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MATERIAL RESEARCH REPORT - SECOND YEAR
CONTRACT NO. NAS 2-3817

STUDY OF PASSIVE TEMPERATURE AND HUMIDITY CONTROL SYSTEMS FOR ADVANCED SPACE SUITS

BY
W. WOO

PREPARED BY

TRW
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FOREWORD

This report presents the results of materials research conducted during the time period 1 July 1967 to 1 September 1968. These research activities were performed as part of the Contract NAS 2-3817, sponsored by the Ames Research Center of the National Aeronautics and Space Administration at Moffet Field, California. James Blackaby of the Environmental Control Research Branch directed this effort for the NASA/Ames Research Center.

The program was performed by the Environmental Sciences and Engineering (ESE) Department of the Systems Engineering and Integration Division of the TRW Systems Group. A. P. Shlosinger, Head of the ESE Research Section was the Principal Investigator and Program Manager. Valuable contributions in the performance of this program were made by E. W. Bentilla, Member of the Technical Staff; G. Hwa, Engineer and Research Specialist; G. B. Patchell and J. G. Morgan, Laboratory Technicians.

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MATERIALS RESEARCH REPORT - SECOND YEAR

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SUMMARY

Material and processes research was performed as part of a study program with the objective of developing techniques for control of temperature in an extravehicular space suit by passive means. This report presents the materials research performed during the second phase of the program with a brief summary of the research performed during the initial phase.

A study directed toward a solution of heat pipe freeze up problems through the use of mixtures as heat pipe working fluid is presented. As part of this study, data on the freezing point of mixtures of 1-propanol and water were experimentally obtained.

Material studies related to noncondensable gases in heat pipes were performed. These included experimental gas emission tests of potential space suit heat pipe materials when exposed to both a vacuum environment, and a water vapor environment. A literature search was conducted to select film enclosure materials suitable for flexible heat pipe application with one of the desired film selection characteristics being impermeability to noncondensable gases.

In order to reduce the thermal gradient from the heat pipe outer surfaces to the active wick surfaces, studies of the thermal conductivity of metallic wicking materials and methods of bonding wicks to substrates were performed.

Techniques are described for the fabrication of an experimental heat pipe device which was used to demonstrate techniques applicable to space suit controllable heat pipe devices.

INTRODUCTION

This report presents the results of analytical and experimental studies of materials and processes related to the performance of a program entitled, "Study of Passive Temperature and Humidity Control Systems for Advanced Space Suits." The primary emphasis of this report is upon the studies performed during the second phase of the program. A brief review of the work performed during the first phase of this study program, as reported in detail in ref. 1, is also presented. Since this report covers only the materials and processes, it is a supplement to the reports which summarize the program (refs. 2 and 3).

The subject of the program was, in general terms, to perform a study leading to a concept which would use the outer shell of a hard space suit as a radiative surface for heat rejection, with variable conductance devices for thermal control of the net heat flow out of an astronaut's body. During the first phase of this program, the major task was the development of these variable thermal conductance devices. Heat pipes were modified such that flow can be controlled in response to externally applied control signals.

During the development of these concepts for space suit temperature control, it became apparent that flexible heat pipes must be developed for efficient removal of heat from the astronaut's skin (refs. 2 and 3). This requires that the flexible heat pipes be located adjacent to the astronaut's skin and, therefore, within the space suit gas pressurizing environment. In present space suits, the pressurizing gas is oxygen at a pressure in the order of 2.4×10^5 dynes/sq cm (3.5 psia). Advanced space suit concepts will use gas mixtures at higher pressures. As the working fluid in a heat pipe is in a saturated condition, the internal pressure in a heat pipe at a nominal skin temperature of 33°C would be in the order of only 4.8×10^4 dynes/sq cm (0.7 psia), if water or a fluid with similar vapor pressure characteristics is used as the heat pipe working fluid. A flexible device using a working fluid such as water in this temperature range would, therefore, tend to collapse as a result of space suit pressurization.

The major effort during the second phase of the program was directed to the development of flexible, heat pipes which would not collapse when evacuated and exposed to space suit pressurizing gas. Several material and process problems had also become apparent during the development of the controllable heat pipes. The material problems and the research efforts directed to their solution are the subject of this report.

Problem Definition

The heat and mass transfer processes which characterize heat pipe operation are described in detail in ref. 4. These processes are the following:

(1) Conductive heat transfer from the outside face of the heat pipe to the liquid-vapor interface at the wick surface.

(2) Endothermic liquid to vapor phase change, i.e. evaporation, at the liquid-vapor interface at the wick surface.

(3) Flow of the vapor from the warmer to the colder regions of the heat pipe.

(4) Exothermic vapor to liquid phase change, i.e. condensation, at the vapor-liquid interface at the wick surface.

(5) Conductive heat transfer from the vapor-liquid interface to the outside surface of the heat pipe.

(6) Capillary flow of the liquid working fluid from the condenser to the evaporator regions of the heat pipe.

An examination of these processes shows that the more important aspects of heat pipe operation are related to working fluid heat and mass transfer and capillary flow. During the first phase of this program, water was selected as the working fluid for use in the space suit controllable heat pipes (ref. 4). Since the space suit heat pipe operating temperature is expected to reach temperatures below the freezing point of water, the problem of either freeze prevention or restart of frozen heat pipes must be solved (refs. 2 and 3). In an attempt to extend the operating temperature range of space suit heat pipes, use of fluid mixture containing water and an anti-freeze solute was studied. This study is presented in the section on "Mixtures of Heat Pipe Working Fluids".

During the initial research activities on heat pipes, it had become apparent that noncondensable gases inside heat pipes can be the cause of significant degradation in heat pipe performance (ref. 4). The source of noncondensable gases inside a heat pipe can either be gas emission from materials inside the heat pipes, or inward leakage of gases into the heat pipe cavity. Since the use of heat pipes for removal of heat from the astronaut's skin requires that heat pipes be located within the space suit pressurizing gas, inward leakage of noncondensable gases into the heat pipes has to be avoided or reduced to an acceptable level. Investigations directed to a solution to the noncondensable gas problem required research in both areas, i.e. material gas emission and inward leakage.

A material screening procedure was performed and materials which could be considered for heat pipe application were tested for gas emission. Inward leakage of suit pressurization gas could be expected to present a serious problem for the flexible heat pipes applied to heat removal from the human skin. Flexible enclosure films impermeable to space suit pressurizing gases were required. The processes which led to the selection of materials and approaches to solutions in remaining problem areas are presented in the section on "Noncondensable Gases".

Since one of the processes of heat pipe operation is the conduction heat transfer between the vapor-liquid interface and the outside surface of the heat pipe, methods of reducing the thermal resistance between the heat pipe outer wall and the vapor-liquid interface were studied. One way of decreasing this thermal resistance is to reduce the contact resistance between the heat pipe wall and the wick surface. This can be accomplished by bonding the wick structure to the heat pipe wall. Problems inherent in putting this into practice are discussed in ref. 3. The primary problem is related to the degradation of capillary flow of heat pipe working fluid resulting from damage to the wick capillaries as a result of bonding. Methods of reducing this degradation through the development of improved wick bonding techniques were studied. The results of a literature search and experimental work on wick bonding are shown in the section entitled "Wick Bonding".

Another way of decreasing the thermal resistance between the heat pipe outer surface and the vapor-liquid interface is to increase the thermal conductance in the capillary structure of the wick-liquid composite. It became apparent that it may be possible to increase the thermal conductance of the composite by using metallic fiber materials as wicks. A study was therefore conducted to investigate the benefits which might be obtained from the use of commercially available metallic wicking materials. The study method and the results of this study are presented in the "Thermal Conductivities of Metallic Felts and Foams" section.

The development of the present concept of a variable conductance space suit panel is described in ref. 2. A prototype working model of this panel was constructed and tested. The application of the knowledge gained in the performance of the material and process studies to the fabrication of this panel is described in detail in the section entitled "Design and Fabrication Techniques used in Construction of Space Suit Prototype Panel".

Review of Material Research Effort - First Phase

During the first phase of this study program, techniques of bonding quartz fiber wicks to fiberglass substrates were studied. These techniques consisted essentially of using viscous adhesives to bond wicks to substrates. Some limited work using thermoplastic films as adhesives was also performed. A successful bonding method using a polyethylene coated polyester was developed for use in space suit heat pipes. This bonding method was tested and found to be satisfactory for an environment consisting of water vapor at a temperature range of 24°C (75°F) to 72°C (160°F) for time periods of up to fifty days. Upper limit temperatures of this bonding material were also experimentally obtained.

Fabrication techniques were developed for use in the construction of the variable thermal conductance space suit prototype panel. This included techniques for fabrication of heat pipe-type devices made of epoxy-fiberglass laminates.

A technique for use in the preliminary screening of potential space suit heat pipe construction materials was developed. The technique was used to eliminate from consideration, for heat pipe construction, materials with undesirable gas emission characteristics.

MIXTURES OF HEAT PIPE WORKING FLUIDS

A review of potential heat pipe working fluids was continued during the second phase of this study program. It was established during the initial phase (ref. 4) that, of the fluids studied, water was the superior working fluid for space suit heat pipe application. But, one of the major problems associated with the use of water for this application is that the freezing point of water is higher than the expected minimum operating temperature of these heat pipes. One of the concepts for freeze prevention generated during this study (ref. 3) was to use mixtures as the heat pipe working fluid. One of the fluids of the mixtures would provide a freezing point depression on the second fluid. Therefore, an analytical study to determine the feasibility of using mixtures as a heat pipe working fluid was performed. In order to place the proper emphasis on this study in relation to other problem areas associated with the development of controllable heat pipes for space suits, a limit was placed on the number of fluids examined.

Several characteristics were used to judge the suitability of fluids for space suit heat pipes. The characteristics used were the following:

- Fluid property parameter
- Toxicity
- Fire hazard
- Vapor pressure
- Freezing point
- Material compatibility

As described in reference 4, the process of capillary flow of the working fluid from the condenser to the evaporator is usually the limiting factor for maximum possible heat flow in a heat pipe. A measure of this capillary flow is given by the fluid property parameter, G_{II}. This parameter, as developed in reference 4, is given by the following:

$$G_{\Pi} = \frac{\rho_{\ell} \lambda \sigma}{\mu}$$

where: ρ = liquid density
λ = heat of vaporization
σ = surface tension
μ = viscosity

Although the fluid property parameter is important for maximizing heat flow in a heat pipe, the other space suit heat pipe selection characteristics must also be considered. Some degradation in capillary flow performance must be accepted so that the requirements of the remaining selection characteristics can be satisfied. Still, this parameter is a convenient starting point in the search for space suit heat pipe working fluid mixtures. The parameter indicates that heat flow will increase as the liquid density, heat of vaporization, and surface tension are increased and viscosity decreased. Due to the availability of a list of fluids which have a high latent heat of vaporization, this fluid property will be used as the initial screening for this fluid mixture study. A manageable list of fluids which have a high heat of vaporization was obtained from reference 5 and is shown on figure 1.

In order to quickly reduce the number of fluids studied, the fluids were examined for their acceptability with respect to the remaining selection characteristics.

Toxicity characteristics for these fluids were obtained from reference 6 and are summarized on Table I. Since the heat pipes would be adjacent to an astronaut and leakage through permeable heat pipe construction materials a distinct possibility, the toxicity level of ethyl alcohol was, for this study, established as the maximum acceptable. From Table I it can be seen that only methanol (methyl alcohol), ethyl alcohol, isopropyl alcohol, 1-propanol (n-propyl alcohol) and water were within this toxicity level. Of these candidate heat pipe fluids, water has the desirable quality of the highest heat of vaporization and the lowest toxicity rating. Therefore, the search for space suit heat pipe mixtures will be directed toward finding a mixture of water and a fluid which will act as a freezing point depressant.

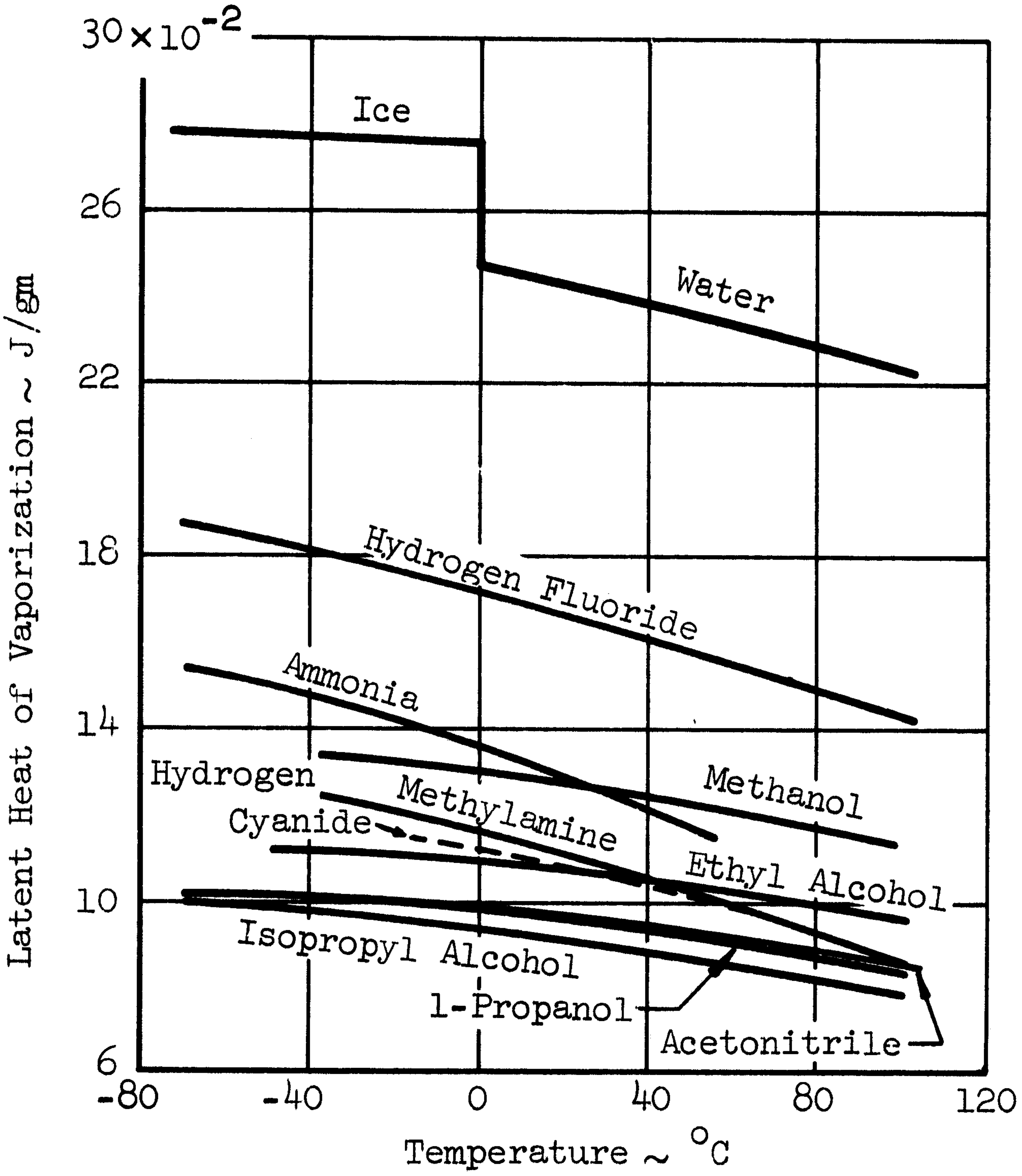


Figure 1 - HIGH LATENT HEAT OF VAPORIZATION LIQUIDS

Table I - TOXICITY CHARACTERISTICS OF SELECTED FLUIDS

Substance	Formula	Toxic Hazard Rating*											
		Acute Local			Acute Systemic			Chronic Local			Chronic Systemic		
		Irritant	Ingestion	Inhalation	Irritant	Ingestion	Inhalation	Irritant	Ingestion	Inhalation	Irritant	Ingestion	Inhalation
Hydrogen Fluoride	HF	3	3	3	0	3	3	2	0	0	0	2	2
Methylamine	CH_3NH_2	U	U	U	U	U	U	U	U	U	U	U	U
Ammonia	NH_3	3	3	3	U	U	U	1	0	0	U	U	U
Methanol	CH_3OH	1	0	1	0	3	2	1	1	0	0	2	2
Ethyl Alcohol	CH_3CH_2OH	1	0	0	0	2	2	1	0	0	0	1	1
Hydrogen Cyanide	HCN	2	2	2	0	3	3	U	U	U	U	U	U
Acetonitrile	CH_3CN	1	0	0	0	3	3	2	0	0	0	1	1

Table I - TOXICITY CHARACTERISTICS OF SELECTED FLUIDS (Cont'd)

Substance	Formula	Toxic Hazard Rating*											
		Acute Local			Acute Systemic			Chronic Local			Chronic Systemic		
		Irritant	Ingestion	Inhalation	Irritant	Ingestion	Inhalation	Irritant	Ingestion	Inhalation	Irritant	Ingestion	Inhalation
Isopropyl Alcohol	$CH_3CHOHCH_3$	1	1	1	0	2	2	U	U	U	0	1	1
1-Propanol	$CH_3CH_2CH_2OH$	1	1	1	0	0	1	U	U	U	0	0	1
Water	H_2O	0	0	0	0	0	0	0	0	0	0	0	0

Note: *The terms in this table are standard medical terms. For definitions, see ref. 6.

Toxicity rating code: 0 = none, 1 = slight, 2 = moderate, 3 = high, and U = unknown.

