



Spacecraft Test Chamber Contamination Study in the AEDC Mark I Facility

J. G. Pipes, D. F. Frazine,
R. P. Young, ARO, Inc., and E. N. Borson, et al., The Aerospace Corporation

January 1980

Final Report for Period April 6, 1977 — July 24, 1977

Approved for public release; distribution unlimited.

**ARNOLD ENGINEERING DEVELOPMENT CENTER
ARNOLD AIR FORCE STATION, TENNESSEE
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE**

NOTICES

When U. S. Government drawings, specifications, or other data are used for any purpose other than a definitely related Government procurement operation, the Government thereby incurs no responsibility nor any obligation whatsoever, and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise, or in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

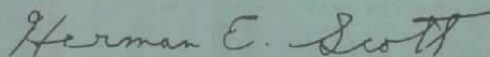
Qualified users may obtain copies of this report from the Defense Technical Information Center.

References to named commercial products in this report are not to be considered in any sense as an indorsement of the product by the United States Air Force or the Government.

This report has been reviewed by the Office of Public Affairs (PA) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

APPROVAL STATEMENT

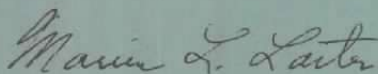
This report has been reviewed and approved.



HERMAN E. SCOTT
Project Manager
Directorate of Technology

Approved for publication:

FOR THE COMMANDER



MARION L. LASTER
Director of Technology
Deputy for Operations

UNCLASSIFIED

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1 REPORT NUMBER AEDC-TR-79-41	2 GOVT ACCESSION NO.	3 RECIPIENT'S CATALOG NUMBER
4 TITLE (and Subtitle) SPACECRAFT TEST CHAMBER CONTAMINATION STUDY IN THE AEDC MARK I FACILITY		5 TYPE OF REPORT & PERIOD COVERED Final Report - April 1977 - July 1977
7 AUTHOR(s) J. G. Pipes, D. F. Frazine, R. P. Young, ARO, Inc., a Sverdrup Corporation Company, and E. N. Borson and L. H. Rachal, The Aerospace Corporation		6 PERFORMING ORG REPORT NUMBER
9 PERFORMING ORGANIZATION NAME AND ADDRESS Arnold Engineering Development Center/DOT Air Force Systems Command Arnold Air Force Station, Tennessee 37389		8 CONTRACT OR GRANT NUMBER(s)
11 CONTROLLING OFFICE NAME AND ADDRESS Arnold Engineering Development Center/DOS Air Force Systems Command Arnold Air Force Station, Tennessee 37389		10 PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS Program Element 65807F
14 MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12 REPORT DATE January 1980
		13 NUMBER OF PAGES 23
		15 SECURITY CLASS. (of this report) UNCLASSIFIED
		15a DECLASSIFICATION/DOWNGRADING SCHEDULE N/A
16 DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		
17 DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18 SUPPLEMENTARY NOTES Available in Defense Technical Information Center (DTIC)		
19 KEY WORDS (Continue on reverse side if necessary and identify by block number) vacuum chamber quartz spectra contamination counting density cryogenics particles level internal reflection detectors residues spectroscopy reflectance drift		
20 ABSTRACT (Continue on reverse side if necessary and identify by block number) Recent results of a contamination study in the Arnold Engineering Development Center Mark I space simulation chamber are presented. The measurements were associated with the Global Positioning System satellite test conducted in the Mark I facility between April 6, 1977, and July 24, 1977. Multiple internal reflection spectroscopy (MIRS) internal reflection elements (IRE's), thermal control surface witness plates, and quartz crystal		

UNCLASSIFIED

UNCLASSIFIED

20. ABSTRACT (Continued)

microbalances (QCM's) were used to measure contaminants condensible during an extended chamber evacuation. An automatic particle counter was used to measure airborne particle density when the chamber was at atmospheric pressure. The nonvolatile residue wipe technique was employed to determine the general cleanliness of the chamber hardware. Temperature of the IRE's during chamber operation was between 150 and 200 K. One QCM was temperature controlled to a temperature of 238 K; the other QCM operated at 135 K. After chamber repressurization, the IRE's were scanned in the 2- to 15- μm wavelength region. Resulting spectra indicated a coating of diffusion pump fluid (DC704) at a concentration level just above the limit of detectability. The cold QCM recorded deposition during chamber evacuation. This deposit sublimed during chamber warmup at a temperature of 170 K, the sublimation temperature of water. Since neither QCM indicated a mass addition after chamber repressurization, minimum detectable mass of the QCM $1.7 \times 10^{-8} \text{ g/cm}^2$ was used as an upper limit for the mass of silicone fluid collected on the IRE's. This upper limit corresponds to two molecular layers of fluid for a 29-day evacuation period. The thermal control surface samples showed no change in IR spectral reflectance during the same 29-day chamber evacuation. Particle counts were taken during periods when the Mark I chamber was at atmospheric pressure. Particle densities measured for particles larger than $0.5 \mu\text{m}$ were nominally 6,000 particles/ft³. Particle densities for particles larger than $5.0 \mu\text{m}$ were nominally 15 particles/ft³. The airborne particle contamination in the Mark I chamber (at atmospheric pressure) was therefore below that of a class 10,000 clean room, as specified by Federal Standard 209.

PREFACE

The work reported herein was conducted by the Arnold Engineering Development Center (AEDC), Air Force Systems Command (AFSC). The results were obtained by ARO, Inc., AEDC Division (a Sverdrup Corporation Company), operating contractor for the AEDC, AFSC, Arnold Air Force Station, Tennessee. The work was done under ARO Project Nos. V32P-P1A and V32S-R1A. Mr. E. N. Borson and L. H. Rachal of The Aerospace Corporation were also authors of this report. Dr. H. E. Scott was the Air Force project manager. The manuscript was submitted for publication on April 30, 1979.

CONTENTS

	<u>Page</u>
1.0 INTRODUCTION	5
2.0 PARTICULATE MEASUREMENTS	
2.1 Instrument Description	7
2.2 Typical Data and Results	8
3.0 NVR (Nonvolatile Residue) MEASUREMENTS	
3.1 Solvents and Procedures	9
3.2 NVR Results	10
4.0 MIRS MEASUREMENTS	
4.1 IRE Description and IR Spectrum	10
4.2 Typical Results	12
4.3 Quantitative Assessment	16
5.0 WITNESS PLATES	
5.1 Description	16
5.2 Results	17
6.0 QCM MEASUREMENTS	
6.1 Instrument Description	18
6.2 Typical Data and Results	18
7.0 SUMMARY	22
REFERENCES	22

ILLUSTRATIONS

Figure

1. Installation in Mark I Chamber	6
2. Block Diagram of Particle Counter	8
3. Particle Size Measurement During a Work Period of Moderate Activity	9
4. Photograph of IRE and Temperature-Controlled QCM	12
5. IRE Spectrum — Post Spin-Drift (IR-8)	13
6. IRE Spectrum — Post Spin-Drift (IR-2)	13
7. IRE Spectrum — Post On-Orbit No. 1 (AEDC-1)	14
8. IRE Spectrum — Post On-Orbit No. 1 (IR-8)	14
9. IRE Spectrum — Prior to On-Orbit No. 3 (IR-8) after Cleaning	15
10. IRE Spectrum — Post On-Orbit No. 3 (IR-10)	15
11. Reference Spectrum — DC-704 Fluid from Al-Mylar® Insulation	15

