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AN ETIOLOGICAL STUDY OF PHTHALATE SELF-CONTAMINATION OF SPACECRAFT AND CONTAMINATION FROM THEIR EARTHLY ENVIRONS

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16. Abstract

Phthalates are very objectionable contaminants in space experiments because of their strong absorption in the UV and IR regions of the spectrum. Thus, even minute amounts of these compounds migrating to optical equipment, in test or space-flight conditions, can seriously compromise the results of experiments by altering the sensitivity of the functional equipment.

Volatility data of the phthalates and other plasticizers are presented in addition to UV spectra of ultrathin films of these same types of compounds. Sources of plasticizer contamination are revealed with special recognition given to di-2-ethylhexyl phthalate (DEHP), the single most ubiquitous plasticizer in use.

A surprisingly large percentage of the outgassing condensates from vacuum testing of spacecraft contain DEHP as well as other plasticizers. IR and gas chromatography/mass spectroscopy methods were used to analyze samples from spacecraft. Methods for successful reduction of plasticizers and other contaminants are mentioned.

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INTRODUCTION

Organic contamination of spacecraft and associated experimental packages is a unique product of modern space technology. It occurs as a result of a complex interplay between onboard components and the environments encountered during construction, testing, launch, and mission operations. These environments include an unprecedented series of extremes including high vacuum, thermal cycling, vibration, and radiation exposures as well as the more conventional handling problems usually associated with clean-room activity. Experience has shown that the primary source of contamination of a spacecraft is internally generated: under the influence of the various phases of development and testing, material from within is distributed on and about other parts of the payload. Secondary sources of contamination include clean-room atmospheres, vacuum pump fluids, and handling materials. In fact, it is necessary to make periodic inspections of clean-room operations to reduce the incidence of spacecraft contamination from the clean-room itself. Contamination caused by backstreaming of diffusion and mechanical pump oils during vacuum testing has been found to be relatively negligible during routine procedures but may become significant during component or pumping failures. Also, multimillion dollar spacecraft have often been contamined by such mundane things as fingerprints, plasticizers from vinyl gloves, plastic tubing or protective covers, and residues from improper cleaning solvents.

The impact of contamination upon unmanned spacecraft missions is becoming more serious as the complexity of experiments increases and as the desired lifetime increases from a few months to several years. All optical experiments are subject to degradation by contamination; however, the vacuum UV experiments are the most sensitive because nearly all organics absorb in this spectral region. Degradation of startracker optics could jeopardize orientation and guidance systems; contamination of thermal-control exterior surfaces could alter absorptance/emittance properties and, thereby, create intolerable temperature changes in the spacecraft. Contamination of other optical experiments and particle detectors on board can result in false data acquisition or failure of that module.

PROPERTIES OF PHTHALATE CONTAMINATION

When phthalates are being discussed, the reference will often be to di-2-ethylhexyl phthalate (DEHP), more accurately referred to as *bis*-2-ethylhexyl phthalate.

Table 1 is a summary of data published by the U.S. Tariff Commission (Reference 1) that illustrate the size of the plasticizer business. It is the latest official published data on the subject that could be readily located. First, note that about 64 percent of the plasticizer market in 1969 was composed of phthalate esters; second, about 40 percent of these were DEHP. It is also interesting to note that although plasticizer production in general and phthalates within this classification are on the increase for the 3 years shown, among the major phthalates only DEHP shows a significant increase. On the other hand, the dollar value of plasticizers sold shows a slight decrease, which presumably stems from more economical production methods. It may be safely concluded that this same production trend has continued, a fact somewhat confirmed by analytical findings from hundreds of samples.

Vaporization is believed by the authors to be the principal means of plasticizer loss and transfer in vacuum testing of aerospace flight packages. Table 2, based on data by Russell (Reference 2), indicates the considerable volatility losses of some common plasticizers when processed in a standard milling test in conjunction with a 95-percent vinyl chloride and 5-percent vinyl acetate mixture at 443 K (170° C).

Table 1—U.S. production and value of plasticizers, 1967-1969.

Material	Production (10^6 kg (10^6 lb))			Sales Value (Million Dollars)		
	1967	1968	1969	1967	1968	1969
Total of all plasticizers	572 (1262)	604 (1331)	627 (1382)	261	280	265
Phthalic anhydride esters	356 (784)	381 (841)	401 (884)	125	133	127
Di-2-ethylhexyl phthalate	133 (293)	150 (330)	161 (355)	43	49	45
Diisodecyl phthalate	56 (123)	62 (137)	62 (137)	18	20	18
Diisooctyl phthalate	44 (98)	43 (94)	38 (83)	16	13	13

Table 2—Plasticizer loss in milling test at 443 K (170° C).

Plasticizer	Rate $\left(\frac{g/m^2}{min}\right)$	Loss per Hour (percent)
N-Octyl-*N*-decyl phthalate	0.5	1.4
Di-2-ethylhexyl phthalate	1.0	2.8
Tri-2-ethylhexyl phosphate	1.2	3.4
Di-*N*-hexyl phthalate	1.3	3.7
Di-2-ethylhexyl adipate	1.8	5.1

Table 3—Temperature/vapor pressure data for some common plasticizers.

Plasticizer	Temperature (K (°C))	
	6.7×10^{-6} N/m^2 (5×10^{-8} mm Hg)	6.7×10^{-4} N/m^2 (5×10^{-6} mm Hg)
Di-2-ethylhexyl sebacate	357 (84)	404 (131)
Di-*N*-octyl phthalate	355 (82)	405 (132)
N-Octyl-*N*-decyl phthalate	353 (80)	411 (138)
Tricresyl phosphate	351 (78)	409 (136)
Diisooctyl phthalate	345 (72)	394 (121)
Di-2-ethylhexyl phthalate	341 (68)	393 (120)
Di-2-ethylhexyl adipate	331 (58)	378 (105)
Tri-2-ethylhexyl phosphate	330 (57)	383 (110)
Di-*N*-hexyl phthalate	327 (54)	393 (120)

Although satellites do not ordinarily operate at such an elevated temperature in test or flight, they do experience as low as 10^{-5} N/m^2 (10^{-7} mm Hg) during test and 10^{-10} N/m^2 (10^{-12} mm Hg) or less for extended periods of up to years in the case of flight conditions at average temperatures of 298 to 313 K (25° to 40° C). The temperature/vapor pressure relationships of several common plasticizers as shown in Table 3 would be very significant if translated to test and, especially, flight conditions. We have detected most of these compounds or some closely related ones in flight preparedness vacuum testing of spacecraft.

Because we must accept the realities of outgassing, which at best has only been minimized through materials selection and proper cleaning procedures, it is very urgent to the success of the programs that we compensate for this phenomenon. To do this properly, it is necessary to understand the mechanisms of deposition and desorption.