The next characteristic considered was the fire hazard rating. All of the remaining candidate heat pipe fluids are dangerous when exposed to heat or flame when not mixed with water. The flammability hazard due to leakage of water and the candidate alcohols decrease as the percentage of water in the mixture is increased. For example, the limits of flammability of ethyl alcohol and water mixtures is presented in ref. 7, and with 80% water it was difficult to inflame any mixture of the vaporized liquid in air. At 105°C (220°F) with 85% water, inflammation was virtually impossible. From this, it was concluded that the fire hazard characteristics of the alcohols when mixed with water would have to be studied further, after a heat pipe fluid mixture ratio and space suit pressurizing environment have been determined. For this reason, the alcohols which are under consideration will not be ruled out at this time due to their fire hazard characteristics.

Vapor pressure is another characteristic which must be examined when selecting a heat pipe working fluid mixture. Vapor pressure as a function of temperature is shown on fig. 2 for the fluids under consideration. It would be desirable to have the mixture exert a pressure inside the heat pipe which is equal to the environmental pressure outside of the heat pipe. This environmental pressure can be as low as 2.4×10^5 dynes/sq cm (3.5 psia) for pure oxygen used as the space suit pressurizing gas, to as high as 1.02×10^6 dynes/sq cm (14.7 psia) for air as the pressurizing gas. As explained in ref. 2, the expected space suit heat pipe fluid nominal temperature would be approximately 33°C (91°F) during normal operation, with the maximum expected being 72°C (160°F) and the minimum expected being -80°C (-112°F). It can be seen from fig. 2, that all the fluids under consideration have a vapor pressure less than 2.4×10^5 dynes/sq cm (3.5 psia) at 33°C (91°). This means that some method of internal support is required for the flexible chambers of the two-chamber heat pipes. This curve also shows no excessive pressures, requiring thick-walled pressure vessels for any of the candidate fluids, over the temperature range to which the heat pipes would be exposed.

The problem of using a heat pipe working fluid consisting of fluids with different vapor pressures is discussed in ref. 3. It would be desirable to use a mixture where water has higher vapor

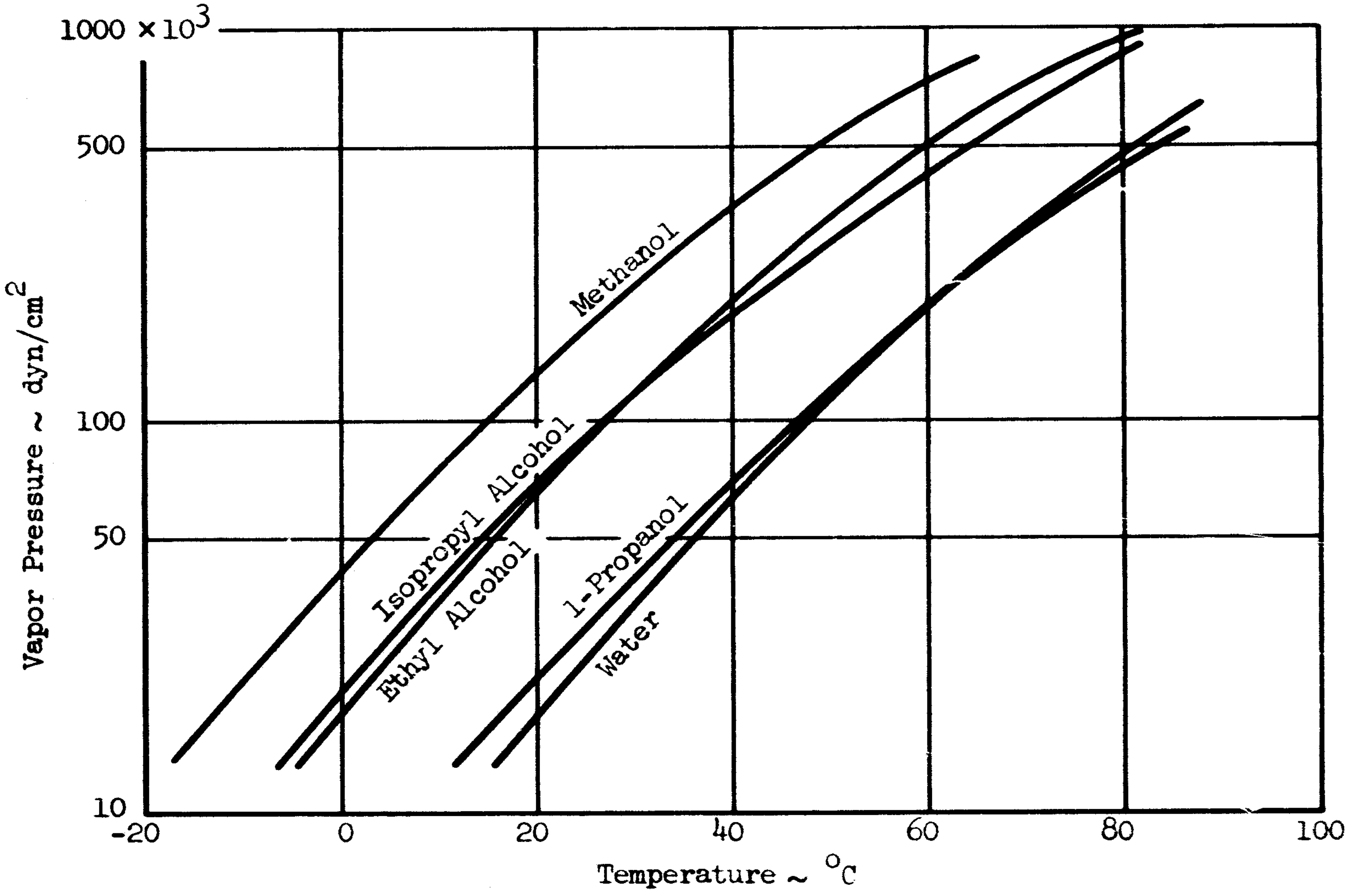


Figure 2 - VAPOR PRESSURES OF LIQUIDS WITH HIGH LATENT HEATS OF VAPORIZATION

pressure than the freezing point depressant. Figure 2 shows that 1-propanol has a vapor pressure curve similar to water at the expected heat pipe operating temperature.

Freezing point curves (ref. 8 and 9) for mixtures of water and the several alcohols being considered for heat pipe application are shown on figure 3. As can be seen from this figure, freezing point data for some of these mixtures was limited. Available data for 1-propanol and water was limited to weight fractions of 32.5% or less. Due to its interesting vapor pressure characteristics, 1-propanol could be a desirable freezing point depressant for water. For that reason, freezing point data were experimentally obtained during this study. These are the data shown on figure 3 for the freezing points of mixtures of 1-propanol and water. No significant freezing point depression is obtained until mixtures of 1-propanol and water reach 90% or more by weight. Therefore, 1-propanol was eliminated as a candidate freezing point depressant for this study.

Freezing point data for isopropyl alcohol was also limited. Since this fluid has a lower latent heat of vaporization and similar vapor pressure characteristics as ethyl alcohol, it was eliminated as a candidate fluid for heat pipe fluid mixtures. As can be seen from figure 3, high percentages of the remaining candidate alcohols are required when mixed with water to lower the freezing point of the mixture to the desired -80°C (-112°F) level.

The material compatibility characteristic cannot be determined until all of the heat pipe construction materials have been selected. But, with water and the alcohols being considered, construction materials can be found which are both suitable for heat pipes and compatible with these fluids.

The fluid property data of viscosity, fluid density and surface tension which are required to compute the heat pipe working fluid property parameters for methanol, and ethyl alcohol, were taken from references 8 and 9. Using these data together with the latent heat of vaporization curve (fig. 1), fluid property performance parameters as a function of fluid mixture ratios at several temperatures were computed. The resulting curves are shown on figures 4 and 5 for methanol and ethyl alcohol. From figure 3, it can be seen that a weight fraction of between 75% and 100% of ethyl alcohol in water is required before the desired freezing point of -80°C (-112°F) can be obtained. Looking at the heat pipe working fluid property parameter for ethyl alcohol (figure 5), there is no

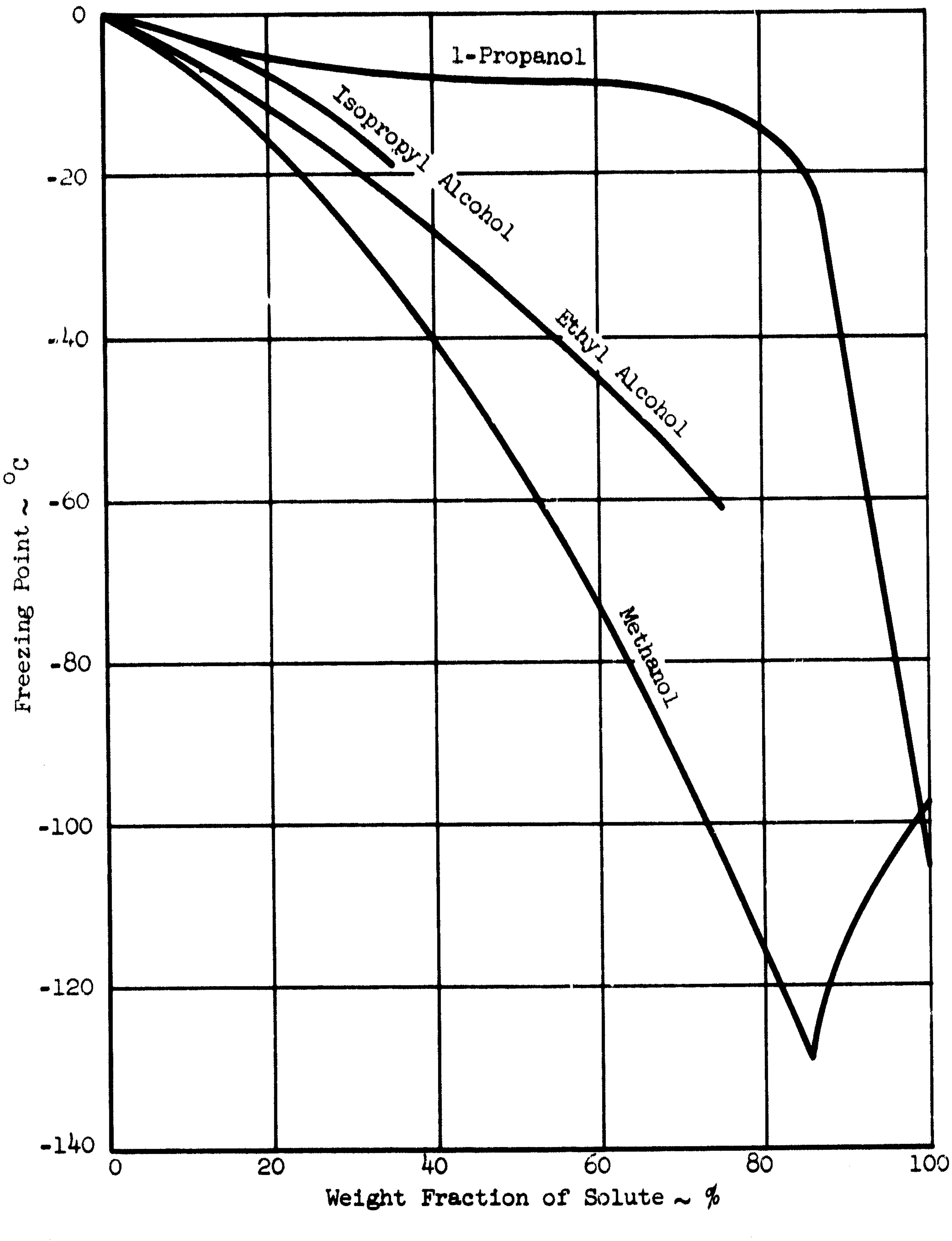


Figure 3 - FREEZING POINTS OF AQUEOUS SOLUTIONS

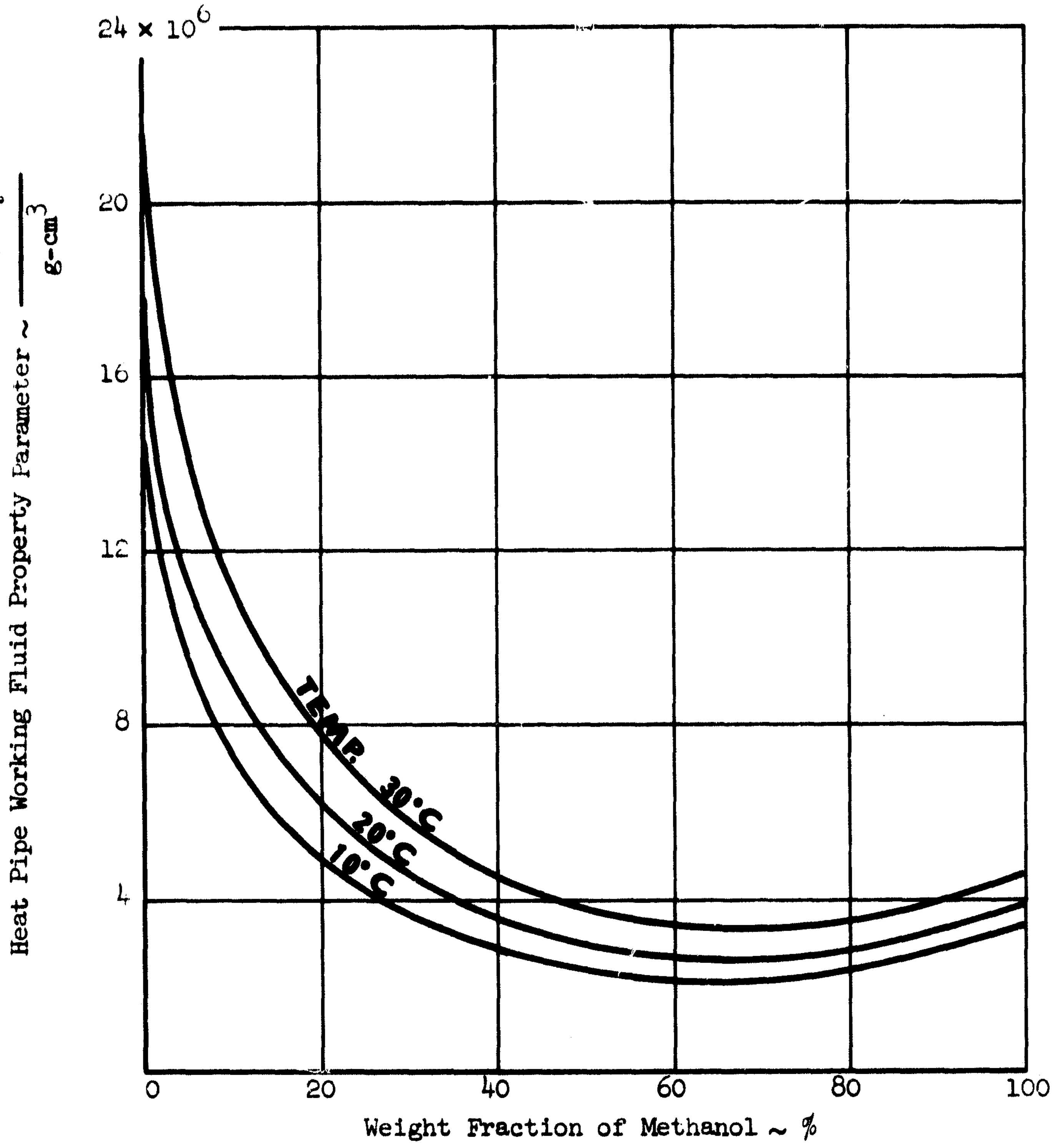


Figure 4 - HEAT PIPE WORKING FLUID PROPERTY PARAMETER FOR MIXTURES OF METHANOL AND WATER AT SEVERAL TEMPERATURE LEVELS

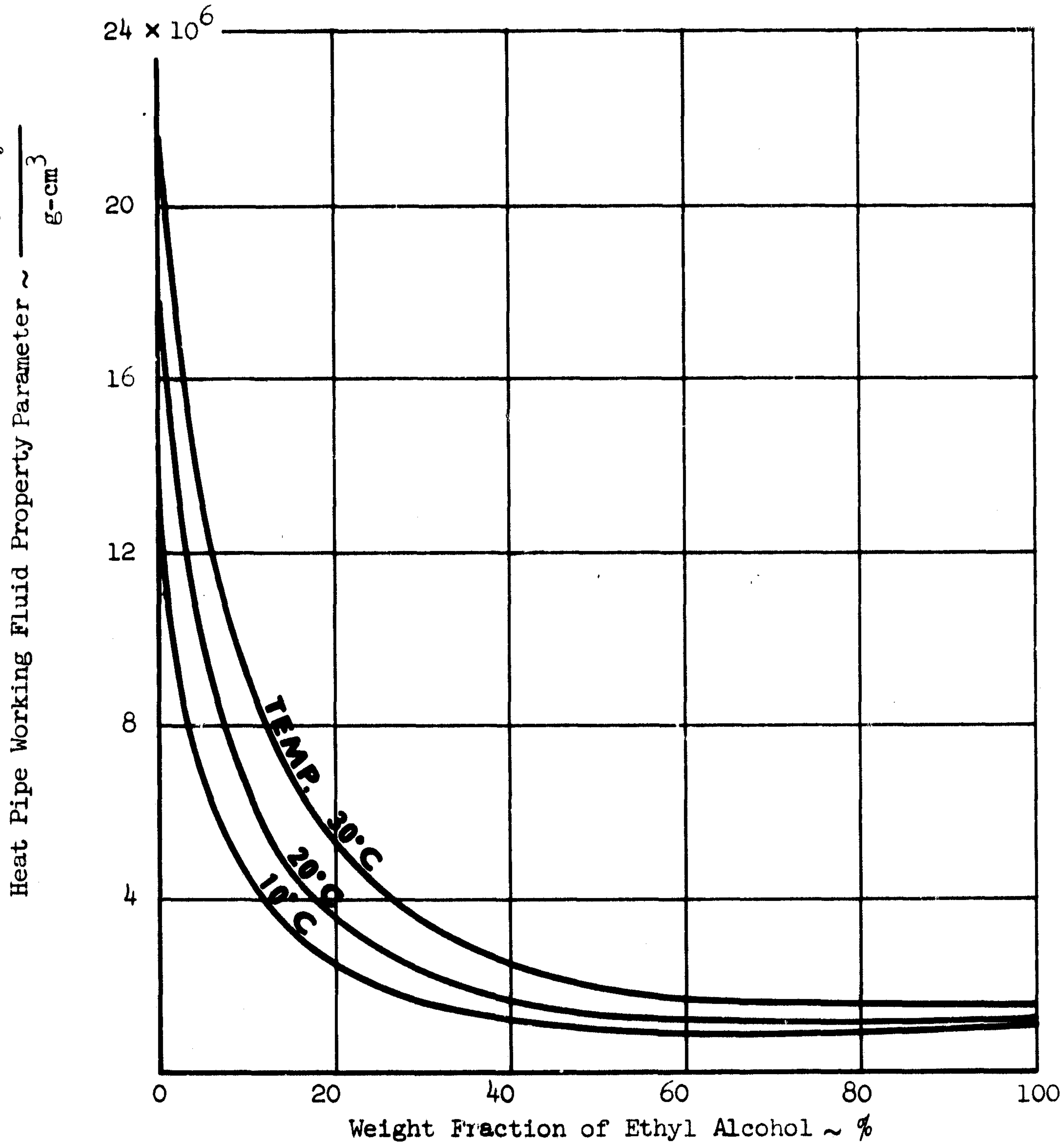


Figure 5 - HEAT PIPE WORKING FLUID PROPERTY PARAMETER FOR MIXTURES OF ETHYL ALCOHOL AND WATER AT SEVERAL TEMPERATURE LEVELS

difference in the parameter between 75% and 100% ethyl alcohol at the nominal space suit heat pipe operating temperature of 33°C (91°F). In order to achieve a freezing point of -80°C (-112°F), a weight fraction of 65% methanol is required (fig. 3). The fluid property parameter for this mixture ratio is approximately the same as 100% methanol at 33°C (91°F).

