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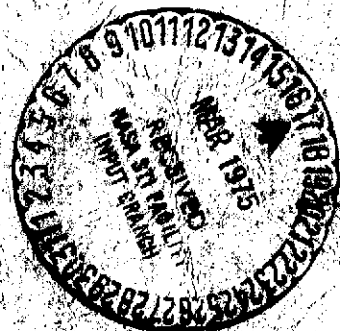
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Final Report

ANALYTICAL STUDY OF SPACECRAFT DEPOSITION CONTAMINATION BY INTERNAL REFLECTION SPECTROSCOPY

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FINAL REPORT
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ANALYTICAL STUDY OF SPACECRAFT DEPOSITION CONTAMINATION
BY INTERNAL REFLECTION SPECTROSCOPY

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ABSTRACT

The in-situ vacuum chamber designed around the IRE has been tested under vacuum. The contamination analyzer system now has enough energy output for in-situ contamination study. The system was tested using two liquid contaminant materials.

The analysis of the contaminants on the SL-4 rendezvous window shows that the material has methyl silicone, hydroxyl, and carbonyl radicals.

Also the analysis of the outgassing material from Shuttle TPS shows silicone as the primary product.

APPROVED:



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1. INTRODUCTION

The contamination of spacecraft due to material outgassing has been recognized as a problem from the beginning of the space program. The specific dangers arising from the presence of contaminants are

- The development of localized atmosphere consisting of cabin leakage products, fuel cell wastes, etc.
- The deposition and adhesion of both particulate matter and gaseous condensates upon sensitive optical, thermal control, and solar cell surfaces
- The interference, through light absorption and scattering, with signal reception.

For Skylab, the column density resulting from venting of water and outgassing products was found to be on the order of 10^4 and 10^{11} , respectively. The scattering due to contaminant cloud was rather high, and bright dust particles produced streaks on SO-52 films. The rendezvous window in the SL-4 mission was found to be badly contaminated.

Deposits of this nature on critical optical, thermal control, and solar cell surfaces of Shuttle and other space programs could adversely affect the very basis of the different experiments.

Plans are now under way to use RCS engines in the Shuttle. Both mono- and bi-propellant RCS engine plumes contain contaminants. Bi-propellant systems give rise to more contaminants, those consisting of water, mono-methylhydrazine hydrate, monomethylhydrazine nitrate, ammonia, ammonium nitrate, hydroxide, etc. All these can very adversely affect the optical surfaces through chemical reaction, abrasion, and deposition of absorbing and reflecting material.

Furthermore, photons in the ultraviolet region and solar protons and electrons possess sufficient energy to cause fission of chemical bonds of the contaminants. The resulting free radicals may react in a wide variety of ways, resulting in polymerization and cross-linking of a very complex nature and ultimately in the formation of a hard carbonaceous

glaze of indeterminate stoichiometry. The effect of ultraviolet irradiation on some representative contaminant materials has already been studied (Refs. 1 and 2). To locate and control the sources of contamination and to devise the appropriate cleaning technique, it is first necessary to identify the substances absorbed on the surfaces and to correlate these contaminants with substances detected in the cabin atmosphere.

The present study is concerned with the design and checkout of an in-situ chamber that will be used for identification of spacecraft connected contaminants and study of UV radiation effects on, and thermal kinetics of, these contaminants.

In addition, two samples supplied by the COR were studied. One was the contaminant found on the rendezvous window of the SL-4 mission; the other was the outgassed contaminant from a Shuttle TPS panel material that was tested in the MSFC Space Sciences Laboratory.

2. APPARATUS

The in-situ chamber and the optical layout are shown in Figure 2-1. The body of the cell consists of a stainless steel cylinder 9 centimeters in diameter and 5 centimeters high. The internal reflection element (IRE) is mounted in a stainless steel block suspended from the 1-centimeter-thick cylinder lid. Two long narrow stainless steel spacers hold the IRE away from the stainless steel block. A stainless steel pressure plate holds the IRE in position. This type of mounting keeps the IRE in good thermal contact with the block and minimizes the temperature gradient within the IRE.

The block is tapped through the lid to accept two heaters and a thermocouple. The thermocouple well is between the two heater wells. A vacuum seal between the lid and the cell body is established by compressing a Viton O-ring between the flat of the lid and the 1-centimeter-wide rim of the cell.

