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(Final Report)

PREPARATION OF S-13 EXPERIMENTAL COATINGS

National Aeronautics
and Space Administration
George C. Marshall Space Flight Center

Report No. IITRI-U6018-1
(Final Report)

PREPARATION OF S-13 EXPERIMENTAL COATINGS

October 29, 1963, through August 31, 1965

Contract No. NAS8-11967
IITRI Project U6018

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FOREWORD

This is Report No. IITRI-U6018-1 (Final Report) of IITRI Project U6018, entitled "Preparation of S-13 Experimental Coatings." The work described in this report was sponsored under two contracts: Contract No. NAS8-11133 (IITRI Project C6025, October 29, 1963, to March 15, 1965) and Contract No. NAS8-11967 (IITRI Project U6018, April 20 to August 31, 1965). Thus the report covers the period from October 29, 1963, through August 31, 1965. The work was administered under the technical direction of the Propulsion and Vehicle Engineering Laboratory, Materials Division of the George C. Marshall Space Flight Center, with Mr. Lowell K. Zoller acting as Project Engineer.

Major contributors to the program include Gene A. Zerlaut, Project Leader; Warren Jamison and Douglas Vance, space-simulation tests; and Douglas Vance and Noel Bennett, paint and specimen preparation and optical-properties measurements.

Data are recorded in IITRI Logbook C14462.

Respectfully submitted,
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I. INTRODUCTION

The heat-transfer analysis (ref. 1) of the three Pegasus spacecraft launched during 1965 showed that portions of the spacecraft, the S-IV stage, the instrument unit (IU), and the Apollo service-module adaptor (SMA) of the Saturn I launch vehicle required a coating with a solar absorptance, α, of 0.18 to 0.36 and an infrared emittance, ε_t, of about 0.9 throughout the spacecraft's 1-yr orbital lifetime.

A zinc oxide-pigmented polydimethylsiloxane coating was chosen after careful consideration of the factors involved. Factors considered were ultraviolet stability, commercial availability of constituents, ease of application of and maintenance of properties, and low-temperature characteristics. Low-temperature characteristics are of some importance since the S-IV stage is fueled with liquid hydrogen. The paint chosen was designated S-13 and was developed by IIT Research Institute under the initial sponsorship of the Jet Propulsion Laboratory of the California Institute of Technology (ref. 2,3). Additional S-13 development efforts were performed under the sponsorship of the Marshall Space Flight Center (ref. 4,5).

S-13 was chosen because of its exceptionally high stability to ultraviolet irradiation in vacuum, a characteristic that it exhibited in numerous tests. The comparatively large amount of ground-testing data that had been accumulated on S-13, especially solar-simulation-effects information, weighed heavily in its choice for use on this mission. The scale-up from laboratory quantities to the production quantities required for the three Pegasus spacecraft represented an important advancement in the state of the art of spacecraft temperature-control coatings for field use. It was recognized early that the large S-IV stage would have to be painted on location at Cape Kennedy and that the painted vehicle would probably stand on the launch pad for a considerable time prior to launch.

As a consequence of this program, a great deal has been learned about the application of thermal-control coatings to large space vehicles and about the maintenance of such coatings during prelaunch and launch operations.

II. HISTORY OF S-13 THERMAL-CONTROL PAINT

Two zinc oxide-pigmented thermal-control coatings developed for the Jet Propulsion Laboratory (ref. 2,3) under Contract No. 950111 (subcontract under NASA Contract No. NAS7-100) were found to have field-application potential. One is IITRI's Z-93 coating, which utilizes potassium silicate as the vehicle, and the other is the S-13 coating, which is based upon a commercial elastomeric methyl silicone polymer.

The inorganic Z-93 coating, while possessing lower solar absorptance than S-13, has a very short shelf-life. S-13, on the other hand, has excellent shelf-life and is relatively easy to apply and maintain. As a consequence, it has received considerable attention as a candidate white thermal-control coating.

For these reasons as well as those discussed earlier, S-13 was chosen as the white thermal-control paint for Pegasus spacecraft and the associated Saturn upper stages. This application required the manufacture of over 145 gal of S-13 paint.

In addition to being used on Pegasus I, II, and III, S-13 was used as the sole thermal-control coating on the Explorer-24 Injun satellite (tinted green to achieve a solar absorptance of 0.55) and on the Air Density Explorers XIX and XXIV (ref. 6). S-13 currently is being considered for use on a number of spacecraft.

III. COMPOSITION

S-13 is a zinc oxide-pigmented polydimethylsiloxane in toluene. The zinc oxide is SP500 obtained from the New Jersey Zinc Company. The methyl silicone vehicle is RTV-602, a linear polymer obtained from the General Electric Company. The curing agent is General Electric's proprietary SRC-05 catalyst. It is added just prior to application of the paint and is 0.4 to 1.0% by weight of RTV-602 in the paint. The lower catalyst concentration is preferred from the standpoint of ultraviolet stability, but a concentration of 0.76% was used for the Pegasus paint as a satisfactory compromise between optimum physical properties and maximum stability of optical properties.

The composition of S-13 paint furnished for application to the Pegasus spacecraft was:

Constituents	Parts by Weight
New Jersey Zinc SP500 zinc oxide	240
General Electric RTV-602 silicone	100
Toluene	170

The SRC-05 catalyst was added at 1 part per 670 parts by weight of the S-13 paint furnished. The catalyst was added as a 3% solution in toluene; the paint was furnished with a solvent holdout of approximately 15 parts by weight based on the formula cited.

The paint can be applied to any surface to which the primer will adhere. The primer, which is General Electric's SS-4044, must be used to prevent stripping of the cured S-13. The primer is applied at a thickness of about 0.5 mil (0.0005 in.) and requires 1 hr of drying time prior to application of the S-13. It usually is recommended that both the primer and the S-13 be spray-applied to obtain best results. More detailed instructions are contained in the S-13 specification, Appendix I.

