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POLYMERS FOR SPACECRAFT HARDWARE
MATERIALS SPECIFICATIONS AND ENGINEERING INFORMATION

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SCOPE

This report covers the work performed during the period December 10, 1965 to January 9, 1966 on "Polymers for Spacecraft Hardware--Materials Specifications and Engineering Information," SRI Project ASD-5046 under JPL Contract No. 950745.

The primary objectives of this program are to assist the Jet Propulsion Laboratory of the California Institute of Technology in the development and preparation of polymeric material specifications to be used in connection with JPL spacecraft, and to provide a study of the effects of simulated space environment on selected commercial polymeric products. The materials and products to be studied and the extent of work to be performed are specified by the JPL Cognizant Engineers.

The program is being conducted as two interrelated phases, running concurrently: The purpose of Phase I, Polymeric Materials Specifications, is to obtain quantitative values for parameters which may be used to assure the performance of a given batch of material in a spacecraft environment. The purpose of Phase II, Engineering Information, is to establish material limitations and to obtain detailed design information.

Since a variety of activities and materials are involved in the overall program, an outline of the work performed and contemplated is given at the beginning of the reports for each Phase.

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Thermal-Vacuum Weight Loss

Chemical Tests

Physical Tests

WORK PERFORMED

Thermal-Vacuum Weight-Loss Determinations

Silicone Elastomers

The vacuum weight-loss determinations made with a standardized procedure are of timely interest since they provide corroborative data for a series of silicone elastomers which have shown relatively superior performance in mechanical property measurements made during exposure in an equivalent thermal-vacuum environment (engineering information, Phase II); the elastomers are General Electric Company SE-3604, -3613, and -3713. (Samples SE-3613 and -3713 had been specially post-cured 24 hours at 250°C.) The results of these determinations are given in Tables I, II, and III. Comparison of these data with those obtained previously for a number of polymeric materials indicates that the silicone elastomers are candidates for space-grade materials, and the following suggestions can be made for weight-loss requirements (125°C and 10^{-6} torr):

SE-3613	0.10% ± 0.05 at 48 hr
SE-3604	0.17% ± 0.05 at 96 hr
SE-3713	0.19% ± 0.05 at 48 hr

Silicone Foamed Rubber

A foamed RTV-type silicone rubber, suggested for use as an ablation material has also been checked for vacuum weight-loss. A block of foamed RTV-560 (General Electric) was provided by the JPL Cognizant Engineer, and a specimen was cut from the center of the block in order to ensure freedom from contamination by mold release or machine tools used in its fabrication; weight-loss data are given in Table IV. As indicated, the weight-loss of 3.2% is in the range of a previously tested RTV-560 slab sample which had received only a normal cure (3.9% weight-loss).

Table I

WEIGHT-LOSS DATA FOR SILICONE ELASTOMER, SE-3613*
(125°C and 10⁻⁶ torr)

Time, Hrs.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
48	3.4211	0.0030	0.09
	3.6081	0.0031	0.09
96	3.5784	0.0043	0.12
	3.3832	0.0035	0.10
192	3.5090	0.0036	0.10
	3.4899	0.0036	0.10

* General Electric Company; post-cured at 250° for 24 hours.

Table II

WEIGHT-LOSS DATA FOR SILICONE ELASTOMER, SE-3604*
(125°C and 10⁻⁶ torr)

Time, Hrs.	S. Wt., G	Wt. Loss, g	Wt. Loss, %
48	3.0715	0.0026	0.08
	3.0900	0.0026	0.08
96	3.1672	0.0055	0.17
	3.0223	0.0050	0.17
192	3.2068	0.0048	0.15
	3.0442	0.0047	0.15

* General Electric Company

Table III

WEIGHT-LOSS DATA FOR SILICONE ELASTOMER, SE-3713*
(125°C and 10⁻⁶ torr)

Time, Hrs.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
48	3.2086	0.0060	0.19
	3.1086	0.0057	0.18
96	3.1521	0.0064	0.20
	3.3769	0.0065	0.19
192	3.1996	0.0063	0.20
	3.3355	0.0058	0.17

* General Electric Company, post-cured at 250°C for 24 hours.

