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OPTICAL EFFECT OF THE CONTAMINATION OF INFRARED WINDOWS
BY THE OUTGASSING OF MATERIALS IN OUTER SPACE

SEMI-ANNUAL STATUS REPORT

FOR THE PERIOD ENDING JUNE 30, 1975

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I. SUMMARY OF OBJECTIVES

The products and kinetics of outgassing from S13G paint were to be studied during this reporting period. This study was to be carried out by infrared absorption measurements on the material outgassed using the evaporation cell and vacuum chamber described in the previous interim report.

II. WORK COMPLETED AND PRESENT STATUS

A. INSTRUMENTATION

The geometry of the test chamber (Figure 1) is such that the gases evaporated from a one-inch diameter paint sample are collected on a one-inch diameter cesium iodide window. The sample and the collecting window are separated by a distance of nine centimeters. The sample can be heated up to 200°C while the infrared collecting window can be cooled to any point between room temperature and 20°Kelvin. A separately heated conical shield helps to prevent the outgassing products from condensing on the walls of the vacuum chamber.

B. EXPERIMENTAL

1. Two separate long-term experimental depositions from heated samples of the S13G paint were made. Before each experiment, the vacuum chamber and sample holder were carefully cleaned and outgassed to ensure that subsequent deposits on the colled-window were due only to the products evaporated from
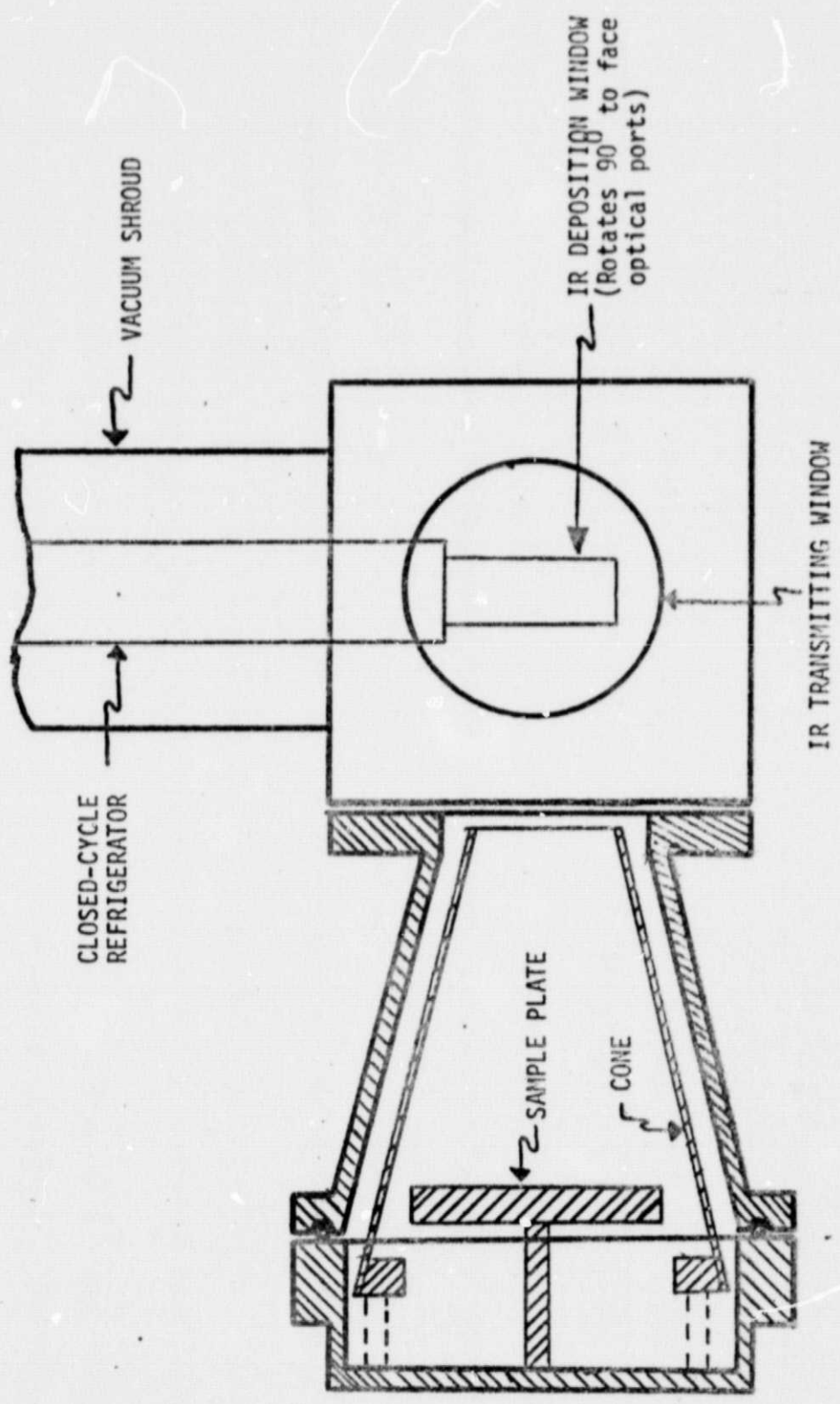
FIGURE 1: LOWER PART OF CONTAMINATION CHAMBER SHOWING SAMPLE ACCESSORY (Approximately Full Scale)

