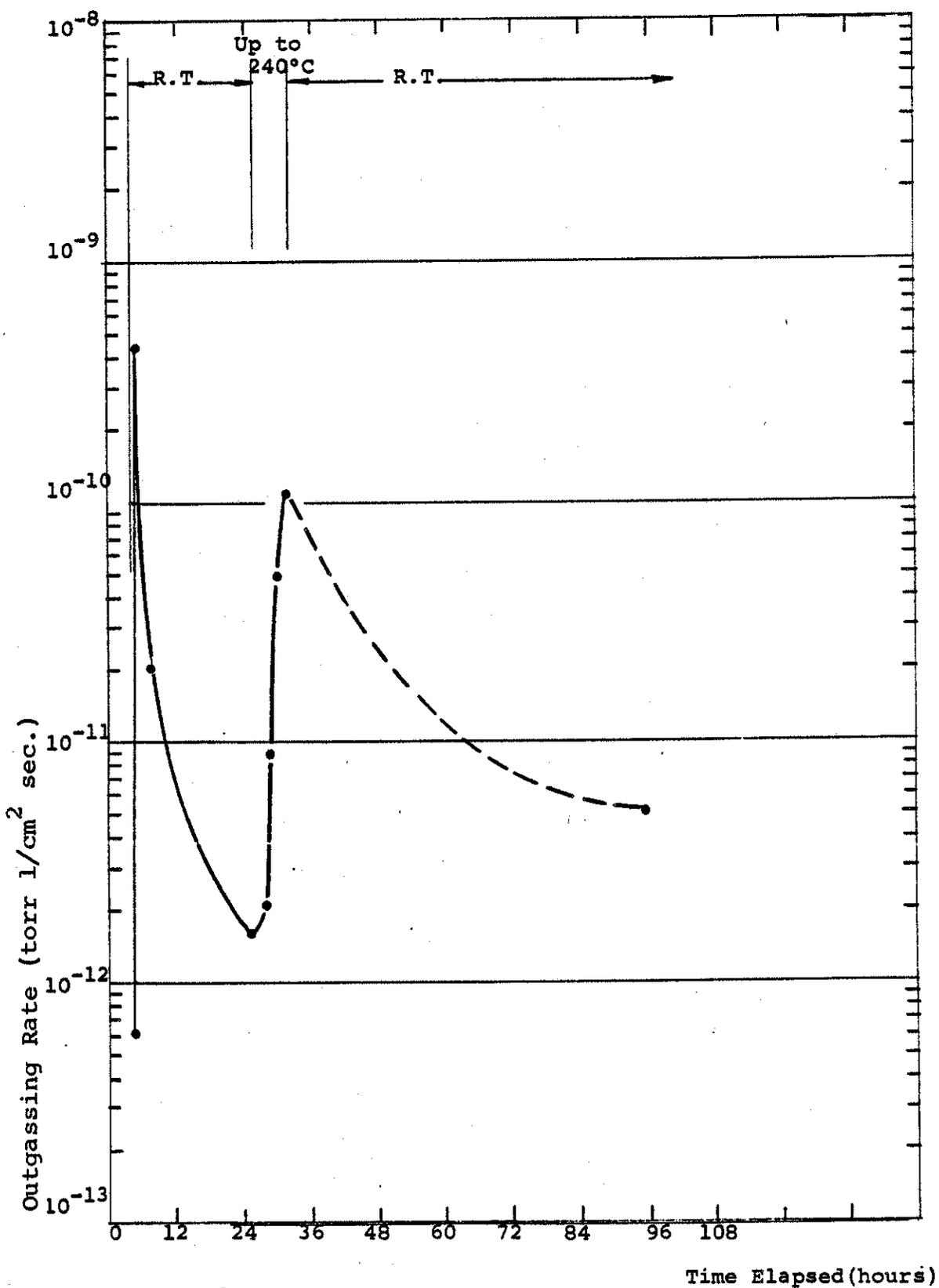


FIGURE 15 BLANK RUN

TABLE VI

Outgassing Rates - Blank Run - Glass System

Time Elapsed Hours	Temp. (°C)	Outgassing Rate (Torr l/sec.cm ²)	Remarks
4.5	R. T.	6.0×10^{-13}	After Thorough Bakeout.
5.5	R. T.	4.3×10^{-10}	He inlet and pump-down.
7.5	R. T.	2.0×10^{-11}	
24.75	R. T.	1.58×10^{-12}	
27.25	+40°C	2.15×10^{-12}	Heating up
28.50	+80°C	8.90×10^{-12}	system.
29.50	+180°C	4.90×10^{-11}	
31.0	+240°C	1.05×10^{-10}	
98.0	R. T.	5.0×10^{-12}	Cooled again to room temperature.

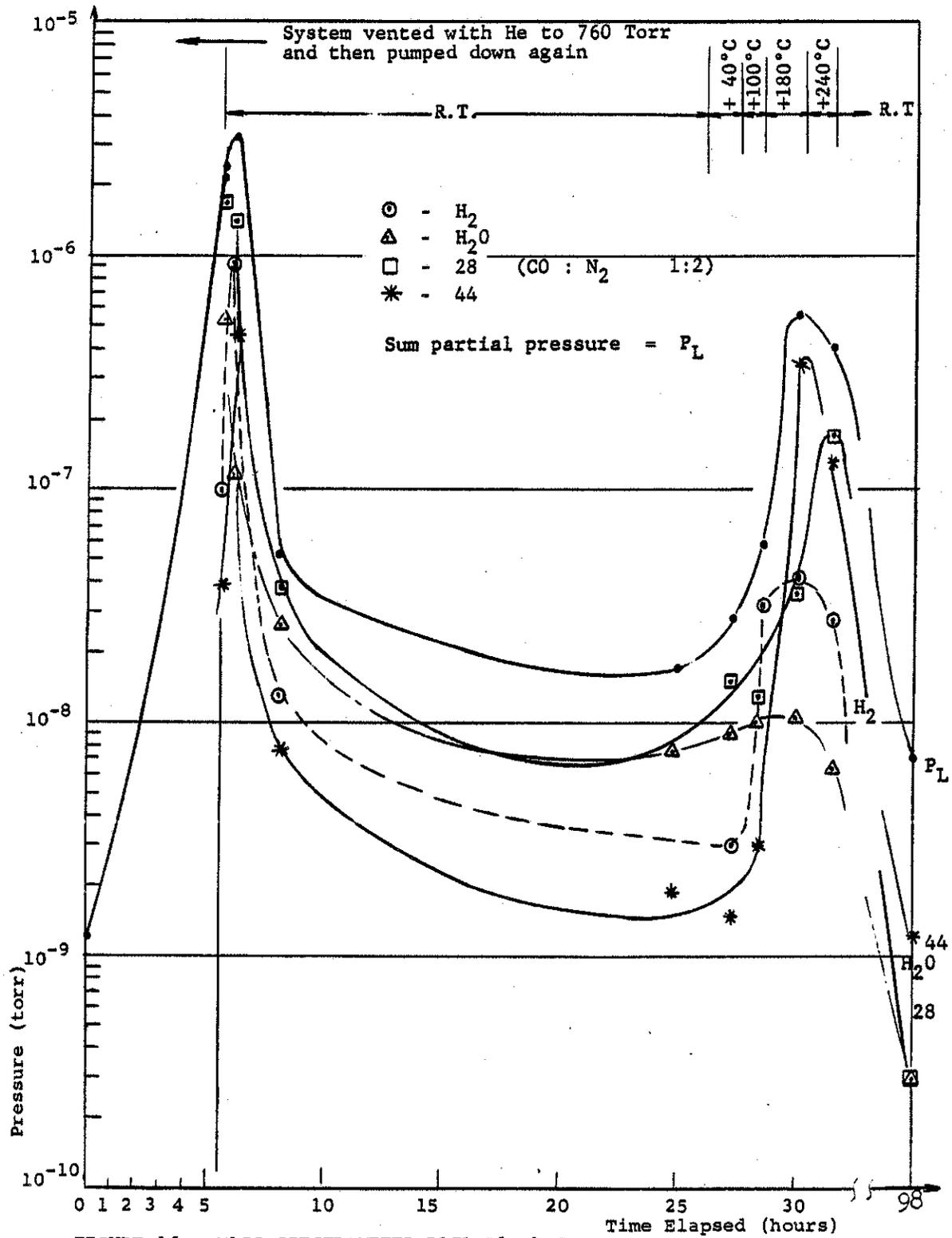


FIGURE 16 - MASS SPECTROMETER DATA Blank Run

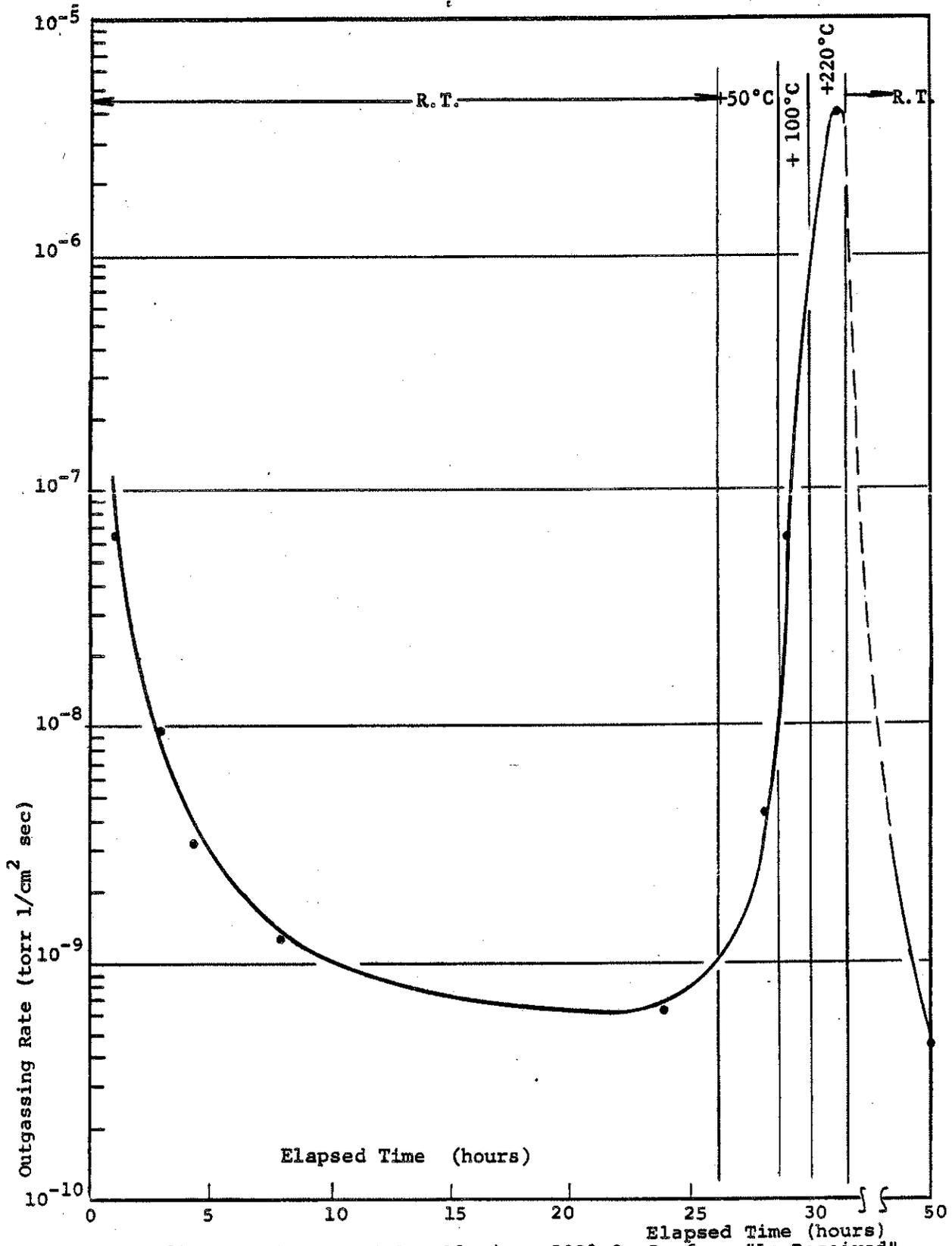


FIGURE-17 - OUTGASSING RATES, Aluminum 5083-0, Surface "As Received"

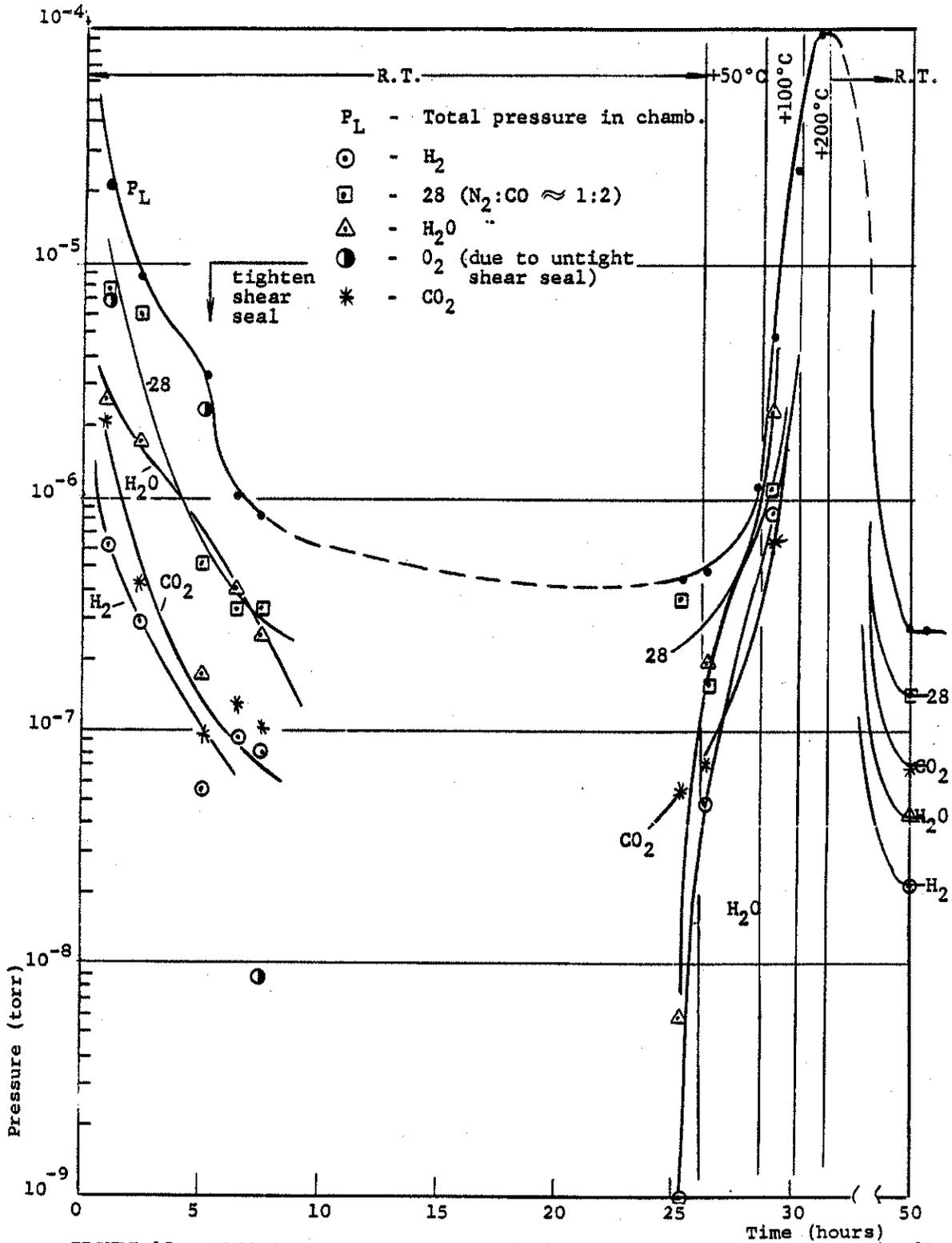


FIGURE 18 - MASS SPECTROMETER DATA, Aluminum 5083-0 Surface "As Received"

TABLE VII

Outgassing Rates - Aluminum 5083-0 ("as received")

Elapsed Time Hours	Temperature	Outgassing Rate Torr 1/cm ² sec.
0.92	R. T.	67.30 x 10 ⁻⁹
2.75	R. T.	9.97 x 10 ⁻⁹
4.25	R. T.	3.33 x 10 ⁻⁹
8.00	R. T.	1.27 x 10 ⁻⁹
24.00	R. T.	0.66 x 10 ⁻⁹
27.00	+ 50°C	1.43 x 10 ⁻⁹
29.00	+100°C	62.30 x 10 ⁻⁹
31.00	+220°C	406.00 x 10 ⁻⁹
50.00	R. T.	0.46 x 10 ⁻⁹

this the sample was cooled to room temperature and the outgassing rate dropped to 4×10^{-10} torr $1/\text{cm}^2$ sec. which was only slightly lower than before heating.

Regarding the partial pressures it can be observed that mass 28 and water are the main components again, followed by hydrogen and carbon dioxide. Mass 28 can be split roughly in a ratio 1:2 for $\text{N}_2:\text{CO}$, concluded out of the minor peaks from mass 12 and 14. At the beginning of the experiment oxygen appeared additionally as a major peak which could not be explained reasonably until the system was checked out once more for leaks. The shear seal was retightened and the oxygen peak dropped almost three decades in the following mass spectrometer reading. During the temperature excursion water and mass 28 were the major gases followed by hydrogen and CO_2 . This relation changed after heating up to $+200^\circ\text{C}$ and back to room temperature where mass 28 and CO_2 were the main components followed by water and hydrogen.

4.3.3 Al-Si-Mag 614

This material was selected from the group of ceramics. Twelve small plates with a total outgassing area of 156 cm^2 were brought into the system under helium atmosphere in the same way as the aluminum sample. The temperature cycle was almost the same as before, namely, pumpdown at room temperature until the pressure reached a final level followed by an excursion up to $+50^\circ\text{C}$, $+155^\circ\text{C}$, $+230^\circ\text{C}$ and back to room temperature. The Figs. 19 and 20 and Tables VIII and VIIIA (see Appendix) show the outgassing rates, partial and total pressures as a function of time and temperature.

Al-Si-Mag 614 is a very dense and hard material. It exhibited a very low initial outgassing rate at 2×10^{-8} torr $1/\text{cm}^2$ sec. and dropped about two orders of magnitude during the pumpdown procedure at room temperature within the next five to six hours. A further pumpdown of twenty hours diminished the outgassing rate only from 8×10^{-10} torr $1/\text{cm}^2$ sec. to 5×10^{-10} torr $1/\text{cm}^2$ sec.

The following temperature cycle brought the outgassing rate up to higher values, 3×10^{-8} torr $1/\text{cm}^2$ sec. at $+50^\circ\text{C}$ and 1×10^{-7} torr $1/\text{cm}^2$ sec. at $+155^\circ\text{C}$. However, the outgassing rate dropped to 5×10^{-8} torr $1/\text{cm}^2$ sec. at 230°C which allows the conclusion that this is certainly more a surface desorption rate than a volume outgassing rate. After the sample was cooled again to room temperature, the outgassing rate was 1×10^{-10} torr $1/\text{cm}^2$ sec. Regarding the various outgassed or desorbed gases, mass 28 ($\text{N}_2:\text{CO}$ 1:1) and water appeared as the main components followed by CO_2

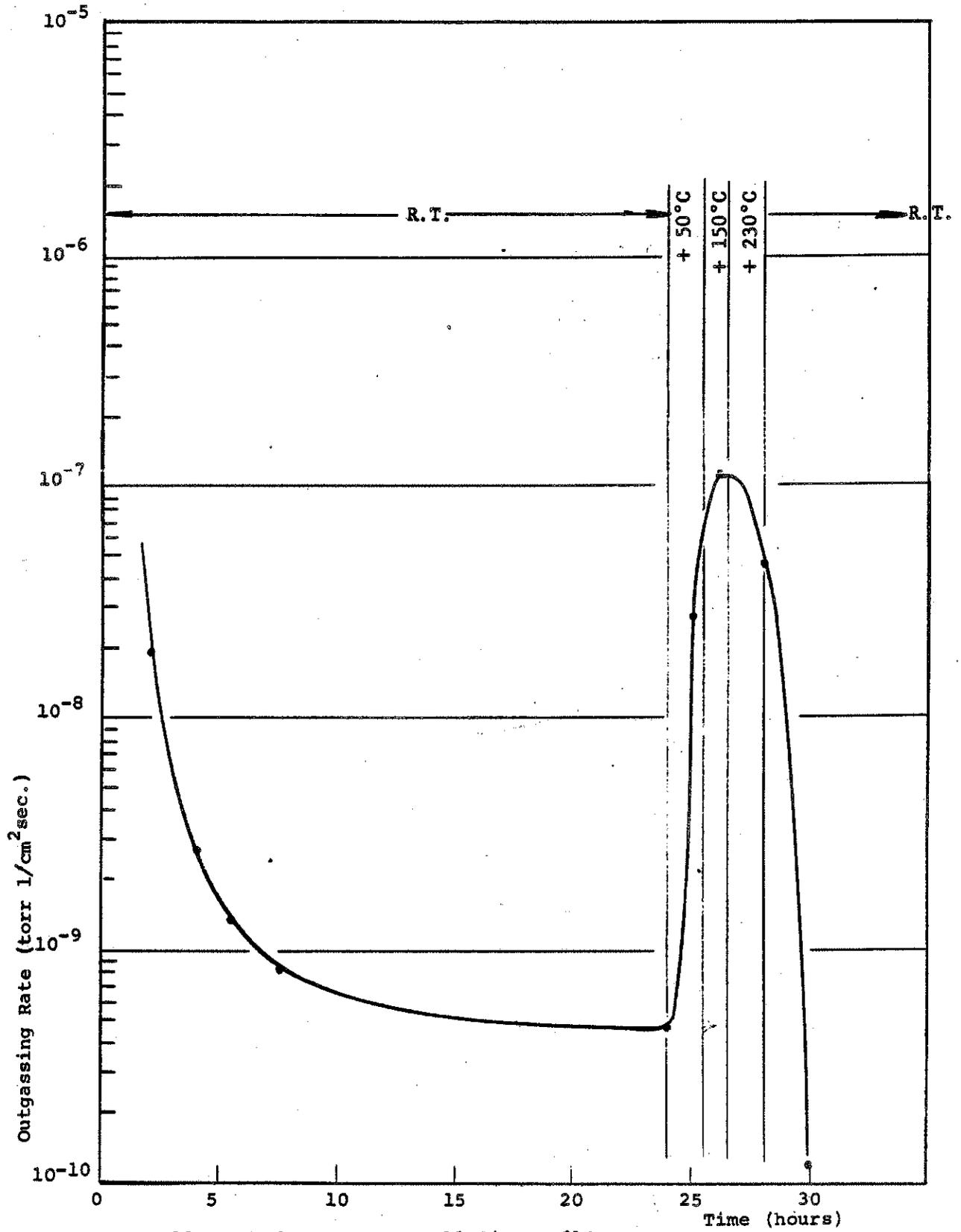


FIGURE 19 - OUTGASSING RATES, Al-Si-Mag 614

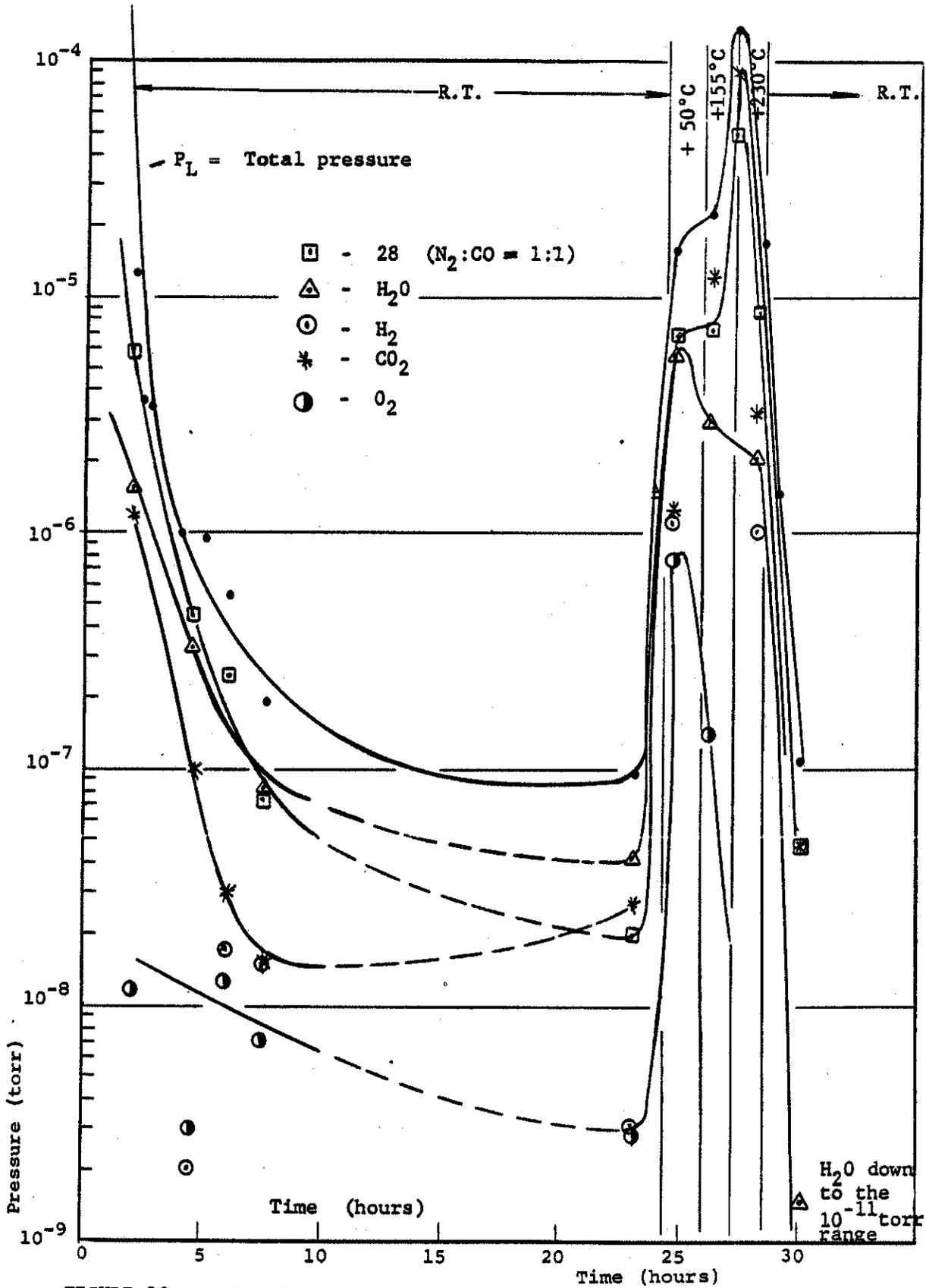


FIGURE 20 - MASS SPECTROMETER DATA, Al-Si-Mag 614

TABLE VIII

Outgassing Rates - Al-Si-Mag 614

Elapsed Time Hours	Temperature	Outgassing Rate Torr l/cm ² sec.
2.00	R. T.	1.9×10^{-8}
4.00	R. T.	2.74×10^{-9}
5.50	R. T.	1.39×10^{-9}
7.50	R. T.	8.1×10^{-10}
24.00	R. T.	4.85×10^{-10}
25.00	+ 50°C	2.8×10^{-8}
26.00	+100°C	1.15×10^{-7}
28.00	+230°C	4.8×10^{-8}
30.00	R. T.	1.7×10^{-10}

some hydrogen and oxygen at concentrations about an order of magnitude lower.