The infrared transparent windows are 1.5-centimeter-diameter KBr plugs with a 2.5-millimeter flat flange. The vacuum seal is made using Viton O-rings. The KBr windows protrude into the cell and leave a very small space between them and the end of the IRE, as shown in Figure 2-1. This design eliminates as much as possible the gas space between the windows and the ends of the IRE.

The chamber's vacuum port has a Leybold type flange. This is connected to the vacuum pump through a "T-connector", butterfly valve, and bellows type hose. The "T-connector" is used to allow electrical feed-throughs to be incorporated. These feed-throughs are necessary for the electrical connection to the quartz crystal microbalance (QCM) and the future installation of an electron gun for the study of electron radiation effects on the contaminants.

A small section of copper pipe with an elbow at one end and a needle valve at the other end is attached to the contaminant dosing port of the chamber to bleed in contaminant. All the joints in this assembly

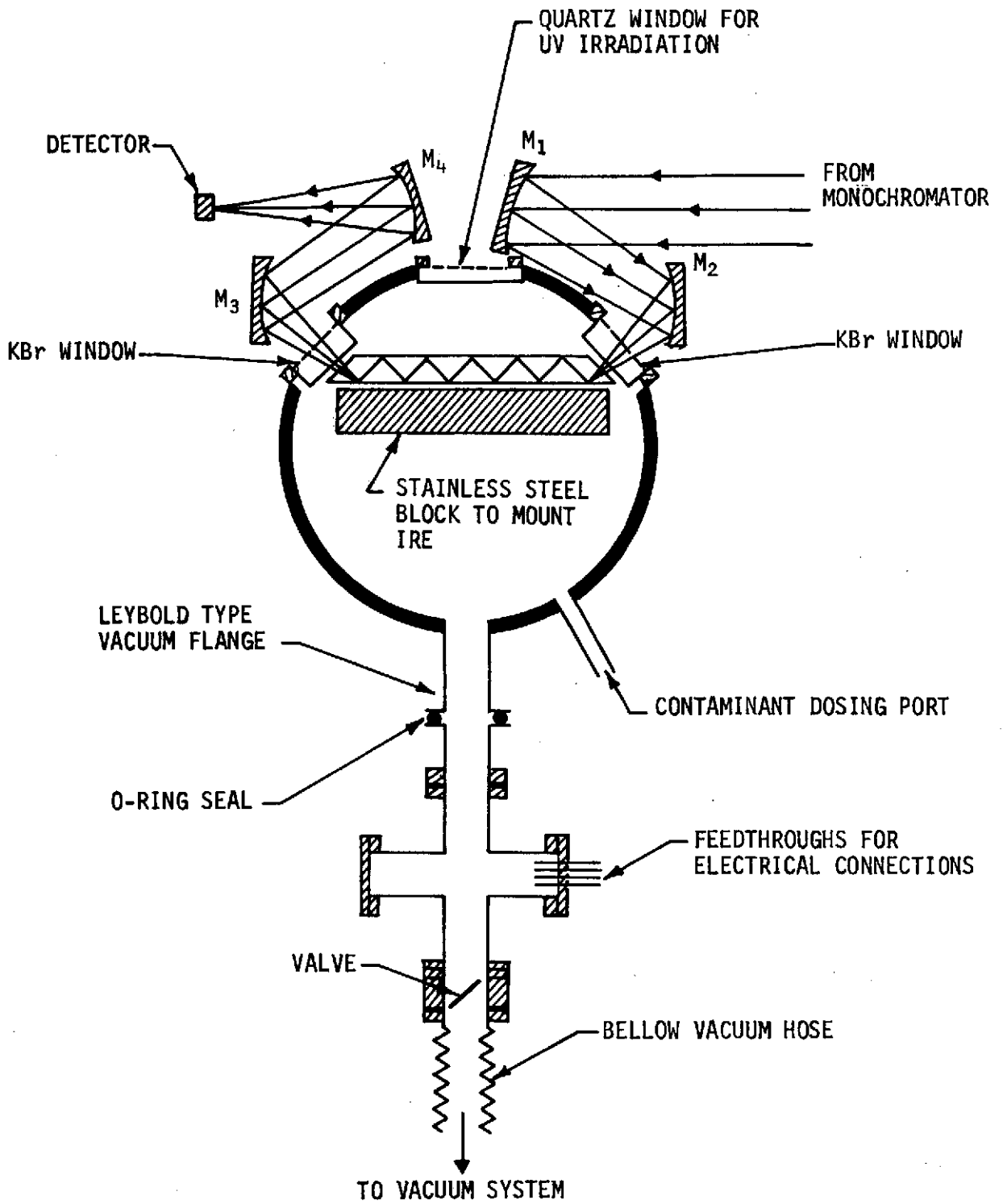


FIGURE 2-1. THE CHAMBER ALONG WITH OPTICAL LAYOUT

are either pressure-fitted or silver-soldered so that the assembly can be heated to drive the contaminants into the chamber.