An interesting study by Baurer and his associates (Reference 3) to determine the various factors that influence surface deposition of contaminants has been performed. A few of the most significant findings of this study are—

(1) Gaseous contaminant transport depends on gas/surface interaction potentials and hydrodynamic, electrostatic, and magnetic fields.

(2) Condensed phase materials are subject to hydrodynamic coupling, induced polarization, and electrostatic field effects.

(3) The nature of the surface is either conducting or dielectric with the dielectric preferentially attracting more of the contaminants.

In another significant and related experiment by Tominaga (Reference 4), a careful study was made of the desorption rate of contaminated surfaces, considering such materials as common esters, hydrocarbons, and silicones used as diffusion pump fluids. It is also noteworthy to relate that these same esters are also used as plasticizers and one is the omnipresent DEHP, sometimes referred to as octoil, as in this instance.

The major findings of this experiment were that—

Oil molecules impinging on a *clean* glass surface are assumed to be absorbed with a fairly large desorption energy such that their sojourn time is considerably longer. Such adsorbed oil molecules can scarcely be desorbed by reducing the vapor pressure over the surface and can only be desorbed when the temperature of the adsorbent is increased. However, further adsorption takes place with a desorption energy which is nearly equal to the latent heat of vaporization.

An illustration of the determination of this principle is shown by the two curves of Figure 1. In curve (a) the characteristics of an ultracleaned surface are demonstrated. Curve (b) is representative of a surface that has already been saturated with DEHP and depicts second and subsequent absorptions and desorptions from this surface.

The implications of these phenomena are quite significant in the theory and solution of our problems. More specifically, for instance, it is known from experimentation that all surfaces of a spacecraft do collect outgassing and other contaminants that are characteristic of the historical environment of the hardware involved. From the surface absorption-desorption studies, calculations can be made to determine the conditions necessary to remove the bulk of surface contamination except for a monolayer or so that will remain unless rather severe conditions are used for removal. In most cases total removal of the contaminant would not be necessary; in fact, many surfaces have been coated purposely for one reason or another. Working with surfaces containing this ultrathin layer of contaminant is standard and calculations usually include this parameter.

Previously it was mentioned that UV optical equipment is particularly vulnerable to contamination and degradation. Some very interesting experiments by Gillette and Kenyon (Reference 5) have been performed on LiF/Al-, MgF_2/Al-, and Pt-mirror surfaces to illustrate this effect. Proton irradiation was used to induce deposition of contaminant film on the optical equipment, which were more markedly affected in some cases in the vacuum UV range. This is particularly important because some satellite experiments are using these optics in this same spectral range.

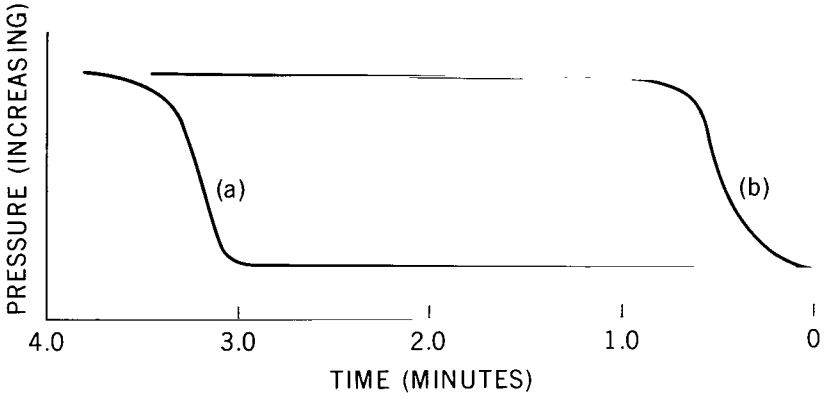

Figure 1—Flowrate of DEHP vapor at 75° C. (a) First flow of DEHP through ultraclean glass tube. (b) Second and subsequent flows through same tube after (a).

Note in Figures 2, 3, and 4 how the reflectances of MgF_2/Al-, LiF/Al-, and Pt-coated glass diminish when a 4- to 5-nm-thick oil film is deposited on them using proton-induced deposition (1×10^{16} protons/cm^2). The MgF_2 and LiF reflectances are affected in a wavelength-dependent manner whereas the Pt reflectance is almost uniformally affected throughout the measured spectral range. An interferometer was used to measure the film thickness of the deposited contaminant oil. Although no chemical analysis was made to determine the molecular species of the oil present, it may probably be safely assumed that the contaminant was a combination of pump oil and various chamber contaminants. It would not be unreasonable to assume that most thin oil films would react similarly in this

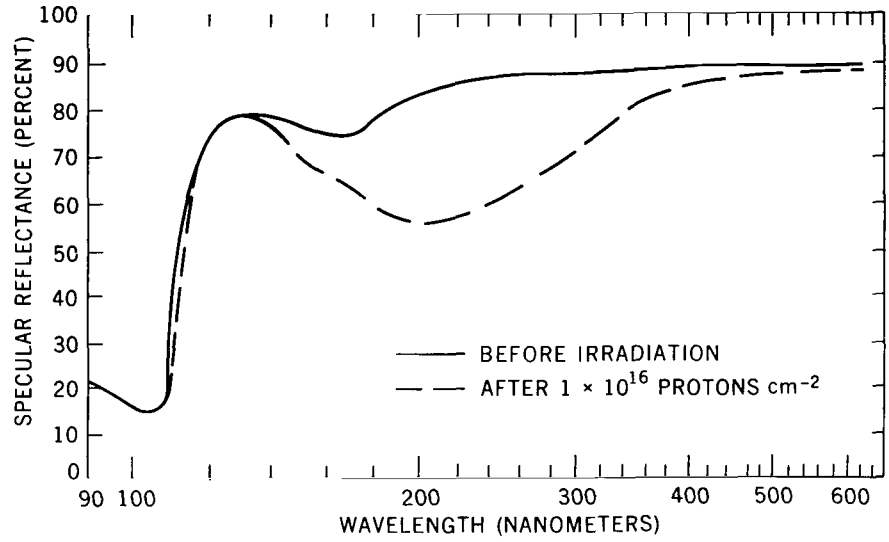

Figure 2—Reflectance change of a MgF_2/Al-coated mirror resulting from deposition of contaminant film during proton irradiation (MgF_2 thickness of 25 nm).