Table IV

WEIGHT-LOSS DATA FOR FOAMED SILICONE RUBBER RTV-560*
(125°C and 10⁻⁶ torr)

Time, Hrs.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
48	3.2640	0.0794	2.43
	2.8331	0.0768	2.71
96	2.9429	0.0795	2.70
	3.5567	0.0898	2.52
192	3.1795	0.1085	3.41
	3.1835	0.0948	2.98

* General Electric Company

Acrylic-Glass Fiber Sleevings

Weight-loss determinations were made for two acrylic-resin treated glass-fiber sleeving materials used for wire and cable insulation, Ben Har Acryl C-2 and Ben Har Acryl A (Grade BAI) supplied by the Bentley-Harris Manufacturing Company. The results of these determinations are summarized in Tables V and VI, and suggestion can be made for a weight loss requirement for Ben Har Acryl C-2 as $0.30\% \pm 0.05$ at 48 hours. The Ben Har Acryl A (Grade BAI) is considered unsuitable at this time in view of the white, powdery coating (not identified) which began depositing on the walls of the sample cell within one-half hour of exposure to the thermal-vacuum environment.

Table V

WEIGHT-LOSS DATA FOR BEN HAR ACRYL A-GRADE BAI*
(125°C and 10^{-6} torr)

Time, Hrs.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
48	2.4724	0.0079	0.32
	2.4726	0.0076	0.31
96	2.4986	0.0106	0.42
	2.4884	0.0088	0.35
192	2.4858	0.0107	0.43
	2.4832	0.0104	0.42

* Bentley-Harris Mfg. Co.

Table VI

WEIGHT-LOSS DATA FOR BEN HAR ACRYL C-2^x
(125°C and 10⁻⁶ torr)

Time, Hrs.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
48	2.2897	0.0054	0.24
	2.2469	0.0058	0.26
96	2.1368	0.0053	0.25
	2.2681	0.0063	0.28
192	2.2320	0.0078	0.35
	2.2532	0.0073	0.33

* Bentley-Harris Mfg. Co.

Epoxy Varnish and Solder Paste

As suggested in Monthly Report No. 18, weight-loss has been greatly reduced by extended curing for Clear Varnish B-276 (Westinghouse Electric) and Eccobond solder 56/C9 (Emerson Cuming, Inc.). The curing time of 4 hours at room temperature plus one hour at 165°C for B-276 was altered to include 16 hours (overnight) at 165°C; the recommended cure of 2 hours at 50°C for 56/C9 was extended to 16 hours (overnight) at 50°C. Since the weight-loss of both materials previously had levelled off at about 48 hours (indicating, perhaps, early release of highly-volatile material), their weight-losses were determined only after 48 hours in the thermal-vacuum environment subsequent to extended curing. Comparative weight-loss data are given below:

	<u>Recommended Cure</u>	<u>Post-cure</u>
Clear Varnish B-276	2.51% (av.)	0.56%
Eccobond Solder 56/C9 (Catalyst No. 9)	0.73% (av.)	0.31%

Chemical Tests

Silicone Hydroxyl Content

Work is in progress on the evaluation of a suitable specifications test procedure for the determination of the hydroxyl content of RTV-type silicone base materials. Representative materials being examined are RTV-11, -60, -560, and -615, supplied by the General Electric Company.

Physical Tests

Silicone Infrared Spectra

Infrared absorbance curves have been prepared for films of RTV-type silicone base materials in order to provide typical spectra for specifications purposes. Curves for RTV-11, -60, -560, and -615 have been submitted to the JPL Cognizant Engineer; the films were prepared by evaporating chloroform extracts of the silicone base materials on optical salt flats.

Silicone Nonvolatile Material

The nonvolatile contents of the silicone bases were determined in accordance with ASTM Method D 1259-61(B), i.e., drying resin films on aluminum foil in a forced-draft oven at 105°C; however, it was found that better reproducibility of results was obtained if the samples were cooled in a desiccator, rather than simply cooling in air and weighing quickly as specified in the ASTM method. Results of these determinations are given in Table VII.

