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FINAL REPORT

FOR

INVESTIGATION OF FABRICATION AND PROCESSING PARAMETERS ASSOCIATED WITH USE OF POLYURETHANE FOAMS IN SEALED CRYOGENIC INSULATION

NASA LeRC Contract NAS 3-5646

GER-12249 June 25, 1965

by: C. B. Shriver

TABLE OF CONTENTS

		Page
List of Illustrations		ii
List of Tables		iv
Section	Title	
I.	Summary	1
II.	Introduction	3
III.	Investigation Discussion	
	A. General	4
	B. Polyurethane Foam Investigations	7
	1. Determination of an Optimum Hole Pattern	7
	2. Investigation of Outgassing Characteristics	22
	3. Effects of Foam Cell Structure	47
	4. Compression Characteristics	52
	C. Vapor Barrier Materials and Fabrication Techniques Investigation	61
	1. Permeability Characteristics	61
	2. Outgassing Characteristics	66

LIST OF ILLUSTRATIONS

Figure	Title	Page
1.	Mechanical Perforation of Calorimeter Sample Foam Separators	9
2.	12" Diameter Calorimeter Sample with Cross Ply Strip Foam Separator	10
3.	Gas-Bubble Perforation of Calorimeter Sample Foam Separators	11
4.	Electrical Test Setup for Heat Shorts between Radiation Shields	13
5.	Manometer and Electrical Resistivity Apparatus for Foam Separator Screening Tests	16
6.	Schematic of Electrical Short Test Setup	17
7.	Foam Separator Strip Configurations for Electrical Short Tests	18
8.	Calorimeter Sample #5	28
9.	Calorimeter Sample #6	29
10.	Calorimeter Sample #8	30
11.	Calorimeter Sample #14	31
12.	High-Vacuum Balance Test Chamber	32
13.	Weight Loss Measurements - GAC Rigid White Foam at Ambient Temp.	38
14.	Weight Loss Measurements - GAC Rigid White Foam at 300°F	39
15.	Weight Loss Measurements - Mobay Chemical Open Cell No. 7 at Ambient Temp.	40
16.	Weight Loss Measurements - Mobay Chemical No. 7 at 300°F	41
17.	Weight Loss Measurements - Mobay Chemical No. 3 at 300°F	42

LIST OF ILLUSTRATIONS (CONT.)

Figure	Title	Page
18.	Weight Loss Measurements - Carwin Chem. Open Cell Rigid Foam at Ambient Temp.	43
19.	Weight Loss Measurements - Carwin Chem. Foam at 300°F	44
20.	Open- and Closed-Cell Foam Structures	48
21.	Compression Test Set Up Foam and Film Samples	53
22.	Typical Compression Load - Deflection Curves Foam and Film at Room Temp.	58
23.	Typical Compression Load - Deflection Curves Mobay #7 Foam and Film at -320°F Room Temp.	59
24.	Ultimate Compression Curve Mobay No. 7 Foam and Film at -320°F	60
25.	Helium Leak- Detector Technique for Evaluating Vapor Barrier Seams	64
26.	Vapor Barrier Seam Leak Test Sample and Test Fixture	65
27.	Weight Loss Measurements - MAAM Strips at Ambient Temp.	68
28.	Vapor Barrier Seam Outgassing Sample	69
29.	Weight Loss Measurements - Sample #1 - MAAM Seams at Ambient Temp.	71
30.	Weight Loss Measurements - Sample #2 - MAAM Seams at Ambient Temp.	72

LIST OF TABLES

Table Title Page

I Aluminized Mylar Electrical Tests for Heat Shorts 12

II List of Foam Separator Specimens for Electrical Short Tests 20

IIA Foam Separator Test Results - Pressure at ∞ Resistivity 21

III Composition of 12 Inch Diameter Calorimeter Test Results 23

IV Summary of Foam Weight Loss Measurements due to Vacuum Preconditioning at Room Temperature and -320°F 37

V Compression Test Values - 2" x 2" Foam and Film Composite Specimens 56

VI Vapor Barrier Film Thickness, Weight and Helium Permeability Data 62

VII Vapor Barrier Adhesive Seam Permeability 63

I. SUMMARY

An investigation of certain fabrication and processing parameters associated with the use of polyurethane foams in sealed cryogenic insulations is presented.

The problems associated with the use of polyurethane foams in sealed cryogenic insulations were investigated by GAC under the following tasks:

a. Optimum hole pattern in perforated foam. Fifteen 12 inch diameter test specimens were fabricated for NASA LeRC evaluation in a thermal conductivity apparatus.

b. Outgassing characteristics. Methods of preconditioning foam to reduce outgassing were investigated. These include vacuum conditioning at room temperature and elevated temperature.

c. The effects of cell structure. Several types of open cell foams were evaluated to obtain a desirable open cell rigid foam. Attempts were made to rigidize several open cell flexible foams.

d. Compression characteristics of open and closed cell foams were investigated at room temperature and -320° F.

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Vapor barrier materials evaluation and fabrication techniques for utilization of vapor barrier materials to seal polyurethane foam insulations were investigated under the following tasks:

a. Vapor barrier films - Helium gas permeability tests utilizing mass spectrometer techniques were conducted on various candidate vapor barrier materials.

b. Adhesives - Helium gas permeability tests utilizing mass spectrometer techniques were conducted on selected adhesive candidates for vapor barrier material seams.

c. Outgassing characteristics - Selected vapor barrier films were preconditioned to reduce outgassing of surface contaminants and evaluated by weight loss measurements in a vacuum environment.

d. Vapor barrier film adhesive seam samples were evaluated for outgassing of a typical glue line edge by weight loss measurements in a vacuum environment.

II. INTRODUCTION

Certain problems are associated with the use of low density polyurethane foam in sealed cryogenic insulations systems such as the Centaur lightweight insulation developed by NASA LeRC and Goodyear Aerospace Corporation (GAC). To reduce or eliminate these problems, further investigations were conducted to optimize the material and configuration of the foam element and to improve the vapor barrier material sealing element of the insulation system. This final report describes the work performed by GAC on NASA LeRC contract NAS 3-5646 Article 1.c. wherein the problems associated with the use of polyurethane foams in sealed cryogenic insulation systems were investigated. Close technical liaison between NASA LeRC and GAC afforded decisions and expeditious attainment of investigation objectives.

III. INVESTIGATION DISCUSSION

A. General

1. The specific items investigated as described in later detail are those associated with upgrading the reliability and thermal performance of the Centaur lightweight sealed foam insulation. The investigations are directed toward the improvement of two major elements of the sealed insulations, (a) the foam element and (b) the vapor barrier foam sealing element.

2. Investigations directed toward foam optimization as an insulation in a sealed foam system included the following tasks:

   (1) Determination of an optimum hole pattern in perforated foam. To determine the maximum percent of open area, hence minimum area of solid conduction, test samples were constructed of alternating layers of foam slices and metallized Mylar film for evaluation by NASA LeRC in a thermal conductivity apparatus. Preliminary screening tests were conducted at GAC to determine the maximum percent of open area possible without contact of metallized Mylar layers when the foam Mylar sandwich is subjected to uniformly distributed pressures ranging from 0 to one atmosphere.

(2) Outgassing characteristics. Candidate foam samples in slices form were analyzed for outgassing characteristics as related to practical techniques for preconditioning the foam to reduce outgassing. These foam preconditioning techniques include:

(a) Vacuum conditioning at room temperature

(b) Vacuum conditioning at elevated temperature

(c) Alteration of foam formulations to reduce or eliminate the elements which theoretically would outgas at very low vapor pressures of 10^{-4} Torr or less.

(3) The effects of cell structure. Rigid closed cell foams have two deficiencies in a sealed self-evacuating cryogenic insulation:

(a) The cells provide a gas source that slowly diffuses through the cell walls creating an outgassing problem.

(b) The flow of cryopumped gas to the cold wall is restricted by the cell walls.

Open cell walls do not have these problems and are therefore desirable. To obtain a rigid open cell foam, two approaches were investigated, (1) obtain a foam that is rigid in the original state by the use of a cell structure having a high percentage of broken cell walls and (2) attempt to rigidize open cell flexible foams by application or impregnation of a rigid coating to the skeleton network of the foam.

(4) Compression Characteristics. The compression properties of deflection, recovery and permanent set were investigated of the candidate foam samples. Tests were conducted at room temperature and -320°F. Consideration of these properties will be a factor in selecting a foam having the desirable properties of low deflection under load and complete recovery after removal of load.

3. The investigations directed toward vapor barrier materials and fabrication techniques for a sealed foam insulation include the following tasks:

(1) Permeability Characteristics: The helium gas permeability of vapor barrier materials and vapor barrier seam adhesives were investigated utilizing a mass spectrometer apparatus. Candidate vapor barrier materials were evaluated in both the unwrinkled and wrinkled condition. By mutual agreement between NASA and GAC technical representatives, the vapor barrier material helium permeability data generated by GAC for NASA Contract NAS 8-11376 (Flexible Vacuum Jacket Development) will provide adequate information on this aspect of the permeability characteristics. The helium permeability data for vapor barrier adhesive seams was conducted on two candidate resin systems Goodyear G-207 polyester resin, and Adiprene L-100/Mocha polyurethane resin.

(2) Ougassing Characteristics. On the basis of permeability tests, vapor barrier materials having a Mylar outer layer appear most promising for sealed self-evacuating cryogenic insulations. The outgassing characteristics of the Mylar surface was evaluated by weight loss measurements conducted after surface preparation by solvent cleaning.

Vapor barrier material seams were evaluated for outgassing characteristics of the adhesive as a function of drying of the adhesive prior to heat sealing of the seam.

B. Polyurethane Foam Investigations

1. Determination of an optimum hole pattern in perforated foam.

By reducing the contact area of the foam on adjacent foam or aluminized Mylar layers, the following benefits will accrue:

(1) The thermal conductivity will be lowered

(2) Weight will be reduced

(3) The breathing capability of the foam will be improved, thus lowering the impedance to cryopumping gas flow.

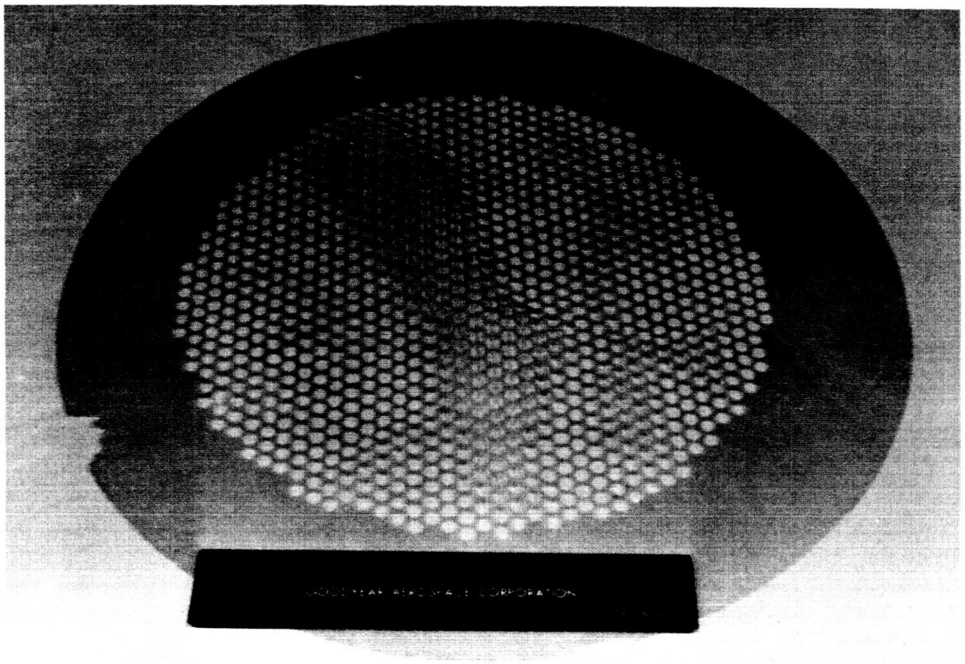
The contact area of the foam may be reduced by addition of perforations to the foam separators by mechanical techniques:

(a) of punching and piercing as shown in Figure 1.

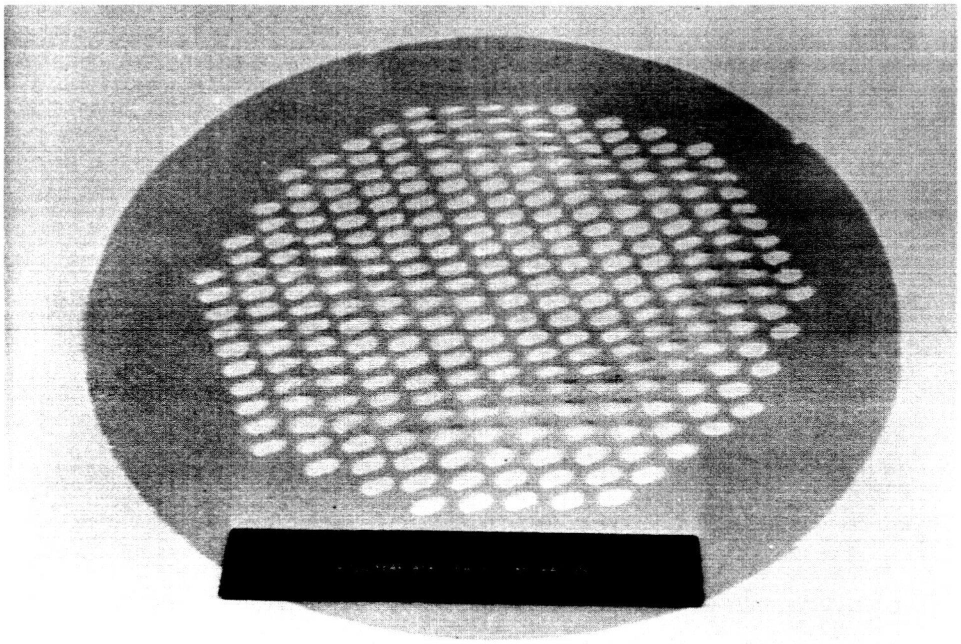
(b) by cross plying foam strips as shown in Figure 2.

(c) by specialized foam formulation to provide uniform distribution of gas bubbles throughout the foam manifesting themselves as holes or perforations when the foam is cut into thin slices as shown in Figure 3.

A paramount consideration in the selection of the perforation size and pattern is the ability of the perforated foam separator to prevent thermal shorts caused by contact of the 0.00025-in. aluminized Mylar radiation shields while under a normal load of one atmosphere pressure. The thermal short characteristics of various separator perforations was investigated by constructing test samples of alternating layers of 0.00025-in. aluminized Mylar shield and foam separators to a minimum thickness of two metallized Mylar layers and three foam separator layers. The foam slices were perforated with various patterns and sizes of holes to determine the maximum percent of open area that is possible without contact of the metallized Mylar shields when the foam-Mylar sandwich is subjected to one atmosphere pressure induced by a flexible vacuum jacket surrounding the sample. The Mylar shields placed with aluminized sides adjacent to each other were wired to an ohmeter.



CIRCULAR HOLES



OBLONG HOLES

FIGURE 1 - MECHANICAL PERFORATION OF CALORIMETER SAMPLE FOAM SEPARATORS

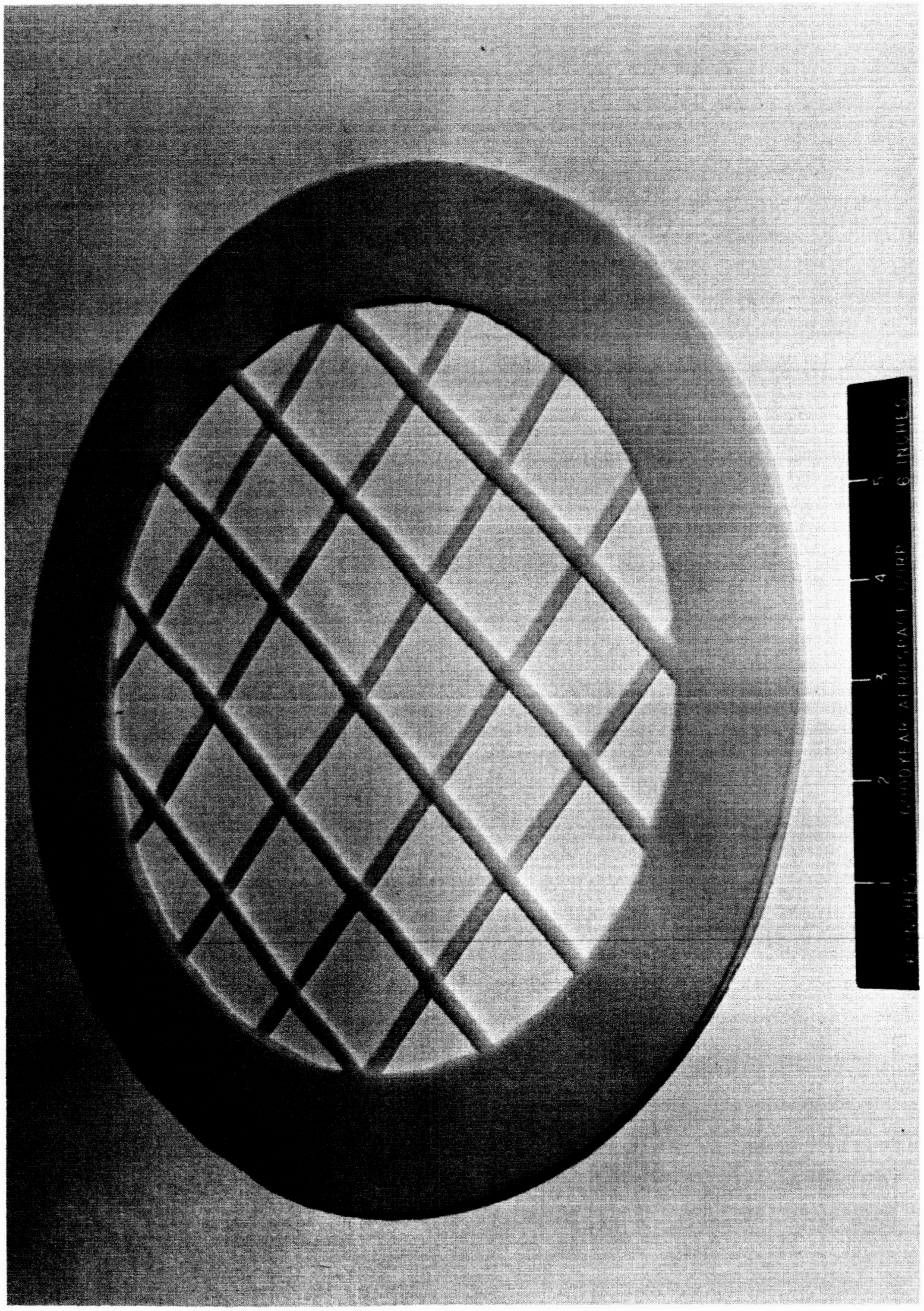
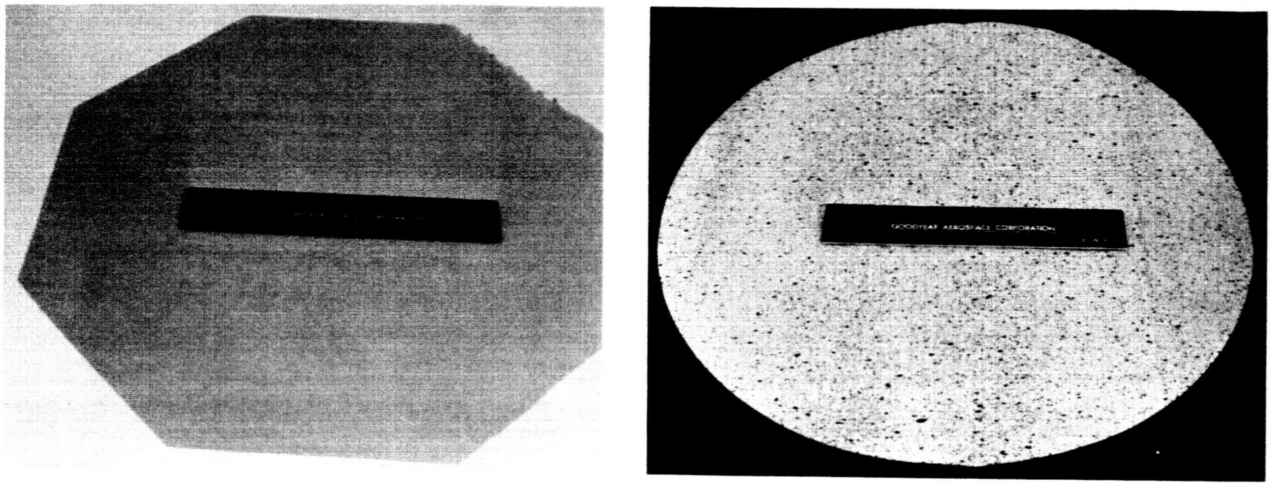
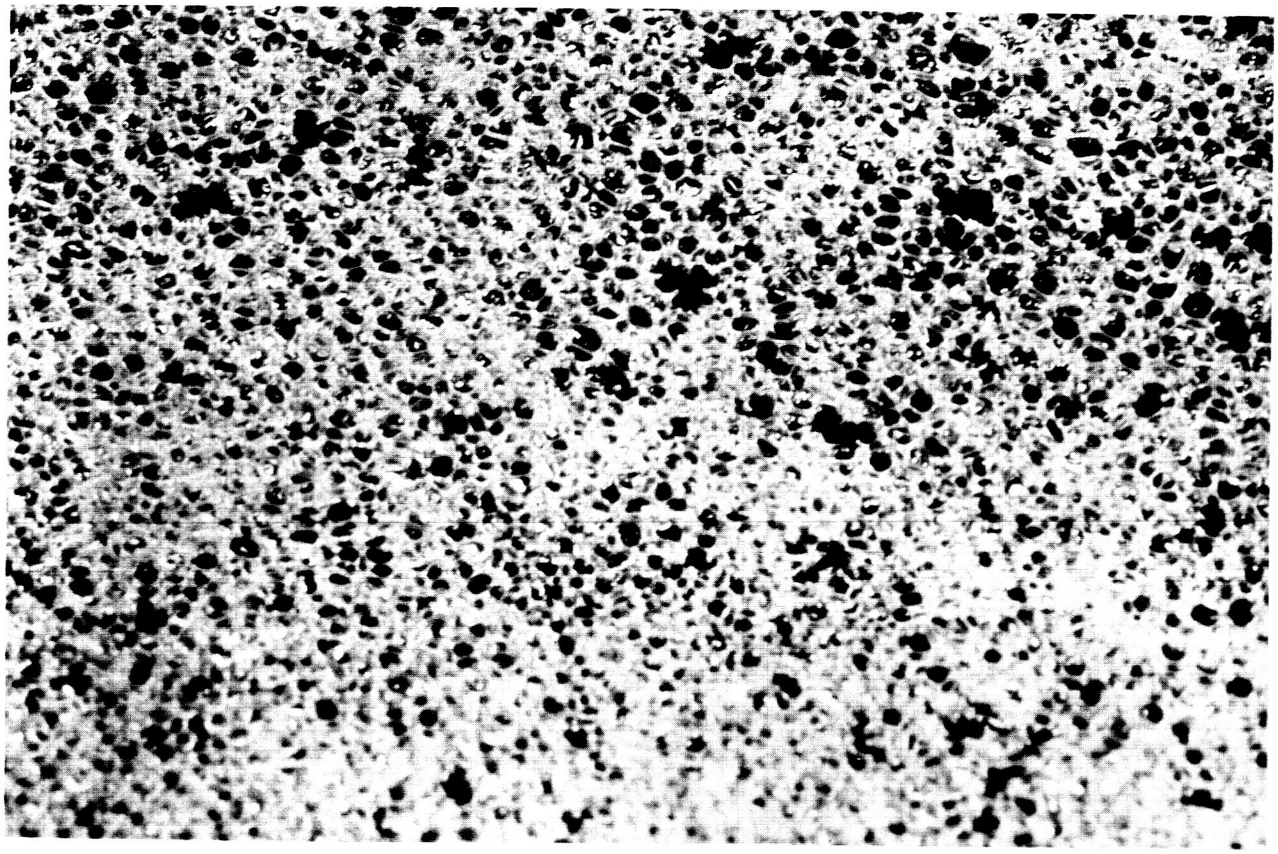