VACUUM SHROUD

IR DEPOSITION WINDOW
(Rotates 90° to face
optical ports)

CLOSED-CYCLE
REFRIGERATOR

IR TRANSMITTING WINDOW

SAMPLE PLATE

CONE

the sample. In the first experiment, the sample was held at $50^{\circ}C$, the cone at $75^{\circ}C$, and deposition window at $240^{\circ}K$ $(-33^{\circ}C)$. Total deposition time was 122 hours. The infrared transmission spectrum of the deposit is shown in Figure 2. The spectrum is primarily that of dimethysiloxane, the source of which would be the RTV-602 (polydimethysiloxane) binder used in the paint. The infrared spectrum was measured at selected times during the deposit so that deposition rate measurements could be made. A plot of peak absorbance for the major bands is given in Figure 3. To a fairly good apporximation, the peak absorbance (A) of an infrared absorption band is directly proportional to the sample thickness $\underline{x}$;

$$A(\nu) = \alpha(\nu)x$$

where $\alpha(\nu)$ is the absorption coefficient at frequency ν. It is evident that after an initial relatively rapid build-up of deposited material, the rate of sample outgassing was reduced and maintained at a much lower level.

In the second experiment, a fresh sample of S13G paint was tested under the following conditions: sample temperature was $40^{\circ}C$, cone temperature $75^{\circ}C$, and deposition window at $240^{\circ}K$. The only change from the previous experiment was the lowering of the sample temperature from $50^{\circ}C$ to $40^{\circ}C$. The infrared spectrum of the deposit was again