Table VII

NONVOLATILE CONTENTS OF REPRESENTATIVE
RTV-TYPE SILICONE BASE MATERIALS*

Sample	Nonvolatile Content, wt.-%
RTV-11	98.57-98.58
RTV-60	99.20-99.23
RTV-560	98.25-98.47
RTV-615	99.66-99.78

*General Electric Company

FUTURE WORK

Thermal-Vacuum Weight Loss

Vacuum weight-loss determinations via standardized procedure will be made on a continuing basis in order to establish limitations for specifications requirements for various polymeric materials. Included during the next working period will be two Lexans for corroboration of Phase II data, and a repeat of Eccobond 56C, using catalyst No. 11 which is recommended for optimum high-temperature properties.

Chemical Tests

Determinations of physical and chemical properties of polymers will be continued as required for incorporation into specifications requirements and test procedures. Selection of the procedure for hydroxyl content of RTV-type silicones will be completed, and work will commence on procedures and determinations for the RTV catalyst, T-12.

Physical Tests

It is anticipated that work will be completed on the evaluation of procedures for determination of viscosity of the RTV-type silicones. Work will be initiated on the preparation of ASTM slabs of these silicones for hardness, density, and tensile strength tests.

PHASE II - ENGINEERING INFORMATION

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 Volatile Condensable Material

 Mechanical Properties

 Physical Properties

WORK PERFORMED

Volatile Condensable Material (VCM)

In order to simplify nomenclature and reference, rapid-maximum-VCM will be called micro-VCM; distinction between VCM and micro-VCM may be made as follows:

	<u>VCM</u>	<u>Micro-VCM</u>
Sample size	up to 1-in. cube	milligrams
VCM value	wt per unit area, length, etc.	wt per wt of sample
Thermal-vacuum exposure	24, 48, 96, and 330 hr	24 hr
Purpose	establish curves for VCM deposition and re- moval with time	screening; estimate maximum VCM values

Micro-VCM

Component parts have been completed and assembled for the micro-VCM unit, and electrical and coolant connectors have been affixed. The unit has been mounted in place in order to determine appropriate values for power input and optimum locations of thermocouples and coolant lines.

Mechanical Properties

Stress Relaxation Tests

Approximately 824 hours of exposure to the thermal-vacuum environment of 125°C and 10^{-6} torr have been logged as of January 9, 1966, for the in situ mechanical property measurements of SE-3613, SE-3713, and Lexan. A preliminary comparison with previous data from tests of SE-3604 indicates that the behavior of SE-3613 is about equivalent to that of SE-3604, and that SE-3713 exhibits a more extensive increase of stress in

