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AMRL-TR-68-27

AD 675177

IDENTIFICATION OF VOLATILE CONTAMINANTS OF SPACE CABIN MATERIALS

J. V. PUSTINGER, JR.

F. N. HODGSON



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FOREWORD

The study was conducted at the Dayton Laboratory of the Monsanto Research Corporation, Dayton, Ohio, under Contract No. F33615-67-C-1357. The principal investigator was Mr. F. Neil Hodgson for the Monsanto Research Corporation. The study was started in January 1967 and was completed in February 1968. The entire study was under the project leadership of Mr. John V. Pustinger, Jr., of Monsanto Research Corporation.

This research was initiated by the Chemical Hazards Branch, Toxic Hazards Division, Biomedical Laboratory in support of Project 6302, "Toxic Hazards of Propellants and Materials," Task 630204, "Environmental Pollution." Mr. Solomon Brokeshoulder and Dr. Gerd A. Kleineberg of the Chemical Hazards Branch were contract monitors for the Aerospace Medical Research Laboratories.

The authors acknowledge the invaluable assistance of Mr. Bruce E. Boggs, Mr. Donald Q. Douglas, and Mr. John E. Strobel; all of Monsanto Research Corporation.

This technical report has been reviewed and is approved.

WAYNE H. McCANDLISS Technical Director Biomedical Laboratory Aerospace Medical Research Laboratories

ABSTRACT

Fifty-three candidate materials for space cabin construction were tested to establish volatile gas-off and oxidation products. Testing was accomplished by two methods:

- (a) preliminary screening by thermogravimetric analysis to determine weight loss during 24 hours at 25°C to 68°C in a nitrogen atmosphere at 5 psia.
- (b) storage tests at 68°C for 72 hours and at 25°C for 30 and 60 days in oxygen at 5 psia, followed by analyses of the chamber gases.

The preliminary screening by measuring weight loss was to determine those materials which exhibit weight losses between 0.001% and 1.0%, exclusive of water. Materials falling within this range were studied further in storage tests to determine the nature of the individual components evolved from the candidate material. Those materials falling outside this range were conditionally excluded from further tests. Weight loss data, thermogravimetric curves, gas chromatograms of volatile contaminants, and the nature and quantities of individual components evolved from the candidate materials are reported.

In addition to the gas-off experiments, gas chromatographic and mass spectrometric analyses were performed on 7 samples of atmospheres from bio-environmental systems.

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SECTION I

INTRODUCTION

Previous material evaluation $\operatorname{programs}^{1,2}$ have shown that certain candidate space cabin materials, when tested under simulated space cabin environmental conditions (5 psia oxygen or air at 25°C or 68°C), yield an abundance of volatile contaminants to the atmosphere. In these programs, storage systems and analytical methods had been devised to identify and to measure quantitatively the gas-off products in the gas phase. As a continuation of these studies, an evaluation program, based on weight-loss measurements and on the storage tests and attendant analyses used previously, has been developed for screening 53 candidate space cabin materials.

Storage tests and subsequent analyses of the gases in the chamber atmosphere are time-consuming. An initial screening of the materials is desirable to discover the materials that give an abundance of volatile contaminants, and those that yield practically none. On this basis, materials could be selected for further testing or conditionally eliminated from additional tests. Materials giving moderate amounts of contamination would be tested further to identify and to measure individual contaminants.

The merit in the use of weight-loss measurements is the comparative simplicity of the equipment and the relatively short measurement time. The intrinsic limitation of this approach is the inability to identify directly the evolved products. On the other hand, the advantage of closed chamber storage tests is the ease with which identification and quantitative analyses of the head gases can be performed; however, the disadvantages are the time-consuming processes of storing specimens for long periods and of performing the identifications of the volatiles. Our program was developed to take advantage of the best characteristics of both approaches.

¹Pustinger, J. V., Hodgson, F. N., Ross, W. D., 1966, Identification of Volatile Contaminants of Space Cabin Materials, AMRL-TR-66-53, Aerospace Medical Research Laboratories, Wright-Patterson Air Force Base, Ohio, pp. XVI + 194.

²Pustinger, J. V., Hodgson, F. N., 1967, Identification of Volatile Contaminants of Space Cabin Materials, AMRL-TR-67-58, Aerospace Medical Research Laboratories, Wright-Patterson Air Force Base, Ohio, pp. XVI + 194 The initial screening procedure, using thermogravimetric techniques for measuring weight loss at moderate temperatures (ambient to $68 \pm 2^{\circ}$ C) for 24 hours in 5 psia nitrogen, was developed to select those candidate materials that lose from 0.001 to 1.0% of their weight, excluding water. The selected materials are then stored in 9-liter chambers at $68 \pm 2^{\circ}$ C for 72 hours and at 25 ± 2°C for periods of 30 and 60 days. Atmosphere in the chambers is oxygen at a pressure of 5 psia. The gaseous contaminants evolved from the test materials are identified by combinations of gas chromatography and mass spectrometry.

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SECTION II

GAS-OFF EXPERIMENTS

A. EXPERIMENTAL METHOD

1. Types of Candidate Materials and Sample Preparation

Table I lists the candidate materials for cabin construction used in these experiments; all materials tested are commercial products provided by the Government. Whenever possible, the candidate materials were studied in the same state as received. Materials such as paints and inks were applied to an aluminum foil substrate and then dried at the designated temperature and for the designated time according to the manufacturer's or the Air Force's directions. Similarly, two-part resins were mixed and cured according to procedures provided by the manufacturers. All calculations were made on the basis of dry sample weight.

Specimens used for thermogravimetric analysis (TGA) were conditioned at 23°C in a desiccator over phosphorus pentoxide for 24 hours prior to testing. For storage tests at 72 hours, 30 days, and 60 days, no pretreatment of samples was performed beyond the curing procedures cited by the manufacturers or the Air Force. The procedure for preconditioning the TGA specimens was devised to minimize adsorbed water and to put all samples on the same basis for comparing relative weight loss.

For storage tests for 72 hours, 30 days, and 60 days, a weighed portion of each sample was placed into a 9-liter chamber in a manner to expose the largest possible surface area. Generally, approximately 10-gram specimens were used; however, in cases where less sample was available, or when the bulk volume of the sample was excessively large, smaller specimens were used. When the bulk volume was too large and subdividing was necessary, freshly exposed surfaces were further cured at ambient conditions, i.e., 23°C and atmospheric air pressure, for 30 days or a minimum of 14 days.

Individual specimens of each candidate material were contained in 9-liter, borosilicate glass chambers for periods of 30 and 60 days at $25 \pm 3^{\circ}$ C, and for a period of 72 hours at 68 $\pm 2^{\circ}$ C, under an oxygen atmosphere at 5 psia and 20-40% relative humidity. The chamber design and pretreatment of the chambers were the same as reported earlier¹. Two-hundred chambers were used (on a staggered schedule) over a span of 13 months to permit analyses to be performed after 72 hours,

¹Pustinger, Hodgson, and Ross, p.1.

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Table I

SPACE CABIN TEST MATERIALS

Material	Irradiated Polyolefin, Thermofit RNF, CRN	Mylar Polyester	Irradiated Polyolefin, Thermofit RNF	Eccobond 70C Adhesive	Thermoplastic Coating,	ll69 A/B Coating	Stycast 1090 Cat-1	Stycast 2651-50 CAF 1	Silicone RTV 521	Adiprene L-100 MCC Adhesive	Silicone RTV 502 Rubber	Silicone Q30079	Silicone RTV 503	Tape, Stycast 2651-Cat 9	O11, Aero Shell #7	Paint, Lacquer Stik White	Conductive Paint	Resin Emerion 310	Varnish E44	Polyurethane PC-18	Ink F-150	Ink #41 Black	Ink Red Marking VF-200	Nylon Cord Style 18	Nylon Cord Style 21	Lastic 60,000 RF1	
Code No.	AF 209	AF 251	AF 266	AF 302	AF 325		AF 402	AF 403	AF 406	AF 421	AF 451	AF 454	AF 459	AF 495	AF 505	AF 515	AF 522	AF 527	AF 528	AF 533	AF 536	AF 537	AF 540	AF 551	AF 552	AF 561	
Material	Schjeldahl (Mylar) X850 WM 238 Nit+vile/Phancito	RTV 731 Silicone	Saran Wrap - Type 18	Aclar Type 33-C	Shonka A-150 Polyolefin/Polyamide	Spandex Lycra Polyurethane	PR 1535 Polyurethane	RTV 615	Stycast 2651/Catalyst II (Epoxy)	Hathone HA 7236	Butyl Rubber 00996-33L	Polyurethane 00996-39B	Silicone 19513-10f	Boltaron ABS Polymer	Epoxy 760A	Merlon 1000 Polycarbonate	Silicone, Type A #428/132	Bondmaster E611 (Modified Epoxy)	Epoxy-Polyamide, V-9 Silver Epon 815	Silicone EMS 323	Viton A EMS 338 (Fluoro Elastomer)	Silicone Rubber EMS 342	Silicone Rubber EMS 345	Hapalon Rubber EMS 355	Urethane EMS 366	Polyester (Dacron) Silicone	Epoxy Primer M-602
Code No.	DAC 001	DAC 003	DAC 005	DAC 006	DAC 007	DAC 008	DAC 011	DAC 012	DAC 013	DAC 014	DAC 017	DAC 018	DAC 019	DAC 020	DAC 021	DAC 022	AF 023	AF 053	AF 054	AF 063	AF 064	AF 065	AF 066	AF 068	AF 071	AF 073	AF 203

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30 and 60 days. Control chambers (containing only aluminum foil) were processed concurrently with those chambers containing the test materials. No contamination was detected from the control chambers.