IV. DELIVERY SCHEDULE

One hundred and fourty-five gallons of paint were furnished to the George C. Marshall Space Flight Center under the terms of the two applicable contracts. The delivery schedule is presented in Table 1. Deliveries 4 through 11 were made to the Kennedy Space Flight Center, Cape Kennedy, Florida.

Table 1

S-13 DELIVERY SCHEDULE

No.	Delivery Date	Quantity	Applicable Contract
1	December 9, 1963	10 gal	NAS8-11133
2	January 8, 1964	30 gal	NAS8-11133
3	April 8, 1964 (paste)	5 lb	NAS8-11133
4	April 29, 1964	25 gal	NAS8-11133
5	May 13, 1964	15 gal	NAS8-11133
6	July 13, 1964 (paste)	20 lb	NAS8-11133
7	March 2, 1965	5 gal	NAS8-11967
8	March 8, 1965	10 gal	NAS8-11967
9	May 3, 1965	10 gal	NAS8-11967
10	May 7, 1965	25 gal	NAS8-11967
11	June 7, 1965	5 gal	NAS8-11967

The delivery of 5 gal of paint on June 7, 1965, was made after it was learned that one 5-gal pail, delivered on May 3, 1965, was defective when opened at the Kennedy Space Flight Center.

V. PAINT PREPARATION

A. Initial Manufacturing Procedure

The first 40 gal of S-13 paint were pebble-milled in 5-gal batches using two 6-gal capacity borundum-fortified porcelain jars. The mills were one-half full of cylindrical porcelain stones 3/4 in. in diameter and 3/4 in. long. The mills were charged with a pigment-vehicle-solvent slurry prepared according to the formula specified in Section III except that 70 parts by weight of the toluene were held out. The slurry was ground at approximately 70% of critical speed (~40 rpm) for 8 hr. The mill was then discharged and the remaining toluene was added. The solvent-rich slurry was ground for approximately 5 min, and the contents were then added to the main charge. The paint was thoroughly mixed.

Test specimens were prepared and subjected to 2000 equivalent sun-hours (ESH) of ultraviolet radiation in vacuum. The facilities for measurement of optical properties and for ultraviolet irradiation in vacuum are described in Appendices II and III, respectively. The results of the space simulation tests on the first 40 gal of S-13 are presented in Tables 2 and 3.

The solar absorptance increase, $\Delta\alpha_s$, exhibited by specimens prepared from the first 40 gal of S-13 was disappointingly high. The increases in solar absorptance were from 0.05 to 0.08 after exposure to 2000 ESH of ultraviolet radiation in vacuum. Little improvement in $\Delta\alpha_s$ was noted when the ultraviolet irradiation was performed with the substrates maintained at a nominal temperature of -38°F.

An investigation showed that the instability of these specimens was directly related to the grinding time and to the shear forces present in the mill. This investigation is described in Section VI.

The George C. Marshall Space Flight Center was subsequently advised to discontinue use of the first 40 gal of S-13 (except for the purpose of developing scale-up procedures for painting

Table 2

EFFECTS OF ULTRAVIOLET IRRADIATION IN VACUUM ON SPECIMENS OF S-13 PEGASUS PAINT (FIRST DELIVERY)

			Exposure		Solar Absorptance	
Specimen	Mill	Batch Size, gal	Solar Factor	ESH[a]	α_s	$\Delta\alpha_s$
1	A	5	0	0	0.181	
			9.2	1700	0.218	0.037
1	B	5	0	0	0.185	
			9.2	1700	0.209	0.024
1	A + B	10	0	0	0.172	
			10.5	2100	0.252	0.080
2	A	5	0	0	0.186	
			9.2	1700	0.217	0.031
2	B	5	0	0	0.199	
			9.2	1700	0.225	0.026
2	A + B	10	0	0	0.179	
			10.5	2100	0.236	0.057

[a] Equivalent sun-hours of ultraviolet exposure in vacuum.

Table 3

EFFECTS OF ULTRAVIOLET IRRADIATION IN VACUUM ON SPECIMENS OF S-13 PEGASUS PAINT (SECOND DELIVERY)

	Substrate Temperature, °F	Exposure		Solar Absorptance	
Specimen[a]		Solar Factor	ESH	α_s	$\Delta\alpha_s$
1	45	0	0	0.209	
		12	2000	0.268	0.059
2	45	0	0	0.204	
		12	2000	0.274	0.070
3	45	0	0	0.202	
		12	2000	0.269	0.067
4	45	0	0	0.197	
		12	2000	0.256	0.059
5[b]	-38	0	0	0.206	
		10.7	2000	0.273	0.067
6	-38	0	0	0.199	
		10.7	2000	0.258	0.059
7	-38	0	0	0.197	
		11.7	2000	0.254	0.057
8	-38	0	0	0.204	
		11.7	2000	0.264	0.060

[a] 30-gal batch. The six 5-gal batches were mixed and the specimens were prepared from the 30-gal composite.

[b] Soiled with pump oil and cleaned with 10% solution of Alconox.

the large Saturn S-IV stage). The paint preparation procedures were modified accordingly; the following paragraphs describe the procedure adopted for the manufacture of the remainder of the Pegasus paint.

B. Final Manufacturing Procedure

The manufacturing procedure adopted for the remaining 105 gal of S-13 involved (1) replacement of the cylindrical stones with spherical grinding media 0.5 in. in diameter, (2) reduction of the volume of the grinding media by 50% (the mills were 1/4 filled with stones), and (3) reduction of the grinding time to 3 hr.