FIGURE 2 - 12" DIAMETER CALORIMETER SAMPLE WITH CROSS PLY STRIP FOAM SEPARATOR



UNIFORM 2-PCF RIGID
POLYURETHANE FOAM

2-PCF RIGID POLYURETHANE
FOAM WITH LARGE GAS BUBBLE



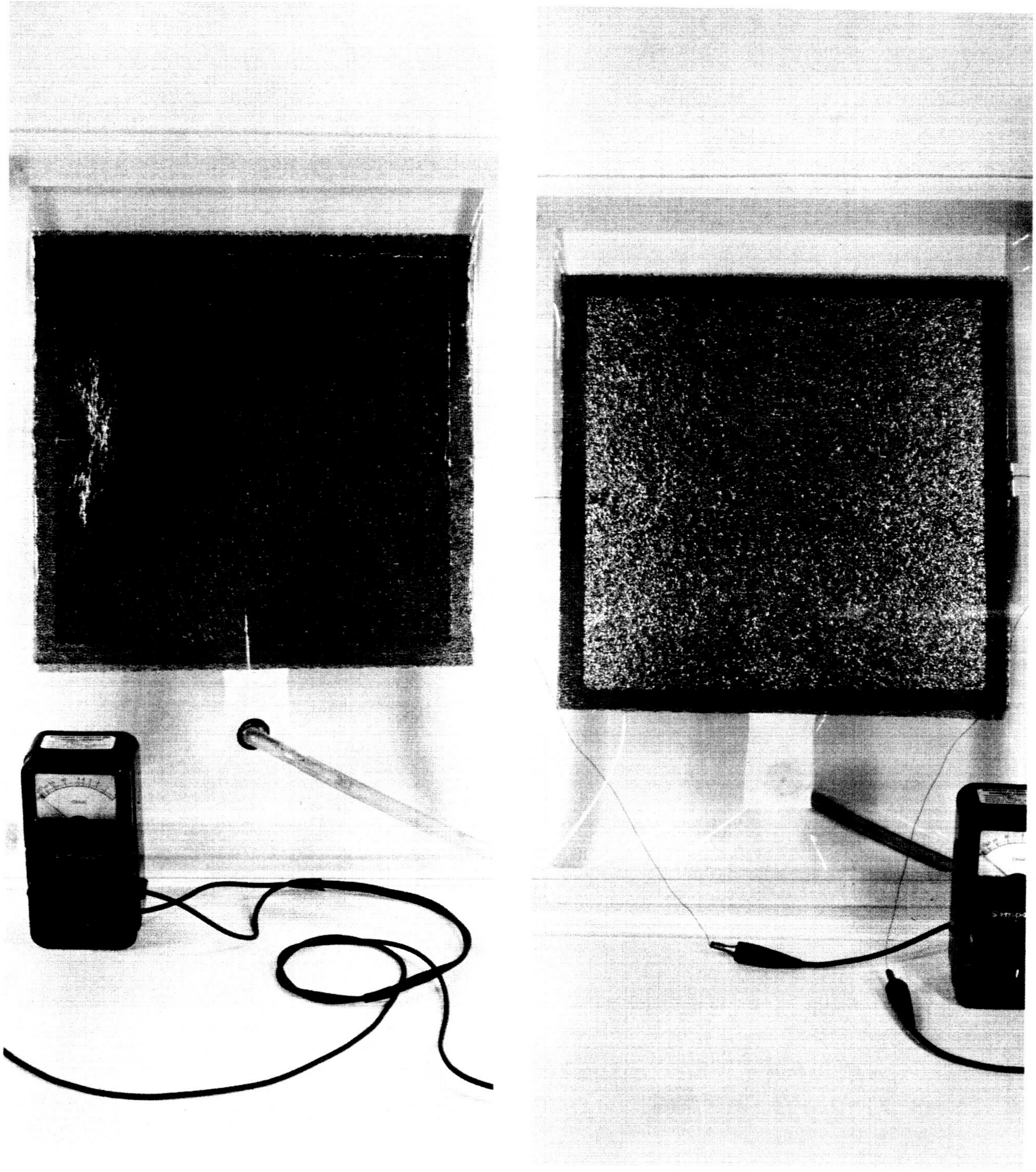
CLOSEUP OF GAS BUBBLES

FIGURE 3 - GAS-BUBBLE PERFORATION OF CALORIMETER SAMPLE FOAM SEPARATORS

Contact between the aluminized shields was checked by the ohmeter resistivity measurements observed during evacuation of the sample. The above testing technique is shown in Figure 4. Test results on several types of foam with and without perforation patterns are noted in Table I.

TABLE I - ALUMINIZED MYLAR ELECTRICAL TESTS FOR HEAT SHORTS

Separator			
Thickness (in.)	Pores per in.	Type	Test Results (15 psi)
0.10	10	Flexible Scott foam	Short
0.10	10	Rigidized Scott foam	No short
0.10	20	Flexible Scott foam	Short
0.10	25	Flexible Scott foam	No short
0.020	..	Rigid polyurethane (Goodyear Aerospace)	No short
0.020	..	Rigid polyurethane (GAC); 1/8 by 1/2 in. slot staggered; 38 percent open; 2 pcf (Figure 1)	Short
0.020	..	Rigid polyurethane (GAC); 2 pcf; 1/8-in.-dia staggered 3/ 8-in. centers; 40 percent open (Fig. 1)	No short
0.020	..	Rigid polyurethane (GAC); 2 pcf; 1/8-in.-dia staggered 1/4-in. centers; 23 percent open	No short
0.020	..	Rigid polyurethane; Zero-cell; 1.5 pcf; 3/16-in. dia staggered 1/4-in centers; 51 percent open	Short
0.020	..	Rigid polyurethane; Zero-cell; 1.5 pcf; 1/4- by 1/2-in. oblong slot, side staggered; 41 percent open	Short



FRONT

REAR

FIGURE 4 - ELECTRICAL TEST SETUP FOR HEAT SHORTS BETWEEN RADIATION SHIELDS

The above test procedure served as a screening method to determine potential perforation patterns that will not allow thermal shorting of aluminzed layers. The following additional considerations will be given to the ultimate selection of perforations:

(1) NASA Lewis Research Center calorimeter test results of thermal performance as a function of compressive load on the foam contact area
(2) Feasibility of perforating foam slices by mechanical means
(3) Strength and handling ability of perforated foam slices

From the test results noted in Table I, the maximum percentage of open area that will not permit thermal shorts of the aluminized Mylar is approximately 40 percent. This percentage is considerably lower than the desired 80 to 90 percent open area objective based on observations from earlier NASA LeRC calorimeter studies.

The preceeding testing was conducted to determine the percent open area in the foam separators wherein no thermal shorts of the Mylar radiation shields would occur under the full range of pressure loading conditions (0-15 psi) encountered by the sealed self-evacuating cryogenic insulation.

Further testing was conducted to determine the maximum open area in the foam separators that would prevent thermal shorting of the Mylar radiation shields at low pressure levels of 0 to 1.5 psi. For this determination the test apparatus was equipped with an inclined tube water manometer to sense the pressure level when the electrical contact is broken between the aluminized layers. The test set up is shown in Figure 5.

The test procedure is as follows:

(1) A seven inch diameter test specimen comprised of foam and aluminized Mylar layers is mounted on a vacuum plate and instrumented as shown schematically on Figure 6. The configuration of typical foam separator specimens (layer # 3 on test setup) are shown in Figure 7.

(2) The Mylar vacuum bag covering the test specimen is evacuated to apply approximately 28" Hg pressure to the specimen. While under the vacuum pressure load, electrical shorting between the aluminized Mylar and the aluminized foam layer is observed by the ohmmeter resistance reading stabilizing at zero. This measurement would represent maximum thermal short condition.

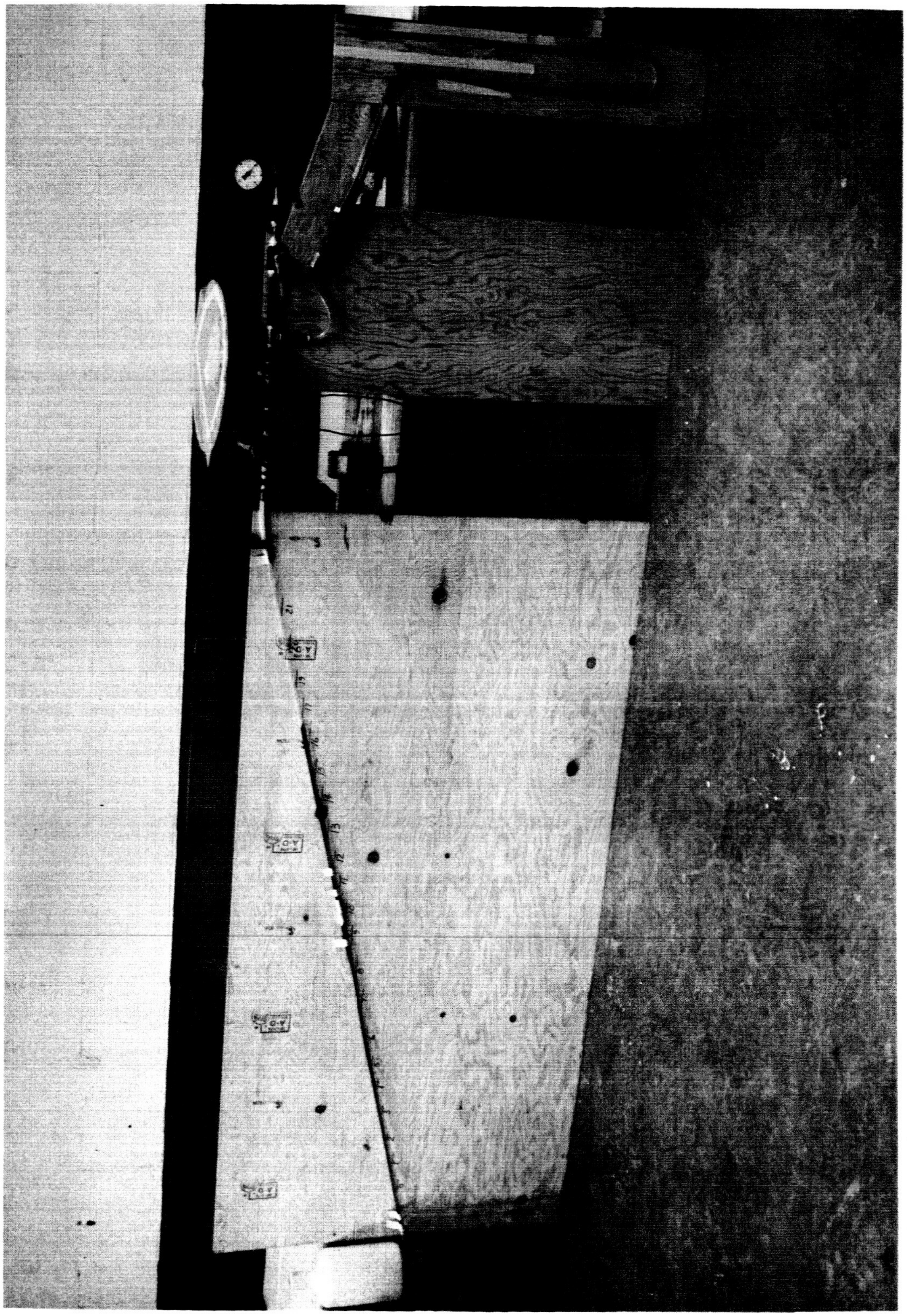


FIGURE 5 - MANOMETER AND ELECTRICAL RESISTIVITY APPARATUS FOR FOAM SEPARATOR SCREENING TESTS

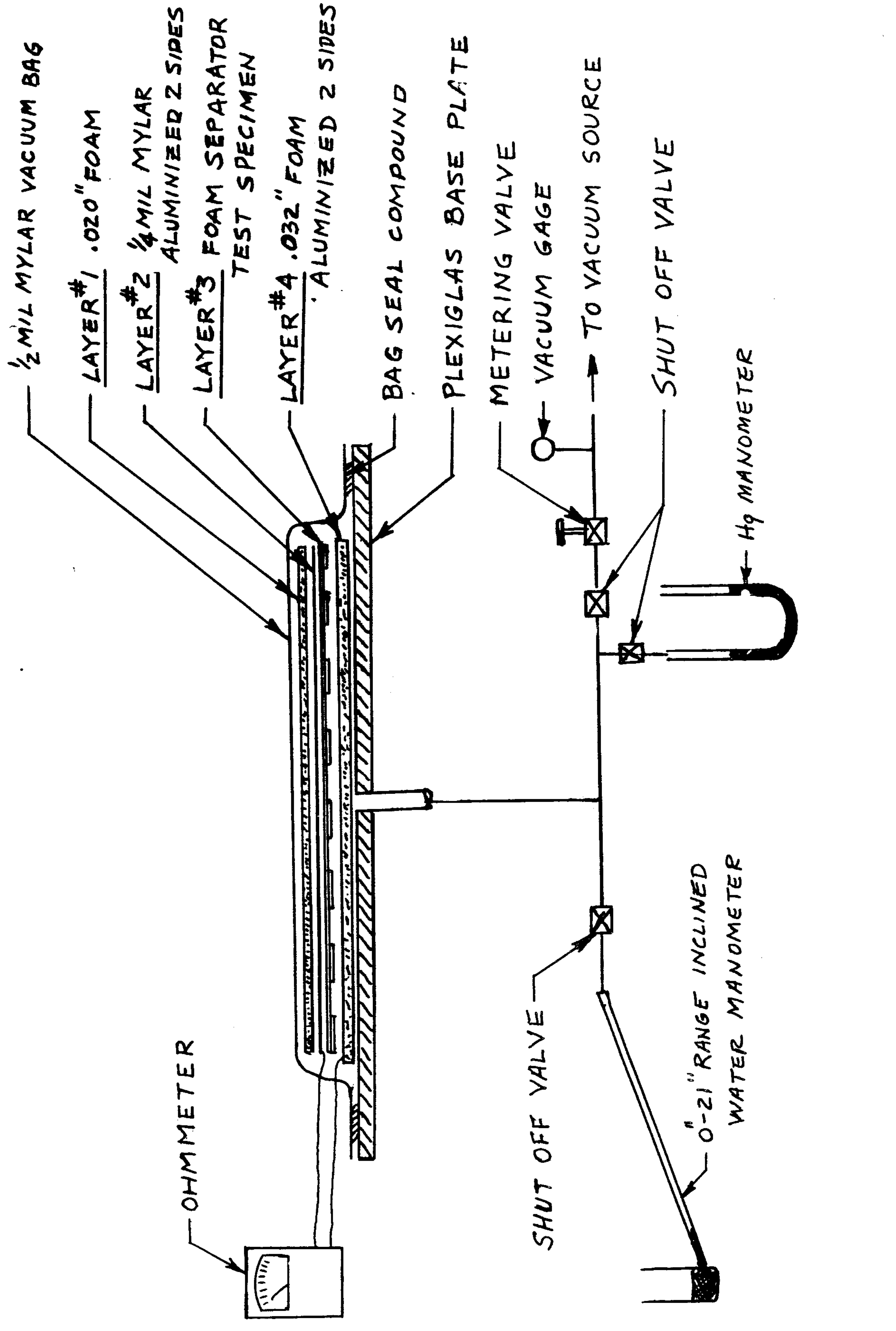


FIGURE 6 SCHEMATIC OF ELECTRICAL SHORT TEST SET UP

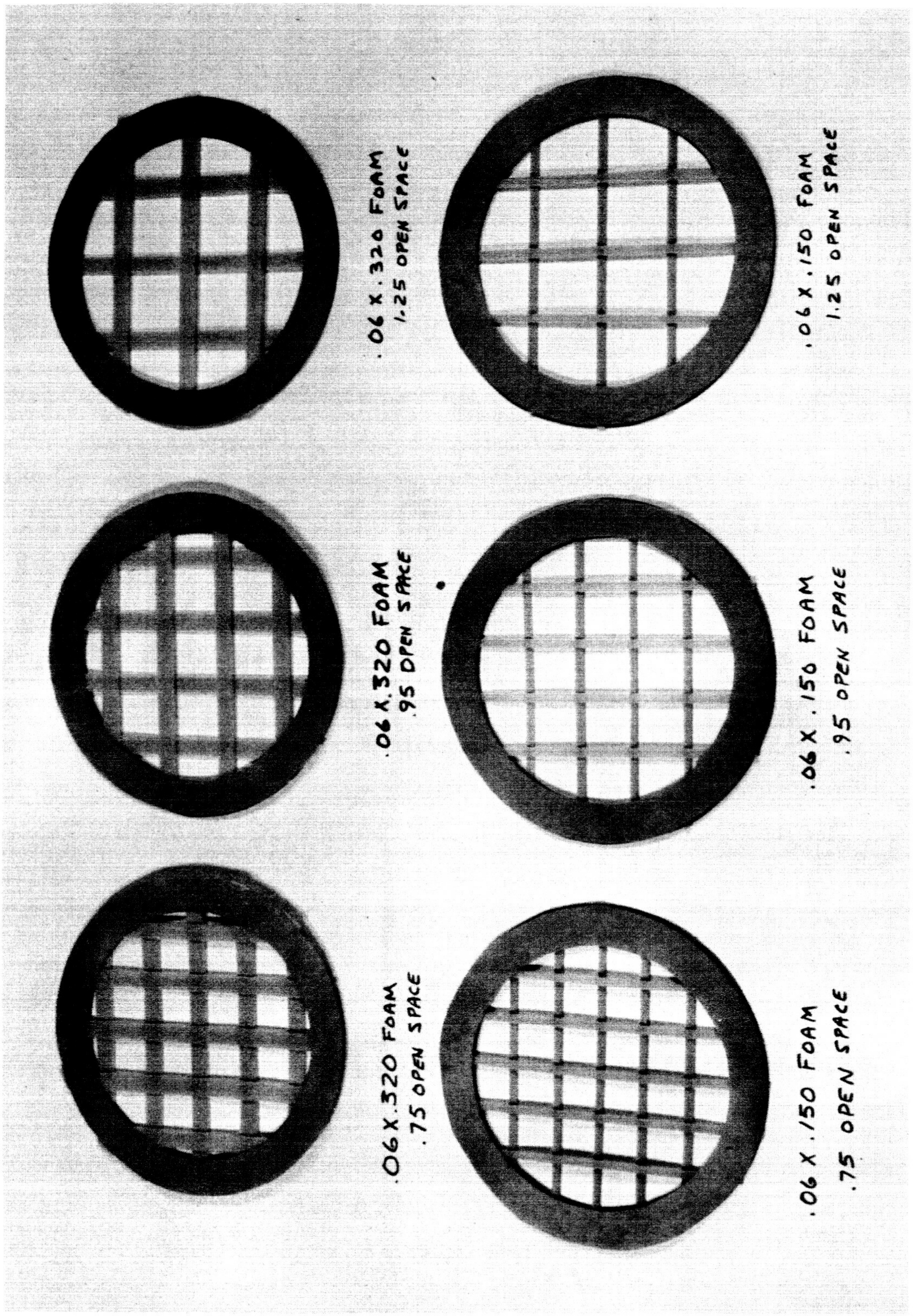


FIGURE 7 - FOAM SEPARATOR STRIP CONFIGURATIONS FOR ELECTRICAL SHORT TESTS

(3) The vacuum pressure is released by backfilling the vacuum bag with air. When the mercury manometer drops to 1.5" Hg the water manometer was opened to the backfill line and monitored during the remaining backfill cycle. The ohmmeter resistivity measurements were simultaneously monitored and the pressure level noted at which the resistivity measurement stabilizes at infinity. Test results are noted in Table II amd IIA.

