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DEVELOPMENT OF THERMAL RADIATIVE SURFACE COATINGS FOR NASA-LEWIS SPACE SIMULATION CHAMBER

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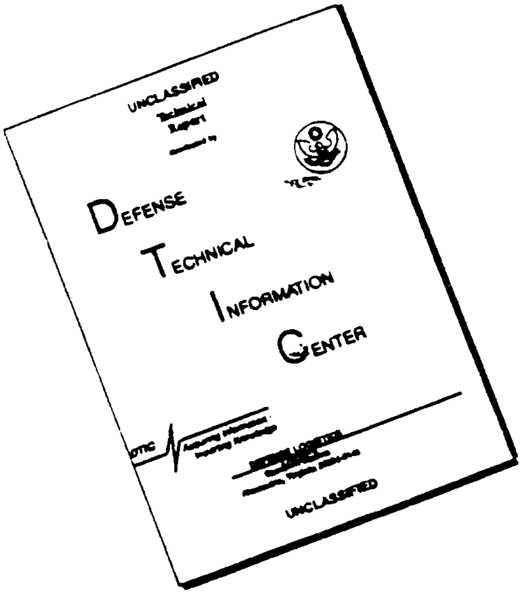

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ABSTRACT

DEVELOPMENT OF THERMAL RADIATIVE SURFACE COATINGS FOR NASA-LEWIS SPACE SIMULATION CHAMBER

Five black polymeric coatings were tested and found almost equally suitable for use in a space-simulation chamber being built for the NASA-Lewis Research Center. These are Dalvor, a fluorocarbon coating, which was the best of the five; Micobond L6X962, a vinyl-modified phenolic coating; a phenolic-modified epoxy coating; and two silicone resin coatings.

These coatings were tested for resistance to abrasion, acids, thermal shock, detergents, live steam, optical radiation and nuclear radiation (under high vacuum while at low temperature). Their spectral properties, were measured, as were their outgassing rates under high vacuum while irradiated by ultraviolet light. Their ease of decontamination when contaminated with radioactive fission products was determined.

Before these five coatings were selected, many others were screened by a literature search or by one or more of the tests noted above.

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FOREWORD

This is Report No. NASA-CR-54274 Report No. IITRI-C6042-10) of IITRI Project C6042, Contract No. NAS3-6019, entitled "Development of Thermal Radiative Surface Coatings for NASA-Lewis Space-Simulation Chamber." This report covers the period from 29 June to 30 May 1965.

Major contributors to the program included J. H. Baldrige, Project Leader; G. A. Zerlaut, general consultation; S. Shelfo, paint formulation, application, and physical testing; W. Courtney, space simulation and high vacuum equipment design; D. G. Vance and N. D. Bennett, optical measurements; A. Brauner and R. Barrall, nuclear and health physics studies; W. Bahmet, decontaminability studies; and J. E. Gilligan, general consultation on nuclear irradiation and associated problems. Mr. R. A. Lindberg, of the Lewis Research Center, served as NASA's Project Manager.

Data are recorded in IITRI Logbooks C15056 and C15549.

Respectfully submitted,

IIT RESEARCH INSTITUTE

DEVELOPMENT OF THERMAL RADIATIVE SURFACE COATINGS
FOR NASA-LEWIS SPACE-SIMULATION CHAMBER

I. INTRODUCTION

Space chambers are an indispensable tool for developing and evaluating materials and hardware for use in space. These chambers supply three conditions: high vacuum (pressures of 10^{-6} torr and below), temperatures from ambient to cryogenic, and energy similar in its spectrum to that received from the sun but often greater in its intensity than that of sunlight in order to accelerate testing. Numerous chambers of this type are now in use, but several larger chambers capable of providing simulated-space conditions for chemical rockets, nuclear space systems, and other large components are being studied or are under construction.

One of these large chambers is being built for the Lewis Research Center. Since it is anticipated that nuclear systems will be tested in it, its walls will be made of aluminum alloy (5083 faced with 3003 alloy inside) in order to minimize residual activity on the walls after tests.

A resistant coating on the aluminum is highly desirable because the chamber will be exposed to corrosive materials such as rocket exhaust products and rocket fuels. Some other requirements, such as low outgassing of the chamber in high vacuum, ease of coating application and replacement, and the desirability of a nonreflecting heat sink, make a polymeric coating highly desirable. A suitable polymeric coating would be expected to absorb much of the ultraviolet, visible, and thermal radiation to which it is exposed, to minimize light reflection to materials or components being tested, and to protect the chamber interior against corrosion.

The coating should have a low outgassing rate when exposed to ultraviolet or nuclear radiation and should be easily decontaminated with standard decontaminating agents such as hot water plus detergent or live steam at 100 psig. In addition, the coating should be nonporous, resistant to acids and alkalis, and sufficiently abrasion-resistant to permit repeated cleaning by scrubbing. A desirable life expectancy of the coating would be the order of ten years.

The relative importance of these chemical, mechanical, and nuclear resistance factors is, of course, highly dependent on the chamber's end use. The conditions anticipated by NASA engineers include:

(1) Temperature

  (a) Steady state of any temperature between -320 and 180°F

  (b) 1000 cycles between -320 and 180°F at a maximum rate of 20°F/hr

  (c) Maximum steady state temperature differential of 33°F between any 3 ft of adjacent surface

  (d) 5-min temperature of 350°F when steam-cleaning contaminates surfaces, if they cannot be decontaminated at lower temperatures

(2) Pressure

  (a) Steady state operation at 10^{-8} to 10^{-6} torr.

  (b) 1000 cycles between atmospheric pressure and 10^{-8} torr, each cycle requiring a 48-hr period.

  (c) 1000 pressure increases from 10^{-8} to 250 torrs, each occurring in 3 seconds.

  (d) Ten cycles between atmospheric pressure and 5 psig; each cycle will involve 1 day to pressurize, 5 day's hold, and 1 day to release pressure.

(3) Radiation

  (a) Fast neutrons ($>$ 0.1 Mev) with a minimum exposure of 2×10^{15} n/cm^2.

  (b) Gamma ray exposure corresponding to a minimum of 10^7 rads (tissue dose).

  (c) Thermal radiation corresponding to a maximum source temperature of 2500°F and a mean temperature of 1500°F, in 2000-hr increments.

  (d) Ultraviolet and thermal radiation from solar simulators with a maximum intensity of 2 earth solar constants.

(4) Miscellaneous exposure

  (a) Mixed chemical rocket combustion products at 250 torrs and 500°F for 1 hr with chamber wall at -300°F; coating may be exposed to liquid or vapor products for 5 or 6 hr after chamber warms up.

  (b) Exposure to cesium and mercury vapor at 10^{-6} torr with chamber wall at -300°F; coating may be exposed to these metals for 5 or 6 hr after chamber warms up.

(c) Live steam at 100 psig.

(d) Hot decontaminating fluids at 240°F maximum.

II. PROGRAM OUTLINE

A. Phase I

The work of this program was divided into two phases, I and II. The thirty candidate coatings listed in Table 1 were screened in Phase I. Nine of these were chosen by NASA engineers and were listed in the Work Statement. The other twenty one were chosen largely on the basis of a study of available literature, with the concurrence of NASA engineers. The literature surveyed is listed in Appendix A.

Four physical tests were used for the initial screening. These tests, described in Section III, were: thermal shock, abrasion resistance, acid resistance, and resistance to a jet of live steam at 1000 psig.

Pigmented commercial coatings were applied according to their manufacturers' recommendations to panels of 3003 aluminum that has been thoroughly cleaned and degreased. If such a coated panel failed under thermal shock or steam jet testing, the coating was reapplied to a conversion-coated[a] 3003 panel and the two tests were run again. Coatings that failed again were dropped from the testing program.

The adhesion of some of these coatings might have been further improved by applying them to a sandblasted surface, but the adhesion of most coatings to a conversion-coated surface was more than sufficient for the purposes of this program.

These four preliminary screening tests were also used to optimize pigment volume concentration (PVC) in unpigmented commercial and experimental coatings and to establish other parameters.

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[a] A chemically bonded phosphate or chromate used to promote adhesion.

Table 1

COMPLETE LIST OF THIRTY CANDIDATE COATINGS

Basis of Selection	Coating Name or Type
Work statement	3M Velvet coating 101-C10
Literature	Glidden styrenated phthalate-alkyd
Work statement	Krylon Black
Work statement	Parsons Optical Black
Work statement	Kemacryl M49 BC12
Work statement	Micobond Black L6X962
Work statement	10043 aluminum silicone
Work statement	Fuller 171A152
Work statement	Modified Du Pont PR229
Work statement	Sicon Black 3X923
Literature	3M Velvet coating 301-C10
Literature	3M Velvet coating 401-C10
Literature	Sherwin-Williams experimental silicone alkyd BMM-80 and PSB-17
Literature	GE SR 112 silicone resin, Raven 40 pigment, SC 3963 curing agent
Literature	GE SR 112, Raven 11 pigment, SC 3963 curing agent
NASA-Lewis	Dalvor fluorocarbon dispersion
Literature	GE SR 112, Neo Spectra pigment, SC 3963 curing agent
Literature	GE SR 112, Molacco H pigment, SC 3963 curing agent
Literature	GE SR 112, Raven 40 pigment, TBPS additive, SC 3963 curing agent
Literature	GE SR 112, Raven 40 pigment, zinc octoate catalyst
NASA-Lewis	Teflon fluorocarbon dispersion
Literature	GE SR82, Molacco H pigment, SC 3963 curing agent
Literature	Du Pont Imron polyurethane coating, Raven 40 pigment
Literature	Spencer-Kellogg Spenko 49-60CX, Raven 40 pigment
Literature	Spencer-Kellogg Spenko XP1531, Raven 40 pigment
Literature	Epoxy-pigmented Shell Epon resin
Literature	Silicone-modified epoxy resin, Plaskon ST874
Literature	Epoxy-modified phenolic resin
Literature	Phenolic resin, BKS 2600
Literature	Epoxy-modified melamine or triazine resin

such as cure time and temperature, particle size and surface chemistry of the carbon black pigment, and application conditions.

When the list of thirty candidate coatings had been approved by NASA-Lewis Project Management, the screening tests specified by the Work Statement were conducted. These additional tests, described in detail in Section III, were measurements of: spectral (reflectance) properties, decontaminability of coated panels on which highly radioactive fission products had been spilled, resistance to a jet of Alconox detergent solution at 240°F, and semiquantitative outgassing characteristics during exposure to ultraviolet light under high vacuum.

B. Phase II

At a meeting held in Cleveland on 16 February 1965, the data obtained thus far were considered by J. H. Baldrige of IIT Research Institute and R. A. Lindberg and H. E. Freedman of the Lewis Research Center. At this time, the five paints to be tested under Phase II were chosen. These were Micobond Black L6X962, a vinyl-modified phenolic coating produced by Midland Industrail Finishes Company (IITRI Coating No. 42-37), a pigmented Dalvor fluorocarbon dispersion furnished by Diamond Alkali Company (IITRI Coating No. 42-34), an epoxy-modified phenolic resin coating (IITRI Coating No. 42-38), a high-temperature-cure silicone resin (IITRI Coating No. 42-48), and a medium-temperature-cure silicone resin (IITRI Coating No. 42-51).

The qualifying tests for the five coatings were: quantitative outgassing measurements, measurements of spectral normal emittance and hemispherical total emittance, tests on the effect of exposure to 2000 equivalent sun-hours (ESH) of simulated extraterrestrial solar radiation, and tests on the effect of exposure to fast neutrons and gamma radiation. These tests and their results are described in detail in Section III.

III. EXPERIMENTAL WORK

A. Preparing Test Specimens

Specimens were prepared by applying coating dispersions with a Paasche AUTF airbrush to 3-in. x 1-in. panels of 3003-0 aluminum alloy previously cleaned with steel wool and an Alconox detergent solution until no water breaks were evident when the panels were rinsed with water. The panels were then dried,washed with adegreasing organic solvent such as trichloroethylene, and again dried. Primers (noted in Table 2) were used only for those commercial coating for which the literature or recommendations made it mandatory. Some formulations that adhered poorly to degreased panels were applied to panels of 3003-0 aluminum previously coated by the Iridite process, as described in Appendix G. The standard panel thickness was 0.061 in., and the standard coating thickness was 0.003 ± 0.001 in., as measured with a Permascope type ECT thickness gauge. For certain tests, such as some spectral or emittance measurements, the standard dimensions could not be used and such exceptions are noted in the individual test descriptions that follow.

Pigment volume concentrations (PVC) had to be adjusted for most unpigmented commercial and experimental binders, in order to optimize adhesion and resistance properties. The PVC of each formulation is given in Table 2. Detailed formulation data are reported in Appendix B: detailed information about products and their sources are in Appendix E.

Table 2

CONDENSED DATA ON PAINT FORMULATIONS

Paint	Substrate Treatment	Binder	Pigment,	% PVC	Cure Agent	Cure Time	Cure Temp., °F
42-1	None	SR 112	Raven 40	13	SC 3963	7 days	75
42-2	None	SR 112	Raven 11	13	SC 3963	7 days	75
42-3	None	SR 112	999	10	SC 3963	7 days	75
42-4	None	SR 112	Neo Spectra Mark 2	10	SC 3963	7 days	75
42-5	None	SR 112	None		SC 3963	7 days	75
42-6	None	SR 112	Raven 40	13	SC 3963	6 hr	220
42-7	None	SR 112	Raven 11	13	SC 3963	6 hr	220
42-8	None	SR 112	999	10	SC 3963	6 hr	220
42-9	None	SR 112	Neo Spectra Mark 2	10	SC 3963	6 hr	220
42-10	None	SR 112	None		SC 3963	6 hr	220
42-11	None	SR 112	Raven 40	13	Zinc octoate	75 min	480
42-12	None	SR 112	Raven 40 + TBPS,[a] 7% w/w of resin solids	13	SC 3963	7 days	75
42-13	None	SR 112	Raven 40 + TBPS, 7% w/w of resin solids	13	SC 3963	6 hr	220
42-14	None	SR 112	Raven 40 + TBPS, 45% w/w of resin solids	13	SC 3963	7 days	75
42-15	None	SR 112	Raven 40 + TBPS, 45% w/w of resin solids	13	SC 3963	6 hr	220

Table 2 (cont.)

Paint	Substrate Treatment	Binder	Pigment,	% PVC	Cure Agent	Cure Time	Cure Temp., °F
42-20	None	BMM-80			0.5% diethanolamine	7 days	75
42-21	None	SP 49-60CX	Raven 40	10	1% DV 1308 + Castor 1066	7 days	75
42-22	None	SP 49-60CX	Raven 40	10	Castor 1066	30 min	250
42-24	None	XP 1531	Raven 40	10	Castor 1066	30 min	250
42-25	None	PSB-17	Raven 40	10	1% diethanolamine	6 hr	220
42-26	None	ST 847	Raven 40	10	Zinc octoate	30 min	350
42-27	None	SR 112	Raven 40	13	SC 3963	6 hr	220
42-28A	None	SR 112	Neo Spectra Mark 2	7.5	Zinc octoate	2 hr	475
42-28B	None	SR 112	Molacco LS	13	SC 3963	6 hr	220
42-29	None	SR 82	Molacco LS	7.5	SC 3963	6 hr	220
42-30	None	3M 101-C10				30 min	250
42-31	None	3M 301-C10				30 min	250
42-32	None	3M 401-C10			401-C10 activator	30 min	250
42-33	None	PSB-17	Raven 40	10	SC 3963	6 hr	220
42-34	None	Dalvor[b]	Neo Spectra Mark 2	5		5 min	500
42-35	None	Imron				15 min	350
42-36	None	Sicon (3X923)				30 min	400
42-37	None	Micobond (L6X962)				15 min	350
42-38	Coated with Iridite 14-2	Phenolic-epoxy	Raven 40	7	H_3PO_4	20 min	365

Table 2 (cont.)

Paint	Substrate Treatment	Binder	Pigment,	% PVC	Cure Agent	Cure Time	Cure Temp., °F
42-39	Coated with Iridite 14-2	SR 112 + TBPS, 5% w/w of resin solids	Raven 40	11	SC 3963	6 hr	220
42-40	Coated with Iridite 14-2	Epon 828	Raven 40	10	Genamid 2000	20 min	365
42-41	Coated with Iridite 14-2	SR 112	Raven 40	11	SC 3963	6 hr	220
42-42	Primed with 850-202 aluminum primer	Teflon (851-205)				5 min	750
42-43	None	Epoxy-melamine	Raven 40	10	Cymel 248-8	20 min	350
42-44	Coated with Iridite 14-2	PSB-17	Raven 40	10	SC 3963	6 hr	220
42-45	Coated with Iridite 14-2	ST 847	Raven 40	10	Zinc octoate	30 min	350
42-46	None	GRV 3171	Raven 40	10		30 min	250
42-47	None	Epon 1001	Raven 40	10	Genamid 2000	20 min	365
42-48	None	SR 112	Raven 40	10	Zinc octoate	75 min	480
42-49	None	ST 847	Raven 40	10	Zinc octoate	30 min	350
42-50	None	BKS 2600	Raven 40	10		30 min	475

Table 2 (cont.)

Paint	Substrate Treatment	Binder	Pigment,	% PVC	Cure Agent	Cure Time	Cure Temp., °F
42-51	None	SR 112	Raven 40	11	SC 3963	4 hr	300
42-52	None	SR 112	Raven 40	11	SC 3963	1 hr	480
42-53	None	SR 112	Raven 40	11	SC 3963	2 hr	350

[a] TBPS, tri-p-biphenylylphenyl silane.

[b] Pigmented by manufacturer.

B. Initial Phase I Screening

1. Thermal Shock

Test panels coated with candidate materials were thermally cycled by alternately dipping them into liquid nitrogen and boiling water to verify their adhesion to the aluminum surfaces and their film integrity under these conditions. The panel remained in each environment for 30 sec during each cycle. (The time to move the panel from liquid nitrogen to boiling water or back was negligible). The results are summarized in Table 3.

2. Abrasion Resistance

Testing of candidate coatings was intended to simulate as closely as possible the abrasive effects to be encountered by the final coating while being scrubbed or decontaminated over the 10-year period for which it is to be used in the Lewis space-simulation chamber. Of the various abrasion tests used for testing organic coatings and plastics, we considered the most suitable to be that specified in Federal Test Method Standard No. 141, Method 6191. This method uses a Gardner falling-sand abrasor and was used in testing coatings that were applied to 4-in. x 6-in. 3003 aluminum panels.