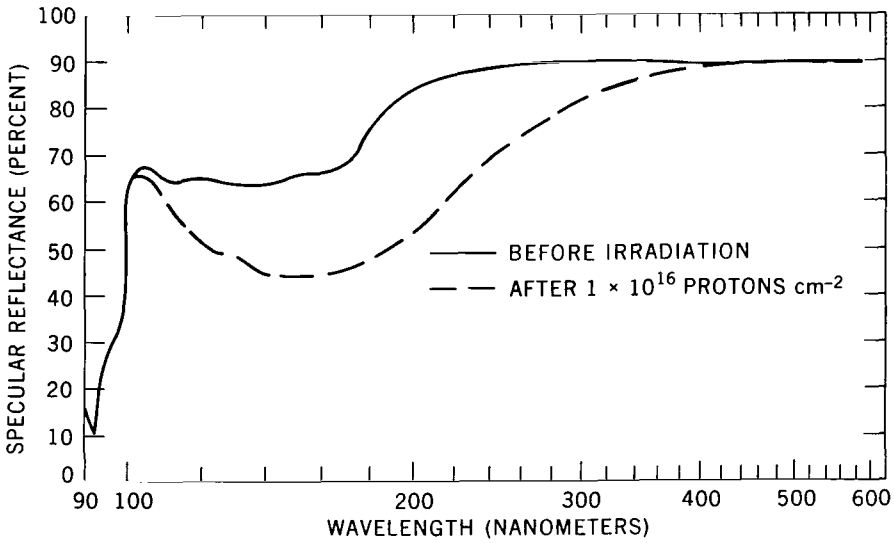

Figure 3—Reflectance changes of a LiF/Al-coated fused-silica mirror resulting from deposition of contaminant film during proton irradiation (LiF thickness of 14 nm).

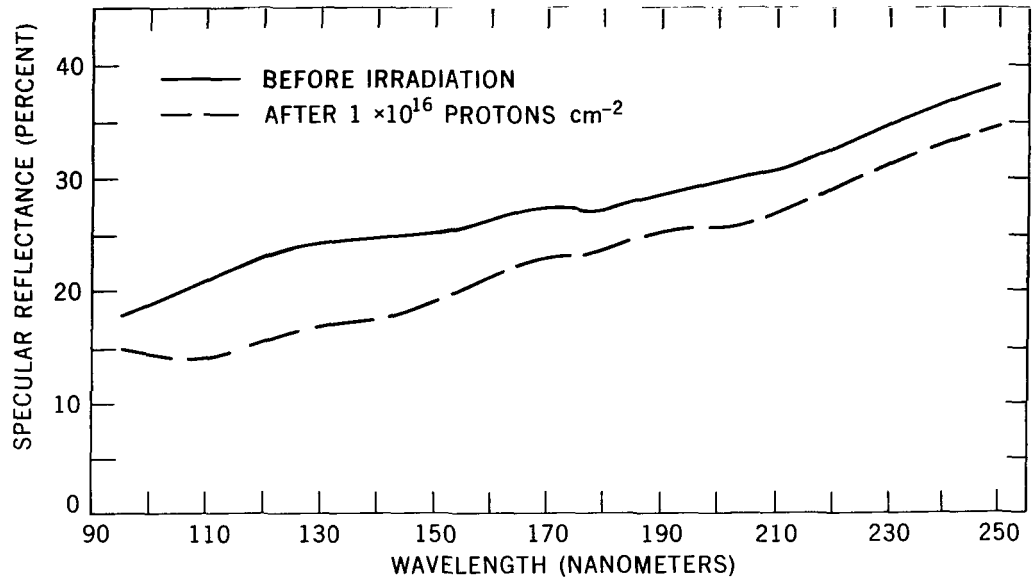

Figure 4—Reflectance changes of a Pt-coated glass mirror resulting from deposition of contaminant film during proton irradiation (Pt thickness of about 10 nm).

region of the spectrum. At GSFC it has been shown that similar polymerization of vacuum chamber contaminants also occurs on surfaces irradiated with either ultraviolet or high-energy electrons. Questions were raised as to how easily this radiation-induced oil film might be removed and whether the radiation had permanently changed the optical properties of the mirrors. As a result several cleaning methods, including Freon and collodion, CCl_4 soaking, and $CaCO_3$ abrasion, were attempted to see what form the oil had taken and how best to remove it. None of these methods was satisfactory, mainly because the oil had polymerized and probably crosslinked. Thus it had become impervious to the solvents and $CaCO_3$ was too harsh for the mirror coatings. Finally the mirrors were exposed to atomic oxygen for 5 min; the result was almost complete recovery to original condition. (See Figure 5.) This was a rather interesting discovery because it showed that the mirror surface itself was changed to a rather minor degree as a result of the proton irradiation and also that the contaminant could be easily removed without damaging the mirror surface. Incidentally, it was concluded that the slight final change experienced by the mirror was caused by a minute reduction in the LiF thickness. These experiments have taught us that a relatively good vacuum (10^{-4} to 10^{-5} N/m^2 (10^{-6} to 10^{-7} mm Hg)) should be obtained before irradiating a spacecraft or piece of hardware in test to prevent polymerization of contaminants on the surface.

EXPERIMENTAL METHODS

A variety of methods (Figure 6) is being used to determine the extent and nature of prelaunch spacecraft contamination. These include in situ residual gas analysis (RGA) and quartz crystal microbalance measurements during vacuum testing. After test completion, contamination areas are sampled, followed by IR analysis of the residues, UV vacuum monochromater measurements of test mirrors placed in strategic areas of the chamber, and collection of outgassing condensates on liquid-N_2-cooled

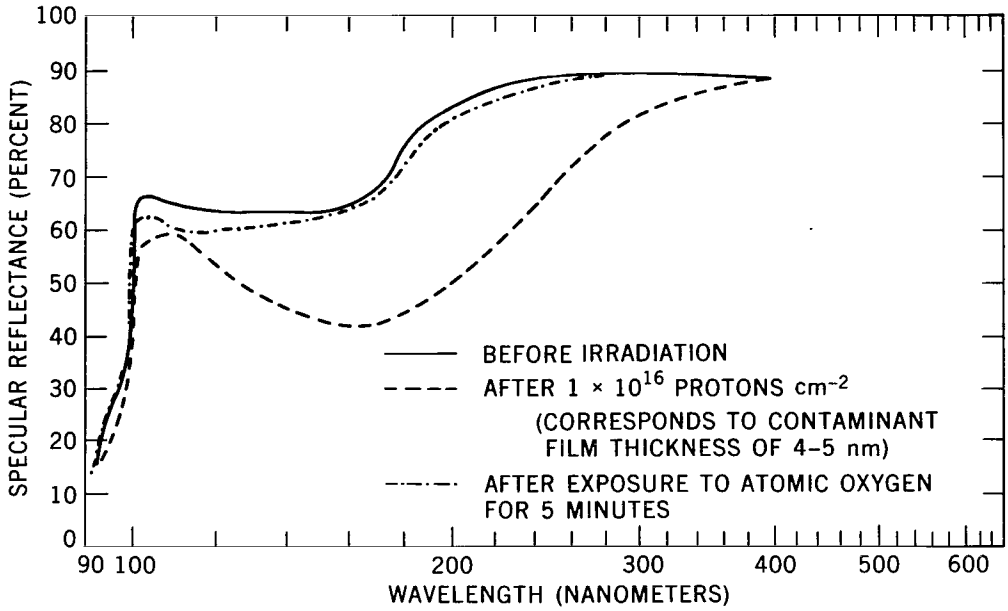

Figure 5—Reflectance recovery of a contaminated LiF/Al-coated fused-silica mirror exposed to atomic oxygen.

