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# Halogen Occultation Experiment (HALOE) Optical Witness-Plate Program

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## **Abbreviations and Acronyms**

ADC	analog-to-digital converter
BGA	biaxial gimbal assembly
CVM	condensable volatile material
DOP	dioctyl phthalate
FTIR	Fourier transform infrared
GEA	gimbal electronics assembly
HALOE	Halogen Occultation Experiment
MLI	multilayer insulation
PEA	platform electronics assembly
UARS	Upper Atmosphere Research Satellite

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## Summary

The Halogen Occultation Experiment (HALOE) will measure atmospheric concentration profiles of various gases from the Upper Atmosphere Research Satellite (UARS). The HALOE instrument has over 100 optical elements and a passive thermal control system with low-absorptivity radiators. Molecular contamination of the optical surfaces could affect measurement sensitivity and radiator performance.

An optical witness-plate program has been implemented to monitor the buildup of molecular contamination in the clean room during the assembly and testing of the HALOE instrument. "Travel" plates to monitor molecular contamination when the instrument is not in the clean room are also measured. The measurement technique is spectroscopy with high resolution in transmission in the 3- $\mu\text{m}$  spectral region using a Fourier transform spectrometer. Witness specimens of infrared transmitting material of low index of refraction are used for contaminant monitoring and for spectral signature analyses.

Spectral signatures of possible molecular contamination are presented. Small absorptions of trace hydrocarbons are present in most virgin infrared optical materials. The witness-plate measurements do not support dioctyl phthalate (DOP) accumulation indicated from residue of aluminum wash plates. No contamination by condensable volatile material (CVM) of HALOE optical witness specimens has yet been found.

## 1. Introduction

### 1.1. Purpose of HALOE

The Halogen Occultation Experiment (HALOE) is a satellite solar occultation experiment to measure, on a global basis, the concentration profiles of a number of trace gas constituents in the stratosphere (ref. 1). The species to be measured are  $\text{O}_3$ ,  $\text{HCl}$ ,  $\text{HF}$ ,  $\text{NO}$ ,  $\text{CH}_4$ ,  $\text{H}_2\text{O}$ ,  $\text{NO}_2$ , and  $\text{CO}_2$ .

The experiment includes the development of the HALOE instrument, the development of the necessary software packages and data reduction procedures, and the analysis of the data for science investigations by members of the HALOE science team.

The HALOE instrument is one of 10 atmospheric and solar instruments to be included on the Upper Atmosphere Research Satellite (UARS). The UARS program is dedicated to the scientific investigation of the Earth's stratosphere, mesosphere, and lower thermosphere.

### 1.2. HALOE Instrument

The HALOE instrument is a two-axis gimballed sensor that acquires the Sun, tracks the Sun through

the atmosphere, and scans the Sun during both an orbital sunrise and a sunset. The HALOE instrument contains three major subsystems—the optics unit, the biaxial gimbal assembly (BGA), and the platform electronics assembly (PEA)—as shown in figure 1.

The HALOE instrument is sensitive to contamination effects in the performance of its optics and its passive thermal control system. The radiators, shown in figure 2, are coated with a silver Teflon<sup>1</sup> film to provide for a beginning-of-mission absorptivity of 0.08 and an allowable end-of-mission absorptivity of 0.20.

The HALOE optics, which are shown schematically in figure 3, consist of 114 discrete elements which include mirrors, lenses, beam combiners and splitters, windows, filters, and detectors. All the optical components are attached to the optical bed with separate mounts that permit individual alignment. The majority of the optical elements are fabricated from germanium. Figures 4 and 5 show typical installations. The optical bed is fabricated from aluminum. A metal cover is designed to fit over the installed optical components in order to provide a dry nitrogen purge during ground testing and to provide protection during the orbital mission.

### 1.3. Instrument Contamination

Spacecraft contamination is generally divided into two classifications: particulate contamination and molecular contamination. Analyses have shown that the effects of particulate contamination are usually negligible, but the effects of molecular contamination during ground operations and in flight may exceed allowable limits unless special precautions are taken to keep radiators and optics clean prior to launch. Molecular contamination includes materials that condense, are transferred onto a surface from the surrounding environment, or migrate over a surface as a thin film. Contamination of optical surfaces may affect the transmittance or reflectance of optical surfaces and also cause scattering. Polymerization of molecular films is a particular hazard because it stabilizes and darkens the deposited contaminant that would otherwise gradually evaporate back off the surface. This could result in reduced measurement sensitivity for certain atmospheric species.

All metal hardware components were cleaned in an ultrasonic Freon<sup>2</sup> bath prior to Chemglaze<sup>3</sup> painting of the surfaces. Only low-outgassing organic materials (total mass loss less than 1 percent and contaminant condensable volatile material

<sup>1</sup> Teflon: Trademark of E. I. duPont de Nemours and Co., Inc.

<sup>2</sup> Freon: Trademark of E. I. duPont de Nemours and Co., Inc.

<sup>3</sup> Chemglaze: Trademark of Lord Corporation.

less than 0.1 percent) were used during the assembly of the instrument for potting and staking the components into place. The assembled instrument will receive two 25-hour exposures at 53°C during the test program in order to outgas and thermally stabilize the hardware components. This high-temperature exposure has been shown to further remove some of the molecular contaminants that can degrade measurement performance. The practice has been to purge the optic cavity with dry gaseous nitrogen at all possible times to further remove outgassed products and to prevent particulate deposition. The flat surface areas around the instrument and the non-optical surfaces of the instrument are regularly vacuumed to prevent a buildup of particles that can adhere to optical surfaces.

Two particular types of condensable volatile material (CVM) were suspected of being capable of changing the spectral transmission of the HALOE instrument over its long assembly, testing, and storage intervals. The types of CVM's of major concern were vacuum pump oil, because of the high incidence of pumps at the Langley Research Center, and dioctyl phthalate (DOP), which was indicated in the residue of aluminum wash plates in the HALOE clean room. It was also felt that a monitoring system should be used that would be able to detect CVM's of unsuspected origin.

#### 1.4. Optical Witness-Plate Program

The monitoring of CVM's relevant to the HALOE instrument was done by placing three sets of three witness specimens at three 90° orientations in the HALOE clean room as shown in figure 6. Transmission spectra of these nine witness specimens and one control specimen are nominally obtained every 6 months. When the HALOE instrument is removed from the assembly and testing clean room, two "travel" witness plates are placed in the immediate vicinity of the instrument. Transmission spectra of the plates were obtained before and after the HALOE instrument was at this location.

Additional witness plates have been doped with small amounts of suspected contaminant CVM's. The effect of planned bakeout of the instrument was measured for one suspected contaminant.

## 2. Measurement Requirements

### 2.1. Resolution in Transmission

The HALOE instrument uses both conventional optical-filter radiometers (O<sub>3</sub>, NO<sub>2</sub>, H<sub>2</sub>O, and CO<sub>2</sub>) and gas-filter correlation radiometers (NO, HCl, CH<sub>4</sub>, and HF). In the gas-filter concept, a specified

quantity of the gas of interest at a known pressure and temperature is placed in a correlation cell in one of two otherwise identical optical paths of the instrument. The full range of signal difference between the two similar channels, due to the absorption of the atmospheric gas of interest, could typically be of the order of 1 percent. This 1-percent gas-difference signal is amplified and digitized by a 12-bit ADC. Thus, the signal-measurement resolution or precision over the full dynamic range is of the order of 10<sup>-5</sup>. This high-measurement resolution can be interpreted as a 10<sup>-3</sup> transmission-measurement requirement over 10-cm<sup>-1</sup> intervals.

A change of 10<sup>-2</sup> percent in transmission in each of the optical elements of the HALOE instrument due to CVM's would result in a change in the measurement signal of the order of 1 percent since the CVM absorptions are accumulative along the optical path. Thus, the high-measurement resolution translates into a requirement for high sensitivity in detecting CVM's during the assembly, testing, and storage of the HALOE instrument.

### 2.2. Spectral Intervals

The spectral interval covered by HALOE optics is 1000 cm<sup>-1</sup> (10 μm) for the O<sub>3</sub> channel to 4100 cm<sup>-1</sup> (2.45 μm) for the HF channels. However, the continuous spectral interval is not of major importance since the width of the optical filters of each gas channel is 100 cm<sup>-1</sup> or less. The spectral regions of interest to HALOE, along with an emittance curve representative of the Sun, are shown in figure 7. An additional spectral selection effect is that the CVM's, being molecular, are expected to be spectrally selective. Three general types of materials are present in the HALOE instrument: (1) metals, which are the materials in the structural components, electrical circuits, and some optics; (2) salts and metal oxides, which are the pure materials of some optics; and (3) hydrocarbon and silicon-based plastics, which make up the wiring harnesses and are used for potting and staking, and alcohols and fluorocarbons, which are used in cleaning. The first two categories of materials do not significantly vaporize or outgas at the expected temperatures (-20°C to 80°C) experienced by HALOE. Thus, the CVM's, including propulsion products, are expected to be primarily hydrocarbons, fluorocarbons, and silicon-based materials. Water is a special case that is not expected to polymerize and hence will always evaporate back off a surface.