2. Preparation of Chamber Atmospheres

After each specimen was inserted into the 9-liter chamber, the test chamber was filled to a pressure of one atmosphere with oxygen, saturated with water vapor. The gas was saturated with water by bubbling 99.5% oxygen (conforming to Type I of MIL-O-27210) through triply distilled water at 23°C. Test conditions were attained by subsequently reducing the pressure in the chamber to 5 psia, resulting in a test atmosphere of oxygen at 5 psia with a relative humidity of approximately 33%.

Measurement of relative humidity was made with an Alnor Type 7300 Dew-Pointer (Illinois Testing Laboratories, Inc., Chicago, Ill.).

Test atmospheres were maintained at $25 \pm 3^{\circ}$ C by storing chambers in a temperature-controlled room for periods of 30 and 60 days. The chambers that were tested at $68 \pm 2^{\circ}$ C were stored in a constant-temperature cabinet (Blue M Electric Co., Stabil-Therm DL132C).

3. Analytical Methods

a. Weight Loss Measurements

Conditional screening of candidate materials was performed by measuring the weight loss of the material using thermogravimetric measurements (TGA). Weight loss from approximately 10 g of a material was recorded continuously as the temperature of its environment was raised from ambient (approximately 23°C) to $68 \pm 1^{\circ}$ C in 4 hours and then maintained at $68 \pm 1^{\circ}$ C for 20 hours or until weight remained constant for 2 hours. All TGA measurements were made in dried, prepurified nitrogen at 5 psia.

Thermogravimetric measurements were made with a Cahn RH Electrobalance equipped with a modified F&M Model 240-00 Power Proportioning Temperature Programmer, Flo-Thru tube, a temperature programmed oil bath, and a 1 mv recorder (Figures 1, 2, and 3). The Cahn RH Electrobalance is a vacuum- and controlledatmosphere automatic recording balance with 100-gram capacity





SCREENING APPARATUS THERMOGRAVIMETRIC

- B. Matheson Absolute Pressure Regulator A. Drierite and P2O5 Drying Tube F. Calcium Carbide Reactor Tube l.Dewar Flask with Liquid N₂ C. Cahn Electrobalance G Gas Sampling Valve J. Sampling Loop H. Toggle Valves K. Needle Valve E. Tare Weight L.Flowmeter D. Sampla
 - O. Auxiliary Evacuation Line Q. Temperature Programmer M.^eump isolation Trap P.Oil Bath N. Pump

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Figure 2. Cahn Electrobalance in TGA System.



Figure 3. Complete TGA System.

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and a sensitivity of 10^{-6} g. This thermogravimetric system will permit the handling of relatively large specimens, 1-10 grams, and the detection of small weight changes, a 10^{-7} fraction of the load.

The arrangement of the components of the TGA system is shown in Figures 1, 2, and 3. To permit greater control and more uniform heating, the sample is heated with an oil bath rather than an oven or furnace. The slow heating rate of one degree every six minutes is more easily achieved by adjusting the temperature of the oil bath than by using a heating unit directly. The temperature was programmed with an F&M Power Proportioning Temperature Programmer that had been modified to permit the slow heating rate.

Water evolved from the sample was monitored by either of two gas chromatographic methods:

- (a) by converting the water to acetylene by a reaction with calcium carbide and analyzing for acetylene;
- (b) by cryogenically trapping the desorbed water and subsequently performing a direct measurement for water.

Gas chromatographic analyses for acetylene were performed with a flame ionization detector (F&M 1609) and a Porapak T column, whereas direct gas chromatographic measurements for water were performed with a thermal conductivity detector (Barber-Colman 61C) and a Porapak T column.

b. Gas Chromatographic Analysis of Gas-Off Products

Carbon monoxide, methane, and gas chromatographic analyses were performed by techniques reported earlier^{1,2}. All atmospheres in the test chamber were sampled for analysis at the temperature of the test, i.e., 25° C or 68° C.

The general analyses of the gas-off products by gas chromatography were performed on an F&M Model 810 Research Gas Chromatograph equipped with dual flame ionization detectors. A general purpose column, 20-ft x 0.25-in. ss., 20% Triton X-305 on 60/80 mesh Gas Chrom Z was used for most samples. Gas-off products from four samples (DAC 006, DAC 017, DAC 018, and DAC 020), tested in the first quarter of the program, were analyzed by means of a double column, 20-ft x 0.25-in. ss., 5% Carbowax 20M on 60/80 mesh Gas-Pack F with a pre-column of 12-ft x 1/8-in. ss., 7% neopentylglycolsuccinate on 60/80 mesh Gas Pack F, used

¹Pustinger, Hodgson, and Ross, p.l. ²Pustinger and Hodgson, p.l.

earlier². The advantage of using the Triton X-305 column is better separation of early eluting components.

Quantitative gas chromatography data were obtained by comparing the peak heights with those of a standard mixture. Gas chromatographic instrument conditions are presented in Appendix III, Table XLV.

Identifications of gas chromatographic components were made by mass spectrometric analysis of the gas chromatographic effluent. In most cases component identification was accomplished by the direct, tandem coupling of a fast scan mass spectrometer, CEC 21-104, to the gas chromatograph. By splitting the effluent, a portion was directed to the flame ionization detector and a second portion was introduced directly into the mass spectrometer. With some samples, a concentration step requiring the cryogenic trapping of the major portion of the total 9-liter volume was necessary. This condensate was subsequently separated into its components and characterized by the coupled gas chromatograph-mass spectrometer system.

c. Mass Spectrometric Analysis of Gas-Off Products

Two types of mass spectrometric analyses were performed for each sample. A composite analysis¹ of the atmosphere of each 9-liter bottle was made on an aliquot (125 cc) of the atmosphere with a Consolidated Electrodynamics Corporation Model 21-103C Mass Spectrometer. As indicated in Section II-A-3-b, a fast scan Consolidated Electrodynamics Corporation Model 21-104 Mass Spectrometer was used in a direct couple with a gas chromatograph to identify the components eluting from the chromatograph. Both approaches are necessary to insure complete characterization of the chamber atmospheres.

Identifications of individual components were made by mass spectrometry and were supported by infrared absorption and by gas chromatographic data as needed. Most of the mass spectra obtained were compared to API (American Petroleum Institute) reference spectra. In cases where the required mass spectrum does not appear in the API collection, comparison was made with spectra from our laboratory files or from the literature.

B. __RESULTS AND DISCUSSION

Table II lists the types of compounds detected in the chamber atmospheres. These data represent compounds exclusive of H_2O , CO_2 , O_2 , and N_2 .