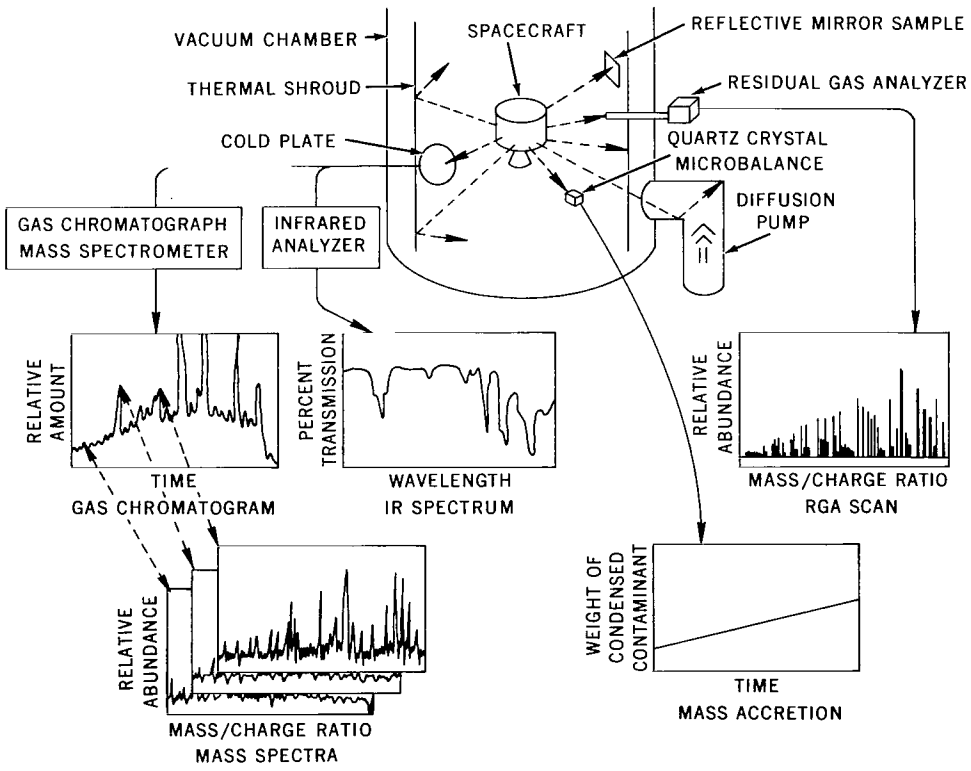

Figure 6—Relationship of measurement methods.

condenser plates that have been installed in the vacuum chambers. These condensates are then subjected to a chemical analysis, the extent of which is based on the particular situation. It is this latter procedure that has been proven most useful because the condensable outgassed materials that are potentially most harmful to the mission are collected in quantity. The analytical procedures necessary for identification include gas and liquid chromatography in addition to IR and mass spectroscopy, which are described in more detail later in this report.

The amount of material collected on a cold finger during thermal vacuum testing of a spacecraft may be as much as several grams and generally consists of three classes of compounds; namely, esters, silicones, and hydrocarbons, with occasional traces of fatty acids and other human outgassing products. Although separation of components reveals as many as 80 to 90 or perhaps even more distinct compounds in a sample, the most ubiquitous of the lot are the phthalate esters. Even after considerable attempts to eliminate this contamination from material lists, over a period of years, it invariably appears in the outgassing products of most spacecraft and many of the individual module tests. In fact, this investigation of sources of phthalates has revealed a veritable multitude of phthalate reservoirs in technology environments. Potential phthalate sources range from polyvinyl chloride tubing, wire insulation, and gloves to alkyd paints, pump oil, and certain rubber formulations. A rather subtle source of phthalate contamination is produced from high-efficiency particulate filters known as HEPA filters used in clean-room and similar-type air purification systems (see Figure 7) where the entire

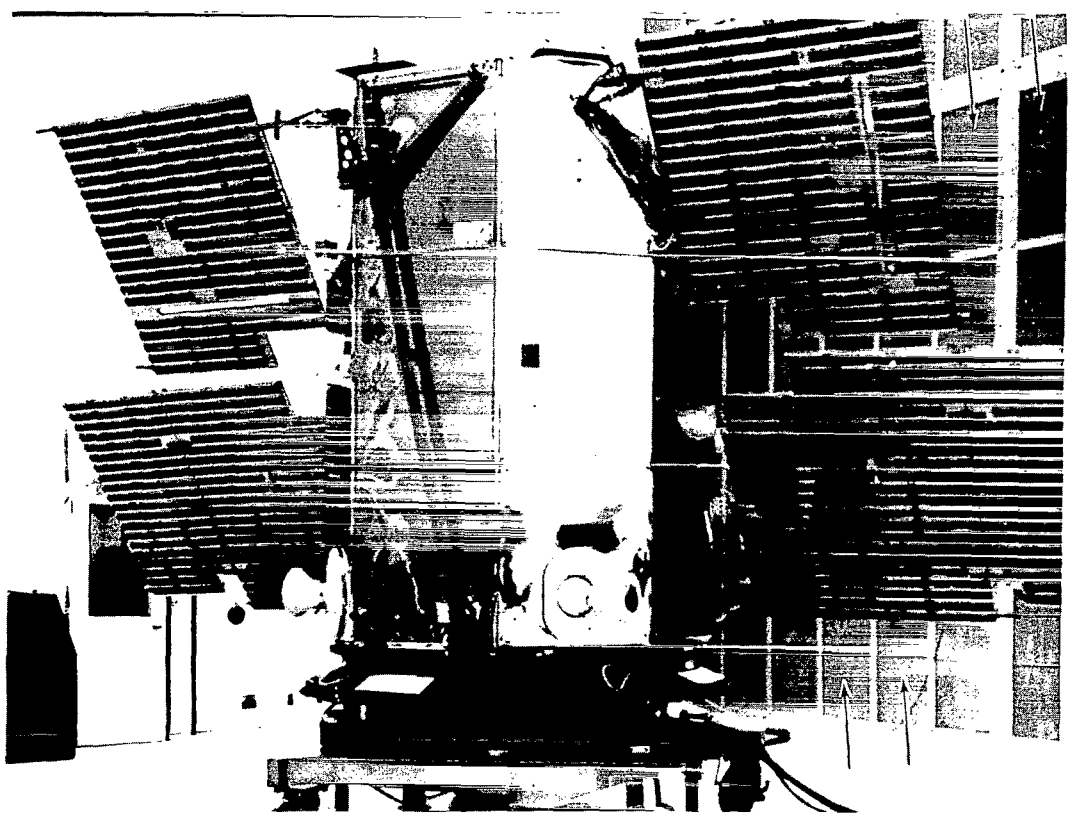

Figure 7—HEPA filters in clean-room walls.