A survey of infrared absorption (ref. 2) indicates that most hydrocarbons have very strong absorption (from CH<sub>2</sub> and CH<sub>3</sub>) near 3000, 1450, and 1250 cm<sup>-1</sup> with very little absorption at 4000

and  $2000\text{ cm}^{-1}$ . Strong hydrocarbon absorptions are sometimes present at  $1600$  and  $1000\text{ cm}^{-1}$ , the other spectral regions of interest to HALOE.

A measurement feasibility study was performed to optimize CVM measurements in terms of sensitivity, signal-to-noise ratio, and repeatability. The signal-to-noise ratio at  $3000\text{ cm}^{-1}$  in the available Fourier transform infrared (FTIR) spectrometers is much greater than at  $1450$  and  $1250\text{ cm}^{-1}$ . Consequently, it was decided to use the  $3000\text{-cm}^{-1}$  absorption features as the fingerprint for measuring CVM's. That this was an appropriate choice was confirmed by the presence of  $\text{CH}_2$  fingerprints in virgin, optical-quality  $\text{Al}_2\text{O}_3$ ,  $\text{CaF}_2$ ,  $\text{MgF}_2$ ,  $\text{LiF}$ ,  $\text{SiO}_2$ ,  $\text{ZnS}$ ,  $\text{Si}$ , and  $\text{N}_2\text{Cl}$  windows used in the HALOE program (figs. 8 and 9). Silicon vacuum pump oil and low-outgassing potting compounds are seen to also have strong absorptions near  $3000\text{ cm}^{-1}$  in the doped witness plates.

The  $3000\text{-cm}^{-1}$  spectral region is the region in which the  $\text{HCl}$  and  $\text{CH}_4$  measurements are to be made, and thus this  $\text{CH}_2$  fingerprint region is the region of concern for two of the most important gas channels (fig. 8).

### 2.3. Measurement Technique

The measurement technique used to monitor CVM accumulation on witness plates was differential-infrared absorption spectroscopy. Two FTIR spectrometers were used early in the HALOE program to verify gas fill and gas stability of the HALOE gas cells (ref. 3). These spectrometers were operated to produce a single-beam sample spectrum and a single-beam background spectrum. The sample spectrum was then divided by the background (reference) spectrum to give a transmission of the sample. Much operational expertise has been acquired to meet the stringent requirements for gas optical-mass and pressure retrievals of the gas cell.

A second measurement program, the optical filter characterization program (ref. 4), also used these FTIR spectrometers. Very high measurement precision ( $10^{-5}$  in transmission) was a critical aspect of the out-of-band transmission measurements of the filter characterization program.

The gas-cell windows and optical filters were nominally 1-in-diameter optics. Since a 1-in. diameter is a standard commercially available size and is a convenient size for placing in the clean room and for inserting into the spectrometers, a 1-in. diameter was chosen for the diameter of the witness specimens. Figure 6 shows three 1-in-diameter witness specimens mounted on 2- by 3-in. aluminum plates.

Transmission spectra of the  $\text{Al}_2\text{O}_3$  windows of the gas cells had shown that optical channeling could

be severe (several percent transmission) in moderate and high refractive-index optical windows. Initially, uncoated germanium windows were used for clean room witness plates, but these windows had 2- to 15-percent channeling. Sample spectra showed this problem was not as severe with low-index  $\text{NaCl}$ ,  $\text{CaF}_2$ , and  $\text{LiF}$  windows. Since several virgin  $\text{NaCl}$  windows were on hand at the inception of the CVM monitoring program and since they are relatively inexpensive, they were chosen for clean room witness specimens and for doping with small amounts of suspected CVM's. Later needs in the program were "travel" plates to monitor CVM's when the HALOE instrument was not in the clean room and a relative humidity that was not as well-controlled; as a result,  $\text{CaF}_2$  windows were chosen for these "travel" witness specimens.

Initially, the witness-specimen spectrum was ratioed to the background spectrum, but as the measurements became more refined, the witness spectrum was ratioed to a spectrum of a control window of the same material. The control windows used for the background spectra were stored in a vessel with a gas purge from boiloff of  $\text{LN}_2$ . The clean room witness plates were periodically scanned at 6-month intervals, and the travel and environmental witness plates were scanned before and after any moving or test period.

## 3. Calibration Procedures

### 3.1. Virgin Plate Signatures

The first use of optical witness plates was during a cleanup of the 8- by 15-Foot Thermal-Vacuum Chamber at the Langley Research Center in 1982-83. However, these measurements were of low resolution in transmission (a transmission range plotted from 0 to 110 percent). During the measurement feasibility study of a method of monitoring CVM's for HALOE, it became apparent that  $\text{CH}_2$  trace contamination was present in most virgin optical material at about a 1-percent level in transmission. Figure 8 demonstrates a typical  $\text{CH}_2$  fingerprint of 0.2 to 0.3 percent in the two  $\text{Al}_2\text{O}_3$  windows of a gas cell. Figure 8 demonstrates a typical  $\text{CH}_2$  fingerprint in a  $\text{NaCl}$  window. The depth of the two  $\text{CH}_2$  absorption features at  $2850$  and  $2920\text{ cm}^{-1}$  would vary by as much as a factor of 2 between individual windows from the same batch. The absorption at  $2960\text{ cm}^{-1}$  results from the  $\text{CH}_3$  bond. The CVM monitoring became a measurement of the change in the  $\text{CH}_2$  fingerprint, not the appearance of a  $\text{CH}_2$  fingerprint.

The initial transmission spectra were plotted on a scale of 1-percent transmission per inch over a spectral range from  $2650$  to  $3100\text{ cm}^{-1}$  with a noise level

and reading uncertainty of the order of 0.1 percent in transmission. It became apparent in 1986 that greater precision was needed in reading the CH<sub>2</sub> signatures. An improvement in signature readability was obtained by increasing the number of scans collected and averaged into an interferogram from 16 to 256 scans and by changing the FTIR sample spacing (resolution) from 8 to 2. The change of sample spacing very effectively averaged out the small (0.1 percent in transmission) optical channeling of the NaCl window without decreasing the CH<sub>2</sub> signatures.

The FTIR optical bed (a 32- by 51- by 1-in. aluminum plate) is very sensitive to temperature. Experience with gas-cell spectra during the first year of operation of an FTIR spectrometer at the Langley Research Center showed approximately 1-percent apparent transmission variances caused by a 1°C temperature instability. The standard procedure evolved, in recognition of this temperature sensitivity, was to take a set of 256 scans of the sample witness plate, 256 scans of the reference witness plate, and a second set of 256 scans of the sample witness plate. As seen in figure 9, the two resulting witness-plate spectra always show nearly identical (to 0.01-percent transmission) CH<sub>2</sub> signatures even though there are significant (0.1-percent) transmission changes in reference-level transmission, source spectral brightness (slope of reference-level transmission), and water vapor (lines of frequency greater than 3000 cm<sup>-1</sup>).

### 3.2. Vacuum Pump Oil and Dioctyl Phthalate Signatures

The 8- by 15-Foot Thermal-Vacuum Chamber at Langley was equipped with diffusion pumps prior to 1982 when the facility was upgraded with cryogenic pumps. Dow Corning 704 (DC-704) silicone fluid (tetra methyl tetraphenyl trisiloxane) was the diffusion pump oil. A spectrum of a light smear of DC-704 on a NaCl specimen is presented as figure 10. The light smear shows several sharp absorption features beyond 2950 cm<sup>-1</sup> and only weak CH<sub>2</sub> absorption at 2850 and 2920 cm<sup>-1</sup>. The wiped smear was obtained by hard wiping of the light smear with a clean cloth to remove as much oil as possible. The background spectrum shows weak CH<sub>2</sub> absorption resulting from the optics of the interferometer. This background CH<sub>2</sub> absorption results from the optics of the interferometer and is removed from the specimen spectra by ratioing. Spectra were obtained from two NaCl witness plates placed at the front door and back door during a bakeout of the 8- by 15-Foot Thermal-Vacuum Chamber. No DC-704 pump oil was identified on the witness plates.

Many of the mechanical (roughing) pumps at Langley are filled with Duo-Seal<sup>1</sup> pump oil. A spectrum of a light smear of the hydrocarbon-based Duo-Seal oil is presented as figure 11. Strong CH<sub>2</sub> absorptions are present at 2850 and 2920 cm<sup>-1</sup>. Again, a wiped smear was obtained by hard wiping of the light smear with a clean cloth, and the background spectrum shows weak CH<sub>2</sub> absorption.

Dioctyl phthalate (DOP) has been used to test clean room air filters and is typically present in vinyl tubing, gloves, wire, insulation, and air-handling duct work. Monitoring of aluminum wash plates in the HALOE clean room yielded evidence of DOP in the clean room. There was a concern that over long periods (years) this material would condense on HALOE optics. A study was conducted in which two concentrations of DOP were placed on both NaCl and Al<sub>2</sub>O<sub>3</sub> specimens. The specimens were baked at several temperatures to simulate the planned bakeout of the HALOE instrument after final assembly. Table I lists the pertinent data of the four specimens. Spectra were taken of the specimens before doping, after doping, and after bakeout.