The test determines the number of liters of sand per mil of coating thickness required to abrade an area of 5/32 sq in. through to the substrate. The results of these tests are summarized in Table 4. It is customary in discussing the results of abrasion tests to include the warning that "these results are relative only, and the suitability of a particular item can be determined only by its behavior in the desired end

Table 3

THERMAL SHOCK TESTS OF PHASE I PAINTS

Paint	Total Number of Cycles	Number of Cycles to First Damage	Extent of Final Surface Damage, %
42-1	10	1	15
42-2	10	1	10
42-3	10	1	15
42-4	10	1	15
42-5	10	1	15
42-6	10	ND[a]	0
42-7	10	3	5
42-8	10	ND	0
42-9	10	ND	0
42-10	10	1	20
42-11	10	ND	0
42-12	10	ND	0
42-13	10	1	15
42-14	10	6	15
42-15	10	3	5
42-20	10	ND	0
42-21	1	1	90
42-22	10	Coating lifted intact	
42-24	10	8	35
42-25	1	ND	0
42-26	10	Coating failed	
42-27	10	ND	0
42-28A	10	2	90
42-28B	10	1	80
42-29	6	7	50
42-30	10	1	100
42-31	10	ND	0
42-32	10	ND	0
42-33	10	ND	0

Table 3 (cont.)

Paint	Total Number of Cycles	Number of Cycles to First Damage	Extent of Final Surface Damage, %
42-34	10	ND	0
42-35	10	ND	0
42-36	10	ND	0
42-37	10	ND	0
42-38	10	ND	0
42-39	10	8	5
42-40	10	ND	0
42-41	10	2	15
42-42	10	ND	0
42-43	10	ND	0
42-44	10	ND	0
42-45	10	ND	0
42-46	10	1	90
42-47	10	ND	0
42-48	10	ND	0
42-49	10	ND	0
42-50	10	ND	0
42-51	10	ND	0
42-52	10	ND	0
52-53	10	ND	0

[a] ND -- No damage.

Table 4

FALLING-SAND ABRASION AND STEAM RESISTANCE OF PHASE I PAINTS

Paint	Sand, liters/mil	Effect of 100 psig of Steam for 5 min
42-21	34	Unaffected
42-22	46	Failed
42-24	59	Unaffected
42-25	16	Slight discoloration
42-26	23	Failed
42-27	24	Failed
42-28A	30	Slight discoloration
42-28B	27	Failed
42-29	18	Failed
42-30	3	Failed after 2 min
42-31	10	Failed
42-32	31	Unaffected
42-33	41	Failed
42-34	>100	Unaffected
42-35	>100	Unaffected
42-36	18	Discolored
42-37	38	Unaffected
42-38	79	Unaffected
42-39	31	Unaffected
42-40	84	Unaffected
42-41	36	Slight discoloration
42-42	>100	Unaffected
42-43	>100	Unaffected
42-44	41	Unaffected
42-45	23	Slight discoloration
42-46	48	Unaffected
42-47	63	Unaffected
42-48	30	Unaffected
42-49	38	Failed after 3.5 min
42-50	25	Unaffected
42-51	29	Very slight discoloration
42-52	29	Slight discoloration
42-53	30	Failed after 4 min

use." With this in mind, our interpretation of the abrasion test values in Table 4 is as follows:

0 to 10 liters/mil	abrasion resistance was very poor, and the coating was unsatisfactory.
11 to 20 liters/mil	resistance was poor to fair; the coating was unsatisfactory.
21 to 50 liters/mil	resistance was fair to good.
51 to 100 liters/mil	resistance was good to excellent.
over 100 liters/mil	resistance was excellent.

3. Steam Resistance

The resistance of candidate coatings to live steam, which is one of the agents for cleaning contaminated surfaces, was measured as specified in the Work Statement by blasting with live steam at 100 psig for 5 min. In order to simulate the most stringent exposure the coatings were likely to encounter, 4-in. x 6-in. panels were supported with a constant distance of 4 in. between the steam jet and the surface of the coating.

The results of these tests are summarized in Table 4. Where "discoloration" is noted, the result observed was evidently due to small amounts of steam absorbed and retained for at least 24 hr after exposure. In most cases, this condition still prevailed 1 week after exposure. Such paint candidates were considered undesirable, primarily because of the difficulty in obtaining a very high vacuum with chamber walls whose paint still retains water vapor. It was anticipated that this result would be probable with any of the candidates that discolored during the steam resistance tests.

4. Acid Resistance

In order to simulate to some extent the effect of rocket combustion products, the surfaces of candidate specimens were wetted continuously for 6 hr with 1 M nitric acid, 1 M hydrochloric acid, and 1 M hydrofluoric acid. In addition, portions of test surfaces were also wetted continuously for 6 hr with concentrated solutions of these acids.

The results of these tests are summarized in Table 5. The ratings used in the table can be elaborated upon to some extent, as follows:

4. No visible damage to coating.
3. Visible change in coating, but no apparent breakthrough.
2. Visible damage to coating and some raising from the substrate, probably indicating acid permeation.
1. Coating penetrated or destroyed, often accompanied by spreading of the acid from the area originally covered.

C. Further Phase I Testing

1. Semiquantitative Outgassing

Since the Lewis Research Center space-simulation chamber is intended for operation at pressures as low as 10^{-8} torr and will have a large wall surface inside, the behavior of a polymeric coating for that wall under high vacuum or high vacuum-ultraviolet conditions may be of great importance.

Table 5

RATING OF PHASE I PAINTS BASED ON 6-HR ACID EXPOSURE

	Rating[a]					
	1 Molar Acids			Concentrated Acids		
Paint	HCl	HNO_3	HF	HCl	HNO_3	HF
42-1	4	3	2	2	2	1
42-2	2	2	1	2	2	1
42-3	3	4	1	2	3	1
42-4	4	3	1	3	3	1
42-5	4	2	1	3	2	1
42-6	4	3	2	4	2	1
42-7	3	4	1	1	2	1
42-8	4	3	2	3	3	1
42-9	4	3	1	2	2	1
42-10	3	2	1	3	1	1
42-11	4	2	1	3	2	1
42-12	4	4	2	3	3	1
42-13	4	4	2	4	3	1
42-14	2	2	1	2	2	1
42-15	4	4	1	4	4	1
42-20	1	1	1	1	1	1
42-21	4	2	1	3	1	1
42-22	4	3	2	3	1	1
42-24	4	1	1	1	1	1
42-25	2	3	1	1	1	1
42-26	4	4	3	4	3	2
42-27	4	3	1	2	2	1

Table 5 (cont.)

Paint	Rating[a]					
	1 Molar Acids			Concentrated Acids		
	HCl	HNO_3	HF	HCl	HNO_3	HF
42-28A	4	4	2	4	4	1
42-28B	4	3	1	3	2	1
42-29	4	4	2	4	3	2
42-30	1	1	1	1	1	1
42-31	4	3	1	2	1	1
42-32	4	1	1	1	1	1
42-33	4	3	1	3	2	1
42-34	4	4	4	4	4	4
42-35	1	1	1	1	1	1
42-36	4	3	2	4	3	3
42-37	2	2	1	2	2	1
42-38	4	2	3	4	1	3
42-39	4	3	1	3	2	1
42-40	1	1	1	1	1	1
42-41	4	3	1	2	2	1
42-42	4	4	4	4	4	4
42-43	4	2	3	3	1	1
42-44	4	3	1	1	2	1
42-45	4	4	3	4	3	2
42-46	4	3	2	3	1	1

Table 5 (cont.)

	Rating[a]					
	1 Molar Acids			Concentrated Acids		
Paint	HCl	HNO_3	HF	HCl	HNO_3	HF
42-47	4	1	1	2	1	1
42-48	3	3	1	2	2	1
42-49	4	4	3	4	3	2
42-50	4	4	1	4	4	1
42-51	4	3	1	2	2	1
42-52	4	3	1	2	2	1
42-53	4	3	1	2	2	1

[a] 4, none; 3, slight; 2, moderate; 1, severe.

Outgassing measurements were made on specimens prepared by coating circular 3003 aluminum alloy panels, 3 in. in diameter, with one of the coating candidates, drying, and curing at the temperatures noted in Table 2. The specimen was then cemented with an epoxy adhesive to a stainless steel table in the sample chamber of a small solar-simulation system used in these laboratories. A high vacuum is maintained in this system with a 400-liter/sec Varian VacIon pump and a mechanical forepump. A quartz window is sealed to a flange at the top of the chamber by means of an O ring, and an AH-6 lamp is mounted over the window. Equivalent solar factors are determined with a temperature-compensated thermopile.

With the sample table cooled by running water circulating through it, the chamber was evacuated initially with the mechanical forepump and the ion pump was then started. The rate at which the pressure fell initially within the chamber was measured to give an indication of the outgassing rate. When the pressure approached equilibrium, the AH-6 lamp was turned on and the rate of pressure change was observed. The pressure change with time was again observed after the AH-6 lamp had been turned off.

Following this procedure with each of several coating candidates, it was found, as was anticipated, that the most that could be done was to arrange them in an order of merit, based primarily on the pressure obtained after the AH-6 lamp had been on for several hours but also on the rate of initial pressure change before illumination with the AH-6 lamp and the rate of pressure change after the lamp had been turned off. The ratings of coating candidates tested are reported in Table 6.

2. Detergent Resistance

Coating candidates were tested for resistance to hot detergents, which are often used for cleaning surfaces contaminated by radioactive spillage, by exposing coated panels for 5 min to a jet of Alconox solution in water (5% w/w of Alconox) preheated to 240 to 250°F. The jet was directed under autogenous pressure against the coating surface at a distance of 1 in. No paint specimen was damaged by this exposure.

3. Alkali Metal Resistance

It is possible, although unlikely, that molten metal could escape from an advanced space power system inside the planned chamber. Therefore, at the request of NASA-Lewis Research Center, such conditions were simulated. Coatings were held at near-cryogenic temperature by immersion of one end of a bent (90°) coated aluminum substrate in liquid nitrogen, while molten potassium metal was poured on the unimmersed painted surface from a height of 1 in.

None of the coatings tested were damaged. The coatings to tested were: 42-34 (Dalvor resin), 42-42 (Teflon resin), 42-37, 42-38, 42-48, and 42-51.

4. Spectral Properties

It is evident that one of the primary requirements for the coating to be used in the Lewis space-simulation chamber is that it have a high energy absorptance over a wide wavelength span, since stray reflection can cause errors in estimating the

Table 6

RATINGS OF PHASE I PAINTS
BASED ON SEMIQUANTITATIVE OUTGASSING EXPERIMENTS

		Outgassing per unit sample area, torr-liters/(sec-cm^2)				
Paint	Time to reach initial equilibrium, min	Prior to irradiation	Highest rate during irradiation	Just before end of irradiation	After irradiation	Rating[a]
42-34	134	4.7×10^{-7}	1.8×10^{-6}	1.3×10^{-6}	3.9×10^{-7}	3
42-36	1000	1.8×10^{-7}	9.5×10^{-6}	2.7×10^{-6}	3.9×10^{-7}	1
42-37	22	1.1×10^{-6}	1.2×10^{-5}	7.7×10^{-7}	1.3×10^{-8}	2
42-38	110	5.2×10^{-7}	6.5×10^{-6}	6.5×10^{-7}	2.6×10^{-9}	1
42-39	140	8.4×10^{-7}	1.1×10^{-6}	7.8×10^{-7}	1.3×10^{-7}	2
42-41[b]	161	3.9×10^{-7}	3.4×10^{-6}	1.6×10^{-6}	3.2×10^{-7}	2
42-42	90	5.8×10^{-7}	2.3×10^{-6}	1.1×10^{-6}	1.3×10^{-7}	2
42-43	139	4.8×10^{-7}	5.2×10^{-6}	1.4×10^{-6}	4.4×10^{-7}	1
42-45	112	1.6×10^{-6}	2.1×10^{-5}	2.3×10^{-6}	3.4×10^{-7}	1
42-48	134	2.6×10^{-7}	1.0×10^{-6}	8.4×10^{-7}	7.1×10^{-8}	3

[a] 3, least likely to outgas under these conditions.
2, more likely to outgas under these conditions.
1, most likely to outgas under these conditions.

[b] Most similar in composition and cure temperature to paint 42-51, which was tested later, in Phase II.

amount of energy delivered by solar-simulation sources to components or systems being tested.

Solar absorptance was measured indirectly by measuring spectral reflectance in the wavelength range from 0.3 to 2.7 μ on a Beckman DK-2A spectrophotometer equipped with an integrating-sphere attachment, with the specimen illuminated with dispersed light at near-normal incidence. These data are integrated with solar spectral energy data for the upper atmosphere (ref. 22, 23, 25) to give normalized solar reflectance. Subtracting solar reflectance from unity gives solar absorptance. This data manipulation is performed by an IBM 7094 computer program used at IITRI.

Spectral reflectance measurements from 2.7 to 10 μ were made at room temperature on a modified Gier-Dunkle heated cavity reflectometer, again with light at near-normal incidence. The data are reported in Table 7.

5. Decontaminability

Since it is expected that nuclear devices will be tested in the Lewis Chamber, with a consequent possibility of contaminating the coating inside the chamber, any one of several decontamination methods may become necessary. These include using a vacuum cleaner, washing with water, washing with an Alconox solution, cleaning with a jet of Alconox or live steam, or literally removing the contaminated coating (ref. 21). Several methods of measuring ease of decontaminating a coated surface are in use (ref. 20, 21), and no one is yet accepted as a standard. The

Table 7

SPECTRAL PROPERTIES OF CANDIDATE PAINTS

Paint	Solar Absorptance, %	Reflectance, Percent							
		0.3 μ	0.5 μ	1.0 μ	2.0 μ	2.7 μ	5.0 μ	7.5 μ	10.0 μ
42-27	97.4	0.04	0.02	0.02	0.03	0.04			
42-28A	97.2	0.04	0.02	0.02	0.04	0.06			
42-32	98.4	0.02	0.02	0.02	0.02	0.02			
42-34	96.8	0.04	0.03	0.03	0.04	0.04	0.09	0.07	0.05
42-35	96.7	0.04	0.03	0.03	0.04	0.05			
42-36	94.7	0.06	0.04	0.05	0.07	0.10			
42-37	97.2	0.04	0.02	0.02	0.04	0.06	0.02	0.02	0.02
42-38	96.0	0.04	0.04	0.04	0.04	0.04	0.03	0.04	0.04
42-39	97.3	0.03	0.02	0.02	0.04	0.04			
42-40	96.7	0.04	0.03	0.03	0.04	0.05			
42-41	97.3	0.03	0.02	0.03	0.04	0.04			
42-42	97.0	0.04	0.02	0.03	0.03	0.04			
42-44	97.2	0.04	0.03	0.02	0.03	0.04			
42-48	96.1	0.04	0.04	0.04	0.04	0.04	0.02	0.04	0.06
42-49	97.1	0.04	0.03	0.03	0.04	0.04			
42-51	96.1	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.05

method used on this project, Kaiser Engineering Test Method 6502, is reproduced in Appendix C with their permission.

Briefly, the method requires contamination with acidic fission products of the specimens to be contaminated and of control specimens, to correct for decay of the fission products during the test. After contamination, the specimens are dried, rinsed with tap water, and dried again. After their activities have been measured, the specimens to be decontaminated are washed in a Gardner model M-105-A washability machine with a solution of 3 M nitric acid, then rinsed again, and dried. The activities of the decontaminated specimens and the controls are again measured. From the activity measurements, the decontamination factor for the coating sample can be calculated.

The fission products used were obtained by irradiating enriched uranium dioxide in the IITRI 75-kw homogeneous research reactor for periods long enough to obtain the required initial activity and then were dissolved in 1.5 N nitric acid. Measurements were made with an ionization chamber developed by Neutronics Laboratory, Tinley Park, Illinois, and a Keithley model 310 micromicroammeter. The calculated decontamination factors are summarized in Table 8. Appendix C also details the method used in calculations for decontamination factors.

D. Phase II Testing

1. Quantitative Outgassing

This set of measurements used essentially the same equipment as that used earlier for the less precise outgassing

Table 8

DECONTAMINABILITY OF PHASE I PAINTS

	Initial Activity, rem/hr		Final Activity, rem/hr		
Paint	Samples R_{1B}	Controls R_{1A}	Samples R_{2B}	Controls R_{2A}	Decontamination Factor (D.F.)[a]
42-34	240	220	110	130	1.29
42-37	170	170	85	105	1.24
42-38	560	550	180	340	1.92
42-48	740	745	130	560	4.28
42-51	115	120	40	70	1.68

[a] Examples of calculations are given in Appendix C.

measurements, except that the chamber containing the test specimen was separated from the pumping system with an orifice whose conductance was small compared with that of other elements of the system. The pressure on either side of the orifice was measured with ionization gauges and the mass flow determined by:

$$Q = U(P_2 - P_1)$$

where U is the conductance of the orifice. Since U is a function of molecular weight, a mass spectrometer residual gas analyzer was incorporated into the system so that appropriate corrections could be made. A sketch of this system is given in Figure 1.

The procedure followed was to pump on the chamber until equilibrium was reached (at a pressure of the order of 3 to 5 x 10^{-8} torr) and then to turn on the AH-6 lamp. By recording the pressures of the two ionization gauges until equilibrium was approached, a plot of outgassing rate per unit time versus time could be made, as shown in Figure 2.