The use of spherical stones greatly reduced the interfacial contact area in comparison to the use of cylindrical stones. Each stone-stone interface became a point contact in contrast to the line contact prevalent with cylindrical grinding media. Reduction of the volume of the grinding media reduced the weight component of the shear forces required to effect pigment dispersion. These changes, coupled with the reduction of the grinding time to 3 hr, resulted in increased stability of the S-13 paint and did not adversely affect the initial solar reflectance characteristics of the S-13 films.

C. Quality-Control Data

The results of the quality-control space-simulation testing of the remaining paint deliveries are presented in Table 4. Each specimen noted in Table 4 was prepared from a composite of

Table 4

EFFECT OF ULTRAVIOLET IRRADIATION IN VACUUM ON SPECIMENS OF S-13 PEGASUS PAINT (DELIVERIES 3 THROUGH 11)

				Exposure		Solar Absorptance	
Specimen	Catalyst Conc., %	Batch Size	Delivery No.	Solar Factor	ESH	α_s	$\Delta\alpha_s$
Paste	0.76	5 lb	3	0	0	0.184	
				12.4	2000	0.220	0.036
4-1	0.76	25 gal	4	0	0	0.174	
				10	2000	0.221	0.047
5010	0.76	25 gal	4	0	0	0.196	
				10	2000	0.217	0.021
B-1	0.4	15 gal	5	0	0	0.197	
				10	2000	0.206	0.009
B-2	0.4	15 gal	5	0	0	0.203	
				10	2000	0.217	0.014
B-3	0.4	15 gal	5	0	0	0.188	
				10	2000	0.205	0.017
B-4	0.4	15 gal	5	0	0	0.198	
				10	2000	0.209	0.011
Paste	0.4	20 lb	6	0	0	0.189	
				10	2000	0.205	0.016
5124	0.76	15 gal	7 & 8	0	0	0.212	
				10	2000	0.234	0.022
5125	0.76	15 gal	7 & 8	0	0	0.217	
				10	2000	0.235	0.018
5150	0.76	10 gal	9	0	0	0.181	
				10	2150	0.205	0.024
5151	0.76	10 gal	9	0	0	0.179	
				10	2150	0.213	0.034
5152	0.76	30 gal	10 & 11	0	0	0.173	
				10	2150	0.208	0.035
5153	0.76	30 gal	10 & 11	0	0	0.175	
				10	2150	0.203	0.028

the entire delivery, or deliveries, as noted. For example, specimens 4-1 and 5010 were prepared from a composite of samples taken from each of the five 5-gal batches that constituted delivery 4. On the other hand, specimens 5124 and 5125 were prepared from a composite of two deliveries, 7 and 8, which were 5 and 10 gal, respectively. Likewise, specimens 5152 and 5153 were made up from a composite prepared from deliveries 10 and 11.

The increases in solar absorptance, $\Delta\alpha_s$, observed for the specimens shown in Table 4 were of the magnitude expected. The modifications in the paint-preparation procedure were sufficient to reduce the effects of grind time and shear forces to a tolerable level. The lower solar absorptance increases observed for the specimens prepared from deliveries 5 and 6 are thought to result from the lower catalyst concentration used to prepare these samples. This lower concentration of 0.4% (based upon RTV-602) was inadvertently used to prepare these specimens.

Specimens subjected to ultraviolet irradiation in vacuum after February 1965 were thermally evacuated (150°F at 10^{-6} torr) for 10 to 16 hr prior to ultraviolet irradiation. This procedure ensured thorough curing of all S-13 specimens and precluded the possibility of photolysis of unreacted polymer and catalyst, which can migrate to the surface.

Test specimens were prepared at Cape Kennedy by Douglas Aircraft personnel when the launch vehicles were painted. The aluminum substrates were located on the SA-9 instrument unit

(IU), the S-IV, and the service-module adaptor (SMA) stages and were coated at the time the stages were painted. The specimens were subjected to the same environment as the vehicle, and two from each stage were subsequently submitted to IIT Research Institute for space-simulation testing. The SA-9 stages (IU, S-IV, and SMA) were cleaned with a 10% Alconox (Alconox Inc.) solution and copious quantities of distilled water 1 week prior to launch. Specimens 5118 through 5123 received the same treatment, were mounted in the Launch Complex 39 Service Tower, and were subjected to the same Cape Kennedy environmental conditions as the entire SA-9 vehicle. The specimens were removed subsequent to launch and submitted to IITRI without further treatment. The results of exposure of these control specimens to 2000 ESH of ultraviolet irradiation in vacuum are presented in Table 5.

Film was removed from SA-9's IU stage by the author after it was cured on November 4, 1964, the day the stage was painted. This detached film is labeled "free film" in Table 5. It was subjected to 450 ESH immediately after SA-9 was launched in order to ascertain whether an anomaly noted in the preliminary temperature data was due to defective S-13. The very low solar absorptance increase, $\Delta\alpha_s$, of 0.005 as well as the results of the exposure tests on the control samples submitted to IITRI by NASA indicate that the S-13, as applied, was as stable as predicted.

Table 5

EFFECT OF ULTRAVIOLET IRRADIATION IN VACUUM ON SPECIMENS OF S-13 PEGASUS PAINT PREPARED AT CAPE KENNEDY FOR SA-9 (PEGASUS I)

			Exposure		Solar Absorptance	
Specimen	Saturn Stage	Thickness, mils	Solar Factor	ESH	α_s	$\Delta\alpha_s$
5118	IU	7	0	0	0.195	
			10	2000	0.217	0.022
5119	IU	7	0	0	0.205	
			10	2000	0.224	0.019
5120	S-IV	6	0	0	0.195	
			10	2000	0.216	0.021
5121	S-IV	6	0	0	0.193	
			10	2000	0.218	0.025
5122	SMA	6	0	0	0.229	
			10	2000	0.248	0.019
5123	SMA	6	0	0	0.226	
			10	2000	0.245	0.019
Free film	IU	8	0	0	0.195	
			10	450	0.200	0.005

The total emittances of specimens of the S-13 paint furnished for the Pegasus program are presented in Tables 6 and 7. The total normal emittance values (Table 6) were obtained on the first delivery of paint. Emittance data were not obtained on the second delivery of paint because of the high $\Delta\alpha_s$s observed after exposure to ultraviolet radiation in vacuum.