Based on this limited study, it appears that little benefit can be gained by using a freezing point depressant for water when it is required that the freezing point of the mixture be -80°C (-112°F). In addition, the use of mixtures presents other problems such as fractioning of the fluids (ref. 3) during heat pipe operation. But, using low weight fractions of methanol as a freezing point depressant, a limited lowering of the freezing point of water can be obtained at a cost of lowering the maximum possible heat pipe heat transport capability. For the fluids studied, it is concluded that use of mixtures would not be advantageous if a heat pipe working fluid freezing temperature of -80°C (-112°F) is a requirement.

MELTING POINT FOR MIXTURES OF 1-PROPANOL AND WATER

As described in reference 3, it appeared that 1-propanol would be a desirable anti-freeze additive to water for space suit heat pipe application. This was due to its vapor pressure versus temperature characteristics being similar to water over the temperature range of interest. Available freezing point data of 1-propanol - water mixtures was, however, limited to weight fractions of 32.5% 1-propanol or less (ref. 8). In order to determine whether or not 1-propanol would be desirable as an additive to water, it was necessary to extend the available freezing point data to 100% 1-propanol. A simple series of tests were, therefore, conducted for this purpose. For ease of testing, melting points rather than freezing point data were obtained.

Test Method. - Test tubes of 100 ml volume were modified as shown on figure 6 to include a copper constantan thermocouple imbedded into the side of the test tube and a trough formed in the side of the test tube to channel the flow of liquid melt over the thermocouple. A wick was placed in this trough to improve the flow of melting mixture. Mixtures of 1-propanol and water were frozen by immersing the modified test tubes containing the mixtures in

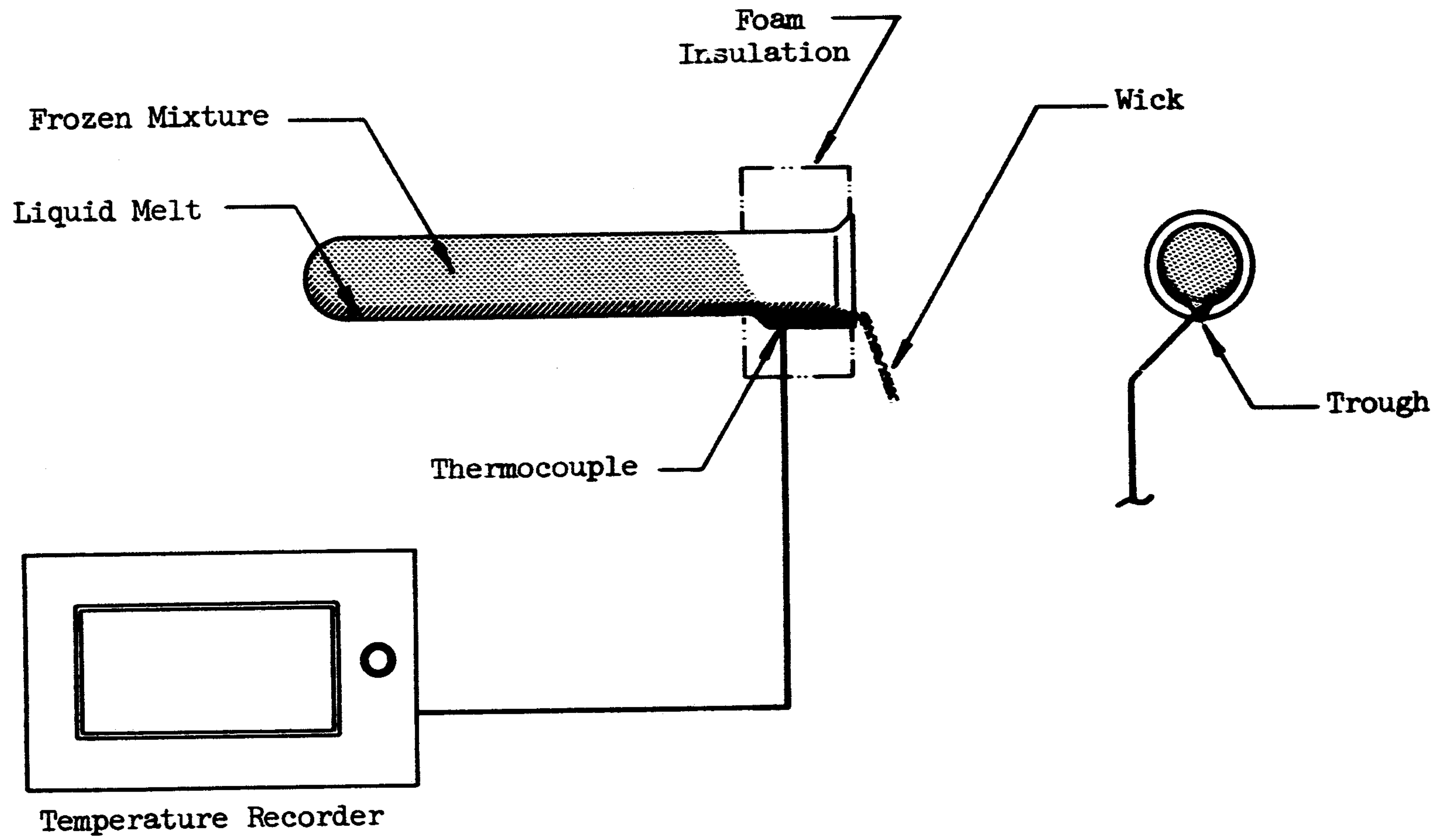


FIG.6 - SCHEMATIC OF 1-PROPANOL AND WATER MIXTURE MELT TEMPERATURE TESTS

liquid nitrogen. The frozen mixtures were then placed in a horizontal position at room temperature and allowed to thaw (fig. 6). Additional heat was applied with a heat gun to increase the flow of melting mixture. Temperature readings were recorded continuously. A typical example of the resulting temperature readout is shown in figure 7, and the results of tests are shown on figure 8. Also shown on this figure are the available data taken from the literature (ref. 8 and 9).

Test Accuracy. - The primary source of error is caused by heat gained by the thermocouple junction. This heat gained results from the higher temperature room environment conducting heat along the thermocouple leads to the thermocouple junction, thus causing an error in the temperature readout. The instrumented end of the test was insulated with foam to reduce this error. If the available data taken from the literature is assumed to be correct, then the experimental results obtained from this test should be within 3°C (5.4°F) accuracy, but on the high side for concentrations up to 80% 1-propanol. Data from 80% to 100% of 1-propanol can be as much as 30°C (54°F) high.

NONCONDENSABLE GASES

Noncondensable gases in the heat pipe vapor passages can be the cause of a deterioration in heat pipe performance (ref. 2 and 4). These noncondensable gases can either be accomodated through proper geometrical design of the heat pipes or be eliminated from the heat pipe. Since accomodating the noncondensable gases would necessitate specialized geometrical designs which may not be applicable to space suit heat pipes, the more generalized solution of eliminating noncondensable gases was the approach pursued in this study. The objective was to eliminate or minimize the two sources of noncondensable gases, namely, material gas emission and inward leakage of space suit pressurizing gases. Selective screening of commercially available materials for suitability to the application was the technique used to find materials with acceptable characteristics. Review of literature data, consultations with experienced material specialists and experimental techniques were applied.

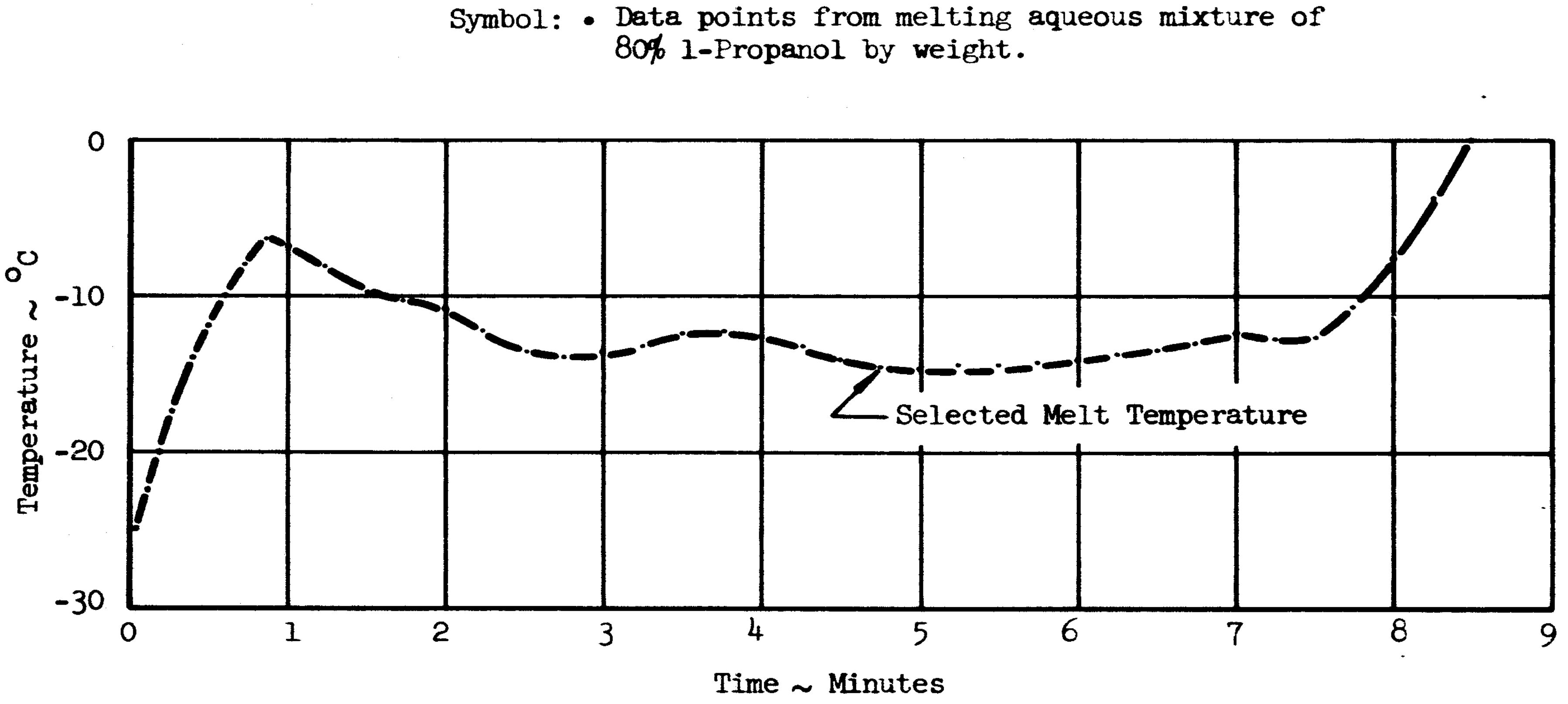


Figure 7 - TIME TEMPERATURE HISTORY OF A MELTING MIXTURE OF 1-PROPANOL/WATER

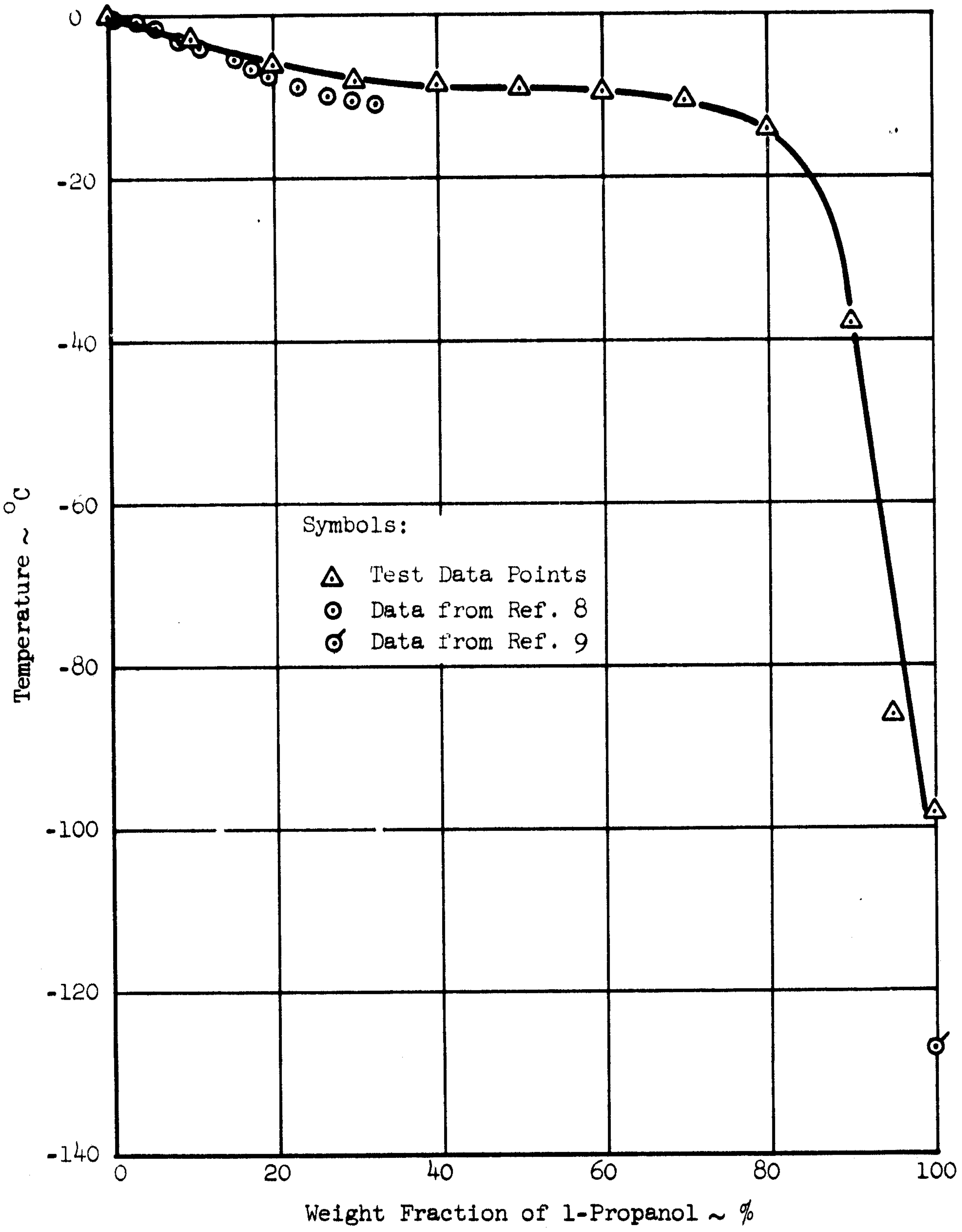


Figure 8 - MELTING POINT OF 1-PROPANOL/WATER MIXTURES

The initial experimental evaluation of material gas emission was performed by exposure to a hard vacuum environment. This technique was used because of the relative simplicity of the experimental arrangements required. It exposed the material sample to a vacuum environment more severe than that expected in a space suit heat pipe. It could therefore be assumed that materials showing only insignificant gas emission in a hard vacuum would also satisfactorily perform in a heat pipe.

These tests did not, however, provide information on material interaction. The possibility of such interaction causing gas emission in an operating heat pipe was recognized. It is, however, believed that interaction effect could best be observed in the actual heat pipe, although to a limited extent testing in a simulated heat pipe internal environment may be performed to advantage.

Material Gas Emission to a Hard Vacuum Environment Tests

Sample Pretreatment. - The samples were pretreated to remove the effects of any easily removable surface contaminants. This pretreatment consisted of a surface cleaning with a halogenated hydrocarbon solvent and vacuum baking. The vacuum baking was performed at 70°C (155°F) at a vacuum $\leq$ 800 dyn/cm^2 for a 24 hour period of time.

Test Method. - The candidate materials which were tested are listed in Table II. These materials were placed in pyrex test tubes in the pretreated condition. All glass thermocouple gages and connecting glass tubing were fused to the pyrex test tubes as shown on figure 9. The test assemblies were connected to a vacuum system and then pumped down until the pressure in the test assemblies was 1.33 dyn/cm^2 (1 μ Hg) abs. At this time, the connecting glass tubings of the test assemblies were heated and drawn closed at point "A" as shown on figure 9. Readings of test tube pressure as a function of time were taken using a thermocouple vacuum gage.