The heat treatment changed the ratios between the different gases. CO_2 and mass 28 became the major gases at $+230^\circ\text{C}$. Water, hydrogen and oxygen concentrations dropped rapidly during the second half of the bake-out period to values which could not be detected with the mass spectrometer. As mentioned above, at $+230^\circ\text{C}$, CO_2 was the main gas present. Its concentration dropped, however, faster than mass 28 during the time held at $+230^\circ\text{C}$. Both CO_2 and mass 28 reached the same value after the sample was cooled to room temperature, the partial pressures of both being 5×10^{-8} torr.

4.3.4 Teflon No. 7

From the group of elastomers, Teflon No. 7 was selected first. As in previous experiments samples in form of small plates were prepared. The total outgassing area of Teflon No. 7 was 370 cm^2 . This area is almost twice as much as in the previous runs; however, a low outgassing rate was expected from the experience made with the gas analysis system for the metal bell jars, where Teflon was used as gasket for the diatron tube of the mass spectrometer. The normal pumpdown procedure was followed by a temperature excursion as soon as the system reached a constant pressure level. In the case of the Teflon No. 7, the temperature could not be raised up to $+250^\circ\text{C}$ because of the expected high outgassing rate at this temperature. Therefore, the temperature excursion was limited to $+50^\circ\text{C}$ and $+100^\circ\text{C}$ and then back to room temperature. The outgassing rates, total and partial pressures as function of time and temperature are presented in Figs. 21 and 22 and Tables IX and IXA (see Appendix).

Teflon No. 7 with its long chains of carbon and fluorine atoms promised to show peaks at mass 31 (CF) and multiplicities of that 62, 93, However, the resolving power of the Omegatron mass spectrometer was limited by about mass 45-50. Therefore, only CF (31) could be observed very distinctly beside oxygen (32).

After the sample was brought into the system the pressure drop as a function of pump time appeared to be very sluggish so that the system was suspected several times to be leaking. Repeated intensive leak checking excluded this possibility. Two groups of gases could be observed. Nitrogen, CF and oxygen in the main group, water and CO_2 in the minor one. The ratios between these different gases stayed reasonably constant over a period of about 30 hours and each single gas dropped steadily. It can be concluded that in this case a real bulk outgassing effect occurred and that the material in spite of its smooth and dense appearance is highly porous at least as far as vacuum

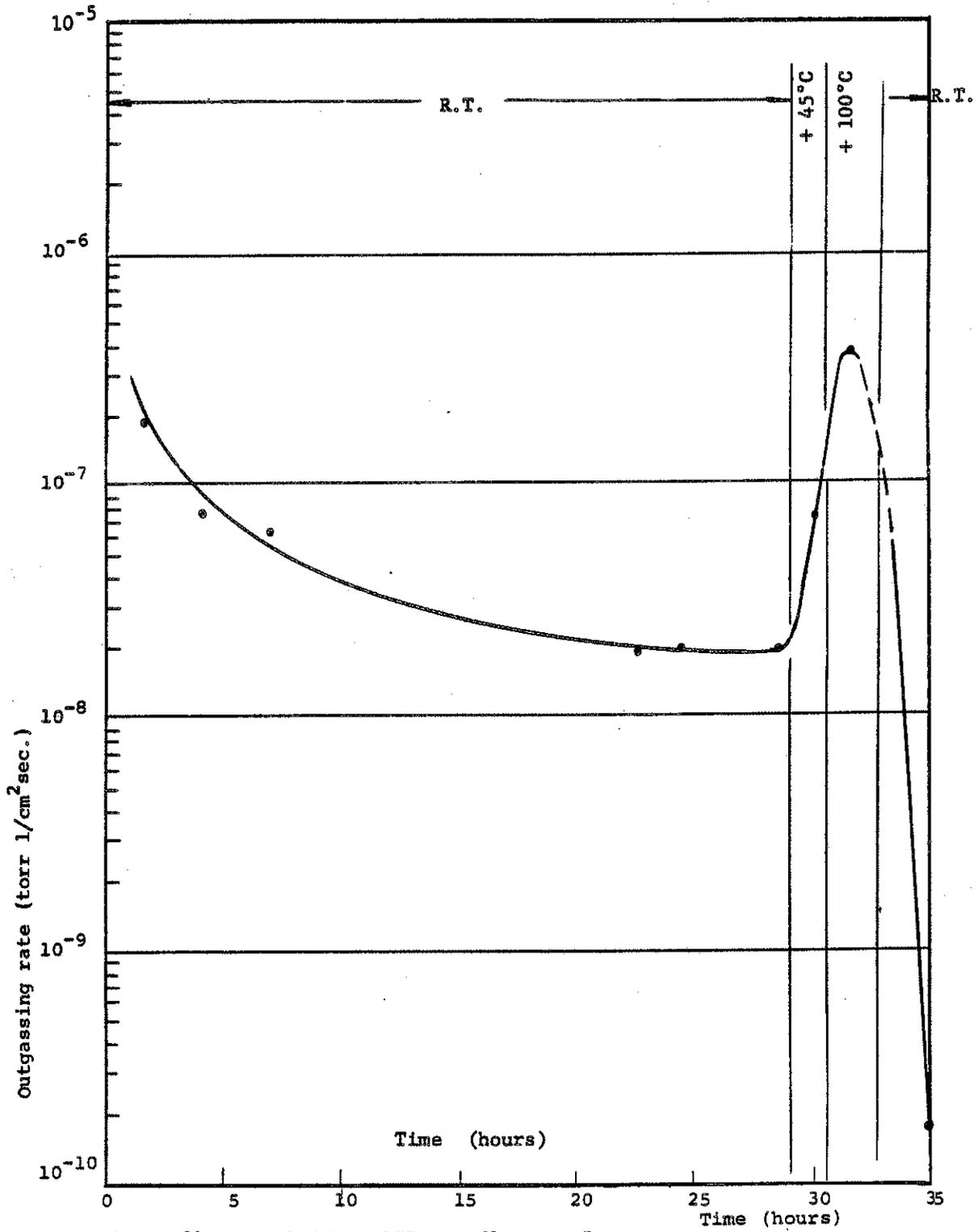


FIGURE 21 - OUTGASSING RATES, Teflon No. 7

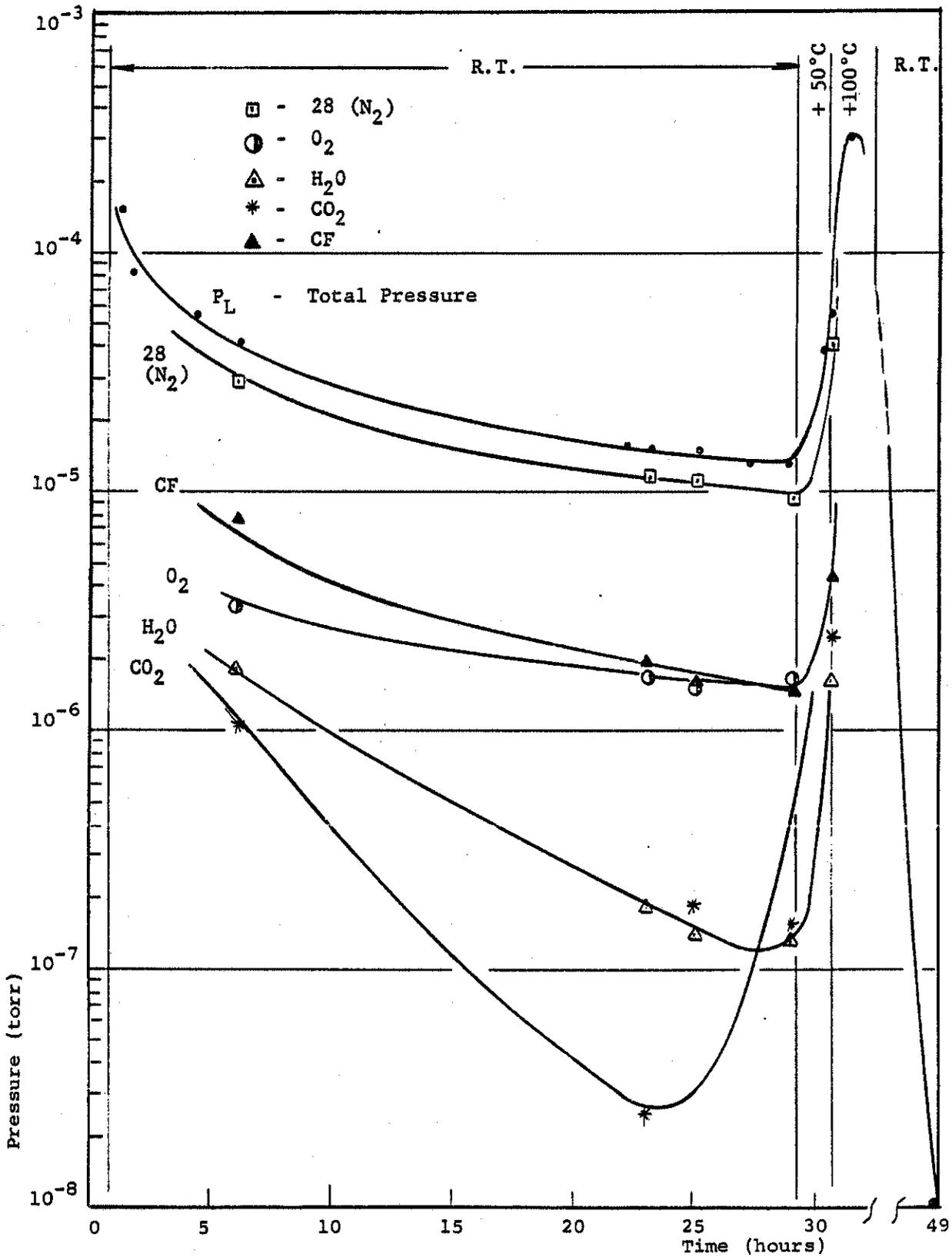


FIGURE 22 - MASS SPECTROMETER DATA. Teflon No. 7

TABLE IX
Outgassing Rates - Teflon No. 7

Elapsed Time Hours	Temperature	Outgassing Rate Torr 1/cm ² sec.
1.50	R. T.	19.00 x 10 ⁻⁸
4.00	R. T.	7.95 x 10 ⁻⁸
6.75	R. T.	6.55 x 10 ⁻⁸
22.50	R. T.	1.96 x 10 ⁻⁸
24.50	R. T.	2.05 x 10 ⁻⁸
28.50	R. T.	2.00 x 10 ⁻⁸
30.00	+ 45°C	7.30 x 10 ⁻⁸
31.50	+100°C	38.00 x 10 ⁻⁸
49.00	R. T.	0.02 x 10 ⁻⁸

requirements are concerned or is capable of dissolving substantial amounts of gas.

The temperature testing did not go as high as with the previous samples. It was limited to +50°C and +100°C. The upper pressure limit for the usability of the mass spectrometer was reached at +50°C with pressure at 5×10^{-5} torr. The following pressure rise to 3×10^{-4} torr during +100°C could only be recorded as total pressures. However, after the Teflon No. 7 sample was cooled down to room temperature the total pressure dropped three decades down to 1×10^{-8} torr. Mass spectrometer data were taken at this time, however, no major mass peaks could be observed. Instead of different peaks of multiplicity of different masses between 10 and 40 were recorded. This measurement seems to be doubtful. It is possible that the mass spectrometer was affected by the high outgassing rate at +100°C.

4.3.5 Mycalex 400

This material is glass-bonded mica and is used for coil supports, arc shields, high frequency transmitter panels, push rods and terminal boards.

The samples used for the outgassing studies were six slabs with a total visible surface area of 147.2 cm². The surface had been cleaned with alcohol and the samples had been 24 hours dustfree stored before they were put into the glass system.

The outgassing rate dropped steadily at room temperature from 1×10^{-7} to 6×10^{-10} torr liters/sec.cm² within the first twenty hours and reached at this point a constant value as can be seen from Fig. 23 and Table X. Water was the main component of the outgassed material followed by mass 28 and carbon dioxide. Mass 28 could be interpreted as nitrogen as long as the sample was at room temperature. Furthermore, mass 32 could be traced also. It was the smallest component. (Fig. 24; Table XA, see Appendix).

The large amount of water explains the steep increase of pressure and outgassing rate when the sample was warmed up to +140°C. The outgassing rate reached its highest value of 1×10^{-7} torr liters/sec.cm² at +100°C and then dropped drastically during further temperature increase. After cooling to room temperature the outgassing rate reached a final value of about 1.5×10^{-10} torr liters/sec. cm². At this time mass 28 was the major component followed by water and CO₂.

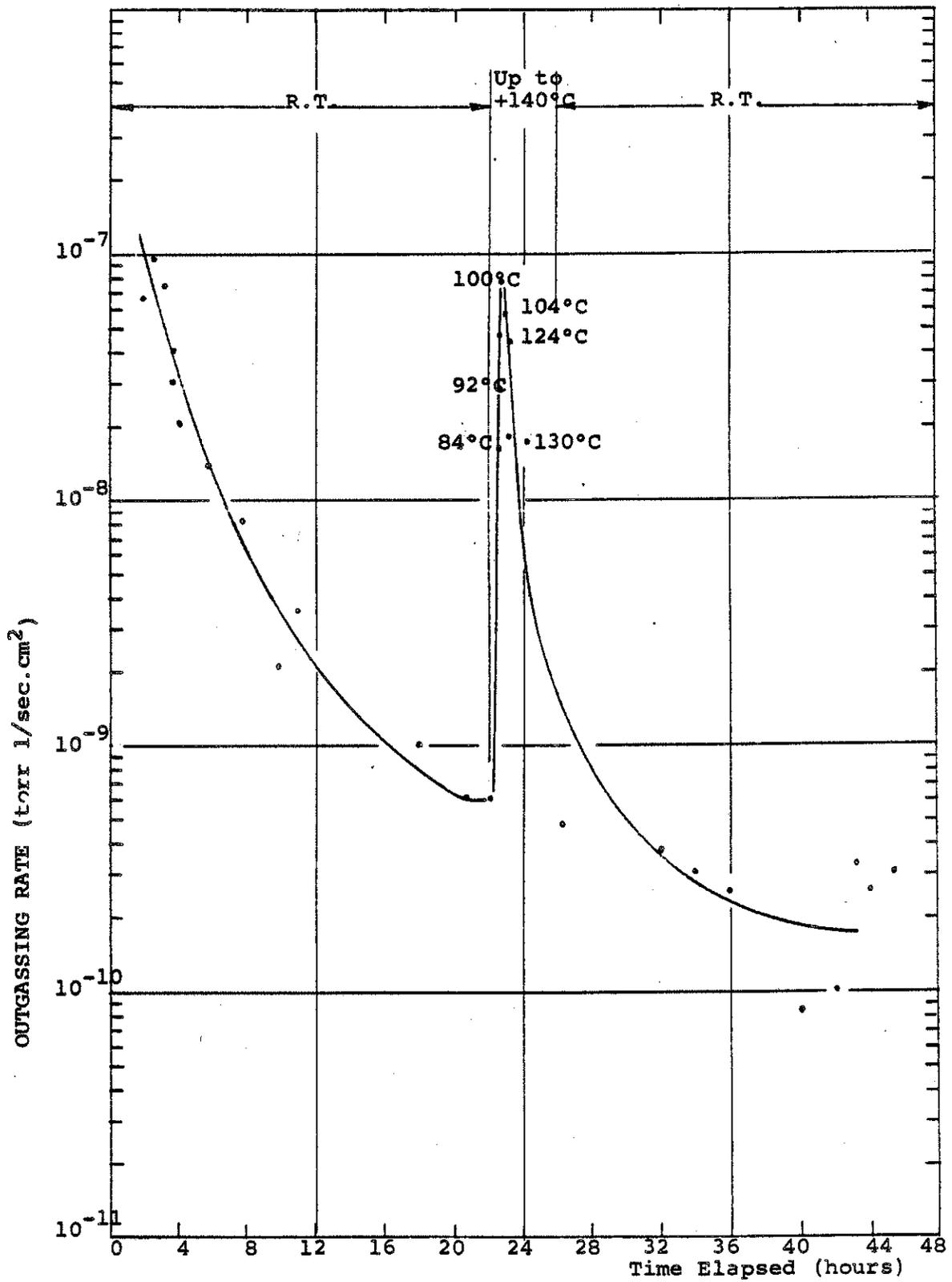


FIGURE 23 - MYCALEX 400

TABLE X

Outgassing Rates - Mycalex 400
Area: 147.2 cm²

Time Elapsed Hours	Temp. (°C)	Torr 1/sec.	Torr 1/sec.cm ²
2	R. T.	9.75 x 10 ⁻⁶	6.63 x 10 ⁻⁸
2.5	R. T.	1.45 x 10 ⁻⁵	9.85 x 10 ⁻⁸
3.5	R. T.	1.1 x 10 ⁻⁵	7.50 x 10 ⁻⁸
3.75	R. T.	4.5 x 10 ⁻⁶	3.06 x 10 ⁻⁸
		6.0 x 10 ⁻⁶	4.08 x 10 ⁻⁸
4.0	R. T.	3.0 x 10 ⁻⁶	2.04 x 10 ⁻⁸
5.8	R. T.	2.1 x 10 ⁻⁶	1.43 x 10 ⁻⁸
7.8	R. T.	1.2 x 10 ⁻⁶	8.2 x 10 ⁻⁹
10.8	R. T.	4.5 x 10 ⁻⁷	2.04 x 10 ⁻⁹
18.1	R. T.	1.5 x 10 ⁻⁷	3.06 x 10 ⁻⁹
20.4	R. T.	9.0 x 10 ⁻⁸	6.12 x 10 ⁻¹⁰
	R. T.	3.55 x 10 ⁻⁷	2.42 x 10 ⁻⁹
22.0	R. T.	9.0 x 10 ⁻⁸	6.12 x 10 ⁻¹⁰
	R. T.	3.84 x 10 ⁻⁷	2.62 x 10 ⁻⁹
22.67	84°C	2.4 x 10 ⁻⁶	1.63 x 10 ⁻⁸
22.74	92°C	4.2 x 10 ⁻⁶	2.86 x 10 ⁻⁸
22.75	100°C	1.14 x 10 ⁻⁴	7.75 x 10 ⁻⁷
22.80	104°C	8.55 x 10 ⁻⁵	5.82 x 10 ⁻⁷
22.90	124°C	6.6 x 10 ⁻⁵	4.5 x 10 ⁻⁷
23.0	130°C	2.7 x 10 ⁻⁵	1.84 x 10 ⁻⁷
24.5	140°C	2.7 x 10 ⁻⁶	1.84 x 10 ⁻⁸
		8.18 x 10 ⁻⁶	5.56 x 10 ⁻⁸
26.5	R. T.	7.2 x 10 ⁻⁸	4.9 x 10 ⁻¹⁰
	R. T.	1.69 x 10 ⁻⁷	1.15 x 10 ⁻⁹
30.0	R. T.	8.1 x 10 ⁻⁸	5.5 x 10 ⁻¹⁰
32.0	R. T.	5.85 x 10 ⁻⁸	3.98 x 10 ⁻¹⁰
34.0	R. T.	4.5 x 10 ⁻⁸	3.06 x 10 ⁻¹⁰
36.0	R. T.	3.9 x 10 ⁻⁸	2.65 x 10 ⁻¹⁰
38.0	R. T.	3.0 x 10 ⁻⁸	2.04 x 10 ⁻¹⁰
40.0	R. T.	1.2 x 10 ⁻⁸	8.16 x 10 ⁻¹¹
42.0	R. T.	1.5 x 10 ⁻⁸	1.02 x 10 ⁻¹⁰
43.6	R. T.	5.1 x 10 ⁻⁸	3.47 x 10 ⁻¹⁰
44.0	R. T.	3.92 x 10 ⁻⁸	2.67 x 10 ⁻¹⁰
45.0	R. T.	4.8 x 10 ⁻⁸	3.26 x 10 ⁻¹⁰

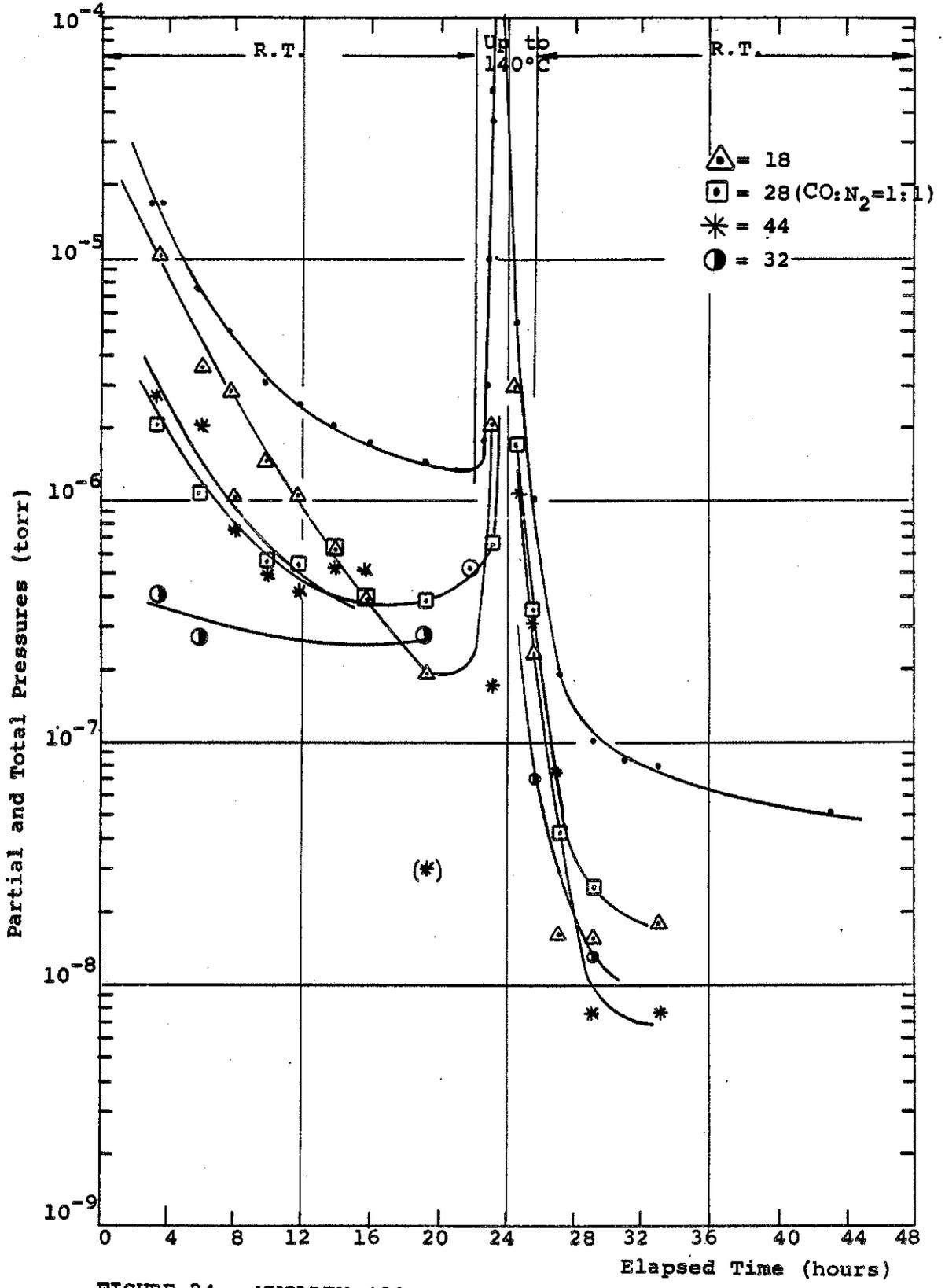


FIGURE 24 - MYCALEX 400

During and after bakeout small peaks belonging to masses 12 and 14 could be recorded. At this time mass 28 could be related to almost equal parts of carbon monoxide and nitrogen.