¹Pustinger, Hodgson and Ross, p.1 ²Pustinger and Hodgson, p.1

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Table II

TYPES OF COMPOUNDS DETECTED

I. Inorganics

Carbon Monoxide

II. Alkanes

C. Hydrocarbon(s) C: Hydrocarbon(s) C: Hydrocarbon(s) Methane Propane

III. Alkenes

Ethylene C. Unsaturated Hydrocarbons Di-isobutylene Isobutylene Propylene Tetra-1sobutylene Trichloroethylene Tri-isobutylene

IV. Alcohols

n-Butanol 2-Butanol tert-Butanol 2-Buten-1-ol 2-r-Butoxyethanol Cyciohexanol Ethanol 2-Ethoxyethanol Isopropanol Methanol 3-Methyl-1-butanol 3-Methyl-1-propanol 2-Phenyl-2-propanol n-Propanol 2-n-Propoxyethanol

V. Alkyl Halides

Chloroform Homologous Series of Chloro-fluorocarbons Trichloromonofluoromethane

VI. Carboxylic Acids and Their _____Derivatives

> Acetic Acid 2-n-Butoxyethylacetate Butylacetate 2-Ethoxyethylacetate Ethylformate Formic Acid Methylformate Propylacetate

VII. Aldehydes

Acetaldehyde Butyraldehyde Formaldehyde Propionaldehyde Valeraldehyde

VIII. Ketones

Acetone Acetophenone 2-Butanone (Methylethylketone) Cyclopentanone Di-isopropylketone Di-n-propylketone Hexanone 4-Methyl-2-pentanone (Methylisobutylketone) Pentanone

IX. Ethers

l,3-Dioxane Propylene Oxide N-Methylmorpholine

X. Aromatic Hydrocarbons

Benzene C: Alkylbenzenes C: Alkylbenzenes Ethylbenzene Methylstyrene Naphthalene Styrene Toluene Xylenes

XI. Aromatic Hydroxy Compounds Phenol

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XII. Silicon Compounds

Various Cyclic and Linear Methylsiloxane Polymers

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Materials that showed no products (exclusive of H_2O , CO_2 , O_2 , and N_2) are listed in Table III.

Materials showing weight loss in excess of 1% and for which no analytical data were obtained are listed in Table IV. Although these materials were conditionally screened and eliminated from further testing based on TGA measurements, all candidate materials in this program were processed in storage tests for 72 hours, 30 days, and 60 days, and their gas-off products were analyzed with gas chromatography and mass spectrometry to confirm the results of the TGA measurements. In most cases, where the weight loss exceeded 1%, gas chromatographic and mass spectrometric data were obtained, only to establish the magnitude of the total off-gases. No specific identifications or measurement of quantities of individual components have been made. Others were analyzed completely for comparison purposes with the TGA data.

Analytical data are presented in Appendix I, Table V, Figures 4 to 56 (Thermogravimetric Weight Loss Data and TGA Curves); Appendix II, Tables VI to LXIV (Analytical Results for Gas-Off Experiments); and Appendix III, Figures 57 to 95 (Gas Chromatograms for Gas-Off Experiments).

All values appearing in the tables of Appendices I and II are calculated on the basis of the dried or cured samples (this is important in the case of paints and coatings where the weight of the material is substantially reduced by drying).

Some gas-off products are identified by compound type only, e.g., alkylbenzene(s), C4 alkylbenzene(s), or C4 hydrocarbon. In these cases several homologues or isomers may be present; however, they have not been identified individually.

Some of the gas-off products from silicone base materials were also calculated collectively. These were the volatile linear and cyclic siloxane polymers (having dimethyl siloxy groups as monomer units), which had been observed in previous gas-off studies^{1,2}. Although separate peaks are noted in the gas chromatograms (Appendix III), these volatile silicones are listed collectively in the tables of gas-off data (Appendix II) as silicone oil.

Although no distinct correlation of composition and the shape of the TGA curve can be derived from the limited data, some similarities in TGA curves are noted, e.g., two materials containing polyamide [DAC 007, Shonka A-150 Polyolefin/Polyamide (Figure 9) and AF 054, Epoxy-Polyamide, V-9 Silver Spon 815 (Figure 23)]. Also, certain similarities in the total weight loss data are observed for materials of a particular type, e.g.,

¹Pustinger, Hodgson and Ross, p.1. ²Pustinger and Hodgson, p.1

Table III

CANDIDATE MATERIALS EXHIBITING NO GAS-OFF PRODUCTS

DAC	001	-	Mylar, Schjeldahl X850
DAC	603	-	Silicone, RTV 731
DAC	005	-	Saran Wrap, Type 18
DAC	800	-	Polyurethane, Spandex Lycra
DAC	011	-	Polyurethane, PR 1535
DAC	012	-	Silicone, RTV 615
DAC	013	-	Epoxy, Stycast 2651/Catalyst II
DAC	022	-	Polycarbonate, Merlon 1000
AF	064	-	Fluoroelastomer, Viton A, EMS 338
AF	561	-	Fabric, Lastic 60,000 RF1

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Table IV

CANDIDATE MATERIALS CONDITIONALLY WITHDRAWN FROM FURTHER TESTING

(Based on Excessive Weight Loss and Preliminary Gas Chromatographic and Mass Spectrometric Measurements)

AF 325 - Thermoplastic Coating, 1169 A/B AF 533 - Polyurethane, PC-18 Coating AF 536 - Ink, F-150 Marking AF 537 - Ink, #41 Black AF 540 - Ink, VF-200 Red Marking

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AF 063 (Silicone Rubber EMS 323), AF 065 (Silicone Rubber EMS 342), and AF 066 (Silicone Rubber EMS 345) lost 0.25%, 0.26%, and 0.26% of their weight, respectively. However, the significance of this observation is not clear since other, non-silicone materials, also exhibit weight loss of similar proportions.

The need for gas chromatographic and mass spectrometric confirmatory evidence in the screening of the candidate materials by TGA measurement results from the inability to measure quantitatively the amount of water evolved during the TGA measurement. The reliability of the two gas chromatographic methods for water, i.e., (a) conversion to acetylene and subsequent acetylene analysis, and (b) direct analysis for water, was established through the use of known water standards. However, difficulty was encountered when attempting to distinguish the water evolved from the sample, from that present in the pre-dried, pre-purified nitrogen used as the atmosphere and from that adsorbed on the inner surface of the TGA apparatus.

Although the amount of water present in the apparatus is very small, apparently there are sufficient quantities adsorbed on the glass at 23 to 68°C to yield a relatively large amount into the gas phase during a 24-hour test. Although not conclusive, an apparent adsorption-desorption equilibrium is established with the sample, metal and glass surfaces of the apparatus, and the gas phase, such that a relatively constant amount of water (20-28 mg) is isolated during a 24-hour test. Experiments performed without sample to establish the background level of water yielded similar quantities of water. To eliminate this problem, the apparatus is being modified to provide a direct measure of water desorbed from the sample by locating a hygrometer probe at the sample site.

A comparison of weight loss data (23 to 68°C) with the quantitative analyses of volatiles from storage tests at 68°C should be made with care. Several opposing effects should be noted. A large number of materials continue to desorb considerable quantities of volatiles after 24 hours. Thus, more gas-off products should be present in the atmosphere during 72-hour tests than gas-off products detected as weight loss during 24-hour TGA measurements. However, adsorption of volatiles on chamber surfaces results in an opposing effect. Considerable quantities of polar and relatively non-volatile gas-off products are retained on the chamber surfaces in the storage tests even at 68°C. Oily films were deposited on chamber walls in many of the tests. This effect was particularly noticeable when testing silicones, in that, relatively low molecular weight silicone oil condensed on the chamber surfaces. Similarly, considerable amounts of butoxyethylacetate (identified by infrared spectra) were collected on the surfaces of test chambers for AF 522.

Material No. DAC 014, Hathone HA 7236, a polymeric foam, showed an unusual behavior during the TGA measurements. Solid material in the form of a fine dust was lost from the crucible. This was perhaps due to the rupture of gas-filled bubbles resulting from the decreased pressure (5 psia). Collection of the dust for weighing was difficult due to the static charge associated with this type of material, but it was estimated to be approximately 2-3 mgs for a 2-g specimen. The formation of this dust could perhaps, in itself, be considered a significant atmospheric contaminant.

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SECTION III

BIO-ENVIRONMENTAL AND SPECIAL ANALYSES

A special study was performed to determine the gas-off products of Hetron Polyester Resin. This material is not a candidate space cabin material, but it is of interest to the Government as a construction material for terrestrial use. Ten grams of the material was stored in a 9-liter gas-off chamber for 30 days in an air atmosphere at a pressure of 1 atmosphere at 25°C. The most abundant product observed in the off-gases was methylethylketone. This compound undoubtedly arises from methylethylketone peroxide used in the formulation of the polymer. However, due to lack of data on the stability of MEK peroxide, it has not been established whether the peroxide decomposes during storage or during analysis. Table XLVI lists the gas-off products from this material.

Special analyses Nos. 2 and 3 were performed on air specimens collected on 28 March 1967 from an Air Force test chamber and from the room air immediately outside the chamber. Data from these analyses are shown in Table XLVII.