Test Variables. - The variables affecting the test results were the following:

(1) Surface area of sample

(2) Volume of sample

Table II - SUMMARY OF CONSTANTS USED IN MATERIAL GAS EMISSION TESTS IN A VACUUM

Sample No. and Name		1	2	3	4	5	6	7	8	9	10
Constants		Epoxy-Glass Sheet	Epoxy Adhesive, Low Density and Thermal Conductivity	Epoxy Adhesive Paste High Thermal Conductivity	Filled Epoxy Paste	Epoxy Resin and Triethylene Tetramine	Epoxy Polyimide	Three Dimensional Fabric - Style 6001	Three Dimensional Fabric - Style 6007	Three Dimensional Fabric - Style 6008	TFE Rod
Sample	Length, cm	1.60	2.00	1.30	1.00	1.00	0.70	10.45	10.15	10.15	4.13
	Width, cm	1.40	1.80	1.30	1.00	1.00	0.70	2.54	2.54	2.54	0.95^D
	Thickness, cm	0.35	0.15	0.20	0.60	0.50	0.10	0.23	0.63	0.31	—
	Volume, cm^3	0.78	0.54	0.34	0.60	0.50	0.05	6.11	16.25	8.00	0.28
	Apparent Surface Area, cm^2	6.74	9.05	4.39	4.64	4.28	1.40	58.30	67.70	59.70	18.00
Volume of Enclosure, cm^3,		139	160	143	147	145	147	126	131	123	134
Volume of Enclosing Gas, cm^3		138	159	143	146	144	147	120	115	115	131

Table II - SUMMARY OF CONSTANTS USED IN MATERIAL GAS EMISSION TESTS IN A VACUUM (Cont'd)

	Sample No. and Name / Constants	11	12	13	14	15	16	17	18	19	20
		Polyethylene-Polyester Film	Polyamide-Vinylidene Chloride Copolymer-Polyethylene film laminate	Fluorohalocarbon	Polypropylene Rope	Plexiglass	Irradiated Polyolefin Tubing*	Irradiated FEP Tubing*	Polyethylene Tubing	Vinyl Tubing	Polyethylene Rod
Sample	Length, cm	9.50	10.20	10.20	11.40	0.950	7.62	8.40	7.62	7.62	7.62
	Width, cm	2.54	2.54	2.54	0.635^{D}	0.950	0.635^{OD}	0.950^{OD}	0.635^{OD}	0.792^{OD}	0.95^{D}
	Thickness, cm	0.0114	0.0089	0.0051	—	0.635	0.762	0.051	0.127	0.159	—
	Volume, cm^3	0.28	0.23	0.13	3.60	0.57	1.02	1.21	1.54	2.40	5.40
	Apparent Surface Area, cm^2	48.30	52.20	51.60	23.10	2.42	28.30	49.60	24.70	31.00	22.80
Volume of Enclosure, cm^3		131	132	132	131	122	135	145	145	144	141
Volume of Enclosing Gas, cm^3		131	132	132	129	121	134	144	143	142	139

*Heat Shrinkable, Recovered Condition

Table II - SUMMARY OF CONSTANTS USED IN MATERIAL GAS EMISSION TESTS IN A VACUUM (Cont'd)

	Sample No. and Name / Constants	21	22	23	24	25	26	27	28	29	30
		Polyethylene-Vinylidene-Chloride Copolymer-Polyester film laminate	Three Dimensional Fabric - Style 6028	Three Dimensional Fabric - Style 6020	Polyester-Al Foil-Proprietary Thermoplastic layer film laminate	FEP-Al Foil-TFE-FEP film laminate	Cellophane-Polyethylene-Al Foil-Polyethylene film laminate	Polyproplene Felt	Three Dimensional Fabric - Style 6027	Three Dimensional Fabric - Style 6022	Vinyl Tubing Special Formulation
Sample	Length, cm	7.70	7.62	7.62	7.62	2.54	7.62	7.82	7.62	7.62	7.62
	Width, cm	2.46	2.54	2.54	2.62	2.54	2.86	2.77	2.54	2.54	0.792^{OD}
	Thickness, cm	0.0081	0.874	1.400	0.0120	0.0254	0.00875	0.00875	0.534	0.920	0.159
	Volume, cm^3	0.15	16.90	27.10	0.24	0.16	0.19	0.19	10.30	17.80	2.57
	Apparent Surface Area, cm^2	19.40	56.40	67.10	40.00	12.90	42.40	53.20	49.50	58.00	25.20
Volume of Enclosure, cm^3		133	139	137	131	135	121	132	140	158	139
Volume of Enclosing Gas, cm^3		133	122	110	131	135	121	132	130	140	136

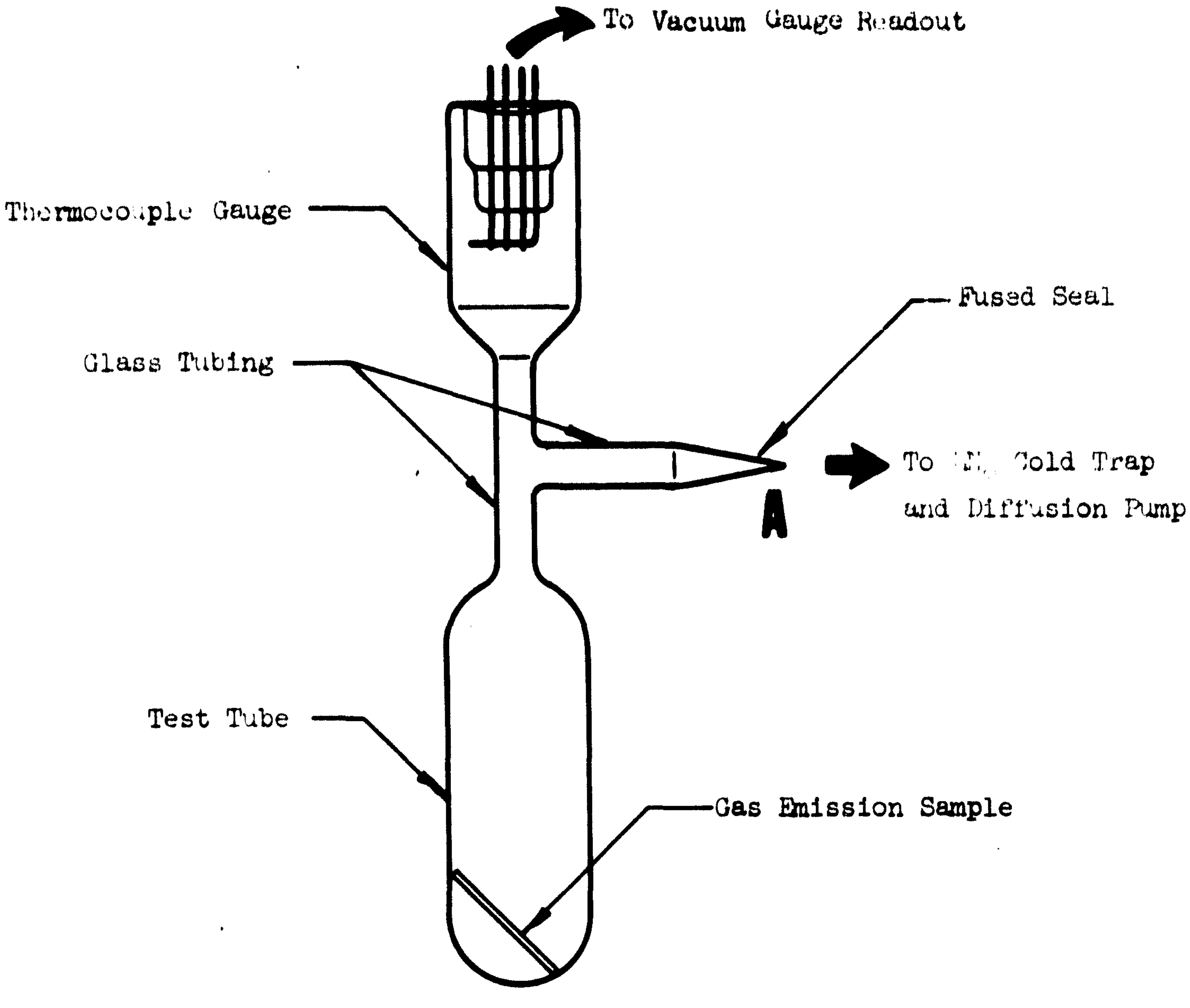


FIG. 9 –SCHEMATIC OF MATERIAL GAS EMISSION TO A VACUUM ENVIRONMENT

(3) Volume of enclosure

(4) Temperature of enclosing gas

(5) Temperature of sample

(6) Composition of enclosing gas

(7) Pressure of enclosing gas

(8) Inward leakage of room air

In order to obtain comparative data between the several candidate suit panel materials tested, data reduction and test method procedures were established to either minimize or account for the effects of the test variables.

Outgassing from the surface of the pyrex test tubes was minimized by a bake out of the test tubes at 560°C (1040°F) for five minutes.

An empty, identically prepared, test tube was included in the tests to provide further correction data for errors introduced into the experiment by the test tube assembly.

A further potential source of error were variations in the initial vacuum obtained by the evacuation and sealing procedure and temperature increases of the gas remaining in the test tubes, resulting from the glass fusing process used to seal the vacuum connection to the test tubes. This increased temperature caused an error in the pressure readings taken immediately after sealing, and prior to cooling of the test tubes at time zero.

In order to minimize these errors, the test data readings taken after the assemblies had stabilized at room temperature (23°C) were plotted and then extrapolated back to time "zero". The curves were then shifted so that the pressure readings of all curves at time "zero" were 1.33 dyn/cm^2 (1 μ Hg).

The pressure readings obtained with the thermocouple gauges were based upon a curve calibrated with air. Therefore, readings of gas emission products other than air must be accounted for. In reference 10, corrections to readings taken with the thermocouple gauges for gases other than air are shown for a number of gases. Using the widest range shown, a reasonable range of possible gas emission constituents would be covered. For this range, a band of data error was determined to be a factor of ± 1.7 relative to air.

The physical dimensions of the samples and volume of the enclosures are shown on Table II. The surface areas listed assume that there are no surface irregularities. This assumption caused an error for samples with surface irregularities such as the quartz fiber wicking material. Due to the difficulty in determining the true surface area of these materials, the outer boundary was used and was called the apparent surface area.

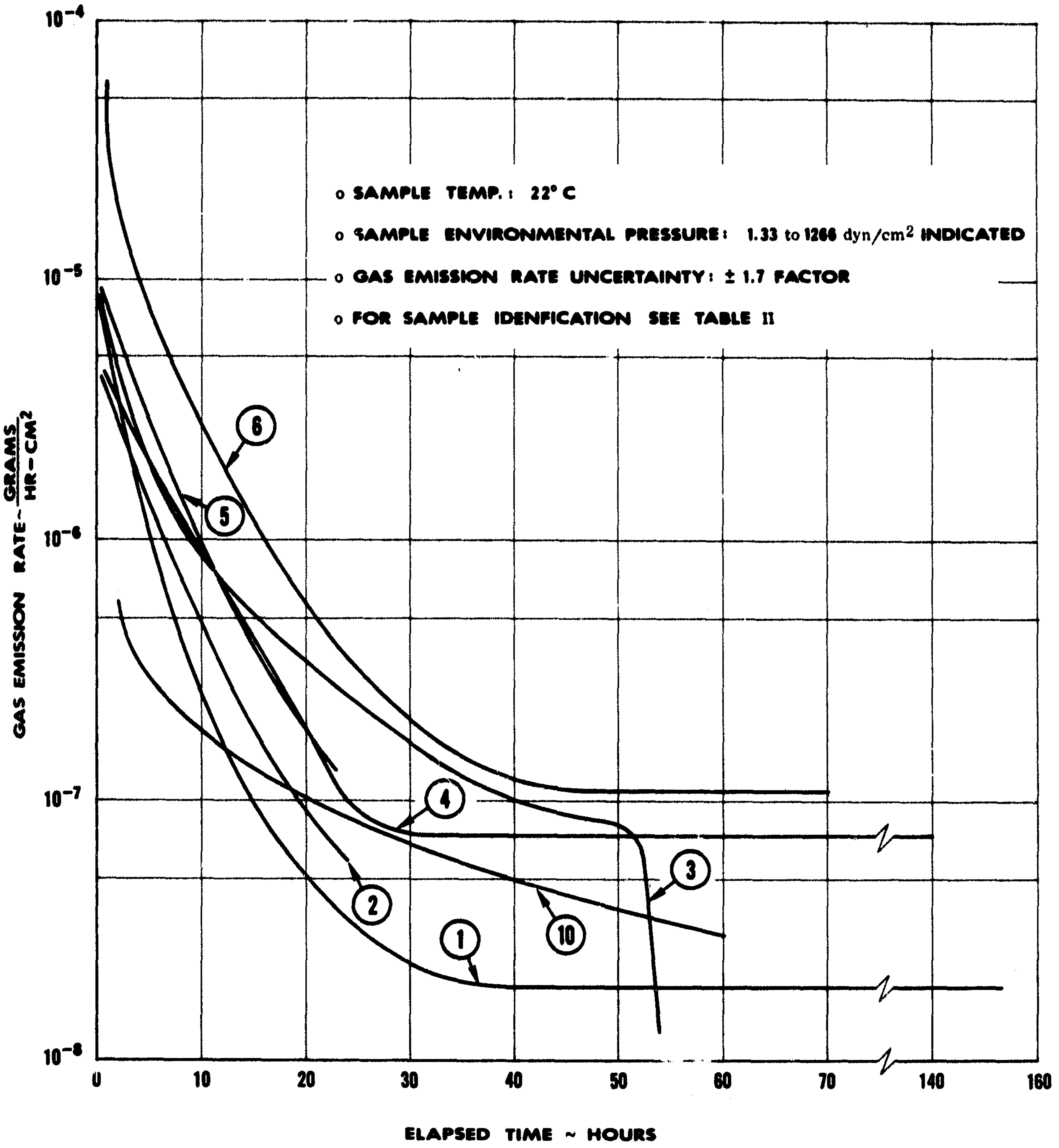
It was assumed that the significant factor for outgassing rate is the sample surface area. Based on this assumption, the effect of the volume of the sample was neglected.

The effect of gas back pressure on the rate of gas emission of this set of samples is not known and cannot be determined from these tests. For this series of tests, the effect of back pressure in the order of magnitude obtained during the tests was assumed to be negligible.

Data Reduction. - Indicated pressure readings as a function of time were taken directly from the test data and plotted using a best fit faired curve. The pressure readings of the empty control tube were also plotted on these figures. The control tube pressure readings represent a measure of gas in-leakage which was subtracted from the outgassing sample pressure readings.

Assuming perfect gas, the mass of the gas emission as a function of time was computed and plotted as a function of time. From these curves, outgassing rates per unit surface area were obtained.

Test Results. - The resulting gas emission rates as a function of time are shown on figures 10 through 13. Based on these results, an effort was made, where feasible, not to apply the following materials in the construction of the space suit prototype panel.