Figure 11 presents three spectra of specimen 34 (0.5 mg of DOP on NaCl) of this study. The 3000-cm<sup>-1</sup> spectra of DOP are quite distinctive in that there are four strong absorption peaks. In addition to the usual CH<sub>2</sub> peaks near 2850 and 2920 cm<sup>-1</sup>, there is a peak near 2865 cm<sup>-1</sup> and a very strong CH<sub>3</sub> peak near 2960 cm<sup>-1</sup>. The middle spectrum was obtained before doping with 0.5 mg of DOP. The top spectrum, plotted at 2-percent transmission per inch (right-hand scale ordinate), was obtained after doping, and the bottom spectrum was obtained after baking the specimen at 45°C for 4 days. Spectra from the other DOP specimens are consistent with those of figure 12. This study of bakeout of DOP was conducted before the measurement technique for high resolution in transmission was implemented. This is reflected in the different transmission scales of figures 9 and 12. This study confirmed that DOP has a very large signature at 3000 cm<sup>-1</sup>, and the scheduled bakeout of the HALOE instrument should cleanse the optics of any condensed DOP.

### 3.3. Other Contaminant Signatures

A set of 18 witness plates identical to the plates used to monitor CVM contamination for HALOE (fig. 6) were sent to the NASA Goddard Space Flight Center for deposition of known amounts of known materials. The amounts and materials are presented

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<sup>1</sup> Duo-Seal: Trademark of Sargent-Welch Scientific Company.

in table II. The materials were deposited in an 18-inch diameter vacuum system pumped by a triode pump. The materials were mounted in a heated effusion cell, and the vaporized material evolved from the effusion cell through a 0.375-in. hole in the lid. A witness plate was mounted to a water-cooled base facing the effusion cell. A quartz crystal microbalance was mounted adjacent to the witness plate to monitor the deposition onto the witness plate, and a residual gas analyzer was mounted to the vacuum system to monitor the mass peaks of the material vapors.

The CH<sub>2</sub> spectral signatures increase in depth with increasing thickness for four of the deposited materials as seen in column 6 of table II. General Electric RTV 142 material and Cohrlastic material (manufactured by Connecticut Hard Rubber Company) do not appear to have CH<sub>2</sub> signatures that correlate well with deposit thickness. This may result from different rates of evolution of volatile components in the sequential heating of the material to obtain the three different deposit thicknesses on three different witness plates. Since the same Cohrlastic specimen was heated sequentially for the three different thickness samples, differences in the composition of the CVM's that evolved from the Cohrlastic material as a function of heating history probably contribute to the nonlinear correlation of deposit thickness with absorption depth. It is noted that the Cohrlastic signatures would correlate well if the order of increasing deposition were for plates 27, 22, and 29. Three of the materials (General Electric RTV 566, RTV 142, and Cohrlastic) have strong CH<sub>3</sub> absorption. Spectra of two of the materials, Solithane<sup>1</sup> 3 and RTV 566, are presented as figures 13 and 14, respectively.

## 4. Results

### 4.1. Clean Room Witness Plates

Ten (nine exposure and one control) 1-inch diameter NaCl windows mounted in 2- by 3-inch specimen holders were placed at three designated locations in the HALOE clean room used for instrument assembly and testing. These witness plates were scanned with a FTIR spectrometer in August 1985, ratioed to a background spectrum, and plotted on a scale of 1-percent transmission per inch. When these plates were rescanned a year later, no indication of CVM deposition was seen. However, this result was in disagreement with CVM results obtained by washing large aluminum plates with a solvent (chloroform) and allowing the solvent to evaporate, after which

the residue was examined with a low-resolution FTIR spectrometer.

A measurement optimization study was conducted in order to improve the sensitivity and resolution-in-transmission measurements of the clean room witness specimens. The number of interferometer scans was increased to 256 per spectrum, the sample spacing (effective spectral resolution factor) was reduced by a factor of 4 to smooth channeling and noise, and the witness specimens were ratioed to a NaCl reference window maintained under a gas purge from boiloff of LN<sub>2</sub>. The resulting clean room witness spectra are then plotted at 0.1-percent transmission per inch, the same scale as that in figure 8. The clean room specimens have been scanned 1 year and 1-1/2 years since this improved measurement technique was adopted, and no indication of CVM's has been seen on the clean room specimens. However, noticeable lint is observed on the windows that are horizontal in the clean room. The clean room witness plates will continue to be scanned at 6-month intervals during use by HALOE.

High confidence in the FTIR investigation of DOP in the HALOE clean room is provided by (1) the excellent repeatability of 3000-cm<sup>-1</sup> spectral signatures of the witness plates resulting from the infrared optical windows, (2) the strength and character of the doped and control specimen spectra, (3) the direct nature of the contamination measurements of the optical witness plate, and (4) the correlation with visual examination.

A possible explanation for the discrepancy between the wash plate results and the optical witness-plate results is that there may be trace amounts of DOP, or a similar hydrocarbon, in the plate wash or introduced from some other aspect of the handling of the wash plates.

### 4.2. "Instrument-Build" Environmental Witness Plates

The HALOE instrument development program used one set of structural components (mainframe) for both the engineering validation and the flight instrument. The "instrument-build" (engineering validation) HALOE was assembled and tested in 1986 and 1987. The instrument-build HALOE was then partially disassembled in the latter part of 1987, and replacement optic and electronic components were installed during early 1988.

The instrument-build HALOE was placed in the 8- by 15-Foot Thermal-Vacuum Chamber at Langley for two periods of several weeks (July 1986 and April 1987). A Sun-look test of several days was also conducted during this period.

<sup>1</sup> Solithane: Trademark of Morton-Thiokol, Inc.

Two NaCl witness specimens were mounted near the fore and aft ends of the Thermal-Vacuum Chamber during the first test. During this test a coolant line ruptured, with the result that the relative humidity reached 100 percent for an extended period of time and the NaCl specimens were destroyed.

Two NaCl witness specimens were also mounted near the fore and aft ends of the Thermal-Vacuum Chamber during the second test. After comparing the before-and-after test-specimen spectra, it was concluded that there was no CVM contamination from the thermal-vacuum test at the 0.01-percent transmission level.

Two NaCl witness specimens were hung in the immediate vicinity of the instrument-build HALOE during Sun-look tests in 1987. No CVM contamination of the plates was noted in comparing the before-and-after spectra. Numerous small, black particles were observed on the witness specimens after this test. These particles are suspected to be soot particles from the nearby heating plant.

#### 4.3. Flight-Instrument Travel Plates

The HALOE flight instrument began testing outside the clean room during mid-1988. Two CaF<sub>2</sub> witness specimens (travel plates) have been mounted in holders and fastened inside the HALOE instrument. These specimens were measured prior to installation and will be remeasured at appropriate times during the testing and moving of the HALOE instrument. CaF<sub>2</sub> specimens are used because they will not degrade during brief periods of high humidity (i.e., greater than 50-percent relative humidity). Various other witness specimens will be placed in the shipping containers and immediate vicinity of HALOE during these tests and operations as required.

#### 5. Concluding Remarks

A program has been developed and initiated to monitor the accumulation of condensable volatile material (CVM) on optical surfaces of the Halogen Occultation Experiment (HALOE) instrument. This program is based on measurements of high sensitivity and high resolution in transmission of hydrocarbon

absorptions in the 3000-cm<sup>-1</sup> region of the infrared spectrum. A Fourier transform infrared (FTIR) spectrometer is being used as the measuring device. A transmission-measurement resolution of 0.01 percent is obtained by optimized scanning and processing of interferograms and by the use of reference witness specimens. Low-index-of-refraction, infrared transmitting windows (NaCl and CaF<sub>2</sub>) are used as witness specimens and "travel" plates for CVM monitoring.

Calibration specimens and control specimens are an integral part of the program. Large absorptions from vacuum pump oils and other suspected CVM's are seen in the doped witness specimens in the 3000-cm<sup>-1</sup> spectral region. The scheduled bakeout of the HALOE instrument is shown to be effective in removing dioctyl phthalate (DOP), a CVM suspected in the HALOE assembly and testing clean room. The optical witness-plate measurements do not indicate the accumulation of DOP on surfaces in the HALOE clean room and, hence, do not support accumulation indicated from residue of aluminum wash plates. Small (1-percent) CH<sub>2</sub> absorptions are in most infrared optical materials. No CVM contamination of HALOE witness specimens of 0.01 percent (10<sup>-4</sup>) or greater in transmission have yet been found.