Special analyses Nos. 4, 5, 6 and 7 were performed on bioenvironmental specimens contained in four 50-liter cylinders which were received from the Air Force on 25 August 1967. The results, given in Table XLVIII, are exclusive of water and carbon dioxide. It will be noted that low molecular weight chlorinated fluorocarbons are present in cylinder #1 (sample No. 4). These appear to be similar to the volatiles which have been observed to arise from certain fluorocarbon lubricants.

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SECTION IV

CONCLUSIONS AND RECOMMENDATIONS

Considerable differences in levels of volatiles as determined from weight-loss measurements (TGA) and from storage tests and analyses of atmosphere of the storage chambers are observed. A major contributing factor is the adsorption of volatiles on the chamber walls.

The application of thermogravimetric measurements for screening candidate materials has proven useful, but a more direct measurement of water at the sample site must be made to provide more reliable data. We recommend the use of a hygrometer probe at the sample site. Such a device is being incorporated in a modification of the TGA system used in this test program. Until a reliable direct water analysis can be performed, we recommend continued 72 hours storage tests at 68°C and attendant atmosphere analyses to confirm the level of volatile products desorbed from the candidate materials.

APPENDIX I

THERMOGRAVIMETRIC PATTERNS OF CANDIDATE SPACE CABIN MATERIALS

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The thermogravimetric analysis (TGA) patterns shown in this appendix were obtained on a Cahn RH Electrobalance. Comparison of the weight loss patterns should be made with care since varying amounts of sample were used to obtain the TGA patterns. The quantity of material used for each TGA measurement is shown on the reproduced pattern.

TGA curves appear in order of their Air Force serial numbers. Names of materials are those submitted by the Air Force.
Table V

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TOTAL WEIGHT LOSS AS DETERMINED BY THERMOGRAVIMETRIC ANALYSIS OF CANDIDATE MATERIALS

Weight Loss (%)	0.75 0.011	0.090	0.17	0.13	2.74	0.053	0.087	0.094	0.22	0.31	0.14	0.17	0.039	0.11	0.32	1.63	1.52	0.85	1.40	3.29	1.03	2.24	0.44	0.52	0.030	
Weight Loss (mg)	39.83 0.92	5.72	10.40	14.12	66.60	5.08	9.16	11.02	25.50	29.96	9.40	16.92	3.96	8.48	10.50	160.80	135.32	56.30	102.72	117.70	44.94	53.50	29.62	46.7	3.40	
Sample Weight (g)	5.2887 8.1098	6.3206	6.2638	11.1985	2.4288	9.5619	10.5348	11.7090	11.3395	9.4696	6.7228	9.9523	10.2746	7.8232	3.2812	9.8416	8.9236	6.5904	7.3303	3.5743	4.3758	2.3888	6.6950	8.9585	ll.2570	
Code No.	AF 203 AF 209	AF 251	AF 266	AF 302	AF 325	AF 402	AF 403	AF 406	AF 421	AF 451	AF 454	AF 459	AF 495	AF 505	AF 515	AF 522	AF 527	AF 528	AF 533	AF 536	AF 537	AF 540	AF 551	AF 552	AF 561	
Weight Loss (%)	0.13 0.12	0.21	0.010	0.016	0.062	3.31	0.076	0.061	0.020	0.34	0.10	0.19	0.19	0.026	0.20	0.016	0.073	0.26	0.042	0.25	0.057	0.26	0.26	0.51	0.10	0.052
Weight Loss (mg)	1.90 10.82	11.41	0.21	06.0	6.60	53.5	7.14	6.1	2.01	6.96	10.44	18.60	13.60	2.44	19.70	0.81	7.24	22.24	4.24	23.40	5.01	15.90	17.00	51.61	6.12	1,50
Sample Weight (g)	1.4217 8.9116	5.3960	2.0140	5.5000	10.4906	1.6140	9.3311	9.9980	10.0375	2.0283	10.1100	1010.01	7.3359	9.3817	9.9900	5.0624	9.8951	8.5453	9.9953	9.3731	8.8647	6.1683	6.6071	10.2164	6.0081	2.8895
Code No.	DAC 001 DAC 002	DAC 003	DAC 005	DAC 006	DAC 007	DAC 008	DAC 011	DAC 012	DAC 013	DAC 014	DAC 017	DAC 018	DAC 019	DAC 020	DAC 021	DAC 022	AF 023	AF 053	AF 054	AF 063	AF 064	AF 065	AF 066	AF 068	AF 071	AF 073

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Figure 4. TGA Curve of Schjedahl (Mylar) X850 (DAC 001). Specimen Weight - 1.4217 grams

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Figure 5. TGA Curve of FM 238 Nitrile/Phenolic (DAC 002). Specimen Weight - 8.9116 grams

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Figure 6. TGA Curve of RTV 731 Silicone (DAC 003). Specimen Weight - 5.3960 grams

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Figure 7. TGA Curve of Saran Wrap Type 18 (DAC 005). Specimen Weight - 2.0140 grams

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Figure 8. TGA Curve of Aclar Type 33-C (DAC 006). Specimen Weight - 5.5000 grams

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Figure 9. TGA Curve of Shonka A-150 Polyolefin/Polyamide (DAC 007). Specimen Weight - 10.4906 grams



Figure 10. TGA Curve of Spandex Lycra Polyurethane (DAC 008). Specimen Weight - 1.6140 grams

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Figure 11. TGA Curve of PR 1535 Polyurethane (DAC 011). Specimen Weight - 9.3311 grams

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Figure 12. TGA Curve of RTV 615 (DAC 012). Specimen Weight - 9.9980 grams



Figure 13. TGA Curve of Stycast 2651/Catalyst II (Epoxy) (DAC 013). Specimen Weight - 10.0375 grams

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Figure 14. TGA Curve of Hathone HA 7236 (DAC 014). Specimen Weight - 2.0283 grams

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Figure 15. TGA Curve of Butyl Rubber 00996-33L (DAC 017). Specimen Weight - 10.1100 grams



Figure 16. TGA Curve of Polyurethane 00996-39B (DAC 018). Specimen Weight - 10.0101 grams



Figure 17. TGA Curve of Silicone 19513-10f (DAC 019). Specimen Weight - 7.3399 grams



Figure 18. TGA Curve of Boltaron ABS Polymer (DAC 020). Specimen Weight - 9.3817 grams



Figure 19. TGA Curve of Epoxy 760A (DAC 021). Specimen Weight - 9.9900 grams



Figure 20. TGA Curve of Merlon 1000 Polycarbonate (DAC 022). Specimen Weight - 5.0624 grams



Figure 21. TGA Curve of Silicone, Type A #428/132 (AF 023). Specimen Weight - 9.8951 grams



Figure 22. TGA Curve of Bondmaster E611 (Modified Epoxy) (AF 053). Specimen Weight - 8.5453 grams







Figure 24. TGA Curve of Silicone EMS 323 (AF 063). Specimen Weight - 9.3731 grams



Figure 25. TGA Curve of Viton A EMS 338 (Fluoro Elastomer) (AF 064). Specimen Weight - 8.8647 grams



Figure 26. TGA Curve of Silicone Rubber EMS 342 (AF 065). Specimen Weight - 6.1683 grams

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Figure 27. TGA Curve of Silicone Rubber EMS 345 (AF 066). Specimen Weight - 6.6071 grams



Figure 28. TGA Curve of Hapalon Rubber EMS 355 (AF 068). Specimen Weight - 10.2164 grams





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Figure 30. TGA Curve of Polyester (Dacron) Silicone (AF 073). Specimen Weight - 2.8895 grams



Figure 31. TGA Curve of Epoxy Primer M-602 (AF 203). Specimen Weight - 5.2887 grams







Figure 33. TGA Curve of Mylar Polyester (AF 251). Specimen Weight - 6.3206 grams







Figure 35. TGA Curve of Eccobond 70C Adhesive (AF 302). Specimen Weight - 11.1985 grams



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Figure 36. TGA Curve of Thermoplastic Coating, 1169 A/B Coating (AF 325). Specimen Weight - 2.4288 grams

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Figure 37. TGA Curve of Stycast 1090 CAT-1 (AF 402). Specimen Weight - 9.5619 grams



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Figure 38. TGA Curve of Stycast 2651-50 CAF1 (AF 403). Specimen Weight - 10.5348 grams


Figure 39. TGA Curve of Silicone RTV 521 (AF 406). Specimen Weight - 11.7090 grams



Figure 40. TGA Curve of Adiprene L-100 MCC Adhesive (AF 421). Specimen Weight - 11.3395 grams