Figure 2 shows a logarithmic plot of flow rate in torr-liters/sec for a 3-in.-diameter disc versus the logarithm of time from 1 min after the AH-6 lamp was turned on until 1000 min after that time, with the lamp being turned off after approximately 150 min. The coatings can be rated by measuring the area under each curve, giving a result that is proportional to the logarithm of material outgassed from t = 1 min to t = 1000 min, in liters, at the temperature and pressure of the system. In order to assign to each paint a rating that is quantitative

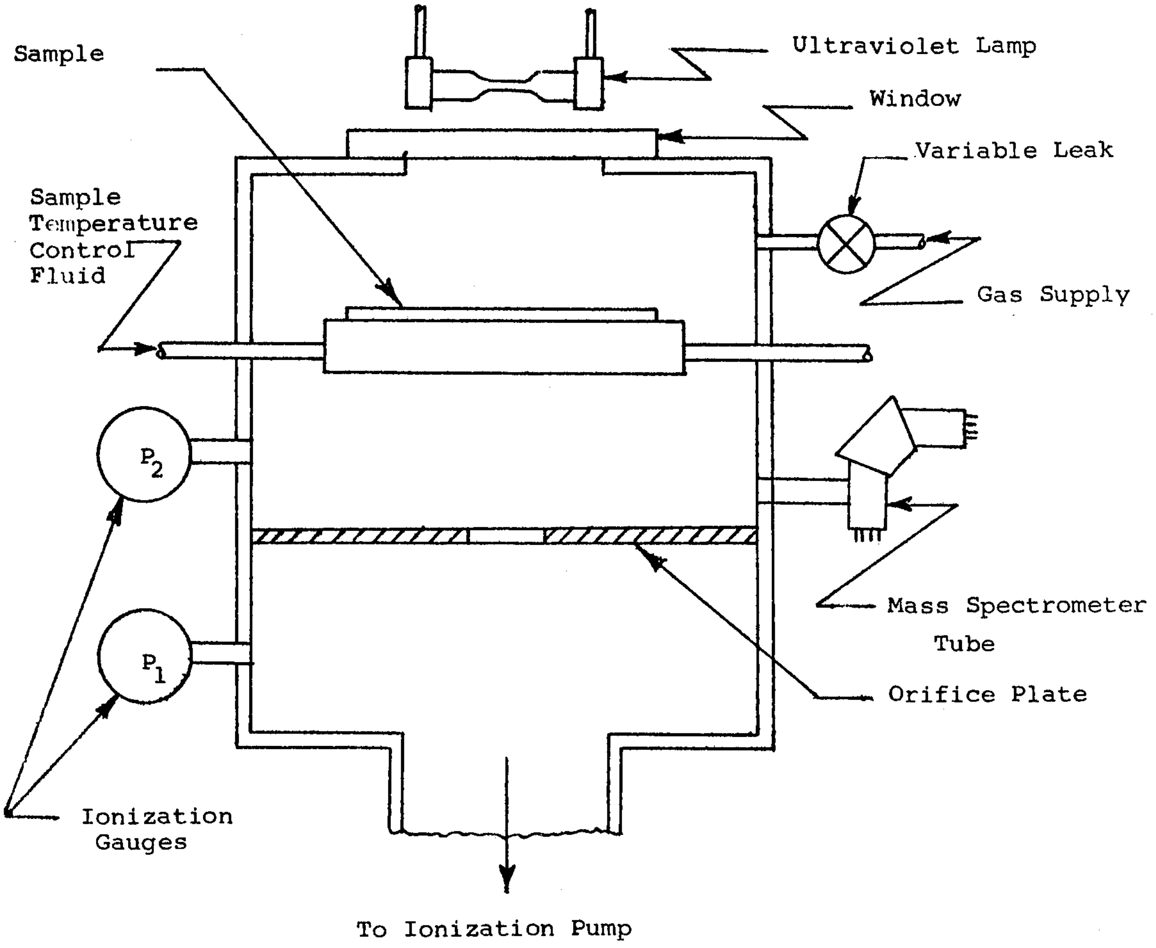

Figure 1

VACUUM CHAMBER FOR OUTGASSING STUDIES

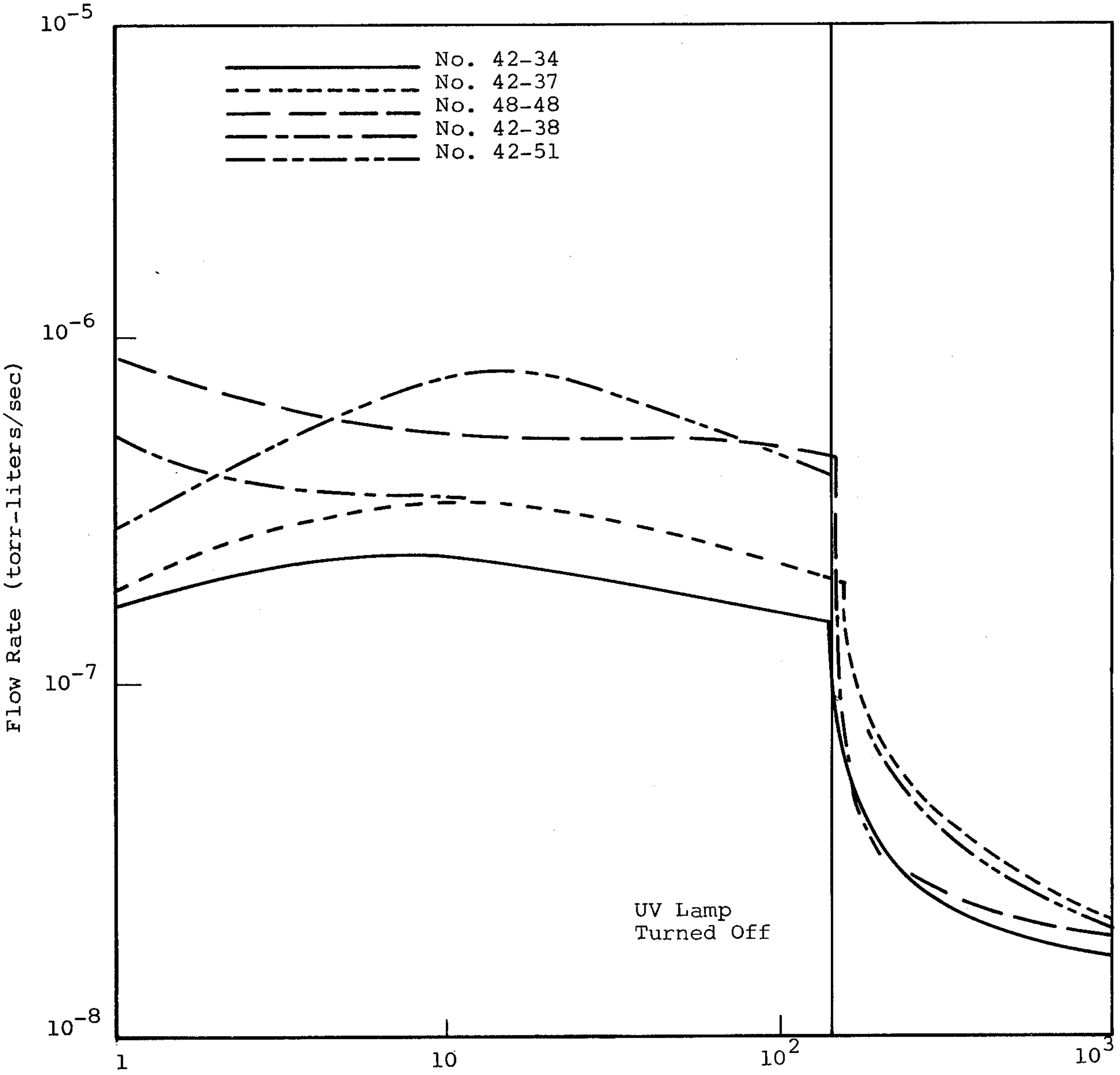

Figure 2

OUTGASSING OF CANDIDATE COATINGS

and also is highest for the most satisfactory paint, the reciprocal of the relative area under each curve is obtained and the results are normalized so that the reciprocal area of the best paint (Coating No. 42-34) equals one. The result is:

Paint	Rating
42-34	1.00
42-37	0.862
42-38	0.862
42-48	0.741
42-51	0.735

2. Normal Spectral Emittance at Room Temperature

Normal spectral emittance was calculated from spectral reflectance by using the equipment described earlier for measurement of spectral properties. Results for these coatings are reported briefly in Table 9. Reflectance curves (5 to 25 μ) are in Appendix F.

3. Total Emittance

Total hemispherical emittance was measured with a calorimetric device that used an equilibrium technique. The coating to be tested was painted and cured onto a 1-in. cube that contained a resistance heater. The thin power leads for the heater also served to support the cube. The cube was suspended inside a liquid nitrogen-cooled sphere that was coated with black paint and that was contained in a vacuum system to eliminate convective losses. Nonradiative losses from the cube were corrected for values of total hemispherical emittance for the five coatings in Phase II are given in Table 10. Total near-

Table 9

NORMAL SPECTRAL EMITTANCE OF PHASE II PAINTS

Paint	Near-Normal (15°) Emittance at 300°K			
	10 μ	15 μ	20 μ	25 μ
42-34	.95	.82	.81	.90
42-37	.98	.98	.95	.94
42-38	.96	.92	.91	.90
42-48	.94	.95	.93	.92
42-51	.95	.94	.93	.91

Table 10

TOTAL EMITTANCE OF PHASE II PAINTS

Paint	Hemispherical at 220°K	Hemispherical at 273°K	Near-Normal (15°) at 330°K
42-34	0.80	0.84	0.89
42-37	0.85	0.85	0.97
42-38	0.75	0.81	0.94
42-48	0.89	0.85	0.94
42-51	0.82	0.84	0.94

normal (15°) emittance is also presented in Table 10 for comparison purposes.

4. Solar Simulation

Samples were exposed to extraterrestrial solar simulation for 2000 equivalent sun-hours (200 hr at 10 sun intensities) in the system used previously for outgassing measurements. After irradiation, its effect on spectral properties was determined by again measuring spectral reflectance in the range from 0.35 to 2.7 μ. The results are given in Table 11. The samples were then tested for adhesion as described in Section III-8-1. All samples withstood 10 cycles with no sign of deterioration.

5. Alkali Metal Exposure

Coatings at cryogenic temperatures were exposed to molten potassium metal as described in Section III-C. None of the coatings were affected by this exposure, either at cryogenic temperatures or after they had warmed to room temperature.

6. Fast Neutron and Gamma Exposure

Since it is expected that the Lewis chamber will be used to test nuclear systems, one qualifying test for the five Phase II coatings was exposure to fast neutrons (greater than 0.1 Mev) and gamma radiation. The minimum exposures required were 2×10^{15} n/cm^2 and a gamma-ray exposure of 10^7 rads.

A fixture was constructed to permit exposure of candidate coatings to nuclear radiation while at near-cryogenic temperatures and under high vacuum. This apparatus is shown in Figure 3 as it appeared during its checkout before being placed in a port of the IITRI reactor. The exterior dimensions of the fixture

Table 11

SPECTRAL PROPERTIES OF PHASE II PAINTS AFTER SOLAR SIMULATION

Paint	Solar Absorptance, %	Solar Absorptance Change, %	Reflectance, after 2000 ESH[a], %				
			0.3 μ	0.5 μ	1.0 μ	2.0 μ	2.7 μ
42-34	96.9	0.1	2.8	3.1	3.1	3.5	4.0
42-37	96.5	-0.7	3.1	3.7	3.4	4.1	5.2
42-38	96.5	0.5	3.6	3.8	3.1	3.2	4.0
42-48	97.2	1.1	2.9	3.0	2.7	2.7	3.1
42-51	96.7	0.6	3.5	3.6	2.9	2.9	3.6

[a] E.S.H. = Equivalent Sun Hours.

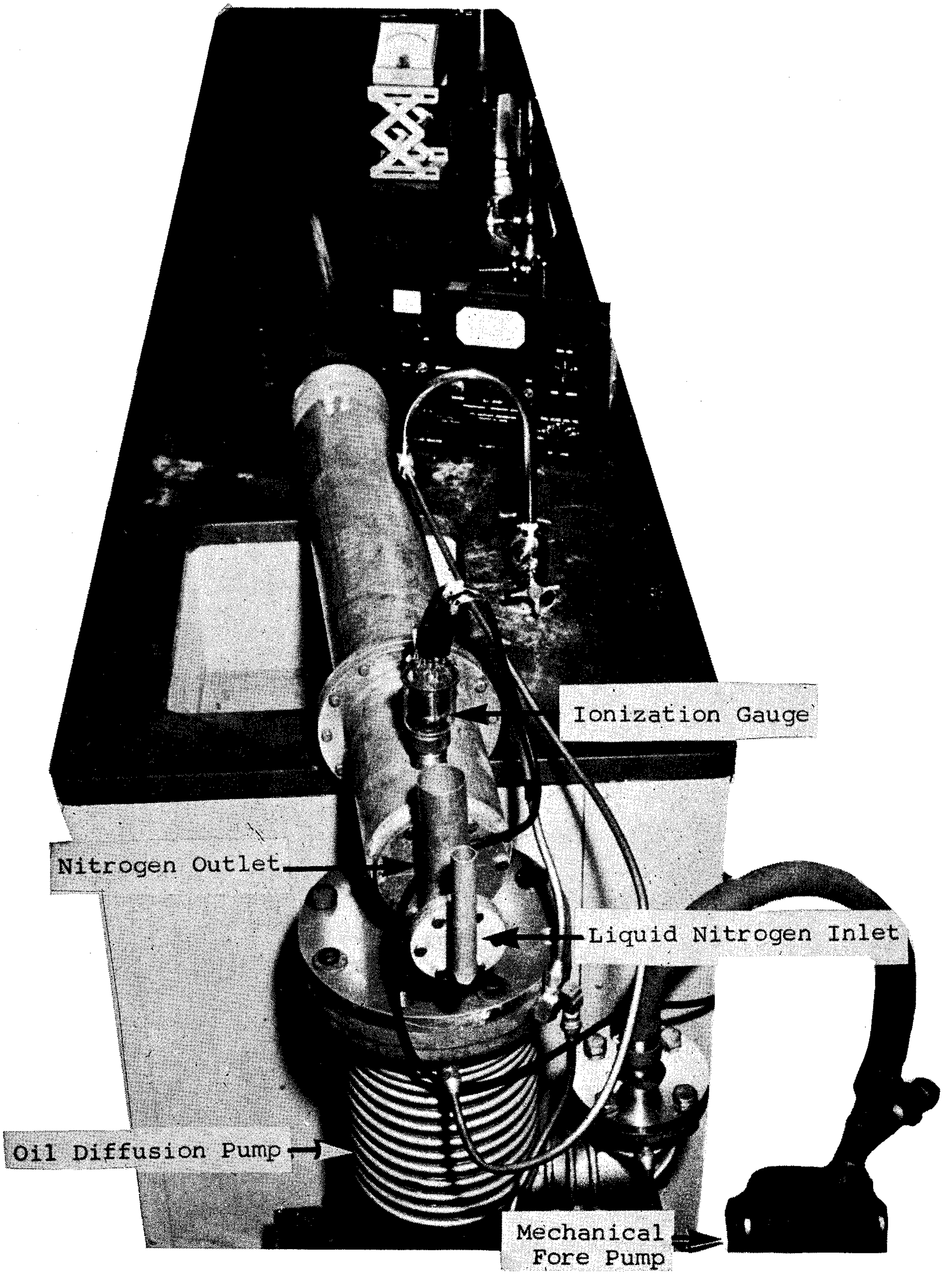

Figure 3

NUCLEAR EXPOSURE FIXTURE

were dictated largely by the geometry of the port in which the fixture was to be placed. The photograph also shows the ionization gauge used to measure pressure inside the fixture, the pumping system, and the entrance and exit tubes for a liquid nitrogen trap. The trap ran concentrically along the length of the fixture and served to cool the coating samples to low temperatures, to trap any materials caused by outgassing of the coatings, and to prevent back-streaming. Almost hidden behind the ionization gauge was a fitting through which a thermocouple entered the fixture. This copper-constantan thermocouple was clamped to the table onto which the coating specimens were bonded and provided an estimate, accurate to about 10°F, of the temperature of the coatings themselves during irradiation. Further details of this apparatus are shown in Figure 4.

Figure 4 shows a cross-sectional view of the exposure fixture at the end nearest the reactor core. In the drawing (not drawn to scale) can be seen details of the sample table, which is made of an octagonal rod unto which the coating samples were bonded with Sauereisen No. 1 cement to a thin aluminum shield at one end to prevent heat transfer from the outside wall (which was at about 120°F) of the fixture to the cold coating samples. At the other end of the sample table was an aluminum rod threaded so that the sample table could be screwed into the solid aluminum cylinder. This cylinder was, in turn, bolted by means of flanges to the outer tube carrying the liquid nitrogen coolant. Indium wire was used at both of these joints

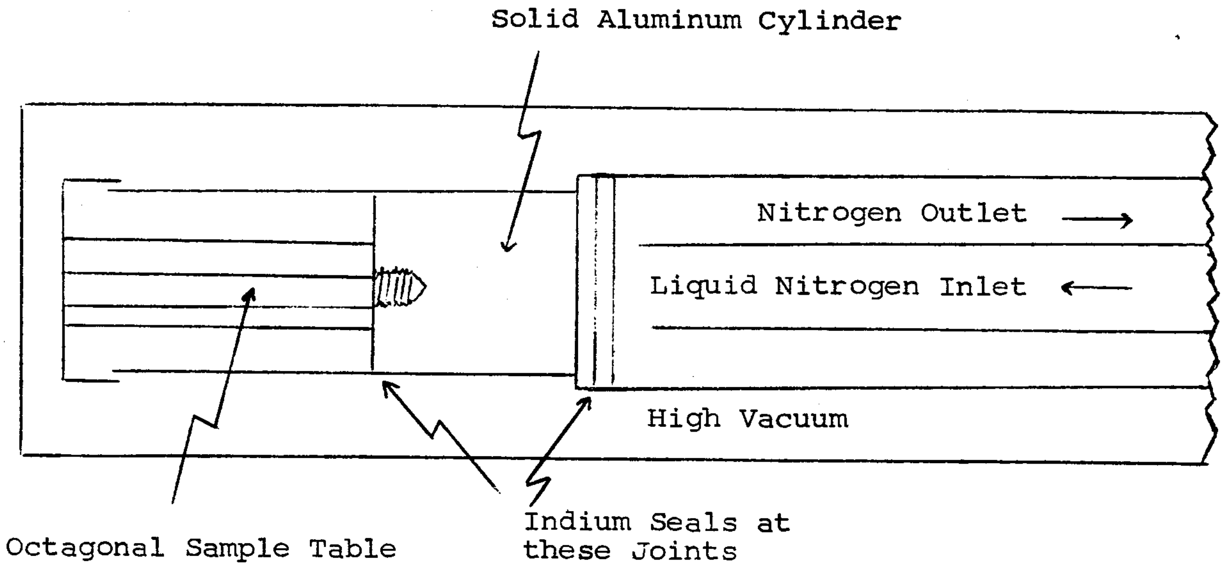

Figure 4

CROSS SECTION OF FIXTURE NEAR REACTOR CORE to ensure good thermal contact. Details not shown in this figure include a polyethylene container with three sulfur pellets attached to one of the sample table's octagonal sides and used for dosimetry and a thermocouple that was bolted to the same side of the sample table. This figure also shows a cylindrical aluminum shield welded to the solid cylinder surrounding the sample table.

The exposure fixture, with coating samples in place, was in the IITRI reactor for a total of 11 hr, with samples under high vacuum and at low temperatures. The sample table temperature ranged generally from -100 to a maximum of -235°F during a run. Pressures in the system (inside the fixture, but immediately outside the reactor port) were in the range of 10^{-6} to 10^{-7} torr. A sample table temperature 10°F above that of liquid nitrogen was attained during the laboratory check of the fixture before the run, but these lower temperatures could not be attained in the reactor partly because of the heat load caused by radiation striking the aluminum in the fixture but chiefly because of a minute leak that occurred only when liquid nitrogen was added quickly and the fitting into which the ionization gauge was inserted became quite cold. The difference in coefficient of thermal expansion between the brass fitting and the aluminum fixture surrounding it caused an opening between the two in spite of the flexible epoxy resin formula used to bond them to one another.