clean-room wall is made up of these filters. The phthalate ester, usually DEHP, is sprayed as a 0.3-μm smoke on the back side of these filters to check their efficiency in removing this size particle. Even though the ester does not emerge immediately and, thus, the filter is considered satisfactory, there is good evidence to show it will diffuse through the filter over a period of time and is eventually carried into the clean room or wherever the air is being delivered to be subsequently deposited as a thin film on all exposed surfaces. As a result, it is always recommended that critical optics, detectors, and other sensitive equipment be exposed in the clean-room atmosphere for the minimum time to accomplish a specific task. Modes of distribution of the phthalates and other related plasticizers include creep along surfaces, solvent extraction and redistribution, contact transfer, and vaporization followed by condensation processes caused by significant vapor pressures of the different esters at characteristic test temperatures and reduced pressures.

ANALYSES OF OUTGASSING RESIDUES

Spacecraft 1, although built by NASA, carried 28 experiment modules assembled by various universities and research centers around the country. Some of these experiment modules were available for individual thermal-vacuum testing but most were not; and because of the diversity of the sources of the hardware, little control of material selection from an outgassing point of view was possible. Thus, it was not surprising that a copious amount of condensable material was collected on the liquid-N_2-cooled condenser plate during thermal-vacuum testing. In fact, this amount of material was so disconcerting to project managers and experimenters that five or six extensive thermal-vacuum soaks were performed to reduce the potential amount of outgassing material before launch. IR spectroscopy of each of these samples showed that although the amount of outgassed material was reduced, its composition remained quite constant, consisting mainly of esters, silicones, and high-molecular-weight hydrocarbons as shown in Figure 8. Our primary means of specific identification of compounds was

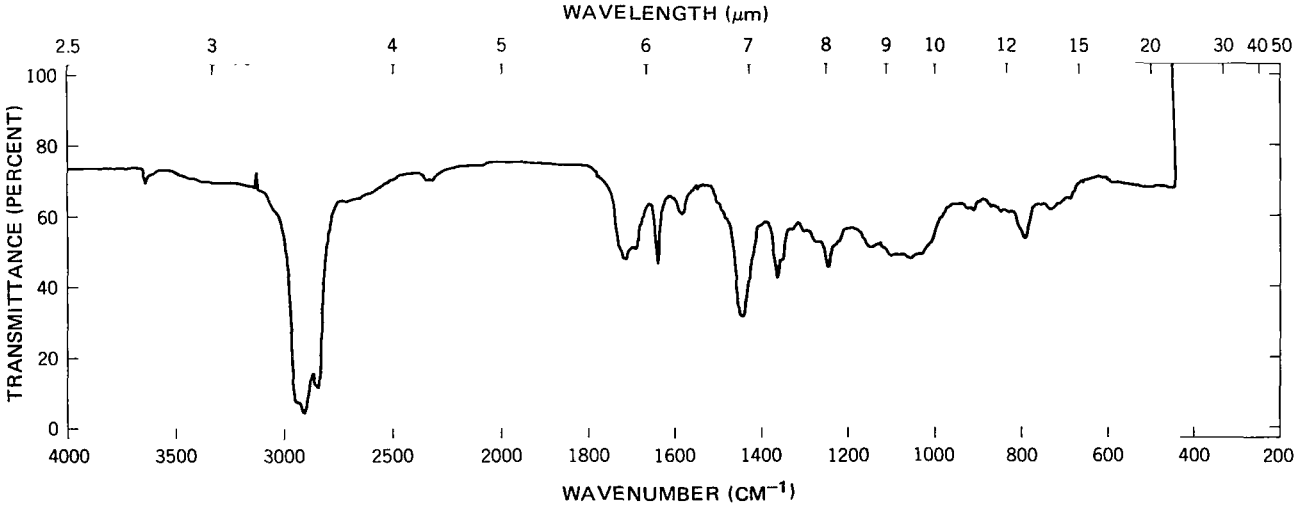

Figure 8—IR spectrum of Spacecraft 1 cold plate condensables.

by combined gas chromatography/mass spectroscopy with a computer interface (Figure 9) and read-out of mass lists and relative abundances by teletypewriter, a typical example of which is the DEHP spectra shown in Figure 10. Analysis of the raw outgassing mixtures from Spacecraft 1 produced the poor chromatogram shown in Figure 11. Although DEHP was identified from this separation, it was apparent that the large unresolved background of hydrocarbons would obscure further identification. To eliminate this problem the crude outgassing mixture was first subjected to a simple liquid chromatographic separation using isooctane followed by chloroform eluting through a silica gel column. The separated fractions were again subjected to the gas chromatography/mass spectroscopy procedure with somewhat more gratifying results. First, the isooctane fraction (Figure 12) contained most of the hydrocarbons (still unresolved) as well as two fumaric acid esters. More polar materials including a large number of silicones and DEHP were contained in the first chloroform fraction (Figure 13); the second chloroform fraction (Figure 14) contained mostly esters of which only a few were definitely identified. Although these separations are somewhat crude and arbitrary, they do indicate the wide variety of volatile plasticizers that can be collected into an experimental package. Attempts to localize the sources of these materials were complicated by the inability to test experimental packages separately