Despite the high density and hardness of Mycalex 400 the surface appears rough and porous. The true surface might be therefore exceeding the visible one and the outgassing rate would be then even lower.

4.3.6 Ceramic Coated Copper Wire

This type of wire can be applied where conductors are exposed to permanent temperatures up to +1000°F (+538°C). It is a nickel clad copper conductor insulated by a ceramic film. The sample of 164 cm² surface area had been taken from a reel from which the outer layers had been removed until the wire appeared visibly clean.

The outgassing rate of the sample dropped rather rapidly within the first 10-12 hours from 2×10^{-8} to 2×10^{-10} torr liters/sec. cm² and reached a constant value at this level. (Fig. 25 Table XI) The mass spectrometer indicated hydrogen as the predominant component. Water, mass 28 and carbon dioxide with almost equal partial pressure of 4×10^{-9} torr were almost one and a half orders of magnitude lower than hydrogen. (Fig. 26; Table XIA, see Appendix).

The subsequent heat treatment up to +197°C resulted in a steep increase in pressure up to 2×10^{-4} torr. The outgassing rate reached its peak value at 64°C with about 1.3×10^{-5} torr liters/sec. cm² and dropped then, oscillating a few times, down to 2.5×10^{-8} torr liters/sec. cm² when the sample was at +197°C. After the sample was cooled to room temperature the outgassing rate dropped to 2.9×10^{-10} torr liters/sec. cm².

In the mass composition hydrogen was still the major component followed by water and mass 28.

The large amount of hydrogen could be due to the copper wire. Only additional outgassing experiments with the bare copper wire or ceramic coating material only would allow reliable conclusions.

4.3.7 Cat-a-lac Paint

(Flat black, Code: 463-1-8)

This type of coating on epoxy base was chosen to study the outgassing characteristics of paint in vacuum. This paint was brushed on Aluminum 5083-0 samples which had been thoroughly

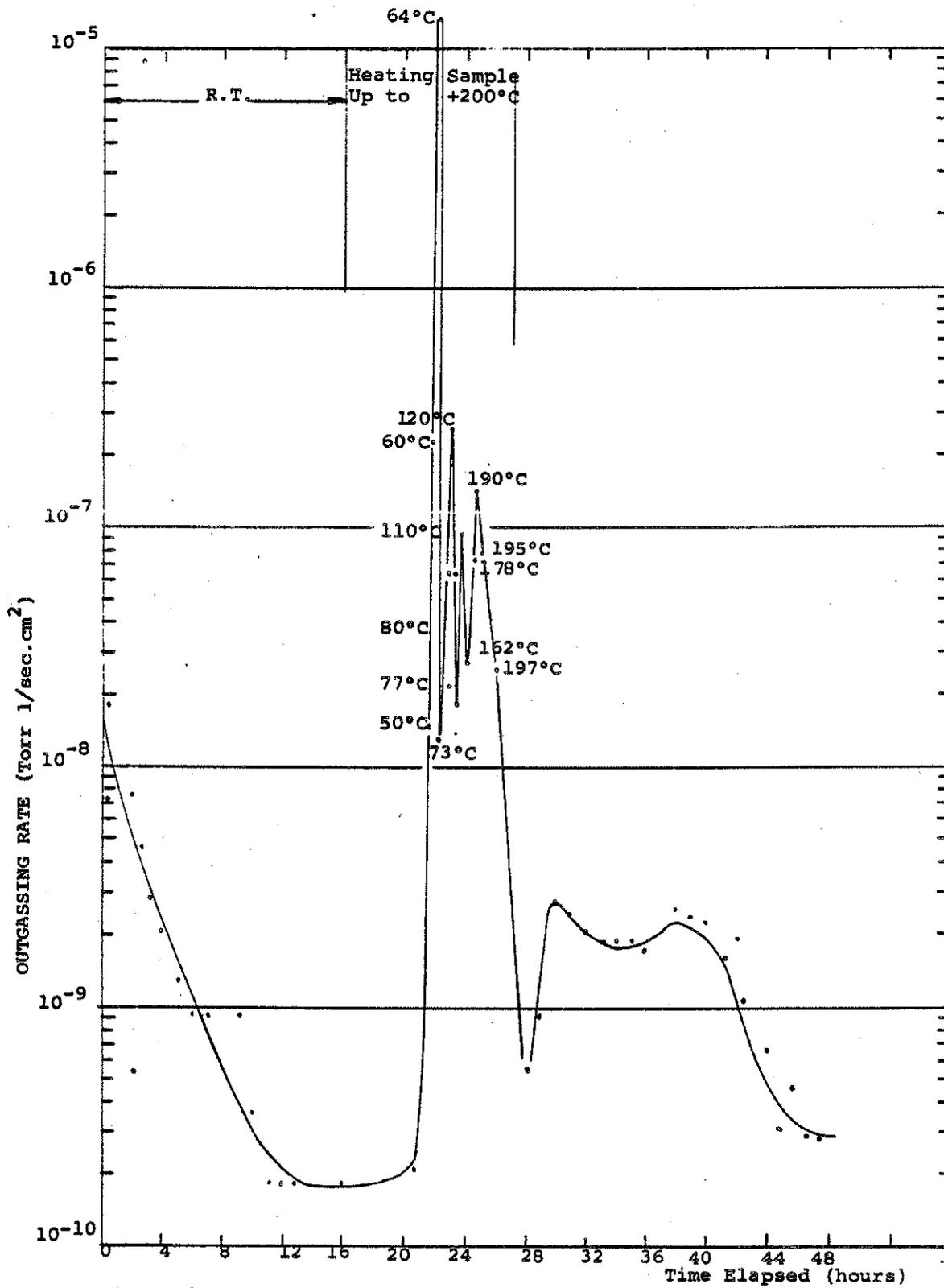


FIGURE 25 - CERAMIC COATED COPPER WIRE

TABLE XI
 Outgassing Rate - Ceramic Coated Copper Wire
 Area: 164.0 cm²

Time		Temp. (°C)		Torr l/sec.		Torr l/sec.cm ²		Time		Temp. (°C)		Torr l/sec.		Torr l/sec.cm ²	
Elapsed Hours	Hours							Elapsed Hours	Hours						
0.5		R.T.		1.2 x 10 ⁻⁶		7.3 x 10 ⁻⁹		23.6		+162°C		1.2 x 10 ⁻⁵		7.3 x 10 ⁻⁸	
2.0		R.T.		2.97x 10 ⁻⁶		1.81 x 10 ⁻⁸		24.0		+162°C		4.5 x 10 ⁻⁶		2.76 x 10 ⁻⁸	
2.5		R.T.		1.24x10 ⁻⁶		7.56 x 10 ⁻⁹		24.5		+178°C		1.2 x 10 ⁻⁵		7.3 x 10 ⁻⁸	
3.0		R.T.		7.5 x 10 ⁻⁷		4.57 x 10 ⁻⁹		24.6		+190°C		2.4 x 10 ⁻⁵		1.46 x 10 ⁻⁷	
4.0		R.T.		4.8 x 10 ⁻⁷		2.93 x 10 ⁻⁹		25.25		+195°C		1.28x 10 ⁻⁵		7.84 x 10 ⁻⁸	
5.0		R.T.		3.3 x 10 ⁻⁷		2.01 x 10 ⁻⁹		26.0		+197°C		4.16x 10 ⁻⁶		2.54 x 10 ⁻⁸	
6.0)		R.T.		2.1 x 10 ⁻⁷		1.28 x 10 ⁻⁹		26.4		+197°C		6.9 x 10 ⁻⁶		4.2 x 10 ⁻⁸	
7:0)		R.T.		1.5 x 10 ⁻⁷		9.15 x 10 ⁻¹⁰		27.5		STOP HEATING		3.49x 10 ⁻⁷		2.13 x 10 ⁻⁹	
8.0)		R.T.						28.1		+35°C		9.0 x 10 ⁻⁸		5.5 x 10 ⁻¹⁰	
9.0)		R.T.						29.0		R.T.		1.5 x 10 ⁻⁷		9.15 x 10 ⁻¹⁰	
10.0)		R.T.		6.0 x 10 ⁻⁸		3.66 x 10 ⁻¹⁰		30.0		R.T.		4.5 x 10 ⁻⁷		2.74 x 10 ⁻⁹	
11.0)		R.T.						31.0		R.T.		3.9 x 10 ⁻⁷		2.38 x 10 ⁻⁹	
12.0)		R.T.						32.0		R.T.		3.36x 10 ⁻⁷		2.05 x 10 ⁻⁹	
13.0)		R.T.		3.0 x 10 ⁻⁸		1.83 x 10 ⁻¹⁰		33.0)		R.T.		1.9 x 10 ⁻⁷			
16.0)		R.T.						34.0)		R.T.					
								35.0)		R.T.					
20.7		START HEATING						36.0		R.T.		2.82x 10 ⁻⁷		1.72 x 10 ⁻⁹	
21.5		R.T.		3.45 x 10 ⁻⁸		2.1 x 10 ⁻¹⁰		37.0		R.T.		3.6 x 10 ⁻⁷		2.2 x 10 ⁻⁹	
21.8		+50°C		2.4 x 10 ⁻⁶		1.46 x 10 ⁻⁸		38.0		R.T.		4.5 x 10 ⁻⁷		2.74x 10 ⁻⁹	
21.95		+60°C		3.75x 10 ⁻⁵		2.29 x 10 ⁻⁷		39.0		R.T.		3.99x 10 ⁻⁷		2.43 x 10 ⁻⁹	
22.05		+64°C		2.1 x 10 ⁻³		1.28 x 10 ⁻⁵		40.0		R.T.		3.75x 10 ⁻⁷		2.28 x 10 ⁻⁹	
22.25		+73°C		2.1 x 10 ⁻⁶		1.28 x 10 ⁻⁸		41.0		R.T.		2.54x 10 ⁻⁷		1.55 x 10 ⁻⁹	
22.4		+77°C		3.3 x 10 ⁻⁶		2.01 x 10 ⁻⁸		42.0		R.T.		3.24x 10 ⁻⁷		1.98 x 10 ⁻⁹	
22.6		+80°C		3.6 x 10 ⁻⁶		2.19 x 10 ⁻⁸		43.0		R.T.		1.65x 10 ⁻⁷		1.05 x 10 ⁻⁹	
22.8		+110°C		1.05x 10 ⁻⁵		6.4 x 10 ⁻⁸		44.16		R.T.		1.14x 10 ⁻⁷		6.95 x 10 ⁻¹⁰	
22.9		+120°C		4.25x 10 ⁻⁵		2.59 x 10 ⁻⁷		44.5		R.T.		5.1 x 10 ⁻⁸		3.1 x 10 ⁻¹⁰	
23.05		+144°C		1.01 x10 ⁻⁴		6.15 x 10 ⁻⁸		44.75		R.T.		7.72x 10 ⁻⁸		4.7 x 10 ⁻¹⁰	
23.25		+148°C		4.4 x 10 ⁻⁵		2.68 x 10 ⁻⁷		46.50		R.T.		4.8 x 10 ⁻⁸		2.9 x 10 ⁻¹⁰	
23.5		+156°C		3.0 x 10 ⁻⁶		1.83 x 10 ⁻⁸		46.75		R.T.		4.8 x 10 ⁻⁸		2.9 x 10 ⁻¹⁰	
		+157°C		1.5 x 10 ⁻⁵		9.15 x 10 ⁻⁸				R.T.					

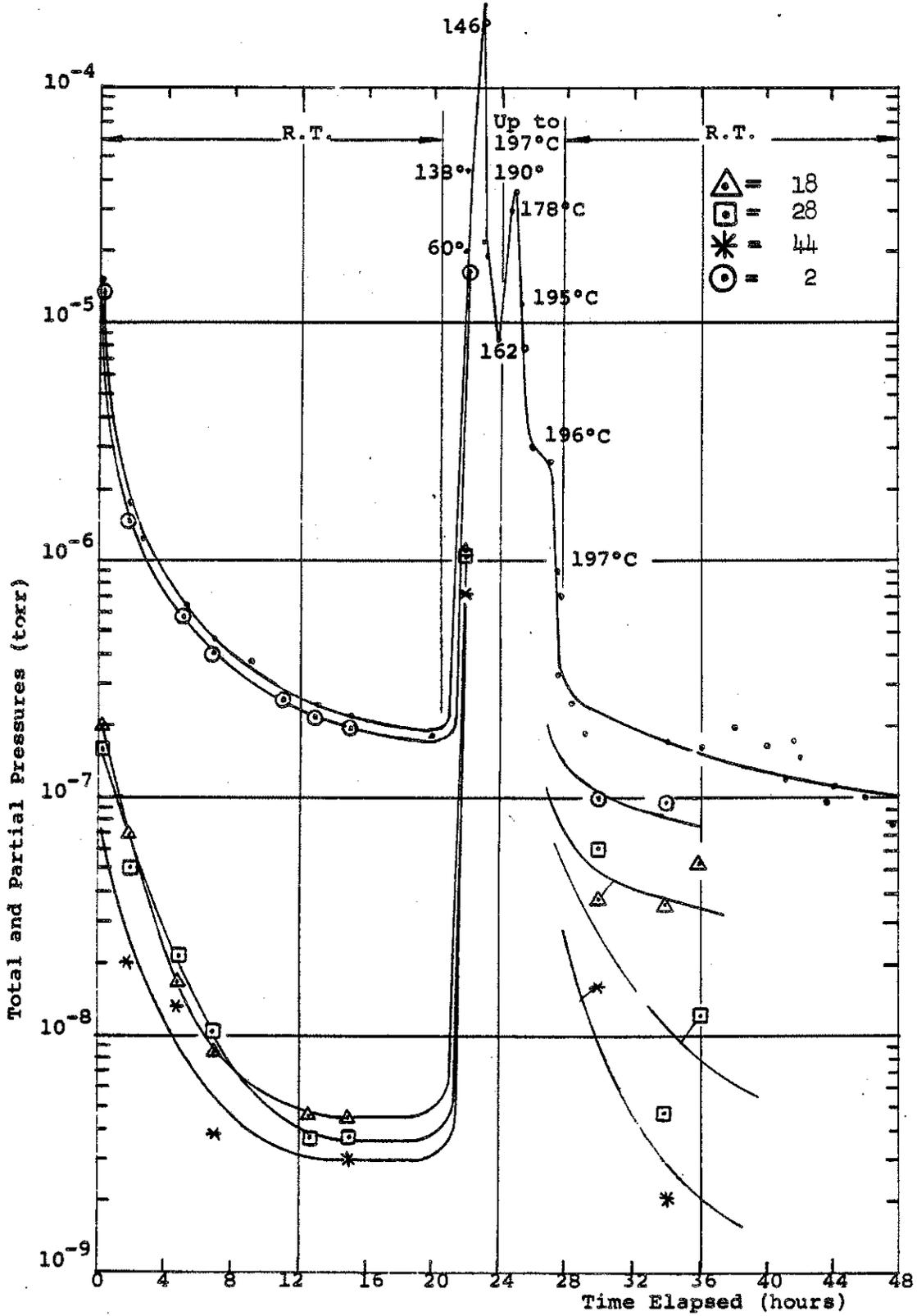


FIGURE 26 - CERAMIC COATED COPPER WIRE

cleaned by Turco* and alcohol. The samples with a total area of 218 cm² were dustfree stored and dried in room air for 96 hours before they were put into the glass system.

The outgassing rate dropped within the first four hours from 4×10^{-6} to 3.4×10^{-7} torr liters/sec. cm² and then decreased very slowly to 2×10^{-7} torr liters/sec. cm² within the next 45 hours at room temperature. (Fig. 27 Table XII). Because of the high pressure in the system of about 4×10^{-5} torr no mass spectrometer could be turned on continuously. The outgassing study which had been planned previously to be carried out only at room temperature had been extended by two brief cooling cycles and one soft bakeout in between. The first cooling of the sample to liquid nitrogen temperature decreased the outgassing rate to 2×10^{-10} torr liters/sec. cm². The subsequent soft bakeout up to +70°C increased the outgassing rate to 1.2×10^{-6} torr liters/sec. cm². At this time the Veeco gauge monitoring the environmental pressure of the samples had to be turned off because of the rapid pressure increase which threatened to poison the filament. The system had been controlled from there on only by the downstream gauge for the length of the heating period.

The heating cycle resulted in lowering the outgassing rate to 1.4×10^{-9} torr liters/sec. cm². This is about 2 orders of magnitude lower than previously measured at room temperature. After this the samples were exposed again to liquid nitrogen temperature. An outgassing rate of 6.9×10^{-11} torr liters/sec. cm² was measured this time.

After the sample was brought back to room temperature 5.5×10^{-10} torr liters/sec. cm² could be measured at the lowest outgassing rate from Cat-a-lac paint at room temperature.

During the test with Cat-a-lac paint no continuous record of the masses could be carried out because of the high pressures which occurred at certain stages in the test. (Fig. 28; Table XIIA, see Appendix). Masses could be checked only a few times between the 46th and 54th hour and at the end of the run. Water and mass 28 and sometimes nitrogen were the major components. Also masses 12 and 14 could be traced, and carbon dioxide was found towards the end of the run.

* Acidic liquid grease and rust remover of Turco, Inc., Newark, N. J.