During the period when the reactor was at full power, 11 hr, the average neutron flux (greater than 0.1 Mev) was 6×10^{11}

n/cm^2-sec, and the gamma flux was 10^6 rads per hour: thus, the irradiated samples received a total dosage of 2.4×10^{16} n/cm^2 and 1.1×10^7 rads.

7. Tests Made after Exposure to Radiation

After the coating samples had been subjected to the irradiation just described, they were tested as required by the Work Statement as follows.

Spectral properties were measured in the wavelength region from 0.3 to 10 μ as described in Section III C. The results are shown in Table 12.

Semiquantitative outgassing measurements were attempted as described in Section III C, but the results were such that this rather limited test was unable to differentiate among outgassing characteristics of the candidate coatings. No evidence could be seen that any of the coatings had a higher outgassing rate as a result of radiation exposure.

The samples were next tested for adhesion by thermal shock, as described in Section III B, and none of the coatings were affected by ten cycles between liquid nitrogen and boiling water.

Finally, the coatings were tested for chemical stability as measured by acid resistance, decontaminability, and resistance to a jet of Alconox solution as described in Sections III B and III C. The results of the first two of these tests are reported in Tables 13 and 14, respectively. All the coatings were unaffected by a 5-min exposure to the jet of Alconox solution.

Table 12

SPECTRAL PROPERTIES OF PHASE II PAINTS AFTER NUCLEAR EXPOSURE

Paint	Solar Absorptance, %	Solar Absorptance, Change, %	Reflectance, after Nuclear Exposure, %				
			0.3 μ	0.5 μ	1.0 μ	2.0 μ	2.7 μ
42-34	96.5	-0.3	3.8	3.6	3.0	3.8	5.0
42-37	95.6	-1.6	4.6	4.7	3.7	4.1	4.4
42-38	95.9	-0.1	4.7	4.3	3.9	3.9	3.7
42-48	96.4	0.3	4.1	3.9	3.3	3.1	3.4
42-51	96.5	0.4	4.1	4.0	3.0	2.8	3.3

Table 13

ACID RESISTANCE AFTER NUCLEAR EXPOSURE

	Rating[a]					
	1 Molar Acids			Concentrated Acids		
Paint	HCl	HNO_3	HC	HCl	HNO_3	HF
42-34	4	4	4	4	4	4
42-37	2	2	1	3	2	1
42-38	4	2	3	4	2	3
42-48	4	3	1	3	2	1
42-51	4	3	1	2	2	1

[a] Rating based on 6-hr contact with acid: 4, none; 3, slight; 2, moderate; 1, severe.

Table 14

DECONTAMINABILITY OF PHASE II PAINTS AFTER NUCLEAR EXPOSURE

	Initial Activity, rem/hr		Final Activity, rem/hr		
Paint	Samples R_{1B}	Controls R_{1A}	Samples R_{2B}	Controls R_{2A}	Decontamination Factor (D.F.)
42-34	350	280	7	14	2.57
42-37	312	420	88	228	1.92
42-38	520	480	125	360	3.12
42-48	156	210	44	118	2.00
42-51	320	630	105	460	2.23

One further test, not required for these coatings, was exposure for 5 min to a jet of live steam at 100 psig. All the coatings, including formulation No. 42-51, were completely unaffected by this exposure.

Although the neutron and gamma exposures received by the coating samples were less than the maximum dosages anticipated for the coating which will ultimately be used in the Lewis chamber, the results reported here (particularly in Table 13) show a likelihood that the coatings tested will not deteriorate under higher dosages than those described here; it seems probable that the predimonant effect of radiation was additional cross-linking.

IV. DISCUSSION

A. Screening of Phase I Candidates

From the list of candidates given in Table 1, a few were not tested, for the reasons given in Appendix D. Table 15 summarizes the screening of candidate formulations tested during Phase I, describes the criterion for rejecting coatings after each test, and lists the coating formulations remaining after each test.

The results of testing coatings for adhesion by thermal shock have been given in Table 3. Twenty-seven of the original fifty-three formulations withstood this test. Coating formulations tested for acid resistance were rejected if they received a damage rating of severe with three or more of the six acid solutions after having been wetted constantly by each solution for 6 hr. This corresponds roughly to a total of twelve or more points for a coating if its numerical ratings (shown in Table 5) for all six acids are added together. Eighteen of the coatings tested remained after this criterion had been applied.

Coatings exposed to live steam at 100 psig for 5 min were rejected if they failed or showed "slight discoloration" as described in Section III. The results of this test are shown in Table 4. Eight coatings remained after these criteria were applied.

The results of testing coating candidates with the Gardner falling-sand abrasor are given in Table 4. Coatings were considered satisfactory if more than 20 liters of sand per mil of coating thickness were required to produce the specified abrasion.

Table 15

SUMMARY OF PHASE I SCREENING
OF COATING FORMULATIONS 42-1 TO 42-53

Test	Criterion	Paints Remaining
Adhesion by thermal shock	No damage after 10 cycles	42-7, 42-8, 42-11, 42-15, 42-24, 42-26, 42-30 to 42-38, 42-40, 42-42 to 42-45, 42-47 to 42-53
Acid resistance	Severely damaged by 3 or more acid solutions	42-8*, 42-11*, 42-15*, 42-26, 42-33, 42-34, 42-36 to 42-38, 42-42, 42-43, 42-45, 42-48 to 42-53
Steam blast	Failure or slight discoloration after 5 min	42-34, 42-37, 42-38, 42-42*, 42-43, 42-48, 42-40*, 42-51**
Abrasion resistance	Over 20 liters/mil coating thickness (Table 4)	42-34, 42-37, 42-38, 42-43*, 42-48, 42-51
Semiquantitative outgassing	Rating in Table 6	42-34, 42-37, 42-38, 42-48, 42-51

*See Section IV-A.

**See Section IV-B.

The six coatings shown opposite this test in Table 15 were judged to be satisfactory.

The selection criterion used for judging coatings subjected to semiquantitative outgassing measurements should be the ratings shown in Table 6. Since ratings of coatings by this test were necessarily imprecise, however, the selection of candidates remaining after this test was actually made on other grounds, which are described elsewhere in this section.

All coatings tested were judged satisfactory with regard to spectral and emittance properties and to resistance to a jet of detergent. Although some differences in decontaminability were evident, this test was not applied for screening purposes, and its results are described along with Phase II testing.

Of the 53 coating formulations tested during Phase I, those dropped from consideration as candidates for Phase II testing are indicated by an asterisk in Table 15. The reasons were as follows.

(1) Coatings 42-5 and 42-10: These were not black coatings but unpigmented formulations used as controls during adhesion and acid resistance testing.

(2) Coating 42-8: This coating,pigmented with carbon black No. 999, performed well in initial adhesion tests only part of the time. These problems might have been eliminated with closer control of formulation or application variables, but this was not necessary, since similar coatings pigmented with Raven 40 gave uniformly good results.

(3) Coating 42-11: The pigment volume concentration of this coating was reduced in order to improve adhesion; the new coating was No. 42-48.

(4) Coating 42-15: This and a few other silicone formulations contained tri-p-biphenylylphenyl-silane. It was believed that this experimental silane, developed under an Air Force contract, might serve as a nonmigrating plasticizer that would be compatible with and improve the low-temperature flexibility and adhesion, and because of its high aromatic content, the radiation stability of silicone resins. These expectations were borne out to some extent, but the amount of this costly additive required to significantly improve resin formulations would have made their cost prohibitive.

(5) Coating 42-42: This pigmented Teflon coating gave excellent results in the tests made on it. At a meeting with Mr. Lindberg and Mr. Freedman at NASA at Cleveland, it was decided that further work on this coating would be discontinued because of its high fusion temperature (750°F).

(6) Coating 42-43: This coating, which had a melamine-epoxy binder, performed as well as coating 42-38, a phenolic-epoxy, in initial tests. At the meeting in Cleveland at which Phase II candidates were selected, it was decided to retain only the latter because its high aromatic content would be expected to provide better radiation resistance.

(7) Coating 42-50: This phenolic formulation exhibited poor resistance in Phase I thermal cycling tests, and furtherwork on it was discontinued. Subsequent work on another project with panels coated with this formulation showed much improved adhesion, so the results obtained initially may have been due to variations in application techniques.

B. Qualification of a Low-Temperature-Curing Paint

With the exception of coating 42-51, all candidates ultimately chosen for Phase II Testing were completely unaffected by live-steam exposure. While this coating showed some sign of containing absorbed water 1 hr after exposure, these traces had disappeared after 24 hr. Because of the desirability of including a coating with a relatively low cure temperature, this coating was included among those tested during Phase II.

V. SUMMARY AND CONCLUSIONS

As will be seen from the conclusions that follow, we believe that the five Phase II coatings (42-34, Dalvor fluorocarbon coating; 42-37 Micobond L6X962 modified phenolic coating; 42-38, phenolic modified epoxy coating; 42-48, high-temperature curing silicone coating; and 42-51, medium-temperature curing silicone coating) are almost equal in overall performances.

The results of tests in both phases must be considered in recommending a coating for use in the NASA-Lewis space chamber. As suggested by Mr. Lindberg of the Lewis Research Center, a quantitative criterion was applied to each test, as shown in Table 16. For go no-go tests, this criterion was set arbitrarily at 1 or 0, except for the live-steam exposure, in which a paint that showed almost no effect after the test received a 0.5 rating. Since all the Phase II paints were required to have excellent optical properties, they were also rated on this basis. The same applies to retention of properties after exposure to solar or nuclear radiation.

Table 17 shows these same ratings weighted according to our own estimate of the importance of each test plus the relative frequency of exposure to the environments encountered. It cannot be overemphasized that these criteria must be subjective and do not include factors such as cure time and temperature, which must also be considered.

Table 16

QUANTITATIVE CRITERIA BASED ON TEST RESULTS

Test	Applicable Rating	Unweighted Paint Rating				
		42-34	42-37	42-38	42-48	42-51
Adhesion (Thermal shock)	1 or 0	1.00	1.00	1.00	1.00	1.00
Acid resistance	Normalized; see note a	1.00	0.42	0.71	0.50	0.54
Live-steam resistance	1 or 0.5	1.00	1.00	1.00	1.00	0.50
Abrasion resistance	1 or 0	1.00	1.00	1.00	1.00	1.00
Detergent jet	1 or 0	1.00	1.00	1.00	1.00	1.00
Alkali metal resistance	1 or 0	1.00	1.00	1.00	1.00	1.00
Spectral properties	1 or 0	1.00	1.00	1.00	1.00	1.00
Decontaminability	Normalized; see Tables 8 and 14[b]	0.61	0.50	0.80	1.00	0.62
Outgassing	Normalized; see Section III-D-1	1.00	0.86	0.86	0.74	0.74
Normal spectral emittance	See Table 9	0.89	0.97	0.94	0.94	0.94
Hemispherical total emittance	1 or 0	1.00	1.00	1.00	1.00	1.00
Solar-simulation resistance	1 or 0	1.00	1.00	1.00	1.00	1.00
Radiation-exposure	1 or 0	1.00	1.00	1.00	1.00	1.00
	Totals	12.50	11.75	12.31	12.18	11.34

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[a] Normalized data from Table 5.
Example of calculation

	Total of individual ratings	Normalized total rating
42-34	6 x 4 = 24	24/24 = 1.00
42-37	4 x 2 + 2 x 1 = 10	10/24 = 0.42

[b] Examples in Appendix C

Table 17

WEIGHTED CRITERIA FROM TABLE 16

Test	Applicable Rating	Weighting Factor	Weighted Paint Rating 42-34	42-37	42-38	42-48	42-51
Adhesion (thermal shock)	1 or 0	0.8	0.80	0.80	0.80	0.80	0.80
Acid resistance	Normalized; see note a	0.8	0.80	0.34	0.57	0.40	0.43
Steam	1 or 0.5	0.7	0.70	0.70	0.70	0.70	0.35
Abrasion	1 or 0	0.9	0.90	0.90	0.90	0.90	0.90
Detergent jet	1 or 0	0.7	0.70	0.70	0.70	0.70	0.70
Alkali metal	1 or 0	0.3	0.30	0.30	0.30	0.30	0.30
Spectral properties	1 or 0	0.9	0.90	0.90	0.90	0.90	0.90
Decontaminability	Normalized; see Tables 8 and 14[b]	0.6	0.37	0.30	0.48	0.60	0.37
Outgassing	See Section III-D-1	0.9	0.90	0.77	0.77	0.67	0.67
Normal spectral emittance	See Table 9	0.9	0.80	0.87	0.85	0.85	0.85
Hemispherical total emittance	1 or 0	0.9	0.90	0.90	0.90	0.90	0.90
Solar-simulation resistance	1 or 0	1.0	1.00	1.00	1.00	1.00	1.00
Radiation-exposure	1 or 0		1.00	1.00	1.00	1.00	1.00
		Total	10.07	9.48	9.87	9.72	9.17

[a] Normalized data from Table 5.
Example of calculation

	Total of individual ratings	Normalized total rating
42-34	6 x 4 = 24	24/24 = 1.00
42-37	4 x 2 + 2 x 1 = 10	10/24 = 0.42

[b] Examples in Appendix C

It is evident that, in our estimation, the Dalvor resin is superior to the other Phase II candidates if the heat required to fuse it can be attained. When all required test criteria are considered, the five Phase II coatings do not differ greatly in their overall suitability. It may well be that more than one paint should be used in different parts of the chamber, and we suggest this as an important option to consider.

One system not tested as a Phase II coating might be considered for at least limited use, namely a silicone paint with SC3963 catalyst cured for at least 7-14 days at room temperature. Such a coating might be expected to have good resistance to simulated solar exposure, detergent cleaning and nuclear radiation. Its drawbacks, however, would probably be in acid resistance and outgassing, and possibly in abrasion resistance.

APPENDIX A

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APPENDIX B

DETAILS OF COATING FORMULATIONS

Number	Type of Binder	Ingredient	Wt. %	Baking Time and Temp.
42-1	Silicone	SR 112	62.5	7 days @ 75°F
		Raven 40	7.2	
		SC 3963	1.6	
		n-Butanol	6.2	
		Xylene	22.5	
42-2	Silicone	SR 112	62.5	7 days @ 75°F
		Raven 11	7.2	
		SC 3963	1.6	
		n-Butanol	6.2	
		Xylene	22.5	
42-3	Silicone	SR 112	65.1	7 days @ 75°F
		No. 999	5.5	
		SC 3963	1.6	
		n-Butanol	6.5	
		Xylene	21.3	
42-4	Silicone	SR 112	65.1	7 days @ 75°F
		Neo Spectra Mark 2	5.5	
		SC 3963	1.6	
		n-Butanol	6.5	
		Xylene	21.3	
42-5	Silicone	SR 112	70.5	7 days @ 75°F
		SC 3963	1.7	
		n-Butanol	6.5	
		Xylene	21.3	
42-6	Silicone	SR 112	62.5	6 hr @ 220°F
		Raven 40	7.2	
		SC 3963	1.6	
		n-Butanol	6.2	
		Xylene	22.5	
42-7	Silicone	SR 112	62.5	6 hr @ 220°F
		Raven 11	7.2	
		SC 3963	1.6	
		n-Butanol	6.2	
		Xylene	22.5	

APPENDIX B (cont.)

Coating Number	Type of Binder	Ingredient	Wt. %	Baking Time and Temp.
42-8	Silicone	SR 112	65.1	6 hrs @ 220°F
		No. 999	5.5	
		SC 3963	1.6	
		n-Butanol	6.5	
		Xylene	21.3	
42-9	Silicone	SR 112	65.1	6 hrs @ 220°F
		Neo Spectra Mark 2	5.5	
		n-Butanol	6.5	
		SC 3963	1.6	
		Xylene	21.3	
42-10	Silicone	SR 112	70.5	6 hrs @ 220°F
		SC 3963	1.7	
		n-Butanol	6.5	
		Xylene	21.3	
42-11	Silicone	SR 112	61.5	75 min @ 480°F
		Raven 40	7.0	
		Zinc octoate	1.6	
		Xylene	29.9	
42-12	Silicone	SR 112	60.0	7 days @ 75°F
		TBPS[a]	4.2	
		Raven 40	6.8	
		SC 3963	1.5	
		n-Butanol	6.0	
		Xylene	21.5	
42-13	Silicone	SR 112	60.0	6 hrs @ 220°F
		TBPS	4.2	
		Raven 40	6.8	
		SC 3963	1.5	
		n-Butanol	6.0	
		Xylene	21.5	
42-14	Silicone	SR 112	47.4	7 days @ 75°F
		TBPS	21.3	
		Raven 40	5.6	
		SC 3963	1.2	
		n-Butanol	4.8	
		Xylene	19.7	

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[a] Tri-p-biphenylylphenylsilane

APPENDIX B (cont.)

Coating Number	Type of Binder	Ingredient	Wt. %	Baking Time and Temp.
42-15	Silicone	SR 112	47.4	6 hrs @ 220°F
		TBPS	21.3	
		Raven 40	5.6	
		SC 3963	1.2	
		n-Butanol	4.8	
		Xylene	19.7	
42-20	Silicone-alkyd	BMM-80	66.3	7 days @ 75°F
		Raven 40	5.6	
		Diethanolamine	0.4	
		Xylene	27.7	
42-21	Polyurethane	SP 49-60CX	49.0	7 days @ 75°F
		Castor 1066	31.2	
		DV 1308	0.5	
		Raven 40	10.1	
		Ethyl acetate	4.6	
		Xylene	4.6	
42-22	Polyurethane	SP 49-60CX	49.2	30 min @ 250°F
		Castor 1066	31.4	
		Raven 40	10.2	
		Ethyl acetate	4.6	
		Xylene	4.6	
42-24	Polyurethane	XP-1531	37.7	30 min @ 250
		Castor 1066	12.2	
		Raven 40	4.9	
		Ethyl acetate	22.6	
		Xylene	22.6	
42-25	Silicone-alkyd	PSB-17	66.3	6 hrs @ 220°F
		Raven 40	5.6	
		Diethanolamine	0.4	
		Xylene	27.7	
42-26	Silicone-epoxy	ST 847	62.1	30 min @ 350°F
		Raven 40	5.9	
		Zinc octoate	0.4	
		Xylene	31.6	
42-27	Silicone	SR 112	62.5	6 hrs @ 220°F
		Raven 40	7.2	
		SC 3963	1.6	
		n-Butanol	6.2	
		Xylene	22.5	

APPENDIX B (cont.)