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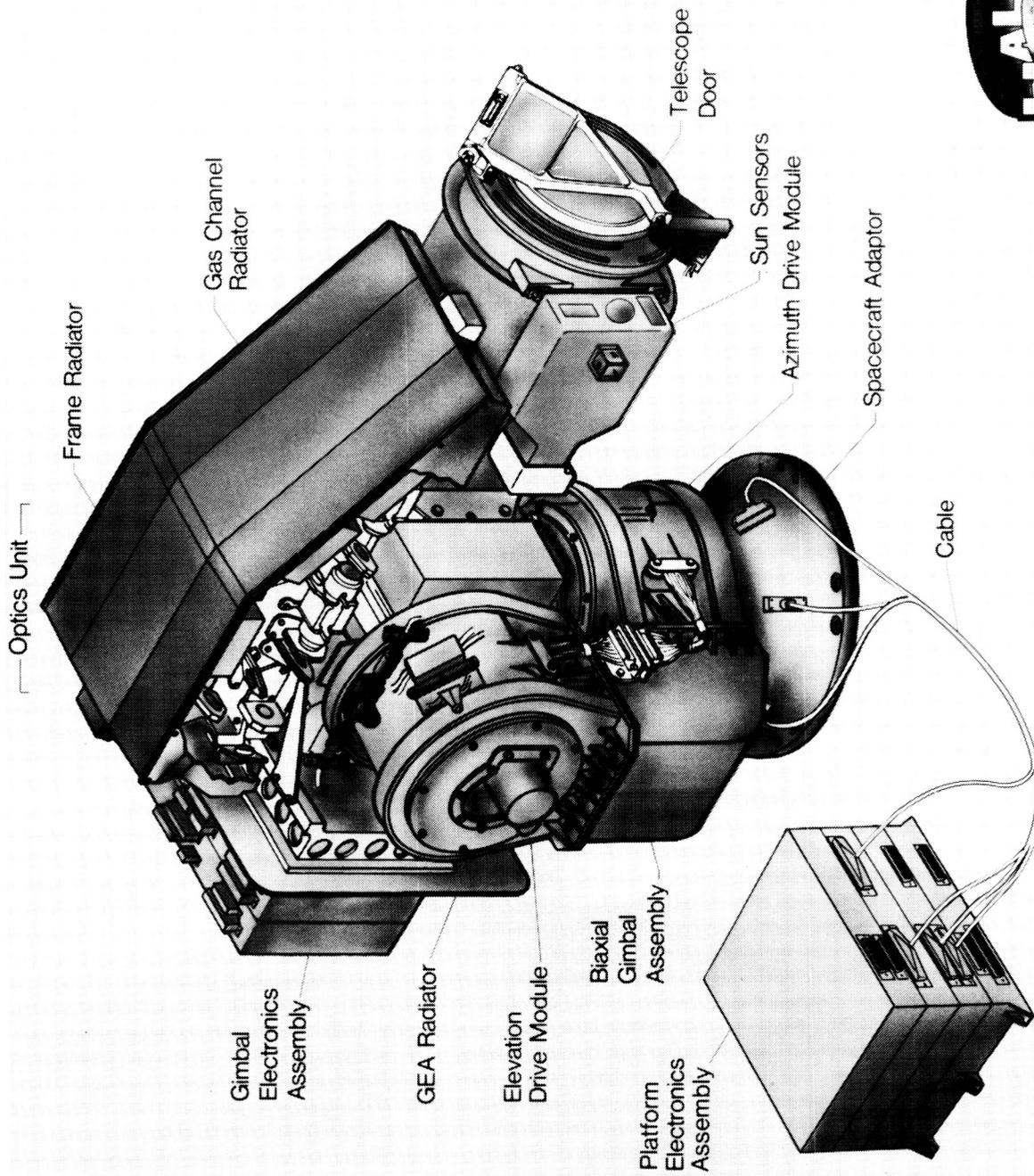
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Table I. DOP Bakeout Study

Specimen	Temperature, °C	Bakeout duration, days	Media	DOP concentration, mg	CH <sub>2</sub> (2929-cm <sup>-1</sup> ) transmission dip		
					Preexposure, percent	Postexposure, percent	Postbake, percent
35	50	7	NaCl	1.0	0.15	17.50	0.36
40	50	7	Sapphire	.5	.14	5.14	.16
34	45	4	NaCl	.5	.21	5.48	.32
41	40	4	Sapphire	1.0	.14	19.25	.15

Table II. Effect of Materials and Amounts in Contamination

Witness	Material	Thickness, Å	CH <sub>2</sub> (2920-cm <sup>-1</sup> ) transmission dip		Transmission change, percent
			Baseline, percent	Postdope, percent	
033	RTV 566	57	0.36	0.68	0.32
016	RTV 566	109	.55	.94	.49
021	RTV 566	189	.34	1.27	.93
031	Solithane 4	63	0.32	0.81	0.49
017	Solithane 4	124	.42	1.13	.71
028	Solithane 4	222	.47	1.58	1.11
018	Solithane 3	77	0.34	1.03	0.69
032	Solithane 3	147	.34	1.50	1.16
026	Solithane 3	336	.73	3.04	2.31
025	RTV 142	75	0.53	0.75	0.22
019	RTV 142	137	.54	.90	.36
020	RTV 142	233	.65	1.02	.37
029	Cohrlastic	57	0.31	0.63	0.32
027	Cohrlastic	129	.46	.54	.08
022	Cohrlastic	213	.34	.54	.20
024	Hysol EA 956	67	0.36	0.69	0.33
023	Hysol EA 956	102	.40	1.09	.69
030	Hysol EA 956	219	.71	1.60	.89



3-10-86

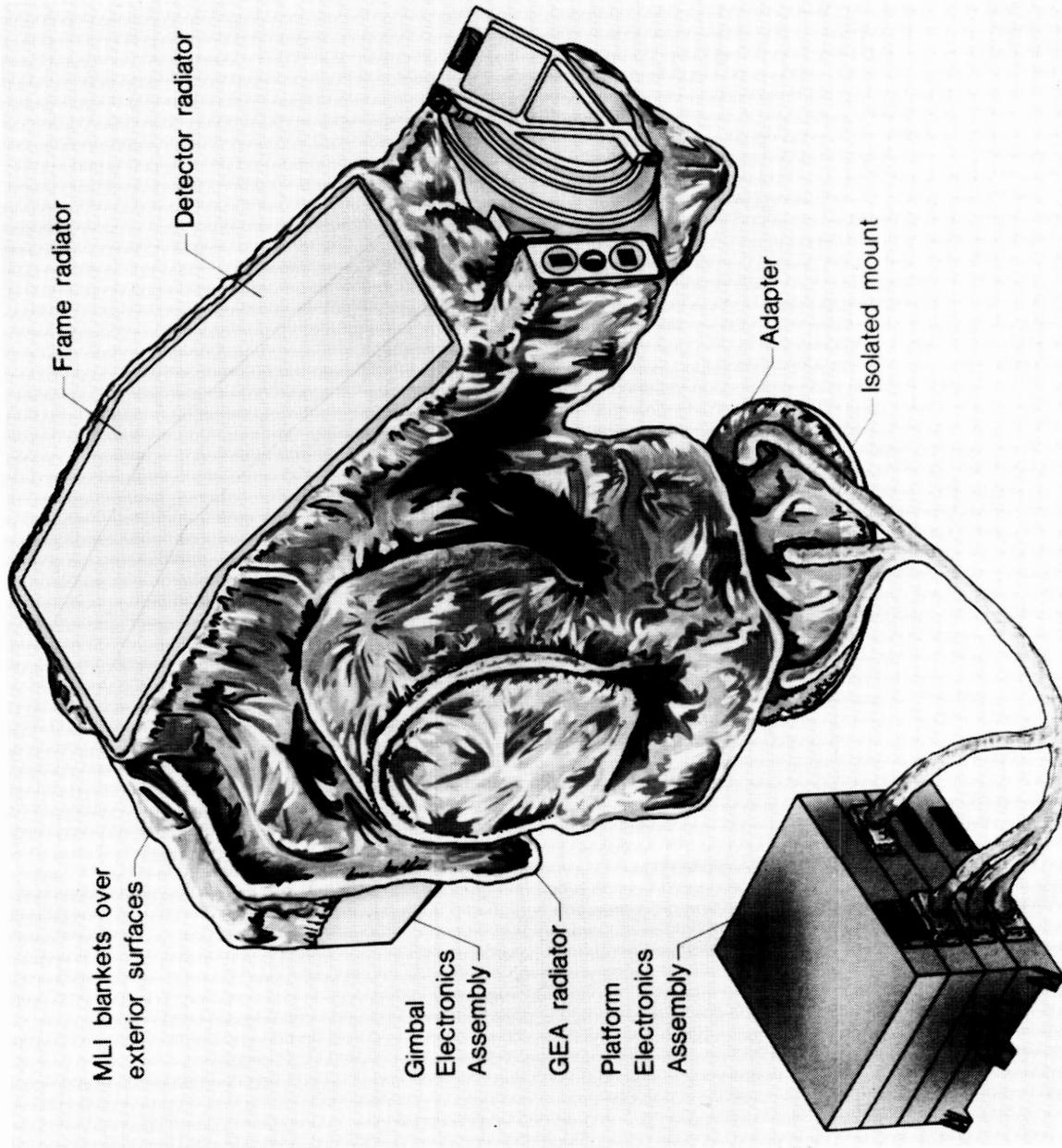


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Figure 1. The HALOE instrument.

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Figure 2. Radiators and thermal blankets of HALOE.