The selection of configurations for further evaluation in 12 inch diameter calorimeter specimens is based on the criteria of open area of foam separators versus pressure at ∞ resistivity.

It should be noted that the resistivity measurement method of testing for thermal shorts as outlined above is a very conservative technique due to the fact that resistivity measurements are at ∞ when only a minute area of contact exists between the aluminized surfaces that are wired to the ohmmeter. As the vacuum pressure load is being reduced, the amount of contact, and hence the thermal conduction path, between layers, may be very small long before the ohmmeter indicates complete loss of contact.

TABLE II

LIST OF FOAM SEPARATOR SPECIMENS FOR ELECTRICAL SHORT TESTS

Specimen No.	Description of Foam Separators		Percent Open Area
#1	GAC - .060 x .320 strips	.75" x .75" openings	(49%)
#2	GAC - .060 x .320 strips	.95" x .95" openings	(56%)
#3	GAC - .060 x .320 strips	1.25" x 1.25" openings	(63%)
#4	GAC - .060 x .150 strips	.95" x .95" openings	(74.5%)
#5	Mobay -7 .060 x .320 strips	.75" x .75" openings	(49%)
#6	Mobay -7 .060 x .320 strips	.95" x .95" openings	(56%)
#7	Mobay -7 .060 x .320 strips	1.25" x 1.25" openings	(63%)
#8	Mobay -7 .060 x .150 strips	.75" x .75" openings	(69%)
#9	Mobay -7 .060 x .150 strips	.95" x .95" openings	(74.5%)
#10	Mobay -7 .060 x .150 strips	1.25" x 1.25" openings	(80%)

TABLE IIA

FOAM SEPARATOR TEST RESULTS - PRESSURE AT ∞ RESISTIVITY

Test No.	Foam Separator Specimen (See Table II)	Release Pressure - Inches of Water Cycle #1	Cycle #2	Cycle #3	Cycle #4	Cycle #5	Cycle #6	Remarks
1	#1	1.1	.5	.5	.75	.75	.45	See Note #1
2	#2	1.11	.8	.6	.5	.52		
3	#3	.01	.01	.01				
4	#4	.01	.01	.01				
5	#5	10.5		8.0	6.5	3.0	1.5	
6	#6	.85	.20	.20	.20	.25		
7	#5 Repeat #1	1.75*	8.0	10	9.5	12.5		See Notes #2 & #4 *Mylar incorrectly oriented
8	#5 Repeat #2	8.0	9.0	10	10.5	10.5		See Note #3
9	#5 Repeat #3	16	14	14				
10	#6 Repeat #1	1.0	2.0	2.0	2.1	2.1		
11	#7	.01	.01	.01				
12	#8	.5	.4	1.0	1.0	1.0		
13	#9	.01	.01	.01				
14	#10	.1	.1	.08	.1	.1		
15	#5 Repeat #4	6.0	5.5					

NOTES:
1. Reference figure 6 for description of test setup.
2. .020" foam (layer #1) spot bonded to Mylar (layer #2) with G-207 adhesive located in center of each open area of foam separator (layer #3).
3. .020" foam (layer #1) 100% bonded to Mylar (layer #2) on tests 8 thru 15.
4. New ohmeter used on fifth cycle. All subsequent tests conducted with new meter.

The calorimeter test samples described in Table III were constructed for evaluation in the NASA LeRC double-guarded cold-plate thermal conductivity apparatus.

Typical calorimeter samples are shown on Figures 8, 9, 10, and 11.

2. Investigation of Outgassing Characteristics

The foam separators used within sealed insulation panels were investigated to determine fabrication and preconditioning techniques necessary to prevent or reduce outgassing. The outgassing characteristics were evaluated by weight loss measurements in a vacuum environment.

These tests were conducted in the specially constructed test facility shown in Figure 12. The facility consists basically of an automatic recording vacuum balance, pumping system, specimen chamber, and instrumentation. The balance has a capacity of 200 grams and can measure weight changes as low as 0.02 mg (0.00002 gram). A heater is built into the specimen chamber to provide controlled elevated temperatures. Continuous information of weight, temperature, and pressure as a function of time were recorded automatically on strip charts.

TABLE III - COMPOSITION OF
12 INCH DIAMETER CALORIMETER TEST SAMPLES

Calorimeter Sample No. GAC	Calorimeter Sample No. NASA	Layer No.	Material Description	Remarks
1 and 2	1066 and 1067	1	MAM .0015"	3 pc. slices
		2	.020" 2 PCF white GAC polyurethane foam	
		3	¼ mil Mylar aluminized two sides	
		4	Same as 2	
		5	Same as 3	
		6	Same as 2	
		7	Same as 3	
		8	Glass fiber mat .008"	
		9	MAM .0015"	
3	2043	1	MAM .0015"	
		2	.062" 45 PPI Scott Foam-Flexible	
		3	¼ mil Mylar aluminized two sides	
		4	Same as 2	
		5	Same as 3	
		6	Same as 2	
		7	Same as 3	
		8	Glass fiber mat .008"	
		9	MAM .0015"	
4	1070	1	.020 2 PCF white GAC polyurethane foam	One pc. foam slices cut from 300°F P.C. foam block. Slices baked 150°F prior to assembly.
		2	¼ mil Mylar aluminized two sides	
		3-21	Same as 1 and 2 alternate layers	
5	2044	1	MAM .0015"	See Figure #8
		2	.062" 45 PPI Scott Foam-Flexible	
		3	¼ mil Mylar aluminized two sides	
		4	Same as 2	
		5	Same as 3	
		6	Same as 2	
		7	Same as 3	
		8	Glass fiber mat .008"	
		9	MAM .0015"	

TABLE III (CONT.)

Calorimeter Sample No.		Layer No.	Material Description	Remarks
GAC	NASA			
6	2045	1	1 mil Mylar-Aluminized side in.	See Figure #9
		2	.020 2 PCF white GAC polyurethane foam	
		3	¼ mil Mylar aluminized two sides	
		4	Same as 2	
		5	Same as 3	
		6	Same as 2	
		7	Same as 3	
		8	Style 116 glass cloth	
		9	1 mil Mylar-aluminzed side in.	
		10	Same as 2	
		11-19	Same as 1 thru 9	
		20	Same as 2	
		21-29	Same as 1 thru 9	
		30	Same as 2	
7	2046	1	MAM .0015"	
		2	.12" rigidized 10 PPI Scott foam	
		3	¼ mil Mylar aluminized two sides	
		4	Same as 2	
		5	Same as 3	
		6	Same as 2	
		7	Same as 3	
		8	Glass fiber mat .008"	
		9	MAM .0015"	
8	1071	1	MAM .0015"	
		2	.020" 2 PCF white GAC polyurethane foam	Perforated 23% open (1/8" dia holes ¼" centers) See Figure #10
		3	¼ mil Mylar aluminized two sides	
		4	Same as 2	
		5	Same as 3	
		6	Same as 2	
		7	Same as 3	
		8	Glass fiber mat .008"	
		9	MAM .0015"	

TABLE III (CONT.)

Calorimeter Sample No. GAC	NASA	Layer No.	Material Description	Remarks
9	1072	1	1 mil Mylar-aluminized side in	
		2	.020 2 PCF white GAC polyurethane foam	
		3	80 PPI Scott foam .06"x.150" strip	Criss cross
		4	¼ mil Mylar aluminized two sides	pattern 1.45" ope
		5	Same as 2	spacing
		6	Same as 3	
		7	Same as 4	
		8	Same as 2	
		9	Same as 3	
		10	Same as 4	
		11	Style 116 glass cloth	
		12	1 mil Mylar-aluminized side in	
10	2047	1	MAM .0015"	
		2	.10" 10 PPI Scott foam-Flexible	White foam
		3	¼ mil Mylar aluminized both sides	
		4	Same as 2	
		5	Same as 3	
		6	Same as 2	
		7	Same as 3	
		8	Glass fiber mat	
		9	MAM .0015"	
11	2048	1	1 mil Mylar-aluminized side in	
		2	.020 1.8 PCF Carwin Chemical Co. foam	98% open cell rigi
		3	¼ mil Mylar aluminized two sides	by the Upjohn Co.
		4	Same as 2	Carwin Chem. Div.
		5	Same as 3	
		6	Same as 2	
		7	Same as 3	
		8	Style 116 glass cloth	
		9	1 mil Mylar-aluminized side in	
12	1073	1	1 mil Mylar - aluminized side in	
		2	.020 2 PCF Mobay #107754-7 foam	96-97% open cell
		3	¼ mil Mylar-aluminized two sides	rigid polyurethane
		4	Same as 2	foam by the Mobay
		5	Same as 3	Chemical Co.
		6	Same as 2	
		7	Same as 3	
		8	Style 116 glass cloth	
		9	1 mil Mylar-aluminized side in	

TABLE III (CONT.)

Calorimeter Sample No. GAC	NASA	Layer No.	Material Description	Remarks
13	1074	1	1 mil Mylar-aluminized side in	
		2	.020" 2 PCF Mobay #107754-7 foam	Bake foam slices overnight at 150° before assembly
		3	¼ mil Mylar-aluminized two sides	
		4	Same as 2	
		5	Same as 3	
		6	Same as 2	
		7	Same as 3	
		8	Style 116 glass cloth	
		9	1 mil Mylar-aluminized side in	
		10	Same as 2	
		11-19	Same as layers 1 thru 9	
		20	Same as 2	
		21-29	Same as layers 1 thru 9	
		22	Same as 2	
14	1075	1	¼ mil Mylar-aluminized two sides 100% bonded to .020" 2 PCF Mobay #107754-7 foam. Foam side out.	3-M #77 Aerosol Adhesive used in bonding Mylar to foam
		2	.060" 2 PCF Mobay #107754-7 foam cutout to 20% solid area (.150" wide strips with 1.25" open spacing) Pattern #1	With respect to hole pattern #1, pattern #2 has holes shifted ¼ space in x & y axes.
		3	Same as 1	
		4	Same as 2 Pattern #2	
		5	Same as 1	In all cutout layers, the strips are aligned in same direction.
		6	Same as 2 Pattern #1	
		7	Same as 1	
		8	Same as 2 Pattern #2	
		9	Same as 1	
		10	Same as 2 Pattern #1	
		11	Same as 1	See Figure 11
		12	Same as 2 Pattern #2	
		13	Same as 1	
		14	Same as 2 Pattern #1	
		15	Same as 1	
		16	Same as 2 Pattern #2	
		17	Same as 1	
		18	Same as 2 Pattern #1	
		19	.060" 2 PCF Mobay #107754-7 foam	

TABLE III (CONT.)

Calorimeter Sample No. GAC	Calorimeter Sample No. NASA	Layer No.	Material Description	Remarks
15	-	1	.020" 2 PCF Mobay #107754-7 Foam	
		2	¼ mil Mylar-aluminized two sides	
		3	.060" 2 PCF Mobay #107754-7 foam cutouts to 20% solid area (.150" wide strips with 1.25" open spacing)	
		4	Same as 1	
		5	Same as 2	
		6	Same as 3 Pattern #2	With respect to hole Pattern #1,Pattern #2 has holes shifted ½ space in x & y axes
		7	Same as 1	
		8	Same as 2	
		9	Same as 3 Pattern #1	
		10	Same as 1	
		11	Same as 2	
		12	Same as 3 Pattern #2	In all cutout layers, the strips are aligned in same direction.
		13	Same as 1	
		14	Same as 2	
		15	Same as 3 Pattern #1	
		16	Same as 1	
		17	Same as 2	
		18	Same as 3 Pattern #2	
		19	Same as 1	
		20	Same as 2	
		21	Same as 3 Pattern #1	
		22	Same as 1	
		23	Same as 2	
		24	Same as 3 Pattern #2	
		25	Same as 1	
		26	Same as 2	
		27	Same as 3 Pattern #1	
		28	.060" 2 PCF Mobay #107754-7 foam	