Coating Number	Type of Binder	Ingredient	Wt. %	Baking Time and Temp.
42-48A	Silicone	SR 112	67.1	2 hrs @ 475°F
		Neo Spectra Mark 2	4.3	
		Zinc octoate	0.5	
		Xylene	28.1	
42-28B	Silicone	SR 112	62.5	6 hrs @ 220°F
		Molacco LS	7.2	
		SC 3963	1.6	
		n-Butanol	6.2	
		Xylene	22.5	
42-29	Silicone	SR 82	54.6	6 hrs @ 220°F
		Molacco LS	3.8	
		SC 3963	1.4	
		n-Butanol	5.5	
		Xylene	34.7	
42-30	Proprietary	Velvet coating 101-C10		30 min @ 250°F
42-31	Proprietary	Velvet coating 301-C10		30 min @ 250°F
42-32	Proprietary	Velvet coating 401-C10		30 min @ 400°F
42-33	Silicone-alkyd	PSB-17	61.6	30 min @ 400°F
		Raven 40	5.4	
		SC 3963	1.6	
		Xylene	31.4	
42-34	Polyvinyl fluoride	Dalvor resin (pigmented with Neo Spectra Mark 2 5% PVC)		5 min @ 500°F
42-35	Polyurethane	Imron		15 min @ 500°F
42-36	Silicone	Sicon black 3X923		30 min @ 400°F
42-37	Vinyl-modified phenolic	Micobond L6X962		30 min @ 350°F
42-38[b]	Phenolic-modified epoxy	Epon 1009	47.8	20 min @ 365°F
		Raven 40	4.6	
		SR 82[c]	4.8	
		Diacetone alcohol	15.6	
		GE 75108 resin	12.8	
		Phosphoric acid[d]	3.3	
		Solvents[e]	11.1	

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[b] Complete data on preparing this mixed resin are given in ref. 19.

[c] 1% solution in xylene, based on resin solids.

[d] 10% w/w in diacetone alcohol.

[e] Mixed diacetone alcohol, xylene, denatured ethanol, and toluene.

APPENDIX B (cont.)

Coating Number	Type of Binder	Ingredient	Wt. %	Baking Time and Temp.
42-39	Silicone	SR 112	58.0	6 hrs @ 220°F
		Raven 40	5.9	
		TBPS[f]	2.9	
		SC 3963	1.5	
		n-Butanol	5.8	
		Xylene	25.9	
42-40	Epoxy	Epon 828	18.0	20 min @ 365°F
		Versamid 2000	18.0	
		Raven 40	6.8	
		Xylene	29.7	
		Methyl ethyl ketone	27.5	
42-41	Silicone	SR 112	62.8	6 hrs @ 220°F
		Raven 40	6.0	
		SC 3963	1.6	
		n-Butanol	6.3	
		Xylene	23.3	
42-42	Poly(tetrafluoro-ethylene)	Teflon 851-205		5 min @ 750°F
42-43[g]	Epoxy-melamine	Epon 1001	32.4	20 min @ 350°F
		Raven 40	4.9	
		Cellosolve acetate	17.8	
		Xylene	17.8	
		Cymel 248-8	23.9	
		Cellosolve acetate	1.6	
		Xylene	1.6	
42-44[h]	Silicone-alkyd			
42-45	Silicone-epoxy	ST 847	62.1	30 min @ 350°F
		Raven 40	5.9	
		Zinc octoate	0.4	
		Xylene	31.6	

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[f] Tri-p-biphenylyl phenyl silane

[g] Method of preparation similar to that used for coating No. 42-38 See ref. 19.

[h] Identical to coating No. 42-33.

APPENDIX B (cont.)

Coating Number	Type of Binder	Ingredient	Wt. %	Baking Time and Temp.
42-46	Styrenated phthalate alkyd	GRV 3171		
		Raven 40		
42-47	Epoxy	Epon 1001	31.2	30 min @ 350°F
		Genamid 2000	10.9	
		Raven 40	4.8	
		Xylene	26.9	
		Methyl ethyl ketone	10.6	
		Cellosolve acetate	15.6	
42-48	Silicone	SR 112	66.2	75 min @ 480°F
		Raven 40	5.4	
		Zinc octoate	0.5	
		Xylene	27.9	
42-49[i]	Silicone-epoxy			
42-50	Phenolic	BKS 2600	37.2	30 min @ 475°F
		Raven 40	5.3	
		Ethanol	56.5	
42-51	Silicone	SR 112	62.8	4 hrs @ 300°F
		Raven 40	6.0	
		SC 3963	1.6	
		n-Butanol	6.3	
		Xylene	23.3	
42-52	Silicone	SR 112	62.8	60 min @ 480°F
		Raven 40	6.0	
		SC 3963	1.6	
		n-Butanol	6.3	
		Xylene	23.3	
42-53	Silicone	SR 112	62.8	2 hrs @ 350°F
		Raven 40	6.0	
		SC 3963	1.6	
		n-Butanol	6.3	
		Xylene	23.3	

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[i] Identical to coating No. 42-45

APPENDIX C

KAISER ENGINEERING TEST METHOD 6502

A. Detailed Test Method

1. Scope

This method covers a procedure for determining the decontaminability of organic and inorganic coating materials by removing radiochemical activity from the surface by standard decontamination procedures.

2. Apparatus

The apparatus shall consist of the following equipment and accessories as required for personnel protection:

2.1 Remote Servo-controlled pipetter, ORNL model Q-1348.

2.2 Ionization chamber and electrometer, ORNL model Q-1102B. This instrument shall be calibrated against radium to detect radiation to ±10% and for conversion of its readings in microamperes to millirems per hour.

2.3 Gardner wear testing machine, model 1725, equipped with a brush with Chinese hog bristles, or approximately 4-1/2 sq. in. surface area, weighted to exert a total pressure of 1 lb on the surface being scrubbed.

3. Reagents

3.1 The contaminant is a radioactive solution, 1.0 to 2.5 N-H^+, containing fission products capable of producing radiation on the panels after procedure 4.5 in the range of 1×10^2 to 3×10^3 rem/hr/0.2 mil of solution. A particular contaminant complying hereto may be specified.

3.2 The acid rinse is an aqueous nitric acid solution, 3 M HNO_3.

4. Procedure

4.1 Four steel panels prepared in accordance with method 2011 shall be coated on all surfaces as specified. Panels shall be marked A, B, C and D, respectively.

4.2 Contaminate each panel with 200 ml of contaminant using a remote Servo-controlled pipetter.

4.3 Dry contaminant at a temperature of 75 to 85°F in air circulating at about 50 cfm over surfaces of the panels.

4.4 Uniformly flush each panel with tap water for 2 min. Do not allow jet effect of water to aid in removal of contaminant.

4.5 Dry the panels as in 4.3.

4.6 Measure and record, as R_{1A}, R_{1B}, R_{1C}, and R_{1D}, the remaining activity on each of the four panels, in millirem/hr, using the ionization chamber and electrometer. Record the time of the measurement.

4.7 Decontaminate panels B and C in fresh acid rinse using the Gardner wear testing machine and scrubbing for 370 passes of the brush. During brushing maintain 1/16 in. minimum acid rinse on panel surfaces.

4.8 Rinse and dry each panel as in 4.4 and 4.5.

4.9 Measure, as close to simultaneously as practical, the remaining activity on each of the four panels and record the results as R_{2A}, R_{2B}, R_{2C}, and R_{2D}, respectively.

4.10 Determine natural decay from formulas 1, 2, and 3:

$$D_A = R_{1A} - R_{2A} \tag{1}$$

$$D_D = R_{1D} - R_{2D} \tag{2}$$

$$D_{Avg} = \frac{D_A + D_D}{2} \tag{3}$$

4.11 Determine decontamination factors for the decontaminated panels from formulas 4 and 5:

$$DF_B = \frac{R_{1B} - D_{Avg}}{R_{2B}} \tag{4}$$

$$DF_C = \frac{R_{1C} - D_{Avg}}{R_{2C}} \tag{5}$$

4.12 Determine and report the overall decontamination factor for the material from formula 6:

$$DF_{Avg} = \frac{DF_B + DF_C}{2} \tag{6}$$

B. Examples of Calculations

The procedure actually used in calculating the decontamination factors shown in Tables 8 and 14 was modified in one respect from that given in the test method. The decay correction factor used was not equal to $(R_{1A} - R_{2A})$, but, instead:

$$D = \frac{R_{1B}}{R_{1A}} (R_{1A} - R_{2A})$$

The rationale for this correction stems from the fact that the initial readings, R_{1A} and R_{1B}, were taken almost simultaneously, while the time between taking the first reading (R_{1A}) and the second (R_{2A}) on the control sample was relatively long (and long compared to the decay rate). Therefore, any differences in the initial reading on sample and control were ascribed to differences in degree of contamination rather than time-related decay. An example of these calculations follows:

R_{1B} = 240 rem/hr
R_{1A} = 220 rem/hr
R_{2B} = 110 rem/hr
R_{2A} = 130 rem/hr

$$D = \frac{240}{220} (220-130) = \frac{240}{220} \times 90 = 98$$

$$DF = \frac{240 - 98}{110} = \frac{142}{110} = 1.29$$

The procedure used to get the normalized ratings of Tables 16 and 17 from the decontamination factors in Tables 8 and 14 is shown here:

	Total of DF from Tables 8 and 14	Normalized Rating
42-48[a]	4.28 + 2.00 = 6.28	6.28/6/28 = 1.00
42-34	1.29 + 2.57 = 3.86	3.86/6.28 = 0.61

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[a] Best paint with respect to this test.

APPENDIX D

JUSTIFICATION OF CANDIDATE COATINGS

Sections I through IV of this part of this appendix are essentially identical with a communication submitted to Mr. R. A. Lindberg of the National Aeronautics ans Space Administration, on March 4, 1965, justifying inclusion of candidate coatings on this project. It is based on available literature and preliminary work on the project. Section V describes some modifications in the list suggested by NASA personnel.

I. Coatings Cited in Work Statement

(1) 3M-Velvet coating 101-C10 optical Black.

Alkyd binder. Flexibility relatively poor (ref. 1). Nuclear and ultraviolet performance probably marginal.

(2) Glidden Missile Black Mil-E-10687B. Glidden Test reference 7353. Government black No. 37038 (3725). Mfgr. Rgl. 22818.

(3) Krylon black.

Acrylic binder. Poor radiation resistance (ref. 2). Reported to suffer physical as well as optical damage when exposed to ultraviolet (ref. 3).

(4) Parsons optical black.

Composition unknown. Acutely nonresistant to mechanical shock and abrasion (3).

(5) Kemacryl M49BC12

Acrylic binder. Damaged by nuclear and ultraviolet radiation (ref 2,3).

(6) Micobond black L6X962

Vinyl-modified phenolic.

(7) 10043 aluminum silicone.

This is a flat reflector and does not meet the optical requirements of the work statement (ref. 4)

(8) Fuller 171A152.

This is a flat reflector and does not meet the optical requirements of the work statement (ref. 4).

(9) Modified DuPont PR 229.

Composition and vendor unknown to DuPont.

(10) Sicon Black 3X923.

Proprietary silicone coating.

II. Discussion of Available Literature

A successful coating for use in the NASA-Lewis space simulation chamber should have optimum resistance to several environments. The most important are probably resistance to nuclear and ultraviolet radiation in vacuo and to vacuum volatilization. Since the walls of the chamber will not operate at temperatures much above 150.F, vacuum volatilization should be a relatively minor problem. It has been reported that vacuum losses of some organic coatings can be severe over long periods even at room temperature, although silicone resins seem quite resistant (ref. 5). Test data are scarce, however, and one source comments that "no rigid conclusions can be drawn which would enable even types of coating polymers to be placed in an order of merit for resistance to vacuum volatilization" (ref. 6).

With regard to ultraviolet stability, much work has been done, but the greatest emphasis has been on optical rather than physical changes (ref. 7). The organic coating materials most widely considered for spacecraft thermal control have been silicone resins, silicone-alkyds, and polyurethanes. Although acrylics show relatively little change in optical properties, there is evidence of slow physical degradation in ultraviolet-vacuum environments (ref. 7,8).

Considerable work has been done on studying physical changes of polymers in nuclear environments (for example, ref. 6,7,9,10) and on mechanisms of nuclear degradation of polymers (ref. 11, 12, 15). Little work has been reported, however, on nuclear irradiation of polymeric coatings. One source (ref. 13) reports coating irradiation in air and another the irradiation of coatings in vacuo (ref. 2). Ref. 13 lists the coatings tested in air, in order of decreasing stability, as phenolic, silicone-alkyd, alkyd, epoxy, chlorotrifluororethylene-vinylidine fluoride copolymer, and nitrocellulose. We concur that black pigments seem to improve radiation stability. Ref. 2 reports the order of coatings irradiated in vacuo as (1) inorganic silicates, (2) silicone resins, (3) epoxy resins, and (4) acrylic resins. Both studies were made on proprietary coatings.

In the absence of extensive studies on coatings themselves, the most useful data in the literature seem to be those on susceptibility to damage of various organic linkages (ref. 12, 14). For organic polymers, such studies indicate the desirability of high aromatic content, low unsaturation, and lack of terminal unsaturation, and point up the decreasing stability of the following groupings: alkanes $>$ ethers $>$ alcohols $>$ esters $>$ ketones.

An additional consideration is the difference in radiative stability sometimes found when polymers are irradiated in vacuum rather than in air. For example, DuPont researchers have reported Teflon to be more stable when irradiated in vacuum.

Additional documentation concerning the stability of siloxanes to nuclear radiation is found in ref. 16, in which very low g_{gas} values for phenyl siloxanes are reported.

Information concerning the radiation stability of polyurethane coatings to nuclear radiation is scarce, but a urethane adhesive is reported satisfactory when irradiated at cryogenic temperatures (ref. 7), and certain polyurethane elastomers are reported to resist nuclear degradation (ref. 17). Irradiation in vacuo is reported to increase the ratio of cross-linking to chain scission of some polyurethanes (ref. 18). From ref. 14, it seems likely that a polyether-based urethane formulation would be more stable than one using a polyester.

There is evidence (ref. 13,15) that carbon black, because of its pseudoaromatic nature, can improve considerably the radiation stability of coatings as well as their optical and physical properties. For this reason, we believe it very important to use carbon black pigments of various concentrations, particle diameters, and surface characteristics in our coating candidates.

III. Additional Candidate Coatings

(1) 3M Velvet coatings.

Data about the resistance of these coatings to chemicals and radiation resistance have not been published, but their superior optical and high vacuum performance should justify studying not only No. 101-C10 but also black coatings 301-C10 (a baking enamel) and 401-C10 (a two-part coating system). All are commercially available.

(2) Silicone-Alkyd coating.

The Sherwin-Williams coating is a special room- or low-temperature-curing silicone-alkyd. In view of the radiation stability of the silicone-alkyd tested by Horrocks (ref. 13), this coating is included.

(3) SR 112 and SR 82 silicone resins.

On the basis of nuclear, ultraviolet, and vacuum stability, along with their other physical and chemical properties, silicone resins show considerable promise. The series of silicone resins listed in Table 18 includes desirable variations in aromatic content (amount of substitutent phenyl groups), type of pigment, and type of catalyst.

(4) Polyurethanes.

The considerations discussed in Section II above were the basis for including three urethane coatings, DuPont's polyester-based Imron and the Spencer-Kellogg polyether-based coatings.

(5) Miscellaneous.

Epoxy and phenolic resins have the disadvantages of radiation-induced outgassing and high cure temperature, respectively. However, it is very important that these be included to serve as chemical-resistant controls for comparison with the modified resins listed as items 27, 29, and 30 in Table 18. Item 27 is commercially available; the epoxy-modified phenolic and melamine resins were prepared at IIT Research Institute. Coatings 26 to 30 in Table 18 were pigmented with Raven 40.

IV. Potential Coatings No Included

Several chemically resistant materials that might meet the nuclear radiation resistance requirments of the Work Statement were not suggested as potential candidates. These include polytetrafluoroethylene (Teflon), polychlorotrifluorethylene (Kel-F), and Dalvor fluorocarbon polymer dispersion. All require extremely high temperature for fusion, in the neighborhood of 500°F or higher.

Table 18

PROPOSED CANDIDATE COATINGS NOT IN WORK STATEMENT

Coating No.	Description
11	3M velvet coating 301-C10
12	3M velvet coating 401-C10
13	Sherwin-Williams experimental silicone-alkyd BMM-80 or equivalent
14	GE SR112 silicone resin, Raven 40 pigment, SC 3963[a]
15	GE SR112, Raven 11 pigment, SC 3963
16	GE SR112, No. 999 pigment, SC 3963
17	GE SR112, Neo Spectra pigment, SC 3963
18	GE SR112, Molacco H pigment, SC 3963
19	GE SR112, Raven 40 pigment, TBPS[b], SC 3963
20	GE SR112, Raven 40 pigment, zinc octoate catalyst
21	GE SR82 silicone resin, Raven 40 pigment, SC 3963
22	GE SR82, Molacco H pigment, SC 3963
23	Du Pont Imron polyurethane coating, Raven 40
24	Spencer-Kellogg Spenko 49-60CX, Raven 40 pigment
25	Spencer-Kellogg Spenko XP1531, Raven 40 pigment
26	Epoxy-pigmented Jones & Dabney Epi-Rez (or equivalent)
27	Silicone-modified epoxy, Plaskon ST847
28	Phenolic resin, GE Methylon 75108 (or equivalent)
29	Epoxy-modified phenolic resin
30	Epoxy-modified melamine or triazine resin

[a] Low temperature curing catalyst.

[b] Tri-p-biphenylyl phenyl silane additive (Weston Chem. Div.)

V. FURTHER COMMENTS

Several of the coatings listed in this appendix have curing temperatures that would make application difficult, since a cure temperature of about 250°F or below would be most desirable. Coating candidates 20, 26 and 20, for example, would have considerably higher cure temperature.

NASA personnel requested that two other high-temperature materials, Dalvor and Teflon fluorocarbon dispersions, be added to the list of candidate coatings. It is believed that the stringent chemical stability requirements that the coating must meet make the consideration of these fluorocarbons advisable. We suggested that these be substituted for items 16 and 21 in Table 18.