Figure 41. TGA Curve of Silicone RTV 502 Rubber (AF 451). Specimen Weight - 9.4696 grams



Figure 42. TGA Curve of Silicone Q30079 (AF 454). Specimen Weight - 6.7228 grams

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Figure 43. TGA Curve of Silicone RTV 503 (AF 459). Specimen Weight - 9.9523 grams

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Figure 44. TGA Curve of Tape, Stycast 2651-CAT 9 (AF 495). Specimen Weight - 10.2746 grams



Figure 45. TGA Curve of Oil, Aero Shell #7 (AF 505). Specimen Weight - 7.8232 grams



Figure 46. TGA Curve of Paint, Lacquer Stik White (AF 515). Specimen Weight - 3.2812 grams



Figure 47. TGA Curve of Conductive Paint (AF 522). Specimen Weight - 9.8416 grams

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Figure 49. TGA Curve of Varnish E44 (AF 528). Specimen Weight - 6.5904 grams



Figure 50. TGA Curve of Polyurethane PC-18 (AF 533). Specimen Weight - 7.3303 grams



Figure 51. TGA Curve of Ink F-150 (AF 536). Specimen Weight - 3.5743 grams



Figure 52. TGA Curve of Ink #41 Black (AF 537). Specimen Weight - 4.3758 grams



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Figure 53. TGA Curve of Ink Red Marking VF-200 (AF 540). Specimen Weight - 2.3888 grams



Figure 54. TGA Curve of Nylon Cord Style 18 (AF 551). Specimen Weight - 6.6950 grams



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Figure 55. TGA Curve of Nylon Cord Style 21 (AF 552). Specimen Weight - 8.9585 grams



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Figure 56. TGA Curve of Lastic 60,000 RF1 (AF 561). Specimen Weight - 11.2570 grams

APPENDIX II

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ANALYTICAL RESULTS FOR

GAS-OFF EXPERIMENTS

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Compounds found as gas-off products from candidate space cabin materials are listed in the following tables. Values for the gas-off product levels are given as: milligrams per 10 grams (mg/10 gms) of the cured candidate material. In some cases, either more or less than 10 g of material was used, but each yield of gas-off products was normalized to that of a 10-g sample.

The order of the tables in this appendix is by Air Force serial number. Names of materials are those submitted by the Air Force.

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Table VI

GAS-OFF PRODUCTS FROM NITRILE/PHENOLIC, FM 238

DAC Serial No. 002

	Ne.	ight of Component	
	(mg/10	gms Candidate Mat	certal)
Component	(2 HOULS (68°C)	30 Days (25°C)	60 Days (25°C)
ropylene	3.5	2.0	2.0
Propylene Oxide	N.D.	N.D.	0.08
Sthanol	0.3	0.02	N.D.
Carbon Monoxide	0.005	0.008	100.0
lethane	0.08	0.004	0.004

N.D. = Not Detected

Table VII

GAS-OFF PRODUCTS FROM ACLAR, TYPE 33-C

DAC Serial No. 006

	We. (mr/10	lght of Component zms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Chlorinated Fluorocarbons	2.1	0.08	1.0
Acetone	N.D.	N.D.	0.004
Ethanol	N.D.	N.D.	0.09
Toluene	N.D.	N.D.	0.002
Carbon Monoxide	10.0	0.009	0,009
Methane	0.08	0.04	0.05

N.D. = Not Detected

Table VIII

GAS-OFF PRODUCTS FROM POLYOLEFIN/POLYAMIDE, SHONKA A-150

DAC Serial No. 007

	Wei (mg/l0 f	lght of Component zms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
ts Unsaturated Hydrocarbon	0.1	N.D.	0.04
cetone	0.08	N.D.	0.05
lethyl Ethyl Ketone	N.D.	N.D.	0.04
loluene	N.D.	N.D.	100.0
lisopropyl Ketone	0.6	0.07	0.3
it-n-propyl Ketone	1.0	0.008	0.01
arbon Monoxide	0.03	0.008	100.01
lethane	0.06	0.04	0.006

N.D. = Not Detected

Table IX

GAS-OFF PRODUCTS FROM FOAM, HATHONE HA 7236

DAC Serial No. 014

	We:	ight of Component	
	(mg/l0 /	gms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Freon-11	4.5	2.7	3.1
Trichloroethylene	0.02	N.D.	N.D.
Xylene	10.0	N.D.	N.D.
Carbon Monoxide	10.0	0.004	100.0
Methane	<0.01	0.04	0.006

N.D. = Not Detected

Table X

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GAS-OFF PRODUCTS FROM BUTYL RUBBER, 00996-33L

DAC Serial No. 017

	We: (mg/l0	ight of Component gms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Isobutylene } Diisobutylene }	0.3	0.08	0.4
Triisobutylene	0.03	10.0	0.3
Tetraisobutylene	0.03	N.D.	N.D.
Toluene	N.D.	N.D.	0.04
Xylene	N.D.	0.002	100.0
Carbon Monoxide	0.05	0.02	0.001
Methane	0.07	0.04	0.006

N.D. - Not Detected

Table XI

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GAS-OFF PRODUCTS FROM POLYURETHANE, 00996-39B

DAC Serial No. 018

	We	ight of Componen	lt
	(mg/10)	gms Candidate Ma	iterial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Ethanol	N.D.	0.08	0.03
Butanol	N.D.	N.D.	10.01
C ₁ -C ₃ Alkylbenzene	0.06	0.08	0.01
Acetophenone	0.2	0.2	10.01
2-Phenyl-2-propanol	0.15	0.1	0.05
Carbon Monoxide	0.05	0.04	0.04
Methane	0.2	0.07	0.07

N.D. = Not Detected.

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Table XII

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GAS-OFF PRODUCTS FROM SILICONE, 19513-10F

DAC Serial No. 019

	We: (mg/10)	ight of Component gms Candidate mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	N.D.	0.02	10.0
Ethanol	0.04	0.1	0.06
Toluene	N.D.	0.06	0.004
Butanol	N.D.	N.D.	0.01
Acetophenone	0.04	N.D.	N.D.
2-Phenyl-2-propanol	0.03	N.D.	N.D.
Carbon Monoxide	0.01	0.02	0.008
Methane	0.08	0.05	0.04

N.D. = Not Detected

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Table XIII

GAS-OFF PRODUCTS FROM ABS POLYMER, BOLTARON

DAC Serial No. 020

	We: (mg/10 p	lght of Component zms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Ethanol	N.D.	N.D.	10.0
n-Propanol	N.D.	N.D.	10.0
Trichloroethylene	0.02	N.D.	0.02
Toluene	0.008	N.D.	0.03
Xylene	0.02	N.D.	0.02
Styrene	0.2	0.04	0.11
Methylstyrene	0.02	N.D.	10.01
Carbon Monoxide	0.006	0.004	100.0
Methane	0.05	0.04	0.006

N.D. = None Detected

Table XIV

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GAS-OFF PRODUCTS FROM EPOXY, 760 A

DAC Serial No. 021

	We (mc/10	ight of Component gms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
2-Butanone	1.8	0.5	0.09
Carbon Monoxide	0.03	0.1	0.008
Methane	0.08	0.02	0.04

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Table XV

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GAS-OFF PRODUCTS FROM SILICONE, TYPE A #428/132

AF Serial No. 023

	We:	ight of Component	(["
Component	$\frac{72 \text{ Hours}}{(68^{\circ}\text{C})}$	(25°C)	60 Days (25°C)
Acetone	0.02	N.D.	10.01
Ethanol	10.0	N.D.	0.002
Benzene	0.02	N.D.	N.D.
2-Methyl-4-pentanone	0.21	0.03	0.03
Toluene	0.17	0.09	0.09
n-Butanol	0.17	0.02	0.02
Kylenes	0.22	0.09	0.09
Carbon Monoxide	10.0	0.006	0.004
Methane	0.04	0.05	0.05

N.D. = Not Detected

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Table XVI

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GAS-OFF PRODUCTS FROM EPOXY, BONDMASTER E611

AF Serial No. 053

	We	ight of Component	
	(mg/10	gms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days
Ethylene	2.8	0.04	0.08
Acetone	0.02	N.D.	N.D.
Ethanol	10.0	N.D.	N.D.
C+-Ce Hydrocarbons	01.0	N.D.	N.D.
Carbon Monoxide	0.05	0.003	0.006
Methane	0.08	0.04	0.05