- o SAMPLE TEMP.: 22° C
- o SAMPLE ENVIRONMENTAL PRESSURE: 1.33 to 1266 dyn/cm^2 INDICATED
- o GAS EMISSION RATE UNCERTAINTY: ± 1.7 FACTOR
- o FOR SAMPLE IDENFICATION SEE TABLE II

FIGURE 10 MATERIAL GAS EMISSION RATES PER UNIT SURFACE AREA

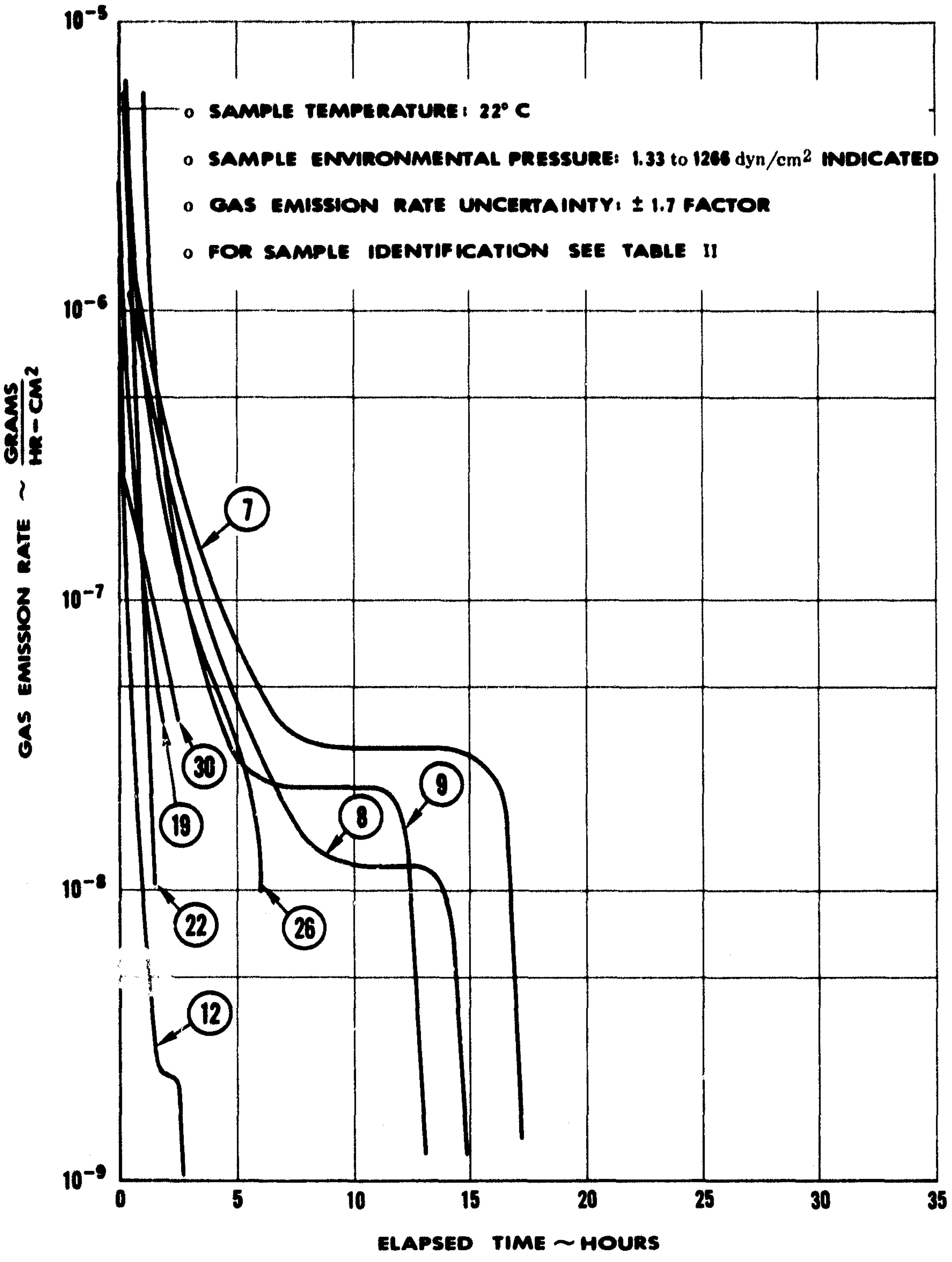


FIGURE 11 MATERIAL GAS EMISSION RATES PER UNIT SURFACE AREA

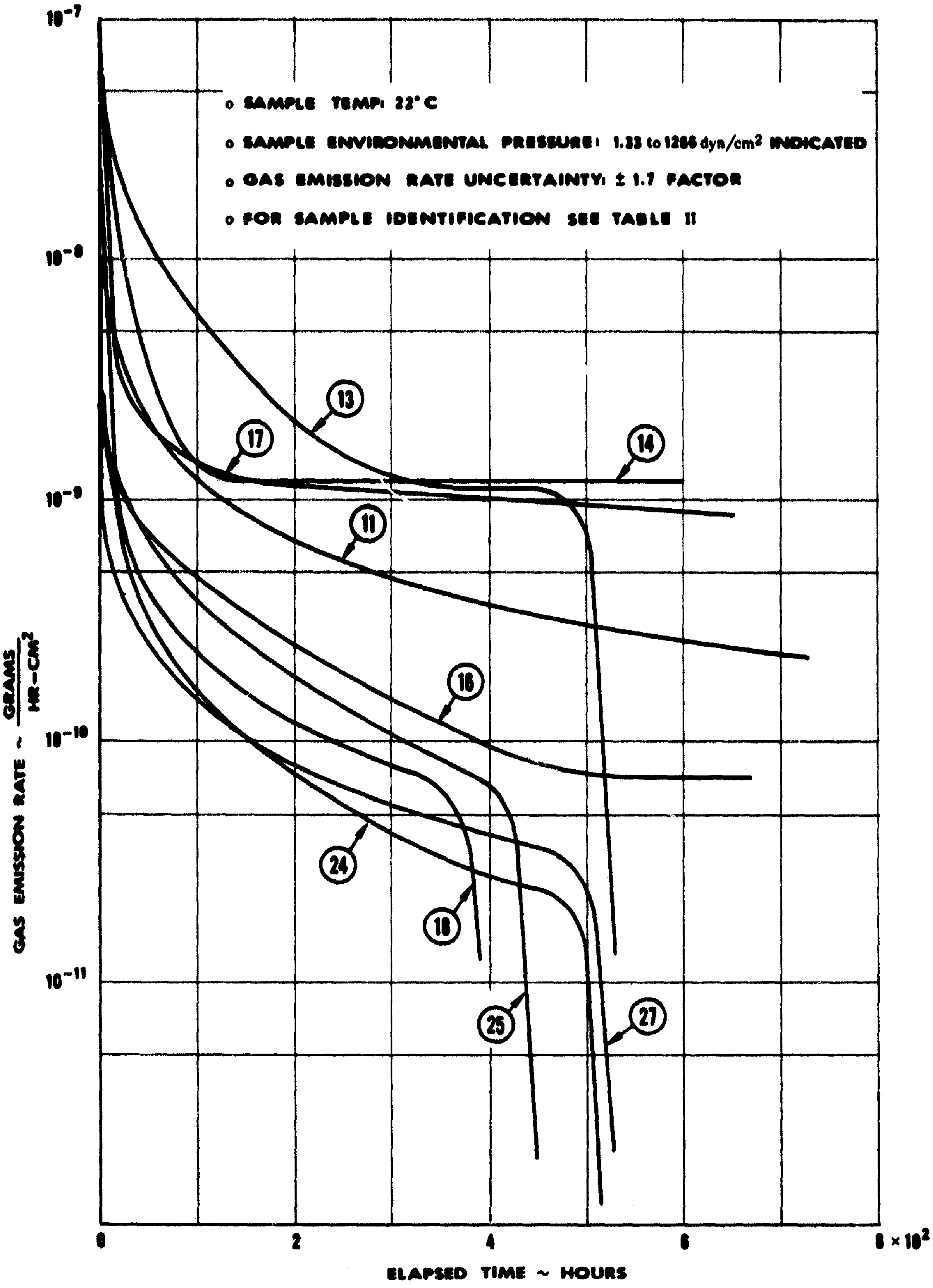


FIGURE 12 MATERIAL GAS EMISSION RATES PER UNIT SURFACE AREA

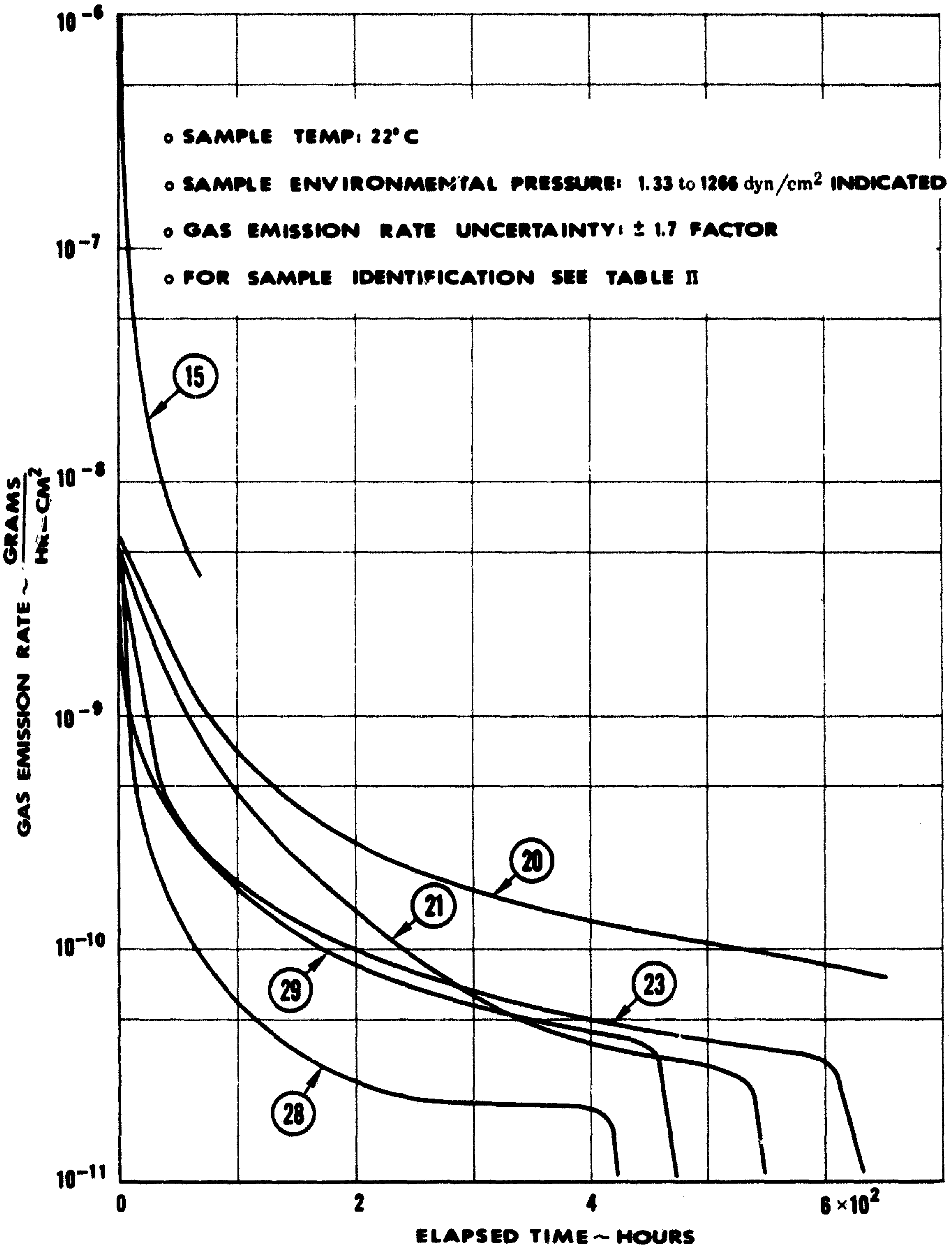


FIGURE 13 MATERIAL GAS EMISSION RATES PER UNIT SURFACE AREA

- Epoxy-Glass Sheet
- Epoxy Adhesive, Low Density and Thermal Conductivity
- Epoxy Adhesive Paste, High Thermal Conductivity
- Filled Epoxy Paste
- Epoxy Resin and Triethylene Tetramine
- Epoxy Polyimide
- TFE Rod
- Plexiglass
- Irradiated Polyolefin Tubing

Looking at figures 10 through 13, it can be seen that gas emission of some of the materials approach zero after exposure to the vacuum environment for a period of time. These materials could probably be pretreated so that their gas emission rate would be zero after the pretreatment. Even the materials which reach a steady gas emission rate could have their initial gas emission rate reduced through proper pretreatment.

Recommendation. - Based on the simple tests developed here, lists of potential heat pipe construction materials can be generated. These lists would present material gas emission rates together with other characteristics such as operating temperature range. In addition, investigations should be performed to determine the necessary pretreatment required to permit use of materials which would otherwise be rejected for use in space suit heat pipes.

Material Gas Emission to a Water Vapor Environment

After the initial preliminary screening of materials was accomplished, the number of materials which could be used as space suit heat pipe construction materials required further screening. A test which more nearly simulated the space suit heat pipe internal environment was devised. In this test, the potential space suit heat pipe construction materials were exposed to the previously selected heat pipe working fluid environment; namely, water vapor.

Sample Pretreatment. - To remove the effects of any easily removable surface contaminants, the samples were pretreated as described previously, and in the same manner as the samples which were tested for gas emission to a vacuum environment.

Test Method. - Each sample was then suspended in one side of a pair of interconnected test tubes. The interconnected test tube assemblies were checked for leaks using a helium leak detector. Inward leakage, if indicated, was eliminated. A vacuum was applied to both sides of the test tube system and outgassed water introduced. Each side of the system was then sealed and separated from the vacuum and fill source by either heat fusion of glass or cold pinch welding of copper tubing. A schematic of the test arrangement is shown on figure 14.

The temperature level of both sides of the test system was maintained equal at 23°C (73°F) by thermally insulating the entire system. Since both sides of this test system are at the same temperature, the pressure, due to the water vapor, would be equal in both sides of the test system and any displacement of water level can be attributed to noncondensable gases evolved from the material test sample. Readings of water level displacement were taken at periodic intervals. These levels were read to within ± 0.01 cm. At the conclusion of the tests, volumes corresponding to the water level readings were obtained. For a water level reading accuracy of ± 0.01 cm, the volumes were accurate to ± 0.06 cm^3.

Test Results. - A summary of the materials tested together with their test results are shown on Table III. The amount of noncondensable gases in a heat pipe which is great enough to cause a serious performance degradation is dependent upon the geometry of the heat pipe. For the controllable space suit heat pipe panel design developed during this program, the volumes of gas emission shown on Table III are acceptable. It was also observed that, in the water vapor environment, all of the materials ceased to emit gases after a finite period of time. This means that these materials can be pretreated so that their gas emission rates in the water vapor environment of a space suit heat pipe, would be essentially zero.

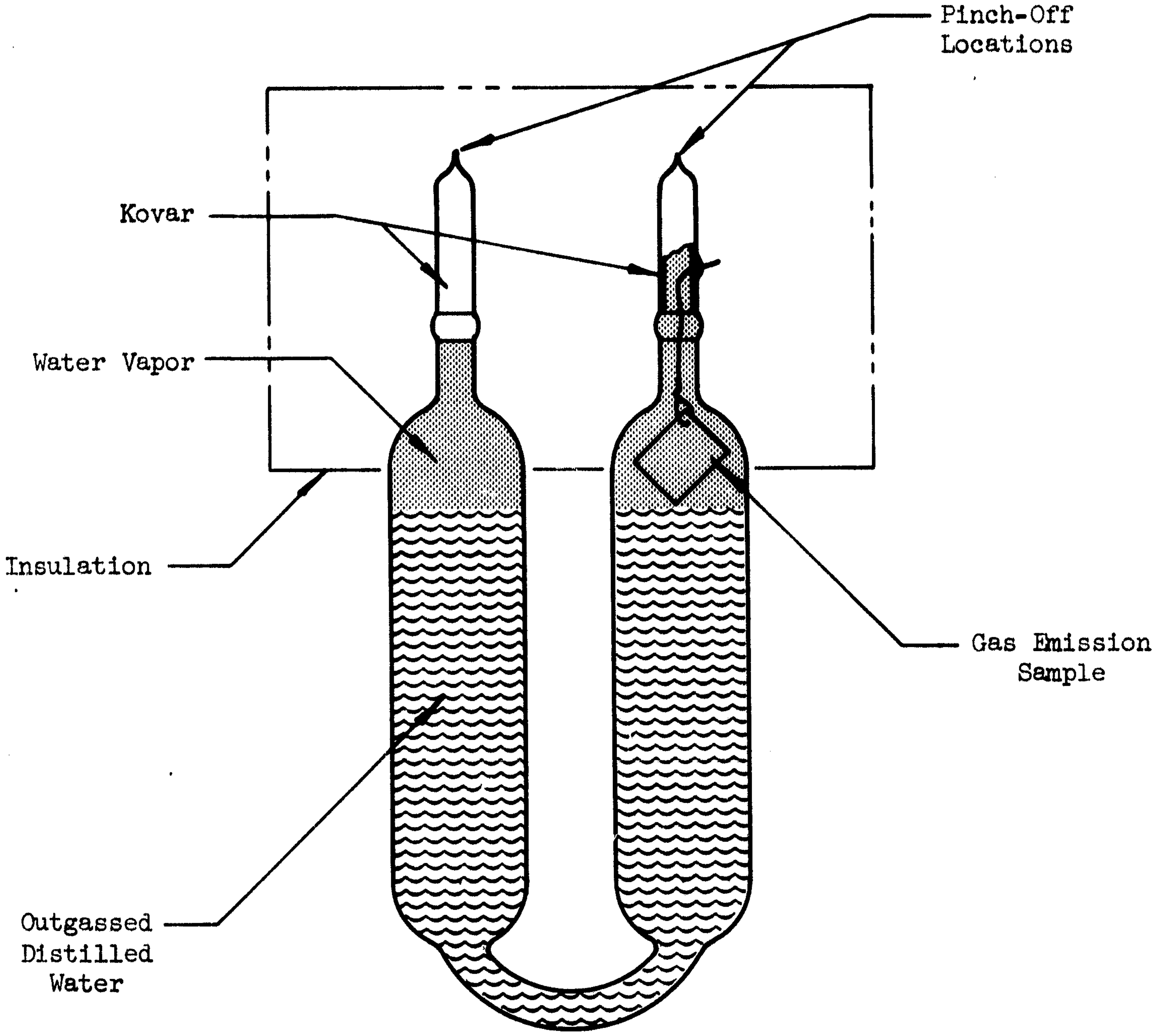


FIG. 14 - SCHEMATIC OF TEST ARRANGEMENT FOR MATERIAL GAS EMISSION IN A WATER VAPOR ENVIRONMENT

Table III - SUMMARY OF CONSTANTS AND RESULTS OF MATERIAL GAS EMISSION TESTS IN A WATER VAPOR ENVIRONMENT

Test Constants and Results		Three Dimensional Fabric - Style 6008	Quartz Fiber Cloth Style C-100-48	Quartz Fiber Sleeving - Style N3/8	Quartz Fiber Sleeving - Style B-24	FEP film	Epoxy Glass Sheet	Vinyl Tubing Special Formulation	Polypropylene Felt	Al Foil-Proprietary Thermoplastic Layer film laminate
Sample	Length, cm	2.54	2.54	2.54	30.40	2.54	2.54	2.54	2.54	2.54
	Width, cm	2.22	2.54	0.95	0.10	2.54	2.22	0.79 OD	2.54	2.54
	Thickness, cm	0.318	0.064	0.071	0.056	0.005	0.318	0.110	0.159	0.010
	Volume, cm^3	1.80	0.413	0.171	0.170	0.030	1.80	0.80	1.03	0.064
	Apparent Surface Area, cm^2	14.50	13.50	5.32	6.43	12.90	14.50	10.60	14.50	12.90
Volume of Sample Material Gas Emission, cm^3		0.95	1.27	0.00	1.07	0.00	0.48	0.00	0.60	0.00
Gas Emission Elapsed Time, hours		242	218	—	960	—	168	—	97	—
Total Test Observation Time Period, hours		842	1290	816	1121	1123	1095	192	546	450
Volume of Material Gas Emission per Unit Surface Area, cm		0.065	0.094	0.00	0.167	0.00	0.033	0.00	0.041	0.00

Heat Pipe Enclosure Materials

Due to the problem of heat pipe performance deterioration resulting from inward leakage of the space suit pressurizing gas, heat pipe enclosure materials should be selected so that their permeability to potential space suit pressurizing gases will be minimized. The present concepts of space suit variable conductance devices include the use of flexible heat pipes located adjacent to the astronaut and, therefore, in the space suit pressurizing gas environment. Inward leakage into the rigid portion of heat pipes is much less severe a problem since the rigidity allows the designer a much greater latitude in material selection. This inward leakage can be minimized through proper selection of materials and thicknesses; but, the problem of finding an enclosure material for the flexible heat pipes is more difficult. This problem and others associated with the development of flexible heat pipes are discussed in more detail in reference 2 and 3. The desired characteristics of flexible heat pipe enclosure films include the following:

- low gas permeability
- low water-vapor permeability
- flexibility
- high tear strength
- low flammability
- ease of fabrication
- compatibility with heat pipe materials and working fluid

Based on these desired characteristics, the search for an enclosure material was initially concentrated on plastic films. A literature search (ref. 11 through 20) was conducted to find a suitable enclosing film.