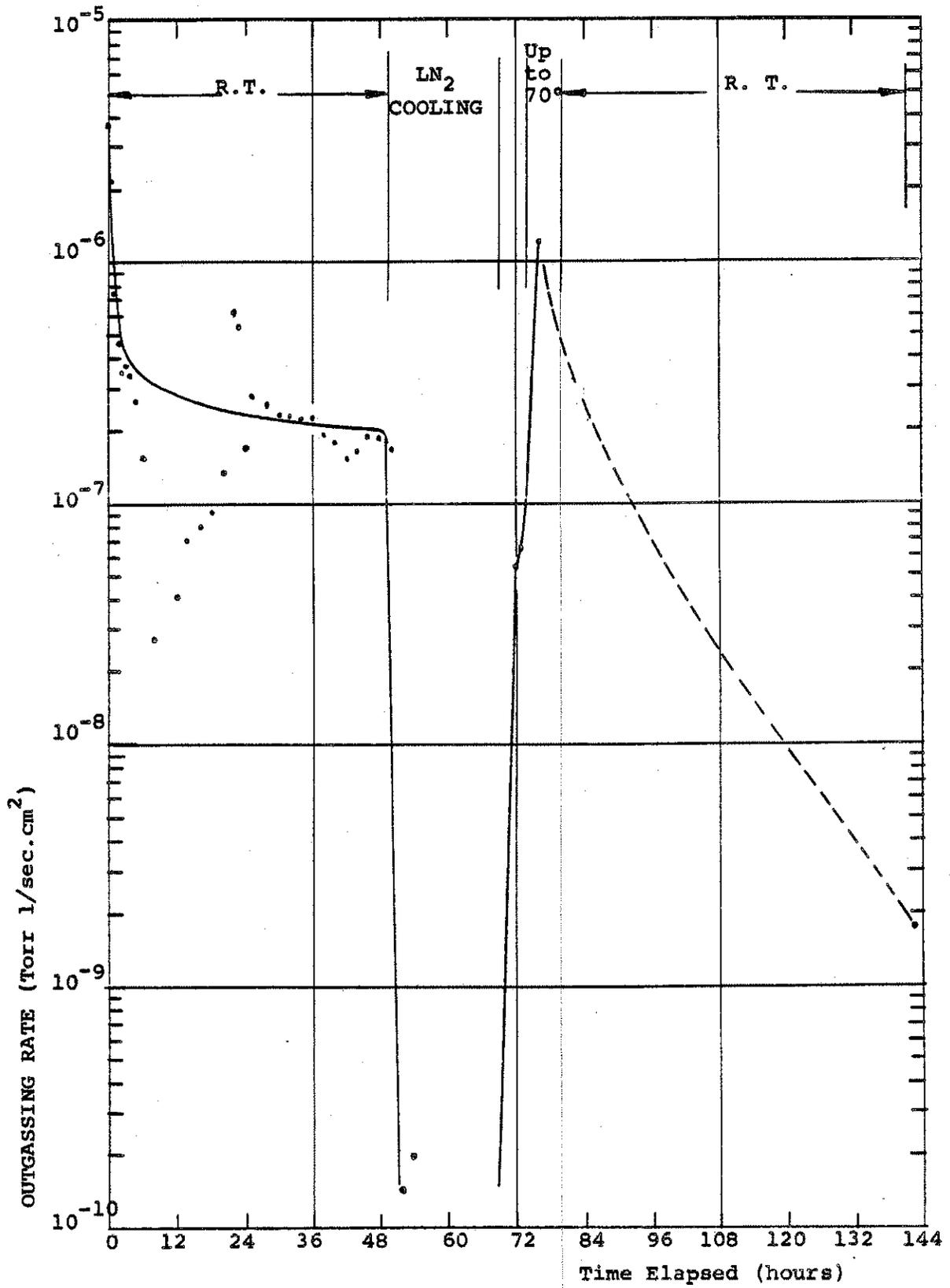


FIGURE 27 - CAT-A-LAC PAINT

TABLE XII
Outgassing rates Cat-a-lac painted on Al 5083-0

Time Elapsed Hours	Temp. (°C)	Orifice (1/sec.)	P (Torr)	Outgassing Rate (Torr 1/sec.)	(Tl/sec cm ²)
0.25		Full Open 31/sec	2.7×10^{-4}	8.1×10^{-4}	3.72×10^{-6}
0.50		" "	3.3×10^{-5}	9.9×10^{-5}	4.54×10^{-7}
1.00		" "	1.6×10^{-4}	4.8×10^{-4}	2.2×10^{-6}
1.50		" "	5.4×10^{-5}	1.62×10^{-4}	7.43×10^{-7}
2.00		" "	3.4×10^{-5}	1.02×10^{-4}	4.68×10^{-7}
2.50		" "	2.6×10^{-5}	7.80×10^{-5}	3.58×10^{-7}
3.00		" "	2.7×10^{-5}	8.1×10^{-5}	3.72×10^{-7}
3.50		" "	2.5×10^{-5}	7.5×10^{-5}	3.44×10^{-7}
4.00		" "	2.4×10^{-5}	7.5×10^{-5}	3.44×10^{-7}
4.50		" "	2.4×10^{-5}	7.2×10^{-5}	3.3×10^{-7}
		0.975 1/sec.	1.4×10^{-4}	1.37×10^{-4})	
				1.26×10^{-4}	5.8×10^{-7}
		0.387 1/sec.	3.03×10^{-4}	1.16×10^{-4})	
5.00		Full Open 31/sec	1.95×10^{-5}	5.7×10^{-5}	2.67×10^{-7}
		0.975 1/sec.	1.43×10^{-4}	1.39×10^{-4})	
				1.25×10^{-4}	5.74×10^{-7}
		0.387 1/sec.	2.85×10^{-4}	1.1×10^{-4})	
5.5		Full Open	1.6×10^{-5}	4.8×10^{-5}	2.2×10^{-7}
		0.975	1.20×10^{-4}	1.17×10^{-4})	
				1.06×10^{-4}	4.86×10^{-7}
		0.387	2.48×10^{-4}	9.6×10^{-5})	
6.0		Full Open	1.1×10^{-5}	3.3×10^{-5}	1.52×10^{-7}
6.5		" "	1.1×10^{-5}	3.3×10^{-5}	1.52×10^{-7}
7.0		" "	1×10^{-5}	3.0×10^{-5}	1.38×10^{-7}
8.0		" "	2×10^{-6})	6×10^{-6}	2.76×10^{-8}
9.0		" "			
10.0		" "			
11.0		" "			
12.0		" "	3×10^{-6}	9×10^{-6}	4.13×10^{-8}
13.0		" "	5×10^{-6}	1.5×10^{-5}	6.9×10^{-8}
14.0		" "	5×10^{-6}	1.5×10^{-5}	6.9×10^{-8}
15.0		" "	4×10^{-6}	1.2×10^{-5}	5.5×10^{-8}
16.0		" "	6×10^{-6}	1.8×10^{-5}	8.25×10^{-8}
17.0		" "	6×10^{-6}	1.8×10^{-5}	8.25×10^{-8}
18.0		" "	7×10^{-6}	2.1×10^{-5}	9.6×10^{-8}
19.0		" "	9×10^{-6}	2.7×10^{-5}	1.24×10^{-7}
20.0		" "	1.0×10^{-5}	3.0×10^{-5}	1.38×10^{-8}
21.0		" "	1.1×10^{-5}	3.3×10^{-5}	1.52×10^{-7}
22.5		" "	3.5×10^{-5}	1.4×10^{-4}	6.4×10^{-7}
		0.975 1/sec.	1.3×10^{-4}	1.27×10^{-4})	
				1.22×10^{-4}	5.57×10^{-7}
		0.387 1/sec.	2.98×10^{-4}	1.16×10^{-4})	
23.0		Full Open	3.7×10^{-5}	1.1×10^{-4}	5.05×10^{-7}
23.5		" "	2.6×10^{-5}	7.8×10^{-5}	3.58×10^{-7}
24.00	dp/dt	2.4 Tl/sec x)			
		2.5 Tl/sec x)	1.5	3.7×10^{-5}	1.7×10^{-7}
24.5		Full Open	2.8×10^{-5}	8.4×10^{-5}	3.85×10^{-7}
25.5		" "	2.1×10^{-5}	6.3×10^{-5}	2.9×10^{-7}
26.5		" "	2.1×10^{-5}	6.3×10^{-5}	2.9×10^{-7}
27.5		" "	2.0×10^{-5}	6.0×10^{-5}	2.76×10^{-7}

TABLE XII - continued

Time	Elapsed Temp.	Orifice	P		Outgassing Rate	
Hours	(°C)	(l/sec)	(Torr)	(Torr l/sec.)	(Tl/sec.cm ²)	
28.0		Full Open	1.9x10 ⁻⁵	5.7x10 ⁻⁵	2.62 x 10 ⁻⁷	
29.0		" "	1.7x10 ⁻⁵	5.1x10 ⁻⁵	2.34 x 10 ⁻⁷	
30.0		" "	1.7x10 ⁻⁵	5.1x10 ⁻⁵	2.34 x 10 ⁻⁷	
31.0		Full Open	1.6x10 ⁻⁵	4.8x10 ⁻⁵	2.2 x 10 ⁻⁷	
32.0		" "	1.7x10 ⁻⁵	5.1x10 ⁻⁵	2.34 x 10 ⁻⁷	
33.0		" "	1.7x10 ⁻⁵	5.1x10 ⁻⁵	2.34 x 10 ⁻⁷	
34.0		" "	1.7x10 ⁻⁵	5.1x10 ⁻⁵	2.34 x 10 ⁻⁷	
35.0		" "	1.7x10 ⁻⁵	5.1x10 ⁻⁵	2.3 x 10 ⁻⁷	
36.0		" "	1.65x10 ⁻⁵	4.95x10 ⁻⁵	2.27 x 10 ⁻⁷	
37.0		" "	1.5x10 ⁻⁵	4.5x10 ⁻⁵	2.06 x 10 ⁻⁷	
38.0		" "	1.4x10 ⁻⁵	4.2x10 ⁻⁵	1.93 x 10 ⁻⁷	
39.0		" "	1.3x10 ⁻⁵	3.9x10 ⁻⁵	1.79 x 10 ⁻⁷	
40.0		" "	1.3x10 ⁻⁵	3.9x10 ⁻⁵	1.79 x 10 ⁻⁷	
41.0		" "	1.2x10 ⁻⁵	3.6x10 ⁻⁵	1.65 x 10 ⁻⁷	
42.0		" "	1.1x10 ⁻⁵	3.3x10 ⁻⁵	1.52 x 10 ⁻⁷	
43.0		" "	1.1x10 ⁻⁵	3.3x10 ⁻⁵	1.52 x 10 ⁻⁷	
44.0		" "	1.2x10 ⁻⁵	3.6x10 ⁻⁵	1.65 x 10 ⁻⁷	
45.0		" "	1.2x10 ⁻⁵	3.6x10 ⁻⁵	1.65 x 10 ⁻⁷	
46.25		" "	1.4x10 ⁻⁵	4.2x10 ⁻⁵	1.93 x 10 ⁻⁷	
47.5		" "	1.3x10 ⁻⁵	3.9x10 ⁻⁵	1.79 x 10 ⁻⁷	
48.0		" "	1.4x10 ⁻⁵	4.2x10 ⁻⁵	1.93 x 10 ⁻⁷	
49.0		" "	1.3x10 ⁻⁵	3.9x10 ⁻⁵	1.79 x 10 ⁻⁷	
50.5		Start 1st Cooling.	1.2x10 ⁻⁵	3.6x10 ⁻⁵	1.65 x 10 ⁻⁷	
		Samples cooled LN ₂ Temp.				
52.0		0.975 l/sec	3.5x10 ⁻⁸	3.4x10 ⁻⁸)		
		0.387 l/sec	9.0x10 ⁻⁸	3.48x10 ⁻⁸)	3.18x10 ⁻⁸	1.46 x 10 ⁻¹⁰
		0.079 l/sec	3.4x10 ⁻⁷	5.1x10 ⁻⁷)		
53.0		0.975 l/sec	4.5x10 ⁻⁸	3.9x10 ⁻⁸)		
		0.387 l/sec	1.4x10 ⁻⁷	5.41x10 ⁻⁸)	4.45x10 ⁻⁸	2.04 x 10 ⁻¹⁰
		0.079 l/sec	5.1x10 ⁻⁷	4.03x10 ⁻⁸)		
54.0		0.975	-	-		
		0.387	3x10 ⁻⁸	1.17x10 ⁻⁷)	1.04x10 ⁻⁷	4.77 x 10 ⁻¹⁰
		0.079	1.1x10 ⁻⁷	8.7x10 ⁻⁸)		
69.0		Warming up sample to room temperature				
72.0	15-20°C	Full Open	4.0x10 ⁻⁶	1.2x10 ⁻⁵		5.5 x 10 ⁻⁸
		0.975 l/sec	2.03x10 ⁻⁵	2.01x10 ⁻⁵		
		0.387 l/sec	5.1x10 ⁻⁵	1.97x10 ⁻⁵	1.62x10 ⁻⁵	7.4 x 10 ⁻⁸
		0.079 l/sec	1.13x10 ⁻⁴	8.9x10 ⁻⁶		
72.5	28°C	Full Open	4.8x10 ⁻⁶	1.44x10 ⁻⁵		6.6x10 ⁻⁸
72.75	33°C	" "	1.0x10 ⁻⁵	1.5x10 ⁻⁵		6.9x10 ⁻⁸
73.15	35°C	" "	8.8x10 ⁻⁵	2.64x10 ⁻⁴		1.21x10 ⁻⁶
		During higher temperatures Veeco gauge turned off cooling down to room temperature and pumping over the weekend.				
143.0	R.T.	" "	1x10 ⁻⁷	3 x 10 ⁻⁷		1.38x10 ⁻⁹
143.5	R.T.	0.975	5x10 ⁻⁷	= 4.86)		
		0.387	1.03x10 ⁻⁶	= 3.99)10 ⁻⁷ =		
				3.83x10 ⁻⁷		1.76 x 10 ⁻⁹
		0.079	3.45x10 ⁻⁶	= 2.63)		
		Start 2nd cooling cycle with LN ₂				
146.5		dp/dt = 1.05 x 10 ⁻⁸ t/sec x 1.51 = 1.5x10 ⁻⁸				6.9 x 10 ⁻¹¹
		Back to room temperature				
148.0	R.T.	Full Open	4 x 10 ⁻⁸	1.2 x 10 ⁻⁷		5.5 x 10 ⁻¹⁰

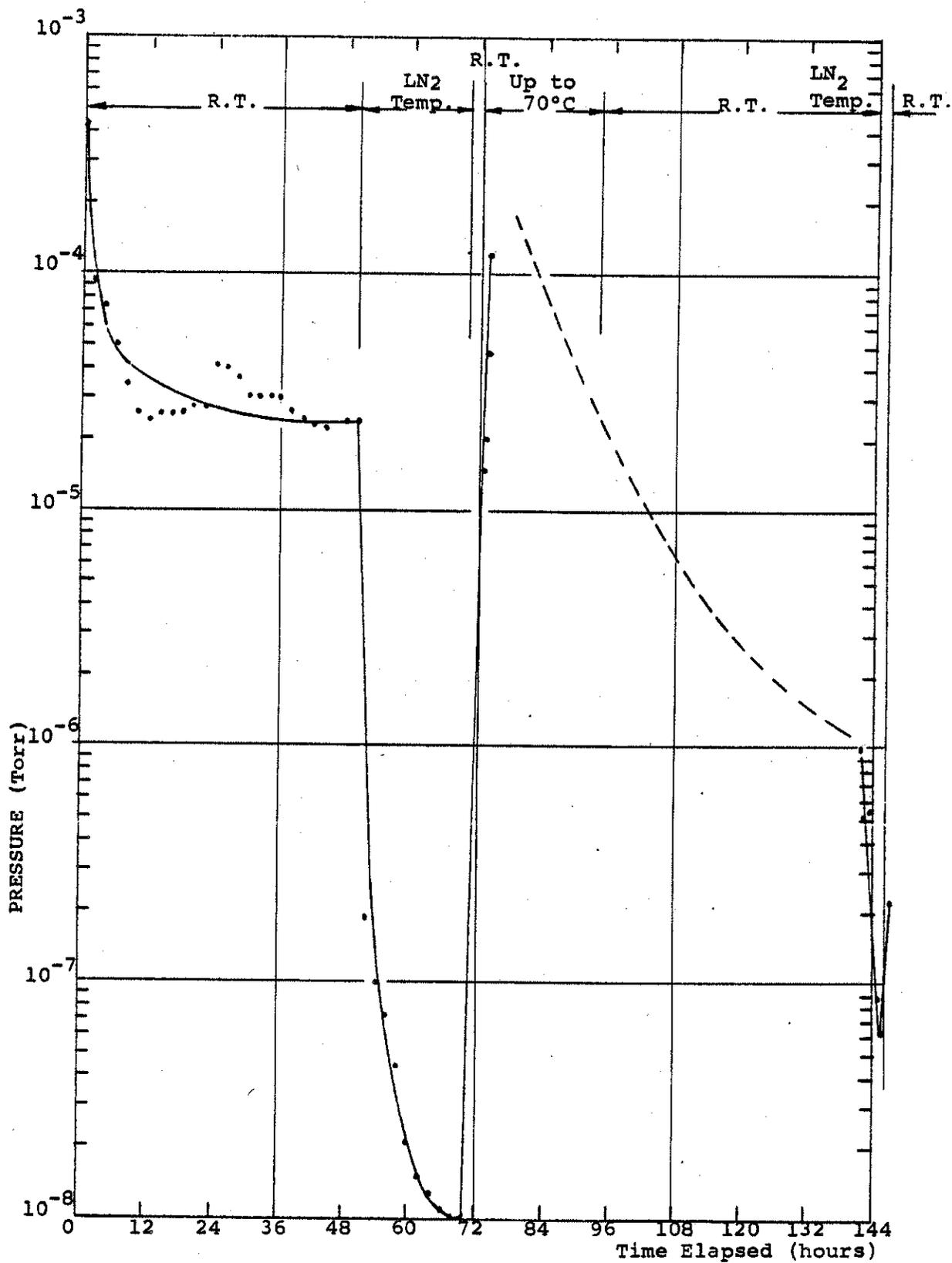


FIGURE 28 - CAT-A-LAC 463-1-8

The effect of the mild bakeout in lowering the outgassing rates suggests that considerable improvements in outgassing of heat sink paints are possible. The high levels of outgassing prior to bakeout indicates the presence of substantial amounts of volatiles trapped in the paint film. The period of cure is less than would normally be involved in normal handling where weeks may elapse between application of the paint and first pumpdown.

4.3.8 Butyl 035

After the Cat-a-lac sample had been removed, it was found that the downstream ion gauge was severely contaminated, especially the glass envelope. Therefore, the gauge was replaced by a new one before the Butyl 035 sample was loaded. This sample had a surface area of 103.4 cm².

The outgassing rate of Butyl 035 dropped within the first 15 hours very slowly and reached an almost constant value of 1×10^{-6} torr liters/sec. cm². (Fig. 29 Table XIII). During the following 10 hours, the outgassing rate dropped about another half an order of magnitude and reached an almost constant value of 3×10^{-7} torr liters/sec. cm². According to the mass spectrometer data hydrogen and water were the main components followed by mass 28 and carbon dioxide. Also mass 32 could be found, however, only at room temperature before warm-up. (Fig. 30; Table XIII A, see Appendix).

The sample was then taken through a heating cycle. The sample temperature was slowly varied to +40°C within 3 hours and was kept at this level for about 10 hours. The highest outgassing rate occurred at the beginning with its highest value of 5.8×10^{-6} torr liters/sec. cm² at +40.5°C. It then dropped towards the end of the heating period to 4×10^{-7} torr liters/sec. cm². After the sample was cooled to room temperature the outgassing rate dropped two orders of magnitude and reached a final value of 4×10^{-9} torr liters/sec. cm².

During the heating cycle, the Omegatron had to be turned off because of the steep pressure increase and was first turned on again when the sample was cooled. At this time water (mass 18) was found to be the main component; mass 28 and mass 44 with almost equal partial pressures were the other ones.

4.3.9 Silastic 1602

This material, used as cable insulation because of its good dielectrical properties, was studied next. Silastic 1602 proved to have very inadequate vacuum properties. The system could be pumped down to about 1×10^{-4} torr as the lowest obtainable pressure. (Fig. 32).

TABLE XIII
Outgassing Rates - Butyl 035

Time Elapsed Hours	Temp. (°C)	P (Torr)	Orifice (l/sec.)	Tl/sec.	Tl/sec.cm ²
0.75	R.T.	7.8x10 ⁻⁵	Full Open	2.34x10 ⁻⁴	2.28 x 10 ⁻⁶
1.0	R.T.	1.0x10 ⁻⁴	3 l/sec.		
1.5	R.T.	2.76x10 ⁻⁴	" " " "	3.0x10 ⁻⁴	2.95 x 10 ⁻⁶
	R.T.	5.28x10 ⁻⁴	0.975	2.69x10 ⁻⁴)	
	R.T.	1.55x10 ⁻³	0.079	2.04x10 ⁻⁴)	1.98x10 ⁻⁴
2.0	R.T.	2.55x10 ⁻⁵	0.975	1.23x10 ⁻⁴)	
	R.T.	4.55x10 ⁻⁵	0.387	2.45x10 ⁻⁴)	
	R.T.	1.23x10 ⁻⁵	0.079	1.76x10 ⁻⁴)	1.73x10 ⁻⁴
3.0	R.T.	2.11x10 ⁻⁵	0.975	9.7 x10 ⁻⁵)	
	R.T.	3.92x10 ⁻⁵	0.387	2.06x10 ⁻⁴)	
	R.T.	1.13x10 ⁻⁵	0.079	1.53x10 ⁻⁴)	1.49x10 ⁻⁴
4.0	R.T.	1.78x10 ⁻⁵	0.975	8.91x10 ⁻⁵)	1.45 x 10 ⁻⁶
	R.T.	3.29x10 ⁻⁵	0.387	1.73x10 ⁻⁴)	
	R.T.	9.91x10 ⁻⁴	0.079	1.27x10 ⁻⁴)	1.027x10 ⁻⁴
5.0	R.T.	6.2 x10 ⁻⁵	3 l/sec.	8.91x10 ⁻⁵)	1 x 10 ⁻⁶
6.0	R.T.	5.4 x10 ⁻⁵	3 l/sec.	1.86x10 ⁻⁴	1.8 x 10 ⁻⁶
7.0	R.T.	4.9 x10 ⁻⁵	3 l/sec.	1.64x10 ⁻⁴	1.59 x 10 ⁻⁶
8.0	R.T.	3.7 x10 ⁻⁵	3 l/sec.	1.47x10 ⁻⁴	1.43 x 10 ⁻⁶
10.0	R.T.	3.2 x10 ⁻⁵	3 l/sec.	1.11x10 ⁻⁴	1.07 x 10 ⁻⁶
11.0	R.T.	2.9 x10 ⁻⁵	3 l/sec.	9.6 x10 ⁻⁵	3.3 x 10 ⁻⁷
12.0	R.T.	3.2 x10 ⁻⁵	3 l/sec.	8.7 x10 ⁻⁵	8.45x 10 ⁻⁷
13.0	R.T.	3.4 x10 ⁻⁵	3 l/sec.	9.6 x10 ⁻⁵	9.3 x 10 ⁻⁷
15.0	R.T.	3.6 x10 ⁻⁵	3 l/sec.	1.02x10 ⁻⁴	9.9 x 10 ⁻⁷
16.0	R.T.	4.1 x10 ⁻⁵	3 l/sec.	1.08x10 ⁻⁴	1.05 x 10 ⁻⁶
17.0	R.T.	5 x 10 ⁻⁵	0.975	1.23x10 ⁻⁴	1.19 x 10 ⁻⁶
	R.T.	1.19x10 ⁻⁴	0.387	4.87x10 ⁻⁵)	
	R.T.	4.72x10 ⁻⁴	0.079	4.60x10 ⁻⁵)	4.4 x 10 ⁻⁵
				3.72x10 ⁻⁵)	4.26 x 10 ⁻⁷
	Dewar under coldtrap				
18.0	R.T.	8 x 10 ⁻⁶	3 l/sec.	2.4 x10 ⁻⁵	2.33 x 10 ⁻⁷
19.0	R.T.	3.6 x10 ⁻⁶	0.975	3.5 x10 ⁻⁵)	
	R.T.	8.7 x10 ⁻⁶	0.387	3.36x10 ⁻⁵)	3.43x10 ⁻⁵
19.25		dp/dt 1.2x10 ⁻⁴	Torr/secx1.5L.=1.8x10 ⁻⁵		3.33 x 10 ⁻⁷
19.50	R.T.	4.3 x10 ⁻⁶	3 l/sec.	1.29x10 ⁻⁵	1.75 x 10 ⁻⁷
20.0	R.T.	3.3 x10 ⁻⁵	0.975	1.29x10 ⁻⁵)	1.25 x 10 ⁻⁷
	R.T.	8.26x10 ⁻⁵	0.387	3.22x10 ⁻⁵)	
	R.T.	3.11x10 ⁻⁴	0.079	3.20x10 ⁻⁵)	2.96x10 ⁻⁵
20.5	R.T.	4.0 x10 ⁻⁶	3 l/sec.	2.45x10 ⁻⁵)	2.82 x 10 ⁻⁷
21.0	R.T.	3.01x10 ⁻⁶	0.975	1.2 x10 ⁻⁵	1.17 x 10 ⁻⁷
	R.T.	7.4 x10 ⁻⁶	0.387	2.93x10 ⁻⁵)	
	R.T.	2.9 x10 ⁻⁴	0.079	2.97x10 ⁻⁵)	2.73x10 ⁻⁵
22.5	R.T.	3.5 x10 ⁻⁵	0.975	2.29x10 ⁻⁵)	2.65 x 10 ⁻⁷
	R.T.	7.2 x10 ⁻⁵	0.387	3.4 x10 ⁻⁵)	
	R.T.	2.5 x10 ⁻⁴	0.079	2.79x10 ⁻⁵)	2.73x10 ⁻⁵
				1.98x10 ⁻⁵)	2.65 x 10 ⁻⁷
	Warming Up Sample				
23.5		5.5 x10 ⁻⁵	0.975	5.36x10 ⁻⁵)	
	30°C	1.48x10 ⁻⁴	0.387	5.72x10 ⁻⁵)	5.54x10 ⁻⁵
24.0		1.63x10 ⁻⁴	0.975	1.59x10 ⁻⁴)	5.37 x 10 ⁻⁷
	34°C	4.02x10 ⁻⁴	0.387	1.54x10 ⁻⁴)	1.57x10 ⁻⁴
					1.52 x 10 ⁻⁶