12. Infrared Reflectance of Al-Kapton® Witness Plate 17

13. Infrared Reflectance of Au-FEP Teflon® Witness Plate 18

14. QCM Measurements (On-Orbit No. 1) 19

15. QCM Measurements (On-Orbit No. 2) 20

16. QCM Measurements (On-Orbit No. 3) 21

TABLE

1. IRE's on GPS Test 11

NOMENCLATURE 23

1.0 INTRODUCTION

The Mark I space simulation chamber (Fig. 1), located at the Arnold Engineering Development Center, Arnold Air Force Station, Tennessee, is one of the largest facilities of its kind in the world. It measures 12.8 m (42 ft) in diameter by 24.99 m (82 ft) high. The chamber is constructed entirely of stainless steel and is equipped with a liquid-nitrogen-cooled liner over the floor, walls, and portions of the ceiling to simulate the thermal environment of outer space. Eleven 0.81-m (32-in.) oil diffusion pumps form the primary pumping system, and each pump has its own liquid-nitrogen baffle and angle valve. This unique facility was recently utilized for the performance of an extensive qualification test of the prototype satellite for the Global Positioning System (GPS), also referred to as the NAVSTAR system. That project is a joint effort under the Department of Defense. It is managed by SAMSO, and Rockwell International is the contractor for satellite construction. When fully operational in 1984 the GPS will provide instant position information accurate to within tens of feet and velocity vectors within tenths of a foot per second to airborne, ship, and ground systems throughout the world.

Contamination of the GPS satellite during the test was of utmost concern since one of the primary objectives of the test was the verification of both the thermal control system on the satellite and a thermal mathematical model derived by Rockwell International personnel. Should contamination adversely affect the α/ϵ (solar absorptance/total emissivity) of the materials, conclusions drawn from the thermal vacuum test would be ambiguous. An additional key motivation for making contamination measurements was the possibility of a follow-on test of a flight vehicle. Thus, a contamination measurement and control program was formulated specifically for the GPS test. Since the test was unique in duration (April 6 through July 24, 1977) and requirements, it presented an excellent opportunity to fully document the environmental conditions of the AEDC Mark I facility, a measurement not previously conducted with state-of-the-art instruments. The threat of contamination was encountered during two distinct test operations, (a) during installation and work periods between the space simulation tests and (b) during the actual space simulation tests. In case (a), the satellite is subjected to particulate fallout and personnel contamination; in case (b), it is subjected to vacuum pump fluid vapors and condensable vapors outgassed from umbilical wiring and general chamber hardware. To measure the degree of contamination originating from each of the possible sources, the following instruments were selected on the bases of sensitivity, applicability, and availability: a particle-sensing instrument, Coulter Model 550, to monitor particulate contamination levels below 10^5 per cubic foot of air; a nonvolatile residue (NVR) wipe test to measure surface cleanliness; a series of infrared multiple internal reflection spectroscopy (MIRS) internal reflection elements (IRE's) for qualitative assessment of condensibles; and a series of witness plates (wafers identical in

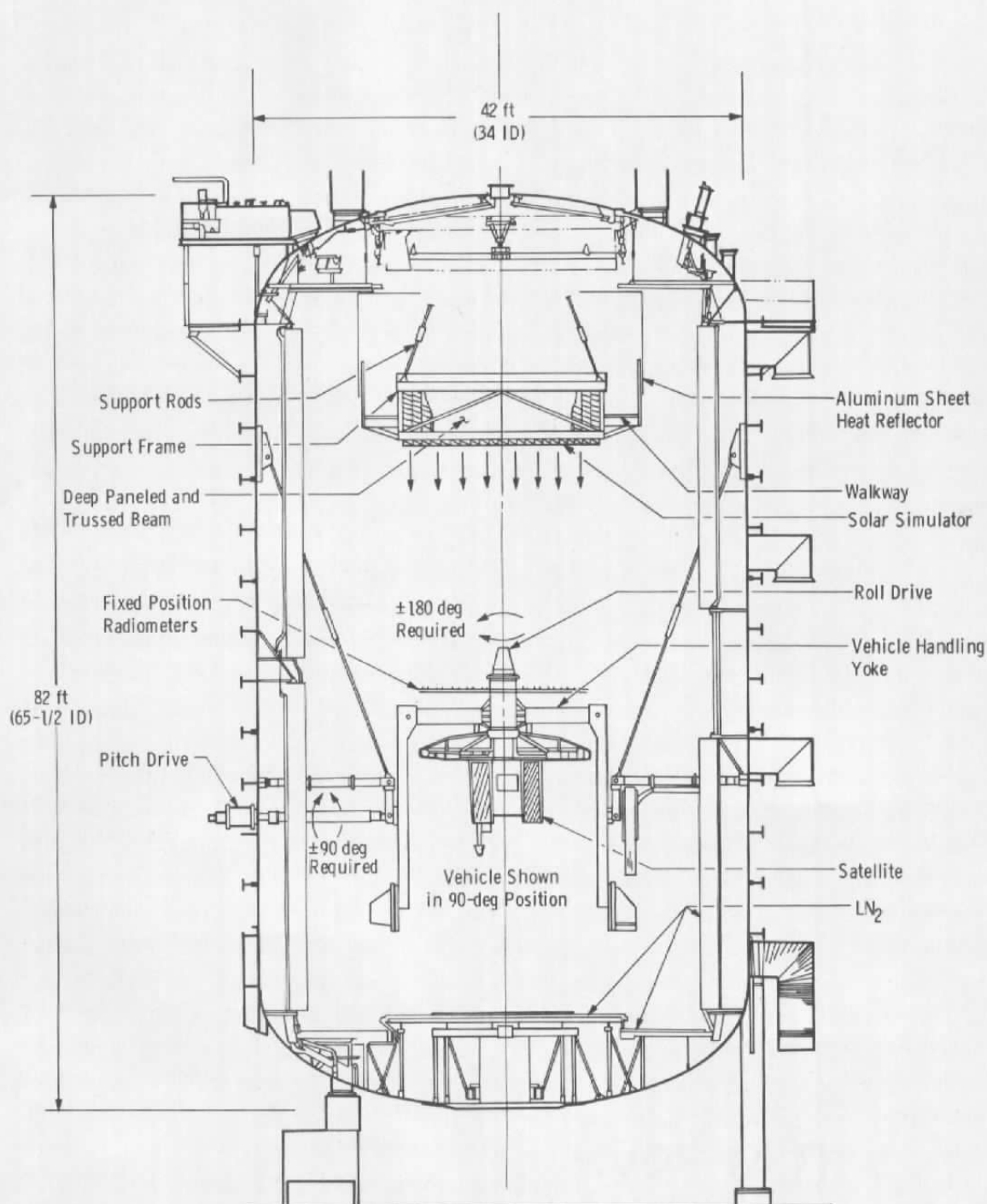


Figure 1. Installation in Mark I Chamber.

form to the satellite's outer skin for α/ϵ measurement of condensibles. Each of the instruments is individually described, and typical data and results are given.

2.0 PARTICULATE MEASUREMENTS

2.1 INSTRUMENT DESCRIPTION

The Mark I chamber is not equipped as a clean room. The GPS requirements of Class 340,000 are not stringent, however, and a series of particle count measurements was made near the beginning of test preparation to determine existing conditions. The series showed that the chamber nearly approximates the required Class 340,000 cleanliness level without special measures. Of particular importance is the fact that the chamber lid was off prior to the measurement. The addition of two dehumidifiers, provided by Rockwell, brought the chamber conditions within the required 340,000 class with 50-percent relative humidity for most of the ambient pressure periods.