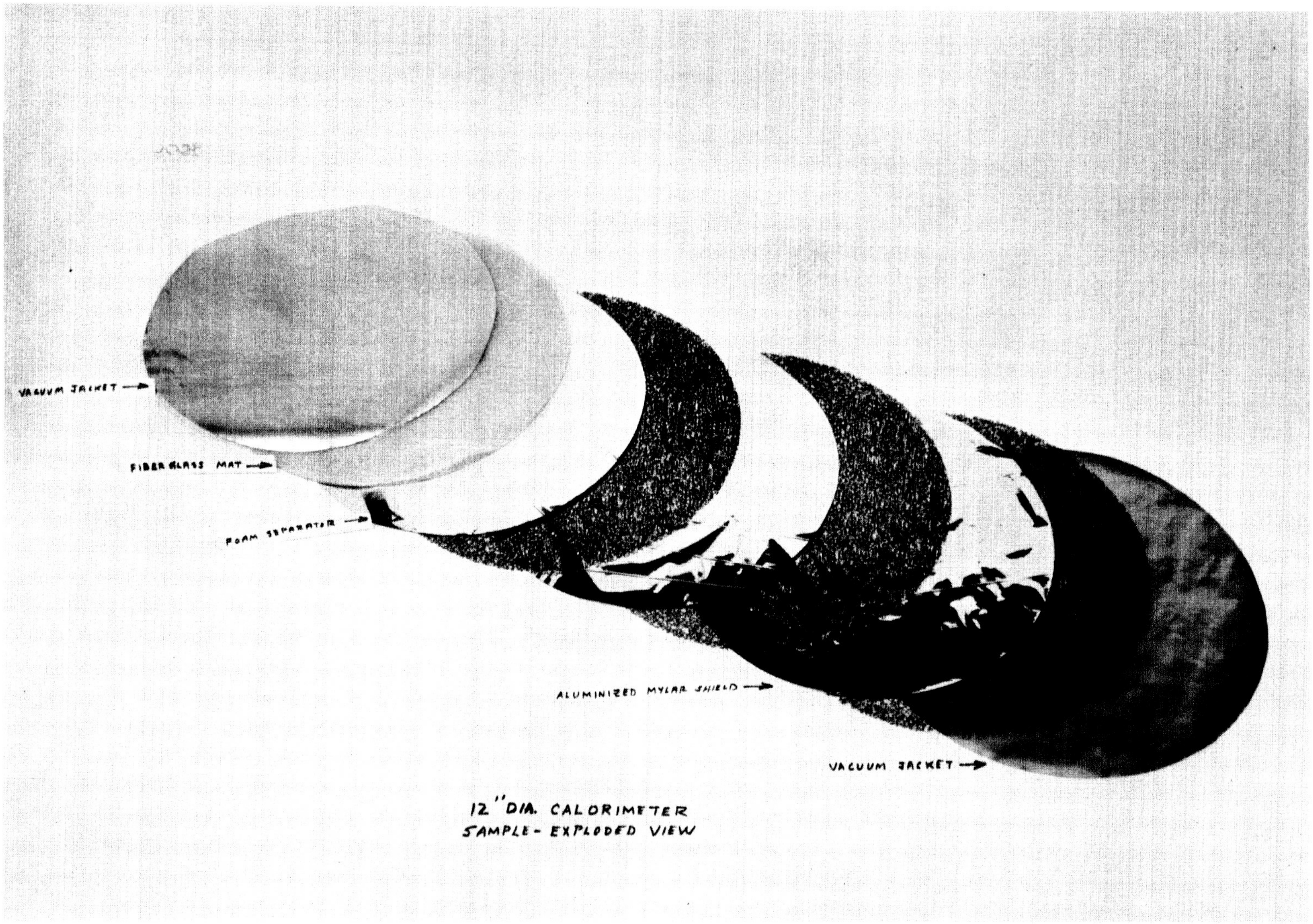


FIGURE 8 - CALORIMETER SAMPLE #5

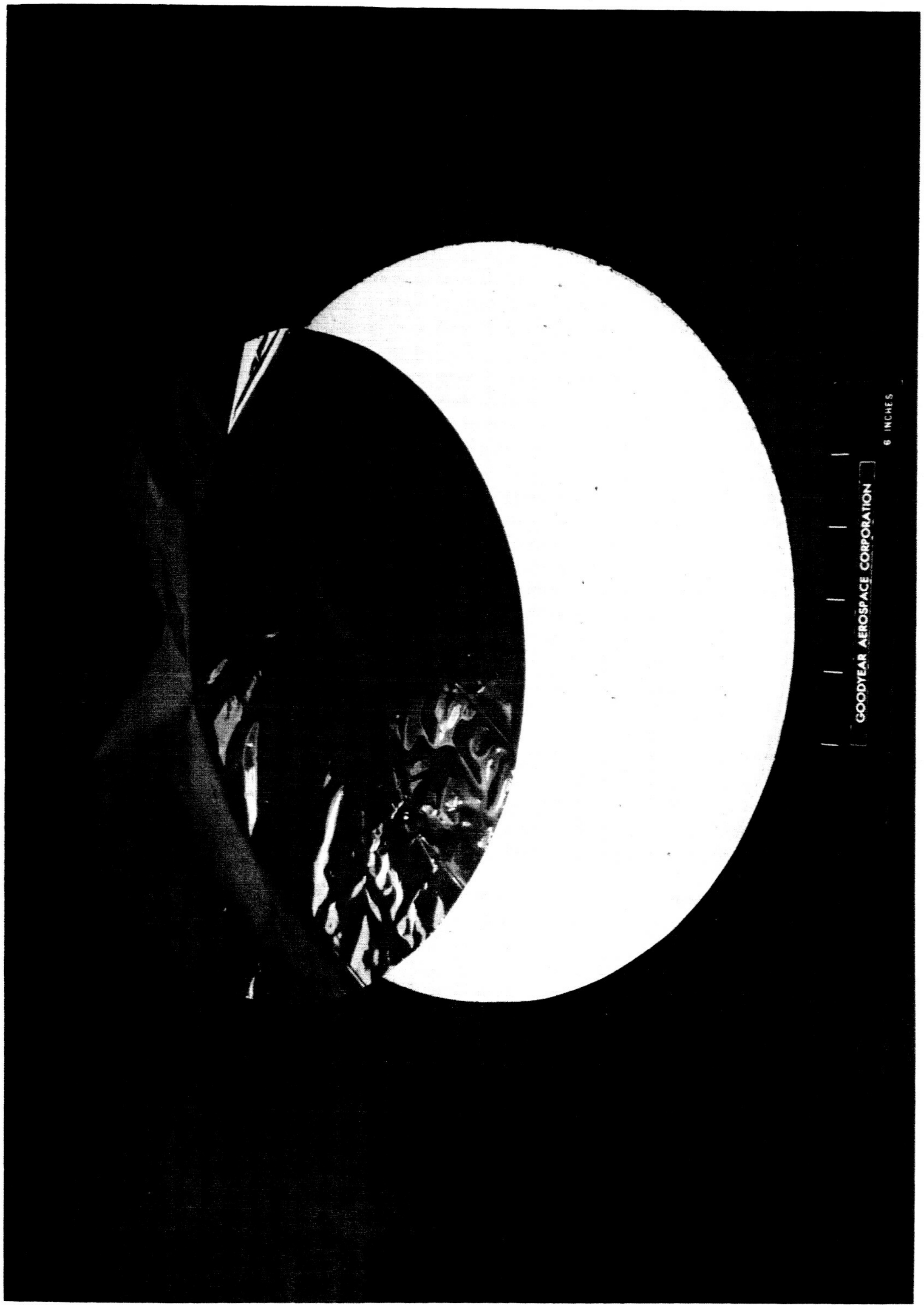


FIGURE 9 - CALORIMETER SAMPLE #6

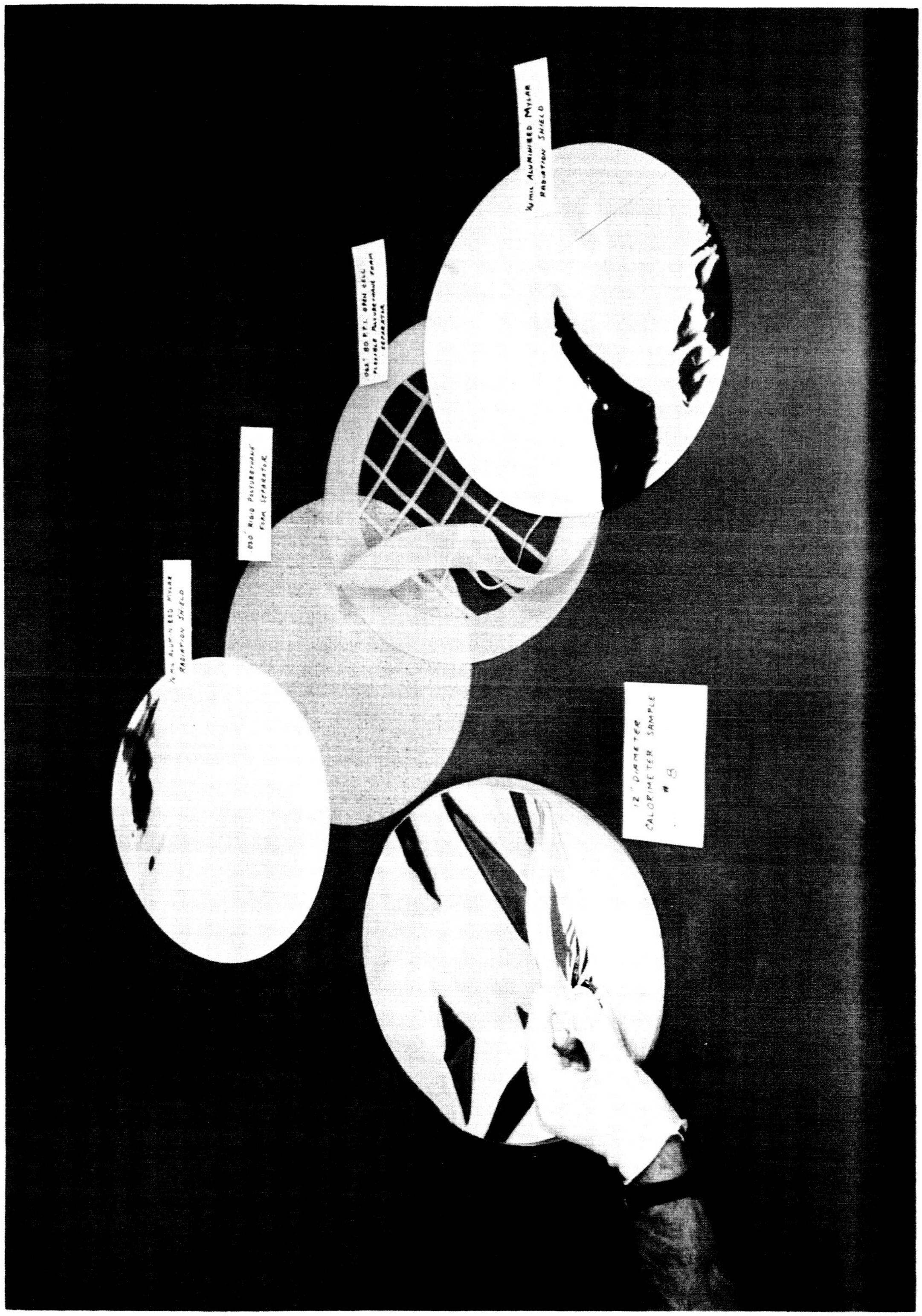


FIGURE 10 - CALORIMETER SAMPLE #8

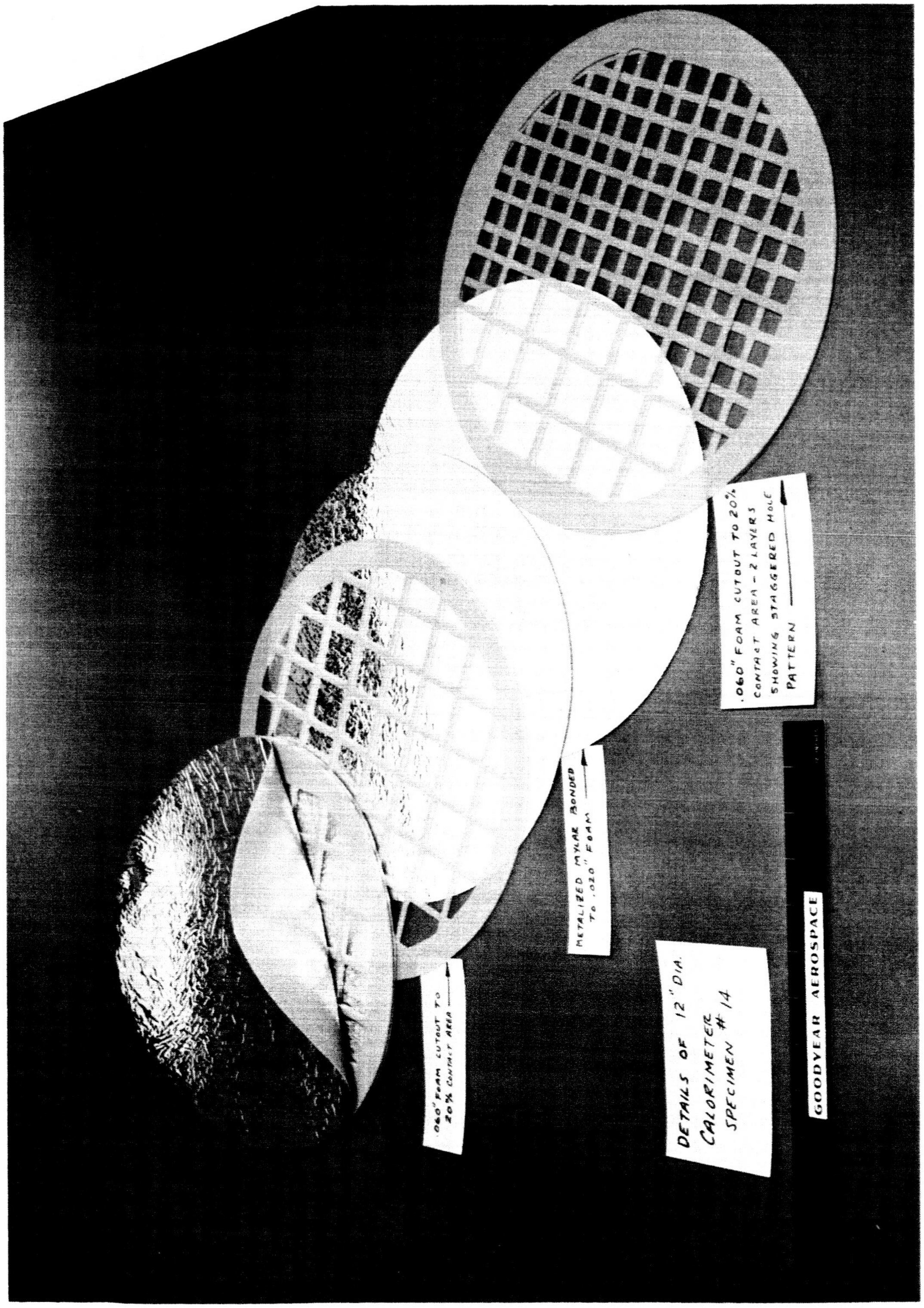


FIGURE 11 - CALORIMETER SAMPLE #14

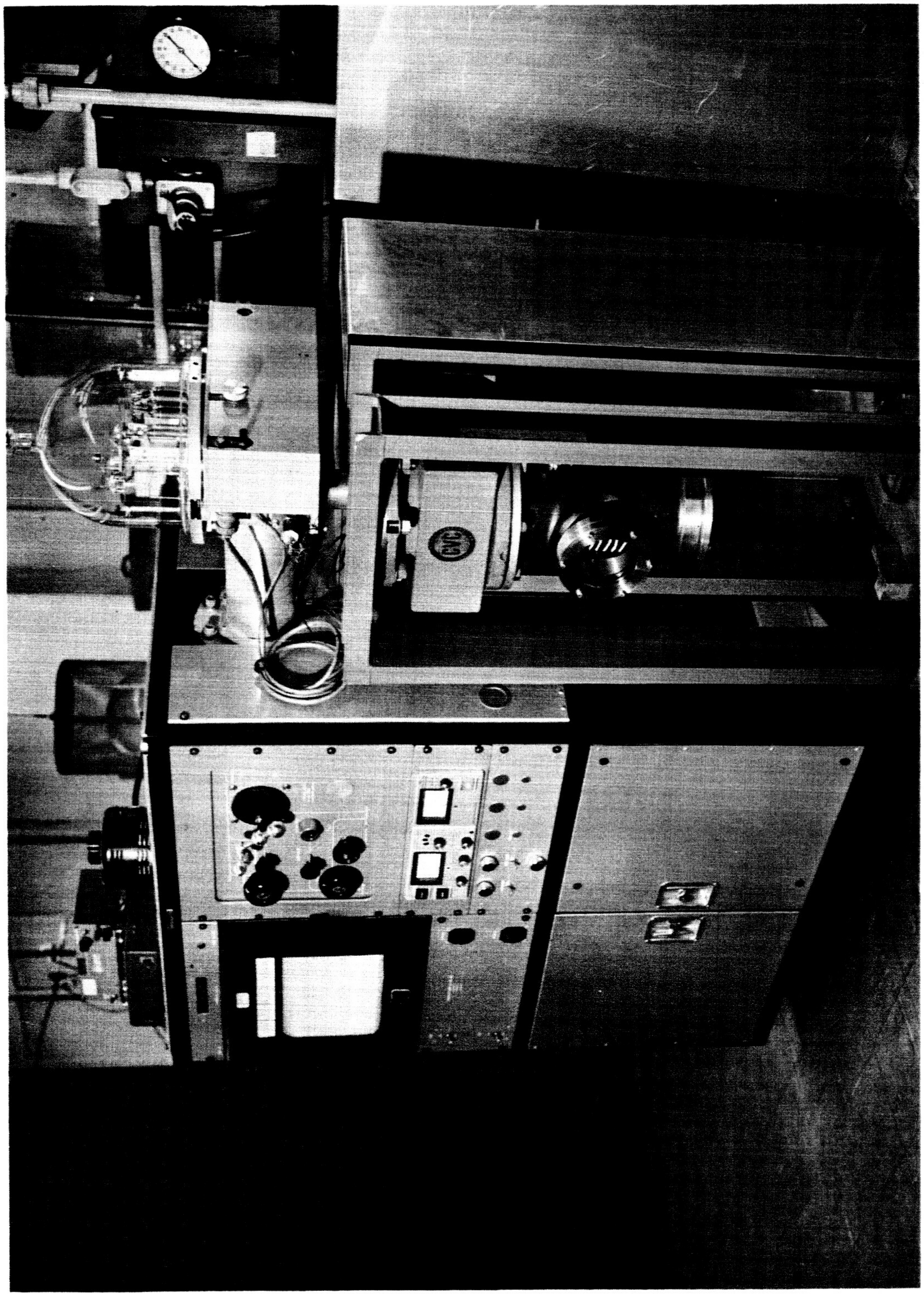


FIGURE 12 - HIGH-VACUUM BALANCE AND TEST CHAMBER

Several preliminary test runs were conducted to establish the test procedure as outlined below:

A. Ambient temperature test

(1) Cut nine strips .020" thick x 2" x 8". Use white cotton gloves in handling foam to prevent finger print contamination of foam.

(2) Precondition foam strips in a controlled 75°F temperature and 45% relative humidity environment for a minimum of 16 hours before test.

(3) Place nine preconditioned specimens in specimen chamber of the high-vacuum balance. Space specimens apart to allow free space between strips.

(4) Record "in air" weight, time, temperature, chart on.

(5) Reduce pressure by roughing to 500 microns- 1 hour.

(6) Reduce pressure to 10 microns, run 1 hour.

(7) Reduce pressure to 5×10^{-4}, run 1 hour (including approach time).

(8) Reduce pressure to 1×10^{-4}, run .5 hour or 1 hour*

(9) Reduce pressure to 5×10^{-5}. " " " " "

(10) Reduce pressure to 1 x 10^{-5}, run .5 hour or 1 hour *

(11) Reduce pressure to 5 x 10^{-6}, " " "

(12) Reduce pressure to ultimate and run overnight.

(13) Record data and secure test.

* Pressure may be reduced when weight loss slope reaches 10 percent.

B. Elevated Temperature Test

(1) Same as ambient test Step (1).

(2) Same as ambient test Step (2).

(3) Same as ambient test Step (3).

(4) Same as ambient test Step (4).

(5) Reduce pressure to 500 microns.

Cool baffles of cold trap with LN_2. Increase temperature of test specimen chamber to 300°F in approximately 1 hour. Hold 500 microns pressure.

(6) Reduce pressure to 10 microns 300°F maintained. *

(7) Reduce pressure to 5 x 10^{-4} 300°F maintained. *

(8) Reduce pressure to 1 x 10^{-4} 300°F maintained. *

(9) Reduce pressure to 5 x 10^{-5} 300°F maintained. *

(10) Reduce pressure to 1 x 10^{-5} 300°F maintained. *

(11) Reduce pressure to 5 x 10^{-6} 300°F maintained. *

(12) Cut off heat and cooling, reduce pressure to ultimate and run overnight.

(13) Record data and secure test.

*Pressure may be reduced in less than 1 hour if weight loss slope reaches 10%.

The test procedures noted above using the principle of weight loss as a function of pressure, temperature, and time have proved to be successful as a screening tool for analyzing the basic outgassing characteristics of various materials. This procedure has enabled detailed examination of the behavior of materials with respect to total weight loss and has provided information concerning the critical areas such as the pressures below which an extremely slow rate of weight change (outgassing) occurs. Although this information is considered to be mandatory in determining preconditioning techniques, additional data must be obtained to more closely compare outgassing characteristics of materials in the environment that results in extremely small weight losses. The automatic weighing system previously described will measure weight changes as small as 0.02 mg and might be considered adequate. However, when this seemingly small weight is converted to volume of gas at pressures of 10^{-5} torr, it is apparent that erroneous conclusions could be reached in the selection of materials in a composite vacuum insulation system. To provide the necessary data to completely define the outgassing characteristics of materials that have been preconditioned, apparatus is required in which a very small rate of outgassing can be readily detected and recorded as a rise in pressure in the vacuum-tight test chamber. This apparatus and test procedure could not be provided within the funds available on this contract.

Four candidate rigid polyurethane foams listed below were evaluated for outgassing characteristics as a function of preconditioning techniques :

(1) 2 pcf white closed cell by GAC.

(2) #107754-3 2.31 pcf open cell by Mobay Chemical Co.

(3) #107754-7 2.18 pcf open cell by Mobay Chemical Co.

(4) TS1080 1.7 pcf open cell by Upjohn Co. Carwin Div.

Test results are summarized on Table IV. Curves of weight loss vs pressure are shown on Figures 13 thru 19. The significant conclusions derived from these tests are:

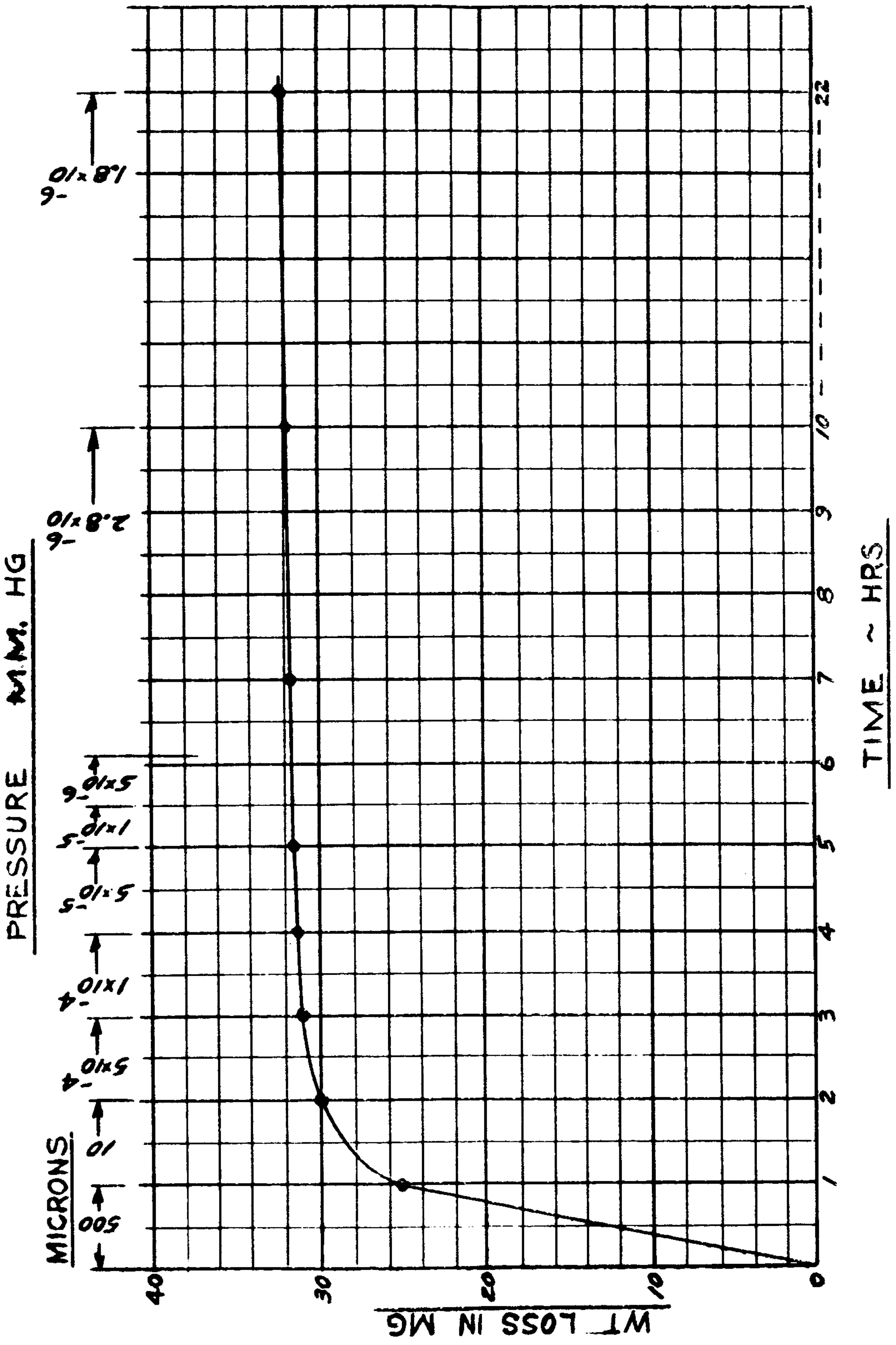
(1) The greatest weight loss occurs in the 500 micron to 10 micron pressure range. In the 10^{-4} to 10^{-6} Torr pressure range the slope of the weight loss curves is below 10 percent and does not change appreciably with reduction of pressure.