The film materials which showed the most promise because of their characteristics are shown, together with selected properties, on Table IV. As this table shows, all of the plastic films are somewhat permeable to atmospheric gases and water vapor. Vinylidene chloride copolymer film material absorbs practically no water, has

Table IV - SELECTED CHARACTERISTICS OF FLEXIBLE PLASTIC FILM MATERIALS

Type of Plastic Base / Characteristics		Fluorinated Ethylene-Propylene	Polyamide (nylon 6)	Fluorohalo-Carbon	Polyethylene (low density)	Polyethylene (high density)	Polyvinyl Fluoride	Polyester	Vinylidene Chloride Copolymer	Polyimide
Water Absorption, 24 hrs, %		<0.01	9.5	0.0	<0.01	Nil	<0.5	<0.8	Negligible	2.9
Permeability to gases, cc/645sq cm/0.025mm thickness/24 hrs/atmos./@ 25° C†	CO_2	316	9.7 to 45	30	4200 @ 30°C	720 @ 30°C	15	16	3.0 to 4.6	45
	H_2	N/A	90 to 250	220 to 330	N/A	N/A	58	100	N/A	250
	N_2	148	0.9 to 6.0	2.5	320 @ 30°C	55 @ 30°C	0.25	1	0.12to0.16	6
	O_2	325	2.6 to 25	12	920 @ 30°C	183 @ 30°C	3	6	0.8 to 1.0	25
Water Vapor Permeability, cc/645sq cm/0.025 mm thickness/24 hrs/atmos./@ 25° C†		0.46	5.4 to 20 @ 38°C	0.055 @ 38°C	14	1.8 to 2.2	3.24	1.8	0.20 @ 38°C	5.4
Resistance Rating*	Strong Acids	E	P	E	E	E	E	G	E	G
	Strong Alkalies	E	E	E	E	E	E	G	E - G	P
	Grease and Oils	E	E	E	P	G - E	E	E	E - G	E
	Organic Solvents	E	E	E	G	G	E	E	G - F	E
	Water	E	E - P	E	E	E	E	E	E	E
	High Relative Humidity	E	E - P	E	E	E	E	E	E	E
	Flammability	NF	SE	NF	SB	SB	SB-SE	SB-SE	SE	SE
Heat Sealing Temp. Range, °C		282 to 371	193 to 232	177 to 204	121 to 204	135 to 204	204 to 219	No	No	288 to 316
Service Temperature Range, °C		-255 to 274	-73 to 204	-196 to 199	-57 to 93	-46 to 121	-73 to 107	-65 to 149	-18 to 93	-268 to 399

Notes: †Permeability data taken at 25° C, except where noted.
*Code: E = Excellent, G = Good, M = Moderate, F = Fair, P = Poor; N/A = Not Available
NF = Non-flammable, SB = Slow burning, SE = Self extinguishing, SB-SE = Slow burning to self extinguishing

the lowest water and gas permeability rates and exhibits excellent chemical resistance qualities, but, it does possess some qualities which are not suited for space suit flexible heat pipe application. This film material is not heat sealable except for very thin films and the propagating tear strength of the material is as low as 10% of polyester, for example. Heat sealability is a desirable characteristic at this time. This is to facilitate fabrication without going into a program to develop methods of sealing various film materials. One of the characteristics of this material is that it can be laminated to other materials through the use of adhesives. Using this quality, vinylidene chloride copolymer films can be laminated to other films with a resulting laminate possessing the best characteristics of each.

Polyamide, fluorohalocarbon, polyvinyl fluoride, polyester and polyimide all possess approximately the same order of magnitude of oxygen, nitrogen, and water vapor permeability. At this point, test models of flexible heat pipes using a 0.11 mm thick film of polyester with a coating of polyethylene for heat sealability were constructed. Also tested was a flexible heat pipe with an enclosure material of polyester, vinylidene chloride copolymer, and polyethylene of 0.0625 mm total thickness. The thickness of the vinylidene chloride copolymer was approximately 0.0025 mm. Heat pipe performance degradation was noted after several hours of operation of both heat pipes (see ref. 3).

These two plastic laminates were typical of the types of laminates generally available as an off the shelf item. It can be seen, however, that an improvement can be made if the laminate were to be tailored to flexible heat pipe requirements. For example, a laminate consisting primarily of vinylidene chloride copolymer with a thin coating of polyethylene for ease of fabrication due to its heat sealability and a thin layer of some strong flexible plastic such as polyester for tear strength can be envisioned. The laminate would be sized to trade-off permeability characteristics with flexibility, and strength characteristics.

Since the search for enclosure materials was limited to available materials, the search shifted to laminates with a metal foil layer as the gas and water vapor barrier. This approach showed promise of being a workable solution as seen by the successful flexible heat pipe test runs described in reference 3. Again, more can be done to tailor these laminates to flexible heat pipe requirements. The quality of metallic films can be better controlled. Flexibility, permeability and strength characteristics can be traded off to obtain an optimized enclosure for flexible heat pipes.

WICK BONDING

In order to improve the effectiveness of heat pipes, the overall thermal resistance from the outside surface to the wick active surface should be minimized. One component of this overall resistance is the resistance from the heat pipe enclosure material to the wick material. To reduce this component's resistance, the wick should be held in close contact with the heat pipe enclosure surface. One method of achieving this is to bond the wick to the enclosure surface.

The problems associated with the bonding of capillary materials or wicks to substrates were discussed in detail in references 1 and 3. Briefly, the problem is to prevent the bonding material from being drawn into the wick by capillary action. This penetration by the bonding material would, upon filling the capillaries, destroy the wicking action of the capillary materials.

<u>Problem Approach</u>. - The approach taken to solve this problem was to develop a technique which would limit the penetration of the bonding material to a small fraction of the wick depth.

Using the experience gained in the first phase of this program; 1) adhesives which are viscous when applied to wicks, and 2) film thermoplastics used as adhesives, were the two types of bonding materials studied.

<u>Literature Search</u>. - A literature search (ref. 11 through 27) was initiated to find suitable bonding materials. Tables IV and V present a summary of data on selected adhesives and thermoplastics which are promising for space suit heat pipe bonding. Due to their chemical resistance, thermoplastics to be used as adhesives were the type of bonding agents selected to be studied further. Of the thermoplastics listed on Table IV, the following were selected to be tested:

- Fluorinated Ethylene Propylene (FEP)

Table V - SUMMARY OF ADHESIVES

Adhesive Type	Melt Temperature Range, °C	Bond or Cure Temperature, °C	Application Method	Rating for Resistance*	Upper Service Temp., °C
Modified Synthetic Rubber	177 - 191	177 - 191	Spray, brush, dip, extrusion, knife, paint, roller, or strip as appropriate.	1	93 - 177
Polyester	146 - 177	146 - 177	↓	4	66 - 93
Polyester	93 - 121	93 - 121		4	66 - 93
Polyester	121 - 146	121 - 146		4	66 - 93
Polyester	138 - 171	138 - 171		4	66 - 93
Curing Agent	—	—		—	—
Acrylic	—	—		6	93
Syntnetic Rubber Resin Base	177 - 191	60 to 90 seconds at 77° C to 105° C	Knife spreader or roll coater.	1	146 - 177
Synthetic Rubber Base	177 - 191	24 hours at room temperature.	Brush, spray, or roll coater.	1	93 - 177

Table V - SUMMARY OF ADHESIVES (Cont'd)

Adhesive Type	Melt Temperature Range, °C	Bond or Cure Temperature, °C	Application Method	Rating for Resistance*	Upper Service Temp., °C
Epoxy	—	7 days at 240° C or 1 hour at 93°C	Knife spreader	3	to 260
TFE Primer for Steel	—	302 - 399	Spray	—	260 - 288
FEP Enamel	282 - 371	302 - 399	Spray	—	274

*Rating for resistance when service environment is steam/hot water.
1 denotes best performance, and 6 denotes poorest performance.

- Fluorohalocarbon
- Polyethylene laminates

As can be seen from this table, fluorinated ethylene propylene, fluorohalocarbon and polyvinyl fluoride are the films with the best chemical resistance rating. Polyethylene is not quite as good in its chemical resistance properties. Although polyvinyl fluoride did possess excellent resistance characteristics, its service temperature range is much narrower and within the band which FEP and fluorohalocarbons can provide. It was therefore not selected for further testing. Polyethylene-polyester laminate was selected for further testing because of its lower heat sealing temperature. Also, because it is a laminate, advantage can be taken of the different fusion temperatures of the materials. The polyester would act as a barrier to the flow of lower melting temperature polyethylene.

Bonding Sample. - Wicks were bonded to approximately 6 cm length by 2 cm width by 0.08 cm thick stainless steel substrates. To facilitate tests which were performed on the wick bonds, the substrate was cut 0.6 cm from the end to form a tab (see fig. 15). The substrate surface was roughened and cleaned by sandblasting before the wicks were bonded; the wicks and thermoplastics were washed in a solvent. In order to effect a good bond, suitable pressure, adequate time at a temperature above the flow point of the thermoplastic, cooling under pressure to a temperature where the thermoplastic becomes hard, and clean surfaces are required. The bonding parameters used for bonding each sample are shown on Table VI.

Wick Bonding Sample Environment Exposure. - In order to determine whether or not these thermoplastics can be used as satisfactory wick bonding materials when in contact with potential heat pipe working fluids, wick bonding tests were conducted. These tests consisted of exposing the wick bonds to the following environments:

- Distilled water immersion at room temperature of 23°C (73°F) for 17 days, then at elevated temperature of 72°C (160°F) for 17 days.
- Distilled water immersion at reduced temperature of 2°C (35°F) for 17 days.

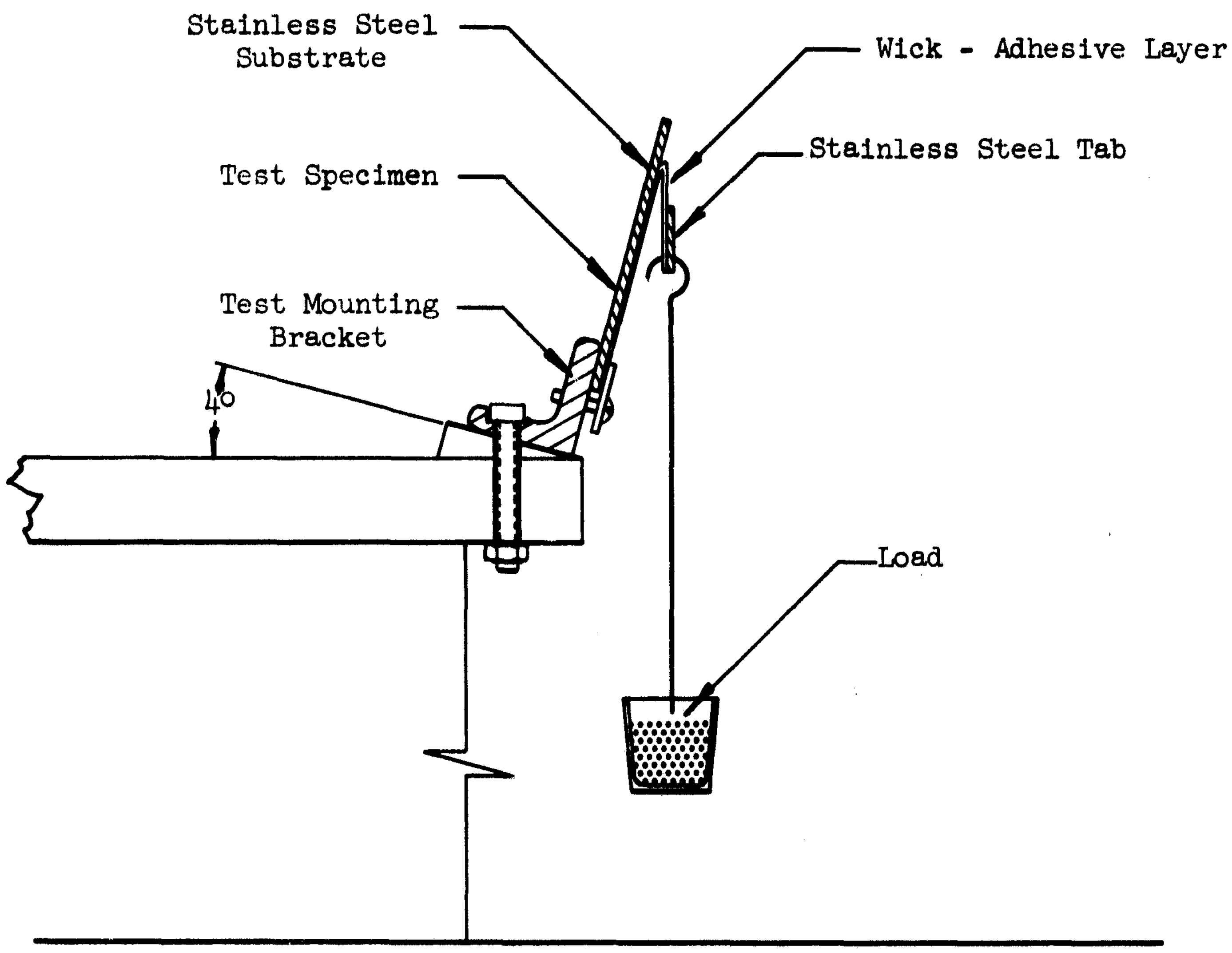


FIG. 15 – SCHEMATIC OF WICK BONDING PEEL STRENGTH TEST APPARATUS

Table VI - WICK BONDING PARAMETERS

No.	Wick Material	Bond[a]			
		Material[b]	Temper-ature ~ °C	Pressure ~ dynes/sq cm	Duration at temp. ~ minutes
111	Quartz Fiber Cloth	FEP	302	2.07×10^6	0.01
112	Polypropylene Felt	PE-PO-PE	148	1.9×10^5	5.00
113	Quartz Fiber Cloth	Fluoro-plastic	193	1.9×10^5	5.00
114	Quartz Fiber Cloth	PE-PO-PE	162	1.9×10^5	5.00
101	Quartz Fiber Cloth	PE-PO-PE	162	1.9×10^5	5.00
103	Quartz Fiber Cloth	Fluoro-plastic	193	1.9×10^5	5.00
105	Polypropylene Felt	PE-PO-PE	148	1.9×10^5	5.00
107	Quartz Fiber Cloth	FEP	302	2.07×10^6	0.01
102	Quartz Fiber Cloth	PE-PO-PE	162	1.9×10^5	5.00
104	Polypropylene Felt	PE-PO-PE	148	1.9×10^5	5.00

Table VI - WICK BONDING PARAMETERS (Cont'd)

No.	Wick Material	Bond[a]			
		Material[b]	Temperature ~ °C	Pressure ~ dynes/sq cm	Duration at temp. ~ minutes
106	Quartz Fiber Cloth	Fluoroplastic	193	1.9×10^5	5.00
108	Quartz Fiber Cloth	FEP	302	2.07×10^6	0.01
G	Quartz Fiber Cloth	PE-PO-PE	165	1.9×10^5	5.00
H	Quartz Fiber Cloth	Fluoroplastic	193	1.9×10^5	5.00
I	Quartz Fiber Cloth	FEP	302	2.07×10^6	0.01
L	Polypropylene Felt	PE-PO-PE	148	1.9×10^5	5.00
A1	Quartz Fiber Cloth	PE-PO-PE	162	1.9×10^5	5.00
B1	Quartz Fiber Cloth	Fluoroplastic	193	1.9×10^5	5.00
C1	Quartz Fiber Cloth	FEP	302	2.07×10^6	0.01
J	Polypropylene Felt	PE-PO-PE	148	1.9×10^5	5.00

Table VI - WICK BONDING PARAMETERS (Cont'd)

No.	Wick Material	Bond[a]			
		Material[b]	Temperature ~ °C	Pressure ~ dynes/sq cm	Duration at temp. ~ minutes
D1	Quartz Fiber Cloth	PE-PO-PE	162	1.9×10^5	5.00
E1	Quartz Fiber Cloth	Fluoroplastic	193	1.9×10^5	5.00
F1	Quartz Fiber Cloth	FEP	302	2.07×10^6	0.01
K	Polypropylene Felt	PE-PO-PE	148	1.9×10^5	5.00

Notes:

[a] Substrate is stainless steel with sandblasted surfaces.

[b] Code: PE-PO-PE = "laminate" of polyethylene, polyester, polyethylene.

FEP = fluorinated ethylene propylene.

Fluoroplastic = fluorohalo carbon.

- Methanol immersion at room temperature for 17 days, then at elevated temperature of 72°C (160°F) for 17 days.

- Methanol immersion at reduced temperature of -68°C (-90°F) for 17 days.

In addition, another set of wick bonding samples were exposed to a room environment. This set of samples were used as standards with which to judge any degradation in wick performance due to long duration exposures at extremes of temperature for two possible heat pipe working fluids.