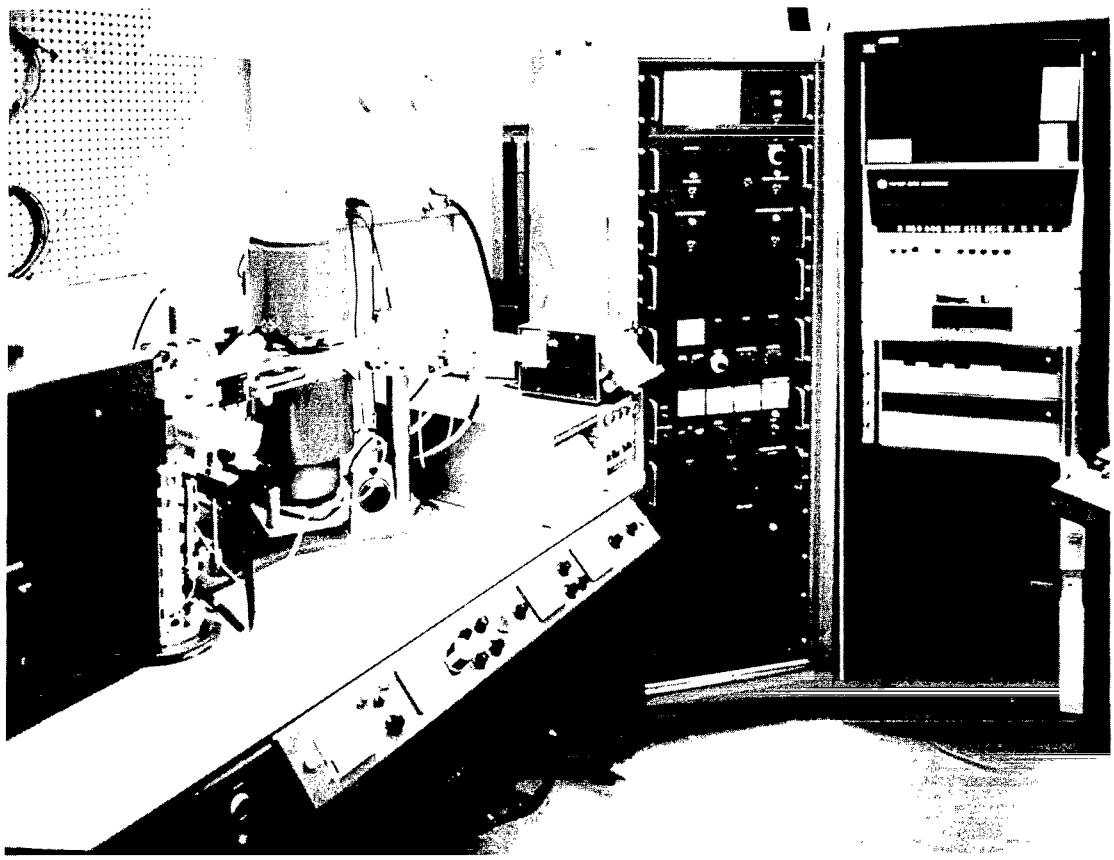

Figure 9—Computerized gas chromatography and mass spectroscopy equipment for identification of compounds.

```
01  MTR, 470,
02  MASCON
03  MTW
04  SUB, 470, 469,
05  MLIST
06
RUN
BACKGRND PEAKS MISSING
PEAKS  BCKGRND     70
6/1/71  /SPEC#  486/LM/
   BASE          SUM
   29031        248730
```

PEAK	INT	I/BASE	MASS	TEST
1	1466	5.04%	18	-1
3	2722	9.37%	27	-2
4	1730	5.95%	28	-2
5	6940	23.90%	29	-1
7	2499	8.60%	39	-2
9	12853	44.27%	41	-1
10	3405	11.72%	42	-1
11	14850	51.15%	43	-1
12	13404	46.17%	55	-1
17	5312	18.29%	56	-1
18	19597	67.50%	57	-3
19	1909	6.57%	68	-1
23	18904	65.11%	69	-0
24	15749	54.24%	70	0
25	3048	10.49%	75	3
28	4434	15.27%	83	-0
33	5227	18.00%	104	-1
39	5669	19.52%	112	-0
45	7904	27.22%	113	-0
46	29031	100.00%	149	-0
60	8852	30.49%	150	-0
61	21882	75.37%	167	-0
63	2499	8.60%	168	-0
75	3453	11.89%	279	0

Figure 10—Mass list computer presentation.

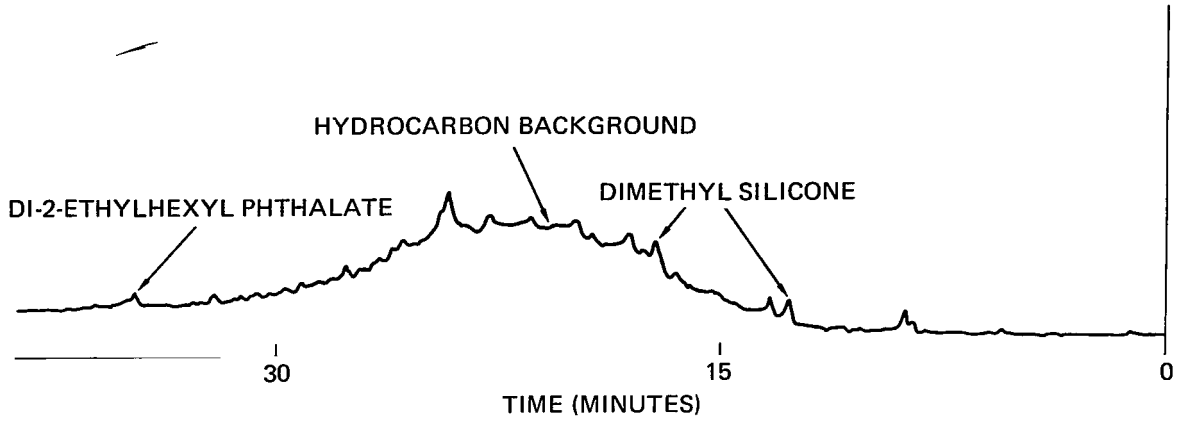

Figure 11—Spacecraft 1 outgassing products. Column length: 30 m (100 ft). Column material: OV17. Program: 373 to 563 K (100° to 290° C) at 5 K/min (5° C/min).

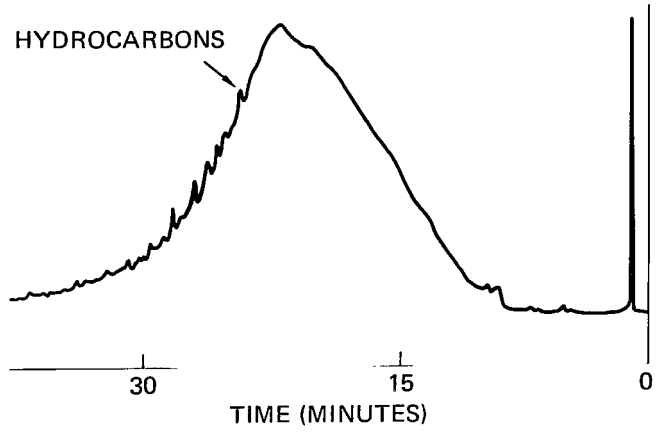

Figure 12—Isooctane fraction of Spacecraft 1 outgassing (contains 65 percent of total sample).