measured at intervals during the deposition period of 263.5 hours so that data could be accumulated on the rate of outgassing by the sample by following the growth of the absorption bands. The spectral data is summarized in Figure 4, in which the peak absorbances for five of the strongest bands are plotted versus time. Inspection of Figure 4 shows that the results are similar to those found in the previous test. The weakness of the
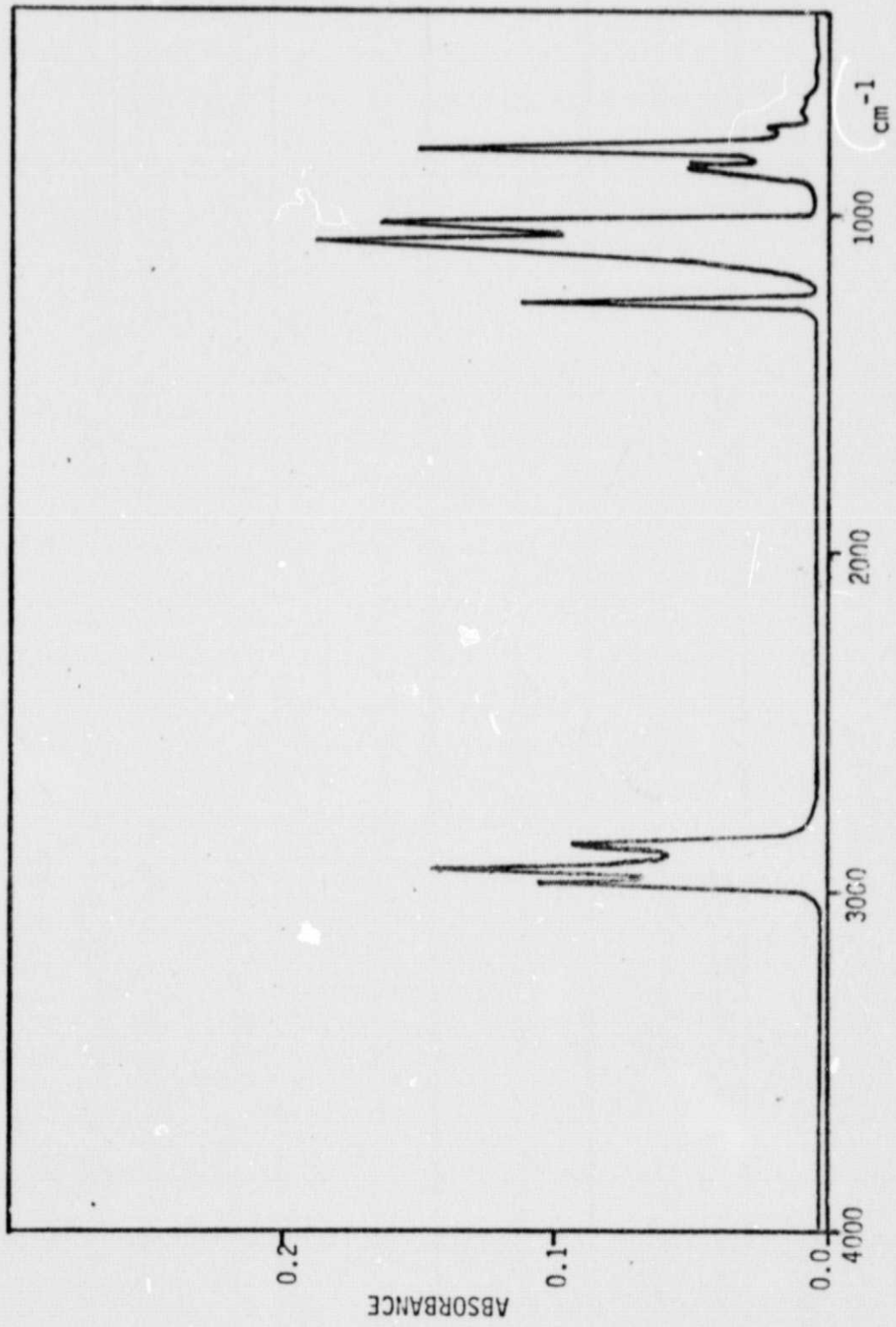
FIGURE 2 : Infrared spectrum of material outgassed from S13G paint and collected on a cooled CsI window.
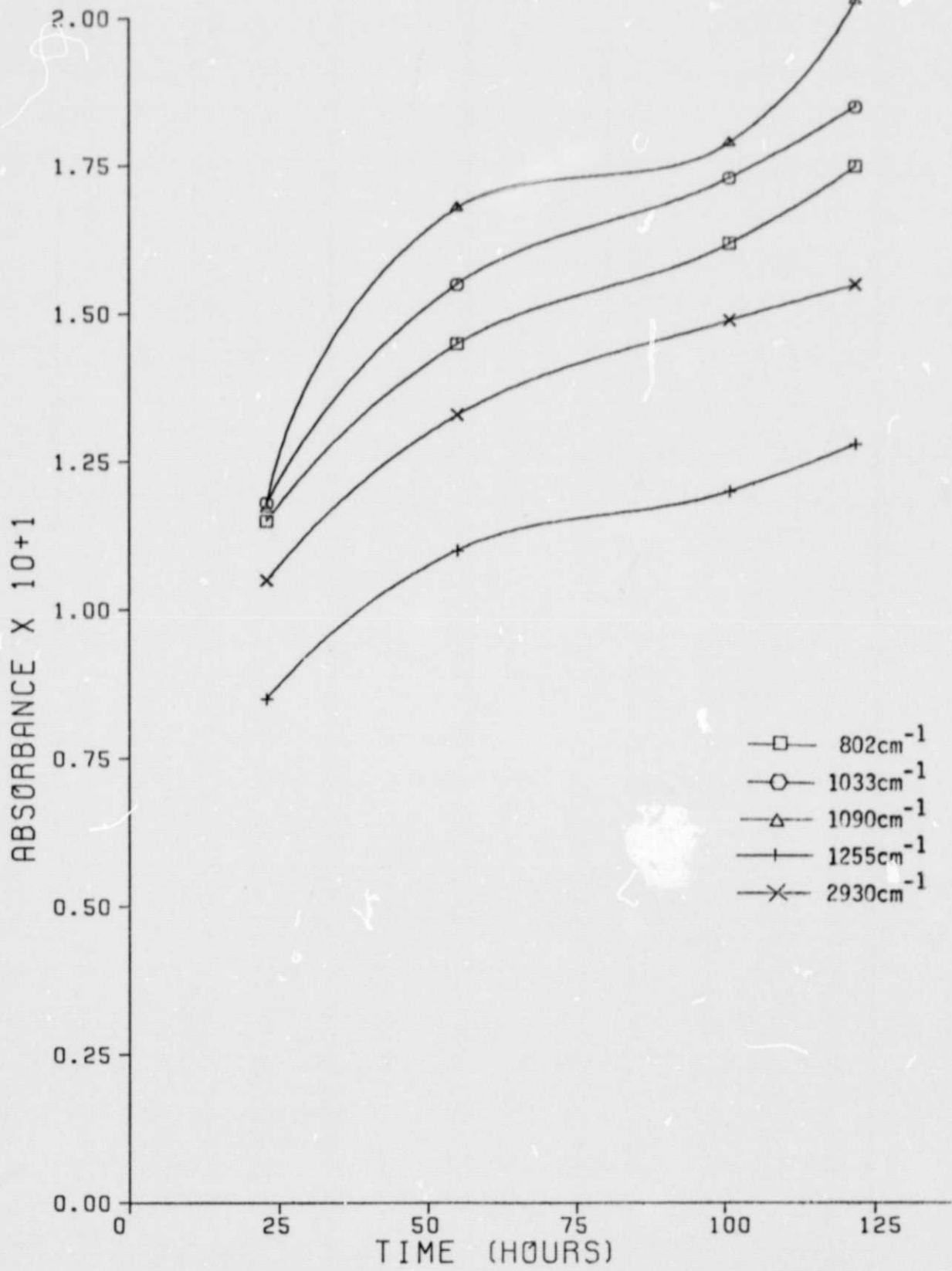
FIGURE 3: Absorbance as a function of time for major absorption
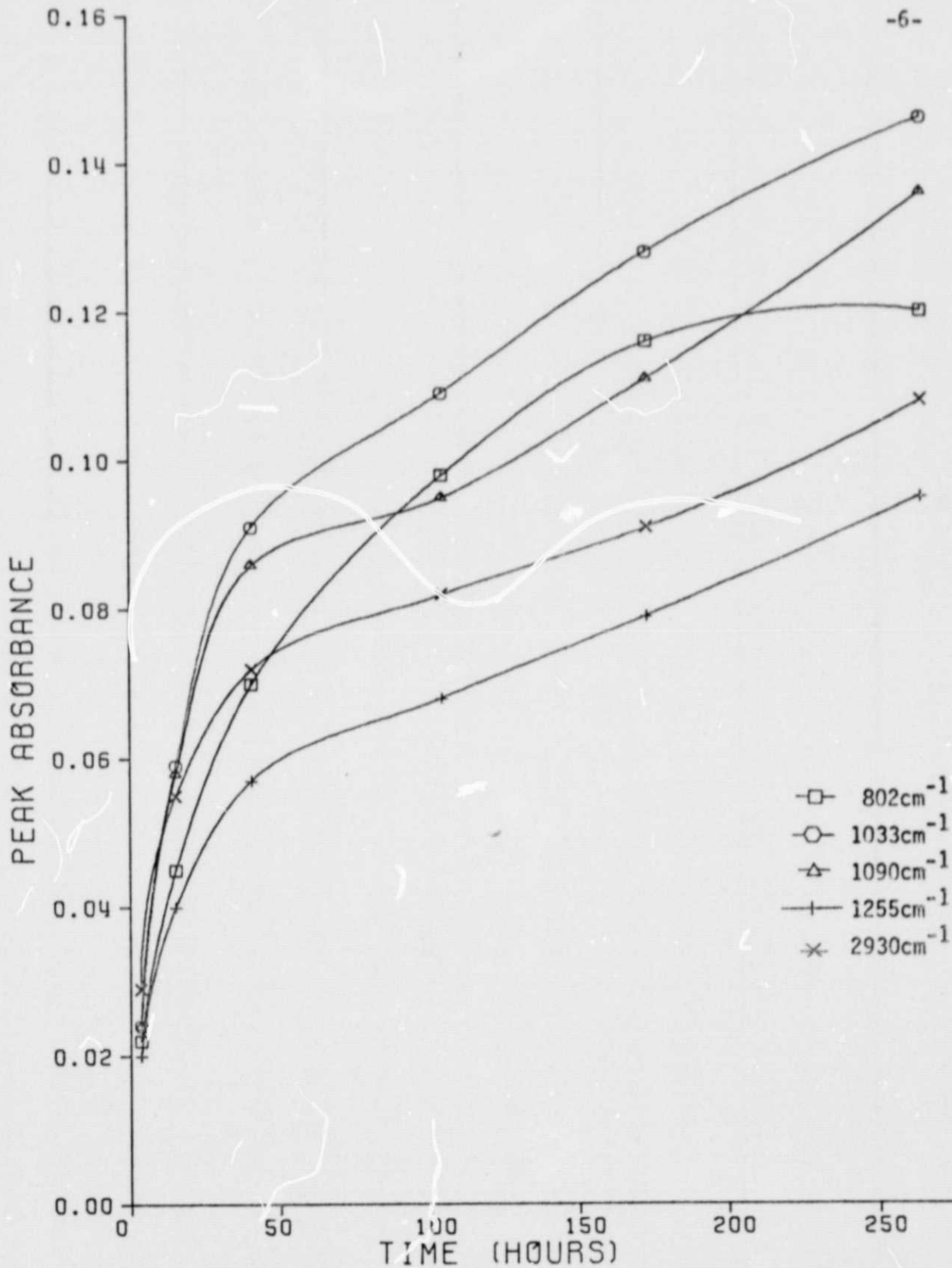bands of outgassing deposit (122 hour test).