TABLE XIII - continued

Time Elapsed Hours	Temp. (°C)	P (torr)	Orifice (l/sec.)	Tl/sec.	Tl/sec.cm ²
24.50	35.5°C	6.9 x 10 ⁻⁵	3 l/sec.	2.07x10 ⁻⁴	2.01 x 10 ⁻⁶
24.75	38°C	1.2 x 10 ⁻⁴	3 l/sec.	3.6 x 10 ⁻⁴	3.49 x 10 ⁻⁶
25.00	40°C	1.7 x 10 ⁻⁴	3 l/sec.	5.1 x 10 ⁻⁴	4.95 x 10 ⁻⁶
25.50	40.5°C	2.0 x 10 ⁻⁴	3 l/sec.	6.0 x 10 ⁻⁴	5.82 x 10 ⁻⁶
27.0	40°C	1.19x10 ⁻⁴	3 l/sec.	3.57x10 ⁻⁴	3.46 x 10 ⁻⁶
29.0	40°C	6.2 x 10 ⁻⁵	3 l/sec.	1.86x10 ⁻⁴	1.82 x 10 ⁻⁶
30.0	40°C	5.3 x 10 ⁻⁵	3 l/sec.	1.59x10 ⁻⁴	1.54 x 10 ⁻⁶
31.0	40°C	4.1 x 10 ⁻⁵	3 l/sec.	1.23x10 ⁻⁴	1.19 x 10 ⁻⁶
32.0	40°C	3.4 x 10 ⁻⁵	3 l/sec.	1.06x10 ⁻⁴	1.03 x 10 ⁻⁶
33.0	40°C	2.2 x 10 ⁻⁵	3 l/sec.	6.6 x 10 ⁻⁵	6.4 x 10 ⁻⁷
34.0	40°C	2.2 x 10 ⁻⁵	3 l/sec.	6.6 x 10 ⁻⁵	6.4 x 10 ⁻⁷
35.0	40°C	1.8 x 10 ⁻⁵	3 l/sec.	5.4 x 10 ⁻⁵	5.24 x 10 ⁻⁷
36.0	40°C	1.5 x 10 ⁻⁵	3 l/sec.	4.5 x 10 ⁻⁵	4.37 x 10 ⁻⁷
	(15 hours at 40°C) Turning off sample heating				
40.0	R.T.	2.00x10 ⁻⁶	3 l/sec.	6.00x10 ⁻⁶	5.9 x 10 ⁻⁸
41.0	R.T.	9 x 10 ⁻⁷	0.975	8.77x10 ⁻⁷)	
	R.T.	2 x 10 ⁻⁶	0.387	7.75x10 ⁻⁷)	7.35x10 ⁻⁷
	R.T.	7 x 10 ⁻⁶	0.079	5.53x10 ⁻⁷)	7.25 x 10 ⁻⁹
42.0	R.T.	6.0x10 ⁻⁷	0.975	5.25x10 ⁻⁷)	
	R.T.	1.5x10 ⁻⁶	0.387	5.01x10 ⁻⁷)	5.23x10 ⁻⁷
	R.T.	5.1x10 ⁻⁶	0.079	4.03x10 ⁻⁷)	5.16 x 10 ⁻⁹
43.0	R.T.	6.0x10 ⁻⁷	0.975	5.85x10 ⁻⁷)	
	R.T.	1.4x10 ⁻⁶	0.387	5.42x10 ⁻⁷)	5.31x10 ⁻⁷
	R.T.	5.9x10 ⁻⁶	0.079	4.66x10 ⁻⁷)	5.24 x 10 ⁻⁹
44.25	R.T.	5.0x10 ⁻⁷	0.975	4.87x10 ⁻⁷)	
	R.T.	1.1x10 ⁻⁶	0.387	4.26x10 ⁻⁷)	3.93x10 ⁻⁷
	R.T.	3.3x10 ⁻⁶	0.079	2.58x10 ⁻⁷)	3.88 x 10 ⁻⁹

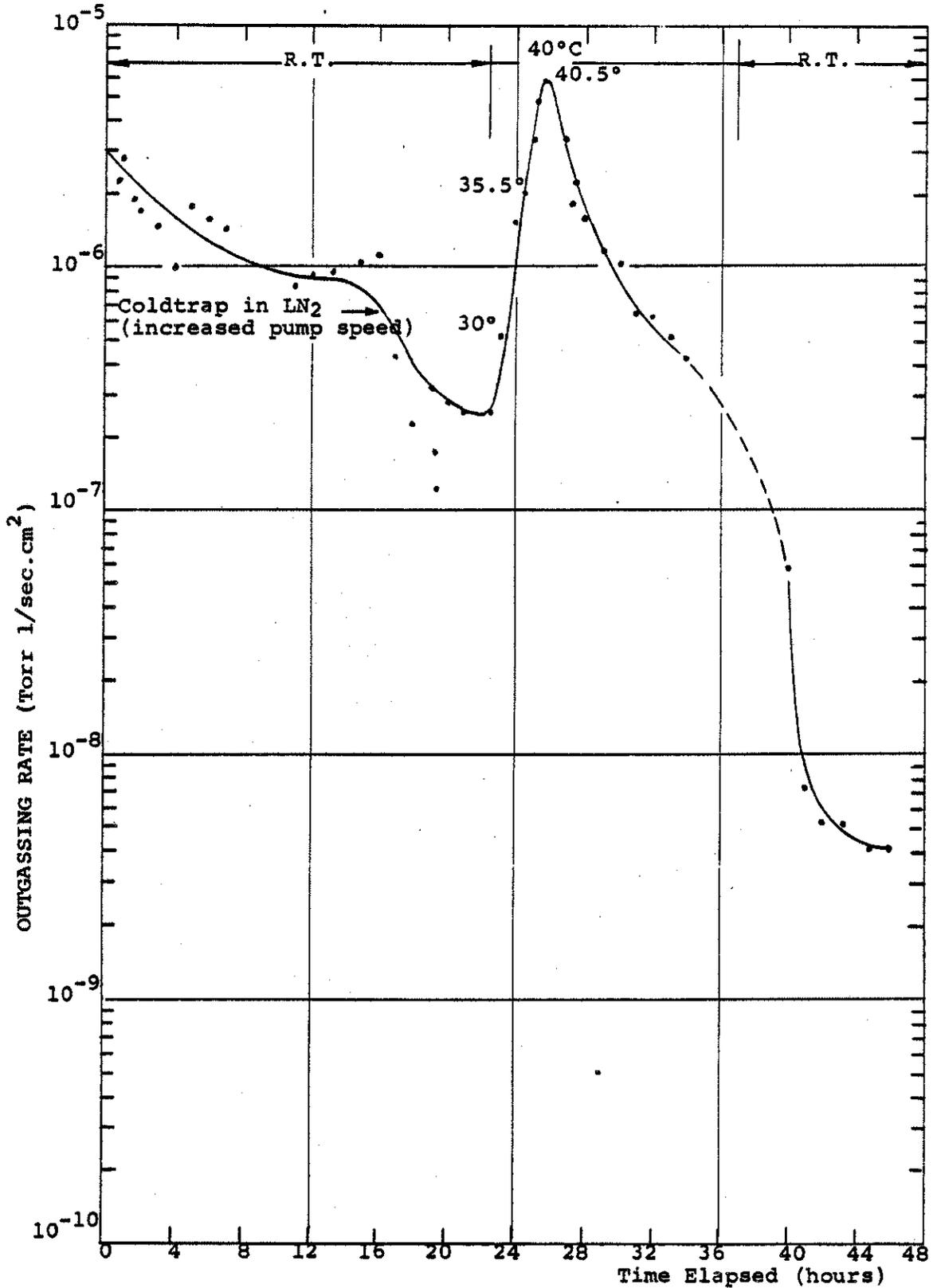
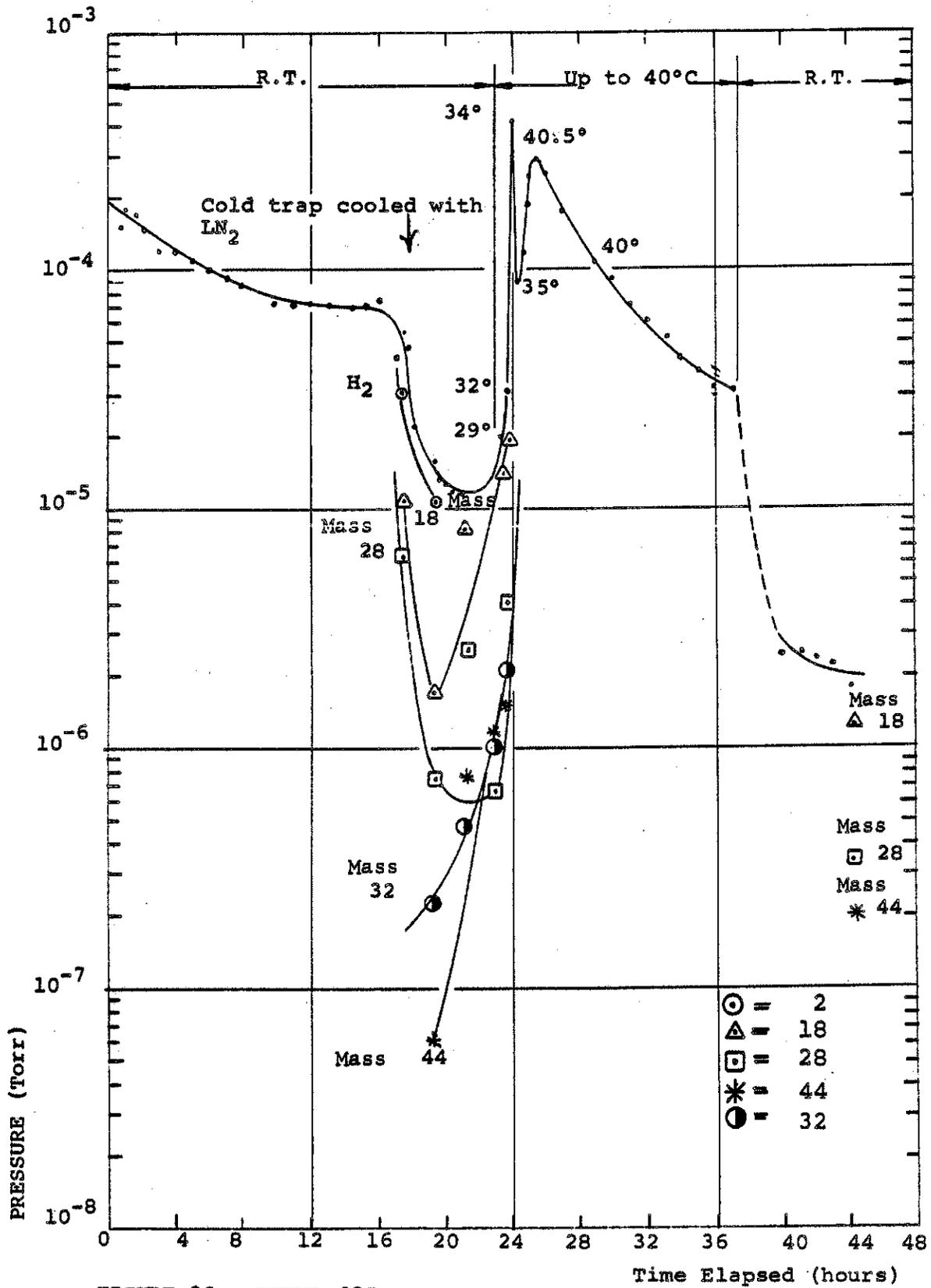


FIGURE 29 - BUTYL 035



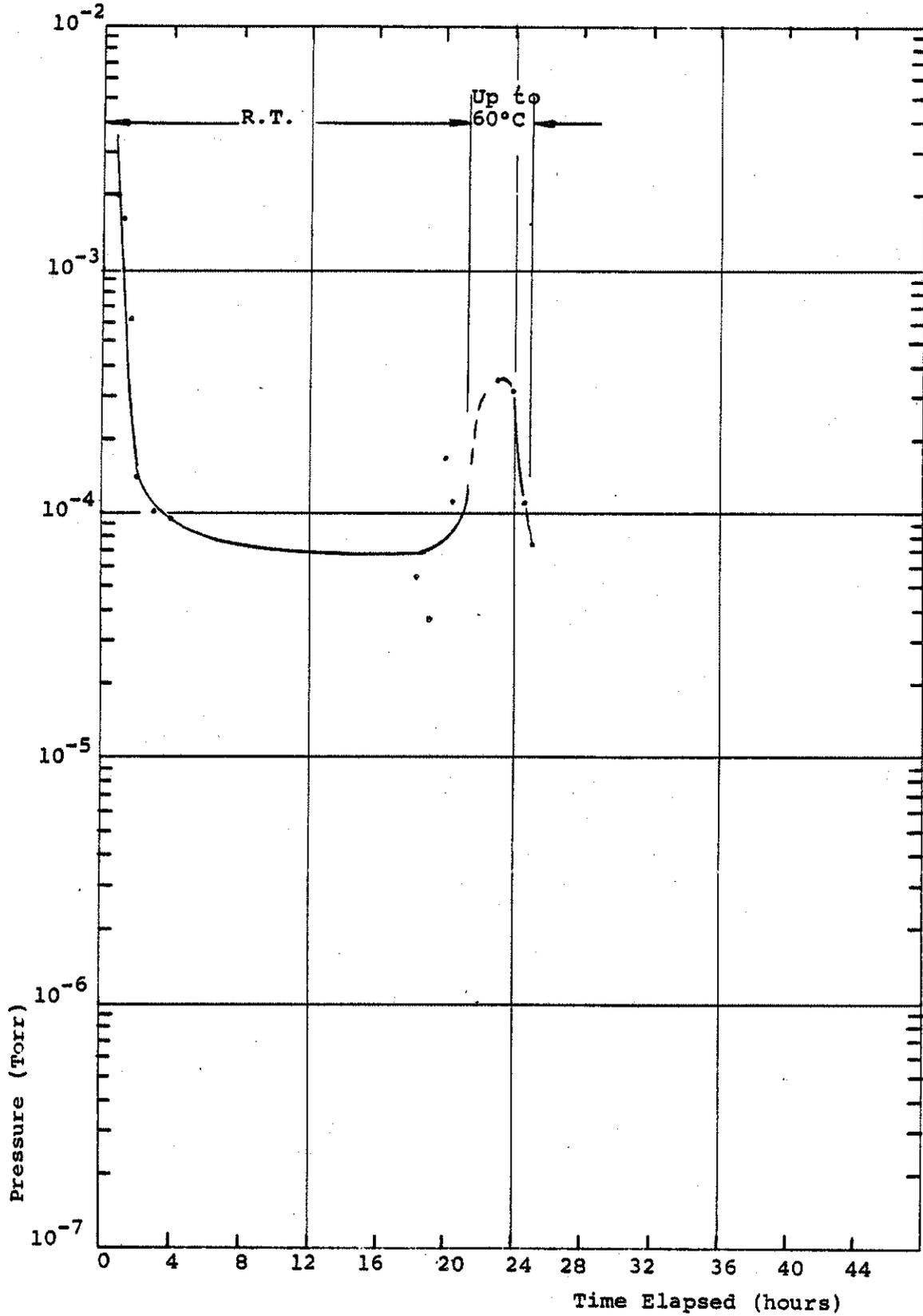


FIGURE 32 - SILASTIC 1602

Ice was observed to completely cover the inner tubing of the coldtrap after approximately one hour of operation. The system was checked several times for leaks; however, was found leaktight. A subsequent mild heat treatment where the sample had been warmed up to 60°C increased the outgassing rate to 3×10^{-6} torr liters/sec. cm² and increased the pressure to about 4×10^{-4} torr. (Fig. 31 Table XIV) These values, however, are doubtful because at this time the entire surface of the glass system was covered with an oily film, which could be seen in even higher concentration at the sample surface.

No mass spectrometer could be turned on during this experiment. After the soft bakeout both gauges ran out of control. The test had to be ended.

After the sample had been removed the system was baked out thoroughly at 400°C and the mass spectrometer section at about 450°C for nearly 24 hours.

The two gauges could not be cleaned again. They had to be removed and replaced by two new ones before the next two samples could be studied. The two new gauges were NRC gauges, Type 553-P.

4.3.10 Silastic 916

Silastic 916 is similar to the previous sample, however, more applicable for mechanical purposes than for electrical ones. The sample tested had a total surface area of 24.75 cm².

Its outgassing rate decreased continuously from 1×10^{-4} to 1×10^{-6} torr liters/sec. cm² within the first twenty hours and stayed at this level. (Fig. 33, Table XV) Mass 28 was the main component of the outgassed material. Peaks corresponding to masses 12 and 14 were also found from which one can conclude that both nitrogen and carbon monoxide were present. Water was the next major component. (Fig. 34; Table XVA, see Appendix)

A heat treatment which brought the sample up to +64°C increased the outgassing rate to 2.5×10^{-5} torr liters/sec. cm² at 60°C. It then decreased rapidly to 5×10^{-7} torr liters/sec. cm² towards the end of the heating period. After the sample was cooled to room temperature a constant value of 3.5×10^{-8} torr liters/sec. cm² was found. At this time water and mass 28 were the main components. Some carbon dioxide was also found.

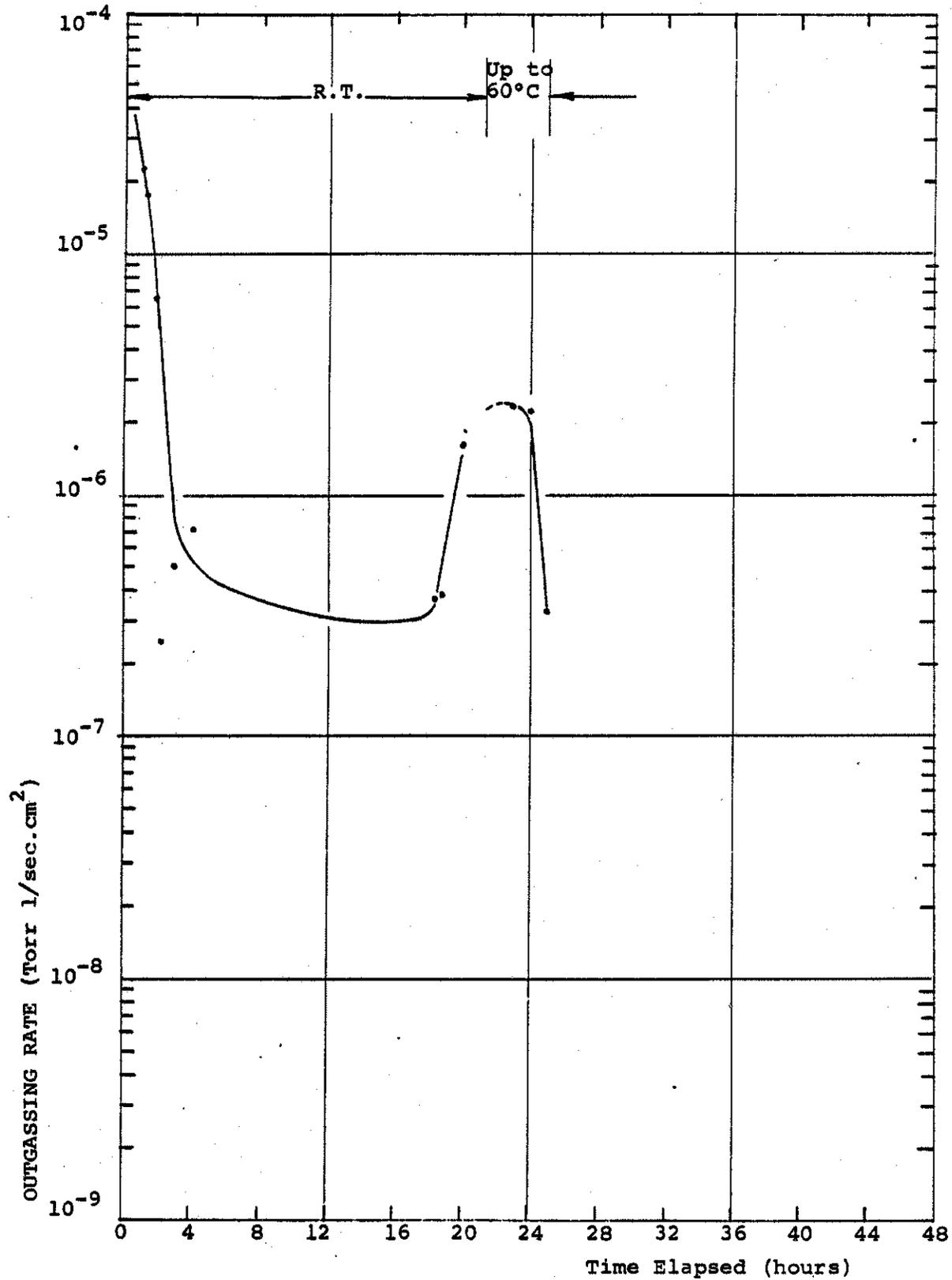


FIGURE 31 - SILASTIC 1602

TABLE XIV
 Outgassing Rates - Silastic #1602 - Area: 242 cm²

Time Elapsed Hours	Temp (°C)	P (Torr)	Orifice (1/sec.)	T1/sec.	T1/sec. cm ²
0.75	R. T.	1.8 x 10 ⁻³	3 1/sec.	5.4 x 10 ⁻³	2.22 x 10 ⁻⁵
1.00	R. T.	1.44 x 10 ⁻³	3 1/sec.	4.32 x 10 ⁻³	1.78 x 10 ⁻⁵
1.50	R. T.	5.26 x 10 ⁻⁴	3 1/sec.	1.57 x 10 ⁻³	6.5 x 10 ⁻⁶
2.00	R. T.	2.0 x 10 ⁻⁵	3 1/sec.	6.0 x 10 ⁻⁵	2.47 x 10 ⁻⁷
3.00	R. T.	4.0 x 10 ⁻⁵	3 1/sec.	1.2 x 10 ⁻⁴	4.9 x 10 ⁻⁷
4.00	R. T.	6.0 x 10 ⁻⁵	3 1/sec.	1.8 x 10 ⁻⁴	7.4 x 10 ⁻⁷
18.5	R. T.	3.0 x 10 ⁻⁵	3 1/sec.	9.0 x 10 ⁻⁵	3.72 x 10 ⁻⁷
19.0	R. T.	2.98 x 10 ⁻⁵	3 1/sec.	8.94 x 10 ⁻⁵	3.70 x 10 ⁻⁷
20.0	R. T.	1.33 x 10 ⁻⁴	3 1/sec.	3.99 x 10 ⁻⁴	1.64 x 10 ⁻⁶
20.25	R. T.	7.8 x 10 ⁻⁵	3 1/sec.	2.34 x 10 ⁻⁴	9.6 x 10 ⁻⁷
Start Warming up Silastic 1602 Sample					
23.0	+55°C	1.9 x 10 ⁻⁴	3 1/sec.	5.7 x 10 ⁻⁴	2.34 x 10 ⁻⁶
24.0	+56°C	1.8 x 10 ⁻⁴	3 1/sec.	5.4 x 10 ⁻⁴	2.22 x 10 ⁻⁶
24.5	+61°C	2.7 x 10 ⁻⁵	3 1/sec.	8.1 x 10 ⁻⁵	3.34 x 10 ⁻⁷
Turn off Heat.					
End of Test					