To ensure that conditions were met around the vehicle at all times, a protective frame and surrounding cover were built for use when the vehicle was at the 0-deg position (antennas pointing up). The frame was attached to the vehicle lifting lug attachments. A cover of polyethylene embedded with nylon mesh was constructed to provide protection from any falling objects. The vehicle was thus contained in a protective enclosure inside the chamber.

Humidity and temperature control within the enclosure were maintained by a trailer-mounted 15-ton air-conditioning unit with attached filter furnished by Rockwell as part of the ground support equipment. This unit was located beneath the chamber; its output was ducted to the protective enclosure. A distribution manifold at the vehicle directed the required output over the batteries, thermal control louvers, and spin-drift drives. Because of the manifold, these units could be cooled with or without the protective enclosure.

Particle count measurements were monitored during ambient pressure operation using a Model 550 counter manufactured by Coulter Electronics, Inc. The manufacturer employs monosized particles in a range of 0.5 μm to 20.0 μm to calibrate the counter. An absolute calibration accuracy for the instrument is not known but is believed to be in the ± 20 -percent range. The counter employs the near-forward light-scattering technique and can detect particles greater than 0.5 μm in size. A simplified block diagram of the counter is given in Fig. 2. An air sample is collected through the positive displacement flow system; particles are detected and are consequently sized as the particle-laden air sample is passed between the light source and photoelectric detector. A resulting pulse, proportional to the particle size, is then amplified and sorted by size in an amplifier card, then subsequently fed to a logic card

for conversion to digital code. The digital code is displayed on the front panel; it is also accessible from a data card. During the installation of the GPS and between-test operations, the particle counter was located on the floor of the chamber since measurements for both the floor of the chamber and under the satellite tent showed no significant difference. As expected, the count level varied considerably depending upon the level of personnel activity in the chamber.

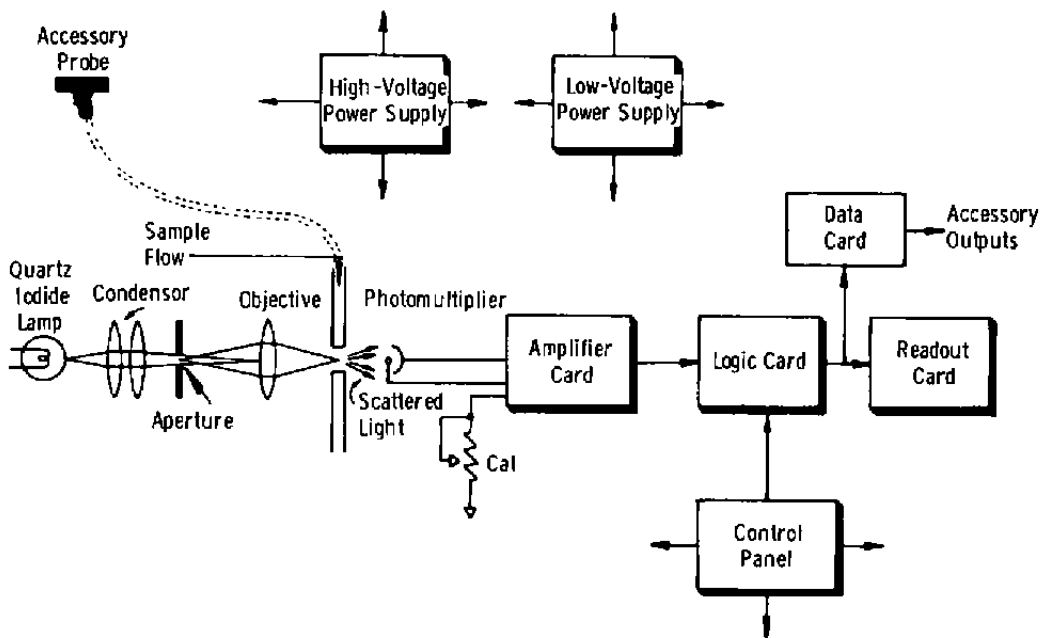


Figure 2. Block diagram of particle counter.

2.2 TYPICAL DATA AND RESULTS

Particle count data were manually recorded. (See Fig. 3 for a typical result during a work period of moderate activity.) As is made clear by Fig. 3, the density of particles greater in size than $0.5\text{ }\mu\text{m}$ was nominally $6,000/\text{ft}^3$ and $15/\text{ft}^3$ for sizes greater than $5\text{ }\mu\text{m}$. The Mark I chamber can therefore be classed as 10,000 for the GPS test. This adequately low count level has been attributed to the pretest cleaning of the chamber and test hardware, the clean room personnel procedures during installation and modification periods (all personnel wore caps, gloves, smocks, and shoe covers), and the operating procedures in general.

A correction of the particle count was made after the test. Originally, class 100,000 was reported, based on a Royce Model 245 counter which was later found to be severely out of calibration. (The Royce calibration was questioned when such a low particle count was indicated and was, furthermore, in disagreement with the Coulter counter measurement.)

Following calibration correction, the Royce measurement closely approximated that of the Coulter; thus reinforced by the Royce data, the Coulter data reported herein is assumed accurate.

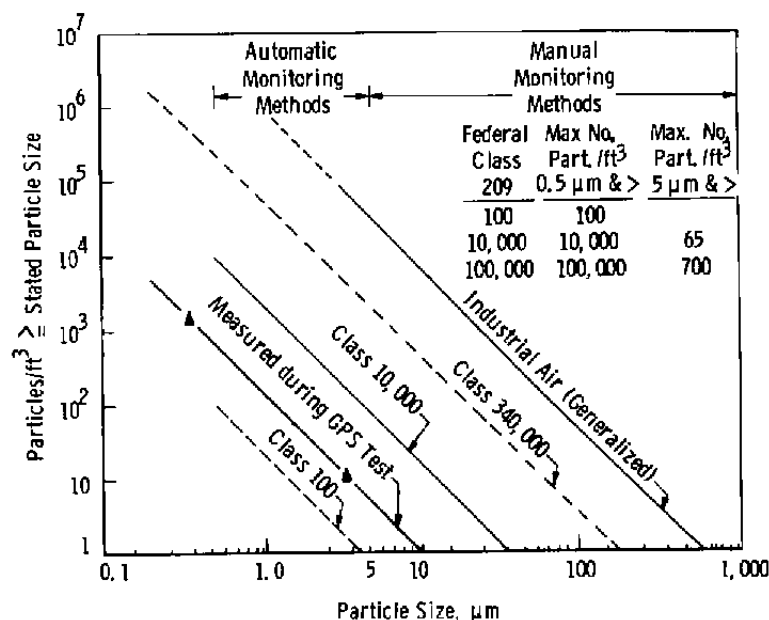


Figure 3. Particle size measurement during a work period of moderate activity.

3.0 NVR (NONVOLATILE RESIDUE) MEASUREMENTS

3.1 SOLVENTS AND PROCEDURES

Nonvolatile residue (NVR) was measured on both the Mark I test chamber and dummy space vehicle surfaces. NVR measurements were performed before and after chamber pumpdown tests with a simulated space vehicle. No additional NVR measurements were performed following the qualification test on the GPS space vehicle because the QCM's and IRE's indicated negligible NVR levels.