FIGURE 4: Absorbance as a function of time for absorption bands of outgassing deposit (264 hours test).

absorption bands made measurement of peak intensities difficult during the initial stage of the test, but all of the peaks show a quite definite change in growth rate after 50 hours. This is shown even more explicitly in Figure 5, which is a graph of deposition rate $\underline{R}$ against time. The deposition rate is defined by the equation

$$R = \Delta A / A_{max} \cdot \Delta t$$

where ΔA is the change in peak absorbance during the time interval Δt, and A_{max} is the maximum intensity of the peak as measured at the end of the deposition period. Division by A_{max} makes R independent of the differing absorption coefficients for different frequency peaks, so R should be the same for all the absorption peaks of a given compound during any time interval. The graph of R versus time shows quite distinctly the change in deposition rate, which is equivalent to the outgassing rate of the paint sample. Within experimental error, the data is consistent with all the bands being due to one molecular species, although there is some uncertainty about the $1090 cm^{-1}$ band, in comparing the data for this test and the previous one.

If the outgassing is due primarily to the presence of trapped monomeric dimethylsiloxane molecules in the paint, the change in outgassing rate may reflect an initial rapid evaporation of monomer molecules at or near the surface of the paint, followed by a slower evaporation rate limited by the diffusion rate of the molecules moving from the interior of the paint film out to the surface. The data also shows that the evaporation was continuing at a relatively constant rate after 264.5 hours, when the test was ended.