TABLE XV
Outgassing Rates - Silastic 916

Time Elapsed Hours	Temp. (°C)	P (Torr)	Orifice l/sec.	Torr l/sec.	Tl/sec. cm ²
0.25	R.T.	6.9 x 10 ⁻⁴	3 l/sec.	2.67 x 10 ⁻³	1.08 x 10 ⁻⁴
0.50	R.T.	4.6 x 10 ⁻⁴	3 l/sec.	1.38 x 10 ⁻³	5.6 x 10 ⁻⁵
1.00	R.T.	2.97 x 10 ⁻⁴	3 l/sec.	8.91 x 10 ⁻⁴	3.6 x 10 ⁻⁵
2.00	R.T.	1.43 x 10 ⁻⁴	3 l/sec.	4.28 x 10 ⁻⁴	1.73 x 10 ⁻⁵
2.50	R.T.	9.37 x 10 ⁻⁵	3 l/sec.	2.82 x 10 ⁻⁴	1.16 x 10 ⁻⁵
3.5	R.T.	7.37 x 10 ⁻⁵	3 l/sec.	2.21 x 10 ⁻⁴	8.96 x 10 ⁻⁶
	R.T.	1.84 x 10 ⁻⁴	0.975	1.795 x 10 ⁻⁴	
	R.T.	4.04 x 10 ⁻⁴	0.387	1.53 x 10 ⁻⁴	5.53 x 10 ⁻⁶
	R.T.	1.0 x 10 ⁻³	0.079	7.9 x 10 ⁻⁵	
4.00	R.T.	6.16 x 10 ⁻⁵	3 l/sec.	1.84 x 10 ⁻⁴	7.46 x 10 ⁻⁶
4.50	R.T.	4.64 x 10 ⁻⁵	3 l/sec.	1.4 x 10 ⁻⁴	5.67 x 10 ⁻⁶
5.5	R.T.	3.53 x 10 ⁻⁵	3 l/sec.	1.06 x 10 ⁻⁴	4.28 x 10 ⁻⁶
6.0	R.T.	3.24 x 10 ⁻⁵	3 l/sec.	9.6 x 10 ⁻⁵	3.88 x 10 ⁻⁶
6.5	R.T.	2.8 x 10 ⁻⁵	3 l/sec.	8.4 x 10 ⁻⁵	3.41 x 10 ⁻⁶
7.5	R.T.	2.3 x 10 ⁻⁵	3 l/sec.	6.9 x 10 ⁻⁵	2.79 x 10 ⁻⁶
9.5	R.T.	1.73 x 10 ⁻⁵	3 l/sec.	5.18 x 10 ⁻⁵	2.1 x 10 ⁻⁶
11.5	R.T.	1.35 x 10 ⁻⁵	3 l/sec.	4.05 x 10 ⁻⁵	1.63 x 10 ⁻⁶
13.5	R.T.	1.07 x 10 ⁻⁵	3 l/sec.	3.2 x 10 ⁻⁵	1.29 x 10 ⁻⁶
15.5	R.T.	9.8 x 10 ⁻⁶	3 l/sec.	2.94 x 10 ⁻⁵	1.19 x 10 ⁻⁶
17.5	R.T.	8.0 x 10 ⁻⁶	3 l/sec.	2.4 x 10 ⁻⁵	9.7 x 10 ⁻⁷
19.5	R.T.	7.0 x 10 ⁻⁶	3 l/sec.	2.1 x 10 ⁻⁵	8.5 x 10 ⁻⁷
22.0	R.T.	7.88 x 10 ⁻⁶	3 l/sec.	2.36 x 10 ⁻⁵	9.56 x 10 ⁻⁷
	R.T.	2.0 x 10 ⁻⁵	0.975	1.95 x 10 ⁻⁵	
	R.T.	3.99 x 10 ⁻⁵	0.387	1.55 x 10 ⁻⁵	6.44 x 10 ⁻⁷
	R.T.	1.4 x 10 ⁻⁴	0.079	1.19 x 10 ⁻⁵	
23.0	R.T.	7.67 x 10 ⁻⁶	3 l/sec.	2.3 x 10 ⁻⁵	9.3 x 10 ⁻⁷
	dp/dt = 1.10 x 10 ⁻⁵ torr/sec. x 1.5 L =				
23.5	R.T.	7.58 x 10 ⁻⁶	3 l/sec.	1.65 x 10 ⁻⁵	6.7 x 10 ⁻⁷
	R.T.	1.9 x 10 ⁻⁵	0.975	2.274 x 10 ⁻⁵	9.2 x 10 ⁻⁷
	R.T.	3.69 x 10 ⁻⁵	0.387	1.85 x 10 ⁻⁵	6 x 10 ⁻⁷
	R.T.	1.49 x 10 ⁻⁵	0.079	1.43 x 10 ⁻⁵	8.2 x 10 ⁻⁷
24.0	R.T.	6.77 x 10 ⁻⁶	3 l/sec.	1.18 x 10 ⁻⁵	6 x 10 ⁻⁷
	Start Warming up sample.			2.03 x 10 ⁻⁵	
24.5	+33°C	1.09 x 10 ⁻⁵	3 l/sec.	3.27 x 10 ⁻⁵	1.32 x 10 ⁻⁶
24.75	+35°C	2.0 x 10 ⁻⁵	3 l/sec.	6.0 x 10 ⁻⁵	2.42 x 10 ⁻⁶
25.0	+40°C	9.6 x 10 ⁻⁵	0.975	9.35 x 10 ⁻⁵	
		3.83 x 10 ⁻⁴	0.387	1.25 x 10 ⁻⁴	4.5 x 10 ⁻⁶

TABLE XV - continued

Time Elapsed Hours	Temp. (°C)	P (Torr)	Orifice 1/sec.	Torr 1/sec.	Tl/sec.cm ²
25.1	+50°C	1.87 x 10 ⁻⁴	3 1/sec.	5.61 x 10 ⁻⁴	2.2 x 10 ⁻⁵
25.5	+60°C	2.15 x 10 ⁻⁴	3 1/sec.	6.45 x 10 ⁻⁴	2.6 x 10 ⁻⁵
26.0	+61°C	1.59 x 10 ⁻⁴	3 1/sec.	4.77 x 10 ⁻⁴	1.92 x 10 ⁻⁵
26.5	+61°C	1.21 x 10 ⁻⁴	3 1/sec.	3.63 x 10 ⁻⁴	1.47 x 10 ⁻⁵
		2.67 x 10 ⁻⁴	0.975	2.54 x 10 ⁻⁴	8.8 x 10 ⁻⁶
		4.41 x 10 ⁻⁴	0.387	1.71 x 10 ⁻⁴	
27.5	+66°C	1.14 x 10 ⁻⁴	3	3.43 x 10 ⁻⁴	1.38 x 10 ⁻⁵
28.0	+64°C	9.54 x 10 ⁻⁵	3	2.86 x 10 ⁻⁴	1.15 x 10 ⁻⁵
28.5	+63°C	6.2 x 10 ⁻⁵	3	1.86 x 10 ⁻⁴	7.5 x 10 ⁻⁶
29.5	+64.5°C	4.22 x 10 ⁻⁵	3	1.27 x 10 ⁻⁴	5.14 x 10 ⁻⁶
30.	+64°C	2.84 x 10 ⁻⁵	3	8.52 x 10 ⁻⁵	3.45 x 10 ⁻⁶
30.5	+64°C	4.66 x 10 ⁻⁶	3	1.398 x 10 ⁻⁵	5.62 x 10 ⁻⁷
		Turn off power, sample cools down			
30.5	44°C	2.5 x 10 ⁻⁶	3	7.5 x 10 ⁻⁶	3.03 x 10 ⁻⁷
	37°C	3.55 x 10 ⁻⁶	0.975	3.46 x 10 ⁻⁶	1.4 x 10 ⁻⁷
	31°C	3.67 x 10 ⁻⁶	0.387	1.44 x 10 ⁻⁶	5.74 x 10 ⁻⁷
31.0	R.T.	5.0 x 10 ⁻⁷	3	1.5 x 10 ⁻⁶	6.06 x 10 ⁻⁸
31.25	R.T.	3.1 x 10 ⁻⁷	3	9.3 x 10 ⁻⁷	3.77 x 10 ⁻⁸
33.5	R.T.	2.1 x 10 ⁻⁷	3	6.3 x 10 ⁻⁷	2.54 x 10 ⁻⁸
35.5	R.T.	2.6 x 10 ⁻⁷	3	7.8 x 10 ⁻⁷	3.15 x 10 ⁻⁸
39.5	R.T.	3.26 x 10 ⁻⁷	3	9.78 x 10 ⁻⁷	3.95 x 10 ⁻⁸
41.5	R.T.	3.15 x 10 ⁻⁷	3	9.45 x 10 ⁻⁷	3.82 x 10 ⁻⁸
43.5	R.T.	2.99 x 10 ⁻⁷	3	8.97 x 10 ⁻⁷	3.62 x 10 ⁻⁸
45.5	R.T.	2.92 x 10 ⁻⁷	3	8.76 x 10 ⁻⁷	3.54 x 10 ⁻⁸
46.5	R.T.	2.85 x 10 ⁻⁷	3	8.55 x 10 ⁻⁷	3.45 x 10 ⁻⁸
		3.88 x 10 ⁻⁷	0.975	3.78 x 10 ⁻⁷	
		5.6 x 10 ⁻⁷	0.387	2.17 x 10 ⁻⁷	9.1 x 10 ⁻⁹
		1.05 x 10 ⁻⁶	0.079	8.3 x 10 ⁻⁸	
	End of Test			2.26 x 10 ⁻⁷	

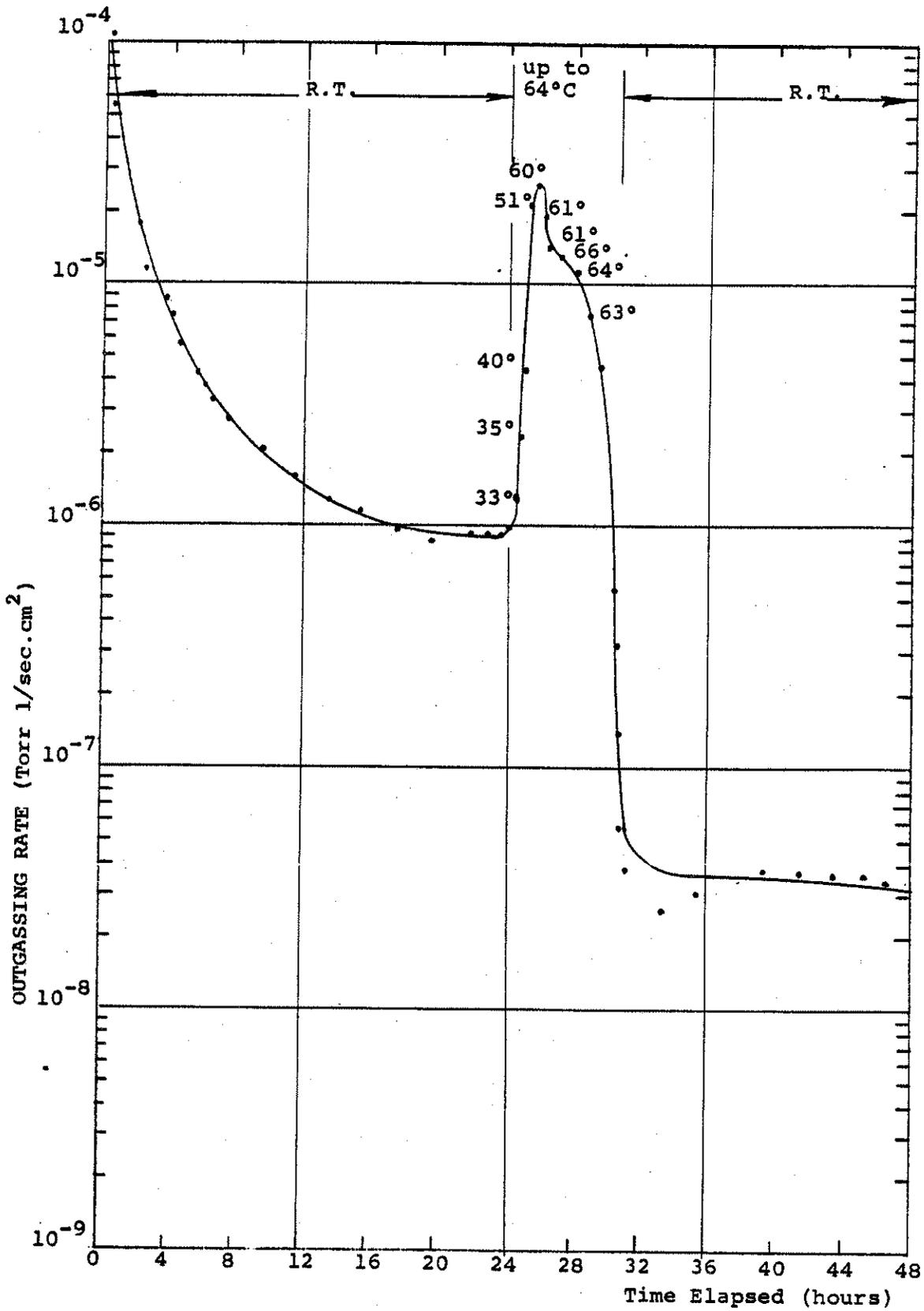


FIGURE 33 - SILASTIC 916

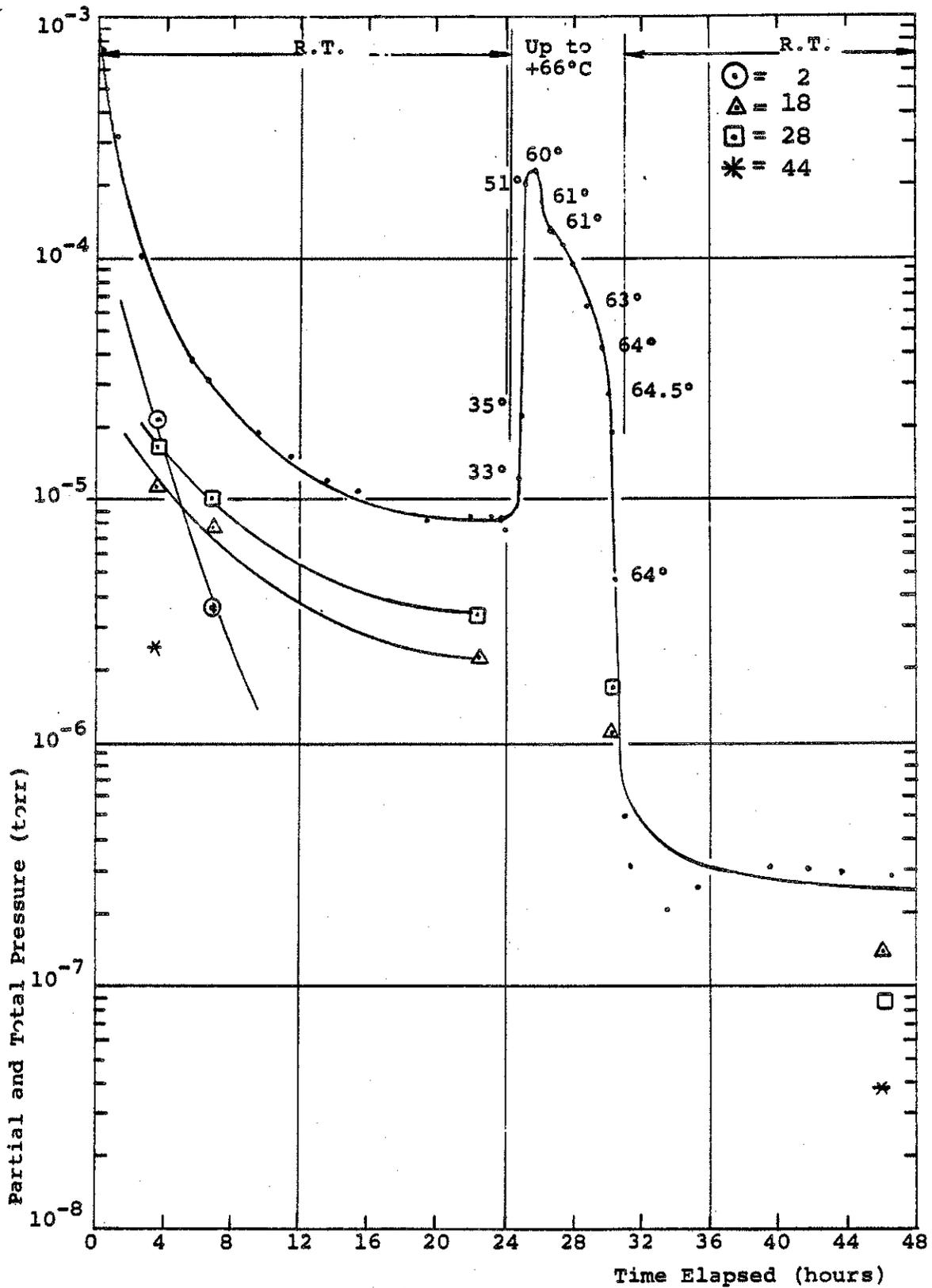


FIGURE 34 - SILASTIC 916

4.3.11 Vibrathane 5004

This sample, representing the polyurethane group, was the last one being studied under this program. With a total outgassing area of 25 cm² the outgassing rate decreased from approximately 1 x 10⁻⁴ to 1 x 10⁻⁷ torr liters/sec. cm² at room temperature within 22 hours and reached a constant value at this level. (Fig. 35 Table XVI). A heating cycle which warmed the sample up to 47°C increased the outgassing rate to 4 x 10⁻⁶ torr liters/sec.cm². It then dropped to 1 x 10⁻⁶ torr liters/sec. cm² at the end of the heating period. When the sample was cooled to room temperature an average value of 4 x 10⁻⁸ torr liters/sec. cm² was the lowest obtainable outgassing rate for this material.

The mass spectrometer, turned on before the heating cycle, indicated main peaks corresponding to masses higher than 44 followed by hydrogen, mass 28 and carbon dioxide. (Fig. 36; Table XVIA, see Appendix). During the next scan new peaks appeared belonging to masses between 18 and 28. After the heating cycle the mass spectrometer seemed to have lost its previous sensitivity; only mass 28 and masses higher than 44 could be traced.

4.4 CONCLUSIONS AND RECOMMENDATIONS

From the experiments carried out with ten different samples a general survey is given of the behavior of a few groups of materials under various vacuum and temperature conditions. Generally it can be said that a mild bakeout up to roughly +100°C decreased the outgassing rate in most cases at least an order of magnitude.

More intense investigations with specific materials will be necessary in order to find their optimum outgassing properties. The experiment with Cat-a-lac paint, e.g. showed that it cannot be said that this material has a certain outgassing rate after so many hours of exposure to vacuum. Pre-history and pre-treatment of the samples are indispensable for this kind of information.

The study of the aluminum alloy showed that this is an adequate material for vacuum chambers, in cases where either no bakeout above +150°C to +200°C are allowed or possible. It would be interesting to investigate samples previously annealed up to temperatures mentioned above under controlled atmospheres with regard to the lowest obtainable final pressure at room temperature.

The ceramic material Al-Si-Mag 614 which was used in the dense modification turned out to be a very good material as soon as the surface contaminations were removed by prebaking.

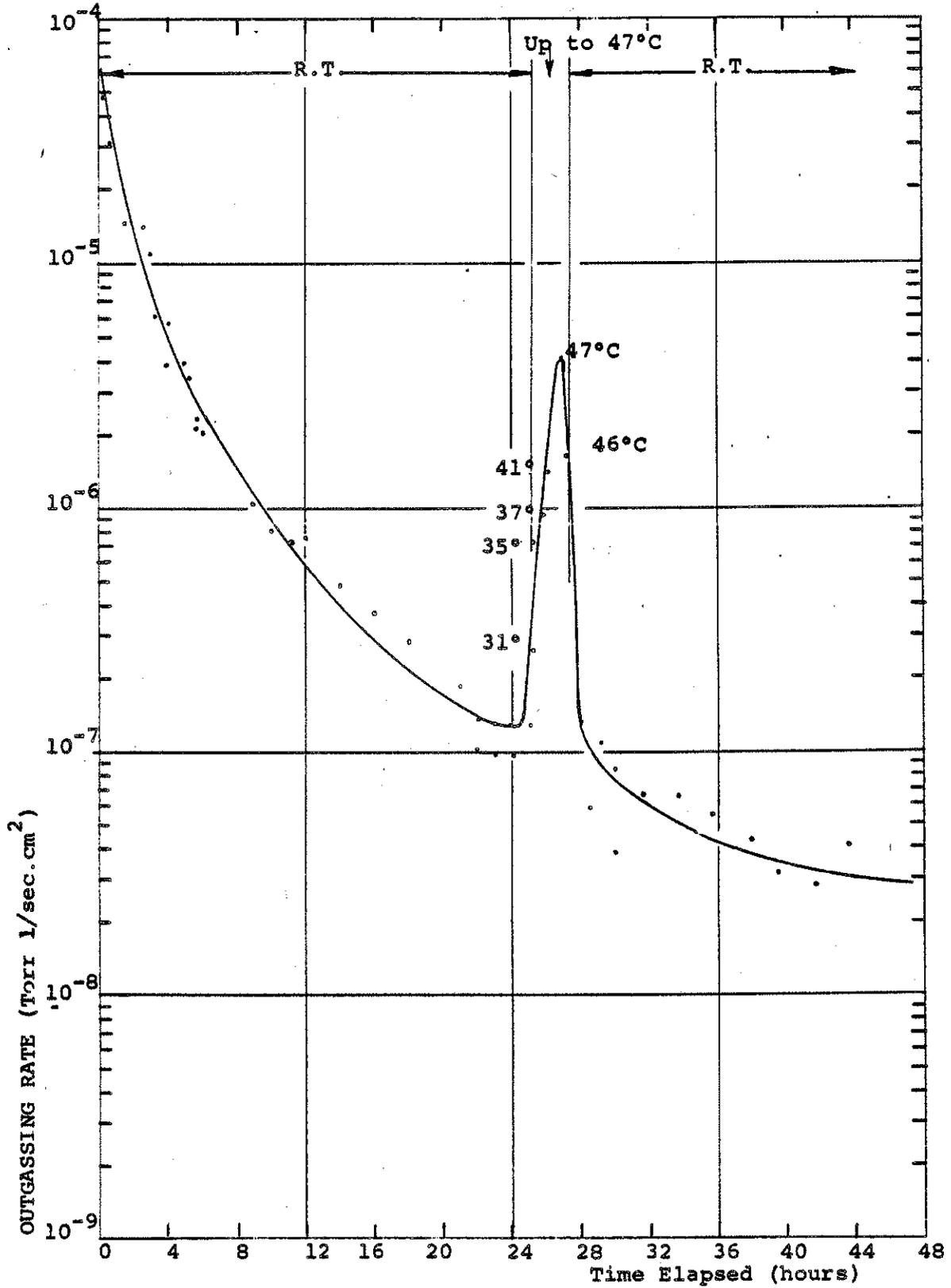


FIGURE 35 - VIBRATHANE 5004

TABLE XVI

Outgassing Rates - Vibrathane 5004

Time Elapsed Hours	Temp (°C)	P (Torr)	Orifice 1/sec.	Outgassing Rate Torr 1/sec.	Tl/sec.cm ²
0.15	R.T.	4.1×10^{-4}	3.0	1.23×10^{-3}	4.92×10^{-5}
0.75	R.T.	2.65×10^{-4}	3.0	7.95×10^{-4}	3.18×10^{-5}
1.75	R.T.	1.97×10^{-4}	3.0	3.96×10^{-4}	1.48×10^{-5}
2.75	R.T.	1.32×10^{-4}	3.0	3.54×10^{-4}	1.42×10^{-5}
3.00	R.T.	1.18×10^{-4}	3.0	3.54×10^{-4}	
	R.T.	2.74×10^{-4}	0.975	2.67×10^{-4}	2.68×10^{-4} 1.07×10^{-5}
	R.T.	4.74×10^{-4}	0.387	1.84×10^{-4}	
3.50	dp/dt = 1×10^{-4} T/sec.		$1.5L = 1.5 \times 10^{-4}$		$6. \times 10^{-6}$
	dp/dt = 6.5×10^{-5} T/sec.		$1.5L = 9.75 \times 10^{-5}$		3.9×10^{-6}
4.0	R.T.	2.1×10^{-4}	0.975	2.05×10^{-4}	
	R.T.	3.62×10^{-4}	0.387	1.39×10^{-4}	1.38×10^{-4} 5.8×10^{-6}
	R.T.	8.31×10^{-4}	0.079	6.6×10^{-5}	
4.5	R.T.	3.58×10^{-5}	3.0	1.07×10^{-4}	4×10^{-6}
5.0	R.T.	1.06×10^{-4}	0.975	5.94×10^{-4}	
	R.T.	2.26×10^{-4}	0.387	8.75×10^{-5}	8.43×10^{-5} 2.2×10^{-6}
	R.T.	6.64×10^{-5}	0.079	5.75×10^{-5}	
5.5	R.T.	1.98×10^{-5}	3.0	5.94×10^{-5}	2.38×10^{-6}
	R.T.	7.48×10^{-5}	0.975	7.30×10^{-5}	
	R.T.	1.48×10^{-4}	0.387	5.72×10^{-5}	5.49×10^{-5} 2.2×10^{-6}
	R.T.	4.38×10^{-4}	0.079	3.46×10^{-5}	
6.0	R.T.	1.7×10^{-5}	3 1/sec.	5.10×10^{-5}	2.04×10^{-6}
9.0	R.T.	8.7×10^{-6}	3.0	2.6×10^{-5}	1.04×10^{-6}
10.0	R.T.	7.5×10^{-6}	3.0	2.02×10^{-5}	8.06×10^{-7}
11.0	R.T.	6.14×10^{-6}	3.0	1.84×10^{-5}	7.36×10^{-7}
12.0	R.T.	6.43×10^{-6}	3.0	1.93×10^{-5}	7.72×10^{-7}
14.0	R.T.	4.08×10^{-6}	3.0	1.224×10^{-5}	4.9×10^{-7}
16.0	R.T.	3.08×10^{-6}	3.0	9.24×10^{-6}	3.70×10^{-7}
18.0	R.T.	2.36×10^{-6}	3.0	7.08×10^{-6}	2.83×10^{-7}
21.0	R.T.	1.53×10^{-6}	3.0	4.59×10^{-6}	1.84×10^{-7}
22.0	R.T.	1.18×10^{-6}	3.0	3.54×10^{-6}	1.32×10^{-7}
	R.T.	3.28×10^{-6}	0.975	3.20×10^{-6}	
	R.T.	6.58×10^{-6}	0.387	2.55×10^{-6}	2.49×10^{-6} 1.00×10^{-7}
	R.T.	2.18×10^{-5}	0.079	1.73×10^{-6}	
23.0	R.T.	1.09×10^{-6}	3.0	3.27×10^{-6}	1.30×10^{-7}
	R.T.	3.19×10^{-6}	0.975	3.11×10^{-6}	
	R.T.	6.08×10^{-6}	0.387	2.26×10^{-6}	2.42×10^{-6} 9.67×10^{-8}
	R.T.	2.28×10^{-6}	0.079	1.80×10^{-6}	
24.0	R.T.	1.09×10^{-6}	3	3.27×10^{-6}	1.30×10^{-7}
	R.T.	3.18×10^{-6}	0.975	3.10×10^{-6}	
	R.T.	6.27×10^{-6}	0.387	2.42×10^{-6}	2.42×10^{-6} 9.60×10^{-8}
	R.T.	2.14×10^{-5}	0.079	1.69×10^{-6}	
25.0	R.T.	1.09×10^{-6}	3.0	3.27×10^{-6}	1.30×10^{-7}