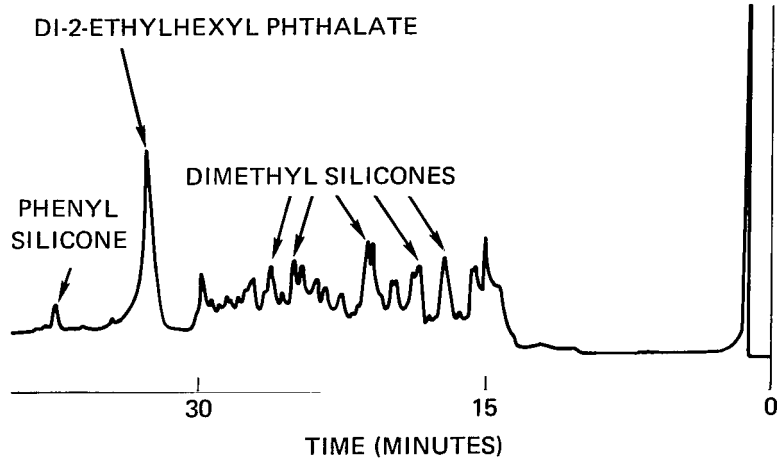

Figure 13—First chloroform fraction of Spacecraft 1 outgassing (contains 8 percent of total sample).

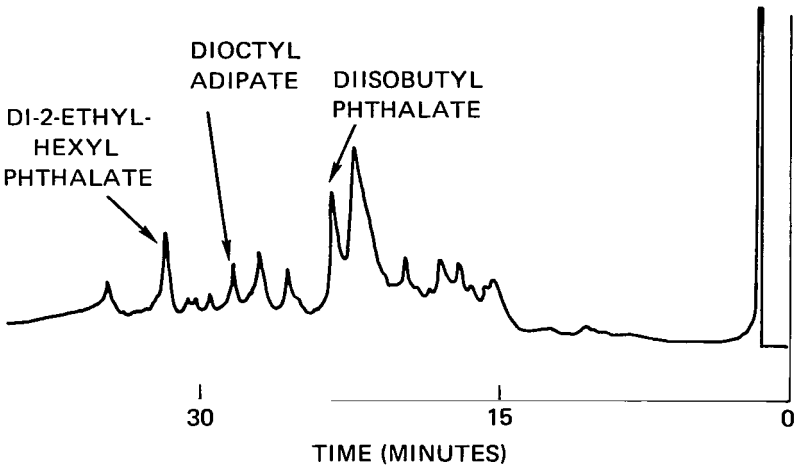

Figure 14—Second chloroform fraction of Spacecraft 1 outgassing (contains 17 percent of total sample).

and the lack of complete materials lists. In the case of DEHP, the complication was that it was found to be an outgassing contaminant from all of the individual modules tested. Outgassing from three of these separate module tests is shown in Figures 15, 16, and 17.

Methyl silicone contamination seen in these outgassing residues is caused by the use of unapproved silicone cements and extensive silicone potting compounds for electronics. Examination of the spacecraft revealed that some of the hydrocarbons may have come from heavily waxed ties used to bundle electronic wiring, incompletely removed machining oil, and various greases. However, because the phthalate contamination emanates from nearly all parts of the spacecraft, it is apparent that future projects must be governed by more strict scrutiny of materials to eliminate, as nearly as possible, all plasticized compounds.

The ubiquitous nature of phthalates as a contaminating species on spacecraft is further demonstrated by similar analyses of outgassing residues from other projects. For example, Spacecraft 2 released large amounts of methyl silicones and DEHP (Figure 18). A dramatic example of phthalate outgassing was demonstrated by a module from Spacecraft 3 (Figure 19) that produced an almost pure DEHP. Similar occurrences of phthalates have been observed in hundreds of samples; and in spite of considerable effort toward controls, the frequency of appearance of phthalates remains somewhat constant.

CONCLUSION

Repeated washing of exposed spacecraft surfaces with high-purity anhydrous isopropyl alcohol followed by vacuum baking has been somewhat successful in reducing the overall contamination levels. Incidentally, no conclusive evidence exists of failure of a spacecraft or a particular module because of phthalate contamination. Of course, it is very difficult to have conclusive evidence of the cause of failure on a nonrecoverable orbiting piece of hardware.

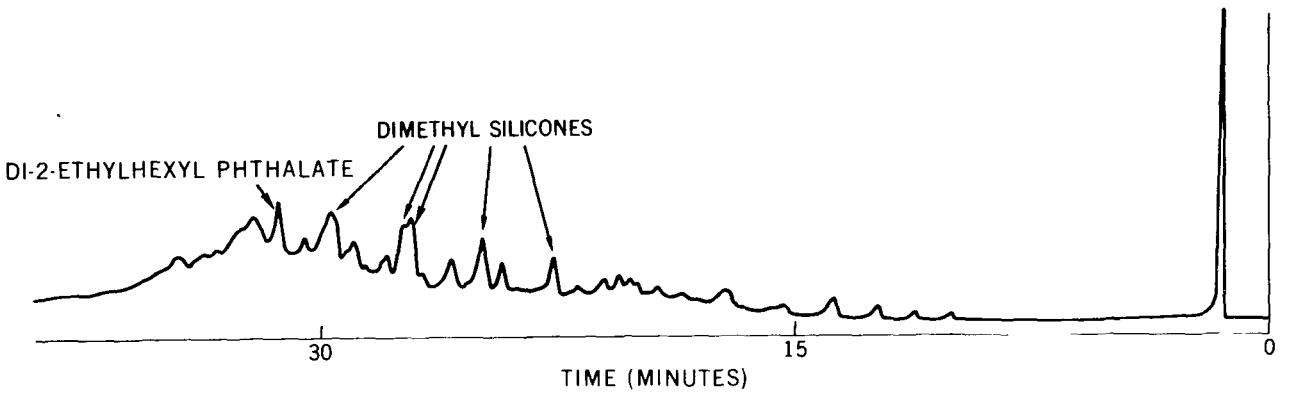

Figure 15—ac fields module outgassing products of Spacecraft 1.

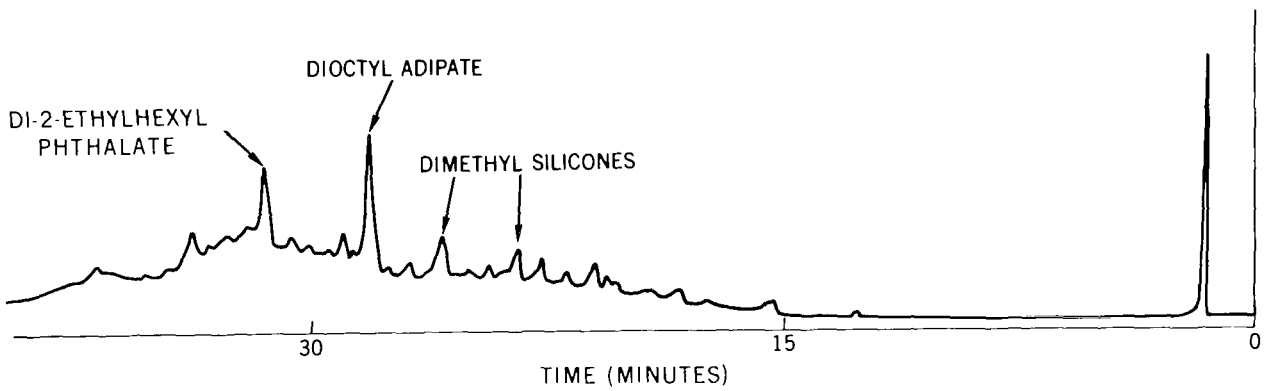

Figure 16—Channeltron module outgassing products of Spacecraft 1.