Table 6

TOTAL NORMAL EMITTANCE OF S-13 SPECIMENS (DELIVERY 1, COMPOSITES)

Thickness, mils	Total Normal Emittance (300°F)
5.0	0.90
5.0	0.82
7.3	0.84
9.7	0.90

Hemispherical emittance data were obtained on composite specimens prepared from delivery 4; the total hemispherical emittance values are more reliable than the total normal values. The less reliable total normal values are a result of low signal-to-noise ratios when the measurements were performed at temperatures below 500°F in the equipment available at IITRI. The data are contained in Table 7.

Table 7

TOTAL HEMISPHERICAL EMITTANCE OF S-13 SPECIMENS (DELIVERY 4, COMPOSITES)

Thickness, mils	Substrate Temperature, °F	Total Hemispherical Emittance
5	35	0.84
	-40	0.86
5	35	0.85
	-40	0.87
6	35	0.86
	-40	0.88

Additional total hemispherical emittance determinations of S-13 specimens were not performed on samples of subsequent deliveries because (1) funds were diverted to underwrite, in part, the studies that were required to solve the mill-yellowing and initial instability problem and (2) these coatings had previously been shown (ref. 2,3) to possess emittances of 0.85 to 0.90 for 5- to 7-mil films.

VI. INVESTIGATION OF MILL-YELLOWING AND PAINT INSTABILITY

Two problems were encountered in scaling up the preparation of the S-13 from small laboratory batches to 5-gal production batches. One was severe mill-yellowing, which occurred during the grinding operation carried out in the large porcelain ball mill; the other problem was decreased stability to ultraviolet radiation in vacuum as a result of grinding in the large mill.

Yellowing of zinc oxide powders results from mechanical distortion. Mechanical distortion of zinc oxide can be produced by scratching the surface of a zinc oxide-silicate coating, grinding the powder with an agate mortar and pestle, or compacting the powder at high pressures. The very process of paint manufacture that involves grinding the pigment into a vehicle in order to wet the particles causes yellowing when shear forces are excessive. Excessive shear forces result from too high a ratio of grinding medium to mill charge or from operation of the mill at too high a speed.

Several paints were prepared by grinding for different lengths of time. Solar absorptance and stability of solar absorptance to ultraviolet radiation in vacuum were determined as a function of grind time. Figure 1 shows that stability is a function of the duration of grinding, regardless of whether a small-capacity or a 5-gal mill was used. Maximum stability is achieved after short grinding times of only 3 hr, which was the grinding time used to prepare the bulk of the Pegasus paint.

The shape of the $\Delta\alpha_s$ curves in Figure 1 is explained as follows. The intial increase in stability (i.e., decrease in effect on solar absorptance) is attributed to an increased degree of dispersion of the pigment. That is, the initial high instability is thought to be due to photolysis of islands of polymer that are not protected by the screening effects of the zinc oxide. The minimum degradation occurs when the benefits