With the in-situ chamber in the optical path of the Beckman Microspec monochromator, the output was found to be quite low. This was due to the additional mirrors and the KBr optical windows. It was therefore necessary to modify the electronics of the Microspec to improve the gain. The instrument now has enough gain to obtain a full-scale deflection on the strip chart recorder with the chamber in the optical path.

3. RESULTS

3.1 IN-SITU FACILITY

With the arrangement shown in Figure 2-1, it is possible to maintain a vacuum of 10^{-4} millimeters of Hg in the chamber. The output of the spectrometer with the vacuum chamber in its path was scanned between 2.5- and 14.5-micrometer wavelengths both before and after the gain improvement as shown in Figure 3-1. This shows that the Microspec now has enough output for use with the vacuum chamber for in-situ studies.

The operation of the contamination port was tested by contaminating the IRE with Dimethyl Phthalate and DC-704. The deposition was detected by monitoring the C=O band at 5.8 micrometers and the silicone band around 9.5 micrometers. The spectra of these two materials are shown in Figures 3-2 and 3-3. The weakness of the spectra is due to the fact that enough material could not be deposited on the IRE. The reason is that the contamination port opens on the back side of the IRE holder and the materials, being of very low vapor pressure, do not reach the front side. This shows that the less volatile material has to be in the line of sight to get deposition at a pressure of 10^{-4} millimeters of Hg and room temperature. To overcome this difficulty, the contamination inlet line has been extended within the chamber so that it opens toward the front side of the IRE.

3.2 CONTAMINANT SPECTRA

3.2.1 SL-4 Rendezvous Window Contaminant

This material was supplied by the COR after being peeled off the window using some "Torr-Seal" on a glass slide. The bonding of Torr-Seal with the contaminant was strong enough to peel it from the window. The contaminant with the glass slide backing was pressed against the IRE to obtain the spectra of the material. The measurement is shown in Figure 3-4. The double-hump band between 9 and 10 micrometers suggests