The NVR measurement procedure consists of wiping the surface with a cloth dampened with 1-1-1 trichloroethane mixed with ethanol. Soxhlet of the cloths is employed to remove soluble residues, and the solvents are triple distilled to reduce the NVR to approximately 1 ppm. A solvent mixture of 75-percent 1-1-1 trichloroethane and 25-percent ethanol by volume is azeotropic (has a single boiling point) and is, therefore, convenient for use in Soxhlet extraction.

A surface area of approximately 0.1 m^2 (1 ft^2) is wiped with the solvent-dampened cloth twice, each time with a fresh cloth. The cloths are retained in a clean, sealed container until removed for the solvent extraction process. For this test, the used cloths were returned to the Aerospace Corporation Ivan A. Getting Laboratories for solvent extraction and NVR determination.

The extraction process consists of removing the NVR from the cloth in an ultrasonic bath using the 75 percent/25 percent (1-1-1 trichloroethane/ethanol) solvent mixture. The solvent is then evaporated and the NVR weighed. Since trichloroethane/ethanol is the solvent, evaporation can be performed at room temperature, rather than at the elevated temperature used in the ASTM standard test methods.

The NVR is usually retained for further analysis, should such information be required. Infrared MIRS is frequently used because of the small quantities of NVR that usually are available.

3.2 NVR RESULTS

The NVR measurements performed during the Mark I chamber tests are as follows for a wipe taken on a cable tray: 33 mg/m^2 (3.1 mg/ft^2) before the warm pumpdown and 15 mg/m^2 (1.4 mg/ft^2) after cold pumpdown. The NVR levels were considered to be high, and additional cleaning of the Mark I chamber was performed prior to starting the qualification test. Infrared spectroscopy had shown the NVR to be primarily a lubricant used on the test fixture.

4.0 MIRS MEASUREMENTS

4.1 IRE DESCRIPTION AND IR SPECTRUM

Multiple internal reflection spectroscopy (MIRS) at infrared wavelengths is one of the more sensitive techniques used for qualitative identification of infrared-active compounds when very low level concentrations are available. The principles of MIRS [also commonly referred to as ATR (attenuated total reflection)], total internal reflection, (TIR), and frustrated total internal reflection (FTIR), as well as the instruments and applications of the technique, are given in Ref. 1. There is also an ANSI/ASTM Standard on the subject (Ref. 2). Basically, polychromatic radiation, in most cases infrared of 2- to $25\text{-}\mu\text{m}$ wavelengths, is directed into a trapezoidal-shaped crystal so that the angles of incidence at the points of internal reflection are greater than the critical angle. The radiation is totally reflected at the crystal-air interface, but the electric field does penetrate the rarer medium (air), a distance not

greater than a few wavelengths, and is damped exponentially. The waves that attempt to penetrate the rare medium are termed "evanescent" or "frustrated," the latter expressed in the concept "frustrated total internal reflection." If the air (a dielectric) is replaced by a conductor (for example, the condensation of a volatile contaminant onto the crystal), the frustrated waves will be attenuated for wavelengths at which the contaminant is active, permitting infrared absorption spectroscopy. This condition leads to attenuated total reflection (ATR), which is the technique employed for contamination detection and identification during the GPS Mark I space simulation test. One other optical measurement was conducted, an IR specular reflection measurement of thermal control witness plates, which is described following the MIRS discussion.

ATR employs a trapezoidal crystal so that multiple reflections occur, amplifying the deflection sensitivity in a manner similar to that of a multipass transmission cell. Many geometric styles of IRE crystals have been fabricated (Ref. 1); however, the trapezoidal shape is the most common commercially available configuration. For the GPS test, Wilks KRS-5 single-pass crystals (52.5 by 20 by 2 mm, with 45-deg face angles) mounted in Delrin® holders were employed. Three AEDC plates and four Aerospace Corporation plates were mounted at locations outlined in Table 1. The temperature history of each plate is also indicated in this table. One IRE and the QCM's are shown in Fig. 4. During the entire course of the test, all MIRS spectra were obtained using the Wilks Model 9 internal

<u>IRE</u>	<u>Location</u>	<u>Temperature</u>
IR-6	On GPS looking along -Y axis	Same as GPS
IR-8	On GPS looking along +Y axis	Same as GPS
IR-2	On radar tower looking down and out at cryopanel	150 to 200 K
IR-10	On radar tower looking up and out at GPS	150 to 200 K
AEDC #1	On pitch yoke looking at GPS	150 to 200 K
AEDC #2	On pitch yoke looking opposite direction of #1	150 to 200 K
AEDC #3	On pitch yoke looking at radar structure	150 to 200 K

Table 1. IRE's on GPS Test

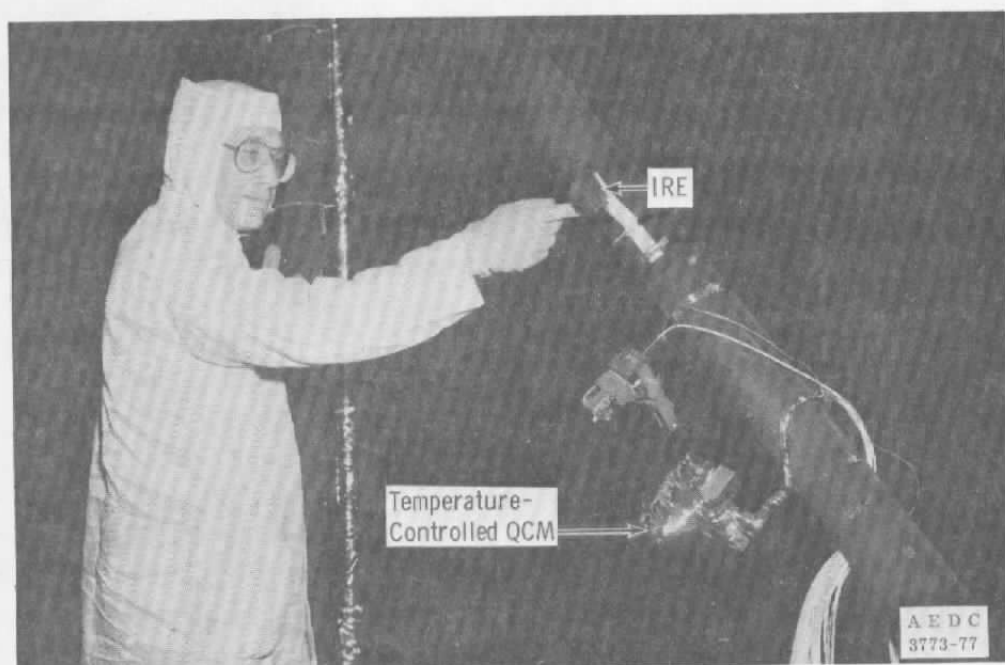


Figure 4. Photograph of IRE and temperature-controlled QCM.

reflection attachment and the Perkin-Elmer Model 21 double-beam spectrophotometer. Because the Model 21 has a sodium chloride prism, a nominal spectral resolution of 1 percent resulted for the slit program used for wavelengths between 2.0 and 15.0 μm .

All of the IRE's were scanned for a baseline or background spectrum before installation. In general, all of the plates showed a smooth transmission spectrum rising from 40 percent at 2.0 μm , to 90 percent near 10 μm , then dropping off to 70-percent transmission at 15 μm . The reference-beam attenuator on the Model 21 spectrophotometer was adjusted to give the 90-percent transmission at 10 μm ; therefore, the transmission values for all MIRS data presented are relative measurements.