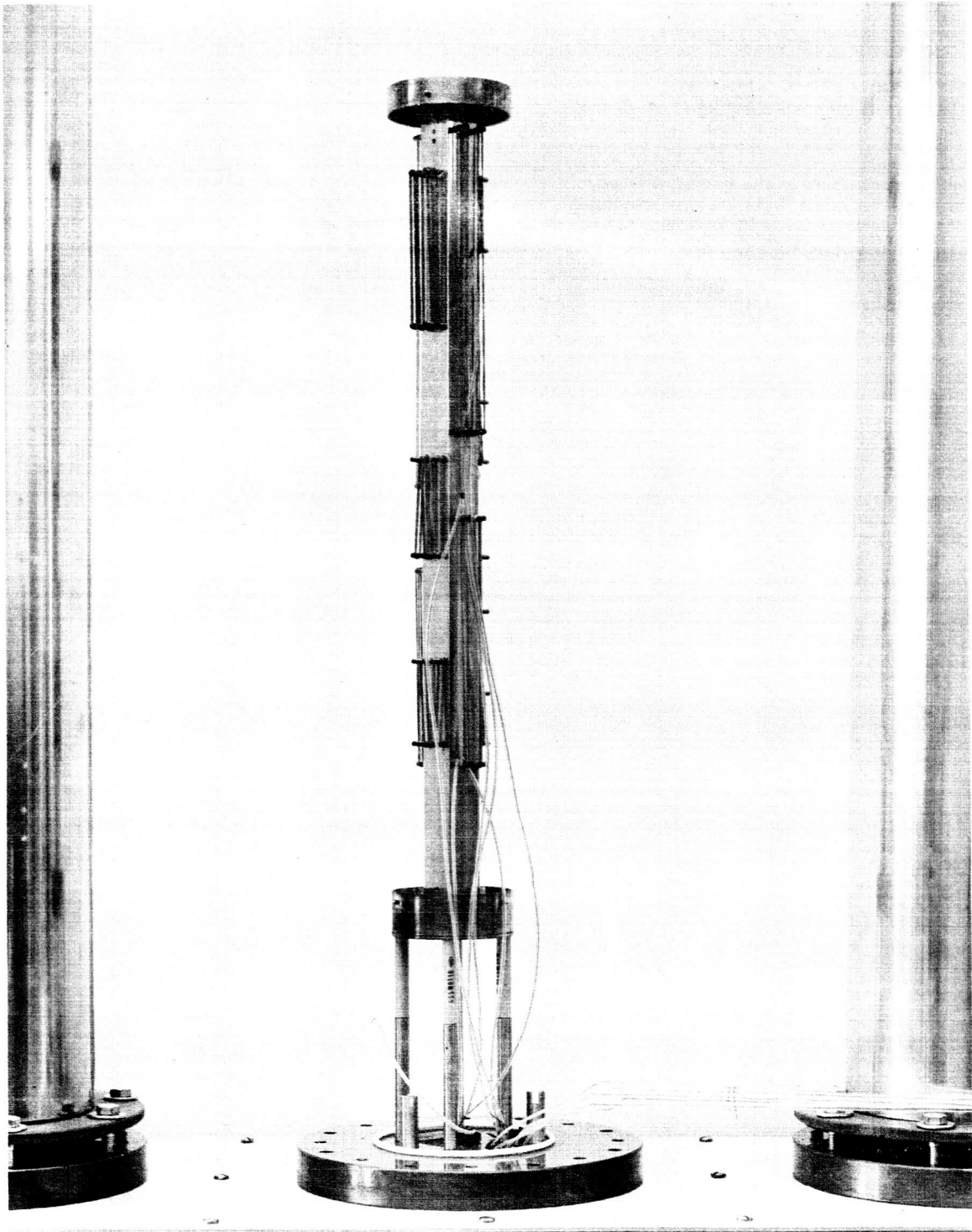
the intermittent tests. At this preliminary stage, neither composition appears to be superior to SE-3604, at least insofar as mechanical behavior is concerned. One of four Lexan specimens, which are being stored under a constant load of 600 psi, has broken; all specimens have elongated more than 10%, indicating that 600 psi is likely to be excessive in a structural application. It appears at the present time that Lexan will not be useful at temperatures of the order of 125°C.

Eight-Month Storage Tests

The assemblies constructed for observing polymeric materials under constant strain during an 8-month exposure to a thermal-vacuum environment (125°C and $< 10^{-2}$ torr) are detailed in Figures 1 to 4. Figure 1 shows the basic structure and the supports on which elastomeric materials (sample rings) can be placed at calculated strains.