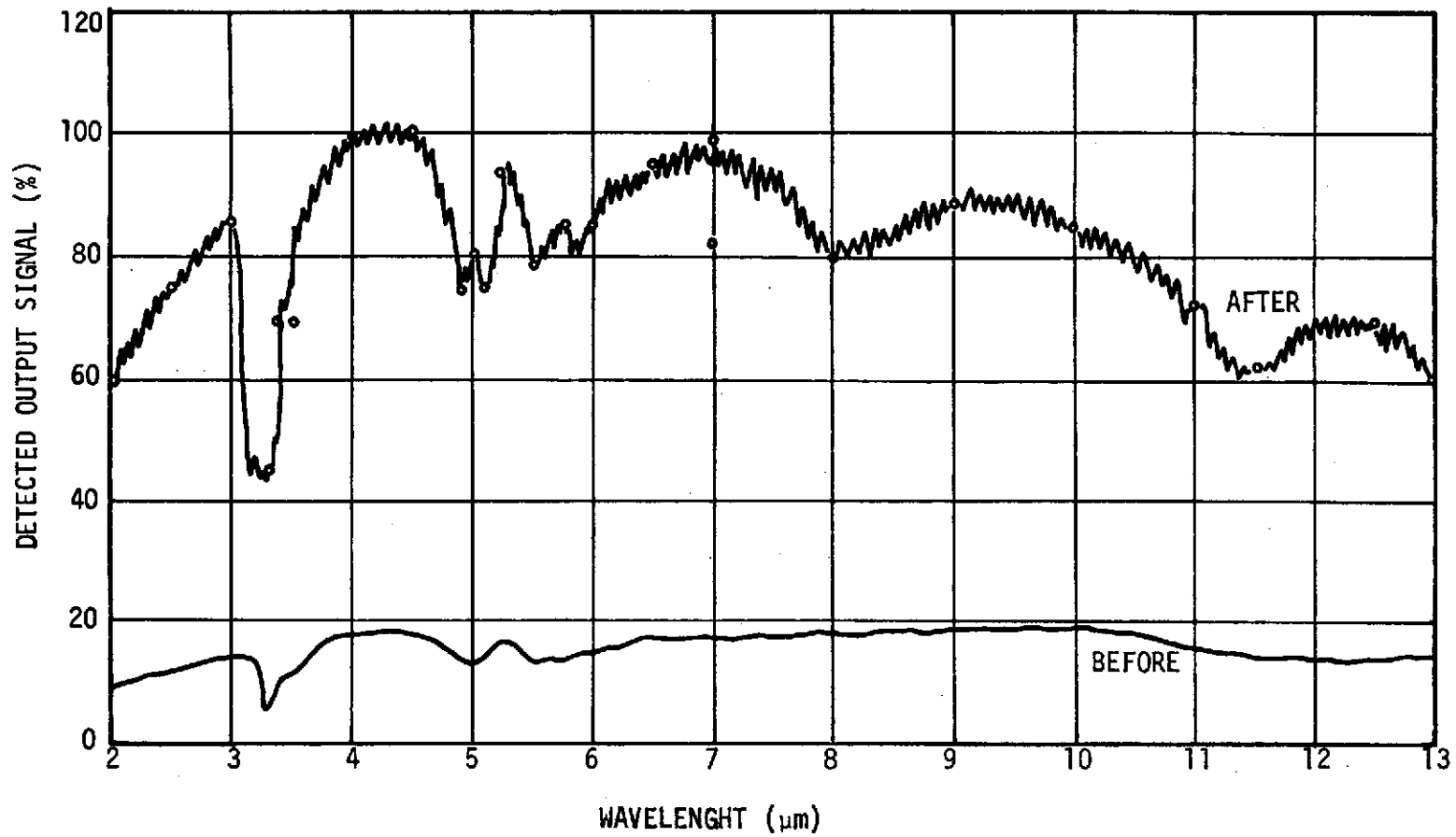


FIGURE 3-1. OUTPUT SIGNAL WITH CHAMBER IN OPTICAL PATH