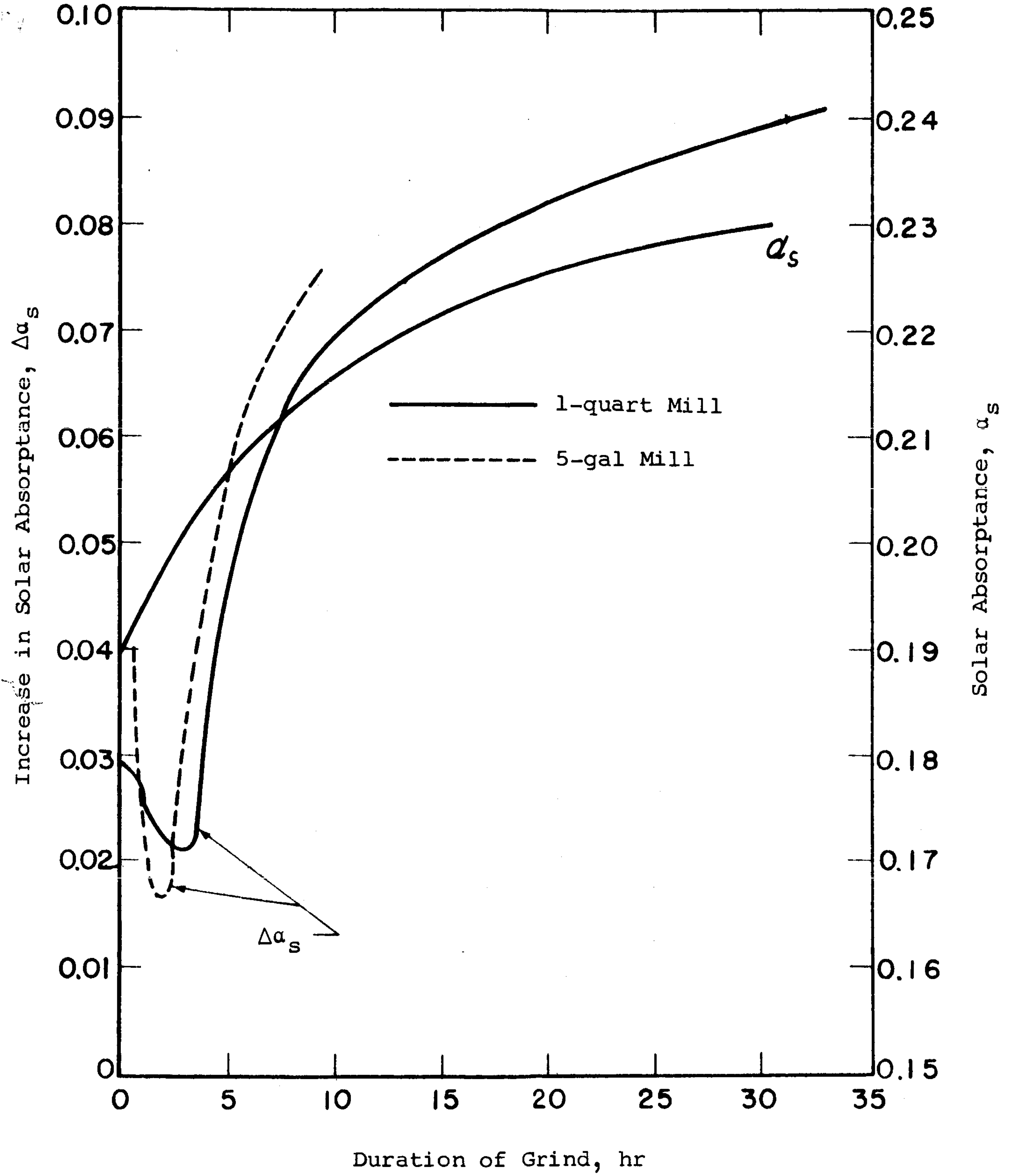

Figure 1

EFFECT OF DURATION OF GRINDING ON STABILITY OF S-13 PAINT of improved dispersion no longer counterbalance the degrading influence of grinding.

Degradation was first thought to be due to silica contamination as a result of shearing forces, the effects of which become important after long grinding. However, shearing forces create lattice distortion in zinc oxide, resulting in the creation of an absorption band centered at 3850 Å, which may be responsible for the observed increase in degradation. The possibility of silica contamination has been ruled out by carefully grinding zinc oxide in an agate mortar and pestle and blending it into the silicone elastomer used in the S-13 formulation. This work was performed for the George C. Marshall Space Flight Center on another program (ref. 4). In a 1500-ESH test the $\Delta\alpha_s$ was 0.049 for the paint made from stressed zinc oxide. This figure compared unfavorably with that of our standard S-13, which degraded by 0.025 in the same test.

VII. COGNATE STUDIES BY OTHER ORGANIZATIONS

A number of studies on S-13 have been performed by other organizations, and their conclusions have been abstracted here as pertinent to this discussion. Zoller (ref. 7) has tabulated available data on specimens of the S-13 paint applied to the SA-9/Pegasus I spacecraft. His data included the results of studies performed by the Materials Division of the George C. Marshall Space Flight Center on control specimens prepared at the Cape Kennedy launch site. The results of these studies essentially corroborate the previous data on S-13 films.

Badin (ref. 8) reported the results of radiative property measurements performed at the Kennedy Space Center on the SA-9/Pegasus I vehicle. The Lion Research model 25-Å emissometer was employed for in situ total normal emittance determinations; the Gier-Dunkle mobile solar reflectometer was employed for in situ solar absorptance measurements. Badin concluded that the effects of contamination, such as fingerprints and dust collection, on the initial optical and radiometric characteristics of the S-13 painted surfaces were negligible. He noted that washing the paint with detergent (Alconox) and water prior to launch removed "the major portion of this contamination." In two other memos (ref. 9,10), Badin presented the results of in situ optical and radiometric measurements on the SA-8/Pegasus II and SA-10/Pegasus III spacecraft, respectively.

Woerner (ref. 6) has recently discussed the results of his studies on S-13 (prepared by NASA's Langley Research Center from IITRI specifications) for use on the Explorer XIX and Explorer XXIV Air Density (inflatable) spacecraft. The distensibility and maintenance of properties at low temperature made the S-13 paint an attractive candidate for application to expandable structures. Woerner concluded that the solar absorptance of S-13 (as well as that of another less stable candidate) was affected much less by electron radiation than by ultraviolet radiation for equivalent exposure times in terms of a given orbital lifetime. His data also corroborate the previously reported data as well as the information contained in this communication.

VIII. CONCLUSIONS

The work described herein has confirmed the excellent stability of the S-13 paint formulation to a simulated space environment. It has shown that the paint applied to the Pegasus I, II, and III spacecrafts and supporting structures exhibited satisfactory, if not excellent, initial solar absorptance and emittance properties as well as excellent stability characteristics. All indications and observations indicate that all S-13 surfaces were in excellent condition at the time of launch.

The major accomplishments resulting from the use of the S-13 paint on the Pegasus spacecrafts, amounting in each of the three cases to the largest single use to date of a thermal-control paint, included (1) the satisfactory sacle-up from small-size laboratory to 5-gal production quantities of a highly specialized thermal-control coating, (2) the completely satisfactory field application of a thermal-control paint to exceptionally large surface areas,* and (3) the successful implementation of on-pad cleaning procedures of the entire upper-stage configuration of SA-8, SA-9, and SA-10.

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*The IU, S-IV, and SMA stages possessed approximately 2500 ft^2 of S-13 painted surfaces, requiring about 20 gal of S-13.