(2) Foams subjected to the elevated temperature plus vacuum conditioning experienced greater outgassing weight loss than vacuum conditioning only.

(3) Open cell foams lose weight at a faster rate than closed cell foams.

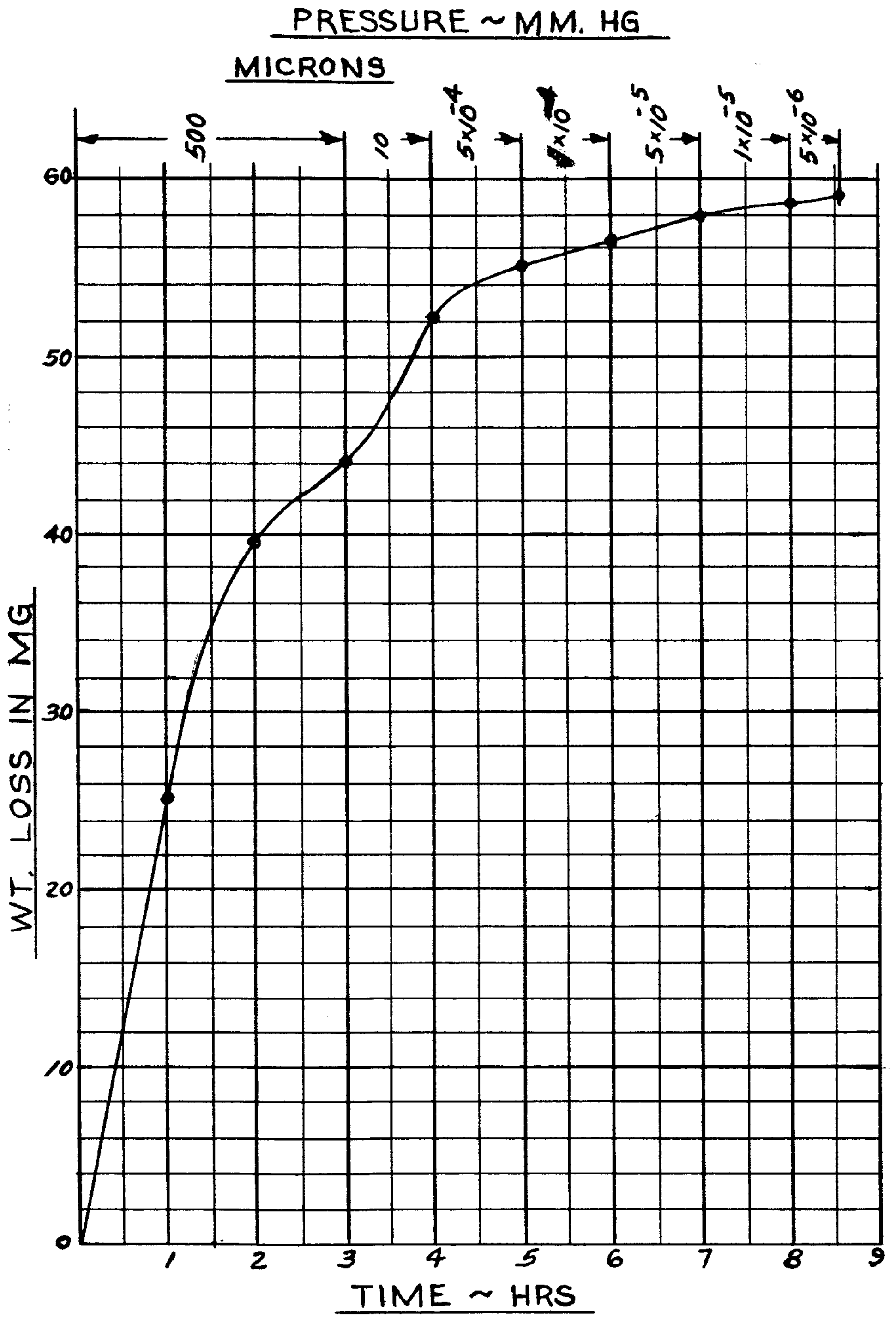
Foam	GAC		Mobay #107754-7		Mobay #107754-3		Carwin #TS1080	
Test Condition	Vacuum Only	Vacuum @ +300°F	Vacuum Only	Vacuum +300°F	Vacuum Only	Vacuum +300°F	Vacuum Only	Vacuum +300°F
Weight In Air (mg)	2695.2	2681.76	3609.7	3548.7	DID NOT RUN	3518.2	3270.5	3257.8
Finish Weight (mg)	2662.92	2622.75	3586.3	3458.51		3449.7	3225.1	3179.7
Weight Loss (mg)	32.28	59.01	23.4	63.19		68.5	45.4	60.1
% Weight Loss	1.21	2.18	0.654	1.78		1.92	1.39	1.85
Weight after N2 Backfill (mg)			3484.28			3448.42		

TABLE IV - SUMMARY OF FOAM WEIGHT LOSS MEASUREMENTS DUE TO VACUUM PRECONDITIONING AT ROOM TEMPERATURE AND 300°F



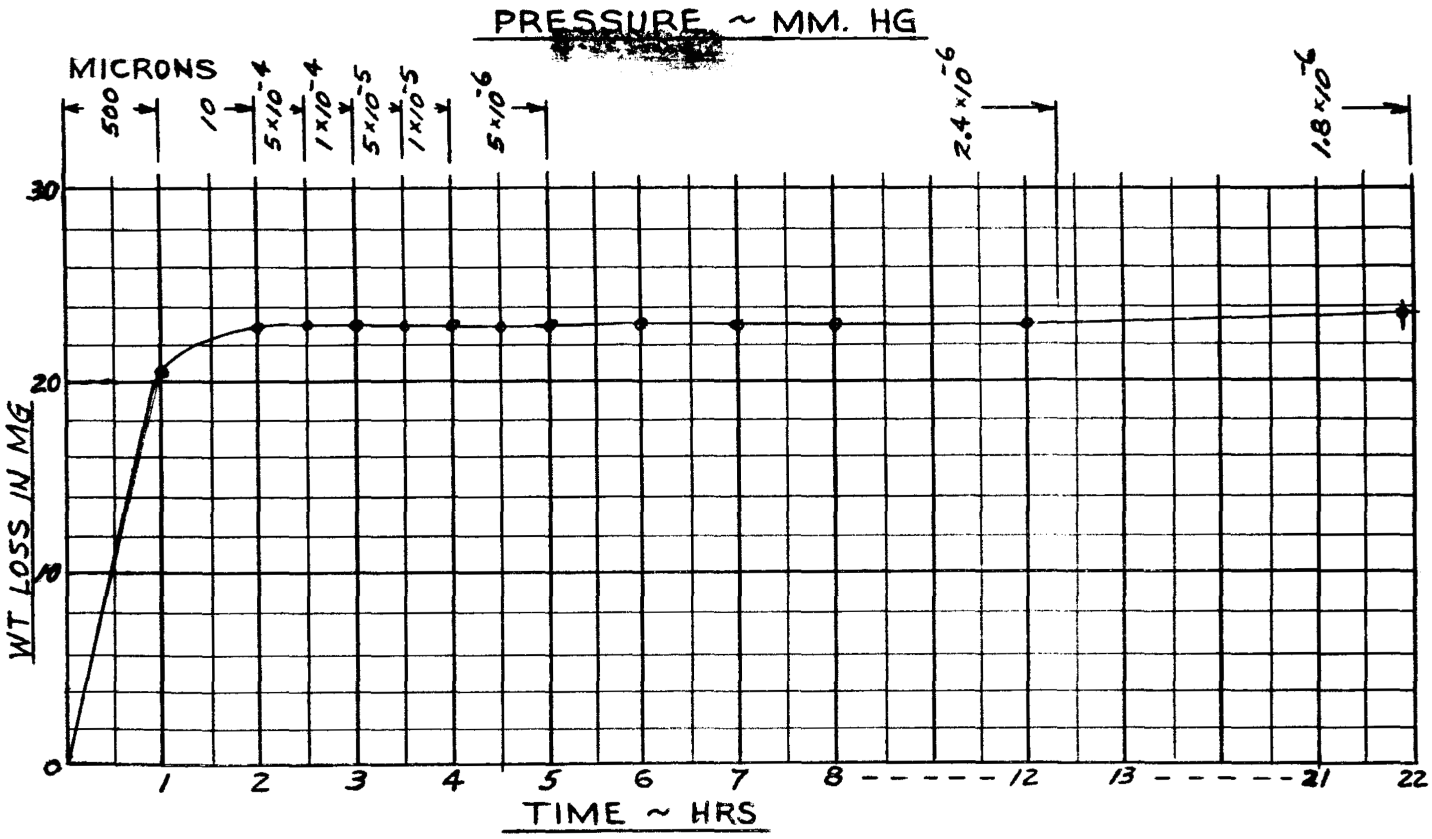
G.A.C. RIGID WHITE FOAM @ AMBIENT TEMP.

FIG. 13



GAC RIGID WHITE FOAM @ 300°F
(TEMP. INCREASED AMBIENT TO 300°F DURING 500μ)

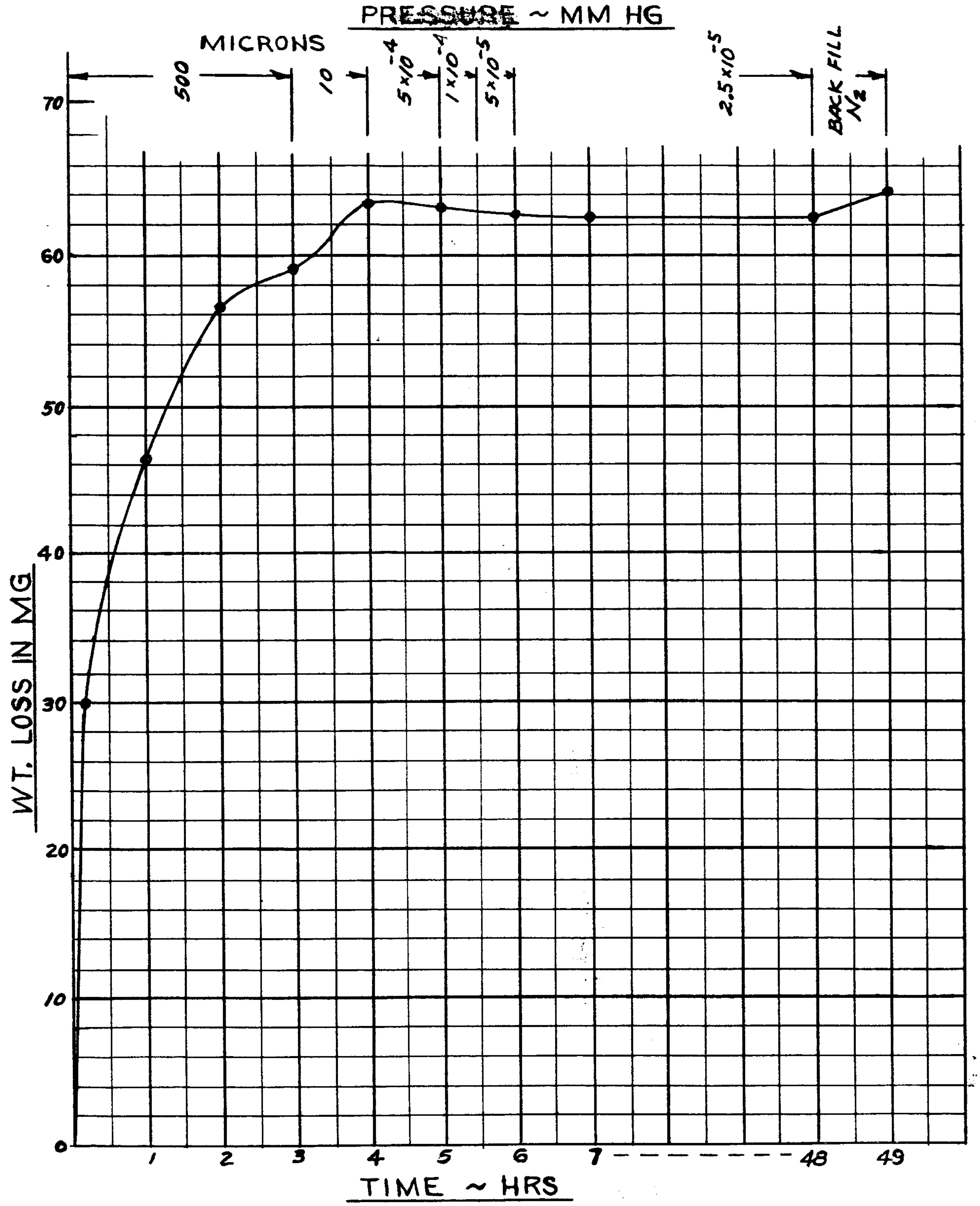
FIG. 14



MOBAY CHEM. OPEN CELL NO 7 @ AMBIENT TEMP.

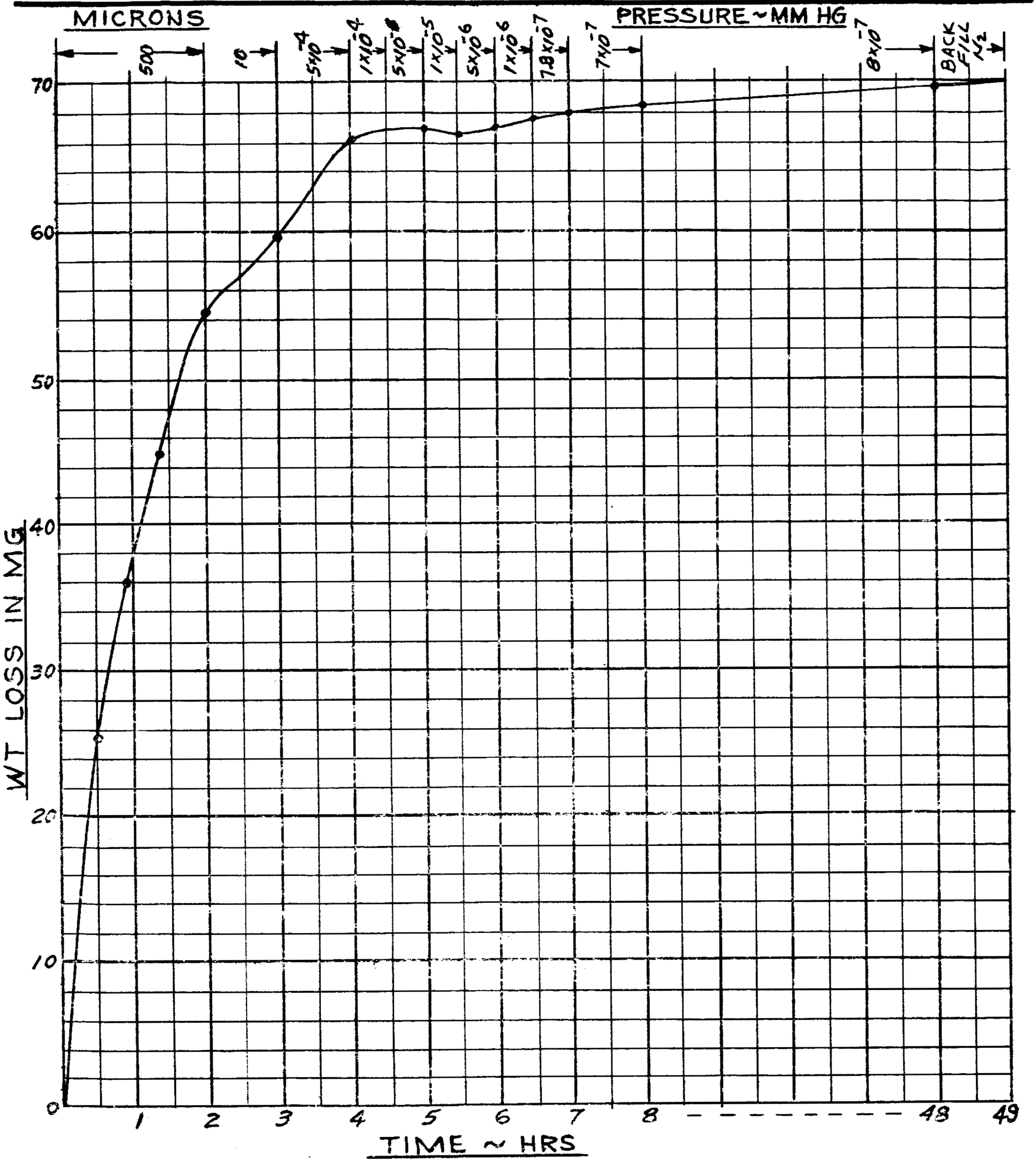
FIG. 15

E-ID-15(7-64)(77-10)
REF: ENGINEERING PROCEDURE S.017



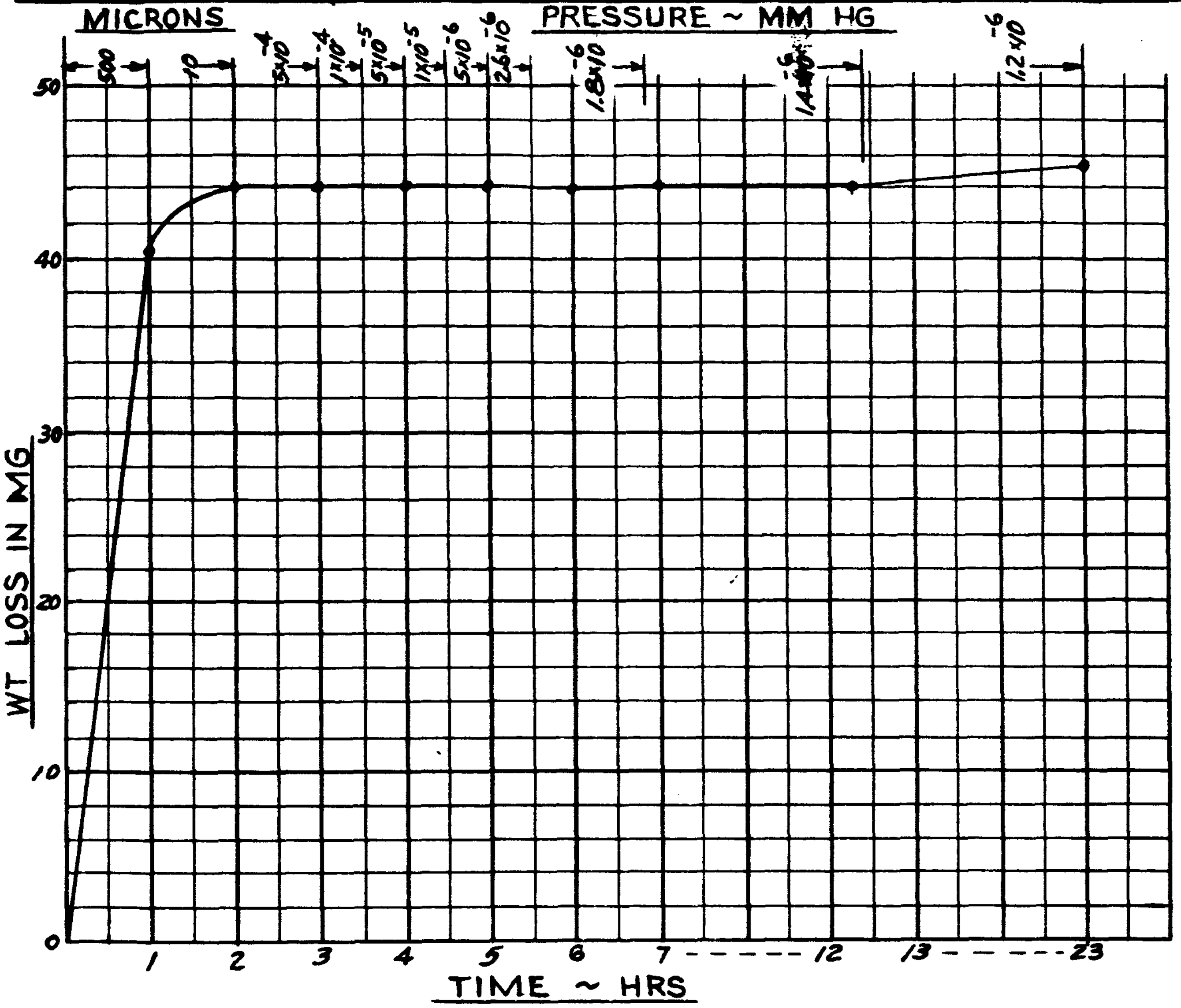
MOBAY CHEM. NO 7 @ 300°F
(TEMP. INCREASED AMBIENT TO 300°F IN 1ST HR)