APPENDIX E

INFORMATION ABOUT MATERIALS USED

Name	Descriptive Data	Source
BKS 2600	Phenolic resin	Union Carbide Plastics Co.
BMS-80	Experimental silicone-alkyd resin	Sherwin-Williams
n-Butanol	1-Butyl alcohol	Fisher Scientific Co.
Castor 1066	Castor oil	Spencer Kellogg Div, Textron, Inc.
Cellosolve acetate	2-ethoxy-ethyl acetate	Union Carbide Chemical Co.
Cymel 248-8	Melamine resin	American Cyanamid Co.
Dalvor resin	Pigmented polyvinyl fluoride dispersion	Diamond Alkali Co.
Diacetone alcohol	4-Hydroxy-4-methyl-2-pentanone	Distillation Products Industries
Diethanolamine	Diethanolamine	E. H. Sargent and Co.
DV 1308	Urethane cure accelerator	Spencer Kellogg Div. Textron, Inc.
Epon 828	Epoxy resin	Shell Chemical Co.
Epon 1001	Epoxy resin	Shell Chemical Co.
Epon 1009	Epoxy resin	Shell Chemical Co.
Ethanol	Ethyl alcohol, 95 percent	----
Ethyl acetate	Ethyl acetate	Fisher Scientific Co.
Genamid 2000	Urethane curing agent	General Mills
GRV 3171	Styrenated phthalate-alkyd resin	The Glidden Co.
Imron	Pigmented polyurethane enamel	E.I. Du Pont de Nemours & Co., Inc.
Methyl ethyl ketone	Methyl ethyl ketone	Fisher Scientific Co.
Methylon 75108	Phenolic resin intermediate	General Electric Co.
Micobond L6X962	Pigmented vinyl-modified phenolic coating	Midland Industrial Finishes Co.
Molacco LS	Carbon black, ink grade	Columbian Carbon Co.
Neo Spectra Mark 2	Carbon black, paint grade	Columbian Carbon Co.
No. 999	Carbon black, paint grade	Columbian Carbon Co.
Phosphoric acid	Phosphoric acid	Fisher Scientific Co.

APPENDIX E (Cont.)

Name	Descriptive Data	Source
PSB-17	Experimental silicone-alkyd resin	Sherwin-Williams
Raven 11	Carbon black	Columbian Carbon Co.
Raven 40	Carbon black	Columbian Carbon Co.
SC 3963	Room-or-low temperature curing agent	General Electric Co.
Sicon black 3X923	Pigmented silicone resin	Midland Industrial Finishes Co.
SP49-60CX	Urethane resin prepolymer	Spencer Kellogg Div, Textron, Inc.
SR 82	Methyl-phenyl silicone resin	General Electric Co.
SR 112	Methyl-phenyl silicone resin	General Electric Co.
ST 847	Silicone-epoxy resin	Allied Chemical Co.
TBPS	Tri-p-biphenylyl phenyl silane	Stauffer Chemical Co. Anderson Chemical Div.
Teflon 851-205	Pigmented tetrafluoroethylene enamel	E. I. Du Pont de Nemours & Co., Inc.
Toluene	Toluene	E. H. Sargent & Co.
XP 1531	Urethane resin prepolymer	Spencer Kellogg, Div. Textron, Inc.
Xylene	Xylene	E. H. Sargent & Co.
Velvet coating 101-C10	Proprietary	Minnesota Mining and Manufacturing Co.
Velvet coating 301-C10	Proprietary	Minnesota Mining and Manufacturing Co.
Velvet coating 401-C10	Proprietary	Minnesota Mining and Manufacturing Co.
Zinc octoate	Zinc octoate	Heydon Newport Chemical Corp. Nuodex Products Div.