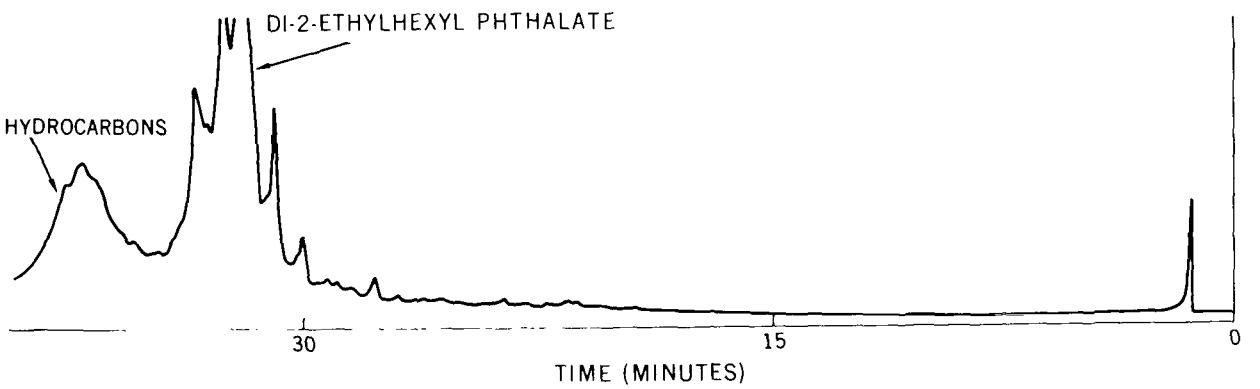

Figure 17—Preamps module outgassing products of Spacecraft 1.

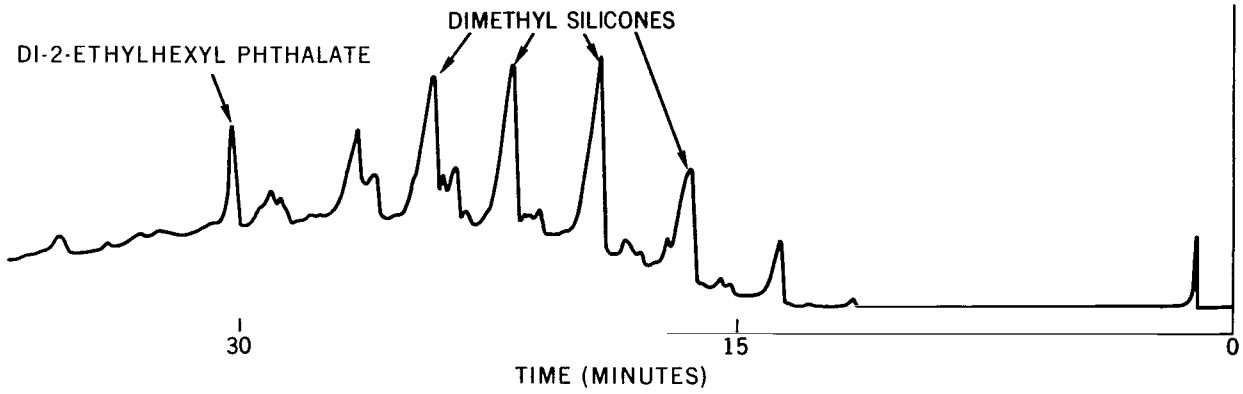

Figure 18—Spacecraft 2 outgassing products. Column length: 30 m (100 ft). Column material: OV17. Program: 393 to 563 K (100° to 290° C) at 5 K/min (5° C/min).

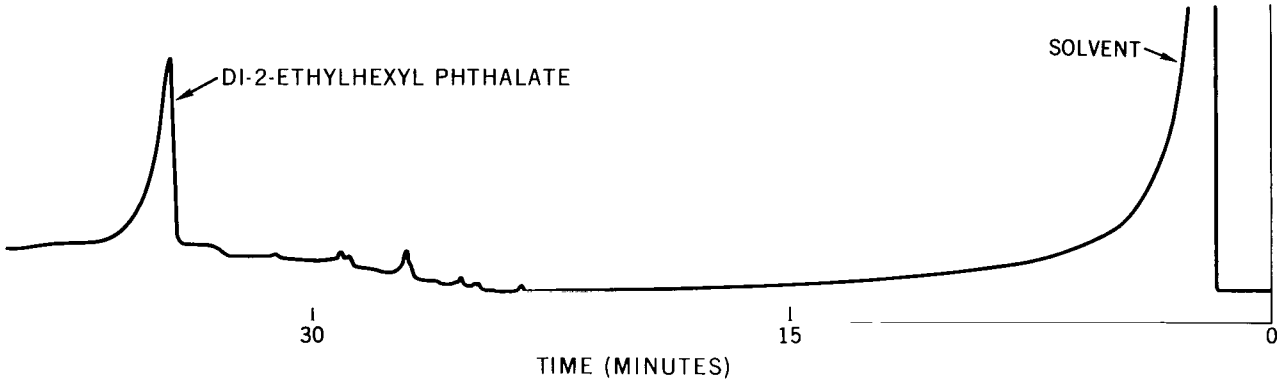

Figure 19—Spacecraft 3 module outgassing products. Column length: 30 m (100 ft). Column material: OV17. Program: 393 to 563 K (100° to 290° C) at 5 K/min (5° C/min).

With the advent of new and advanced space programs, the size and complexity of payloads, launch vehicles, and test chambers continue to grow. Those working on the development of a manned orbiting laboratory such as Skylab must consider not only these problems but in addition the problems of long-term environmental stabilization and control for the well-being of personnel. As a result of these developments it can be anticipated, and, in fact, preliminary evidence exists, that phthalate as well as other types of contamination problems will emerge on even a larger scale than previously experienced. This does not seem like the type of problem for which there is any straightforward solution; therefore, people connected with all aspects of the space program must be made fully aware of the contamination pitfalls and work to minimize them so that they will no longer pose a threat to the success of a program.

Goddard Space Flight Center
National Aeronautics and Space Administration
Greenbelt, Maryland, February 11, 1972
831-41-75-01-51

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