4.2 TYPICAL RESULTS

The IRE's were used during each pumpdown-vent cycle with exceptions of the "warm bakeout" and "cold pumpdown" of the Mark I. Spectra were recorded after each venting of the Mark I; if contamination was indicated to a significant degree, the IRE's were cleaned and rescanned before the next pumpdown. After the GPS was installed, there were four pumpdowns and vents during the test sequence: (1) spin drift, (2) on-orbit No. 1, (3) on-orbit No. 2, and (4) on-orbit No. 3. The spin-drift test took five days, after which the IRE's

were scanned, the first scanning since being under vacuum. A number of weak features were observed in the spectra of all of the plates; these features were not recognized as significant until after the on-orbit No. 1 MIRS measurements. The spectral features for IR-6 and IR-8 were interpreted as baseline fluctuations; the bands of all other plates (e.g., IR-2) could not be explained at the time (see Figs. 5 and 6). No contaminant was detected on the IRE's, and they were reinstalled for the on-orbit No. 1 sequence. After the 15-day on-orbit No. 1, MIRS spectra were obtained, it became clear that the spectral features were real, and a search for a matching spectrum (from the data by F. C. Gross, NASA Goddard Space Flight Center, "Infrared Characterized Spacecraft Contaminants and Related Compounds") revealed that DC-704 was identifiable. The broad weak band in the MIRS spectra at $9.4\text{ }\mu\text{m}$ is, no doubt,

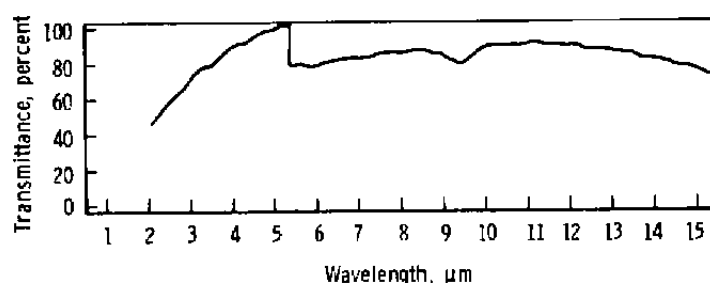


Figure 5. IRE spectrum — post spin-drift (IR-8).

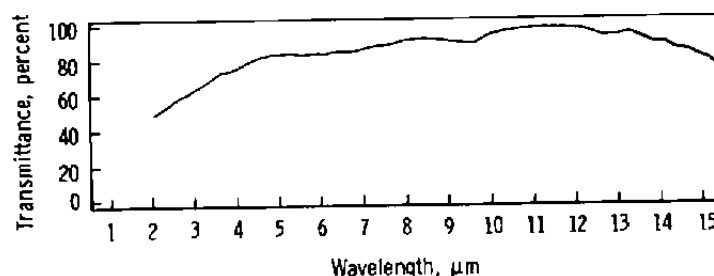


Figure 6. IRE spectrum — post spin-drift (IR-2).

the DC-704 reference spectrum strong band of the same measurement. The absorption bands at 3.5 , 12.5 , and 13.75 , and $14.4\text{ }\mu\text{m}$ were also just identifiable in the MIRS spectra (see Fig. 7). IR-6 and IR-8 located on the GPS showed a slightly different spectrum: The $9.45\text{-}\mu\text{m}$, triangularly shaped feature contrasted to the square $9.40\text{-}\mu\text{m}$ band shape in Fig. 7 (see Fig. 8). Although this $9.45\text{-}\mu\text{m}$ band could indicate another contaminant, the DC-704 fluid is the most probable candidate.

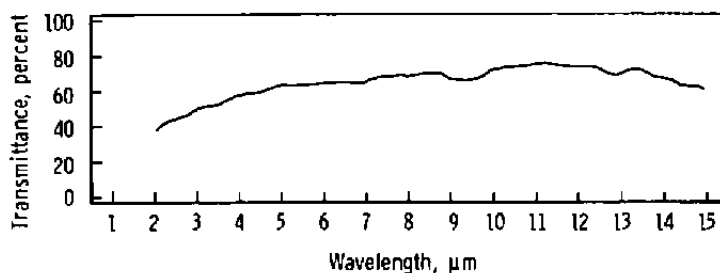


Figure 7. IRE spectrum — post on-orbit No. 1 (AEDC-1).

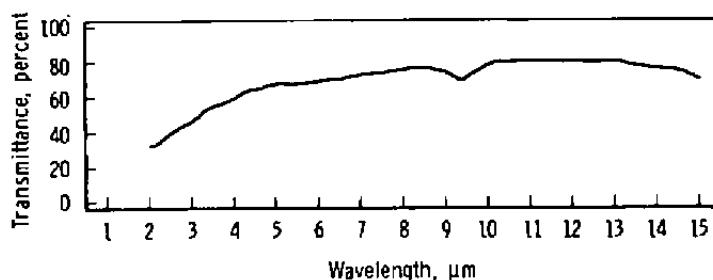


Figure 8. IRE spectrum — post on-orbit No. 1 (IR-8).

For the on-orbit No. 2 sequence the IRE's were not cleaned before installation so that a check for accumulation could be made. It was questionable whether the DC-704 was deposited during the pumpdown or vent sequence or whether it was a gradual accumulation during the entire test sequence. After the on-orbit No. 2 the MIRS spectra did not show a significant increase (doubling) in the DC-704 absorption bands; thus it was concluded that the DC-704 deposit is a gradual, continuous accumulation not primarily occurring during either the pumpdown or vent cycle. Also, during the on-orbit No. 2, IR-6 and IR-8 showed no increase in the 9.4- μm line strength, as determined by using neighboring transmission values as baselines.

Before on-orbit No. 3 the IRE's were cleaned with acetone, rinsed in ethanol, and scanned. The cleaning removed all evidence of DC-704, as is made clear by Fig. 9. On-orbit No. 3 lasted 30 days; again the IRE's showed evidence of DC-704 of the same concentration as the scan of on-orbits No. 1 and No. 2 (total of 25 days) (Fig. 10). The plates located on the GPS (i.e., IR-6 and IR-8) also showed a slight transmission loss near 9.4 μm , but this time the band was more strongly indicative of DC-704, (i.e., a square-shaped transmission loss between 9 and 10 μm).

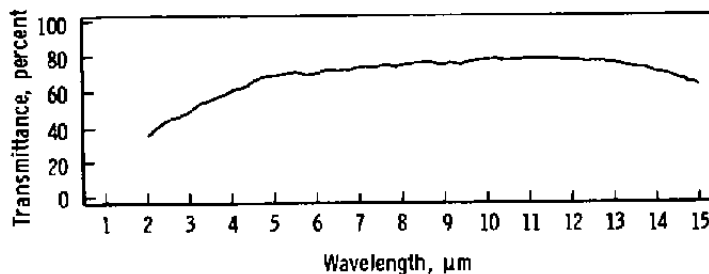


Figure 9. IRE spectrum — prior to on-orbit No. 3 (IR-8) after cleaning.

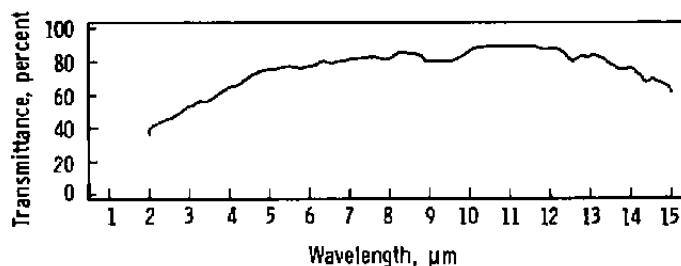


Figure 10. IRE spectrum — post on-orbit No. 3 (IR-10).