Wick Bonding Tests. - At the conclusion of the environmental exposures, tests to determine adhesion, fluid retention, wicking capability and peel strength were performed. Adhesion data obtained were qualitative and the degree of adhesion was described in the following manner:

- Good - no noticeable change in the bond.

- Separated 0.32 cm (1/8 inch) - wick separated at least 0.32 cm from entire substrate edge.

- Completely separated - wick is completely separated from the substrate.

Fluid retention data were obtained on a beam balance accurate to ± 0.0001 gm. The samples were weighed to the nearest ± 0.01 gm or ∓ 5% of the minimum gain in weight due to wick bond fluid retention of a sample.

The wicking capabilities of the bonded samples were tested quantitatively by comparing the vertical lift rate of the sample exposed to temperature and immersed in either water or methanol (test samples) to the sample of similar construction which was exposed only to room air environment (control sample). The following notations were used in describing the results of these tests:

- Good - no noticeable difference between the test sample and the control sample.

- Fair - test sample wicked from 60% to close to 100% as fast as the control sample.
- Poor - test sample wicked less than 60% as fast as the control sample.

Peel strength data were obtained by using an apparatus as shown on figure 15. The value obtained was the average load per unit width of bond line required to separate the wick from the substrate at a separation angle of 176 degrees from the vertical. The load was applied by progressively adding weight to a container with an initial weight of 51 gms. This weight was added at the rate of 90 gms/minute with the container being suspended from the wick bonding sample tab. The loads obtained were measured on a platform balance with an accuracy of ± 0.1 gms. The error due to the accuracy of the load was then as great as 0.2%. For the samples which failed, the peel strength was denoted by an equal to or greater than notation of peel strength value, and for samples which peeled when the initial load was applied, the peel strength value was denoted by an equal to or less than notation.

Test Results. - The results of these tests are shown on Table VII. Very little degradation in fluid retention of the wick bond samples was observed in these tests with the exception of the samples which used polypropylene felt as the wicking material and water as the fluid to be retained in the wick. It was found that prolonged immersion of the polypropylene felt in water removes the wetting agents in the felt, thus causing the wick to be water proofed. This problem was also the cause of the poor performance of the polypropylene felt samples when tested for wicking capability with water. The wicking capability of the remaining samples were not affected by the test environmental exposures. The results of the adhesion observations indicates that the bonding material of polyethylene polyester laminate should be avoided for wick bonds which will be exposed to methanol and temperatures as high as 72°C (160°F).

A reduction in peel strength was noted for the wick bond samples which were exposed to the elevated temperature of 72°C (160°F) environment. The reduction in peel strength was less when FEP was used as the bonding material and the reduction in peel strength was the worst for samples which used the polyethylene-polyester

Table VII - WICK BONDING TEST PARAMETERS AND RESULTS

No.	Environment Exposure			Adhesion^b	Data Taking Pretreatment	Fluid			Peel Strength ~ gms/cm
	Immersion Fluid	Fluid^a Temperature ~ ^oC	Test Duration ~ days			Type	Fluid^c Retention ~ %	Wicking^d Capability	
111	Room Air	23	34	Good	None	Distilled Water	4.2	—	535
						Methanol	3.4	—	
112	Room Air	23	34	Good	None	Distilled Water	10.3	—	481
						Methanol	4.3	—	
113	Room Air	23	34	Good	None	Distilled Water	7.5	—	≥433
						Methanol	3.3	—	
114	Room Air	23	34	Good	None	Distilled Water	6.0	—	462
						Methanol	10.2	—	
101	Distilled Water	23	17	Good	None	—	—	—	—
		72	17	Good	Room Air Dry	Distilled Water	6.3	Good	≤23
103	Distilled Water	23	17	Good	None	—	—	—	—
		72	17	Good	Room Air Dry	Distilled Water	4.5	Good	≥26
105	Distilled Water	23	17	Good	None	—	—	—	—
		72	17	Good	Room Air Dry	Distilled Water	0.0	Poor	44
107	Distilled Water	23	17	Good	None	—	—	—	—
		72	17	Good	Room Air Dry	Distilled Water	10.0	Good	110
102	Methanol	23	17	Good	None	—	—	—	—
		72	17	Completely Separated	Room Air Dry	Methanol	13.0	Good	-0-
104	Methanol	23	17	Good	None	—	—	—	—
		72	17	Completely Separated	Room Air Dry	Methanol	6.7	Good	-0-

Table VII - WICK BONDING TEST PARAMETERS AND RESULTS (Cont'd)

No.	Environment Exposure			Adhesion[b]	Data Taking Pretreatment	Fluid			Peel Strength
	Immersion Fluid	Fluid[a] Temperature ~ °C	Test Duration ~ days			Type	Fluid[c] Retention ~ %	Wicking[d] Capability	~ gms cm
106	Methanol	23	17	Good	None	—	—	—	—
		72	17	Good	Room Air Dry	Methanol	3.4	Good	≥28
108	Methanol	23	17	Good	None	—	—	—	—
		72	17	Good	Room Air Dry	Methanol	3.3	Good	56
G	Room Air	23	17	Good	Room Air Dry	Distilled Water	—	—	259
						Methanol	5.3	—	
H	Room Air	23	17	Good	Room Air Dry	Distilled Water	—	—	272
						Methanol	5.5	—	
I	Room Air	23	17	Good	Room Air Dry	Distilled Water	—	—	352
						Methanol	2.7	—	
L	Room Air	23	17	Good	Room Air Dry	Distilled Water	—	—	234
						Methanol	12.0	—	
A1	Distilled Water	2	17	Good	Room Air Dry	Distilled Water	5.0	Good	176
B1	Distilled Water	2	17	Good	Room Air Dry	Distilled Water	4.1	Good	≥204
C1	Distilled Water	2	17	Good	Room Air Dry	Distilled Water	4.7	Good	314
J	Distilled Water	2	17	Good	Room Air Dry	Distilled Water	-0-	Poor	255

Table VII - WICK BONDING TEST PARAMETERS AND RESULTS (Cont'd)

No.	Environmental Exposure			Adhesion[b]	Data Taking Pretreatment	Fluid			Peel Strength ~ gms/cm
	Immersion Fluid	Fluid[a] Temperature ~ °C	Test Duration ~ days			Type	Fluid[c] Retention ~ %	Wicking[d] Capability	
D1	Methanol	-68	17	Good	Room Air Dry	Methanol	3.7	Good	139
E1	Methanol	-68	17	Good	Room Air Dry	Methanol	3.3	Good	293
F1	Methanol	-68	17	Good	Room Air Dry	Methanol	3.7	Good	323
K	Methanol	-68	17	Good	Room Air Dry	Methanol	14.6	Good	547

Notes:

[a] Where there were two different environmental temperatures to which samples were exposed, the exposures were conducted sequentially in the order shown.

[b] The qualitative observations of adhesion of the bond were taken at the conclusion of the environmental exposures. The following notations were used to describe the degree of adhesion:

- good - no noticeable change in the bond.
- separated - wick separated 0.32 cm (1/8") or more from the entire substrate edge.
- completely separated - wick is completely separated from the substrate.

[c] Fluid retention is the weight increase of the bonded wick when in a wet-by-wicking-action condition compared to a room-air-dry condition.

[d] The wicking properties of the bonded samples were tested quantitatively by comparing the vertical lift rate of the sample exposed to temperature and immersed in either water or methanol (test sample) to the sample of similar construction which was exposed only to room air environment (control sample). The following notations were used in describing the results of these tests:

- good - no noticeable difference between the test sample and the control sample.
- fair - test sample wicked from 60% to close to 100% as fast as the control sample.
- poor - test sample wicked less than 60% as fast as the control sample.

laminate as the bonding material with the fluorohalocarbon bonded samples falling somewhere between.

Based on the results of these test exposures, FEP and fluro-halocarbon are superior to the polyethylene polyester laminate for use as materials for bonding wicks to substrates.

THERMAL CONDUCTIVITIES OF METALLIC FELTS AND FOAMS

It is recognized that the thermal conductivity in the heat pipe wick layer when soaked with working fluid, is one of the factors affecting heat pipe performance. Therefore, a limited study was undertaken to try to optimize this factor.

Based on capillary performance as the selection criterion, quartz fiber textile wicks had been selected for use in space suit heat pipes (ref. 4). Thermal conductivity of the working fluid - wick composite could, however, be increased if the thermal conductivity of the wick material can be increased.

Since metals possess higher thermal conductivities than textiles, an attempt was made to determine the order of improvement in composite thermal conductivity which might be obtained by the use of commercially available metallic fiber felts in place of the quartz fiber wick materials. Samples of metallic felts and thermal conductance data were obtained from vendors.

Working fluid - wick layer composite thermal conductivities were determined analytically. Preliminary thermal conductivity information for sintered metallic felts were obtained from a vendor. Since this vendor data is of a preliminary nature, conclusions on the use of sintered metallic felts for heat pipe application which were based on this data are, however, also only preliminary.

As described in reference 4, water had been selected as the space suit heat pipe working fluid. The thermal conductivity of sintered metallic felts and water were combined analytically to obtain the thermal conductivity of wick - water composites. Parallel conductive paths were assumed. This assumption resulted in optimistic values for the thermal conductivities of these composites because of the implied assumption of the shortest heat flow path length in the metallic fibers, perpendicular to the wick surface. The results of this analysis are shown on figure 16 for several sintered metallic felt wicks and water composites.

The styles of quartz fiber wicks considered for space suit heat pipe application are very porous. Therefore, the thermal conductance of these wick - water composites can be assumed to be close to that of a layer of water of equal thickness. At a temperature of 33°C (91°F), the thermal conductivity of quartz is approximately three times as high as water. This assumption is, therefore, pessimistic in regard to the thermal conductance of quartz fiber - water composites. Both assumptions therefore favor metallic fiber wicks. Even with these assumptions, figure 16 shows only little increase in thermal conductivity of wicks and water composites, obtained through the use of sintered metallic fiber wicks.

These results were not encouraging enough to justify further study of other types of metallic wicks within the scope of this program.

DESIGN AND FABRICATION TECHNIQUES USED IN CONSTRUCTION OF SPACE SUIT PROTOTYPE PANEL

An experimental prototype of a suit panel was developed and fabricated in order to verify techniques of application of flexible heat pipe panels and flexible wick and vapor interconnections for two-chamber heat pipes. This prototype includes a rigid condenser chamber of stainless steel, vinyl tube interconnections between the condenser and the evaporator chamber and a flexible evaporator chamber, as schematically shown in figure 17. Design approach and results of tests performed with this prototype are reported in reference 2. Details of fabrication techniques and materials are reported as follows.

Fabrication Techniques

As figure 17 shows, the prototype panel assembly consists of a flat rigid chamber serving as condenser, and of a flat flexible chamber serving as evaporator of a two-chamber heat pipe. The two chambers are interconnected by flexible wick and vapor passages. These passages consist of vinyl tubing. There was one vapor passage

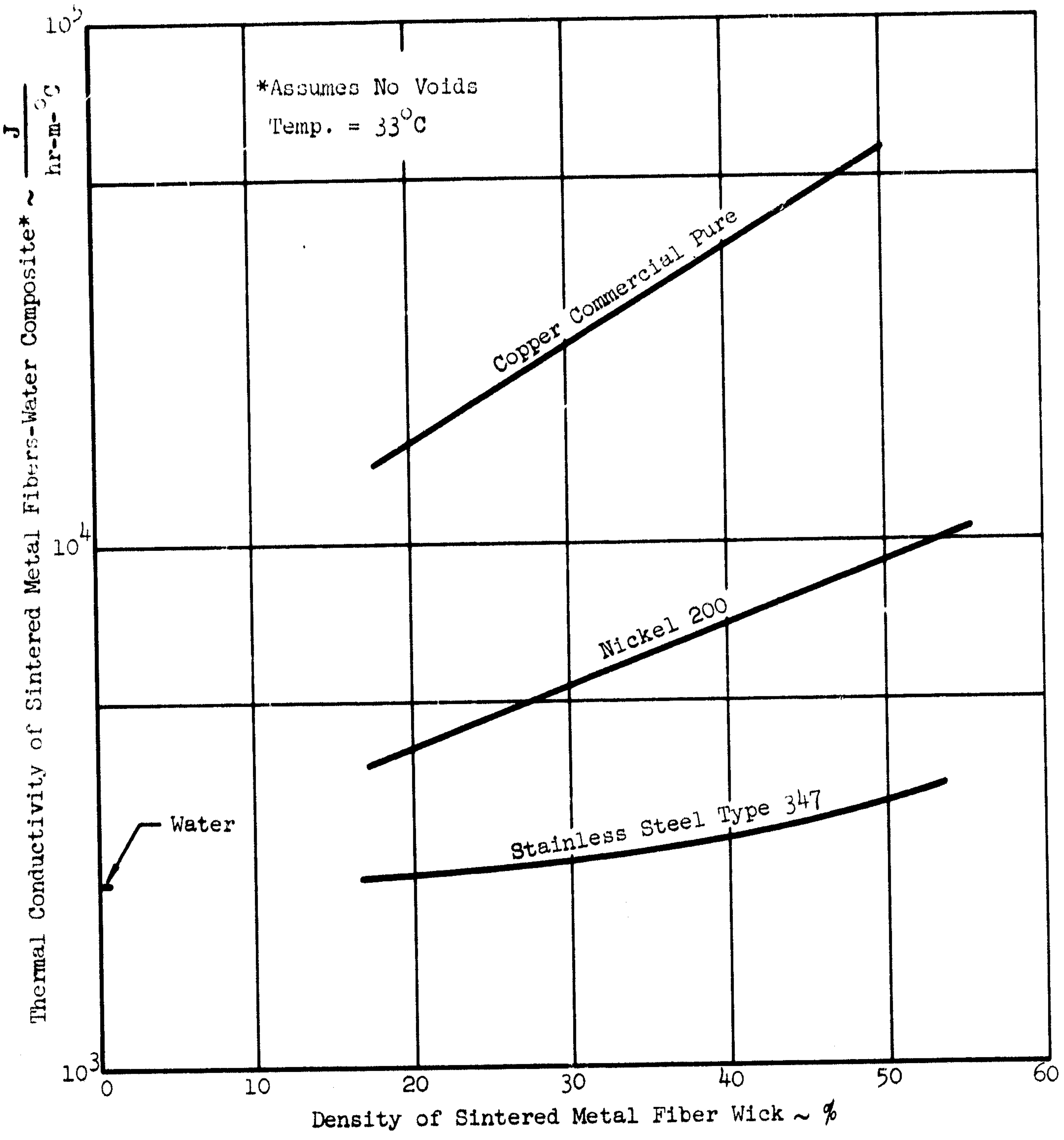


Figure 16 - THERMAL CONDUCTIVITY OF SEVERAL SINTERED METAL FIBER WICK-WATER COMPOSITES AS A FUNCTION OF DENSITY

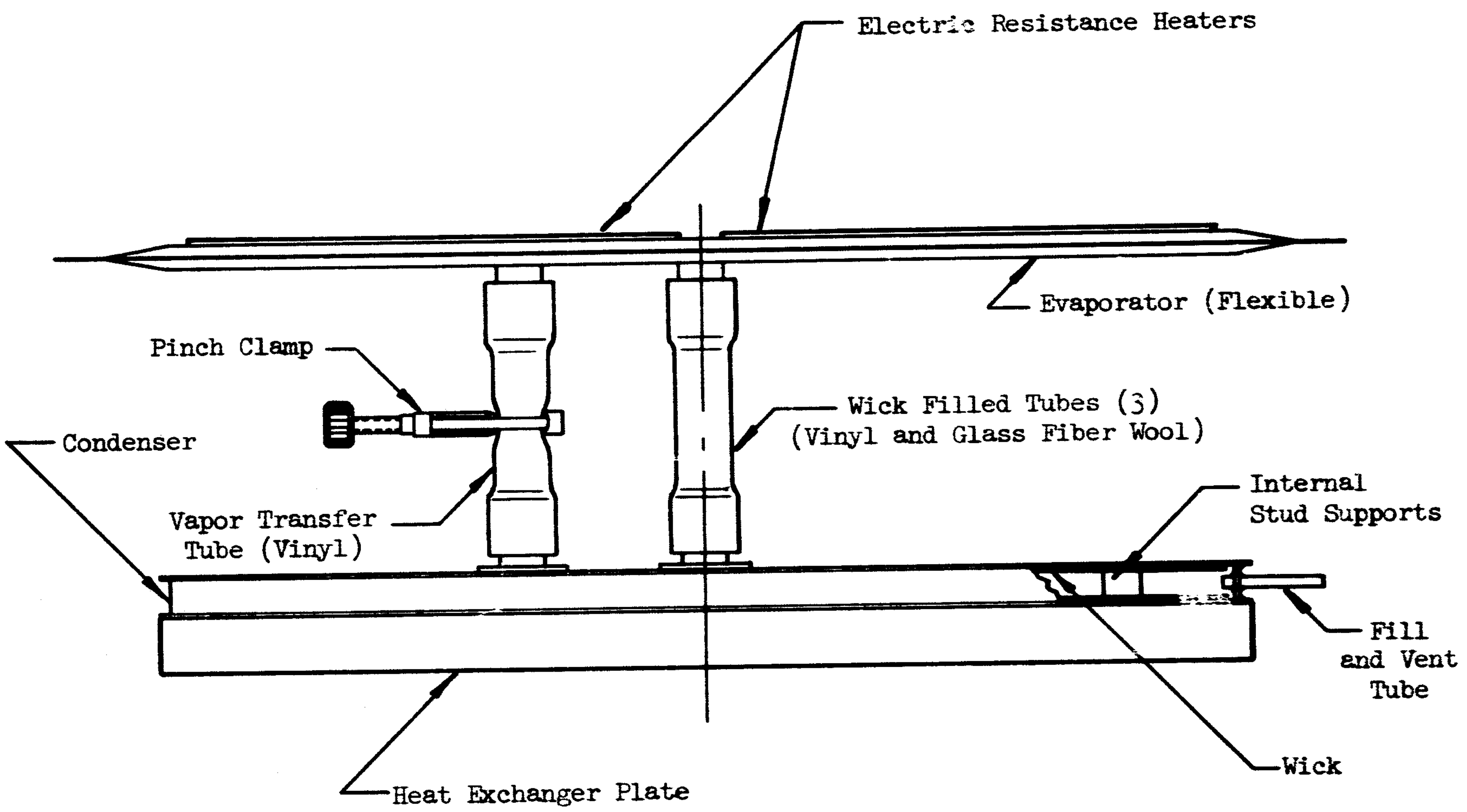


FIGURE 17 - PROTOTYPE PANEL ASSEMBLY

required, and as shown in figure 17, flow of vapor through the vinyl tube can be controlled by a pinch clamp, thereby providing thermal switching capability. Three feeder wicks were required for interconnection of the evaporator and condenser chamber wicks. They consist of vinyl tubing filled with a packing of glass fiber wool. Connection between the flat chambers and the vapor transfer and wick tubes is made with the help of machined fittings attached to the two chambers. Figure 17 also indicates electric resistance heaters and a heat exchanger plate, which were provided for test purposes.