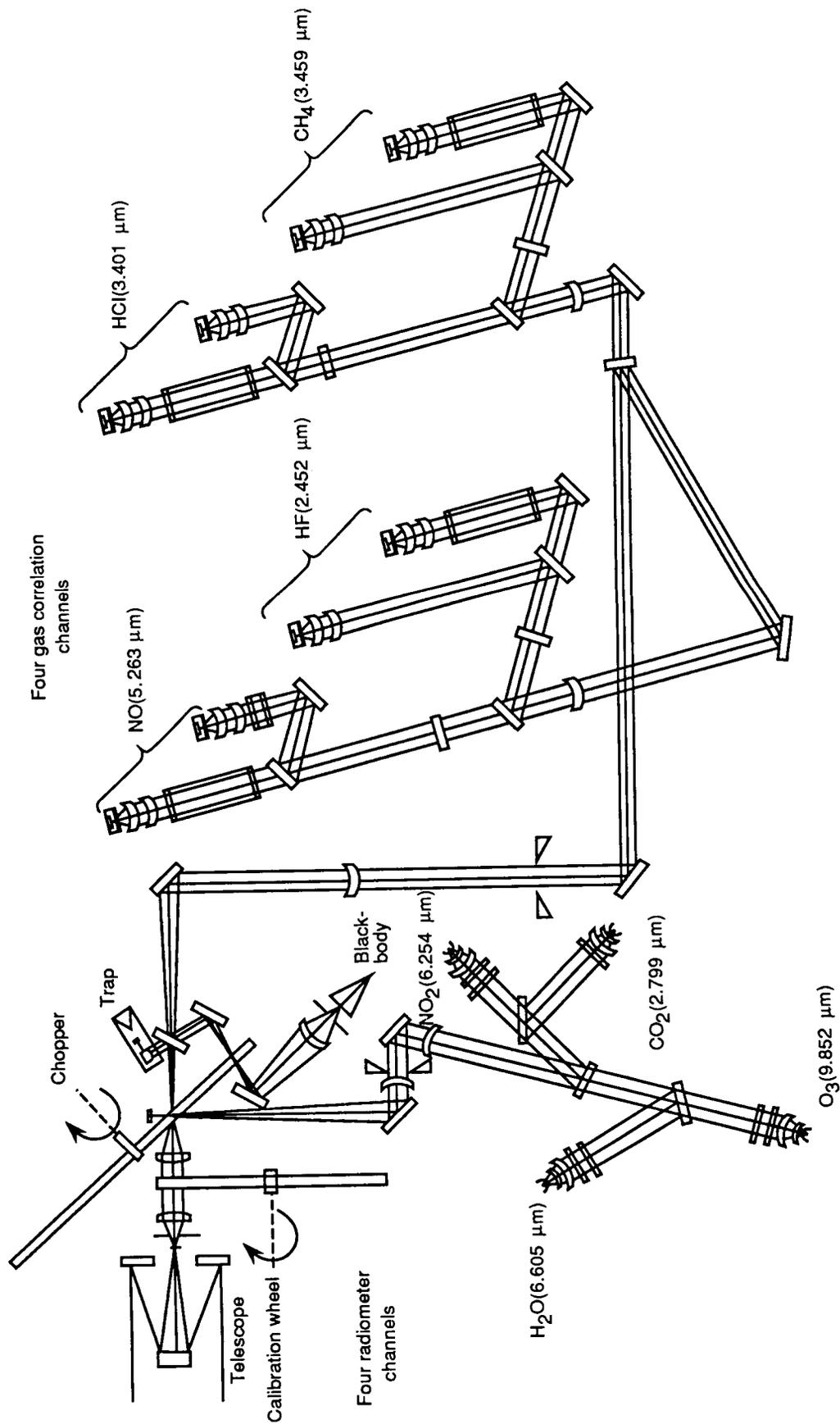
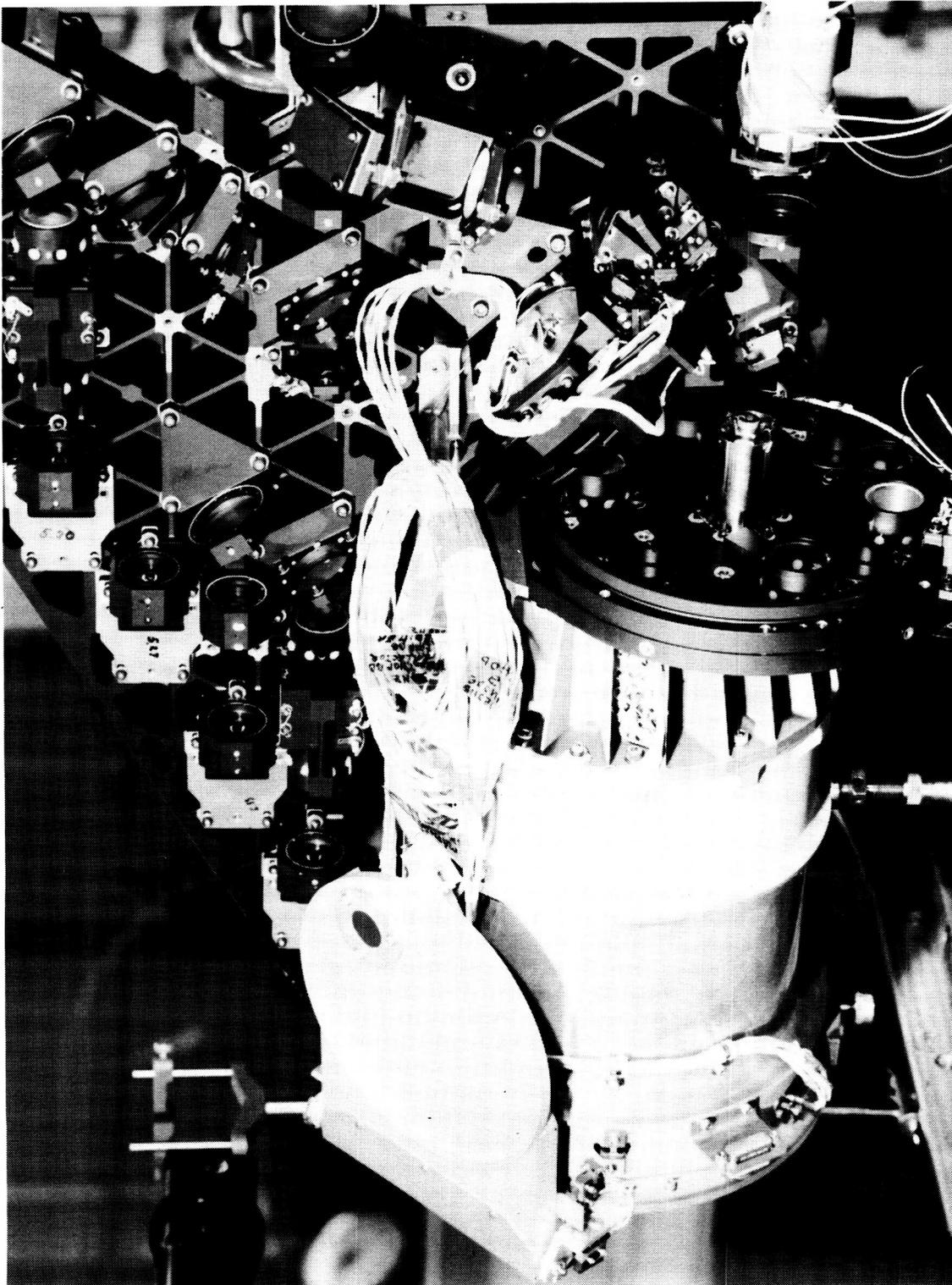


Figure 3. Optical schematic of HALOE.



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Figure 4. Optical bed assembly of HALOE.