During a chamber inspection after the on-orbit No. 1 test, the aluminized Mylar[®] thermal control insulation behind the cryopanel manifolds manifested haze, especially on the area facing the diffusion pumps. A wipe sample was taken, the results of which are shown in Fig. 11. Apparently, the combination of the aluminized Mylar and the LN₂ wall separating the diffusion pumps from the test volume is an effective baffle. However, the LN₂-cooled chevron baffles immediately above the pumps are not sufficient by themselves.

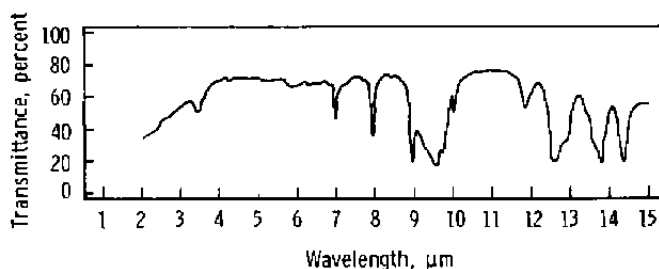


Figure 11. Reference spectrum — DC-704 fluid from Al-Mylar[®] insulation.

4.3 QUANTITATIVE ASSESSMENT

A quantitative assessment of DC-704 deposition required to produce the observed spectrum can be made using the quartz crystal microbalances (QCM's) results. The QCM's, temperature-controlled at 290 ± 10 K and one uncontrolled ($T = 116$ K after equilibration with the Mark I environment) showed that a deposition took place during each period under vacuum; however, the deposits sublimed immediately following the return to atmospheric pressure and temperature (see Section 6.2). Therefore, the deposit was probably water and definitely not DC-704 since DC-704 would not have sublimed. Thus, the minimum detectable mass deposition of the QCM may be used as an upper limit for DC-704 deposition, assuming that the DC-704 background was uniform throughout the chamber, exposing equally the IRE and QCM crystals. The QCM's have a sensitivity of 1.7×10^{-8} g/cm²-Hz. A change of 10 Hz is needed for positive deposition indication. It therefore follows, from the combination of these two, that there is an upper limit of 17×10^{-8} g/cm² of DC-704 contamination or, dividing by a density of 1.04 g/cm³, a thickness of 16×10^{-8} cm (16 Å). If it is assumed that the DC-704 was uniformly distributed across the plate, then the DC-704 layer on the IRE's could not have been more than two molecular layers in thickness, since DC-704 is a pentaphenyl-trimethyl-trisiloxane molecule, and the CH and SiO groups are approximately 3 Å in characteristic size. For the size of the IRE's employed, the number of multiple reflections is 25; thus, the loss of energy per reflection needed to yield a final loss of 5 percent is 0.25 percent. The thermal control engineer must determine whether such a small loss (0.0025) in reflectance at 9.4 μm (reflectance loss at other wavelengths is negligible) is detrimental to a thermal control surface. If it is not detrimental, then the GPS (or any similar hardware placed in the Mark I chamber) was completely free of contamination during the test.

5.0 WITNESS PLATES

5.1 DESCRIPTION

The witness plates were 1-in.-diam laminated wafers identical in form to the outer thermal control skin of the satellite. Two styles were employed: (1) aluminum with 0.001 in. of Kapton®, coated with aluminum, and (2) gold-coated Inconel® coated with FEP Teflon®. Seven witness plates were mounted on the satellite with double-stick tape at strategic locations. The α/ϵ (solar absorptance/total emissivity) ratio of the plate was

determined by measuring the reflectance at visible and near-IR wavelengths. Rockwell specifications of 0.002 for the Au-FEP Teflon plates and 0.004 for the Al-Kapton plates were set as acceptable upper limits for change in reflectance, for all wavelengths. This criterion is below the absolute accuracy limit on the Cary 90 and the Beckman DK-2A reflectometers employed to measure the reflectance. The reflectometers are more nearly 0.01 to 0.02 accurate in reflectance measurement capability. Nevertheless, the samples were scanned after the first test period.

5.2 RESULTS

The reflectance of all the witness plates was consistently within the measurement accuracy of the reflectometers. Thus, the reflectance had not changed more than one percent during a test period. Furthermore, following a review of the MIRS results, it was concluded that the reflectance measurements were unnecessary, since the IRE's, which are 25 times more sensitive than the reflectance measurements, showed a very small level of contamination. The witness plates were still included on each test phase but were later stored for future reference. Typical infrared reflectance scans of Al-Kapton and Au-Teflon witness samples are shown respectively, in Figs. 12 and 13.

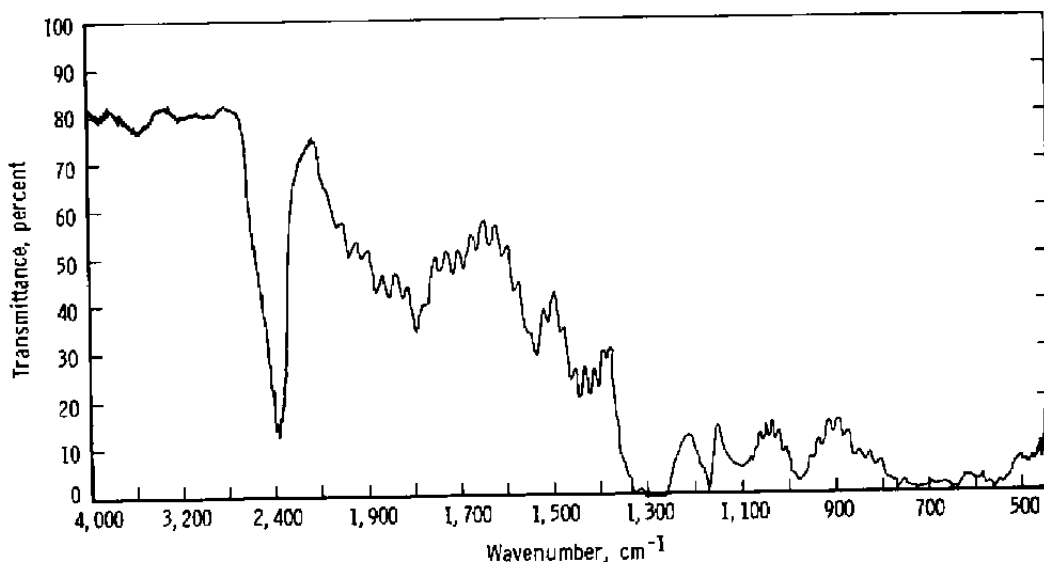


Figure 12. Infrared reflectance of Al-Kapton® witness plate.