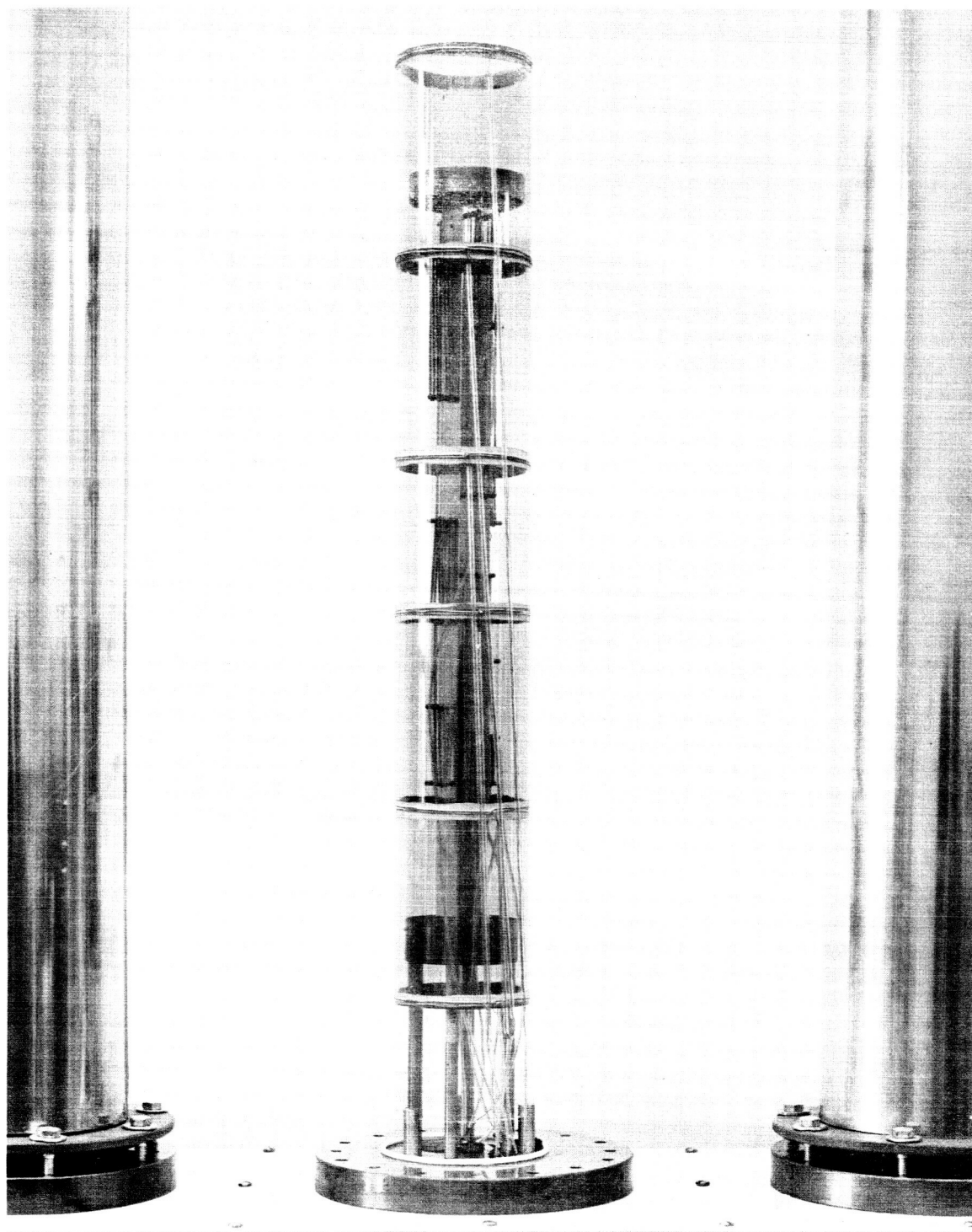
In Figure 2, the glass cylinder heater is shown around the basic structure. The glass has been coated at SRI with a bismuth oxide-gold-bismuth oxide laminate; at intervals, current-carrying copper wires have been affixed to conducting silver paint which is deposited under and over the laminate; after deposition of the laminate over initial bands of silver paint, the assembly was annealed at 400°C, and then the final bands of silver paint were applied. The gold layer is of the order of 90-100 Å in thickness. As shown in Figure 2, clear visibility is provided through the heater.

The bell-tube (vacuum chamber) for each unit is shown in place in Figure 3, and an assembly of four units is shown in Figure 4. Aluminum shields serve a dual purpose, i.e., maintenance of thermal equilibrium and protection from radiation (fluorescent fixtures). Each unit has a separate heating control, three-position thermocouple read-out, and vacuum system. The thermocouple beads are inserted in pieces of the same elastomer as being tested; thus, the observed temperatures at different locations within the unit is representative of the temperature of the materials under test.