FIG 16



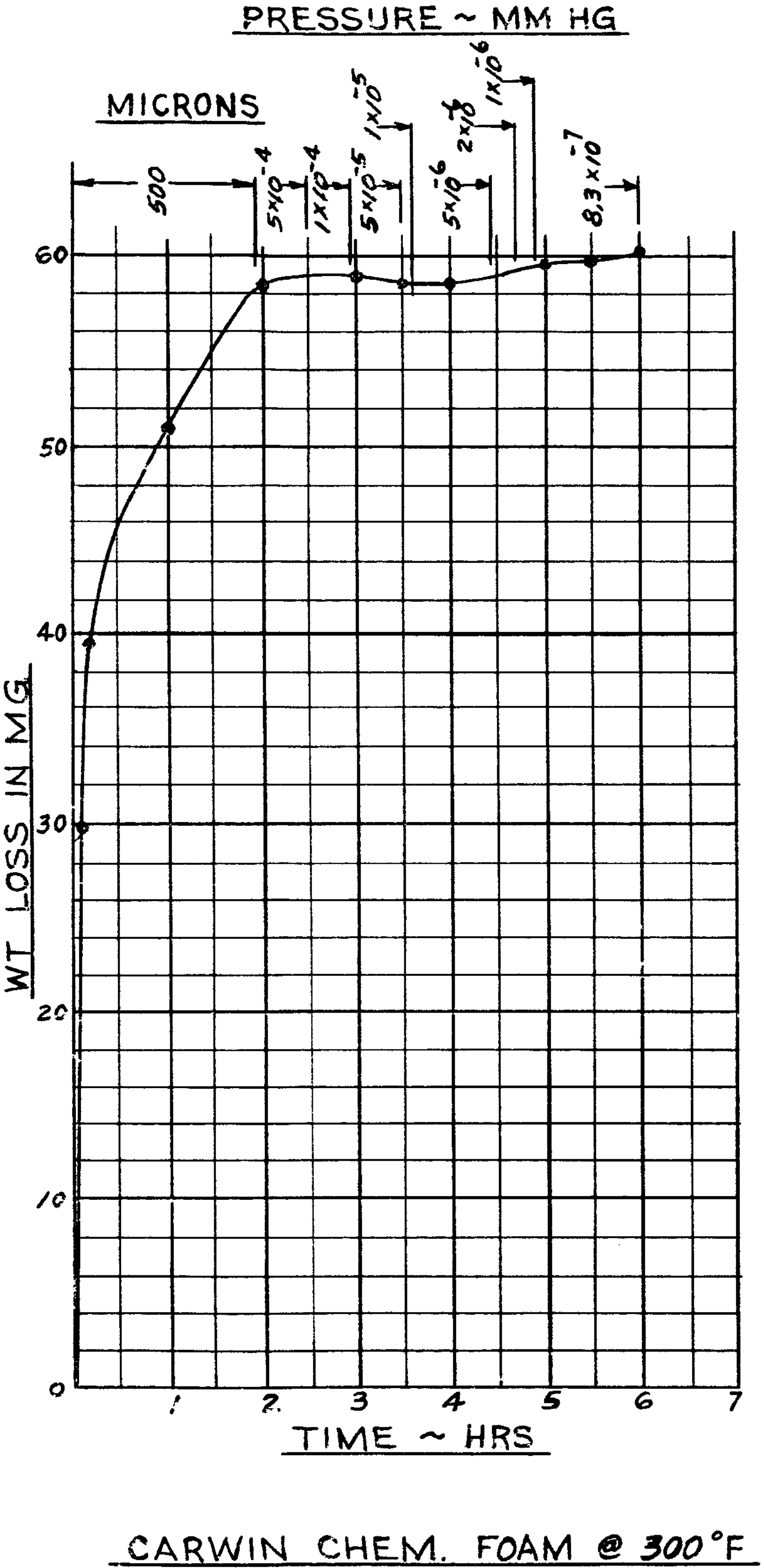
MOBAY CHEM. NO 3 @ 300°F
(TEMP INCREASED AMBIENT TO 300°F DURING 500μ)

FIG. 17



CARWIN CHEM. OPEN CELL RIGID FOAM
AT AMBIENT TEMP.

FIG. 18



CARWIN CHEM. FOAM @ 300°F
(TEMP INCREASED AMBIENT TO 300°F DURING 500μ)

FIG. 19

77-10 (5-63)
REF: ENGINEERING PROCEDURE S-017

(4) The Mobay Chemical Company #107554-7 foam loses approximately 50 percent less weight than GAC or Carwin Chemical foam.

(5) All foams tested do not outgas appreciably at 10^{-4} to 10^{-6} pressure range.

A technical meeting was held at the Research Laboratories of Mobay Chemical Company for the purpose of investigating the possibility of obtaining foam formulations specifically tailored to eliminate elements which theoretically outgas at vapor pressures of 10^{-4} Torr or less. Preparation of open cell foam structures was also discussed and will be covered in subsequent sections of this report.

It was the opinion of the Mobay personnel that there should be little if any outgassing problem with the basic urethane polymers when exposed to space vacuum but there is a definite chance that it will occur with some catalysts, stabilizers and blowing agents. Foams in the past have exhibited outgassing problems but the exact source has not been defined. The following opinions were offered by Mobay on this subject:

(1) Although there are some low molecular weight low boiling portions present in the base resins, once the isocyanate reaction is completed during foaming these portions also become thoroughly tied up in the polymer.

(2) Certain catalysts notably the tertiary amines may remain as unreacted volatiles in the foam. This can be overcome by substituting a reaction amine such as methyl diethanolamine and/or possibly metal organic catalysts such as dibutyl tin dilaurate.

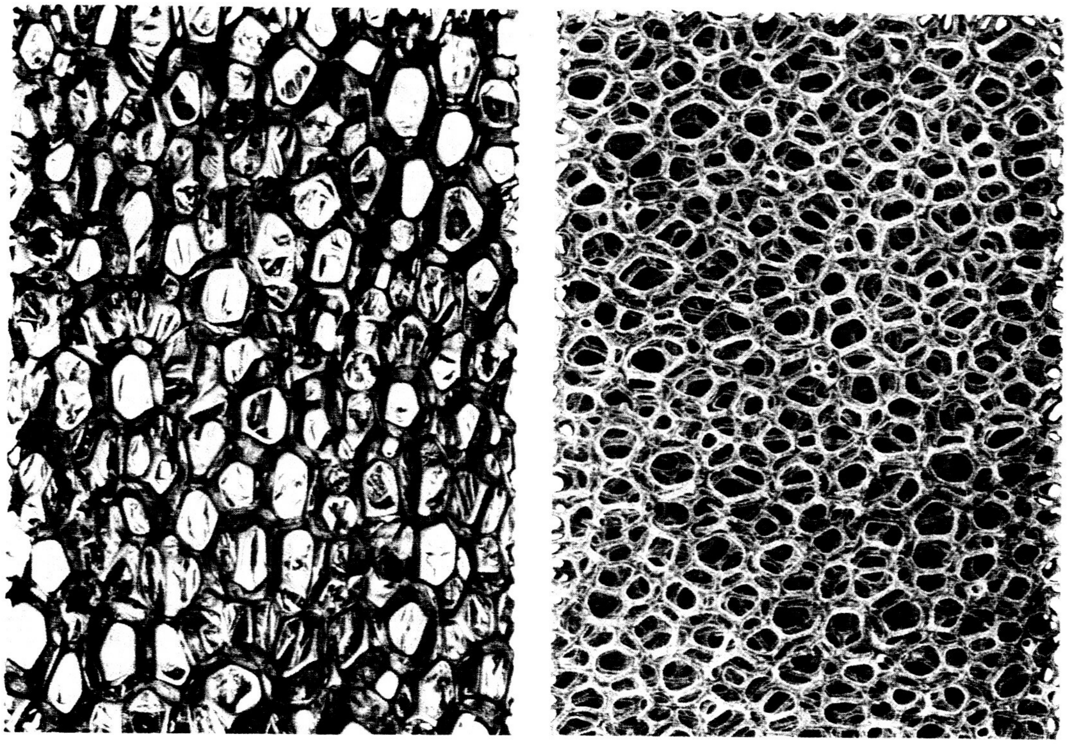
(3) Some of the non reactive stabilizers such as some silicones may volatilize in a vacuum but there are silicone compound with reactive hydroxyl group that can be tied in to the polymer.

(4) Any blowing agent when trapped in a closed cell can diffuse through the cell walls when exposed to vacuum. In fact CO_2 generated by the water reaction in foams will diffuse readily at atmospheric pressure. An open cell foam would provide rapid exit for the gas in the cells.

3. The Effects of Foam Cell Structure

The desirability of an open cell structure foam for self-evacuating insulations has been previously explained. To obtain a rigid open cell polyurethane foam, two basic approaches were investigated:

(1) Rigidization of open cell flexible polyurethane foam. The flexible polyurethane foams are characteristically open cell or reticulated, some having a greater degree of reticulation than others. For the purpose of this investigation a 100 percent reticulated flexible polyurethane foam manufactured by the Foam Division of Scott Paper Company was chosen for the rigidizing experiments. The reticulated structure of this foam is shown on Figure 20 in comparison with the structure of a conventional closed cell rigid foam. The reticulated foam possesses the ideal characteristics of minimal surface area for outgassing and low impedance to the flow of cryopumped gases. The reticulated foam structure is however soft and sponge-like due to its basic flexible foam formulation and the elimination of structural rigidity associated with closed cell foams. The flexibility of the foam is undesirable due to excessive deflection when compressively loaded to one atmosphere pressure. The foam also will stiffen when exposed to the cold wall of a cryogenic tank. Simultaneous occurance of these undesirable features will increase the insulation density and rate of heat transfer.



CLOSED

OPEN

FIGURE 20 - OPEN- AND CLOSED-CELL FOAM STRUCTURES

Several approaches have been made to the problem of rigidizing the reticulated Scott foam.

(a) Scott commercially offers two grades of foam that have been resin coated to alter either the stiffness or the chemical characteristics. One type was a polyehtylene coated 10 pore per inch foam sold under the Skelcoat trade name and AM 175 foam was a 45 pore per inch foam coated with a stiff urethane (Adiprene) elastomer. Although both samples showed a marked increase in stiffness over the original foam, the compressive strength was still low by rigid foam standards. The density of the rigidized foam was approximately three times greater than untreated foam.

(b) Several samples of reticulated foam from both Scott and Chemotronics were impregnated at GAC with a moisture curing urethane varnish PP-1502-E6 by Wyandotte Chemical. Increased stiffness was obtained with a lower weight penalty than that of the Scott samples. However the cure rate was very slow since it was dependent of moisture in the air.

(c) The third approach to rigidizing flexible foam was illustrated on samples supplied by Mobay Chemical. On these a rigid foam formulation was prepared without blowing agent and diluted with ketone solvents to impregnate a flexible foam. Of the three methods this appeared to be the most efficient but still was not equivalent to a rigid foam in compression strength on an equal weight basis.

Considering the factors of increased density and relatively high flexibility after rigidization, further efforts to rigidize flexible foams were curtailed in favor of more promising efforts of obtaining rigid foams with open cells.

(2) Fabrication of rigid polyurethane foams having a high percentage of open cells.

A vendor survey was conducted to obtain a source of rigid polyurethane foam having the desirable open cell configuration. Response was limited to two vendors due to the unusual nature of the requested foam. Vendors supplying samples were the Carwin Chemical Division of the Upjohn Company and the Mobay Chemical Company. Both vendors supplied rigid foams with greater than 90 percent open cells which were open by virtue of ruptured cell walls rather than the completely removed cell walls of reticulated foam.

As noted earlier in this report, the outgassing tests showed the open cell rigid foams to lose weight faster than closed cell foam. This desirable feature is attributed to the ruptured cell walls allowing rapid escape of gasses within the cells.

The Mobay Chemical Company open cell rigid foams exhibited better mechanical properties than the Carwin Company foam. The formulation for the Mobay Company foam was basically a rigid foam formulation which achieved the open or ruptured cell walls by the introduction of a controlled amount of aluminum flake with little deleterious effect on the mechanical properties of the foam. The Carwin foam formulation was proprietary and without this formulation it can only be conjectured that the open cell wall was achieved by a chemical adjustment of the foam formulation which created a weak and more fragile foam. The Carwin foam was difficult to cut into thin slices whereas the Mobay foams were cut into thin slices as readily as conventional rigid closed cell foam. The Mobay foam formulations for samples received are as follows:

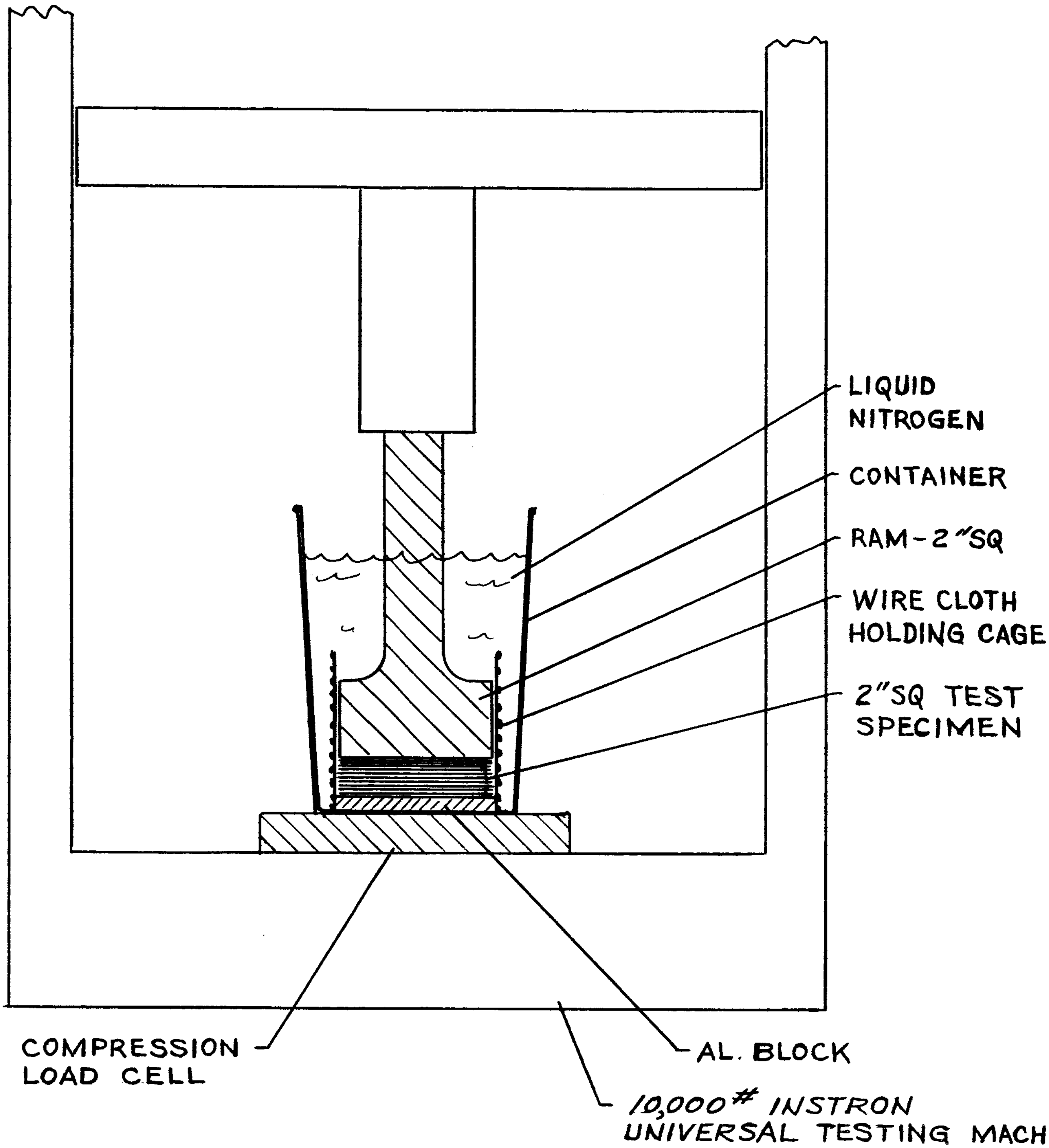
Sample No. 107754-3		107754-7	
Matl Designation	Parts by weight	Matl Designation	Parts by weight
DMG 460 Polyol	100.0	PeP 450 Polyol	100.0
F-11 Blowing Agent	30.0	F11 Blowing Agent	30.0
L-5320 Stabilizer	2.0	L530 Stabilizer	2.0
TMBDA Catalyst	1.5	TMBDA Catalyst	1.5
#422 Aluminum Cell Opener	0.5	#422 Aluminum Cell Opener	1.0
Mondur MR Isocyanate	115.0	Mondur MR Isocyanate	136.0

The choice of stabilizer, level of stabilizer and level of aluminum are very important in preparing an open cell foam which will not collapse. The PeP 450 system (107754-7) is the better of the two largely because of better molding characteristics.

4. Compression Characteristics

The compression characteristics of candidate foam material in slices form were evaluated on test specimens comprised of alternating layers of .020" thick foam slices and 1/4 mil aluminized Mylar. The test samples measures 2" x 2" in area and were approximately .350" total thickness of ten layers of foam and nine layers of Mylar. Test procedure:

(1) Place multi layer sample in the holding cage of an open mouthed container mounted on load cell of 10,000# Instron Universal Testing machine as shown on Figure 21.



COMPRESSION TEST SET UP
FOAM & FILM SAMPLES

FIG. 21

(2) With the specimen at room temperature, at a crosshead speed of .10" per minute, apply compression load on sample to 15 psi (60# total load) and return to 0 psi. Record load, deflection and recovery data on strip chart.

(3) Allow five minute recovery of specimen at zero load at room temperature. Repeat load cycle as described in (2).

(4) Repeat operation (3) for third room temperature compression test.

(5) Allow five minute recovery of specimen at zero load at room temperature. Fill container with LN_2 and keep specimen immersed for 10 minutes before applying compressive load. With specimen immersed in LN2 apply compression load cycle as described in (2). Allow specimen to warm up to room temperature overnight.

(6) Repeat operation (2) for fourth room temperature cycle.

The following foams were evaluated by the technique described above:

GAC white 2 pcf rigid polyurethane
Mobay Chemical #107754-7 open cell rigid polyurethane
Mobay Chemical #107754-3 open cell rigid polyurethane
U. S. Gypsum "Zero Cell" 1.7 pcf rigid polyurethane
Carwin Chemical #TS 1080 open cell rigid polyurethane

Test specimen thickness as a function of load is tabulated in Table V. Typical load-deflection curves for room temperature and -320°F tests are shown on Figures 22 and 23. An ultimate compression load-deflection curve for Mobay 107754-7 foam is shown on Figure 24.

Conclusions derived from the compression tests of foam-film composites are:

(1) At room temperature and at -320°F all specimens exhibited elastic recovery after release of 15 psi compressive load. (Figures 22 and 23).

(2) The modulus of the specimens increases with decrease in temperature (Figure 23).

(3) Closed cell foams are more rigid than open cell foams (Figure 22).

(4) Of the open cell foams tested, the Mobay Chemical #107754-7 is structurally superior and has 50 percent less deflection than the Carwin Chemical foam at 15 psi.

(5) The ultimate compression test of Mobay #107754-7 foam at -320°F showed a yield point at approximately 32 psi (See Figure 24). This value is slightly greater than twice the maximum pressure on self evacuating insulation panels.