APPENDIX I

S-13 PAINT SPECIFICATION

Modification II

Materials	Parts by Weight
New Jersey Zinc SP500 zinc oxide	240
General Electric RTV-602 silicone	100
Toluene	170

Formulation: The zinc oxide, the RTV-602, and 100 parts by weight of the toluene are premixed and charged to a porcelain ball mill in a quantity sufficient to just fill the void space when the mill is one-fourth full of grinding stones 0.5 in. in diameter. The paint is ground for 3 hr at approximately 70% critical speed. The critical speed (rpm) is given by: $w_{c_s} = \frac{54.2}{\sqrt{R}}$, where R is the radius of the mill in feet. The basic charge is then removed, and 70 parts of toluene are added to the mill. The mill residue and the solvent are ground until the contents are uniformly thin, but not for more than 5 min. The contents are then added to the main charge, and the whole charge is mixed thoroughly. NOTE: THE SRC-05 CATALYST IS NOT ADDED UNTIL THE PAINT IS APPLIED.

Preparation of Paint for Application: The paint is furnished without the SRC-05 catalyst. The catalyst is added as 1 part SRC-05 in 20 parts of toluene per 670 parts of S-13 (as formulated). This concentration represents 0.76% catalyst based on polymer solids. A lower concentration is recommended in order to ensure optimum stability to ultraviolet irradiation in vacuum. A concentration of 0.4% based upon RTV-602 provides optimum stability without greatly sacrificing terminal cure properties, although a coating prepared at this concentration represents the lower limit without sacrificing cure and physical properties. Somewhat better physical properties are obtained with a catalyst concentration of 0.5% based on RTV-602. A catalyst concentration of 0.4% of resin solids corresponds to 1 part SRC-05 per 1275 parts of S-13; 0.5% catalyst requires 1 part catalyst per 1020 parts of S-13. The catalyst should be added as a 20:1 reduction in toluene. The catalyst solution is added <u>only</u> as the paint is used and to only the amount that can be applied in about 30 min. The bulk paint was furnished in 5-gal epoxy-lined metal pails. The paint should be thoroughly stirred before transfer to other containers or before addition of catalyst. Allow the catalyst paint to set for 10 min before application to the primed surfaces.

<u>Preparation of Surfaces for Painting:</u> Standard surface cleaning procedures should be used to prepare the surface for application of the S-13 paint. S-13 paint can, in general, be applied to any surface to which the required primer can be applied. The primer, General Electric's proprietary SS-4044, can be applied to either anodized or zinc chromate-primed surfaces. It is preferable that it be applied to clean bare metal or to anodized surfaces, however. Greasy surfaces should be cleaned with standard detergent and water prior to priming; they should be thoroughly dry.

Application of Paint: The primer can be spray-applied (Binks model 18 or comparable gun) at about 30 psi. Only about 0.5 mil of primer is required (just enough to provide a base for the S-13 paint). The primer should be allowed to air-dry for 1 hr before application of the S-13 paint.

The S-13 paint can be spray-applied with a Binks model 18 spray gun (or comparable gun) at a gas pressure of about 60 psi. Unless missile-grade air is available, prepurified nitrogen or prepurified air must be used. The S-13 paint should be allowed to air-cure 16 hr. IT IS IMPERATIVE THAT DUST AND DEBRIS BE KEPT OFF THE SURFACE DURING THE CURING PROCESS.

The wet film thickness of the paint can be measured by either the Pfund or the Interchemical wet-film thickness gage, or a suitable bridge-type gage. Dry film thickness can be measured with a Fischer Permascope nondestructive thickness tester, type ECTH.

Reapplication: Soiled or damaged areas can be recoated. Soiled areas must be cleaned thoroughly with detergent and water and dried before application of additional S-13 paint. Damaged or gouged areas can be recoated by making a paste of S-13 in which the bulk of the solvent is omitted. Such a material can be trowelled or brushed over the damaged areas and cures can be tack-free within a few hours.

Storage of the Paint: The paint is supplied in 5-gal quantities. Since the paint cannot be mixed with catalyst solutions in 5-gal lots because of the pot life of the catalyzed paint, it may be desirable to store the paint in small containers in order to avoid contamination when the 5-gal container is opened and closed a number of times. If smaller containers are utilized, only glass, nickel, or unlined unleaded unsoldered steel cans can be used. The caps, tops, or closures of these containers should not possess gummed seals or any material soluble in toluene.

Physical Properties: Paint S-13 is rubbery and resilient. Therefore, it can be gouged by a sharp tool with little effort. Its adherence is excellent when a primer is used but is very poor when applied directly to a metal substrate, in which case it can be stripped from the substrate in one piece. Because of the resiliency of the surface, dirt tends to cling to the surface. Dirt can be easily removed by wiping with a water-moistened CLEAN, SOFT cloth. NOTE: S-13 SHOULD NEVER BE CLEANED WITH ORGANIC SOLVENT. Paint S-13 withstands more than 10 thermal-shock cycles consisting of immersion in liquid nitrogen followed by rapid heating to 200°F. The paint can be torsionally stressed to 90° without failure and withstands repeated bending to 180°.

Optical Properties: Minimal solar absorptance is not obtained until a thickness of nearly 10 mils is reached. The following tabulation is provided as a guide.

Thickness (±0.25 mil)	Solar Absorptance (±0.01)
1	0.30
2	.25
3	.23
4	.21
5	.20
6	.19
8	.18
9	.17
10	0.17

A working range of 5 to 8 mils is recommended. For coatings of 5 mils or thicker, the total hemispherical emittance is 0.85 or better at 300°K.

Stability to Ultraviolet Irradiation in Vacuum: The severest test to which this material has been subjected was that of 7000 ESH; the solar absorptance increased from 0.20 to 0.23, with a $\Delta\alpha = 0.03$. Cleaning with water or with detergent and water before irradiation does not affect stability. CLEANING WITH ACETONE AND OTHER ORGANIC SOLVENTS, HOWEVER, SEVERELY AFFECTS STABILITY. Therefore, cleaning only with water or with water and detergent is required. Until another detergent or wetting agent that does not affect stability is found, the use of a 10% solution of Alconox (Alconox Inc.) followed by rinsing with copious quantities of demineralized or distilled water is recommended.