TA-5046-40

FIG. 1 BASIC STRUCTURE FOR SUBJECTING SELECTED ELASTOMERS TO SPECIFIC INITIAL STRAINS



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FIG. 2 VIEW OF CONSTANT STRAIN APPARATUS WITHIN GLASS CYLINDER HEATER

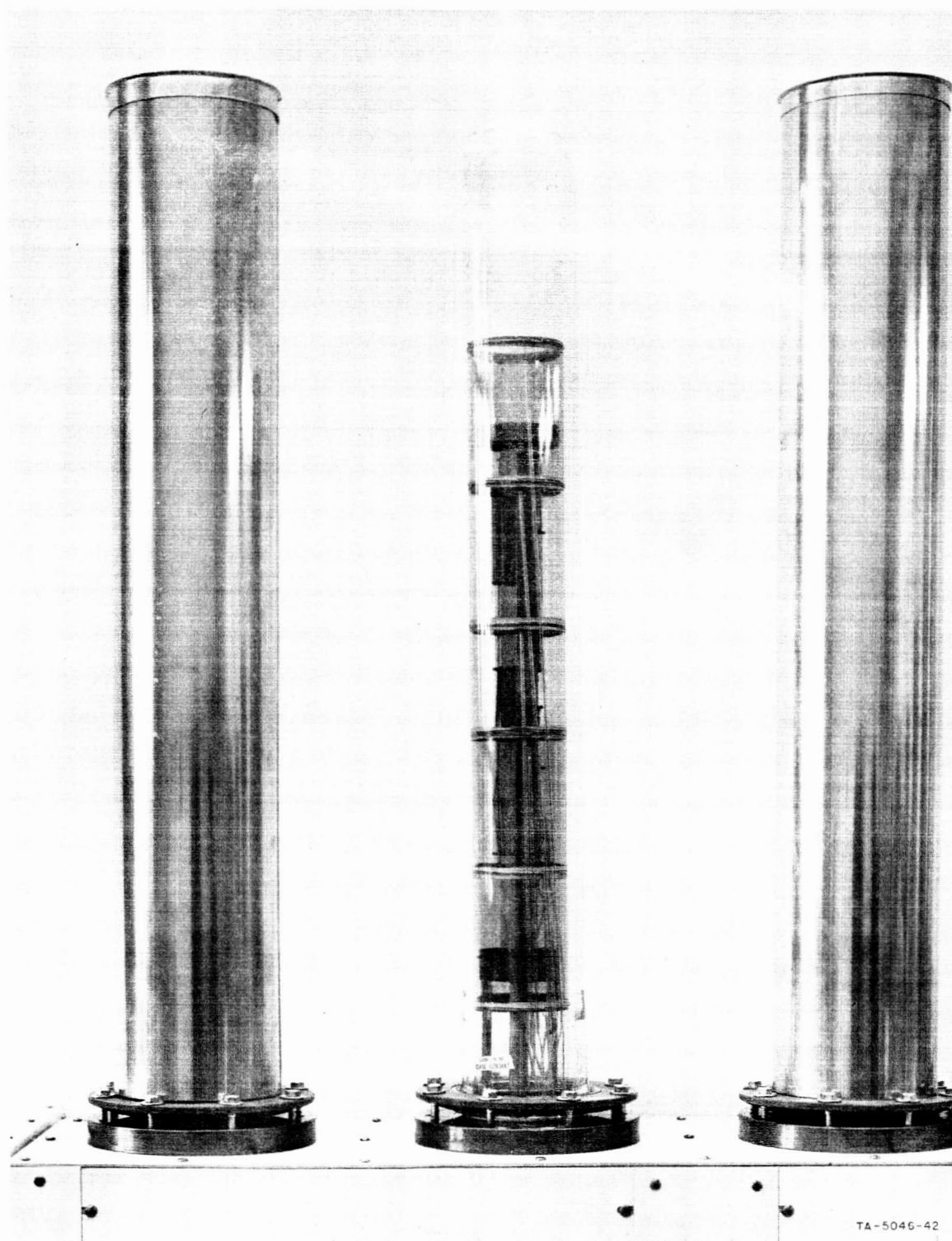


FIG. 3 VIEW OF CONSTANT STRAIN APPARATUS AND GLASS CYLINDER HEATER WITHIN BELL-TUBE (Vacuum Chamber)