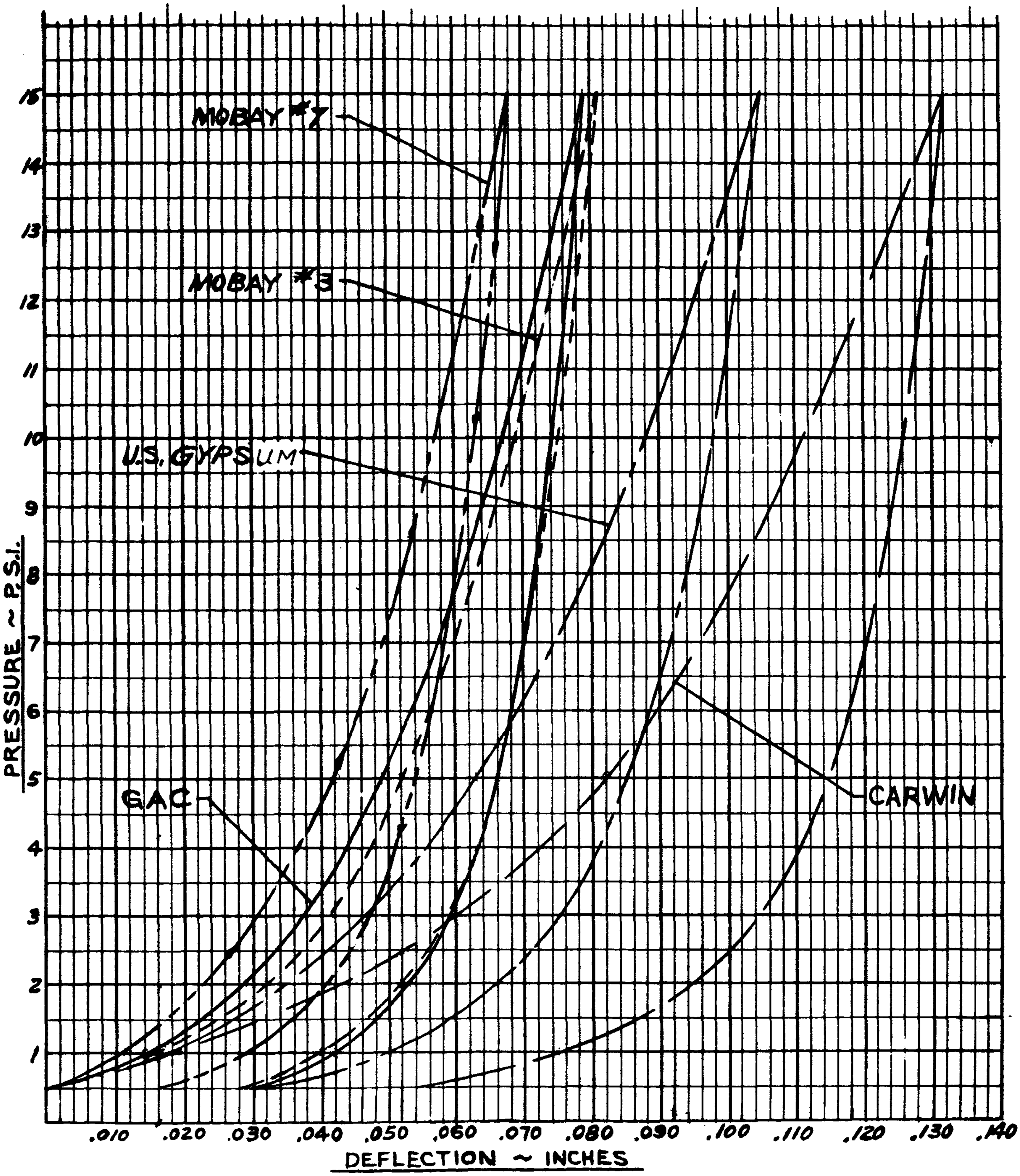
TABLE V

COMPRESSION TEST VALUES - 2" x 2" FOAM AND FILM COMPOSITE SPECIMENS

		Test Specimen Thickness				
Type Foam	PSI	RT #1	RT #2	RT#3	-320°F	RT#4
GAC	0.5	.419	.353	.344	.338	.360
	1.0	.393	.339	.330	.325	.346
	2.0	.365	.323	.315	.312	.328
	5.0	.328	.301	.295	.293	.303
	10.0	.297	.282	.277	.279	.281
	15.0	.272	.267	.265	.270	.265
	10.0	.277	.272	.270	.276	.270
	5.0	.286	.281	.278	.287	.278
	2.0	.300	.294	.291	.302	.291
	1.0	.314	.306	.302	.314	.302
	0.5	.329	.318	.314	.329	.313
Mobay #107754-7	0.5	.315	.297	.292	.278	.301
	1.0	.301	.286	.281	.270	.291
	2.0	.286	.272	.269	.260	.277
	5.0	.263	.253	.250	.246	.257
	10.0	.243	.237	.235	.234	.237
	15.0	.226	.225	.224	.226	.222
	10.0	.232	.230	.229	.232	.227
	5.0	.241	.239	.238	.241	.236
	2.0	.255	.253	.251	.253	.249
	1.0	.267	.264	.263	.262	.260
	0.5	.278	.276	.276	.270	.269
Mobay #107754-3	0.5	.344	.301	.290	.287	.306
	1.0	.320	.283	.275	.271	.288
	2.0	.293	.265	.258	.255	.268
	5.0	.259	.242	.238	.236	.244
	10.0	.232	.223	.221	.220	.223
	15.0	.212	.211	.209	.211	.207
	10.0	.217	.216	.215	.217	.212
	5.0	.227	.225	.224	.228	.221
	2.0	.240	.239	.238	.243	.234
	1.0	.255	.251	.250	.256	.245
	0.5	.270	.265	.262	.270	.255

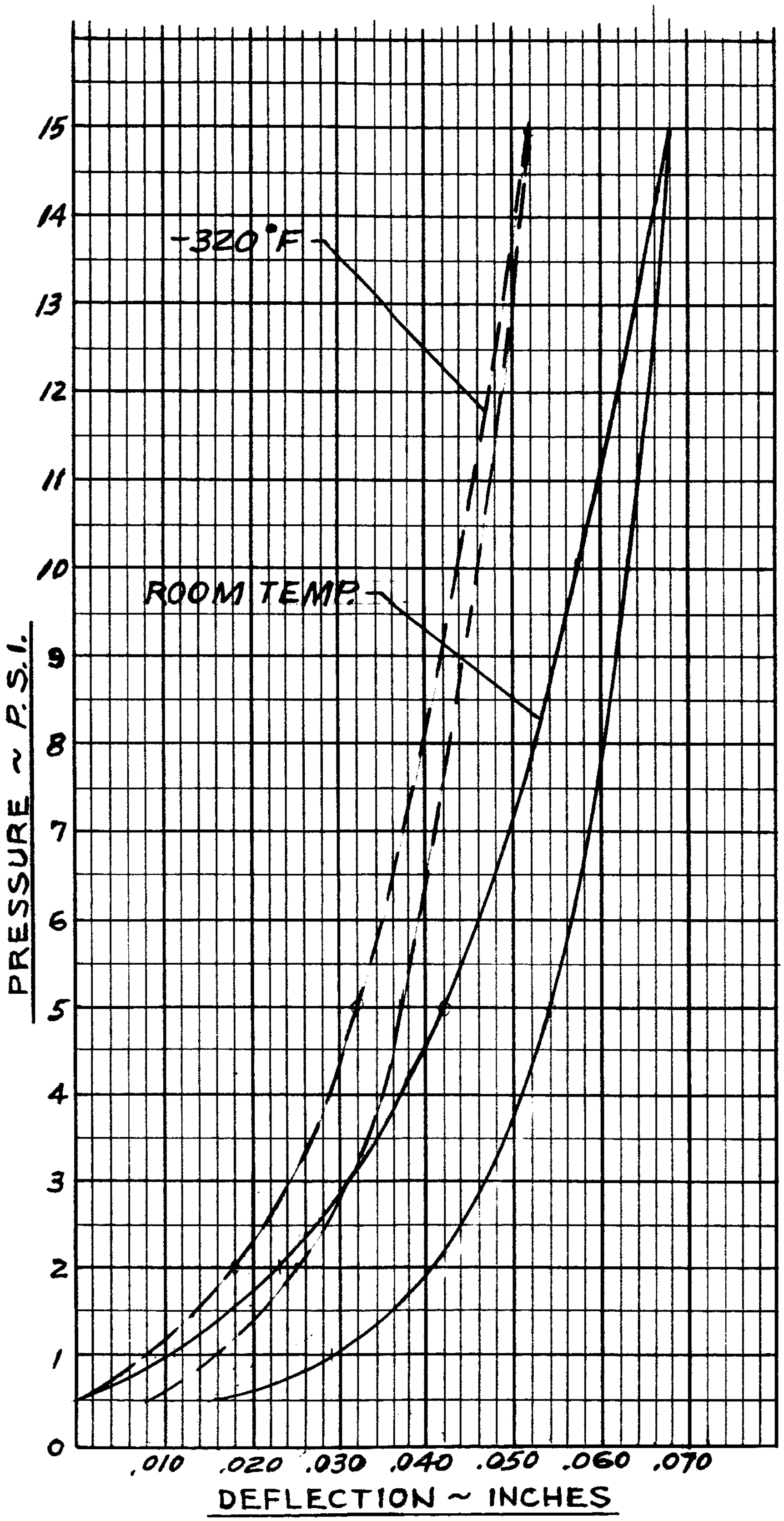
TABLE V (CONT.)

Type Foam	PSI	Test Specimen Thickness				
		RT #1	RT #2	RT #3	-320°F	RT #4
U. S. Gypsum Zero Cell	0.5	.362	.310	.300	.291	.320
	1.0	.340	.294	.283	.280	.305
	2.0	.321	.276	.265	.263	.287
	5.0	.290	.246	.237	.237	.255
	10.0	.251	.218	.212	.217	.221
	15.0	.201	.197	.195	.204	.197
	10.0	.208	.205	.202	.213	.205
	5.0	.222	.218	.215	.229	.218
	2.0	.242	.237	.234	.249	.237
	1.0	.260	.255	.250	.265	.255
	0.5	.278	.270	.270	.277	.270
Carwin TS 1080	0.5	.490	.396	.372	.350	.370
	1.0	.470	.379	.355	.338	.350
	2.0	.445	.355	.329	.321	.323
	5.0	.409	.312	.290	.294	.290
	10.0	.352	.275	.261	.268	.267
	15.0	.259	.246	.240	.252	.249
	10.0	.264	.252	.246	.260	.253
	5.0	.277	.264	.257	.276	.263
	2.0	.299	.285	.277	.300	.279
	1.0	.321	.305	.297	.317	.296
	0.5	.345	.327	.318	.330	.315



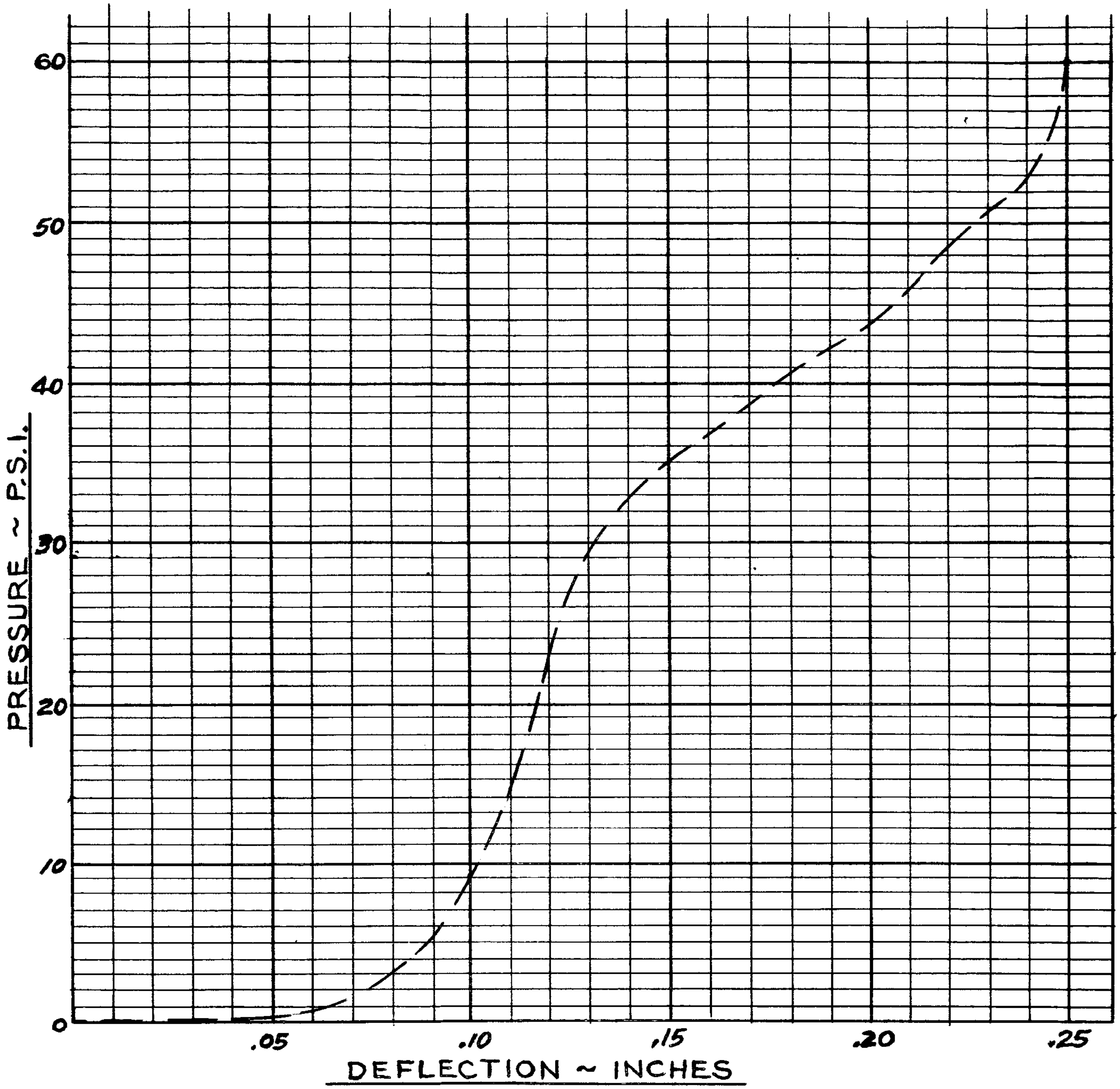
TYP. COMPRESSION LOAD ~ DEFLECTION CURVES
FOAM & FILM AT ROOM TEMP

FIG. 22



TYP. COMPRESSION LOAD-DEFLECTION CURVES
MOBAY #7 FOAM ¢ FILM AT -320°F ¢ ROOM TEMP

FIG. 23



ULTIMATE COMPRESSION CURVE
MOBAY NO.7 FOAM & FILM AT -320°F

FIG. 24

C. Vapor Barrier Materials and Fabrication Techniques Investigation

1. Permeability Characteristics

To avoid duplication of effort and divert funds to other important areas of the contract, the task of conducting helium gas permeability tests on candidate vapor barrier materials was deferred by mutual agreement between NASA and GAC technical representatives. The vapor barrier helium permeability data generated by GAC during the development of a flexible vacuum jacket under NASA Contract NAS 8-11376 has provided sufficient test data to permit the selection of an improved vapor barrier material. The maximum allowable helium permeability rate for qualification of the vacuum jacket is 2.2×10^{-10} cc helium per second per square inch at standard atmosphere pressure and temperature. On the basis of over 240 helium permeability tests, the following vapor barrier material selections are made in decending order of preference:

(1) MAAM (Mylar-aluminum-aluminum-Mylar) type 34321 by the Dobeckmun Company.

(2) MAMAM (Mylar-al.-Mylar-Al.-Mylar) type 34322 by the Dobeckmun Company.

(3) MLM (Mylar-lead-Mylar) X283 by G. T. Schjeldahl Company.

TABLE VI - VAPOR BARRIER FILM THICKNESS, WEIGHT AND HELIUM PERMEABILITY DATA

Material Designation	Film Total Thickness	Film Weight lbs/ft^2	Helium Permeability cc/sec/Atn/2.9" Dia. Test Area*			
			Sample No.	Unwrinkled	Sample No	Unwrinkled
MAAM, Type 34321 Dobeckmun Co. ½ mil Mylar, 0.35 mil aluminum 2 plies each	.002"	.0187	24 P-1	$< 1 \times 10^{-10}$	24 PW-1	$< 1 \times 10^{-10}$
			-2	$< 1 \times 10^{-10}$	-2	$< 1 \times 10^{-10}$
			-3	$< 1 \times 10^{-10}$	-3	$< 1 \times 10^{-10}$
MAMAM, Type 34322 Dobeckmun Co. ½ mil Mylar, 0.35 mil aluminum	.0027"	.023	25 P-1	$< 1 \times 10^{-10}$	25 PW-1	5×10^{-9}
			-2	$< 1 \times 10^{-10}$	-2	1×10^{-9}
			-3	$< 1 \times 10^{-10}$	-3	1×10^{-10}
MLM, X283 G.T. Schjeldahl Co. 1 mil Mylar 0.8 mil lead	.0047"	.0703	22 P-1	$< 1 \times 10^{-10}$	22 PW-1	$< 1 \times 10^{-10}$
			-2	$< 1 \times 10^{-10}$	-2	$< 1 \times 10^{-10}$
			-3	$< 1 \times 10^{-10}$	-3	$< 1 \times 10^{-10}$
			22_a P-1	1.5×10^{-10}	22_a PW-1	4.5×10^{-10}
			-2	$< 1 \times 10^{-10}$	-2	2.5×10^{-10}
			-3	7.0×10^{-9}	-3	9.0×10^{-10}
			22_b P-1	8.0×10^{-10}	22_b PW-1	9.0×10^{-10}
			-2	2.0×10^{-10}	-2	5.0×10^{-10}
			-3	6.0×10^{-10}	-3	8.0×10^{-10}
			22_c P-1	4.0×10^{-10}	22_c PW-1	4.4×10^{-9}
			-2	3.0×10^{-10}	-2	6.7×10^{-9}
			-3	1.2×10^{-9}	-3	7.0×10^{-8}
MAM, Type 12-1169 Continental Can Co. ½ mil each layer	.0017"	.0132	1 P-1	8.6×10^{-9}		
			-2	9.0×10^{-9}		
			-3	7.17×10^{-10}		

*Helium permeability data taken from Contract NAS 8-11376 Progress Report #10 GER-11706 S/9 dated 5 May 1965.

Permeability and weight data for these vapor barrier materials is presented in Table VI. Also given are these data for MAM vapor barrier material used on the lightweight sealed foam Centaur insulation.

The helium permeability characteristics of vapor barrier material seams was conducted on a specially constructed test fixture designed to present the edge of a seam to the flow of helium gas into a mass spectrometer apparatus as shown on Figures 25 and 26.

Helium permeability values for two adhesives are presented in Table VII.