N.D. = Not Detected

Table XVII

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GAS-OFF PRODUCTS FROM EPOXY/POLYAMIDE, V-9 SILVER EPON 815

AF Serial No. 054

	We: (me/10	ight of Component rms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.009	N.D.	N.D.
Sthancl	0.009	N.D.	N.D.
2-Butanol	0.1	N.D.	100.01
loluene	0.002	N.D.	N.D.
n-Butanol	0.1	N.D.	N.D.
Kylenes	0.005	N.D.	N.D.
Carbon Monoxide	0.06	0.005	0.006
Methane	0.05	40.0	0.05

N.D. = Not Detected

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Table XVIII

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GAS-OFF PRODUCTS FROM BLACK SILICONE, EMS 323

AF Serial No. 063

	We: (mc/10	ight of Component ems Candidate Mat	ertal
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
C, Unsaturated Hydrocarbons	0.07	N.D.	N.D.
Acetone	0.05	N.D.	N.D.
Ethanol	0.01	N.D.	N.D.
Benzene	0.007	N.D.	N.D.
Silicone Oil	0.03	N.D.	N.D.
Toluene	10.0	N.D.	N.D.
Xylenes	10.0	N.D.	N.D.
Carbon Monoxide	10.0	0.005	0.006
Methane	0.05	0.05	0.04

N.D. = Not Detected

89

Table XIX

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GAS-OFF PRODUCTS FROM SILICONE, 342 RUBBER

AF Serial No. 065

	We	ight of Component	
	(mg/10	gms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Methanol	0.03	10°0	0.02
Silicone Oil	0.30	0.10	0.12
Toluene	0.007	0,005	10.0
Xylene	0.021	10.0	0.02
Carbon Monoxide	0.006	0.001	0.00
Methane	0.09	0.1	0.05

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Table XX

GAS-OFF PRODUCTS FROM SILICONE, EMS 345 RUBBER

AF Serial No. 066

	We: (mg/10	ight of Component gms Candidate Mat	; cerial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.007	N.D.	N.D.
Methanol	0.15	0.008	0.02
Silicone Oil	1.5	0.06	0.12
t-Butanol	0.050	0.003	0.007
Toluene	0.007	<0°01	0.001
Carbon Monoxide	0.007	0.007	0.003
Methane	0.1	0.07	0.05

N.D. = Not Detected

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Table XXI

GAS-OFF PRODUCTS FROM RUBBER, EMS 355

AF Serial No. 068

	We: (me/l)	ight of Component rms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.009	0.002	0.005
Toluene	0.013	0.003	0.005
Xylene	0.021	0.002	0.005
Carbon Monoxide	0.007	0.003	0.003
Methane	0.04	0.04	0.04

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Table XXII

GAS- 'FF PRODUCTS FROM EMS 366 URETHANE

AF Serial No. 071

	Wei (mg/l0 gi	ght of Component ns Candidate Mate	rtal)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.009	N.D.	100.0>
Methanol	0.018	N.D.	<0.001
2-Propanol	0.010	N.D.	<0.001
Carbon Monoxide	0.02	0.002	0.004
Methane	0.09	0.04	0.04

N.D. = Not Detected

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Table XXIII

GAS-OFF PRODUCTS FROM SILICONE, DACRON

AF Serial No. 073

	Wej (mg/l0 p	lght of Component zms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.03	N.D.	N.D.
Silicone Oil	0.03	N.D.	N.D.
Sthanol	0.06	0.01	0.04
Toluene	0.009	100.0	0.003
Carbon Monoxide	0.01	0.008	0.02
Methane	0.1	0.2	0.2

N.D. = Not Detected

Table XXIV

GAS-OFF PRODUCTS FROM EPOXY, PRIMER M-602

AF Serial No. 203

	Wei (mg/l0 gr	ght of Component ns Candidate Mate	rial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
lcetone	5.0	0.02	0.03
U, −C. Hydrocarbons	0.4	N.D.	N.D.
:-Butanone	81.0	2.0	5.0
I-Propanol	0.9	0.04	0.1
:-Methyl-l-propanol	0.7	0.1	0.2
loluene	1.0	0.4	0.7
I-Butanol	92.0	6.1	10.0
(y lenes	120.0	0.3	0.7
larbon Monoxide	0.03	10.0	0.02
lethane	0.06	0.02	0.03

N.D. = Not Detected

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Table XXV

GAS-OFF PRODUCTS FROM TUBING, THERMOFIT RNF, CRN

AF Serial No. 209

	Wei (mg/l0 gr	ght of Component ns Candidate Mate	rial
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
lcetaldehyde	0.009	N.D.	N.D.
lcetone	0.007	N.D.	N.D.
čε Hydrocarbon(s)	0.002	N.D.	N.D.
thanol	0.014	N.D.	0.005
loluene	0.002	N.D.	N.D.
l-Butanol	0.006	N.D.	N.D.
Jarbon Monoxide	0.007	0.003	0.002
le thane	0.04	0.04	0.04

N.D. = Not Detected

96

Table XXVI

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GAS-OFF PRODUCTS FROM POLYESTER, MYLAR FILM

AF Serial No. 251

	Weil	ght of Component	
	(mg/10 gr 72 Hours	ms Candidate Mate 30 Dava	rfal) 60 Davs
Component	(68°C)	(25°C)	(25°C)
Chloroform	0.10	10°0	0.02
Acetone	0.12	N.D.	0.004
Benzene	0.004	N.D.	<0.001
Toluene	0.001	N.D.	<0.001
Carbon Monoxide	0.002	0.002	0.002
Methane	0.04	0.04	0.04

N.D. = Not Detected

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Table XXVII

GAS-OFF PRODUCTS FROM TUBING, THERMOFIT RNF

AF Serial No. 266

	Wei (mg/l0 g)	ght of Component ms Candidate Mate	rial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.005	N.D.	100°0>
Ethanol	0.005	N.D.	N.D.
Benzene	0.002	N.D.	N.D.
Toluene	0.009	N.D.	N.D.
Xy lene	0.003	N.D.	N.D.
Carbon Monoxide	0.003	0.002	0.002
Methane	0.04	0.04	0.04

N.D. = Not Detected

98

Table XXVIII

GAS-OFF PRODUCTS FROM ADHESIVE, ECCOBOND 70C

AF Serial No. 302

	Wei (mg/l0 p	ght of Component ms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Formaldelyde	0.004	N.D.	N.D.
Acetaldehyde	0.008	N.D.	N.D.
Methyl Formate	0.004	N.D.	N.D.
Ethanol	0.04	<0.01	0.001
Benzene	0.002	N.D.	N.D.
Formic Acid	0.002	N.D.	N.D.
Toluene	0.002	N.D.	N.D.
Carbon Monoxide	0.1	0.005	0.005
Methane	0.06	0°04	0.04

N.D. = Not Detected

99

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Table XXIX

GAS-OFF PRODUCTS FROM STYCAST, 1090 CAT-1

AF Serial No. 402

	Wet	ght of Componen	t,
	(mg/10 g	ms Candidate Ma	terial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone			
	0.00		TTN.0
Benzene	0.004	N.D.	<0.001
Carbon Monoxide	0.007	0.002	0.003
Methane	0.04	0.04	0.04

N.D. = Not Detected

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Table XXX

GAS-OFF PRODUCTS FROM STYCAST, 2651-50 CAF1

AF Serial No. 403

		ght of Component	
		ms candidate Mate	rial)
Component	(68°C)	50 Jays (25°C)	60 Days (25°C)
Acetone	0.012	N . D .	100 02
Benzene	0.005		
Carbon Monoxide	0.01	100.0	N.U.
Me thane	0.04	0.05	0.04

N.D. = Not Detected

Table XXXI

GAS-OFF PRODUCTS FROM SILICONE, RTV 521

AF Serial No. 406

	We: (mg/10_	ight of Component gms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Methanol	10.0	N.D.	N.D.
Acetone	0.04	0.01	10.01
sthanol [sopropanol]	0.18	0.02	0.03
Silicone Oil	1.4	0.81	0.77
Carbon Monoxide	0.002	0.003	N.D.
lethane	0.04	0.05	0.03

N.D. = Not Detected

102

Table XXXII

GAS-OFF PRODUCTS FROM ADHESIVE, ADIPRENE L-100 MCC

AF Serial No. 421

	We: (mg/10	ight of Component zms Candidate Mat	; erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Methanol	0.06	0.018	0.022
Ethanol	0.018	0.009	0.012
Benzene	0.002	N.D.	N.D.
Toluene	100.0	<0.01	<0.001
Xylene	0.003	N.D.	100.01
Carbon Monoxide	0.002	100.0	100.01
Methane	0.04	0.04	0.03