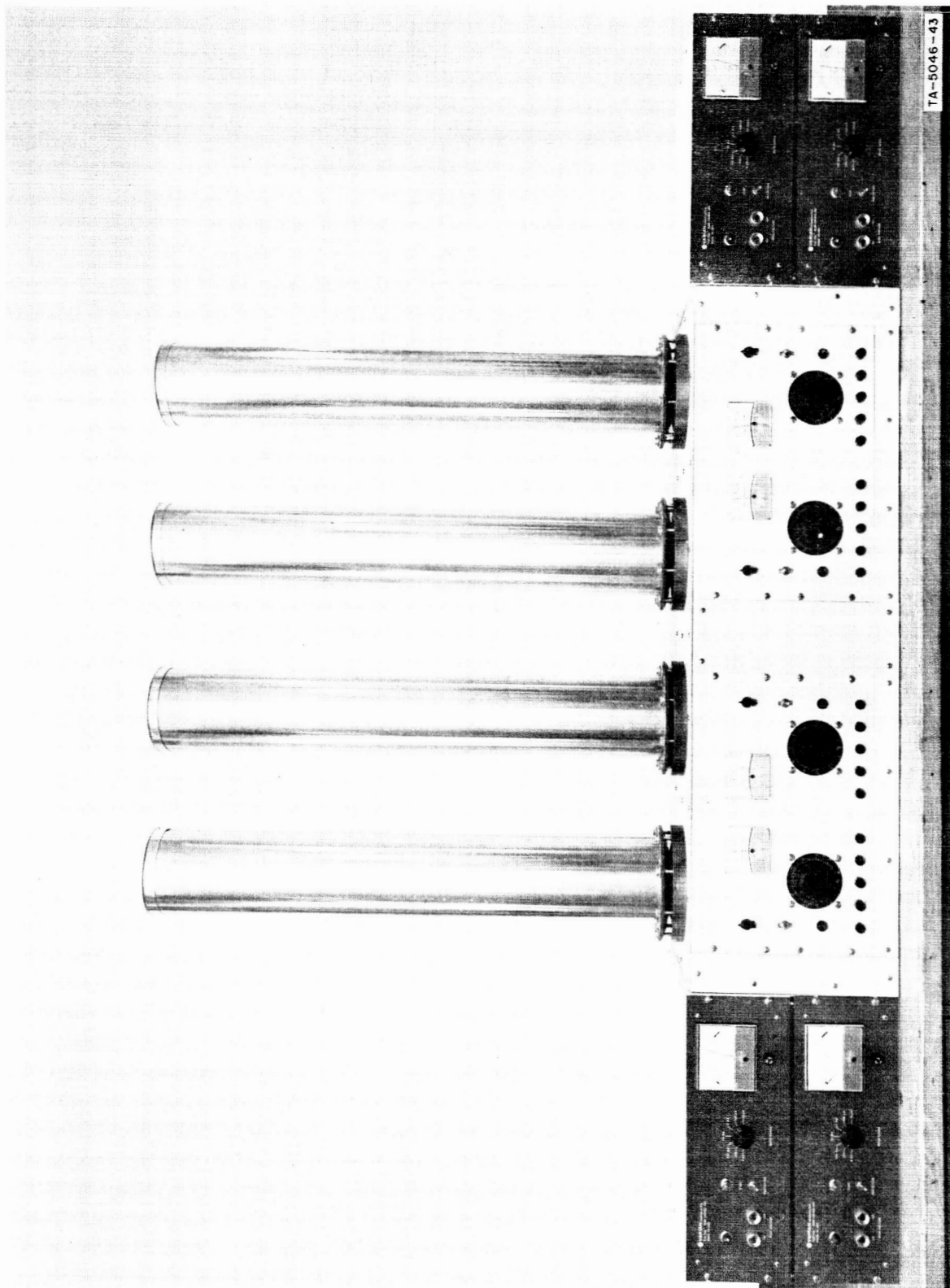


FIG. 4 PHOTOGRAPH OF TOTAL ASSEMBLY FOR 8-MONTH THERMAL-VACUUM TESTS OF SELECTED POLYMERIC MATERIALS

Three units were loaded with elastomers SE-3604, Viton A-4411A-990, and Hycar-520-67-108-1 (Hycar-1) at various strains. Rings of each material were stretched at room temperature between support pins to the largest strains which could be sustained. In most instances, four rings were placed at each level of strain, and at least four rings were stored unstrained as controls in each unit. Only one elastomer species was used for each unit in order to prevent cross-contamination by volatilized substances.

At start-up, each unit was evacuated to less than 10^{-3} torr with a mechanical pump, and then its ion pump (Varian, 15 liter/sec) was turned on. When a pressure of less than 10^{-5} torr was indicated, each unit was permanently sealed off by crimping the fore pump line. Then the temperature of the units was raised gradually as described below.

Preliminary outgassing of the materials was accomplished at about 40°C for about 175 hours, during which time the pressure was less than 10^{-5} torr. Approximately 10 hours were required to raise the temperature from 40°C to 125°C ; the pressure did not exceed 10^{-4} torr during that period. Storage time is computed from the approximate time of first exposure to 125°C , which was at about 6:00 P.M. on December 27, 1965. From 0 to 50 hours, the pressure was less than 10^{-4} torr; from 50 to 150 hours, the pressure had decreased to less than 10^{-6} torr. Preliminary data on the 8-month tests are summarized in Table VII.

Table VII

PRELIMINARY DATA FOR EIGHT-MONTH STORAGE TESTS

Material	Approx. Strain, in/in	No. of Rings	Approx. Rupture Time, hrs at 125°C
SE-3604 (General Electric)	0	5	1 at -160; 1 at -7 1 at 0
	0.25	4	
	0.35	4	
	0.50	4	
	0.75	4	
	1.00	4	
	1.50	4	