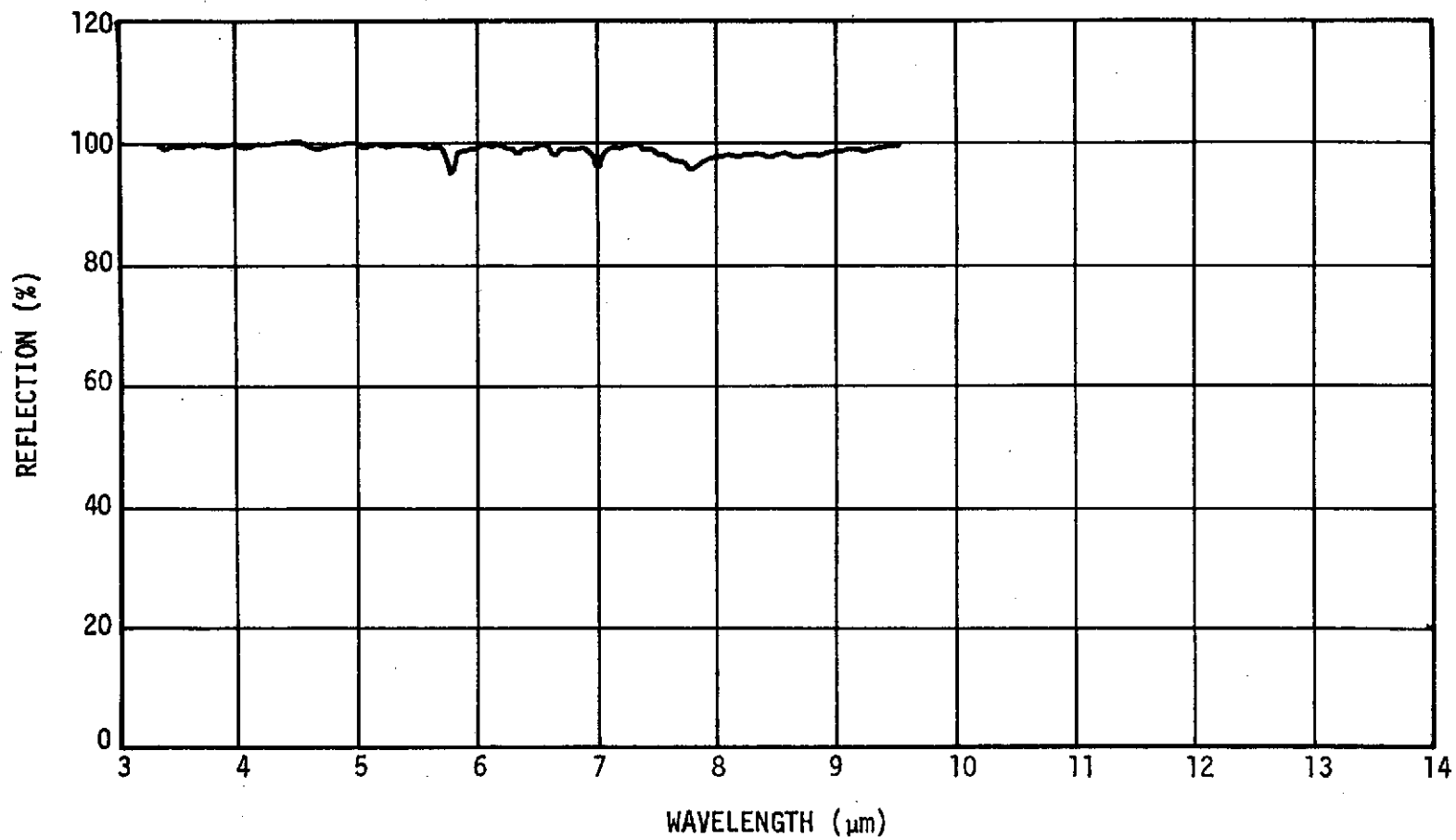


FIGURE 3-2. SPECTRA OF DIMETHYL PHTHALATE

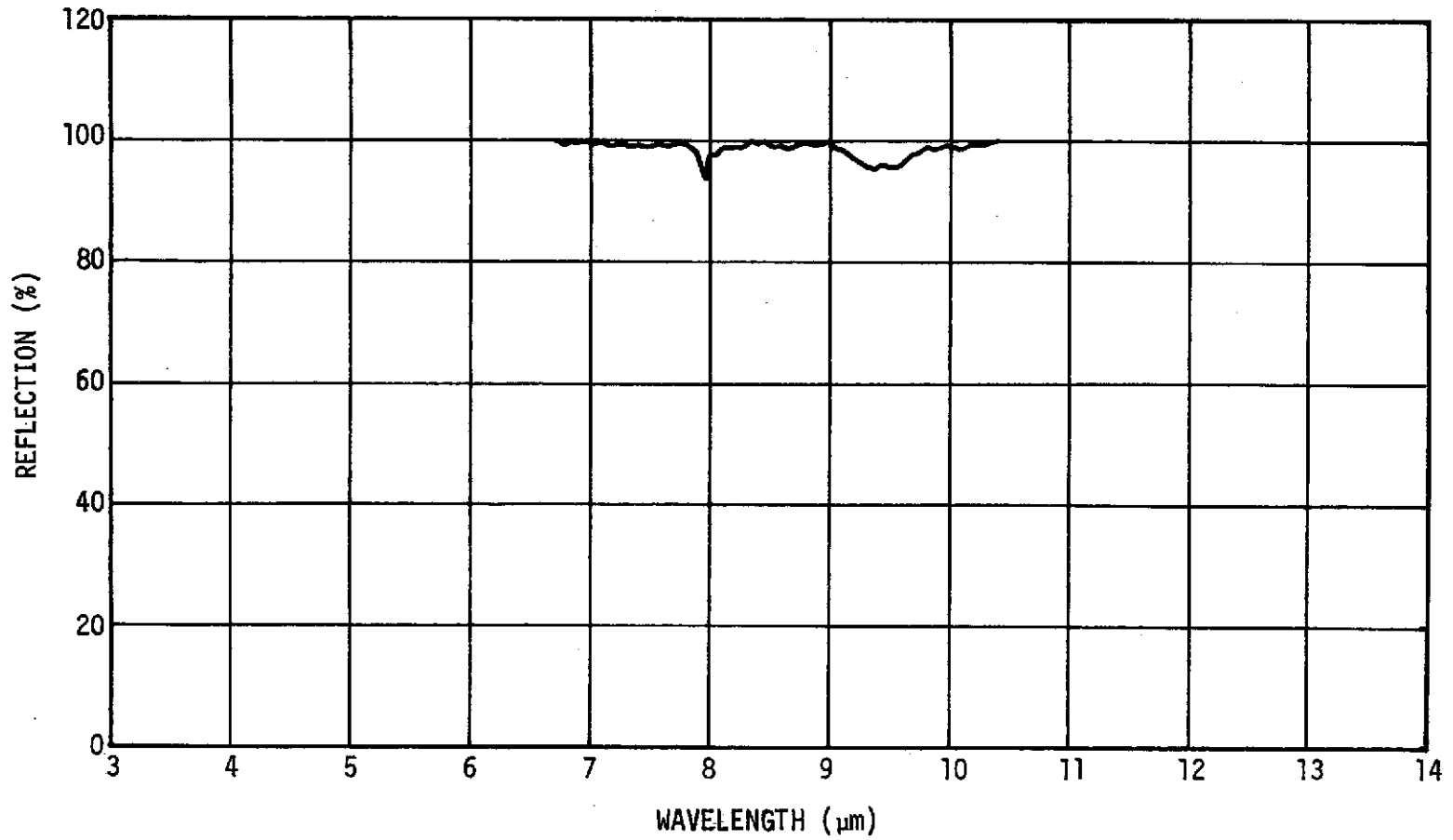