N.D. # Not Detected

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Table XXXIII

GAS-OFF PRODUCTS FROM SILICONE, RTV 502 RUBBER

AF Serial No. 451

	We (mg/l0 p	ight of Component gms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.04	N.D.	N.D.
Silicone Oil	2.1	0.05	0.15
n-Propanol	3.9	1.2	5.6
n-Butanol	0.03	N.D.	N.D.
Ethylbenzene	0.03	N.D.	N.D.
Xylenes	0.03	N.D.	N.D.
Carbon Monoxide	0.02	0.002	0.004
Methane	0.05	0.05	0.05

N.D. = Not Detected

Table XXXIV

GAS-OFF PRODUCTS FROM SILICONE FOAM, Q30079

AF Serial No. 454

	We1 (mg/l0 p	Ight of Component tms Candidate Mat	ertal)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.02	100.0	100.0
sthanol	0.009	N.D.	N.D.
Silicone Oil	0.15	0.009	0.02
ropanol	0.006	N.D.	N.D.
loluene	0.035	0.009	τα•ο
Sutanol	0.02	0.002	0.004
Kylenes	0.09	0.008	0.010
Carbon Monoxide	0.02	0.002	0.002
lethane	0.05	0.05	0.05

N.D. = Not Detected

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Table XXXV

GAS-OFF PRODUCTS FROM SILICONE FOAM, RTV 503

AF Serial No. 459

	Wei (mg/l0 g	ght of Component ms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.02	N.D.	N.D.
Ethanol	5.0	2.8	4.9
Silicone Oil	2.2	N.D.	N.D.
Xylenes	0.04	N.D.	N.D.
Carbon Monoxide	0.02	0.001	0.002
Methane	0.05	0.04	0.05

N.D. = Not Detected

Table XXXVI

GAS-OFF PRODUCTS FROM TAPE, STYCAST 2651-CAT 9

AF Serial No. 495

	We	ight of Component	, , ,
Component	$\frac{(mg/10)}{(68°C)}$	gms canaldate Mat 30 Days (25°C)	erial) 60 Days (25°C)
Acetone	0.004	0.003	0.007
Ethanol	0.04	<0.01	10.01
Toluene	0.06	N.D.	100.0
n-Butanol	0.006	N.D.	N.D.
Xy lenes	0.003	N.D.	N.D.
Carbon Monoxide	0.02	0.004	0.003
Methane	0.05	0.05	0.05

N.D. = Not Detected

Table XXXVII

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GAS-OFF PRODUCTS FROM OIL, AERO SHELL #7

AF Serial No. 505

	Wei (mg/l0 p	ight of Component gms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.007	N.D.	N.D.
Ethanol	0.02	N.D.	N.D.
n-Propanol	0.02	N.D.	N.D.
2Butanol	10.0	N.D.	N.D.
Toluene	0.02	0.001	0.002
n-Butanol	0.02	N.D.	N.D.
Xylene	0.01	N.D.	N.D.
Naphthalene	0.02	N.D.	N.D.
Carbon Monoxide	c.008	0.005	0.008
Methane	0.05	0.05	0.07

N.D. = Not Detected

Table XXXVIII

GAS-OFF PRODUCTS FROM PAINT, LACQUER STIK WHITE

AF Serial No. 515

	Wei (mg/l0 gn	ght of Component ns Candidate Mat	t terial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Formaldehyde	2.3	4.D.	N.D.
Ct Unsaturated Hydrocarbon(s) _} Acetaldehvde	0.32	n.28	0.39
Cs Unsaturated Hydrocarbon(s)	0.77	0.20	0.21
rropionalgenyge Ethvlformate	0.72	0.27	L.5 0.32
Acetone	0.36	0.09	0.09
C ₆ Unsaturated Hydrocarbon(s)	0.81	0.09	0.08
Butyraldehyde 2-Butanona	0.36	0.22	0.22
C, Unsaturated Hydrocarbon(s)	2.0	0.43	0.36
Valeraldehyde	4.3	0.36	0.22
Pentanone	0.43	0.33	0.43
Ce Unsaturated Hydrocarbon(s)} 2-Buten-l-ol	0.79	0.09	11.0
Hexanone	0.02	N.D.	N.D.
Cyclopentanone	0.58	N.D.	N.D.
3-Methyl-l-butanol	0.26	N.D.	N.D.
Co Unsaturated Hydrocarbon(s)	0.03	N.D.	D.N.
cycruiteranoi 3-Methylcycloheranol	0.10	N.D.	N.D.
Acetic Acid	0.32	N.D.	N.D.
Carbon Monoxide	25.2	8.6	14.5

N.D. = Not Detected

Table XXXIX

GAS-OFF PRODUCTS FROM PAINT, CONDUCTIVE

AF Serial No. 522

	Wei (mg/l0 e	ght of Component ms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
C ₃ -C ₄ Hydrocarbons	0.03	N.D.	N.D.
Acetone	2.2	0.4	1.1
Ethanol	3.2	0.9	7.5
Propanol	0.08	0.02	0.7
Propylacetate	1.3	0.3	1.2
Toluene	0.3	0.07	0.2
2-Butanol	0.9	0.7	2.7
l-Butanol	5.2	1.2	3.8
Xylene	0.04	N.D.	N.D.
2-Ethoxyethylacetate	0.09	0.02	0.09
2-n-Propoxyethanol	0.3	0.04	1.0
2-n-Butoxyethanol	2.8	0.2	1.2
2-n-Butoxyethylacetate	0.04	N.D.	N.D.
Carbon Monoxiúe	1.1	0.2	0.03
Methane	0.03	0.06	0.07

N.D. = Not Detected

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Table Xü

GAS-OFF PRODUCTS FROM RESIN, EMERLON 310

AF Serial No. 527

	Weig (mg/l0 gn	ght of Component ns Candidate Mate	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
A1r			
Acetone	0.4	0.04	0.1
Ethanol	21	1.8	6.9
Propylacetate	0.8	0.1	0.6
Toluene	0.6	0.08	0.3
2-Butanol	2.3	0.3	1.2
l-Butanol	26	2.5	8.9
Butylacetate	7.2	0.8	3.3
Xylene	0.7	0.06	0.4
2-Ethoxyethanol	1.6	0.08	0.3
2-Ethoxyethy lacetate	9.2	1.6	4.1
Carbon Monoxide	0.02	10.01	0.005
Methane	0.05	0.05	0.07
Phenol	3.3	N.D.	N.D.

N.D. = Not Detected

Table XLI

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GAS-OFF PRODUCTS FROM VARNISH, E44

AF Serial No. 528

	We: (mg/10 p	ight of Component zms Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.21	0.02	0.01
Ethanol	2.2	0.40	0.30
2-Butanone	0.20	0.01	0.007
Benzene	0.05	0.003	0.002
Methyllsobutylketone	717	18.4	18.0
Toluene	25.2	3.4	3.5
Butanol	8.9	0.02	0.02
Xylenes	5.7	0.36	0.38
C ₃ -C ₄ Alkylbenzenes	0.58	N.D.	N.D.
Carbon Monoxide	0.05	0.007	0.003
Methane	0.05	0.05	0.06

N.D. = Not Detected

Table XLII

GAS-OFF PRODUCTS FROM COATING, POLYURETHANE PC-18

AF Serial No. 533

	Wei	lght of Component	
	(mg/10 f	gms Candidate Mat	crial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
lcetone	0 . 61	0.005	0.007
thanol	0.08	N.D.	0.005
lenzene	0.02	N.D.	100.0
-Butanol	0.72	N.D.	N.D.
oluene	0.22	0.004	0.008
I-Butanol	0.84	N.D.	N.D.
ylenes	11.8	1.4	2.7
!-Ethoxyethy lacetate	6.9	0.32	0.68
3-Alkylbenzenes	0.4	N.D.	N.D.
arbon Monoxide	0.008	0.005	100.0
lethane	0.04	0.06	0.08

N.D. = Not Detected

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Table XLIII

GAS-OFF PRODUCTS FROM CORD, STYLE 18 NYLON

AF Serial No. 551

	We:	ight of Component	
Component	72 Hours (68°C)	gms canatuate mat 30 Days (25°C)	erial) 60 Days (25°C)
Acetone	0.5	0.005	0.016
Toluene	0.009	N.D.	0.003
n-Butanol	0.03	N.D.	600.0
1,3-Dioxane	0.03	N.D.	N.D.
n-Methylmorpholine	0.03	N.D.	N.D.
Carbon Monoxide	0.03	0.006	0.002
Methane	0.06	0.05	0.06