Viton A-4411A-990 (Du Pont)	0	8	2 at -3
	0.25	4	
	0.35	4	
	0.50	4	
	0.75	4	
	1.00	4	
	1.50	4	
	2.50	4	
Hycar-1 (B.F. Goodrich)	0	4	2 between 100-160 1 between 170-185
	0.25	2	
	0.35	2	
	0.50	2	
	0.75	4	
	1.00	4	
	1.50	4	

FUTURE WORK

Volatile Condensable Material

Check-out and preliminary runs will be made with the micro-VCM unit. Initial samples will include several materials for which VCM data already have been established at SRI on a macro scale and several polymers of current interest in both Phases of this program.

Work will be resumed on the assembly of the clam-shell units which will be used for establishing VCM data. Investigations are being made into the availability from JPL of a vacuum system of sufficient size to accommodate 12 clam-shell units.

Mechanical Properties

Work will continue on the measurements of stress relaxation changes for selected polymers during their exposure to a thermal-vacuum environment. The present run (see Work Performed) will be completed and work will be initiated on a new set of materials as confirmed by the JPL cognizant Engineer.

Regular observations will be made of the status of the elastomers which are stored under constant strain for 8 months. It is anticipated that detailed drawings for these units will be available for the next report. Work will commence on the fabrication of a constant-load device to be accommodated in the 4th unit; the plastic to be tested has not yet been selected.

Physical Properties

Work will continue on the spectral studies of polymeric materials which have been exposed to the thermal-vacuum environment. Every attempt will be made to complete the mass spectrometric identification of the 4 to 6 substances evolved from a series of Lexans (polycarbonates). Various instrumental difficulties have delayed the examination of polymers in the far infrared; it is hoped that these will be overcome shortly.