FIGURE 3-3. SPECTRA OF DC-704

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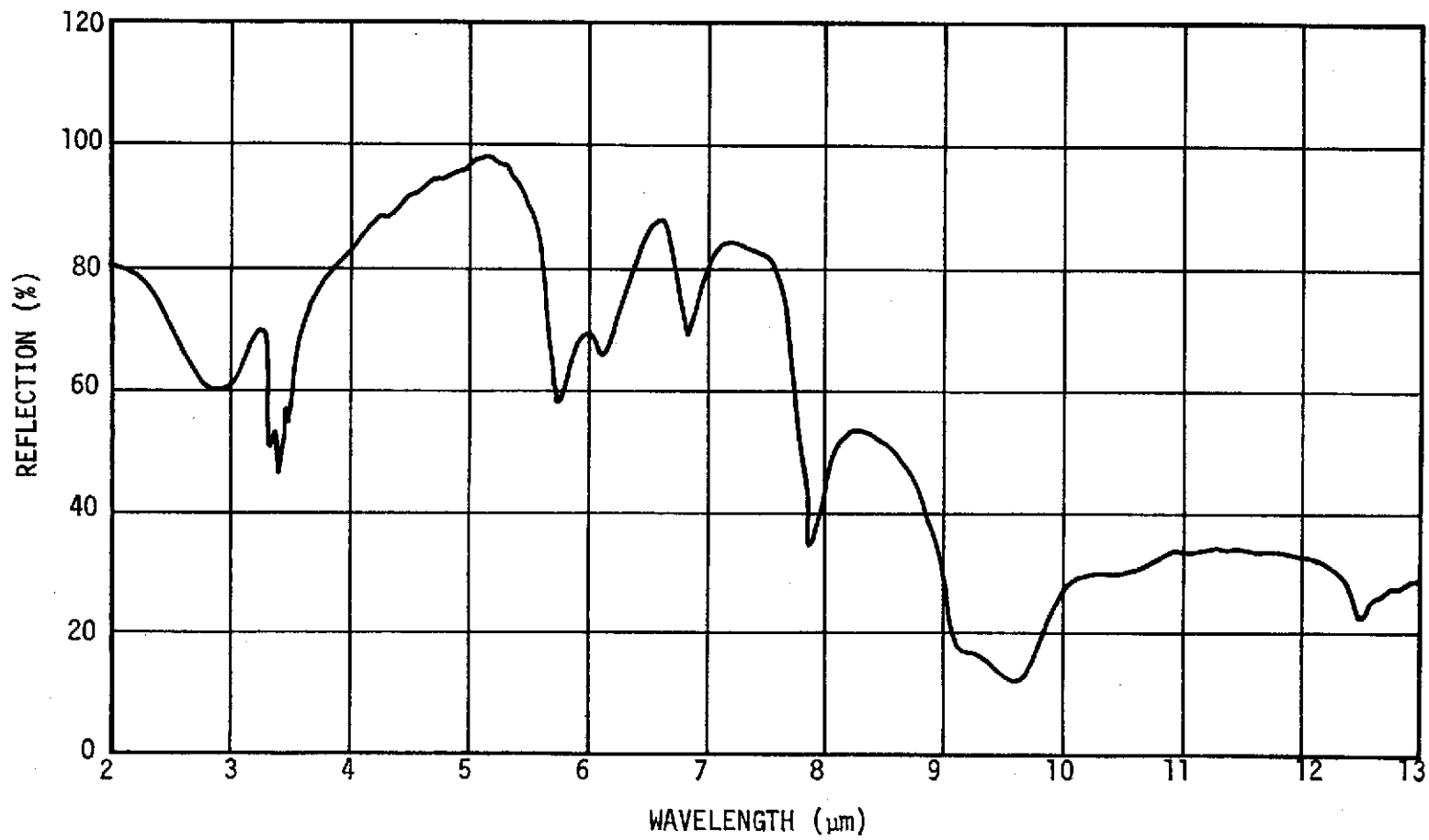