Condensing Chamber. - The rigid condensing chamber is constructed of stainless steel. Soft solder, containing 60% tin and 40% lead by weight, with a melting point of 183°C (362°F) was used for assembly of the stainless steel parts. This design and fabrication technique was selected because it was desirable, for the planned experimentation, that the condenser chamber provide a reliable "sink" for heat and vapor transmitted to it. This removed a potential variable and permitted evaluation of the flexible elements of the device. This construction is, however, obviously not suggested for application to a space suit.

The top plate of the chamber consists of 0.25 mm (0.01 in) thick, No. 303 stainless steel. The bottom plate is of the same material, 0.890 mm (0.035 in) thick. The purpose of a heavier bottom plate is to provide improved flatness for thermal contact with the heat exchanger plate used for experimentation. The flat design of the panel also requires internal stud supports to prevent collapsing of the chamber due to the external pressure which is much higher than the vapor pressure of the working fluid inside the cavity.

The edge of the panel consists of a 6.35 mm (0.25 in) high frame, made from 0.635 mm (0.025 in) thick stainless steel. The internal support studs were cut from 6.35 mm (0.25 in) o.d. stainless steel tubing with 0.375 mm (0.015 in) wall thickness. They were of the same height as the frame, i.e. 6.35 mm.

The wick material selected is a woven quartz fiber cloth. As described in the section on wick bonding, several methods of bonding wicks to metallic substrates had proven to be satisfactory. Considering the melting points of the thermoplastics which can provide satisfactory wick bonding, it can be seen that fluorinated ethylene propylene (FEP) has a melting point well above the melting point of the soft solder used. The melting point of fluorohalocarbon film is also above that of the solder, but much closer and FEP was therefore preferable.

The side of both plates to be covered with the wick was roughened by sandblasting. A layer of FEP, 0.05 mm (2 mils) thick, was put on the roughened side of the stainless steel plates. A layer of quartz fiber cloth was put on top of the FEP film and the "sandwich" so formed placed between steel and silicon rubber slip sheets. The assembly was then put into a heated press. Bonding was performed at 302°C (575°F), at a pressure of 2.07×10^6 dyn/cm^2 and with a dwell time of 5 seconds. The bond was cooled to room temperature prior to removal from the press.

In order to assemble the wick covered plates with the frame and support studs, the wick and FEP film was scraped from the plates at the location of attachment to the frame and to the support studs. Clearance of approximately 2.5 mm (0.1 in) was allowed between the wick and the frame and the support studs. The support studs and the frame were then soldered to the bottom stainless steel plate using the low melting point, 60/40 solder. In order to provide continuation between the wicks attached to the top and bottom plate, three (3) sets of nine (9) discs each of quartz fiber cloth wick, 1.25 cm (0.5 in) diameter were placed between the two plates at three (3) locations corresponding to the three (3) feeder wick tube connections. The top plate was then soldered to the frame. A copper fill and vent line was soldered to the frame and stainless steel tube fittings for the feeder wicks and the vapor passage were soldered to the top plate as shown in figure 17.

Evaporating Chamber. - The flexible evaporating chamber is of flat panel design. The problems of flexible heat pipe design are discussed in detail in references 2 and 3, prepared under the same program. Figure 18 shows a schematic crossection through the type of flexible heat pipe used as evaporating chamber.

A three dimensional fabric was used for internal support, required because, just as in the condenser chamber, internal pressure would be much lower than the pressure of the external environment. A three dimensional fabric is a woven flexible structure of the approximate appearance of corrugated cardboard, made of plastic fiber screen material. Three dimensional fabrics and their application to flexible heat pipes are discussed in references 2 and 3. Three dimensional fabric is manufactured and commercially available in a number of different styles.

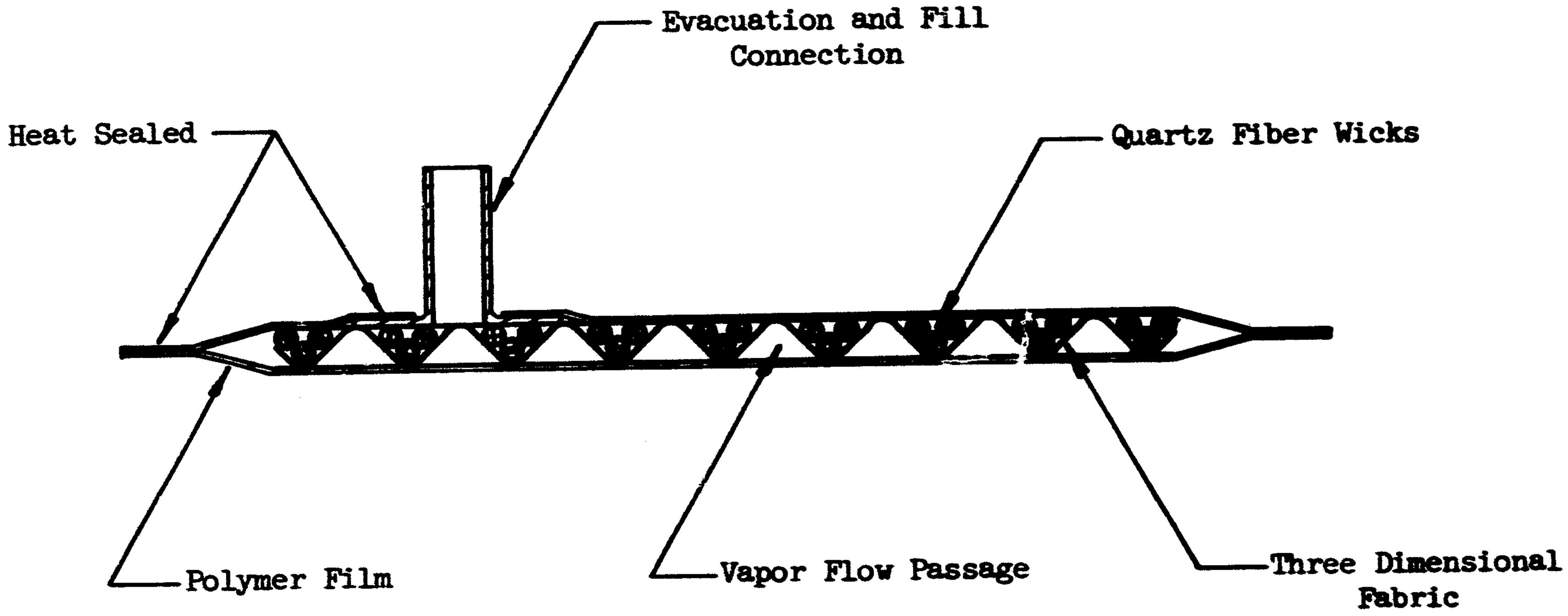


FIG.18 - FLEXIBLE HEAT PIPE PANEL, SCHEMATIC CROSSECTION

Using the results of simple compression tests, three styles of the three dimensional fabric were found to be acceptable for supporting the maximum expected pressure load of 1.02 x 10^6 dyn/cm^2 (14.7 psi). These styles are indentified in other sections of this report as styles 6001, 6007, and 6008. All of these styles consist of woven polyethylene and vinylidene chloride copolymer filaments. Gas emission tests on these styles were performed as described in the section on "Noncondensable Gases" of this report. They were found in these tests to be satisfactory for space suit heat pipe application (see fig. 11). A Material Gas Emission to Water Vapor Environment test, performed on the three dimensional fabric style number 6008 was also found to be satisfactory (Table III). Style 6008 has a geometry which provided convenient location for wick material, and was selected for application in the flexible heat pipes.

A braided quartz fiber tube, 7 mm o.d. was selected as wick. Alternate channels of the style 6008 three dimensional fabric were filled with wick (fig. 19). The remaining open channels provide vapor passages. At the ends of the three dimensional fabric and perpendicular to the channels formed by the corrugations (fig. 18 and 19), header strips of three dimensional fabric, approximately one (1) cm wide and without wick are provided as interconnections between the vapor passages. Additional vapor passages of three dimensional fabric and wick headers are provided at strategic locations to provide unrestricted vapor flow and working fluid distribution. The flexible spacer and wick assembly is encased in a flexible heat sealable aluminum foil - polymer laminate. This laminate was selected after a search for suitable enclosure film materials, as discussed in the section on "Heat Pipe Enclosure Materials". Heat sealing, as shown in figure 18 was used for assembly of the external enclosure and for attachment of four (4) polyethylene fittings to which one (1) vapor transfer and three (3) wick filled tubes are attached (fig. 17).

Feeder Wick. - The three feeder wicks were made by packing glass fiber filtering wool into the polyethylene fittings attached to the flexible chamber, the stainless steel fittings attached to the rigid chamber and into clear flexible vinyl tubing of 1.11 cm (0.438 in) o.d. and 0.635 cm (0.25 in) i.d. The packed vinyl tubing was force pushed onto the polyethylene and stainless steel fittings and was held by force fit. The glass fiber filtering wool was packed to a density of 0.5 gms/cc.

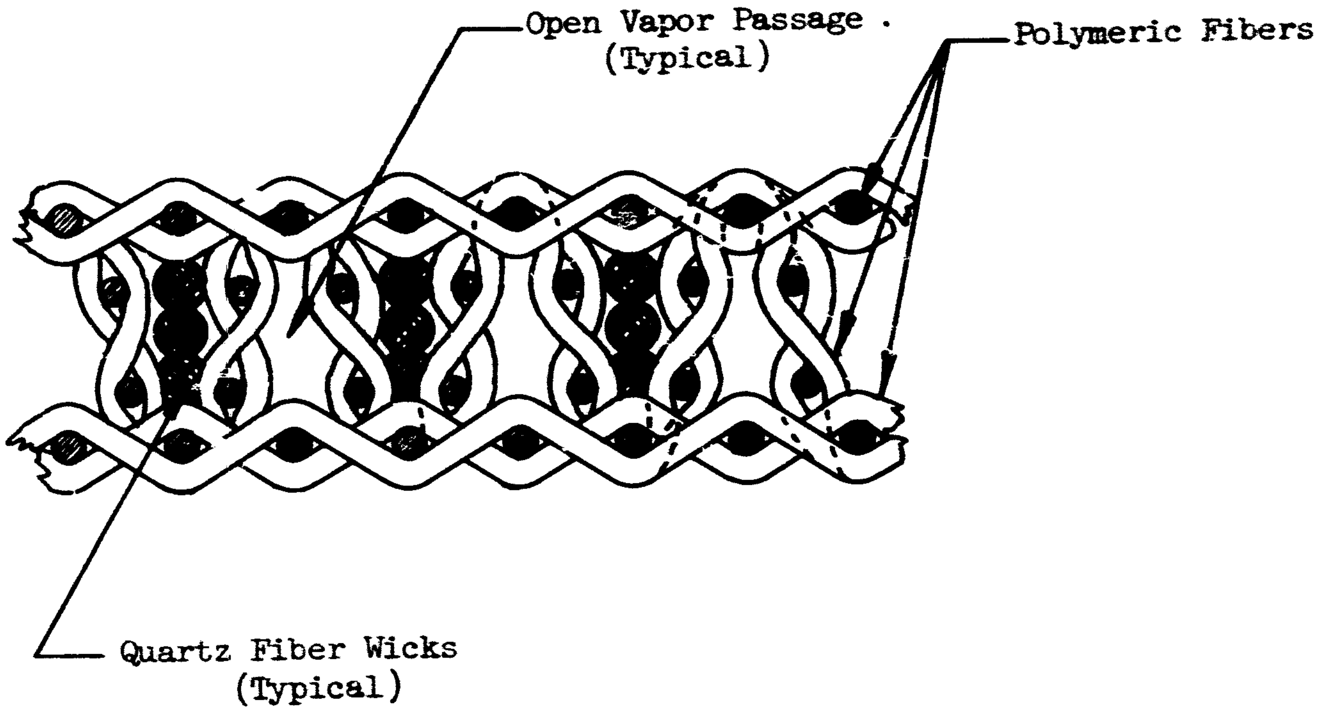


FIGURE 19 - SCHEMATIC CROSS SECTION THROUGH TYPICAL THREE-DIMENSIONAL FABRIC WITH WICKS IN ALTERNATING CHANNELS

Vapor Passage Tube. - The vapor passage tube connecting the rigid and the flexible chambers was made from a piece of clear flexible vinyl tubing of the same type and dimensions used to encase the feeder wick. The method of attachment was also the same, i.e. force fit over polyethylene and stainless steel fittings. The variable conductance (thermal switching) capability is provided by a pinch clamp valve, which can be used to control the vapor flow through the vapor passage tube.

REFERENCES

1. Shlosinger, A. P.; and Jacobi, E. F.: Study of Passive Temperature and Humidity Control Systems for Advanced Space Suits - Material Research Report - First Year. NASA No. CR-73169, September, 1967.

2. Shlosinger, A. P.: Study of Passive Temperature and Humidity Control Systems for Advanced Space Suits - Final Second Phase Report. TRW Systems Report No. 06462-6006-R000, November, 1968.

3. Shlosinger, A. P.: Heat Pipe Devices for Space Suit Temperature Control. TRW Systems Report No. 06462-6005-R000, November, 1968.

4. Shlosinger, A. P.: Study of Passive Temperature and Humidity Control Systems for Advanced Space Suits. NASA No. CR-73168, September, 1967.

5. Mackay, D. B.: Design of Space Powerplants. Prentice Hall, Inc. Englewood Cliffs, N. J., 1963.

6. Sax, N. I.: Dangerous Properties of Industrial Materials. Reinhold Publishing Corp. New York, 1957.

7. Coward, H. F.; and Jones, G. W.: Limits of Flammability of Gases and Vapors. Bulletin 503, Bureau of Mines, 1952.

8. West, C. J., ed.: International Critical Tables of Numerical Data, Physics, Chemistry and Technology. McGraw-Hill Book Co., Inc., 1933.

9. Hodgman, C. D., ed.: Handbook of Chemistry and Physics. 42nd edition. Chemical Rubber Publishing Co. Cleveland, Ohio, 1960.

10. Anon.: Instruction Manual - Hastings Vacuum Gauge Model VT-6. Hastings-Raydist, Inc. Hampton, Virginia.

11. Anon.: Summary of Properties - Du Pont Mylar Polyester Film. Technical Information Bulletin M-1F. E. I. DuPont de Nemours and Co., Inc.

12. Anon.: Aclar Fluorohalocarbon Film. Allied Chemical, Technical Data. Allied Chemical, General Chemical Division.

13. Anon.: Modern Plastics Encyclopedia (1968). Vol. 45/No. 1-A. McGraw-Hill Book Co., Inc., September, 1968.

14. Anon.: Summary of Properties. Du Pont Tedlar PVF Film, Technical Information Bulletin TD-1D. E. I. DuPont de Nemours and Co., Inc.

15. Anon.: Technical Information - Tedlar - Laminating Adhesives. Bulletin TD-10. E. I. DuPont de Nemours and Co., Inc.

16. Anon.: Technical Information - Teflon FEP - Heat Sealing. Bulletin T-14A. E. I. DuPont de Nemours and Co., Inc.

17. Anon.: A New and Different Kind of Fluorocarbon Film. . . DuPont Teflon FEP Fluorocarbon Film. E. I. DuPont de Nemours Co., Inc., November, 1967.

18. Anon.: Technical Information - Tedlar - Heat Sealing. Bulletin TD-14. E. I. DuPont de Nemours and Co., Inc.

19. Anon.: Technical Information - Teflon FEP - Heat Bonding. Bulletin T-13B. E. I. DuPont de Nemours and Co., Inc.

20. Anon.: Saran Wrap. Technical Information Brochure. The Dow Chemical Company.

21. Anon.: DuPont Adhesives for Mylar and Other Polyester Films. Bulletin No. 17. E. I. DuPont de Nemours and Co., Inc.

22. Anon.: Technical Data Sheet - Bondmaster K218-34. Pittsburgh Plate Glass Company - Adhesive Products Division. 13 September 1967.

23. Anon.: Technical Data Sheet - Bondmaster P431. Pittsburgh Plate Glass Company - Adhesive Products Division. 2 March 1967.

24. Goldman, E. J.; Rosenberg, R. A.; Lee, W. E.: How to Use Fluorocarbon Plastics as Bonding Agents. Adhesives Age, February, 1967.

25. Anon.: Shell Epon Adhesive 934. Shell Chemical Company, Adhesives Department.

26. Anon.: Shell Epon Adhesive 9261 Primer. Shell Chemical Company, Adhesives Department.

27. Anon.: DuPont Teflon and FEP Non-Stick Finishes Products and Application Techniques. Bulletin No. 1; Twelfth Edition. E. I. DuPont de Nemours and Co., Inc.