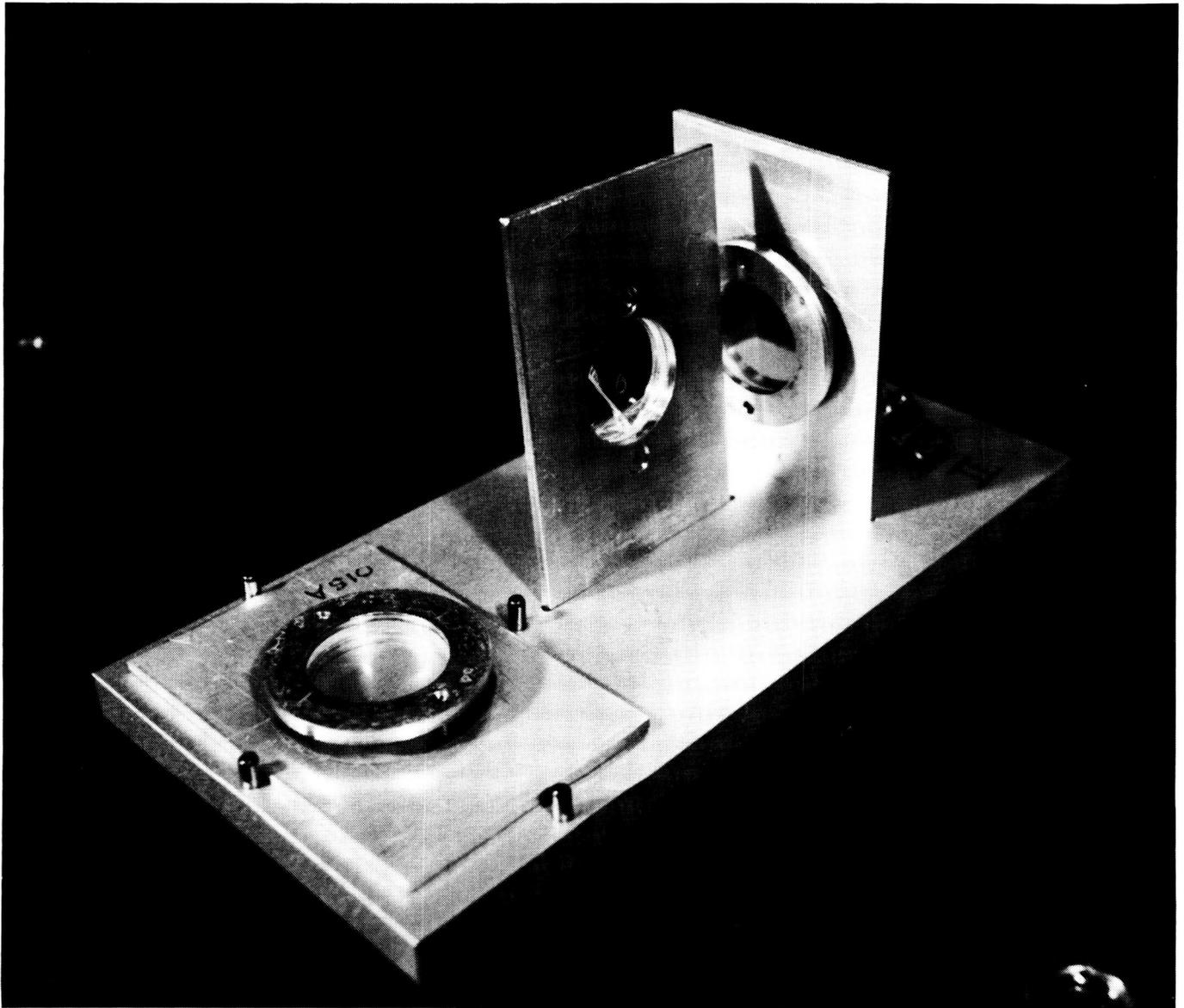
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Figure 5. Radiometer-detector assembly of HALOE.

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Figure 6. One-inch-diameter witness specimens mounted on 2- by 3-in. aluminum plates.

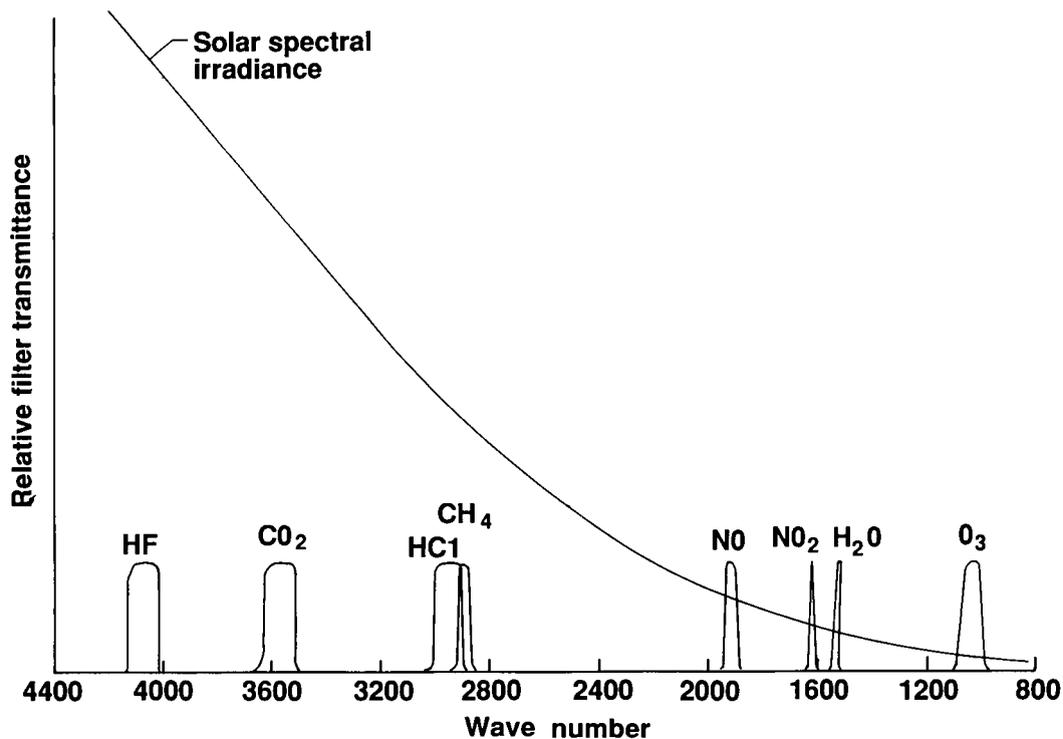


Figure 7. Spectral intervals of HALOE.

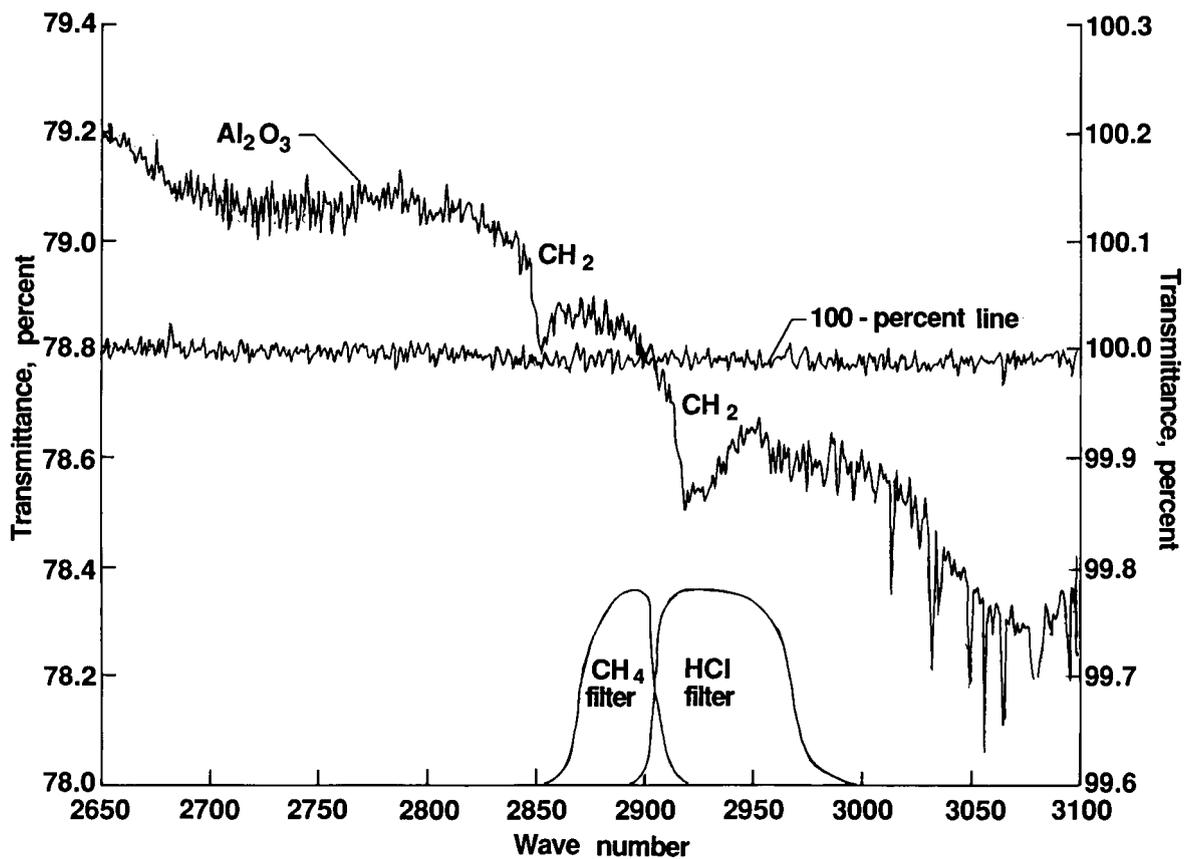


Figure 8.  $\text{CH}_2$  absorptions in  $\text{Al}_2\text{O}_3$  windows of a HALOE gas cell. The 100-percent line corresponds to the right ordinate.