N.D. = Not Detected

Table XLIV

GAS-OFF PRODUCTS FROM CORD, STYLE 21 NYLON

AF Serial No. 552

	Weig (mg/l0 gn	cht of Component is Candidate Mat	erial)
Component	72 Hours (68°C)	30 Days (25°C)	60 Days (25°C)
Acetone	0.02	<0°01	100.0>
Ethanol	0.009	<0.001	100°0>
sec-Butanol	10.0	<0.001	0.002
Toluene	0.04	0.002	0.004
n-Butanol	11.0	0.003	0.006
Xylene	0.01	N.D.	N.D.
Carbon Monoxide	10.0	0.007	0.002
Metháne	0.05	0.05	0.06

N.D. = Not Detected

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APPENDIX III

REPRESENTATIVE GAS CHROMATOGRAMS

FOR

GAS-OFF EXPERIMENTS

The gas chromatograms shown in this appendix were obtained on an F&M Scientific Corporation Model 810 Research Gas Chromatograph. Instrument conditions and column specifications are listed in Table XLV. Since retention times tended to shift somewhat due to column aging, a standard mixture was used as a day-to-day reference.

The gas chromatograms are representative of a particular candidate material. Comparison of peak intensities in chromatograms for different candidate materials should be made with care, since sensitivity factors and quantities of atmosphere taken for analysis vary.

Chromatograms appear in order of their Air Force serial numbers. Names of materials are those submitted by the Air Force.

Table XLV

GAS CHROMATOGRAPHIC INSTRUMENT CONDITIONS

All samples were analyzed using a flame ionization detector and a F&M Model 810 Research Gas Chromatograph.

Instrument Conditions

12-ft x 1/8-in O.D. Stainless Steel, 7% neopentyl-glycolsuccinate on 60/80 mesh Gas-Pack F + 20-ft x I. Column: 1/4-in. O.D., 5% Carbowax 20M on 60/80 mesh Gas-Pack F. Column Temperature: programmed 50°-185°C @ 8°C/min. Detector Temperature: 275°C Injection Port Temperature: 250°C Flow Split: 1:9 Flow Rate: 60 ml/min. Range: 10 Attenuation: X8, or as noted Sample Size: 25 cc of gas II. Column: 20-ft x 1/4-in O.D. Stainless Steel, 20% Triton X-305 on 60/80 mesh Gas Chrom Z. Column Temperature: programmed 50°-170°C @ 8°C/min. Detector Temperature: 300°C Injection Port Temperature: 250°C Flow Split: none Flow Rate: 60 ml/min. Range: 10 Attenuation: X8, or as noted Sample Size: 50 cc of gas



Figure 57. Gas Chromatogram of Gas-Off Products from Nitrile/Phenolic, FM 238 (DAC 002) (72 hours @ 68°C).





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Figure 60. Gas Chromatogram of Gas-Off Products from Foam, Hathone HA 7236 (DAC 014) (72 hours @ 68°C).

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Figure 61. Gas Chromatogram of Gas-Off Products from Butyl Rubber, 00996-33L (DAC 017) (50 days @ 25°C).



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Figure 62. Gas Chromategram of Gas-Off Products from Polyurethane, 00996-39B (DAC 018) (30 days @ 25°C).



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Figure 63. Gas Chromatogram of Gas-Off Products from Silicone, 19513-10f (DAC C19) (30 days @ 25°C).

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Figure 64. Gas Chromatogram of Gas-Off Products from ABS Polymer, Boltaron (DAC 020) (72 hours @ 68°C).

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Figure 66. Gas Chromatogram of Gas-Off Products from Silicone, Type A #428/132 (AF 023) (72 hours @ 68°C).


Figure 67. Gas Chromatogram of Gas-Off Products from Epoxy, Bondmaster E611 (AF 053) (72 hours @ 68°C).

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Figure 68. Gas Chromatogram of Gas-Off Products from Epoxy/Polyamide, V-9 Silver Epon 815 (AF 054) (72 hours @ 68°C).

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Figure 69. Gas Chromatogram of Gas-Off Products from Black Silicone, EMS-323 (AF 063) (72 hours @ 68°C).



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Figure 71. Gas Chromatogram of Gas-Off Products from Silicone, EMS 345 Rubber (AF 066) (72 hours @ 68°C).



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Figure 78. Gas Chromatogram of Gas-Off Products from Tubing, Thermofit RNF (AF 266) (72 hours @ 68°C).



Figure 79. Gas Chromatogram of Gas-Off Products from Adhesive, Eccobond 70C (AF 302) (72 hours @ 68°C).







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Figure 81. Gas Chromatogram of Gas-Off Products from Stycast, 2651-50 CAF1 (AF 403) (72 hours @ 68°C).



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Figure 84. Gas Chromatogram of Gas-Off Products from Silicone RTV 502 Rubber (AF 451) (72 hours @ 68°C).



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Figure 85. Gas Chromatogram of Gas-Off Products from Silicone Foam, Q30079 (AF 454) (72 hours @ 68°C).



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Figure 86. Gas Chromatogram of Gas-Off Products from Silicone Foam, RTV 503 (AF 459) (72 hours @ 68°C).



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Figure 89. Gas Chromatogram of Gas-Off Products from Paint, Lacquer Stik White (AF 515) (72 hours @ 68°C).



Figure 90. Gas Chromatogram of Gas-Off Products from Paint, Conductive (AF 522) (72 hours @ 68°C).



Figure 91. Gas Chromatogram of Gas-Off Products from Resin, Emerlon 310 (AF 527) (72 hours @ 68°C).



Figure 92. Gas Chromatogram of Gas-Off Products from Varnish, E44 (AF 528) (72 hours @ 68°C).

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Figure 93. Gas Chromatogram of Gas-Off Products from Coating, Polyurethane PC-18 (AF 533) (72 hours @ 68°C).



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Figure 94. Gas Chromatogram of Gas-Off Products from Cord, Style 18 Nylon (AF 551) (72 hours @ 68°C).





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APPENDIX IV

ANALYSES OF BIO-ENVIRONMENTAL ATMOSPHERES AND SPECIAL ANALYSES

Table XLVI

SPECIAL ANALYSIS NO. 1

GAS-OFF FRODUCTS FROM HETRON POLYESTER RESIN

Components	Weight of Components After 30 Days at 25°C (mg/10 gm Candidate Material)		
Styrene	0.1		
Methylethylketone (or methylethylketone peroxide)*	0.4		
Divinylbenzene	0.02		
Xylene	0.005		
Acetone	0.01		
Ethanol	0.1		
Methane	0.02		
Carbon Monoxide	0.1		

*Due to lack of data on the stability of MEK peroxide, it has not been established whether the peroxide decomposes during storage or during analysis.

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Table XLVII

SPECIAL ANALYSES NOS. 2 AND 3

BIO-ENVIRONMENTAL SAMPLES

	No.2 Ex Fro Test Ch	haust m amber	No.3 Room Air Outside Test Chamber	
Carbon Dioxide	500	ppm	500 ppm	
C ₁ -C ₂ Chlorinated Fluorocarbons*	20	ppm	15 ppm	
C1-C; Alkylbenzenes	0.1	. ppm	(not detected)	
Carbon Monoxide	3	ppm	2 ppm	
Methane	1-2	ppm	1-2 ppm	

*Similar to gas-off products observed from partially chlorinated fluorocarbon lubricants.

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Table XLVIII

SPECIAL ANALYSES NOS. 4, 5, 6 AND 7

ANALYSIS OF FOUR BIO-ENVIRONMENTAL SPECIMENS

			Component	Level (mg/liter)
Cylinder	No.	1	Acetone Ethanol Benzene Trichloroethylene Toluene C ₁ -C ₂ Chlorofluorocarbons Methane Carbon Monoxide	0.02 <u>5</u> 0.03 0.0002 0.002 0.006 5.6 0.0008 0.0005
Cylinder	No.	2	Benzene Toluene Methane Carbon Monoxide	0.001 0.0007 0.004 0.0002
Cylinder	No.	3	Acetone Trichloroethylene Toluene Methane Carbon Monoxide	0.01 0.002 0.002 0.006 0.001
Cylinder	No.	4	Acetone Ethanol Benzene Toluene Methane Carbon Monoxide	0.001 0.0008 0.0005 0.002 0.002 0.003

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