Start warm up sample

TABLE XVI - continued

Time Elapsed Hours	Temp. (°C)	P (Torr)	Orifice 1/sec.	Outgassing Rate Torr 1/sec.	Outgassing Rate Tl/sec.cm ²
25.25	+31°C	2.19x10 ⁻⁶	3 1/sec.	6.57x10 ⁻⁶	2.66 x 10 ⁻⁷
		7.7 x10 ⁻⁶	0.975	7.54x10 ⁻⁶)	
	+33°C	1.67x10 ⁻⁵	0.387	6.45x10 ⁻⁶)	6.6x10 ⁻⁶ 2.64 x 10 ⁻⁷
		7.34x10 ⁻⁵	0.079	5.80x10 ⁻⁶)	
25.60	+35°C	6.06x10 ⁻⁶	3.0	1.818x10 ⁻⁵	7.28 x 10 ⁻⁷
25.75	+37°C	7.91x10 ⁻⁶	3.0	2.37x10 ⁻⁵	9.50 x 10 ⁻⁷
26.25	+41°C	1.19x10 ⁻⁵	3.0	3.57x10 ⁻⁵	1.43 x 10 ⁻⁶
26.75	+47°C	3.4 x10 ⁻⁵	3.0	1.02x10 ⁻⁴	4.08 x 10 ⁻⁶
27.0	+46°C	2.74x10 ⁻⁵	3.0	8.12x10 ⁻⁵	1.65 x 10 ⁻⁶
	End of warming up sample				
28.0	R.T.	1.12x10 ⁻⁶	3.0	3.36x10 ⁻⁶	1.34 x 10 ⁻⁷
28.25	R.T.	2.32x10 ⁻⁶	0.975	2.26x10 ⁻⁶)	
	R.T.	3.71x10 ⁻⁶	0.387	1.44x10 ⁻⁶)	1.49x10 ⁻⁶ 5.96 x 10 ⁻⁸
	R.T.	9.9 x10 ⁻⁶	0.079	7.82x10 ⁻⁷)	
29.0	R.T.	9.32x10 ⁻⁷	3.0	2.8 x10 ⁻⁶	1.12 x 10 ⁻⁷
29.75	R.T.	7.18x10 ⁻⁷	3.0	2.15x10 ⁻⁶	8.60 x 10 ⁻⁸
	R.T.	1.44x10 ⁻⁶	0.975	1.41x10 ⁻⁶)	
	R.T.	2.34x10 ⁻⁶	0.387	9.05x10 ⁻⁷)	9.46x10 ⁻⁷ 3.79 x 10 ⁻⁸
	R.T.	6.63x10 ⁻⁶	0.079	5.22x10 ⁻⁷)	
31.75	R.T.	5.85x10 ⁻⁷	3.0	1.76x10 ⁻⁶	6.79 x 10 ⁻⁸
33.75	R.T.	5.66x10 ⁻⁷	3.0	1.70x10 ⁻⁶	6.79 x 10 ⁻⁸
35.75	R.T.	4.62x10 ⁻⁷	3.0	1.39x10 ⁻⁶	5.54 x 10 ⁻⁸
37.75	R.T.	3.71x10 ⁻⁷	3.0	1.11x10 ⁻⁶	4.45 x 10 ⁻⁸
39.75	R.T.	2.78x10 ⁻⁷	3.0	8.34x10 ⁻⁷	3.34 x 10 ⁻⁸
41.75	R.T.	2.41x10 ⁻⁷	3.0	7.23x10 ⁻⁷	2.89 x 10 ⁻⁸
43.75	R.T.	3.52x10 ⁻⁷	3.0	1.05x10 ⁻⁷	4.21 x 10 ⁻⁸
	End of Test				

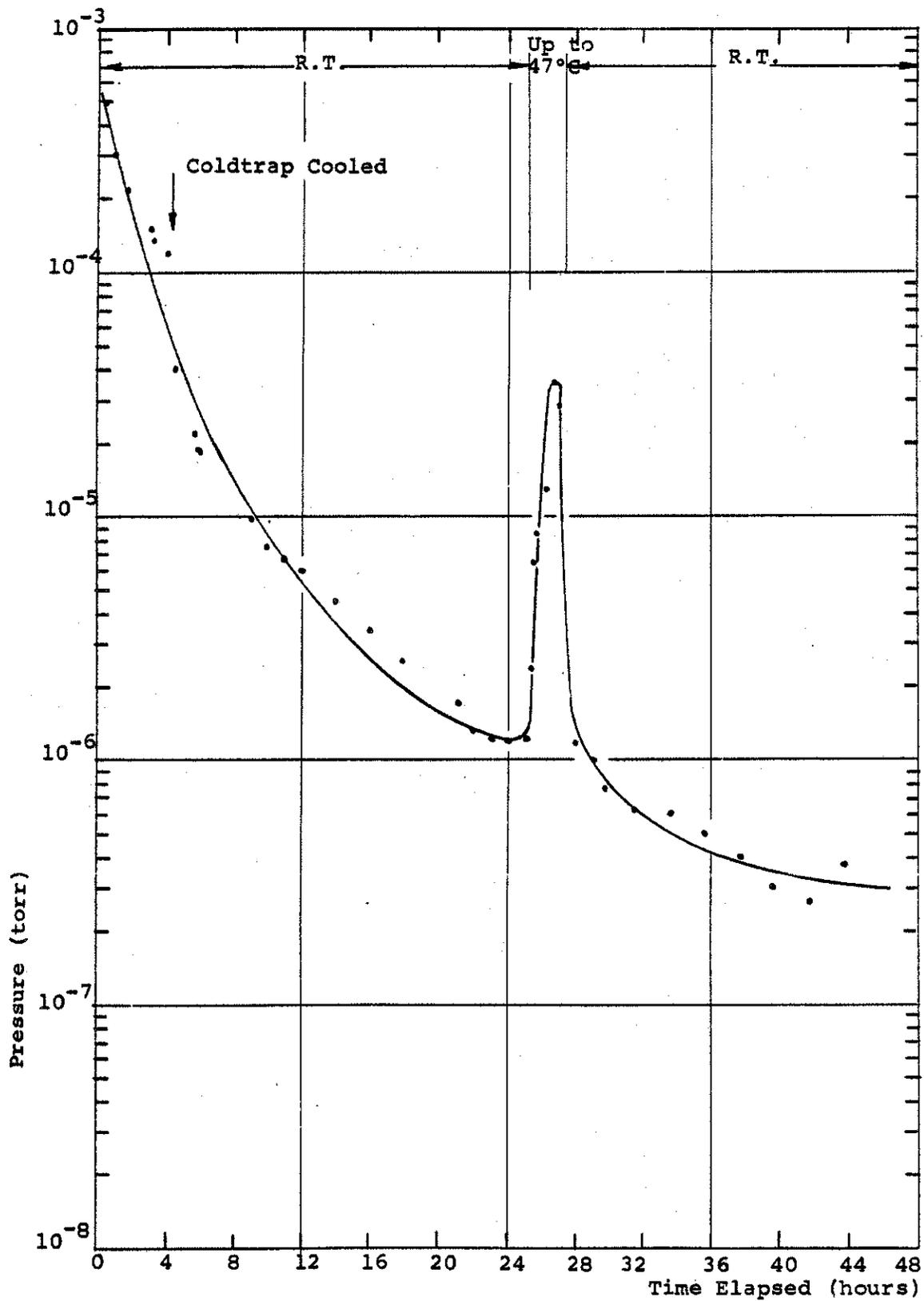


FIGURE 36 - VIBRATHANE 5004

Samples prebaked under controlled dry atmosphere should be further investigated; also samples with different surface finishes should be compared.

The Teflon No. 7 sample, as mentioned previously seemed to be a very porous material, which is therefore able to store all kinds of gases for a long time and which will even swell during exposure to humid atmosphere. As shown in the experiment a mild bakeout under a controlled atmosphere will improve its vacuum usefulness considerably, especially in regard to ultimate pressures. As mentioned by the manufacturer, Teflon No. 7, in its final stage of processing is heated up to about +250°C. However, in molding, extruding or swaging the material in the desired form of sheets, tubes or rods, stearine compounds are used as lubricants. This might explain the multiplicity of peaks recorded after +100°C bakeout.

It would be worthwhile also to investigate Teflon No. 6 and Teflon No. 100 which represent together with Teflon No. 7 the three major varieties of about 30 Teflon species.

Mycalex 400, representing the group of ceramics, showed excellent properties for construction elements in vacuum chambers. The high water content which was still found after +100°C bakeout could certainly be reduced by heat treatments at higher temperatures or preheating the materials under controlled atmosphere. Mycalex 400 can be heated up to 340°C without changing its electrical and mechanical properties.

The Ceramic-Coated Copper Wire was similar. As previously mentioned, the large amounts of hydrogen could only be explained by further experiments with the uncoated wire and with the ceramic coating material alone.

The outgassing characteristics of the Mycalex were similar to those of the ceramic coated copper wire, and can be recommended as construction materials.

Cat-a-lac flat black (type 463-1-8) was selected to represent the group of paints. As mentioned above, the values for the outgassing rates are only made meaningful by indicating pre-history and pre-treatment. Preconditioning of the samples by 96 hours of drying at one atmosphere at room temperature is insufficient. Either more extended drying time or a prebake under controlled atmosphere would certainly lower the initial barrier of high total pressure in the system and high outgassing rate. It certainly would also less affect pressure gauges and mass spectrometer. It should be mentioned that LeVantine [45] obtained the same outgassing rates by a different method. His sample had a preconditioning time of only 48 hours.

One of the elastomers was Butyl 035. The initial outgassing rate determined by hydrogen and water could be improved after the sample was heat treated. The slight increase of temperature resulted in a considerable decrease of the outgassing rate. Since the rubber can be permanently exposed to dry heat up to 175°C and for short periods up to 280°C it would certainly be interesting to know how far the outgassing rate could be lowered under those conditions. The low outgassing rate resulting from a mild bakeout allows this elastomer to be recommended as gasket material in vacuum chambers.

The elastomer Silastic 1602 proved to be an inadequate material applied as construction material in space chambers. The material seemed to decompose under vacuum conditions as could be seen from the condensed oil film at the inside of the system. Even a mild bakeout did not improve its properties for space chamber applications. It cannot be recommended.

Like Silastic 1602 the same high initial outgassing rate could be observed with Silastic 916 which was studied subsequently. However, no evaporation of this material or of its components could be observed either at room temperature or during the heat treatment. The bakeout improved the outgassing properties considerably though the full temperature range from -90°C to +250°C was not applied. As in many of the other experiments the upper temperature was limited by the system pressure which could be tolerated without damage to the ion gauges. Silastic 916 treated to obtain its lower limit outgassing of 3×10^{-8} torr liters/sec. cm^2 can be recommended as a construction material.

The series of outgassing studies was completed by studying Vibrathane 5004, basically a polyurethane. This material proved to have a relatively high outgassing rate. The heat treatment did not improve the outgassing properties. The steep increase of the outgassing rate resulting to only a slight temperature increase and the indication of high masses by the mass spectrometer might mean that some weight loss occurred. By exposing the material to higher temperatures and longer times it could be determined whether outgassing or decomposition or evaporation was occurring.

Vibrathane 5004 could not be recommended unless further testing indicates a pre-treatment which would reduce the outgassing.

4.5 APPENDIX

4.5.1 Theory of Outgassing

The relationship of several parameters such as outgassing rate, chamber size, pumping speed, etc. is very important in the region of high vacuum and has been the subject of investigation of a number of publications. The earliest and highly intensive mathematical treatment of the subject is due to Dayton [9, 10]. In "1961 International Congress on Vacuum Science and Technology", a number of interesting papers were presented in the section "Limitations in the Attainment of Ultra-High Vacuum" [22, 39, 40]. It is universally agreed that in a leak-free system the gas load is caused by 1) desorption of adsorbed gas molecules on surfaces and 2) by the diffusion of dissolved gases in the bulk material. There is a third source - permeation of gases through the enclosure walls from the outside ambient - which is of significance in some cases. Since the present program is directed towards the behavior of the aluminum alloy with special emphasis on the low temperature region the theoretical analysis is confined to the phenomenon of desorption of surface gases.

If the surface has a real area, A , (cm^2) and N_a (torr liters) represents the gas adsorbed on the surface, then the rate of desorption is:

$$-\frac{dN_a}{dt} = \frac{n_a A_1}{\tau_0} \exp(-E_d/RT) \quad (1)$$

N_a = total gas on surface - torr liters

t = time - secs.

n_a = surface concentration adsorbed gas - torr $1/\text{cm}^2$

A_1 = actual surface area (cm^2)

$-E_d$ = desorption activation energy

T = absolute temperature

R = gas constant

τ_0 = time period of thermal oscillation of adsorbed molecules $\sim 10^{-13}$ secs.

Adsorption from the gas phase also is simultaneously occurring and its rate can be expressed by the following equation:

$$+ \frac{dN_a}{dt} = \nu \sigma A_2 \quad (2)$$

where

ν = is the rate of impingement of gas on unit area of surface - $\frac{\text{torr l}}{\text{cm}^2 \text{sec.}}$

σ = sticking probability

A_2 = geometric surface area (cm^2)

Now at equilibrium in an isolated system, adsorption rate equals desorption rate and therefore

$$\frac{n_a A_1}{\tau_0} \exp(-E_d/RT) = \nu \sigma A_2 \quad (3)$$

The impingement rate may be expressed in terms of other parameters by the following equation:

$$\nu = 3.513 \times 10^{22} \frac{P_{\text{torr}}}{\sqrt{MT}} \quad (4)$$

and substituting in equation (3) we obtain

$$\frac{n_a A_1}{\tau_0} \exp(-E_d/RT) = 3.513 \times 10^{22} \frac{\sigma A_2 P}{\sqrt{MT}} \quad (5)$$

Solving for P, the equilibrium pressure, we obtain

$$P = \frac{\sigma n_a}{\tau_0} \frac{A_1}{A_2} \frac{\sqrt{MT}}{3.513 \times 10^{22}} \exp(-E_d/RT) \quad (6)$$

when $A_1 = A_2$ this reduces to

$$P = \frac{\sigma n_a}{\tau_0} \frac{\sqrt{MT}}{3.513 \times 10^{22}} \exp(-E_d/RT) \quad (6a)$$

A typical value for monolayer coverage is 5×10^{14} molecules/ cm^2 . Sticking coefficient may range from 1.0 to very low values. For chemisorption of many gases on metallic surfaces, initial sticking coefficient is about 0.5. Sticking coefficient decreases as saturation is approached for a given monolayer.

Equations (1) and (6) permit calculating two parameters of interest for gases adsorbed on surfaces with different binding energies. Equation (1) permits calculation of the rate of desorption of gases from surfaces. Equation (6) permits calculation of the equilibrium pressure in a system as a function of temperature and binding energy of the adsorbed gas.

Table XVIA presents a series of values of outgassing rates calculated as a function of binding energies and temperature. Values of desorption energy of 10, 15, 20 and 25 kcal/mole were used in these calculations. The temperature values chosen were those which were expected to be used in the experimental program. The effect of possible temperature variations at each test temperature were also calculated. Outgassing values were reported in torr liters per cm^2 per sec. It should be noted that the area here is the true area and not geometric area. From Table XVIA, one can readily see the extreme sensitivity of desorption to variation in temperature and also the very appreciable effect of binding energy on desorption rate. These data provide a frame of reference for evaluating experimental results.

For example, one can see that for the hypothetical adsorbate whose desorption energy is 20 kilocalories per mole, an increase in temperature from room temperature to 100°C increases the rate of desorption by a factor of 1000. Cooling of the same system from room temperature to 0°C decreases the desorption rate by more than one order of magnitude and cooling to dry ice temperature reduces the rate by almost eight orders of magnitude as compared to the room temperature rate.

The calculated values must be used with considerable caution, however. One might deduce from the desorption value of 3.6×10^{-7} torr liters per cm^2 per sec. for the room temperature desorption rate for the 20 kcal binding energy that this species would be desorbed in a very short time (~40 sec.) since a monolayer coverage corresponds to only 1.5×10^{-5} torr liters per cm^2 of surface.

Several factors must be considered in dealing with real surfaces. The effective surface area is always greater than the geometric area and this factor will vary according to the surface finish up to orders of magnitude. In Equation (1), the area assumed is the true area. The calculated desorption rates are thus calculated on the basis of unit of true area. The quantities of gas adsorbed per unit of geometric surface area will be the adsorption concentration per unit area multiplied by the roughness factor (A_1/A_2).

Secondly, adsorption is not necessarily monomolecular, nor, in the case of multilayer adsorption would all the layers be bound with the same energy. Thus, even for a given molecule (e.g. H₂O) one can imagine a spectrum of binding energy with the energy of binding decreasing with the distance of the multilayer from the surface.

Nonetheless, Table XVII points out the probability that cooling of surfaces below ambient may be a useful technique in substantially reducing outgassing rate and the gas load resulting therefrom.

In Table XVIII, the equilibrium pressure in a closed system containing a single gas species was calculated for various values of adsorption binding energy utilizing Equation (6a) and assuming a value of 0.5 for the sticking coefficient and assuming a molecular weight of 18 (H₂O). The strong effect of temperature on the equilibrium pressure may be observed from Table XVII, and also the strong effect of binding energy. In Equation (6a), a roughness factor of unity was assumed. In Equation (6), roughness factors greater than unity will lead to higher calculated values of pressure for a given binding energy and temperature. Roughness factors of 100X higher are not unusual. The value of 0.5 for sticking coefficient may also be unrealistic in many structures. Values of 0.1 to 0.001 for sticking coefficient are not untypical for many gas-solid surface interactions. Therefore, we can see that the combined effects of surface roughness and variable sticking coefficient may result in equilibrium pressure values which are 10² to 10⁴ higher than those calculated in Table XVIII.

These considerations point clearly to the need for experimental evaluation to determine outgassing (desorption) rates in real systems.

TABLE XVII
Rate of Outgassing Due to Desorption

Temperature	Outgassing Rate in $\text{l}/\text{cm}^2 \text{ sec.}$ for Different Desorption Energies*					
	$^{\circ}\text{K}$	$^{\circ}\text{C}$	25 kcal/mole	20 kcal/mole	15 kcal/mole	10 kcal/mole
Liquid Nitrogen	80 120	-193 -153	1.5×10^{-60} 6.0×10^{-33}	6.0×10^{-47} 1.1×10^{-28}	3.0×10^{-33} 9.5×10^{-20}	6.0×10^{-20} 1.1×10^{-10}
Dry Ice	125 205	-148 -68	1.9×10^{-20} 4.7×10^{-19}	7.5×10^{-15} 9.5×10^{-14}	2.3×10^{-9} 1.9×10^{-8}	3.9×10^{-4} 3.8×10^{-3}
Eth. Glycol and Water	243 253	-30 -20	1.5×10^{-14} 4.8×10^{-14}	1.6×10^{-10} 9.3×10^{-10}	5.6×10^{-6} 1.9×10^{-5}	1.6×10^{-1} 3.9×10^{-1}
Ice and Water	273 278	0 5	1.8×10^{-12} 3.8×10^{-12}	1.8×10^{-8} 3.0×10^{-8}	1.7×10^{-4} 3.0×10^{-4}	1.7 2.1
R. T.	298	25	9.4×10^{-11}	3.8×10^{-7}	2.0×10^{-3}	7.5
Boiling Water	373 363	100 90	3.8×10^{-7} 1.9×10^{-7}	3.8×10^{-4} 1.9×10^{-4}	2.7×10^{-1} 1.5×10^{-1}	2.3×10^2 1.5×10^2

* Surface coverage of 5×10^{14} molecules/ $\text{cm}^2 = 1.5 \times 10^{-5}$ torr l/cm^2

TABLE XVIII

Calculated Pressures in Chamber
At Various Temperatures and Desorption Energies

Temperature	Equilibrium Pressure* in Torr at Desorption Energies				
	°K	25 kcal/mole	20 kcal/mole	15 kcal/mole	10 kcal/mole
80		1.5×10^{-44}	2.6×10^{-31}	2.4×10^{-22}	2.6×10^{-13}
195		3.8×10^{-21}	1.4×10^{-15}	4.1×10^{-10}	7.2×10^{-5}
243		2.3×10^{-15}	2.5×10^{-11}	3.5×10^{-7}	2.4×10^{-2}
273		2.6×10^{-13}	2.6×10^{-9}	2.4×10^{-5}	2.4×10^{-1}
298		7.5×10^{-12}	5.1×10^{-8}	2.7×10^{-4}	1.0
373		4.0×10^{-8}	4.0×10^{-5}	2.9×10^{-2}	7.9

* Assumes only single species adsorbed. Surface coverage 5×10^{-14} molecules/cm² assumes roughness factor of unity and sticking coefficient of 0.5.

4.5.2 Tables

TABLE I (UNABRIDGED)

Pressure Rise on Closing Aperture in 304 ELC
Stainless Steel Test Chamber with As-Received Surface

Elapsed Time (Hrs.)	Pressure in Test Chamber (Torr x 10 ⁷)					Pressure Rise dp/dt (Torr/sec) x 10 ⁷	Outgassing Rate (Fl/sec cm ²) x 10 ⁹	Room Temp.
	0 sec	5 sec	10 sec	20 sec	30 sec			
0.75	75.0	130.0	160.0	220.0	280.0	11.0	5.86	
2.5	22.0	42.0	64.0	100.0	130.0	4.8	2.55	
3.5	16.0	-	50.0	72.0	110.0	3.4	1.81	
4.5	13.0	28.0	40.0	59.8	82.0	3.21	1.73	
5.5	12.0	22.0	32.0	51.0	72.0	2.0	1.06	
-	-	-	-	Liq. N ₂ Added to the Cold Trap		-	-	-
7.0	3.4	9.0	14.0	22.0	28.0	1.1	0.586	
8.0	3.7	8.4	12.0	19.0	25.0	0.94	0.50	
9.5	0.35	1.2	1.4	1.6	1.7	0.22	0.117	
10.0	0.32	1.1	1.3	1.5	1.7	0.22	0.117	
11.25	0.30	0.9	-	1.3	1.5	0.14	0.0745	0°C
13.5	0.24	0.8	-	-	1.1	0.13	0.0692	
15.2	0.23	0.6	-	-	1.0	0.081	0.043	
16.5	0.12	0.5	-	-	0.68	0.076	0.041	
20.0	0.062	0.15	0.39	-	-	0.046	0.0008	-30°C
23.5	0.044	-	-	-	-	-	-	-
24.5	0.042	0.60	0.50	0.40	0.38	0.046	0.024	-78°C
25.5	0.044	-	-	-	-	0.043	0.023	

After the dry ice run, liquid N₂ was started. The pressure rose rather than dropped. Removed liquid nitrogen and located a large leak at the glass-metal seal of the Redhead gauge. The experiment was, therefore, abandoned at this point.