APPENDIX II

MEASUREMENT OF THERMOPHYSICAL PROPERTIES

Determination of Solar Absorptance: Solar absorptance is determined indirectly by measurement of spectral reflectance in the wavelength range 0.21 to 2.7 μ. The reflectance data are integrated with solar spectral energy data for the upper atmosphere (ref. 11) to yield normalized solar reflectance. Subtraction of the solar reflectance from unity yields solar absorptance. Comparison of these solar spectral energy data with the data of Nicolet (ref. 12) and Johnson (ref. 13) shows that solar absorptance agrees within 0.005. An IBM 7090 Fortran computer program performs this integration according to the mathematical definition given below:

$$\alpha_s = \frac{\int_{0.2\mu}^{2.7\mu} (1 - R_\lambda)_\lambda \, W_{s,\lambda} \, d\lambda}{\int_{0.2\mu}^{2.7\mu} W_{s,\lambda} \, d\lambda}$$

where $W_{s,\lambda}$ is the solar radiation intensity in the wavelength interval between λ and $\lambda + d\lambda$. The value $(1 - R_\lambda)_\lambda$ is the spectral absorptance obtained by subtracting the spectral reflectance from unity.

Spectral Solar Reflectance Measurements: Two systems were used during this program. One of these is the Cary model 14, which is used for spectral reflectance measurements in the 0.25 to 2.0-μ region. The Cary model 14 is equipped with a model 1411 integrating sphere accessory and a recording device. The reflectometer is normally used in Type II operation; that is, the sample is illuminated with diffuse, nondispersed light and is viewed through a small solid angle at near normal direction. The Cary 14 was used on Project C6025 (Contract No. NAS8-11133), which involved the first six deliveries of S-13 paint.

The second unit is a Beckman DK-2A instrument equipped with the standard Beckman integrating-sphere attachment. It is used in Type 1 operation; that is, the specimen is illuminated with dispersed light at near-normal incidence and is viewed hemispherically. It is used in the 0.21- to 2.7-μ region. The DK-2A spectroreflectometer was used on Project U6018 (Contract No. NAS8-11967), which involved deliveries 7 through 11.

Emittance Measurements: An evacuated calorimetric device was used for measuring the total hemispherical emittance of the S-13 specimens in the -40 to +40°F range. This instrument utilizes the equilibrium technique for determination of emittance. The sample is deposited on a 1-in. cube, which contains a resistance heater. Thin power leads support the cube inside a liquid nitrogen-cooled sphere, which is coated with a black paint. The sphere is, in turn, contained in a vacuum system to eliminate convection losses. Nonradiative losses from the cube are taken into account.

APPENDIX III

SPACE (SOLAR) SIMULATION CHAMBERS

The workhorse, the ion-pumped space simulation chamber, is shown in Figure 2. The chamber is provided with a quartz window and a liquid-cooled table. The table can be cooled with liquid nitrogen, ice water, or tap water (or heated fluids) and can accommodate twelve 1 x 0.5-in. specimens. The system consists of a 400-liter/sec Varian VacIon pump and a mechanical forepump. An AH-6 lamp is mounted over the quartz window. Reproducible equivalent solar factors, as determined with a temperature-compensated thermopile, can be achieved from 1 to 15 solar intensities in the 2000- to 4000-Å wavelength region.

In addition to the workhorse chamber described above, a multiple-chamber ion-pumped system, called the Quad-Ion system, was used for studies in which different exposures, different intensities, and different temperatures are required with the same set of AH-6 lamps. This system has been particularly useful in studying reciprocity effects and filter effects on engineering coatings. A photograph of the system is given in Figure 3.