APPENDIX F

REFLECTANCE OF PHASE II PAINTS

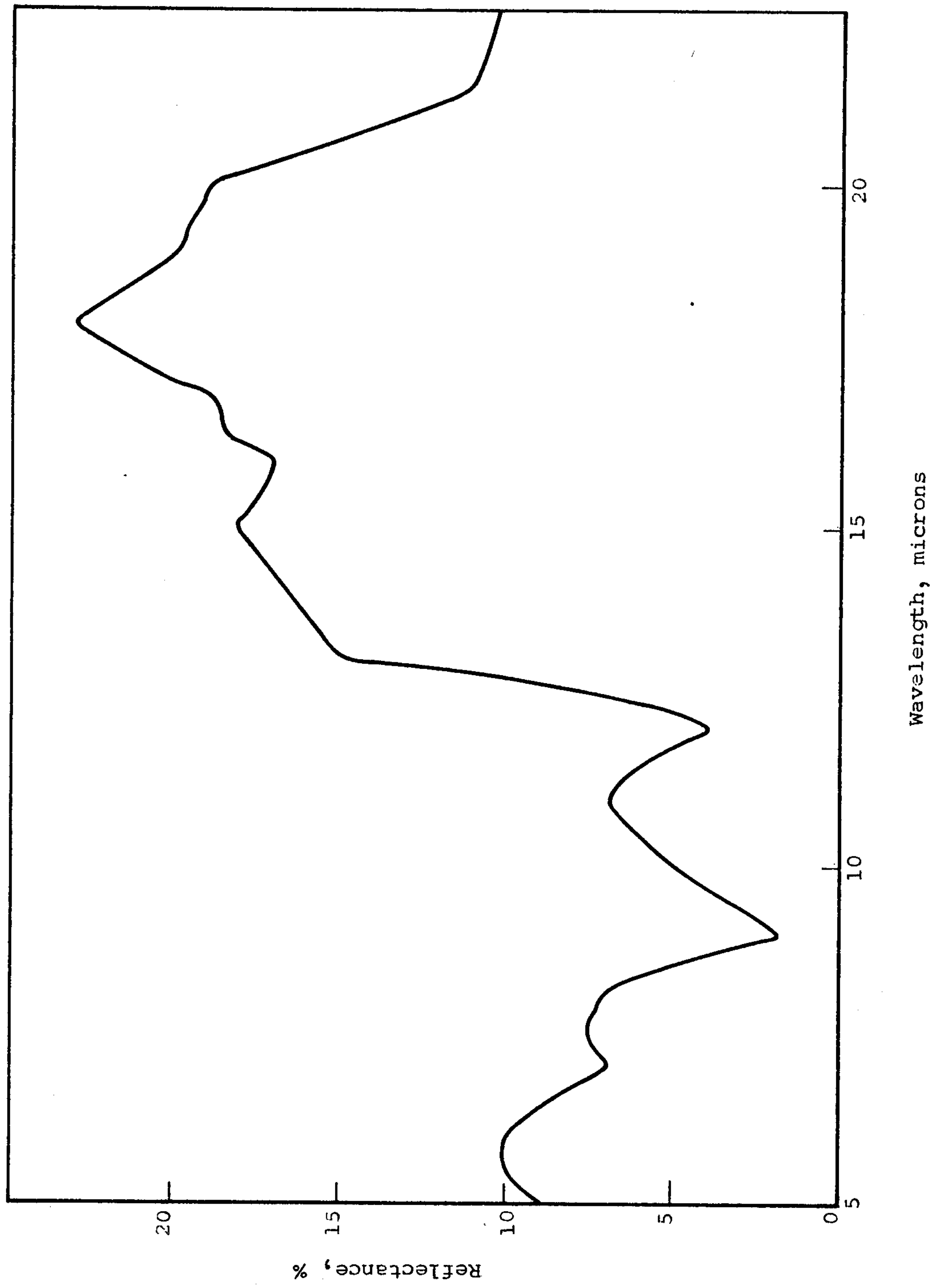


Figure 5

REFLECTANCE OF PAINT NUMBER 42-34

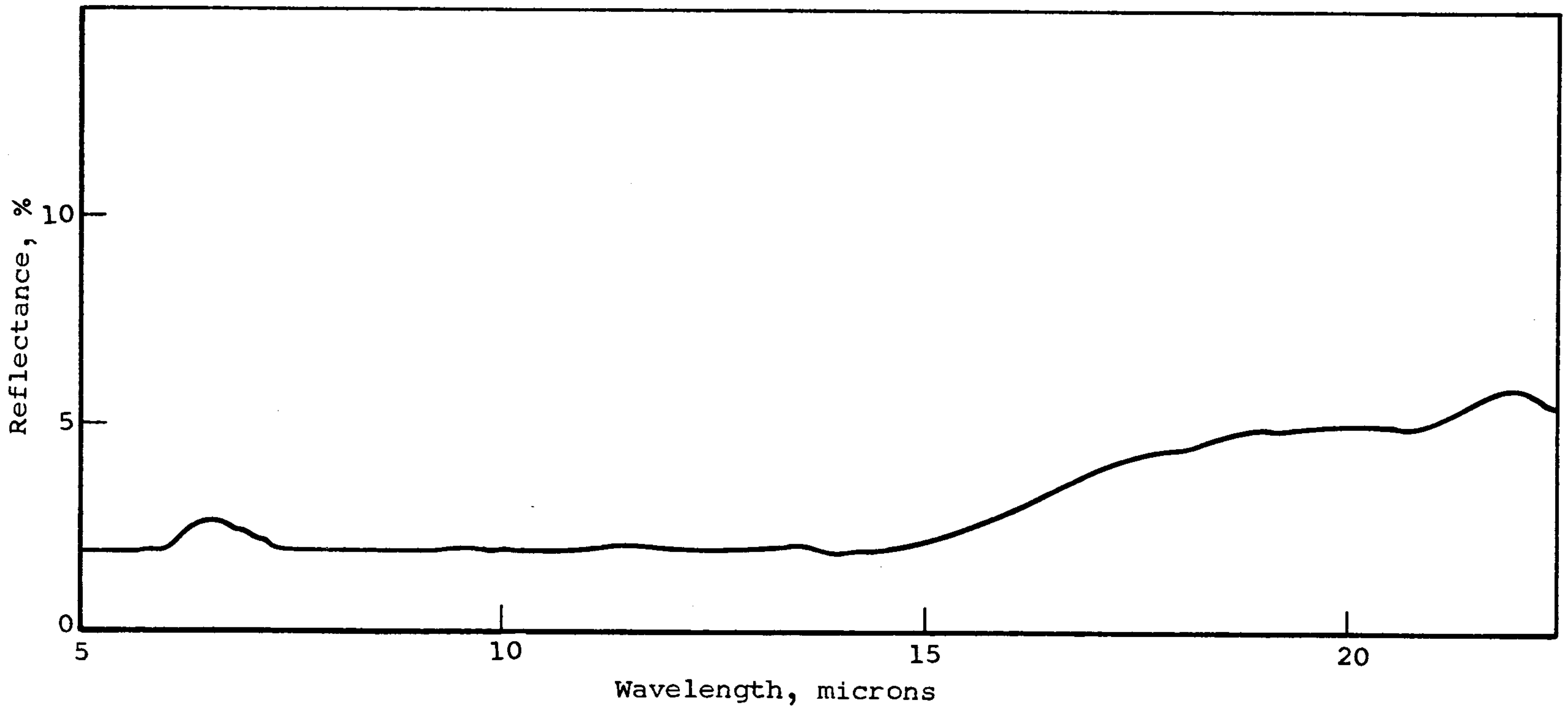


Figure 6

REFLECTANCE OF PAINT NUMBER 42-37

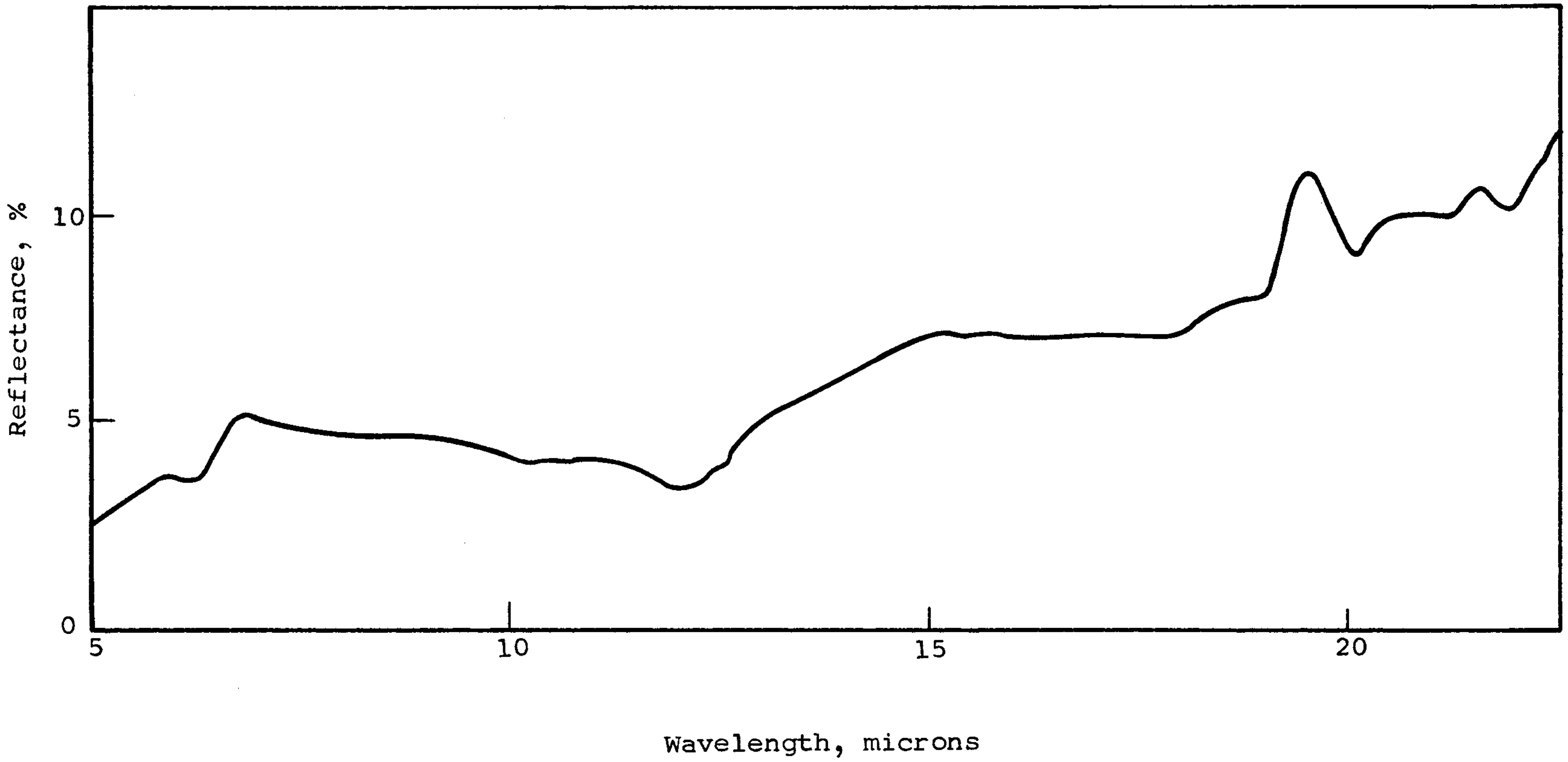


Figure 7

REFLECTANCE OF PAINT NUMBER 42-38

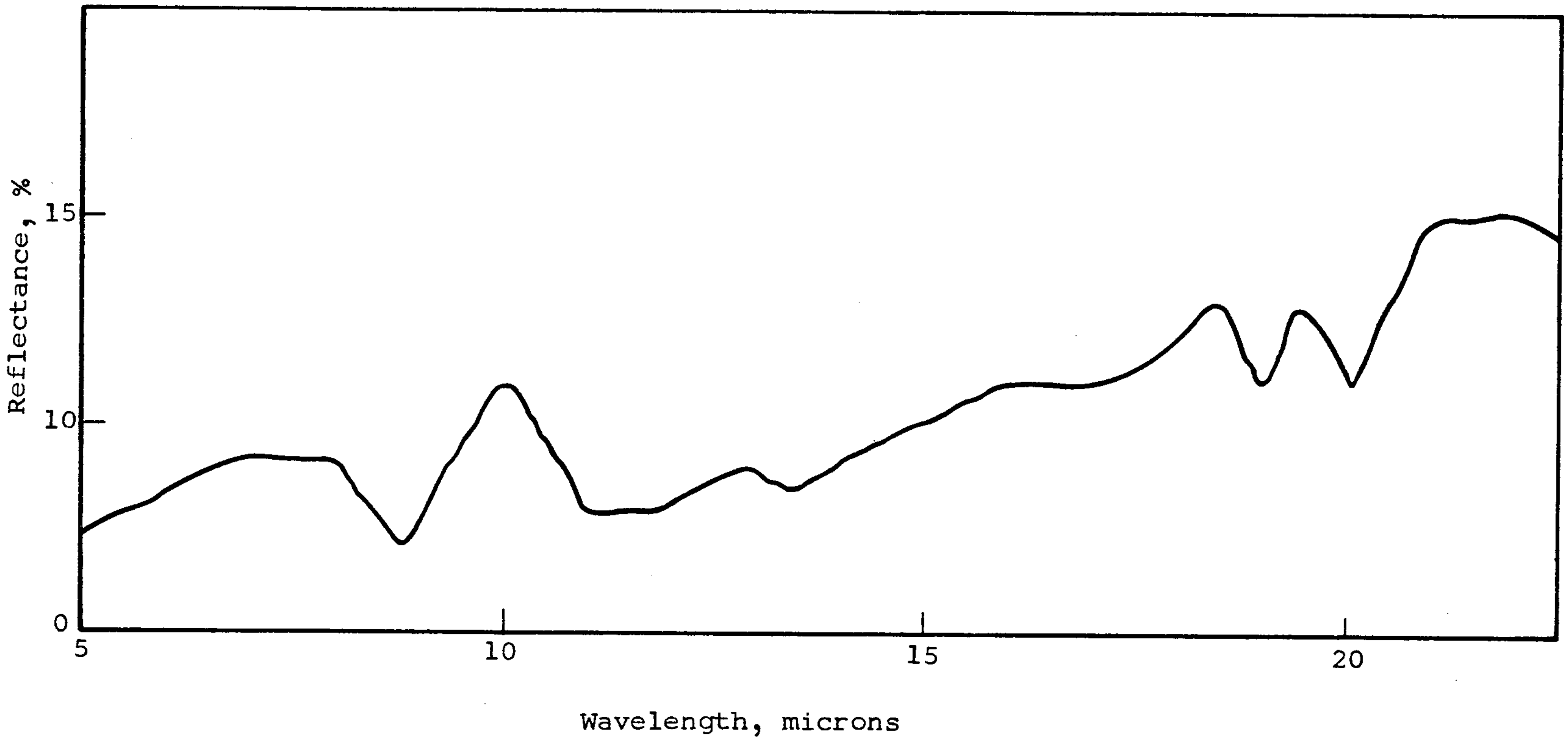


Figure 8

REFLECTANCE OF PAINT NUMBER 42-48

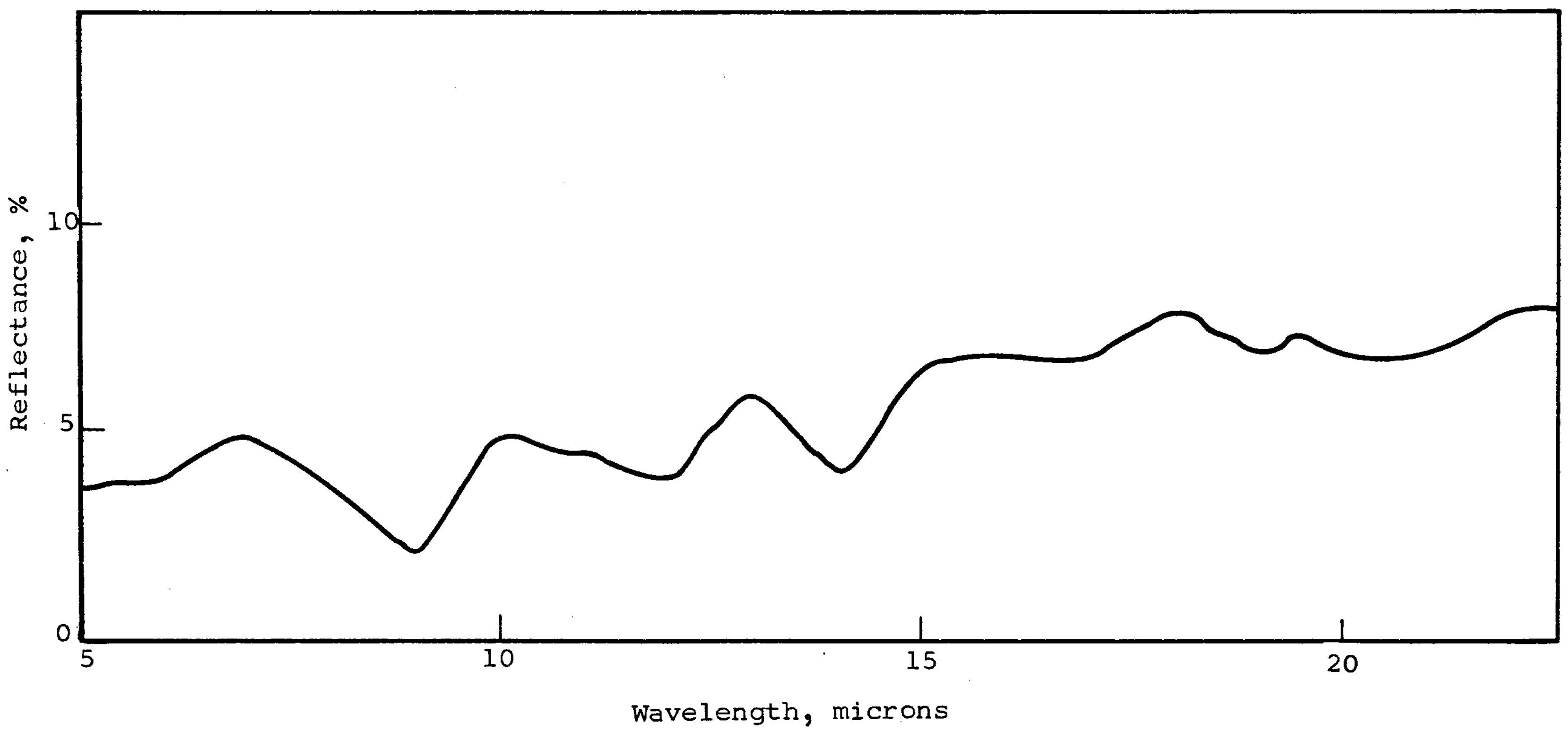


Figure 1

REFLECTANCE OF PAINT NUMBER 42-51

APPENDIX G

CONVERSION-COATING ALUMINUM SUBSTRATES

Iridite No. 14-2, manufactured by Allied Research Products, Inc., is a processing agent for conversion-coating aluminum or aluminum alloy surfaces, which can be applied by dip, brush, swab or spray. For this purpose, the dip technique was used, according to the following procedure:

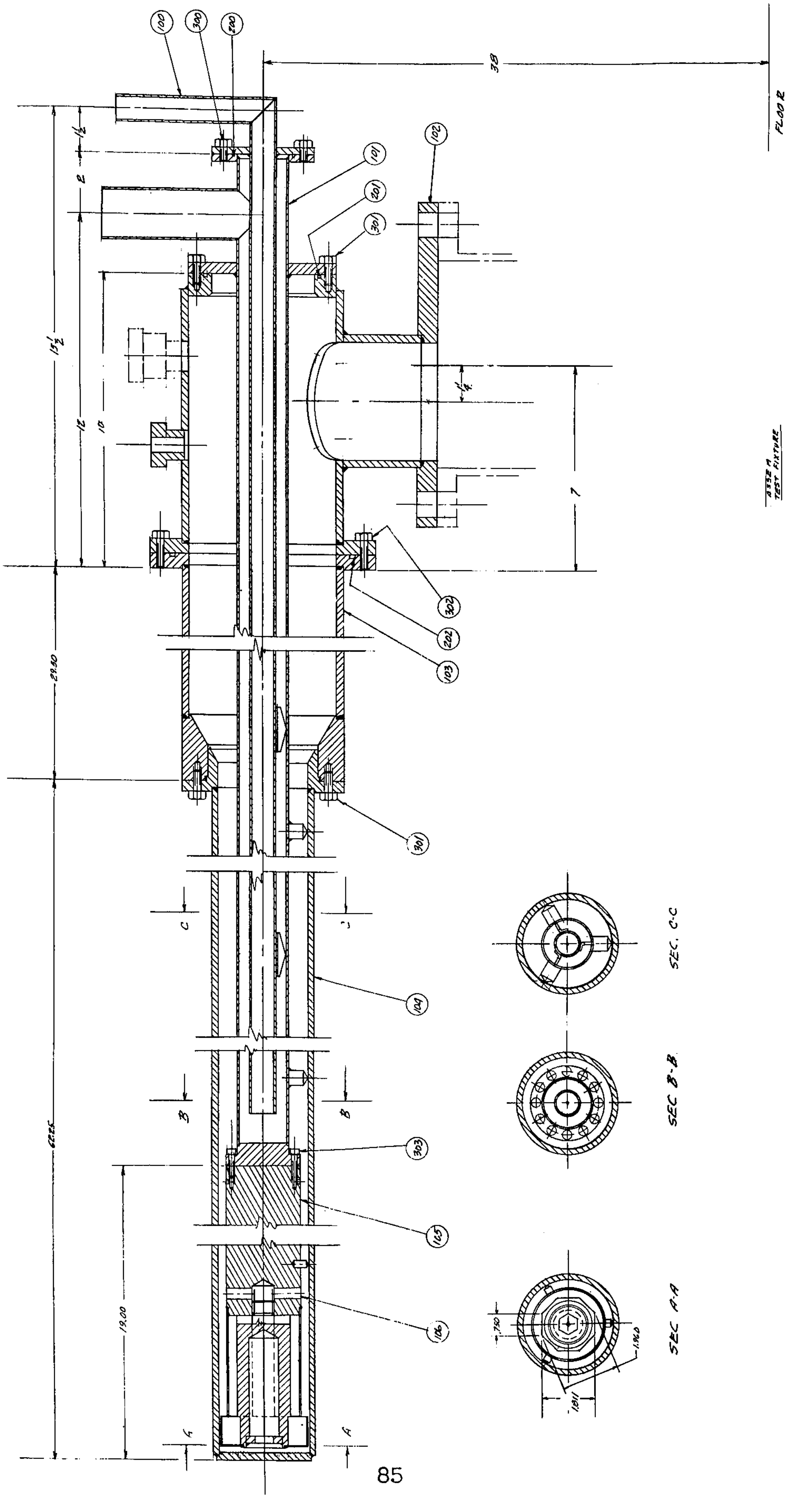
1. The substrate was washed with trichloroethylene and allowed to dry.
2. The substrate was washed with Alconox, rinsed in water and allowed to dry.
3. The substrate was next cleaned by dipping the panel into a solution of chromate-type deoxidizer (3-5 min at 70 to 90°F). The deoxidizer consisted of:

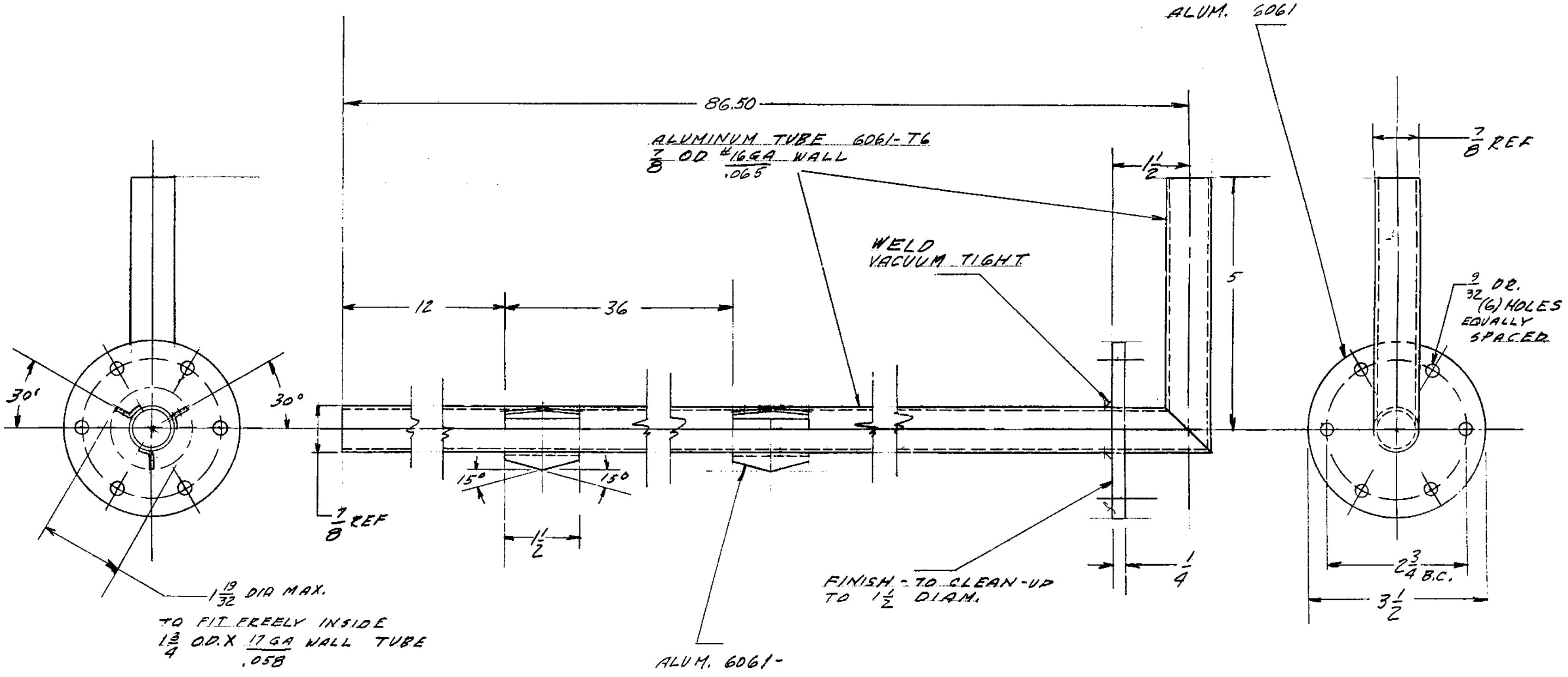
ARP No. 170	6 oz/gal
Alconox	1.5 oz/gal

4. The panel was again rinsed in water, then dipped (5 min at 70 to 90°F) in a solution of Iridite No. 14-2 (concentration 2 oz/gal). The specimen was again rinsed, then dried in an air-circulating oven at 120°F.

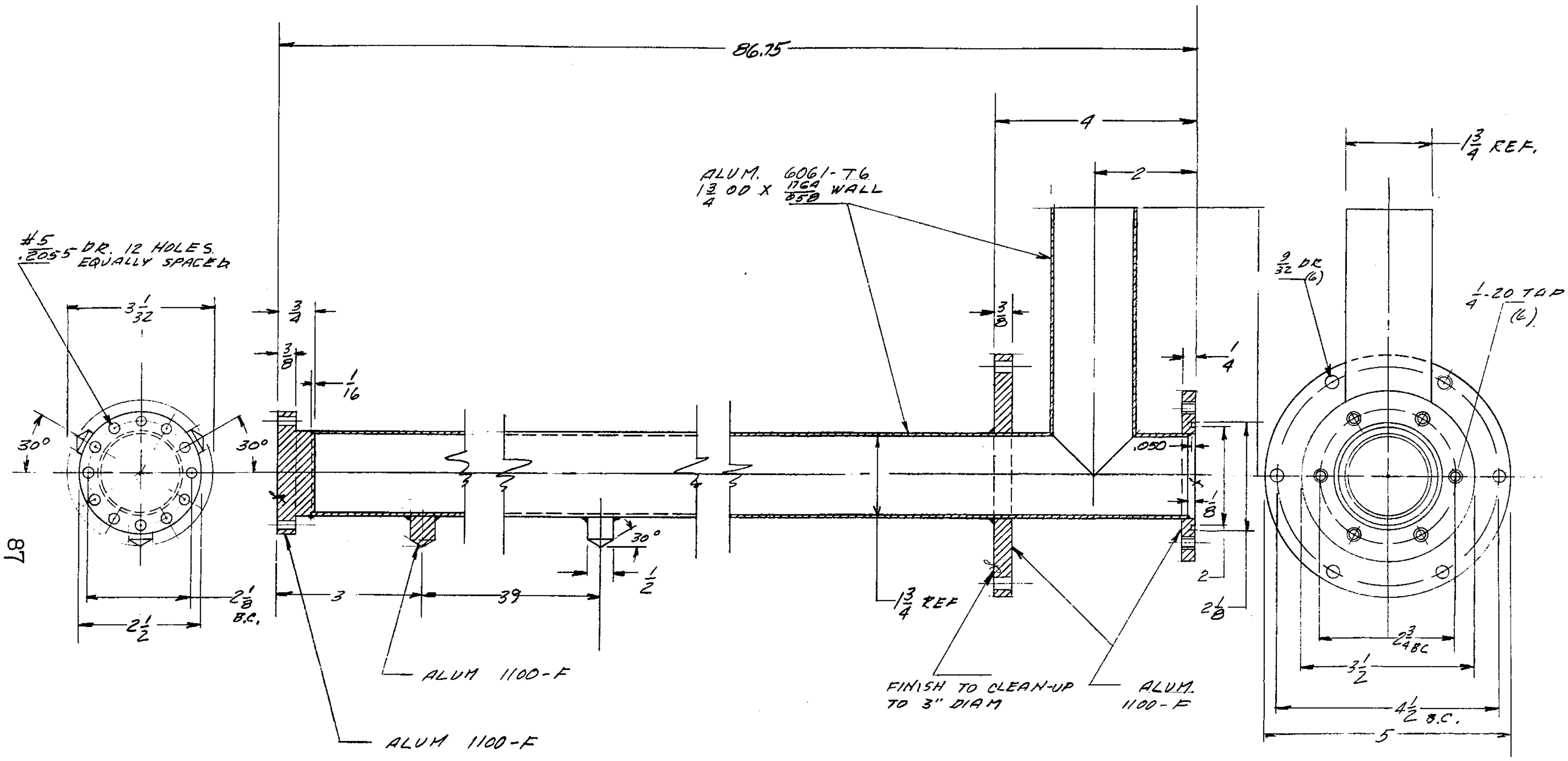
APPENDIX H

ENGINEERING DRAWINGS OF NUCLEAR EXPOSURE FIXTURE


ASSEM
TEST FIXTURE
100
300
200
101
201
301
102
38
FLOOR
13½
12
10
2
1½
1¼
7
302
202
103
29.30
301
104
62.25
C
C
B
B
303
105
106
19.00
A
A
SEC. C-C
SEC. B-B
SEC. A-A
.750
1.811
1.340

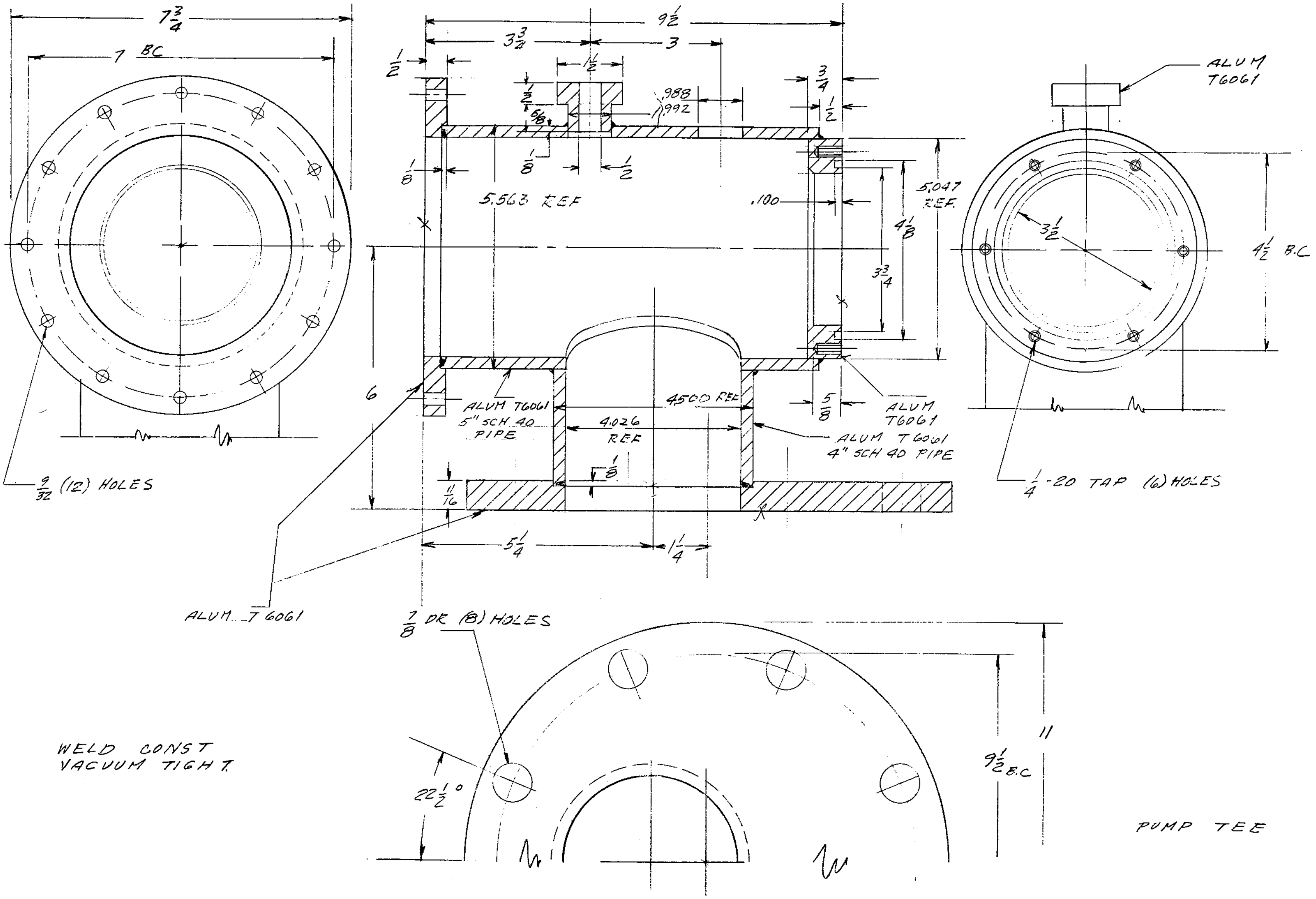
ALUM. 6061
86.50
ALUMINUM TUBE 6061-T6
7/8 OD #16GA WALL .065
1 1/2
7/8 REF
5
WELD
VACUUM TIGHT
9/32 DR. (6) HOLES EQUALLY SPACED
12
36
30'
30°
15°
15°
1 1/2
7/8 REF
1 19/32 DIA MAX.
TO FIT FREELY INSIDE
1 3/4 O.D. X 17 GA WALL TUBE .058
ALUM. 6061-
FINISH - TO CLEAN-UP TO 1 1/2 DIAM.
1/4
2 3/4 B.C.
3 1/2