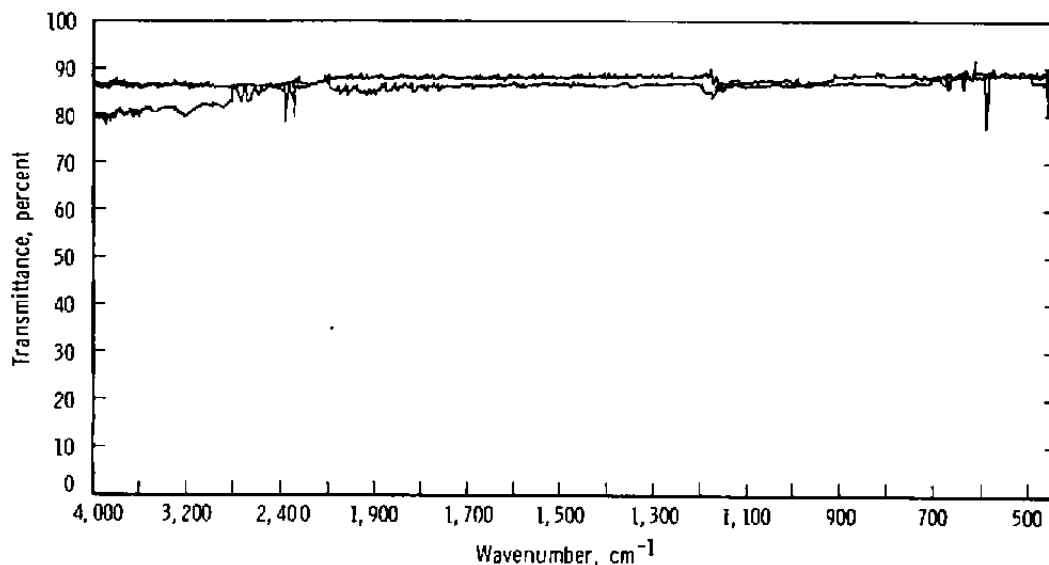


Figure 13. Infrared reflectance of Au-FEP Teflon® witness plate.

6.0 QCM MEASUREMENTS

6.1 INSTRUMENT DESCRIPTION

During the entire GPS test in the Mark I chamber, two quartz crystal microbalances viewed the central volume of the chamber containing the satellite. These Jet Propulsion Laboratory (JPL) QCM's incorporated a 5-MHz doublet crystal (measuring and reference oscillators on the same 3/4-by 1-1/2-in. crystal) with both crystals cut at 40°8' for cryogenic operation. Reference 3 gives a more complete description of the JPL-designed QCM's.

QCM 1 was strapped to the LN₂-cooled frame of the fixed-earth support tower and eventually reached a minimum temperature of 116 K; temperature variations were caused by changing solar simulator irradiance. QCM 2 was similarly mounted but was wrapped with aluminized Mylar and included a heater that maintained a crystal temperature of 284 K. (See Fig. 4 for mounting arrangement of QCM's.)

6.2 TYPICAL DATA AND RESULTS

QCM output signals were recorded continuously by frequency-to-voltage converters and a dual-pen strip chart; hourly readings from a frequency counter were noted on the chart. Frequencies taken from the chart and counter and crystal temperatures are plotted in Figs. 14 through 16. The actual frequency shown in the figures is a frequency signifying difference

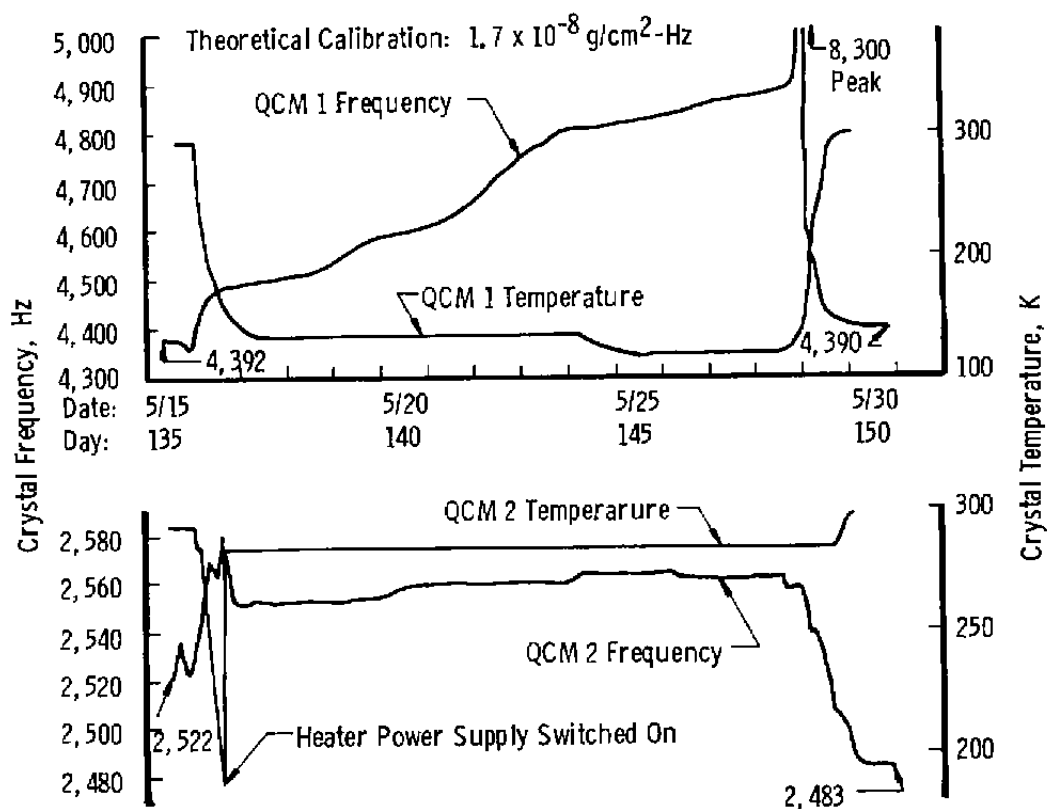


Figure 14. QCM measurements (on-orbit No. 1).