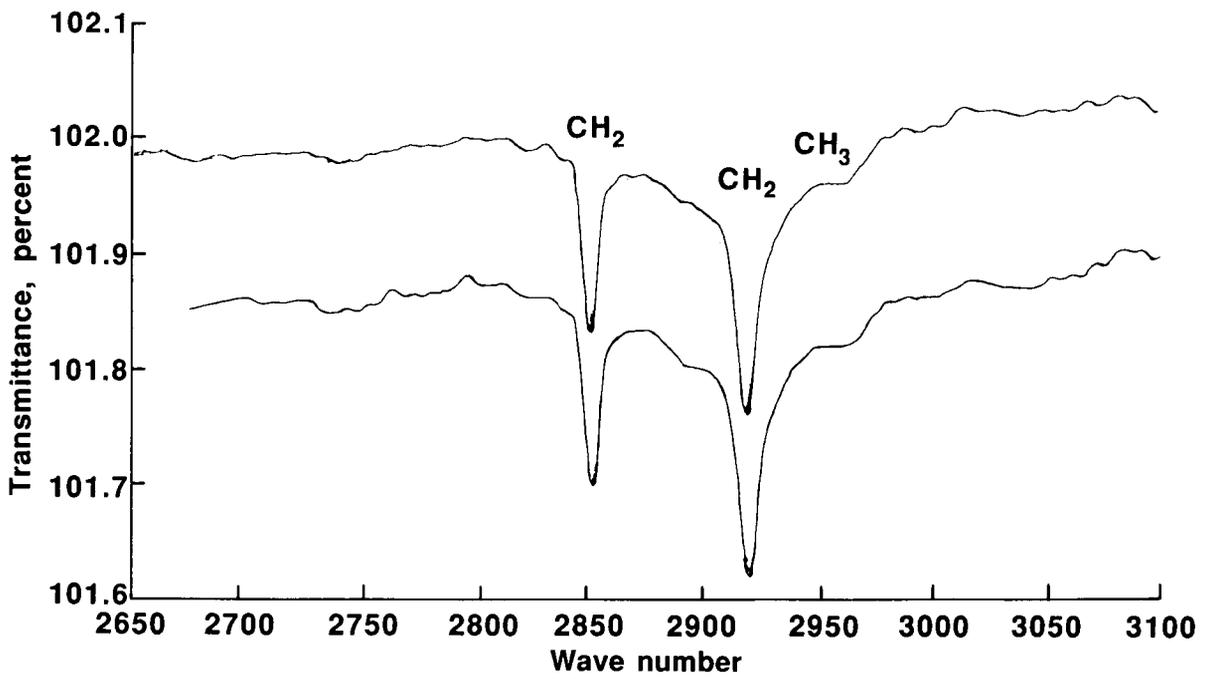


Figure 9. CH<sub>2</sub> absorptions in a NaCl specimen.

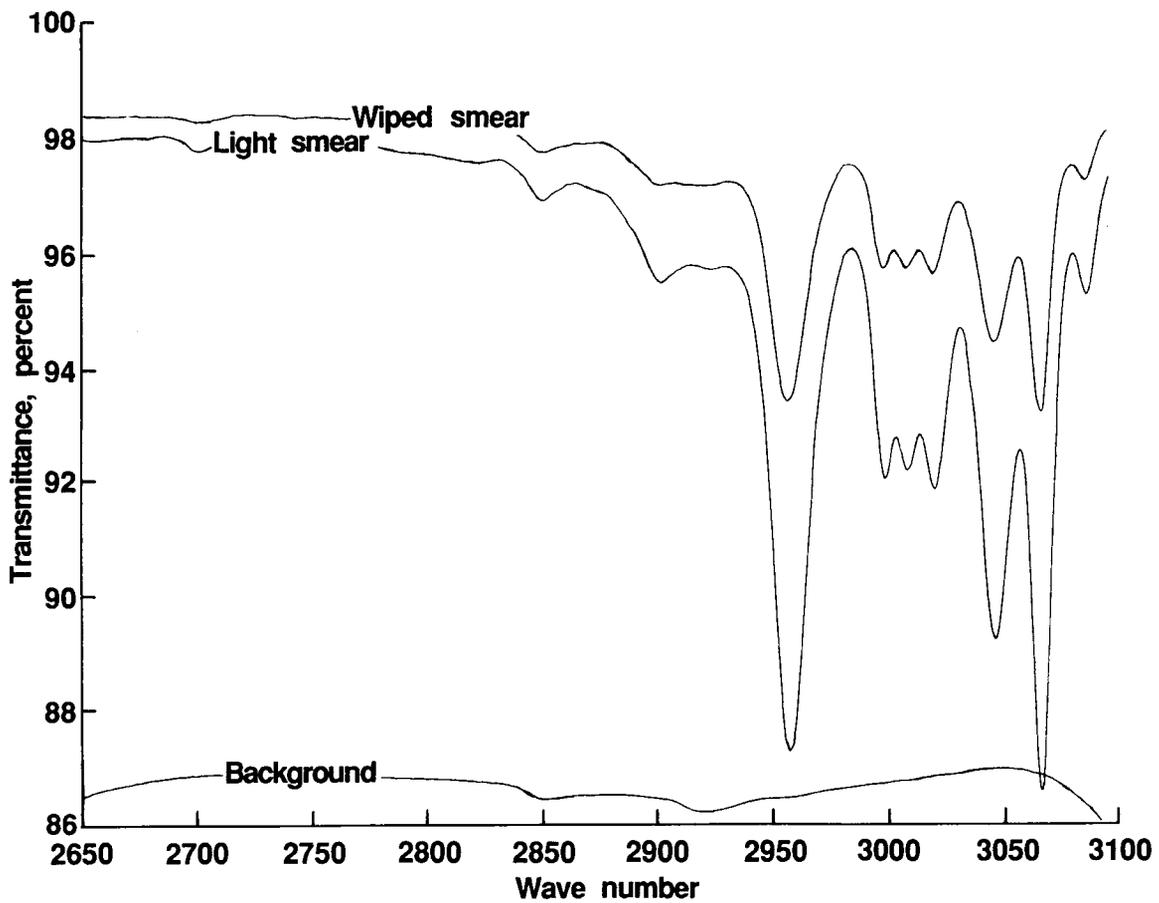


Figure 10. Spectra of Dow Corning 704 diffusion pump oil.

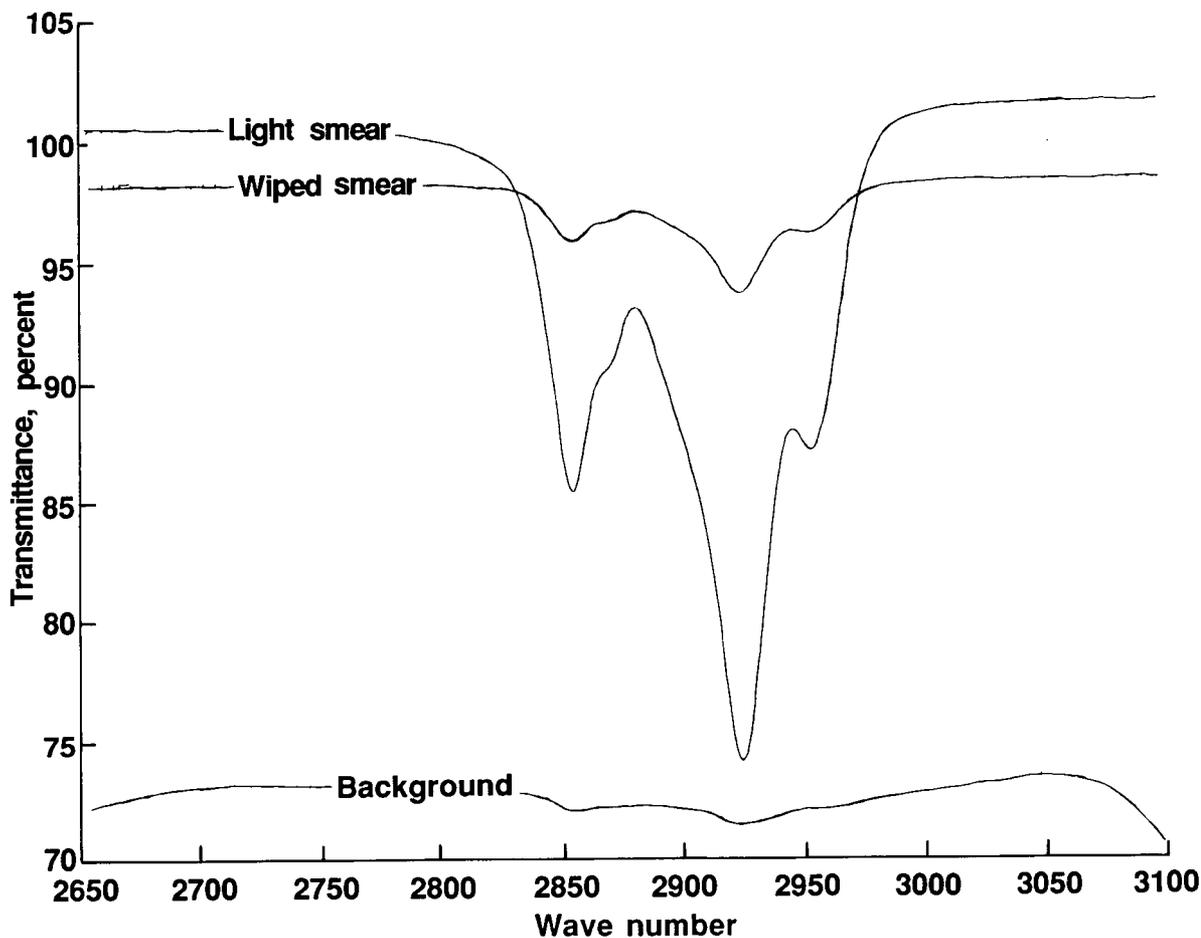


Figure 11. Spectra of Duo-Seal vacuum pump oil.