FIGURE 3-4. SPECTRUM OF SL-4 WINDOW CONTAMINATION

the presence of silonane. It appears that it is probably a methyl silicone, as the band around 7.8 micrometers is characteristic of the Si-CH₃ group. The band around 5.75 micrometers shows the presence of the C=O group. The broad band around 2.9 micrometers appears to be due to the OH group.

From this evidence, it appears that the contaminant is probably a methyl silicone along with another substance which has C=O and OH groups. It is to be noted that this material is the space radiation modified form of a contaminant which makes identification impossible until additional information on formation can be obtained.

3.2.2 Shuttle TPS Panel

The outgassed product from this panel was dissolved in acetone and was supplied through the COR. The panel had been tested in a vacuum chamber and the outgassed material contaminated the chamber wall from where it was washed down with acetone.

Upon settling the material showed some greyish-white sedimentation. Hence, two spectra were run: one, the film left from the clear solution after the acetone evaporated, Figure 3-5, upper curve; the other, the clear solution along with sediments, Figure 3-5, lower curve.

Both spectra are similar except for the bands at 6.5 and 11.1 micrometers. These two bands are absent in the clear solution spectra.

The double band around 3.5 micrometers is the characteristic of the CH₃ group. This could also be due to the CH₂ group, but the presence of the Si-CH₃ bands at 7.95, 11.95, and 12.5 micrometers excludes the possibility of the CH₂ group attached to Si-O. The double band between 9.0 and 9.5 micrometers is characteristic of Si-O-Si. In addition to those bands, both spectra show a band at 5.8 micrometers. Silicon compounds, in general, do not absorb at this wavelength. Aryl group and C=O show absorption in this region, but their association with the epoxy used in the panel is not clear at present.

As mentioned before, the sedimental part of the contaminant gives two additional bands. This means the sedimental part has some additional