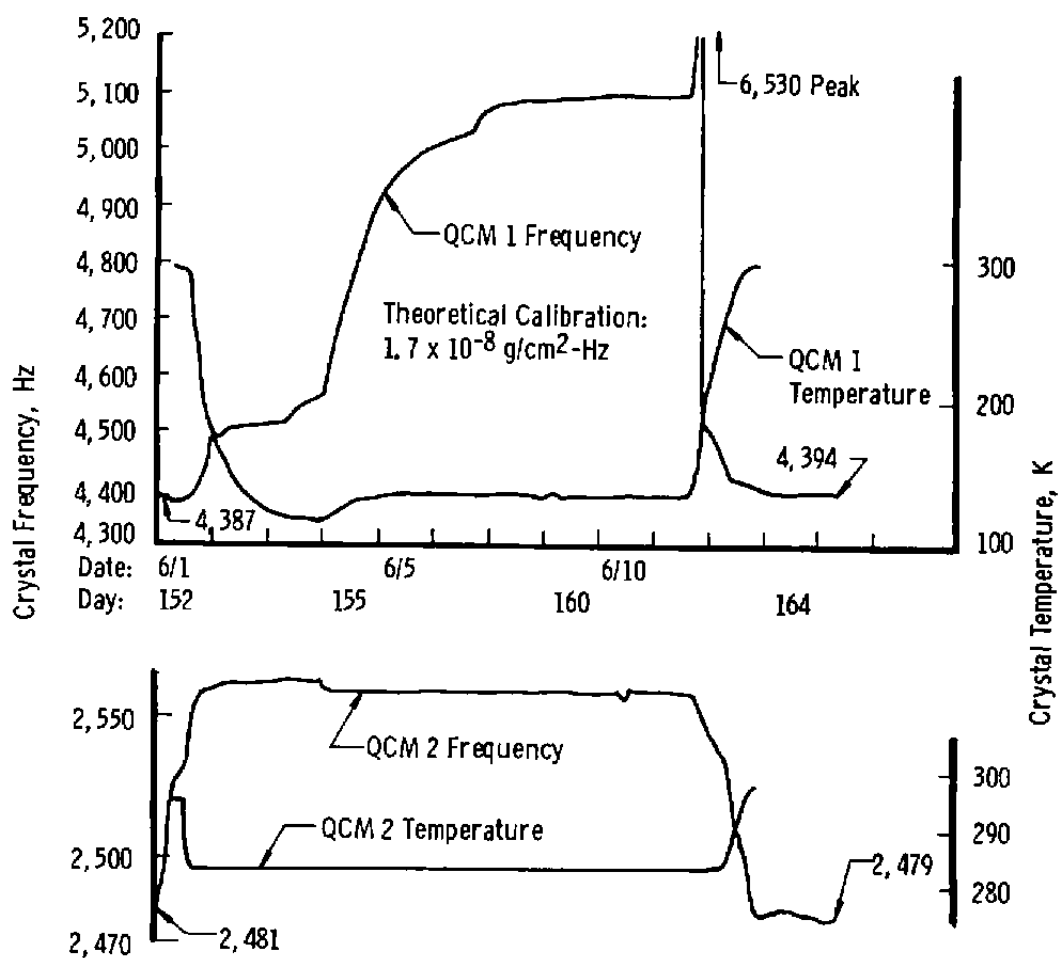


Figure 15. QCM measurements (on-orbit No. 2).

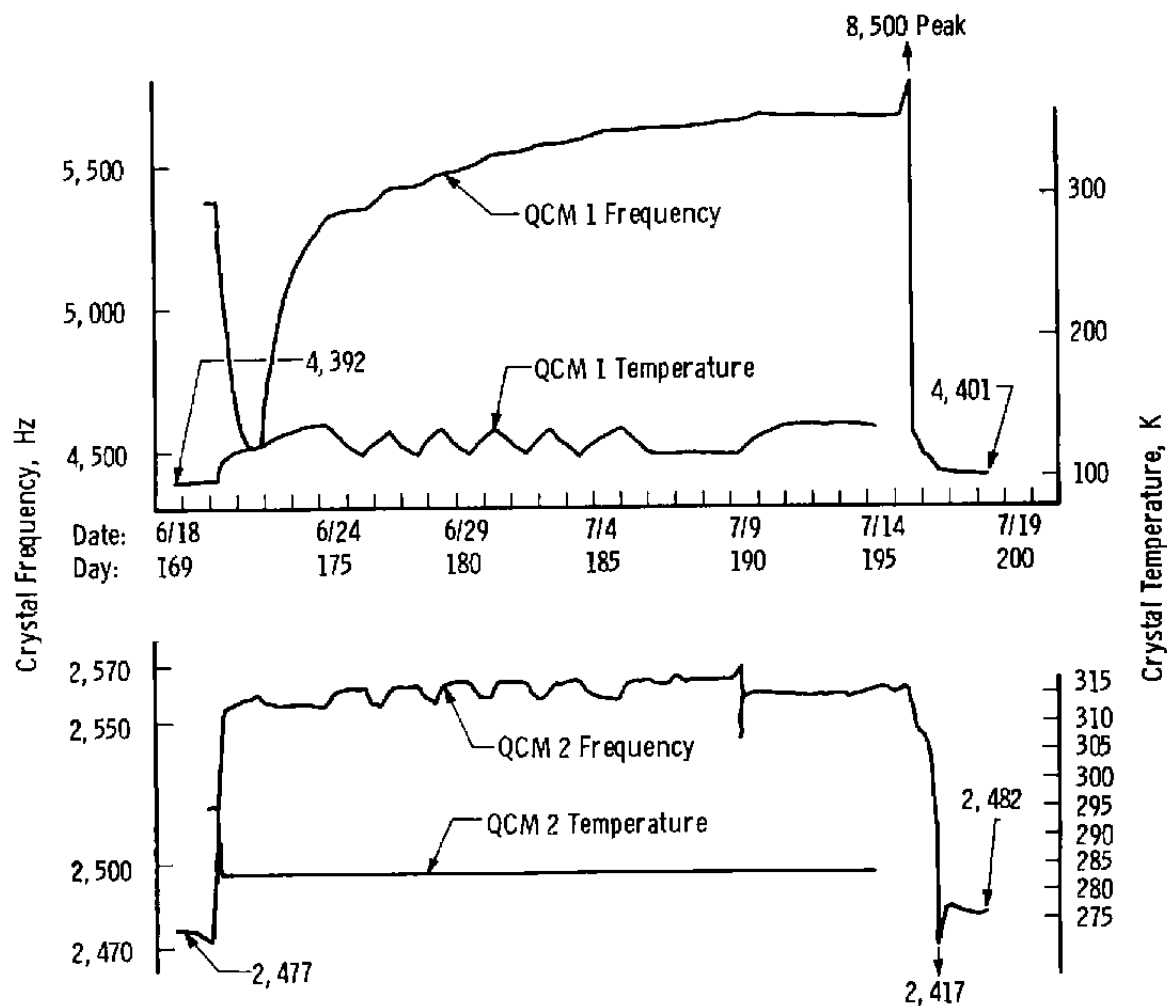


Figure 16. QCM measurements (on-orbit No. 3).

between the measuring and reference oscillators on each QCM. The temperature-controlled QCM 2 showed a small change in difference frequency of about 70 Hz (or a deposition of 1.2 μg) in contrast to the cooled QCM 1, which indicated a 4,000-Hz maximum frequency change (or a deposition of 70 μg). The maximum deposition occurred during warmup of the chamber; this deposition is caused by a release of water vapor from portions of the chamber that warm at a higher rate than the QCM. Given the facts that the final frequencies (after return to ambient conditions) were within 10 Hz of the original frequencies and that the QCM's indicated rapid loss of mass near 175 K (H_2O has a 10^{-5} torr vapor pressure at this temperature), it appears that the mass deposited on the QCM 1 was water vapor. Since the satellite itself was maintained at 300 K, it was not subjected to contamination by water vapor.

7.0 SUMMARY

Through the use of modern, sensitive contamination-measuring instruments, a complete assessment of contamination levels and types has been conducted for the AEDC Mark I space chamber. The general conclusion is that the Mark I facility has a very low background contamination level of diffusion pump fluid of, at most, two molecular layers over an extended evacuation period. Assessment of the effect of this level of contamination on the GPS test results is beyond the scope of this study. This contamination level is regarded as acceptable for most Mark I tests.

REFERENCES

1. Harrick, N. J. *Internal Reflection Spectroscopy*. John Wiley & Sons, Inc. New York, 1967.
2. ANSI/ASTM E 573-76, *Standard Recommended Practices for Internal Reflection Spectroscopy*, Annual Book of ASTM Standards, Part 42, American Society for Testing and Materials, Philadelphia, Penn., 1978, pp. 442-465.
3. Chirivella, J. E. "Hydrazine Engine Plume Contamination Mapping." AFRPL-TR-75-16 (AD-A018729), October 1975.

NOMENCLATURE

ATR	Attenuated total reflection
FTIR	Frustrated total internal reflection
GPS	Global Positioning System (also known as the NAVSTAR—Navigation System using Time and Ranging—System)
IRE	Internal reflection element
JPL	Jet Propulsion Laboratory
MIRS	Multiple internal reflection spectroscopy
NVR	Nonvolatile residue
QCM	Quartz crystal microbalance
TIR	Total internal reflection