TABLE IA
 Mass-Spectrometer Data - Partial Pressures
 SS304 EIC "As-Received" All Pressures in Units of 10⁻⁷ Torr

Time Elapsed Hours	2	4	12	14	15	16	17	18	20	22	28	32	40	44	Chamber
1.0	7.14	-	-	-	1	0.40	11.48	22.33	-	-	4.47	-	-	3.20	49
1.5	-	-	0.43	0.50	0.80	1.33	1.74	19.80	1.08	-	14.10	-	-	-	40
1.5	7.70	5.85	-	-	2.69	2.69	2.69	-	-	-	15.40	-	3.00	-	40
2.5	4.29	0.72	-	0.31	0.19	0.77	0.34	1.32	-	-	12.07	-	0.40	1.56	22
3.5	5.54	1.27	0.74	1.06	0.95	1.27	-	1.43	2.22	-	0.26	-	-	1.27	16
5.5	5.71	0.84	0.42	0.42	0.75	0.96	0.36	0.73	1.44	0.29	0.12	-	0.48	1.47	14
6.0	2.43	0.26	0.16	0.16	0.33	0.45	0.18	0.54	0.54	-	(6.35)	-	0.14	0.61	12
7.0	0.98	0.06	0.03	0.03	0.06	0.16	0.03	0.11	0.15	-	(1.53)	-	0.04	0.19	3.4
8.0	1.09	0.05	0.04	0.04	0.07	0.08	-	0.19	0.16	-	(1.60)	-	0.08	0.25	3.7
END ROOM TEMPERATURE RUN															
10.0	0.07	0.01	-	-	0.02	0.01	-	0.02	0.03	-	0.12	-	0.01	0.02	0.32
12.0	0.08	0.005	-	0.006	0.006	0.01	0.006	0.015	0.018	0.012	0.12	0.006	0.006	0.027	0.30
13.5	0.068	0.006	0.007	-	0.007	0.007	-	0.007	0.019	-	0.105	-	-	0.016	0.24
END 0°C RUN															
15.0	0.067	0.007	-	-	-	-	-	-	0.011	0.007	0.137	-	-	-	0.23
16.5	0.009	0.004	-	-	0.01	0.01	0.01	0.009	0.014	-	0.054	-	-	-	0.12
21.0	0.015	0.003	-	-	-	0.006	-	0.01	0.01	-	0.019	-	-	-	0.062
END -30°C RUN															

TABLE II (UNABRIDGED)

Pressure Rise on Closing Aperture in 304 ELC
Stainless Steel Test Chamber with No. 4 Surface Finish

Elapsed Time (Hrs.)	Pressure in Test Chamber(Torr $\times 10^7$)					dp/dt (Torr/sec) $\times 10^7$	Outgassing Rate (Fl/sec cm^2) $\times 10^9$	Temperature
	0 sec	5 sec	10 sec	20 sec	30 sec			
0.5	42.0	80.0	110.0	160.0	200.0	7.2	3.83	Room Temp
1.75	13.0	24.0	32.0	48.0	59.0	2.6	1.38	
2.75	6.0	-	18.0	25.0	33.0	1.4	0.74	
4.5	3.8	8.5	12.0	17.0	20.0	1.0	0.554	0°C
6.0	1.8	3.0	3.8	4.5	5.6	0.28	0.149	
7.0	1.1	2.2	2.7	3.8	4.6	0.26	0.138	
10.0	0.75	1.3	1.5	1.9	2.1	0.12	0.064	-27°C
13.0	0.17	0.39	0.47	0.59	0.69	0.076	0.040	
14.0	0.15	0.36	0.44	0.56	0.65	0.060	0.032	
15.25	0.12	0.31	0.39	0.46	0.52	0.054	0.0287	-78°C
16.25	0.094	0.29	0.35	0.42	0.47	0.054	0.0287	
18.25	0.088	0.26	0.32	0.39	-	0.054	0.0287	
20.0	0.040	0.10	0.13	0.14	0.15	0.014	0.00745	- 196°C
21.0	0.038	0.10	0.13	0.14	0.15	0.014	0.00745	
31.0	Liq. N ₂ Cooling maintained up to this time							
32.8	0.82	2.8	4.5	6.0	7.5	0.36	0.192	Room Temp
35.3	0.97	2.3	3.2	4.6	5.9	0.306	0.163	
37.0	34.0	140.0	260.0	440.0	620.0	22.6	12.8	100°C
38.0	9.1	50.0	80.0	120.0	170.0	8.2	4.36	
39.2	7.1	37.0	60.0	100.0	130.0	6.2	3.30	
40.2	1.6	12.0	21.0	34.0	44.0	2.2	1.17	
41.0	Hot water removed							
43.5	0.097	0.48	0.58	0.74	0.88	0.095	0.0505	Room Temp
44.7	0.054	0.20	0.30	0.38	0.42	0.031	0.0165	
52.0	0.05	0.20	0.28	0.36	0.4	0.032	0.017	-196°C
52.75	0.03	0.10	0.12	0.14	0.15	0.017	0.0089	

TABLE IIA
 Mass-Spectrometer Data - Partial Pressures
 SS304 ELC No. 4 Finish. All Pressures in Units of 10^{-7} torr

Time Elapsed Hours	2	4	12	14	15	16	17	18	20	22	28	32	40	44	Chamber P
0.0	36.39	0.82	-	1.08	1.33	2.03	1.46	5.07	1.84	-	37.47	-	0.76	1.78	90.00
1.75	0.04	-	0.15	0.23	-	0.38	0.15	0.53	0.34	-	11.17	-	-	-	13.00
4.50	-	-	0.15	0.14	0.18	0.31	0.58	1.72	-	0.05	-	-	-	0.66	3.80
	END ROOM TEMPERATURE RUN														
No mass-spectrometer data during low temp. runs because of breakdown of mass-spectrom.															
32.5	0.01	-	-	0.03	0.05	0.08	0.14	0.04	0.01	0.39	-	0.01	0.06	0.82	
35.0	0.09	0.02	0.02	0.04	0.04	0.05	0.04	0.10	0.06	-	0.48	-	0.04	0.04	1.00
	END 2nd ROOM TEMPERATURE RUN														
37.0	2.42	-	-	0.50	0.75	3.33	7.56	1.36	-	9.60	-	-	-	2.49	28.00
38.0	1.19	-	0.19	0.11	0.29	0.41	0.49	1.49	0.40	-	4.04	-	-	0.48	9.10
38.5	0.78	0.14	-	-	0.11	0.27	0.43	0.83	0.34	-	3.68	-	0.05	0.48	7.10
39.5	0.17	-	0.04	0.04	0.07	0.09	0.07	0.16	0.14	-	0.69	-	0.04	0.01	1.60
	END +100°C RUN														
45.0	0.03	-	-	-	-	-	-	-	-	-	0.03	-	-	-	0.054
	END 3rd ROOM TEMPERATURE RUN														
55.0	0.001	-	-	-	-	-	-	-	-	-	0.001	-	-	-	0.029
	END 2nd IN ₂ RUN														

TABLE III (UNABRIDGED)
 Pressure Rise on Closing Aperture in 5083-0 Al Alloy ("as received") Test Chamber with As-Received Surface

Elapsed Time (Hrs)	Pressure in Test Chamber (Torr x 10 ⁷)					dp/dt (Torr/sec) x 10 ⁷	Outgassing Rate (Fl/sec cm ²) x 10 ⁹	Temperature
	0 sec	5 sec	10 sec	20 sec	30 sec			
0.8	370.0	600.0	890.0	1300.0	-	100.0	53.2	
2.0	110.0	170.0	190.0	200.0	210.0	30.0	15.9	
3.8	49.0	85.0	96.0	110.0	120.0	14.2	7.55	Room Temp.
5.2	35.0	60.0	65.0	72.0	75.0	7.0	3.71	
5.75	31.0	54.0	59.0	65.0	68.0	6.8	3.61	
7.3	15.0	29.0	31.0	38.0	41.0	4.5	2.39	
10.3	7.6	19.0	22.0	25.0	27.0	4.1	2.18	
11.3	3.7	4.5	4.6	4.7	4.75	0.25	0.133	
12.5	2.2	3.0	3.1	3.25	3.30	0.24	0.128	0°C
13.7	1.9	2.6	2.8	2.9	3.0	0.18	0.0957	
14.5	1.6	2.3	2.4	2.5	2.6	0.18	0.0957	
15.7	1.4	1.6	1.65	1.65	1.65	-	-	-30°C
16.5	1.0	1.2	1.2	1.2	1.2	-	-	
17.3	1.2	1.1	1.1	1.1	1.1	-	-	
18.6	0.68	1.0	0.9	0.82	0.82	-	-	-78°C
22.3	0.60	0.7	0.7	0.70	0.70	-	-	-196°C
25.0	0.06	0.3	0.2	0.1	0.1	-	-	
26.0	3.6	18.0	26.0	38.0	48.0	3.7	1.97	Room Temp.
27.3	2.6	12.0	16.0	22.0	28.0	2.3	1.22	
30.0	2.3	7.0	8.3	9.6	12.0	2.0	1.06	Room Temp.

TABLE IIIA

Mass-Spectrometer Data - Partial Pressures
 AL-5083-0 "As-Received". All Pressures in Units of 10^{-7} torr.

Time Elapsed Hours	2	4	12	14	15	16	17	18	20	22	28	32	40	44	P Chamber
2.0	23.52	1.18	-	1.76	4.03	8.04	36.88	90.72	6.22	-	16.80	-	1.43	-	190.00
2.5	34.89	-	-	-	6.86	30.03	108.97	-	-	-	-	-	-	-	180.00
11.0	-	-	-	-	1.22	-	3.17	-	-	-	3.29	-	-	-	7.60
END ROOM TEMPERATURE RUN															
12.5	-	-	-	-	-	-	-	3.22	-	-	1.29	-	-	-	4.51
13.0	-	-	-	-	-	-	-	-	-	-	-	-	2.20	-	2.20
END OOG RUN															
16.5	0.13	-	-	-	-	-	-	-	-	0.18	0.40	-	0.18	0.51	1.40
17.5	0.30	-	-	-	-	-	-	0.33	0.33	-	0.23	-	-	-	1.20
END -30°C RUN															
No mass-spec data during -78°C and -196°C Runs															
26.5	2.26	-	-	-	-	-	0.29	0.29	-	-	-	-	-	0.70	3.60
27.0	0.45	0.06	-	0.08	0.09	0.12	0.38	1.17	0.15	-	0.70	-	0.03	0.35	3.60
29.5	0.15	0.10	0.06	0.04	0.07	0.17	0.75	0.19	0.10	-	0.18	-	0.13	0.66	2.60
31.5	0.35	-	-	-	-	-	0.46	0.88	0.22	-	0.37	-	-	-	2.30
END ROOM TEMPERATURE RUN															

TABLE IV (UNABRIDGED)

Pressure Rise on Closing Aperture in 5083-0 Al Alloy Test Chamber with No. 4 Finish

Elapsed Time (Hrs)	Pressure in Test Chamber (Torr x 10 ⁷)						dp/dt (Torr/sec) x 10 ⁷	(Tl/sec cm ²) x 10 ⁹	Temperature
	0 sec	5 sec	10 sec	20 sec	30 sec	30 sec			
0	280.0	1700	3100	4400	5300	5300	284.0	151.0	
1	180.0	1300	2300	4100	5200	5200	224.0	119.0	
2	180.0	1000	2800	4400	5400	5400	164.0	87.2	Room Temp
3.25	Found and sealed a leak								
3.26	24.0	24.0	42.0	56.0	67.0	67.0	2.2	1.17	
5.0	3.7	8.6	14.0	22.0	29.0	29.0	0.98	0.521	
6.0	2.5	5.0	7.6	10.0	14.0	14.0	0.54	0.287	
7.0	0.86	1.7	2.4	3.3	4.1	4.1	0.189	0.10	
8.0	0.89	1.8	2.4	3.2	3.9	3.9	0.189	0.10	0°C
9.0	0.64	1.0	1.2	1.5	1.7	1.7	0.092	0.0489	
12.0	0.51	1.2	1.6	2.0	2.2	2.2	0.168	0.0895	
13.25	0.39	0.78	1.1	1.3	1.6	1.6	0.084	0.0446	-30°C
14.5	0.32	.63	0.78	0.93	1.1	1.1	0.072	0.0382	
16.0	0.17	.42	0.53	0.67	0.76	0.76	0.062	0.0329	
17.5	0.13	.40	0.54	0.68	0.79	0.79	0.046	0.0244	-78°C
18.5	0.14	.40	0.54	0.70	0.80	0.80	0.046	0.0244	
19.5	0.063	.16	0.20	0.24	0.28	0.28	0.0244	0.0129	
20.5	0.052	.15	0.19	0.22	0.25	0.25	0.0244	0.0129	-196°C
24.0	0.71	1.5	1.8	2.3	2.8	2.8	0.18	0.0956	Room Temp
25.0	64.0	310.0	500.0	840.0	1100.0	1100.0	51.2	27.4	
26.0	13.0	80.0	100.0	190.0	240.0	240.0	15.4	8.2	100°C
27.0	7.2	36.0	54.0	80.0	100.0	100.0	6.56	3.48	
28.0	0.21	0.50	0.67	0.83	0.95	0.95	0.066	0.035	Room Temp
29.0	0.22	0.49	0.64	0.78	0.87	0.87	0.064	0.034	Room Temp

TABLE IVA

Mass-spectrometer Data - Partial Pressures
 A1-5083-0 No. 4. All pressures in units of 10^{-7} torr.

Time elapsed hours	2	4	12	14	15	16	17	18	20	22	28	32	40	44	P Chamber	
1.5	6.56	-	10.39	-	-	-	-	16.41	5.47	-	136.75	4.38	-	-	180.00	
2.0	6.53	-	11.04	3.01	-	3.01	-	8.03	3.01	-	140.56	5.02	-	-	180.00	
4.0	1.92	-	-	0.43	-	1.87	-	0.89	-	-	16.08	-	0.43	2.52	24.00	
5.0	1.20	-	0.01	0.04	0.08	0.09	-	0.08	0.10	-	1.72	-	-	0.33	3.70	
6.0	0.27	-	-	-	0.07	0.08	-	0.15	0.18	-	1.39	-	0.07	0.30	2.50	
								END ROOM TEMPERATURE RUN								
7.0	0.14	-	-	-	-	-	-	-	-	-	0.72	-	-	-	0.86	
								END 0°C RUN								

TABLE VIA

Glass System - Blank Run Mass Spectrometer Data
(all pressures in units of 10^{-7} torr)

Elapsed Time Hours	Temper- ature	Mass							P (Chamber)
		2	12	14	18	28	32	44	
5.5	R.T.	1.06	-	-	5.30	17.25	-	0.44	24.0
6.0	R.T.	9.15	1.15	1.62	1.15	14.7	0.48	4.77	33.0
8.0	R.T.	0.13	-	0.04	0.26	0.39	-	0.08	0.89
24.75	R.T.	-	-	-	0.08	0.08	-	0.02	0.17
27.25	+ 40°C	0.03	-	-	0.09	0.15	-	0.15	0.28
28.5	+100°C	0.32	-	-	0.10	0.13	-	0.03	0.58
30.0	+180°C	0.43	0.06	0.12	1.04	0.37	-	3.56	5.60
31.5	+240°C	0.27	0.03	0.06	0.63	1.70	-	1.30	4.70
98.0	R.T.	-	-	-	0.03	0.03	-	0.01	0.07

TABLE VIIA
 Total and Partial Pressures (Mass Spectrometer Data) - Aluminum 5083-0 "as received"

(all pressures in units of 10^{-7} torr)

Elapsed Time Hours	Temp.	Mass											P Chamber
		2	12	14	18	28	32	44	15	16	17	17	
1.00	R. T.	6.30	1.14	0.76	21.00	76.00	76.00	21.00	21.00	1.90	2.86	4.75	210.00
2.40	R. T.	2.98	1.36	0.65	13.55	59.60	-	4.61	-	-	3.25	86.00	
5.15	R. T.	0.57	0.95	1.71	1.78	2.54	24.00	0.95	-	-	-	33.00	
6.50	R. T.	0.92	0.09	0.03	4.25	3.24	-	1.30	-	0.19	-	10.00	
7.50	R. T.	0.80	0.09	0.17	2.60	3.10	0.19	1.06	-	0.35	-	8.30	
25.25	R. T.	0.01	0.14	0.06	0.06	3.67	-	0.57	-	-	-	4.50	
26.25	+50°C	0.49	-	-	1.95	1.55	-	0.73	-	0.08	-	4.80	
29.00	+100°C	8.60	0.46	0.27	23.00	10.00	-	6.30	-	-	-	49.00	
50.00	R. T.	0.22	0.03	0.03	0.44	1.37	-	0.71	-	-	-	2.80	

TABLE VIII A

Total and Partial Pressures Mass Spectrometer Data - Al-Si-Mag 614
(All pressures in units of 10^{-7} torr)

Elapsed Time Hours	Temp. (°C)	Mass										P Chamber
		2	12	14	18	28	32	44	16	16		
2.00	R. T.	-	8.25	9.00	15.00	39.50	0.12	11.80	0.08	85.00		
4.50	R. T.	0.02	0.10	0.10	3.20	4.20	0.03	1.00	-	8.80		
6.00	R. T.	0.27	0.03	0.03	2.20	2.40	0.13	0.30	-	5.30		
7.50	R. T.	0.15	-	-	0.80	0.73	0.07	0.15	-	1.90		
23.00	R. T.	0.03	0.03	0.07	0.10	0.03	0.2	0.28	-	0.98		
24.50	+ 50°C	11.5	1.15	1.87	57.50	67.00	8.05	12.50	-	160.00		
26.00	+100°C	-	0.37	0.25	29.8	74.40	1.49	124.00	-	230.00		
26.50	+155°C	-	-	-	-	500.00	-	800.00	-	1300.00		
28.00	+230°C	10.50	0.53	0.44	21.80	87.50	5.30	34.00	-	160.00		
30.00	R. T.	-	-	-	-	0.55	-	0.55	-	1.10		

TABLE IXA
 Total and Partial Pressures (Mass Spectrometer Data) - Teflon No. 7
 (All pressures in units of 10^{-7} torr)

Elapsed Time Hours	Temperature	Mass									
		2	12	14	18	28	32	44	41	31	
6.00	R. T.	-	2.82	31.00	18.00	258.00	33.50	10.80	-	77.40	430.00
23.00	R. T.	-	-	15.00	1.90	101.00	16.50	0.25	-	19.00	150.00
25.00	R. T.	-	-	11.80	1.38	102.50	15.80	1.97	-	16.75	150.00
29.00	R. T.	-	2.00	5.60	1.34	85.00	16.80	1.56	2.70	15.20	130.00
30.50	+ 47°C	-	-	16.60	16.60	415.00	-	24.90	22.20	44.2	540.00
31.5	+100°C)	No reliable values obtainable-see text									
49	R. T.)										

TABLE XA

Mass Spectrometer Data

Total and Partial Pressures of Mycalex 400
(All Pressures in units of 10^{-7} torr)

Elapsed Time Hours	Temp. (°C)	Mass							Total
		2	12	14	18	28	32	44	
3.00	R. T.	102.0	4.2	4.2	39.9	7.0	3.5	10.0	170.00
3.75	R. T.	-	-	16.2	102.0	20.4	4.1	26.9	170.00
5.80	R. T.	-	-	4.3	35.8	10.9	2.7	21.5	75.00
7.80	R. T.	-	-	5.8	27.5	9.5	-	7.5	50.00
9.80	R. T.	-	-	7.2	14.3	5.5	-	5.0	32.00
11.80	R. T.	-	-	5.2	10.4	5.2	-	4.2	25.00
13.80	R. T.	-	-	3.2	6.4	6.4	-	5.1	21.00
15.80	R. T.	-	-	3.8	3.8	3.8	-	5.1	17.50
19.0	R. T.	-	1.9	3.8	1.9	3.8	2.8	0.3	14.50
22.74	+ 92°C	-	-	-	21.8	6.5	-	1.7	30.00
24.50	+140°C	-	2.0	5.9	29.3	17.0	-	10.4	65.06
25.40	+100°C	-	0.5	0.5	1.8	3.50	0.7	3.1	10.00
27.25	R. T.	-	0.34	0.23	0.16	-	0.44	0.78	1.95
29.00	R. T.	-	0.18	0.21	0.16	0.24	0.14	0.08	1.00
31.00	R. T.	-	0.23	0.16	0.17	-	0.2	0.10	0.85
33.00	R. T.	-	0.17	0.20	0.17	0.17	-	0.08	0.80
43.00	R. T.	-	-	0.04	0.04	0.04	-	0.40	0.50

TABLE XIA

Mass Spectrometer Data

Partial and Total Pressures of Ceramic Coated Copper Wire
(All pressures in units of 10^{-7} torr)

Elapsed Time Hours	Temp. (°C)	Mass							Total
		2	12	14	18	28	32	44	
0	R. T.	145.5	-	-	2.0	1.6	-	0.6	150.00
1.8	R. T.	15.7	-	0.5	0.7	0.5	-	0.2	17.50
2.5	R. T.	11.8	-	0.1	0.33	0.3	-	0.1	12.50
5.0	R. T.	5.9	-	0.1	0.18	0.2	-	0.14	6.50
7.0	R. T.	4.4	0.08	0.06	0.01	0.1	-	0.04	4.80
11.0	R. T.	2.9	0.002	0.004	0.006	0.003	-	0.004	2.90
22.0	60°C	16.8	-	4.0	11.0	10.2	-	7.30	200.00
33.0	156°C	-	-	-	32.2	121.0	-	67.1	220.00
30.0	R. T.	1.08	0.36	0.84	0.37	0.50	-	0.16	2.80
32.0	R. T.	1.95	0.01	0.01	0.02	0.004	-	0.01	2.00
34.0	R. T.	0.99	0.14	0.16	0.36	0.05	-	0.02	1.70
36.0	R. T.	-	0.32	0.26	0.57	0.12	-	0.32	1.60

TABLE XIIA

Mass Spectrometer Data

Total and Partial Pressures Cat-a-lac Paint (463-1-8)

Elapsed Time Hours	Temp. (°C)	2	12	14	Mass 18	28	32	44	Total
46.25	R. T.	-	57.7	50.7	-	80.7	50.7	-	240.0
51.00	R. T.	42.2	-	-	1.90	1.90	-	-	46.0
52.50)	LN ₂ Temp.	-	0.69	0.31	0.72	0.19	-	-	1.90
53.50)		0.71	-	-	0.27	-	-	0.06	0.74
143.5	R. T.	-	1.11	0.70	2.52	-	0.70	0.37	5.40

TABLE XIII A

Partial and Total Pressures Butyl 035

Elapsed Time Hours	Temp. (°C)	(All pressures in units of 10^{-7} Torr)							Total
		2	12	14	Mass 18	28	32	44	
17.75	R. T.	309.0	-	-	107.0	64.2	-	-	480.0
19.50	-	108.0	-	-	17.2	7.66	2.36	0.61	135.0
21.00	-	-	-	-	81.6	25.5	4.90	7.90	120.0
23.00	R. T.	-	-	-	14.2	6.5	10.2	11.60	170.0
23.00	32°C	-	14.6	46.0	190.0	42.0	23.0	15.0	320.0
44.25	R. T.	-	-	-	12.5	3.46	-	2.08	18.0

TABLE XVA

Mass Spectrometer Data

Partial and total Pressures - Silastic 916
 (All pressures in units of 10^{-7} torr)

Elapsed Time Hours	Temp. (°C)	12	14	16	18	28	32	44	Total
3.75	R. T.	65.2	42.5	-	113.5	161.8	221.0	255.0	630.0
6.50	R. T.	37.5	57.5	-	77.5	100.0	37.5	-	310.0
22.5	R. T.	14.4	19.2	-	22.8	33.6	-	-	90.0
30.75	R. T.	-	-	11.32	11.32	17.36	-	-	40.0
46.00	R. T.	0.24	0.72	-	1.4	0.85	-	0.39	3.6

TABLE XVIA

Mass Spectrometer Data
 Partial and Total Pressures - Vibrathane 5004
 (All pressures in units of 10^{-7} Torr)

Elapsed Time Hours	Temp. (°C)	Mass								
		2	12	14	18	28	32	44	44	Total
4.50	R. T.	2.60	13.6	22.0	23.2	5.42	-	15.6	64.2	400.00
6.00	R. T.	-	-	11.58	-	11.58	-	6.11	142.0	190.0
(Also mass 30: 9×10^{-7} ; 21-22: 10.3×10^{-7})										
27.0	+46°C	-	15.8	31.0	124.0	-	-	65.0	-	360.0
28.0	R. T.	-	-	-	-	6.0	-	-	6.	120.0

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