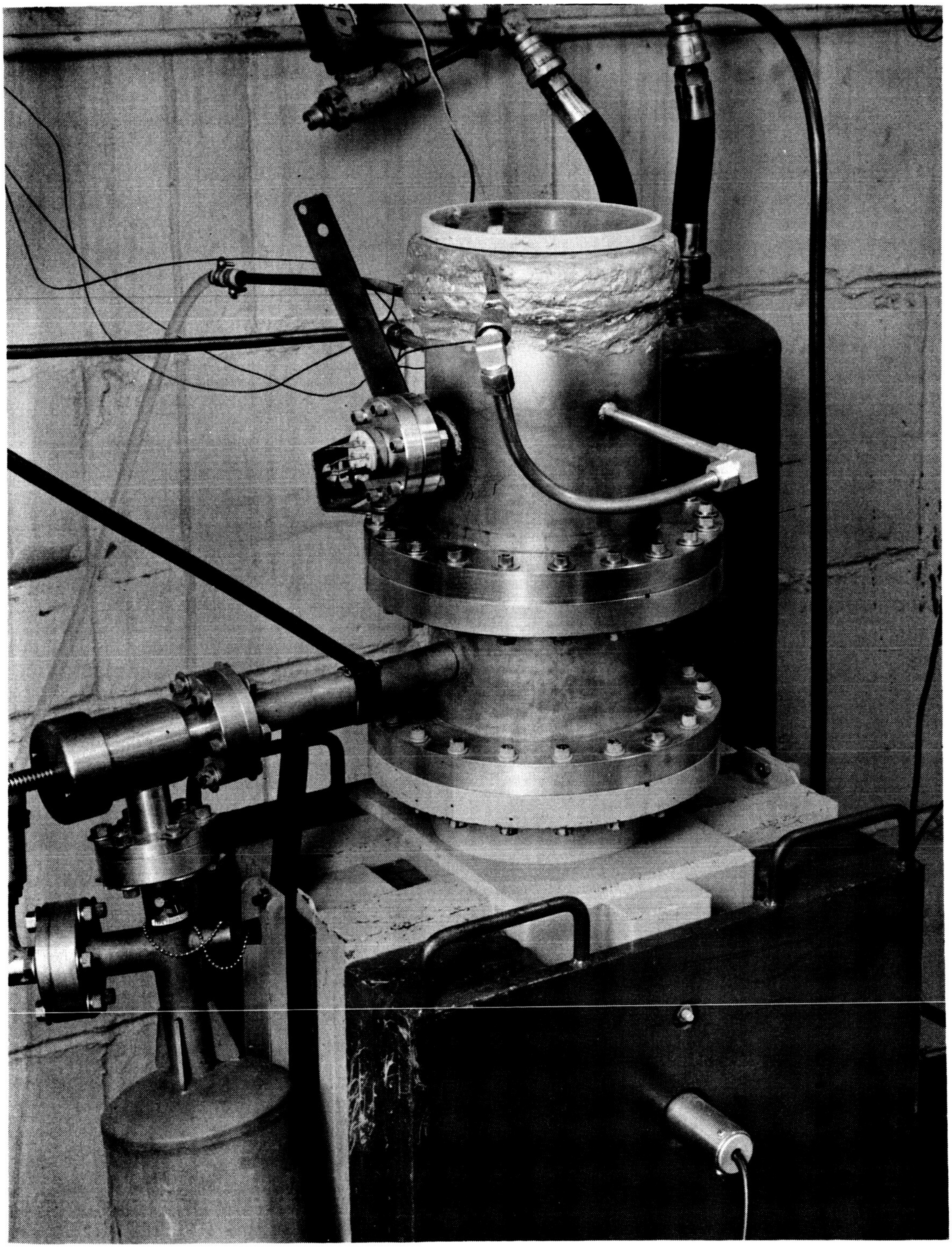

Figure 2

VACION SOLAR SIMULATION CHAMBER

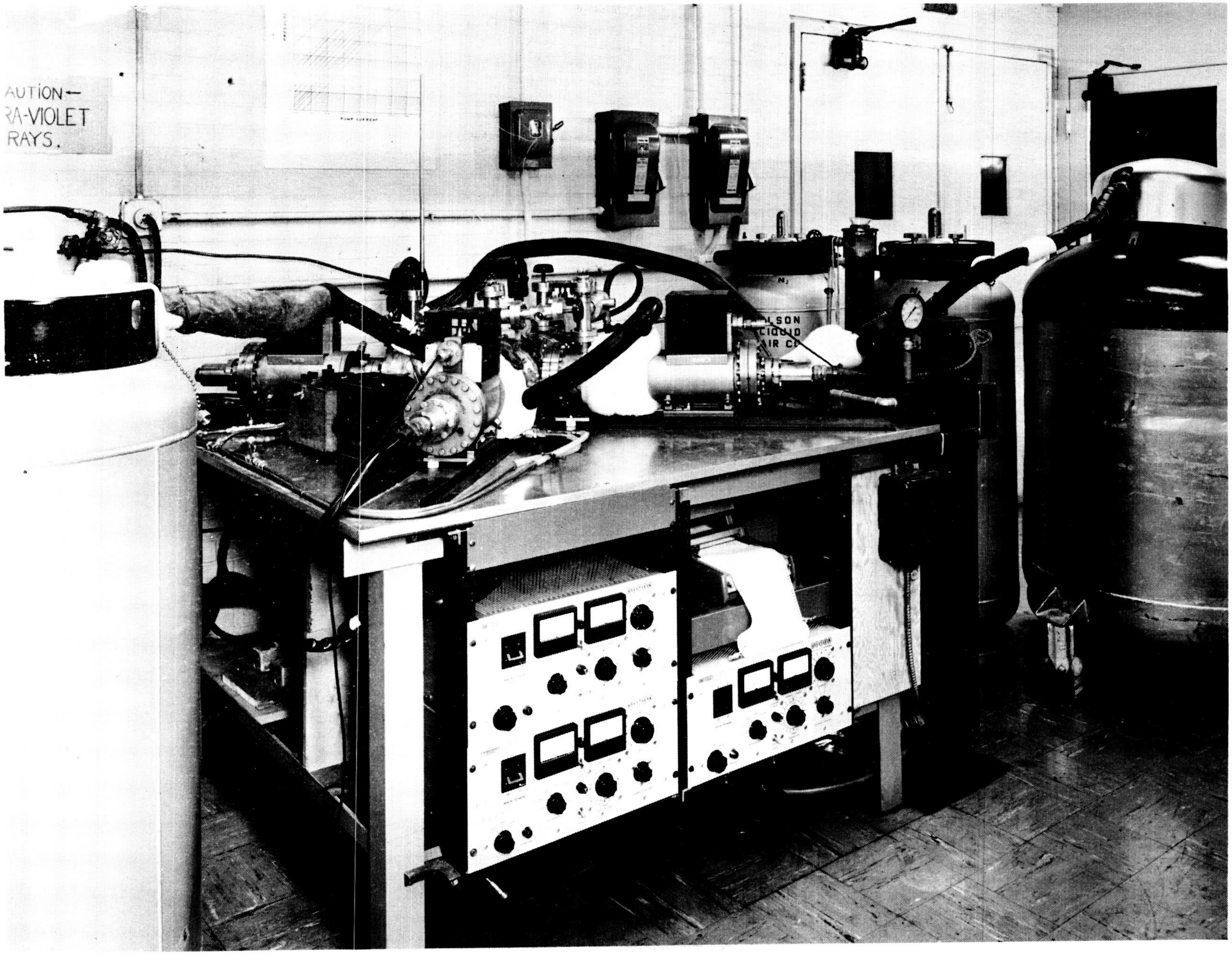

Figure 3

QUAD-ION SOLAR SIMULATION CHAMBER

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