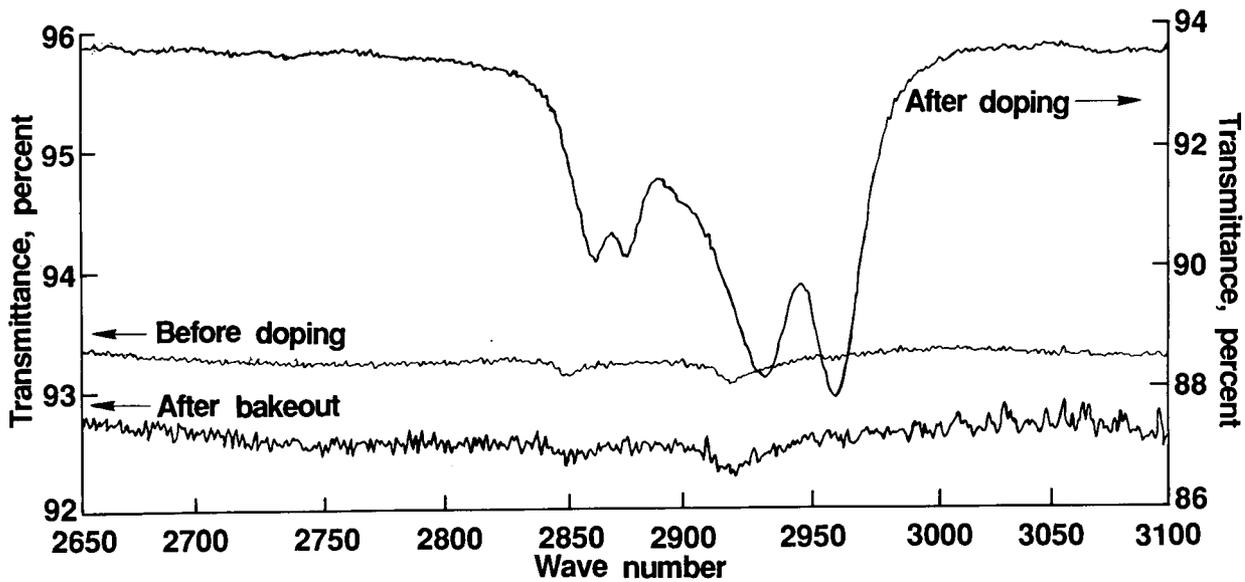


Figure 12. Diocetyl phthalate (DOP) spectra. The after-doping line corresponds to the right ordinate.

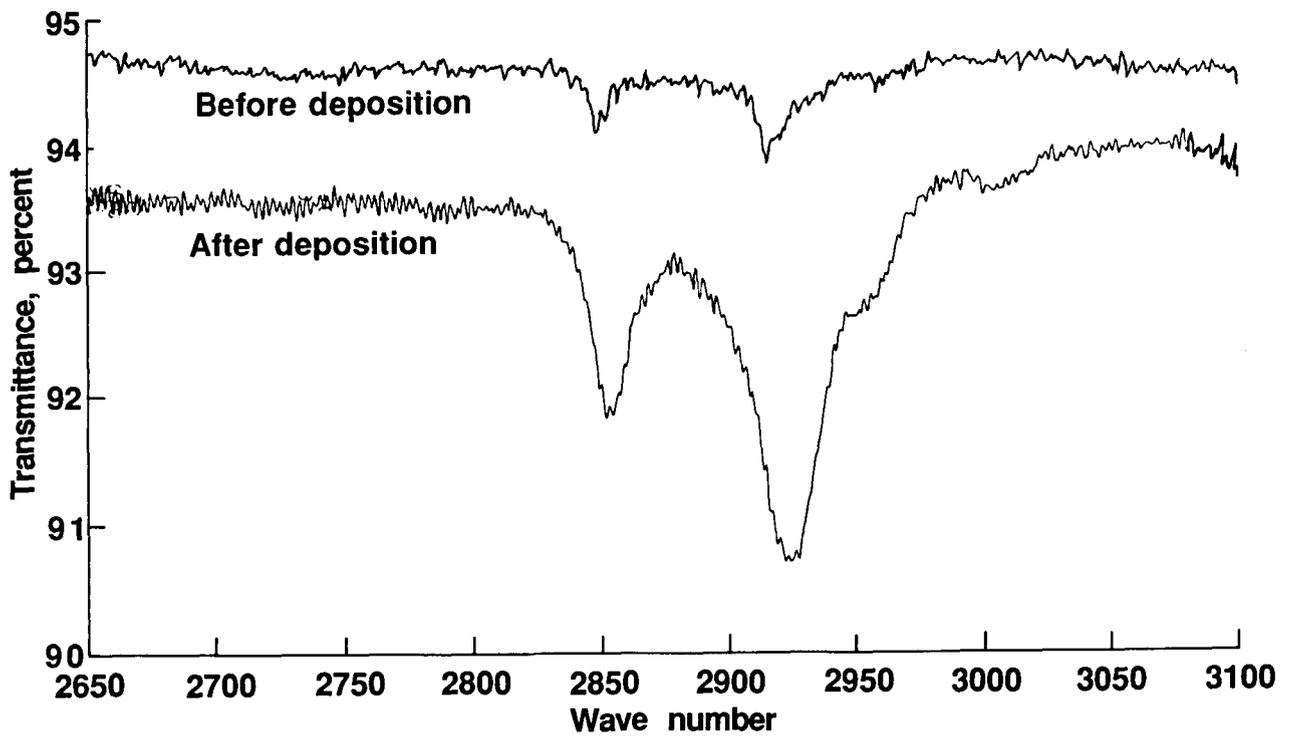


Figure 13. Spectra of 336-Å Solithane 3.

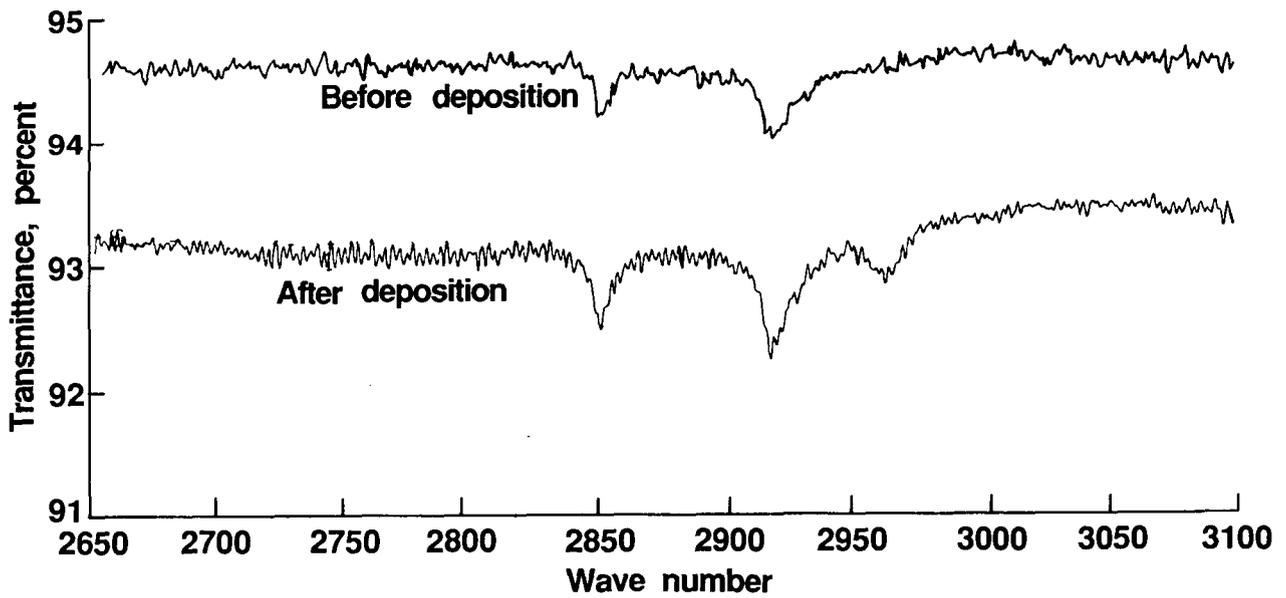


Figure 14. Spectra of 109-Å RTV 566.



Report Documentation Page

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16. Abstract The Halogen Occultation Experiment (HALOE) spacecraft instrument has over 100 optical elements and a passive thermal control system with low-absorptivity radiators. Molecular contamination of the optical surfaces could affect measurement sensitivity and radiator performance. An optical witness-plate program has been implemented to monitor the buildup of molecular contamination in the clean room during the assembly and testing of the HALOE instrument. "Travel" plates to monitor molecular contamination when the instrument is not in the clean room are also measured. The measurement technique is spectroscopy with high resolution in transmission in the 3- $\mu$ m spectral region using a Fourier transform spectrometer. Witness specimens of infrared transmitting material of low index of refraction are used for contaminant monitoring and for spectral signature analyses. Spectral signatures of possible molecular contamination are presented. No contamination of condensable volatile material (CVM) of HALOE optical witness specimens has yet been found.					
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