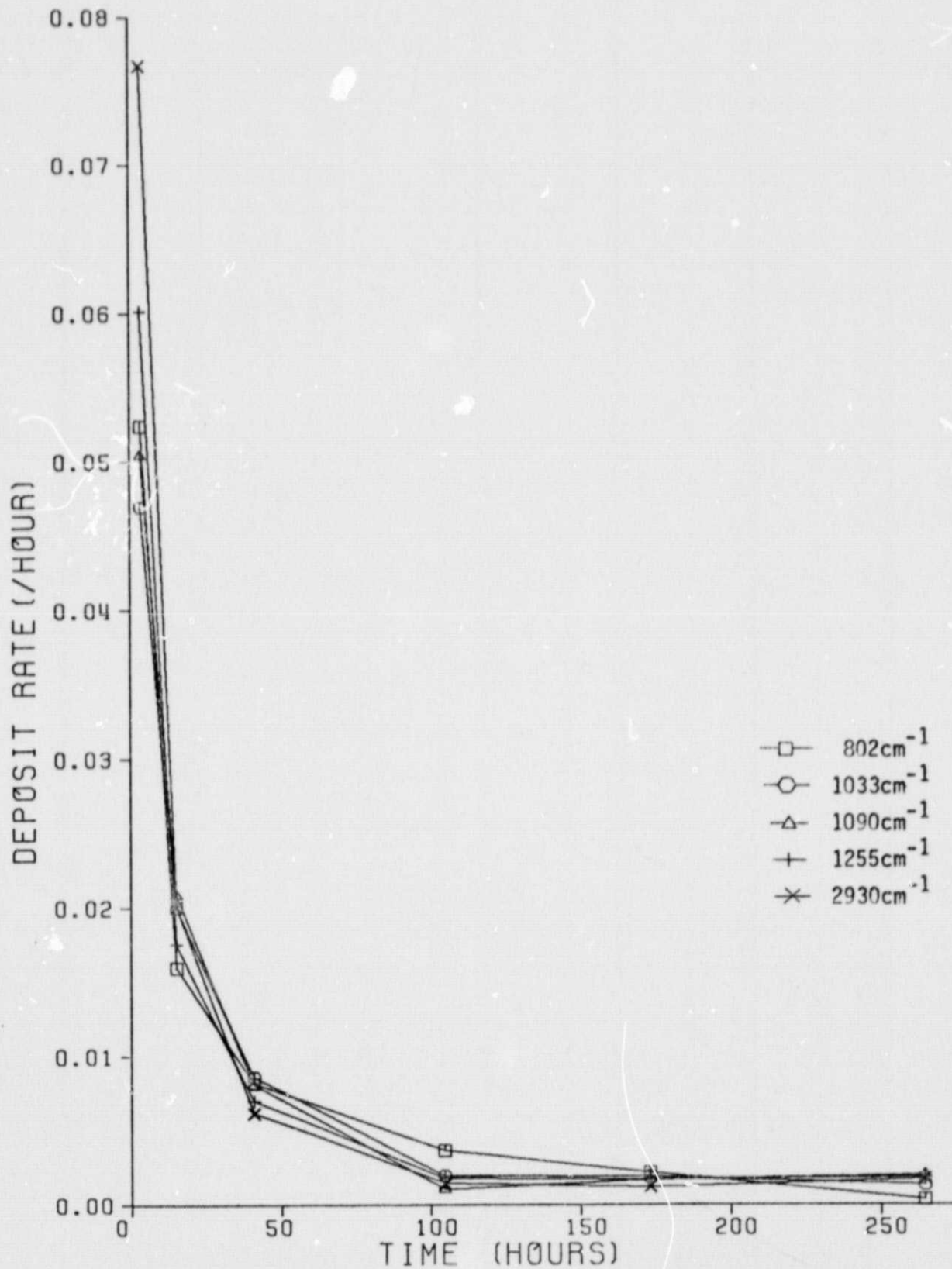
FIGURE 5: Deposit rate as a function of outgassing time for S13G paint (264 hour test).

The material collected on the deposition window during the test showed no measureable evaporation rate when held at $240^{\circ}K$ $(-33^{\circ}C)$. As the window temperature was raised, however, the deposit began to evaporate, as indicated by the slow disappearance of the infrared absorption bands. This is shown in Figure 6, in which the evaporation rate from the window is plotted against window temperature. Again, the evaporation rate $\underline{R}$ is defined similarly to the deposition rate given above, although in this case the negative sign introduced by the fact that $\triangle A$ is negative rather than positive has been dropped for clarity. The fact that the curves for all the bands fall within a narrow envelope further indicates that only one compound was present in the accumulated deposit. The values of R were calculated at 10° intervals from the change in absorption with time for each of the major absorption peaks. The rate of evaporation can be seen to have increased almost linearly with increasing temperature. This type of curve along with a similar curve for outgassing by the sample could be used to calculate the equilibrium build-up of contaminant layers for situations in which the contaminant source was at one temperature and the contaminated surface at another.