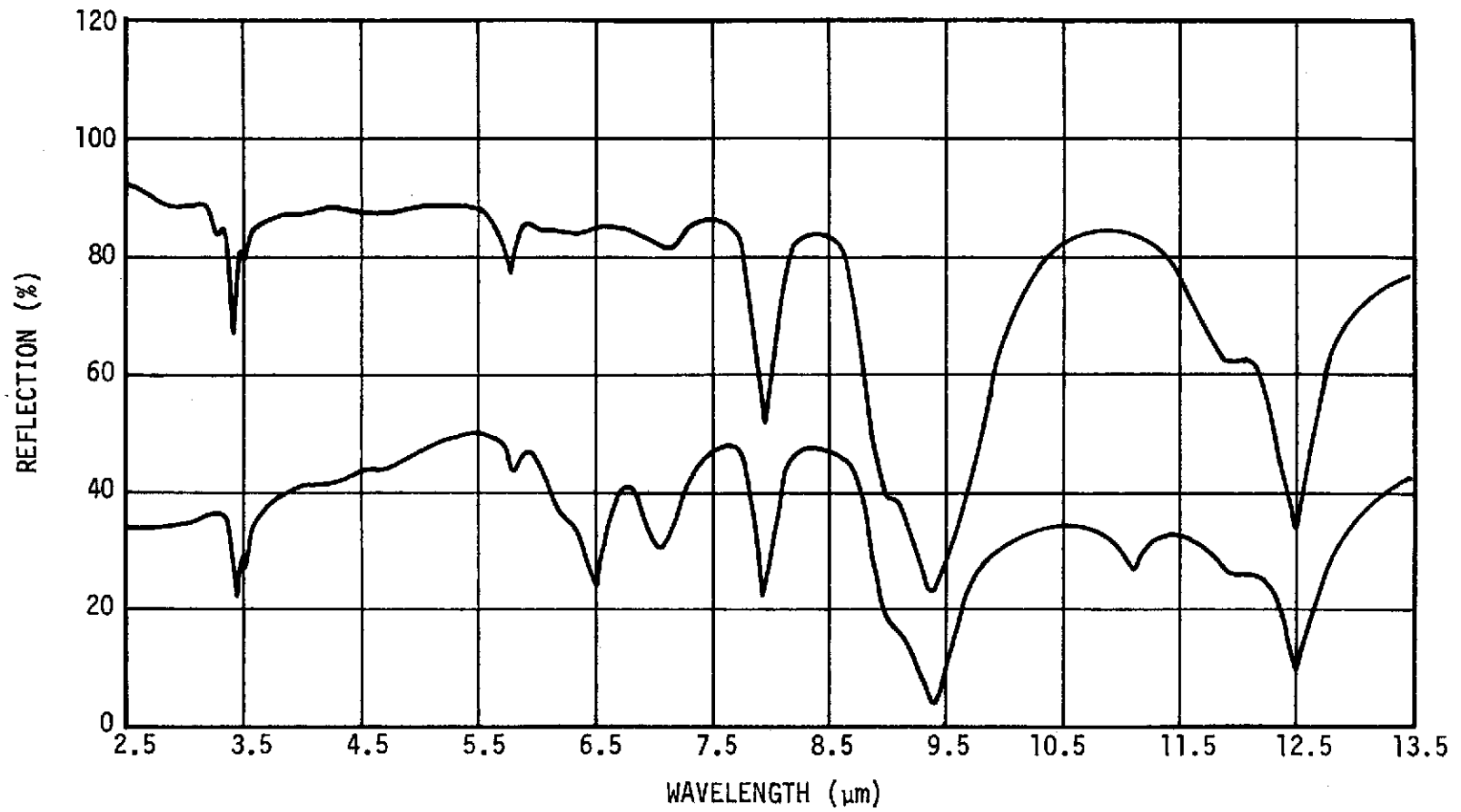


FIGURE 3-5. SPECTRA OF SHUTTLE TPS PANEL OUTGASSED PRODUCT

material. The band at 6.5 micrometers could be because of some kind of aromatic ring and the band at 11.1 micrometers is that due to the presence of Si-OH. It is possible that the sedimentation part formed because of simultaneous UV radiation and thermal heating of the panel material.

4. RECOMMENDATIONS

The major sources of contamination, as understood now, appear to be a combination of silonanes, "coolenol", and other complex organic materials. Presently, it is not possible to identify which one and/or combination is responsible for a particular problem and to what degree.

Moreover, no study seems to be available on the effect of solar radiation on these elemental contaminant materials. It is not known what kind of chemical changes, polymerization, fragmentation, etc., occur when these materials are irradiated with UV and/or electrons and protons.

The most appropriate approach to the understanding of contamination problems is to proceed systematically and study step-by-step in-situ the following for each elemental contaminant:

- Identify the IR spectrum of each material in the wavelength region of 2.5 to 14.5 micrometers
- Study the effect of UV radiation on them
- Examine thermal desorption kinetics of the material due to heating of the IRE
- Study changes produced in the thermal desorption kinetics due to UV irradiation of the material.

These studies will help understand contamination mechanisms and pave the way to the design and construction of a real-time ATR contamination monitor for space use.

5. REFERENCES

1. Mookherji, T., "Analytical Study of Spacecraft Deposition Contamination by Internal Reflection Spectroscopy", Teledyne Brown Engineering Final Report SE-SSL-1678, December 1972
2. Mookherji, T., "Analytical Study of Spacecraft Deposition Contamination by Internal Reflection Spectroscopy", Teledyne Brown Engineering Interim Report EE-SSL-1781, December 1973