LN_2 TUBE



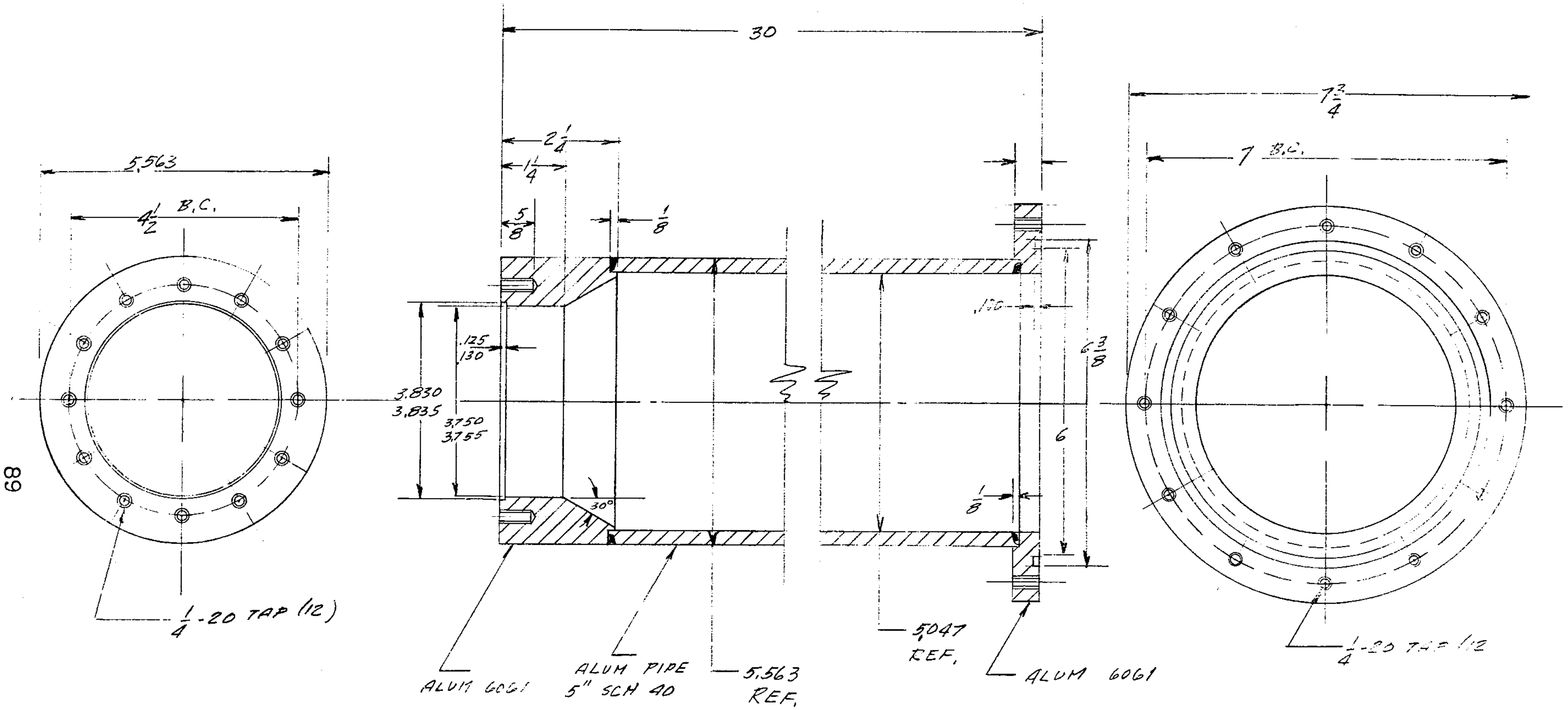
WELDED CONST.
VACUUM TIGHT.

GN_2 TUBE



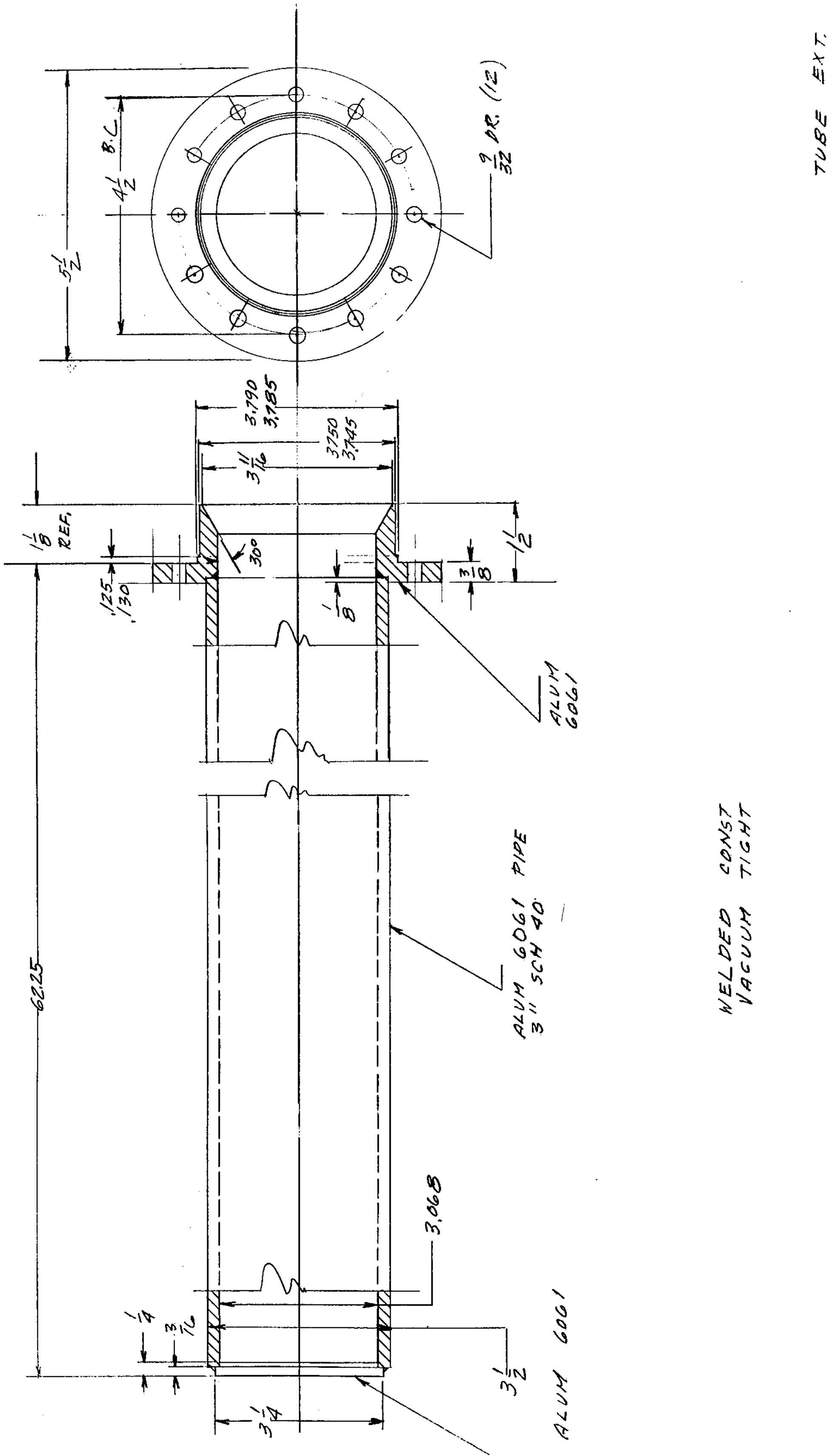
WELD CONST
VACUUM TIGHT.

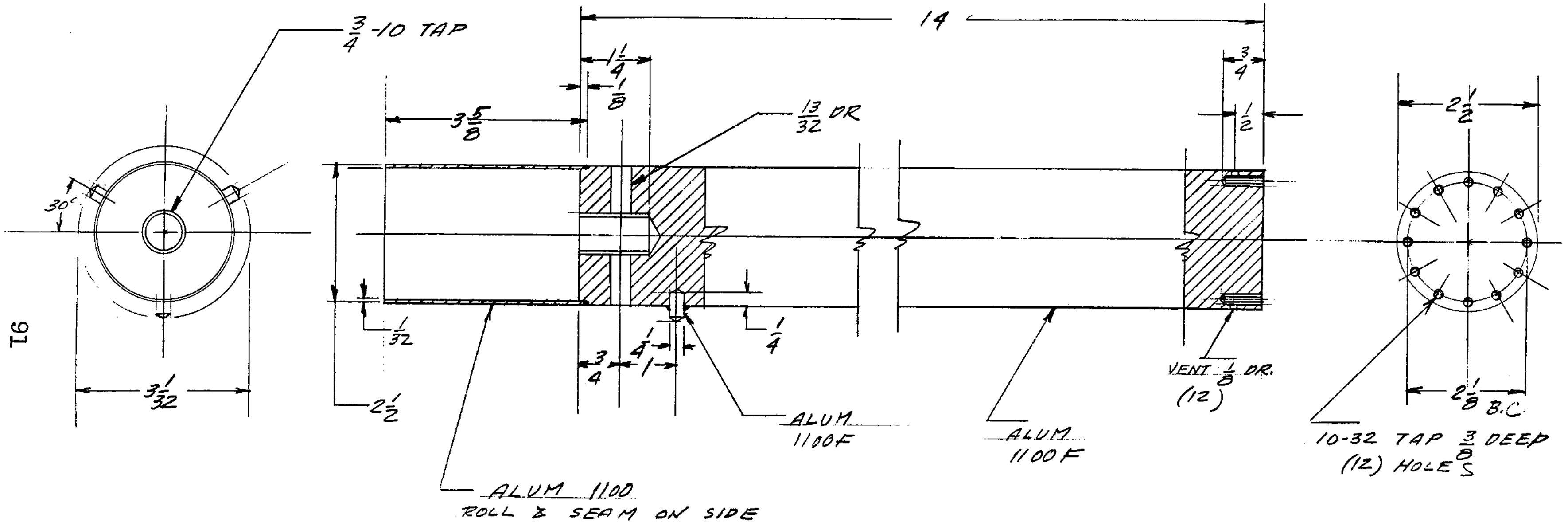
PUMP TEE



WELDED CONST.
VACUUM TIGHT

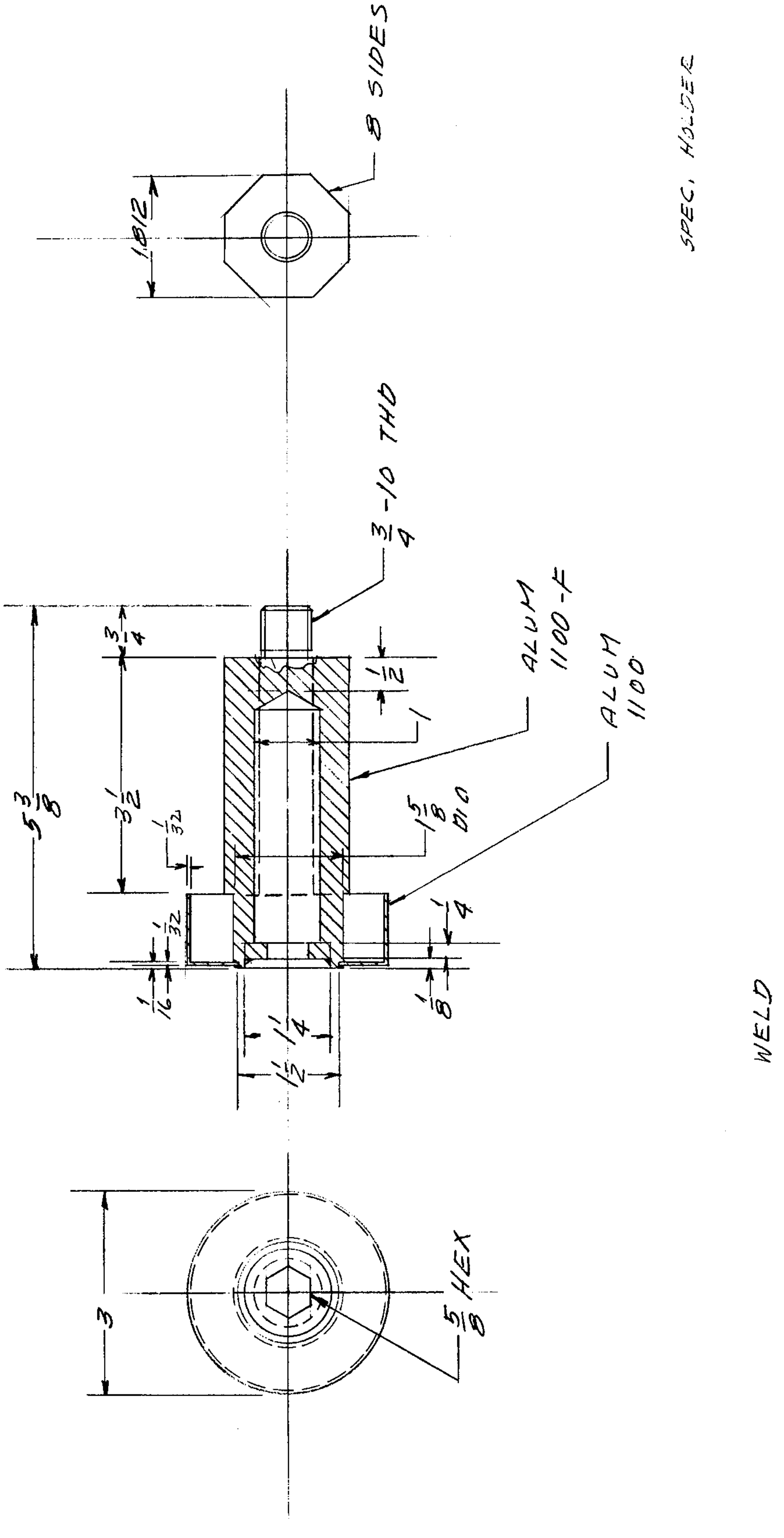
TUBE


TUBE EXT.
WELDED CONST
VACUUM TIGHT
5 1/2
4 1/2 B.C.
9/32 DR. (12)
3.790
3.785
3.750
3.745
3 11/16
1/8 REF.
1 1/2
3/8
.125
.130
1/8
30°
ALUM 6061
62.25
ALUM 6061 PIPE
3" SCH 40
3.068
1/4
3/16
3 1/2
3 1/4
ALUM 6061



WELDED CONST

HEAT CONDUCTOR


8 SIDES
1.812
SPEC. HOLDER
3/4-10 THD
3/4
1/2
1
ALUM 1100-F
ALUM 1100
5 3/8
3 1/32
1/32
1 5/8 DIA
1/4
1/32
1/16
1/8
1 1/4
1 1/2
WELD
3
5/8 HEX

APPENDIX I

SUMMARY OF DATA ON SELECTED PHASE II COATINGS

Table 19A

COATING SYSTEMS-IDENTIFICATION AND RELATIVE ENVIRONMENTAL RESISTANCE RATINGS

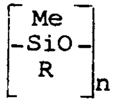
Order of Recommended System	Binder Trade Name	Coating-Generic Composition	Order of Coating System	Curing Agent	Carbon-Black Volume %	Curing Temperature	Curing Time, at Temperature
I	Dalvor	Polyvinyl Fluoride $(CH_2CHF)_n$	10.1		5	500°F	5 min.
II	G.E. SR 112	Polysiloxane  $\left[-\overset{Me}{\underset{R}{Si}}O-\right]_n$	9.7	Zinc octoate	10	480°F	75 min.
III	G.E. SR 112	Polysiloxane	9.2	SC 3963 (Amine type low temperature curing agent)	11	300°F	4 hours

Table 19B

COATING SYSTEMS-IDENTIFICATION AND RELATIVE ENVIRONMENTAL RESISTANCE RATINGS

G.E. SR 112 Resin 300°F Cure Code 42-51	G.E. SR 112 Resin 480°F Cure Code 42-48	Dalvor-Code 42-34	Environment or Property
Fair to Good	Good	Excellent	Acid (HF, HCl, HNO_3) Resistance
Fair to Good	Good	Excellent	Superheated Steam
Good	Good	Good	Jet Cleaning (Hot Alconox)
Satisfactory	Satisfactory	Satisfactory	Spectral Requirements
Good	Good	Superior	Abrasion Resistance
Satisfactory	Satisfactory	Satisfactory	Thermal Shock
Acceptable	Acceptable	Acceptable	Emissivity (Hemispherical, total)
A-Satisfactory B-Satisfactory C-Fair D-Satisfactory	A-Satisfactory B-Satisfactory C-Good D-Satisfactory	A-Satisfactory B-Satisfactory C-Superior D-Satisfactory	Nuclear Radiation Exposure-Effects: (A-Visual; B-Spectral; C-Out-gassing: D-Thermal Shock)
Superior	Superior	Superior	Exposure to Ultraviolet light (2000 Equivalent Sun-Hours)
Fair to Good	Good	Fair to Good	Decontamination

NASA-Langley, 1967 —— 18 E-3709