TABLE VII - VAPOR BARRIER ADHESIVE SEAM HELIUM PERMEABILITY

Vapor Barrier	Adhesive	Cure	Helium Perme. cc/sec/atmosphere
MAAM	G-207	Heat sealed by 275°F Hand Iron	1.2×10^{-9} *
MAAM	Apidrene L100/ Mocha	Minimum 16 hours at room temperature	1×10^{-10} *

*These values for 3" length of seam

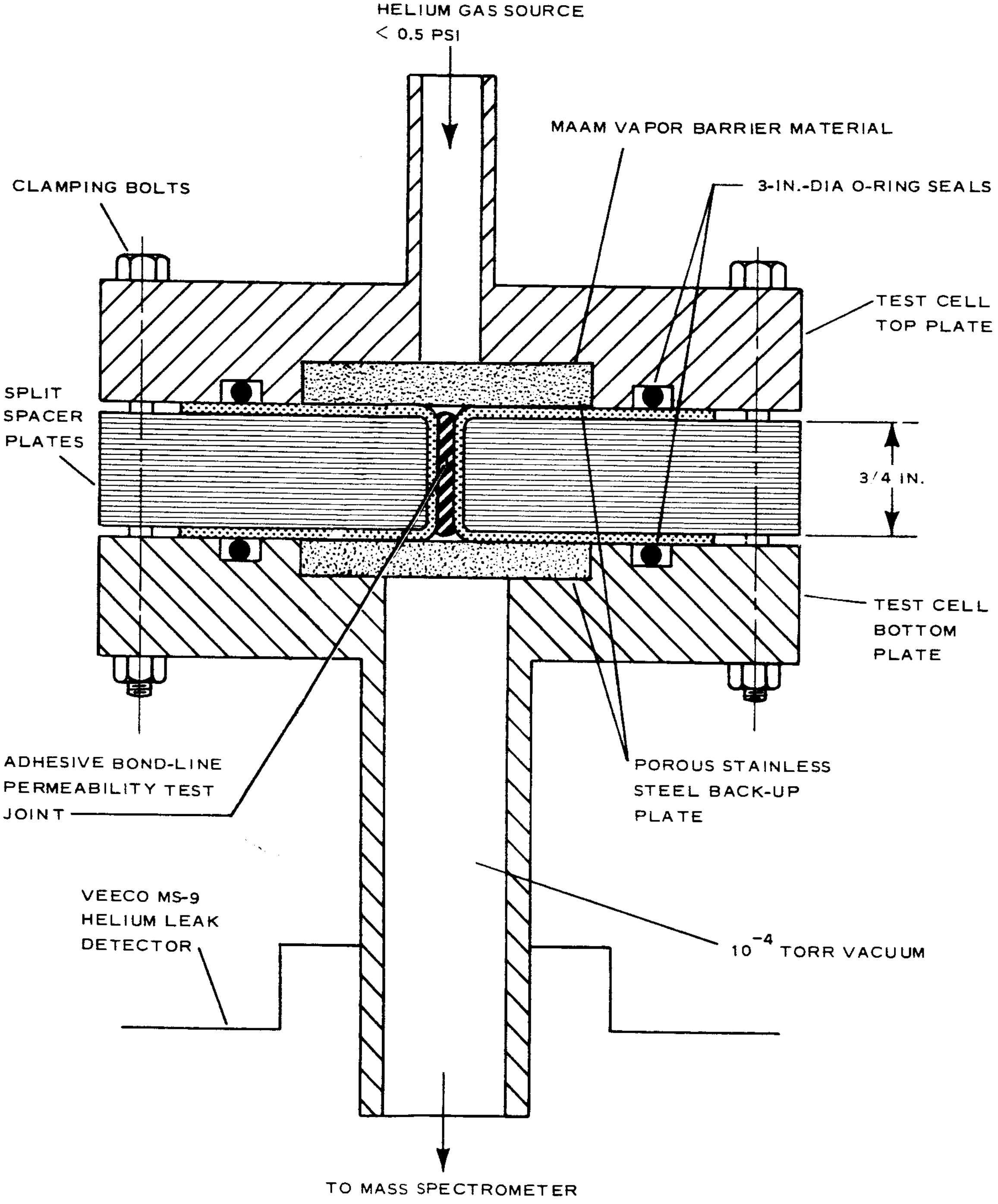
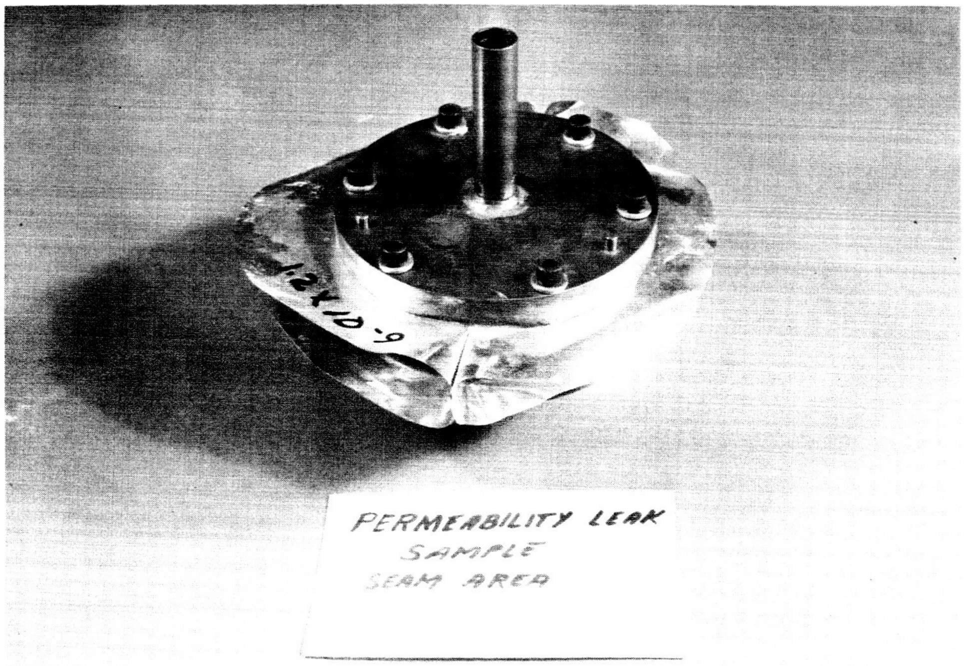


FIGURE 25 - HELIUM LEAK-DETECTOR TECHNIQUE FOR EVALUATING VAPOR BARRIER SEAMS

Fixture Closed



Fixture Open

FIGURE 26 - VAPOR BARRIER SEAM LEAK TEST SAMPLE AND TEST FIXTURE

It may be concluded from the seam leak tests that vapor barrier seams properly made with either heat sealed G-207 polyester adhesive or room temperature cured Adiprene L-100/ Mocha polyurethane adhesive will have a per linear inch helium leak rate equivalent to or lower than the vapor barrier film leak rate per square inch. With doubler strips added to the vapor barrier seam, an order of magnitude reduction of seam permeability is expected.

2. Outgassing Characteristics

The outgassing characteristics of the Mylar surface of MAAM vapor barrier material was evaluated by weight loss measurements conducted in the automatic recording vacuum balance shown in Figure 12. The test procedure is as follows:

(1) Nine strips 2" x 8" were cut from a MAAM sheet and surface cleaned with MEK solvent wipe.

(2) After the MEK wipe flashed off, strips were hung in the specimen chamber of the vacuum balance. The balance was monitored for approximately two hours until no weight loss was observed at ambient pressure indicating evaporation of all solvent volatiles prior to evacuation cycle.

(3) Record "in air" weight, time temperature chart on.

(4) Reduce pressure by roughing to 500 microns for 1 hour.

(5) Reduce pressure to 10 microns - 1 hour*.

(6) Reduce pressure to 5×10^{-4} run 1 hour (including approach time)*.

(7) Reduce pressure to 1×10^{-4}, run .5 hour or 1 hour*.

(8) Reduce pressure to 5×10^{-5}, " " " " .

(9) Reduce pressure to 1×10^{-5}, " " " " .

(10) Reduce pressure to 5×10^{-6}, " " " " .

(11) Reduce pressure to ultimate and run overnight.

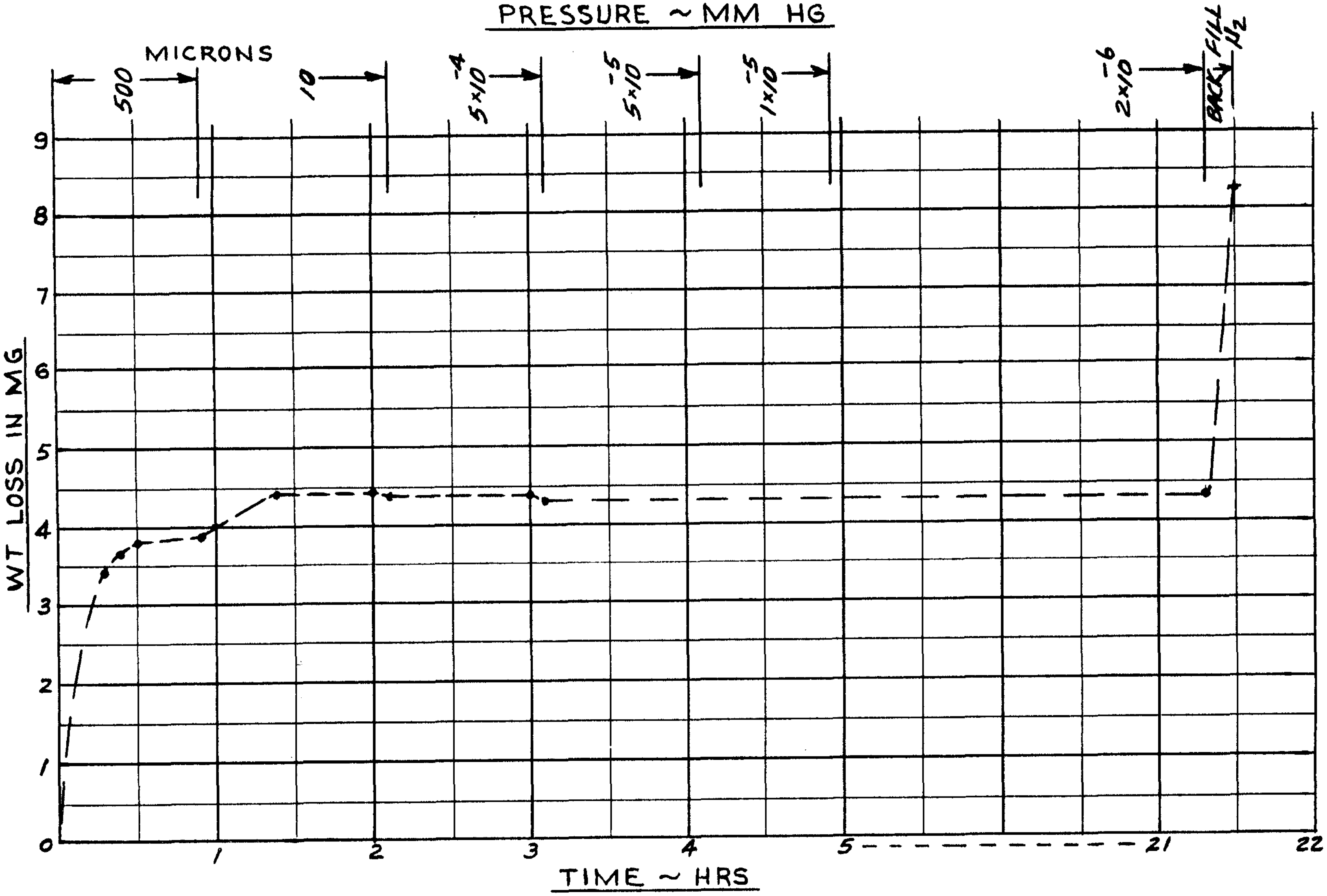
(12) Record data and secure test.

(13) Backfill with dry Nitrogen and record final "in air" weight.

*Pressure may be reduced when slope of weight loss curve reaches 10 percent.

Test results are shown on Figure 27. No significant weight loss is observed after the 10 micron pressure range therefore it may be concluded that MAAM vapor barrier material does not outgas appreciably at 10^{-4} to 10^{-6} Torr pressure range.

The outgassing characteristics of vapor barrier material seams were evaluated by weight loss measurements conducted in the automatic vacuum balance by the same techniques described above for vapor barrier materials. Two seam test samples were fabricated as shown in Figure 28. This sample configuration will expose to the vacuum balance test chamber an area of adhesive seam per unit length of seam identical to an equivalent



MAAM STRIPS @ AMBIENT TEMP.

FIG. 27

length of vapor barrier seam presented to the vacuum environment of a self evacuated insulation panel. The exterior surfaces of the samples were cleaned by MEK solvent wipe and thoroughly dried before testing.

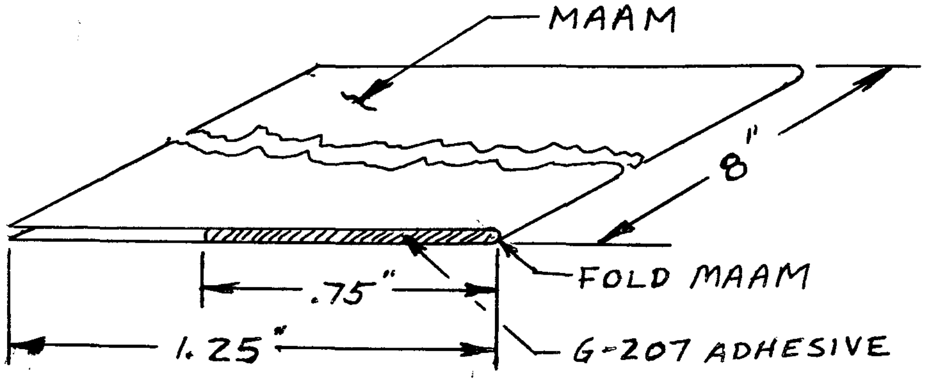


FIGURE 28 - VAPOR BARRIER SEAM OUTGASSING SAMPLE

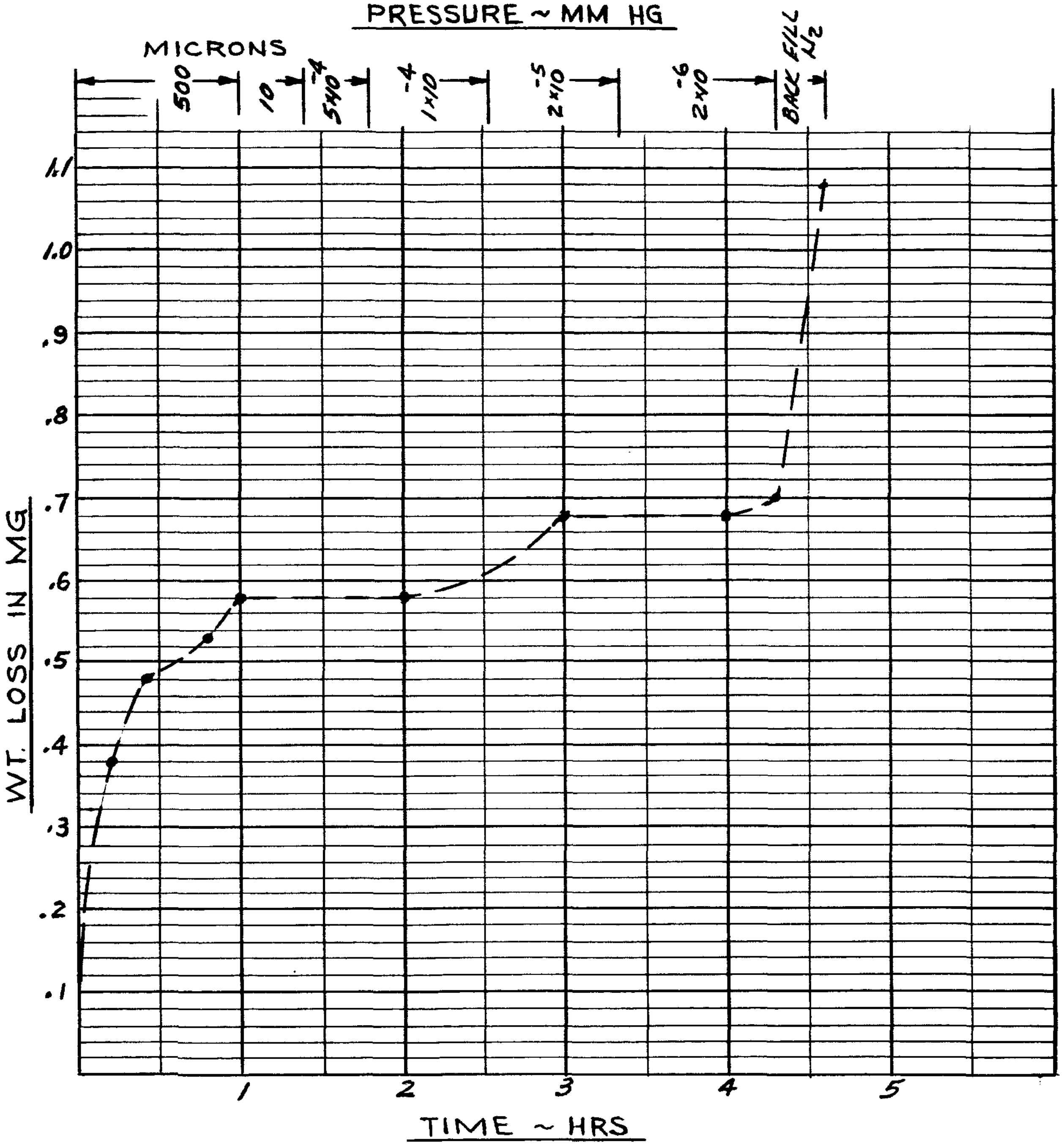
Sample #1 was prepared by allowing the G-207 adhesive to air dry approximately one hour at room temperature prior to heat sealing the folded MAAM material with a 275°F hand iron. Sample #2 was prepared by oven drying the G-207 adhesive at 120°F for two hours prior to heat sealing the folded MAAM material with a 275°F hand iron.

A comparison of weight loss (outgassing) versus pressure for both types of seams is shown on Figures 29 and 30.

The following conclusions are derived from the seam outgassing evaluation:

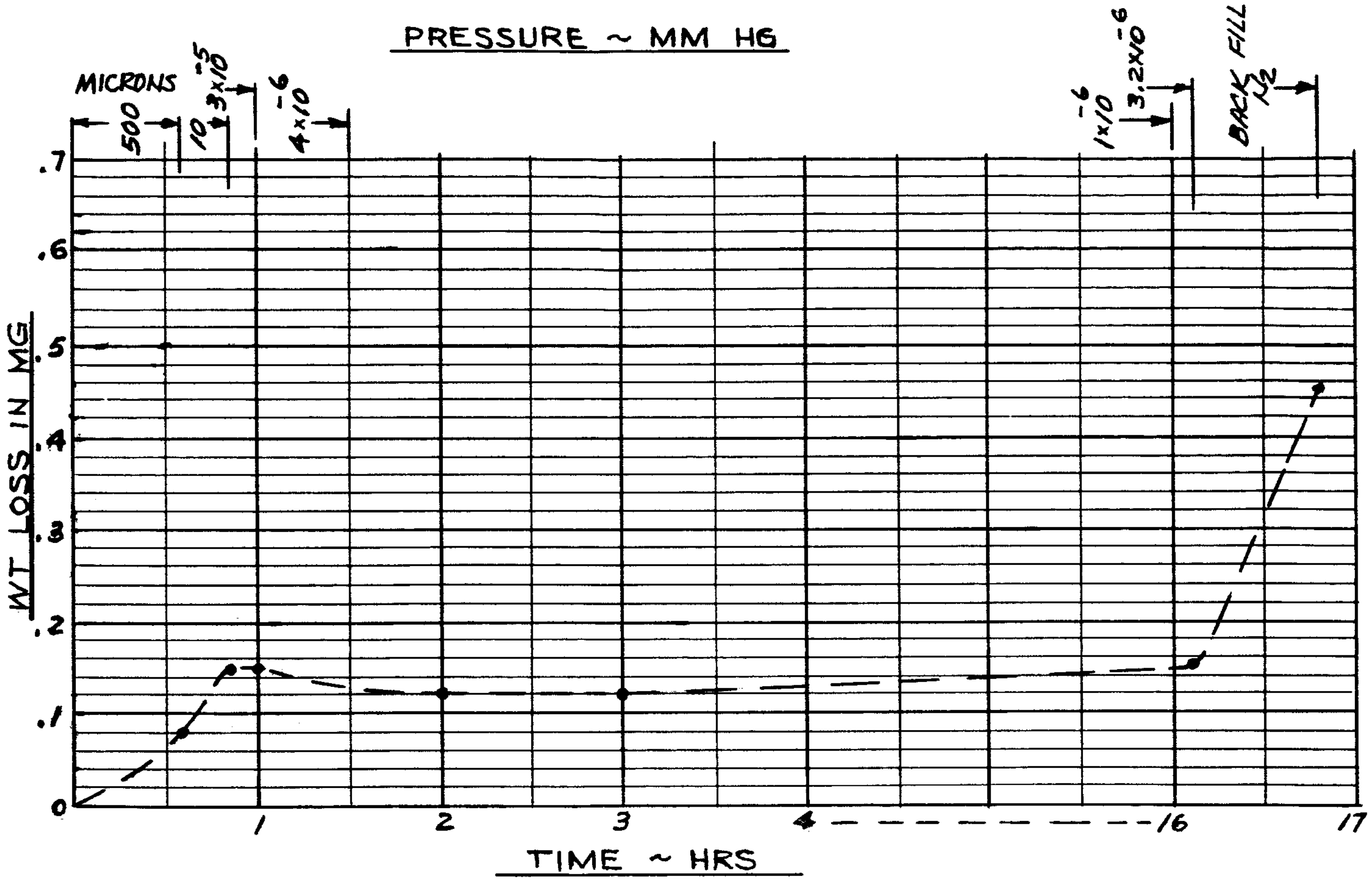
(1) The MAAM vapor barrier seams joined with G-207 adhesive processed with a 120°F oven dry cycle before heat sealing will appreciably reduce the amount of volatiles outgassed to the evacuated insulation.

(2) Very little outgassing of G-207 adhesive seams occurs in the 10^{-4} to 10^{-6} range on both types of seams.



SAMPLE #1
MAAM SEAMS @ AMBIENT TEMP

FIG. 29



SAMPLE #2

MAAM SEAMS @ AMBIENT TEMP.

FIG. 30