2. Since the assumption of proportionality between intensity and sample thickness is used in the above analysis, we have performed experimental and theoretical tests of this assumption using deposits of ethanol and acetone whose thicknessses were measured by an interferometric technique. In particular, we have considered the errors due to multiple reflection effects in thin films which have been shown to cause errors in integrated intensities of 50% in some cases. The results of this work are not complete and will be reported later.
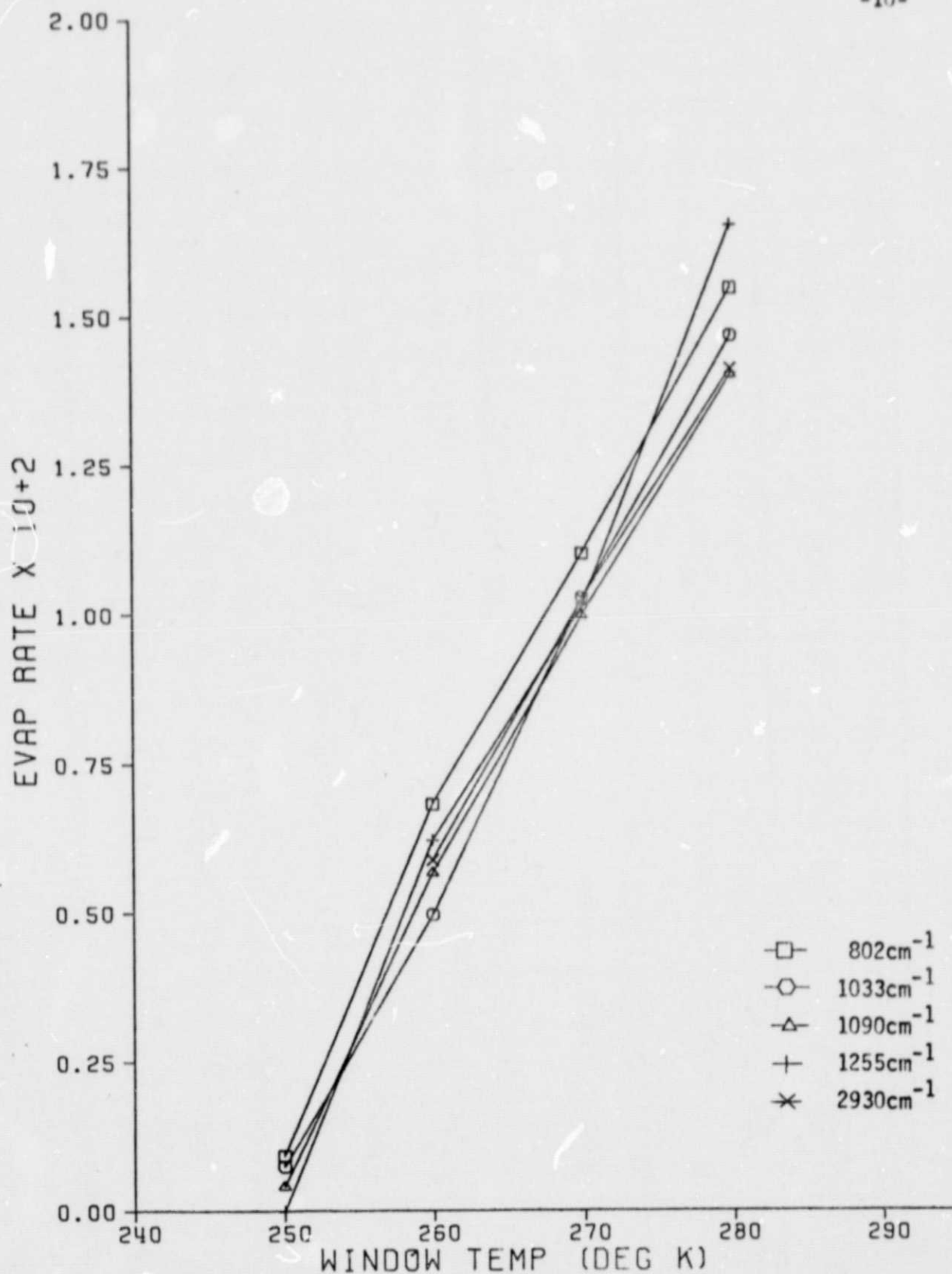
FIGURE 6: Evaporation rate versus temperature for outgassed material collected on CsI window (264 hour test).

III. WORK IN PROGRESS

A series of outgassing tests of S13G paint are being undertaken to further measure its outgassing rate as it is affected by changes in sample temperature, and to find the length of time required to completely outgas the paint. Further measurements will be made to determine the effect of ultraviolet radiation on the outgassing properties of the paint and on the evaporation rate and composition of the material deposited on the collecting window. This work will also be extended to encompass two siloxane polymers used as adhesives, RTV-560 and RTV-566.