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DEVELOPMENT OF LOW TEMPERATURE DIELECTRIC COATINGS FOR ELECTRICAL CONDUCTORS

Thirteenth Quarterly Report

BY

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October 15, 1964

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DEVELOPMENT OF LOW TEMPERATURE DIELECTRIC COATINGS FOR ELECTRICAL CONDUCTORS

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DEVELOPMENT OF LOW TEMPERATURE COATINGS for ELECTRICAL CONDUCTORS

INTRODUCTION

This report includes additional information on the dielectric loss of insulating films at low temperatures which was first reported in the 12th quarterly report. As these studies progressed it became apparent that moisture exposure influenced the loss characteristics of H-film - even at very low temperatures. Consequently, studies of the dielectric loss of ice by itself have been made.

Dielectric loss measurements made earlier in the program on the cryogenic liquids were found to be in error. These measurements have been repeated using the best techniques now available.

In this report an investigation of the factors most likely to influence the performance of connectors and terminations at cryogenic temperatures is described. The preliminary work on the removal of ML and H-film insulation, as well as the initial work on developmental models of cryogenic cable connectors, are described.

SUMMARY AND CONCLUSIONS

Dielectric Loss at Cryogenic Temperatures

The dielectric measurements at cryogenic temperatures continue to produce fundamental as well as very useful information on dielectric characteristics. It is startling to discover that the dielectric loss of ice at liquid helium temperatures is too low to be measured and yet "wet" H-film has about twice the loss of dry H-film at the same temperature. It is obvious that water attached to some polymer molecular structures may have a considerable degradative effect. In contrast, ice, by itself, at very low temperatures, possesses lower dielectric loss than any other solid material yet measured! However, ice close to its melting point has high dielectric losses which are influenced by dissolved ionic contaminant. These losses may be important in connection with the performance of connectors when frost is deposited on them.

The dielectric losses of the cryogenic liquids, too, are so low that they can just be detected at frequencies between 2 and 10 KC. It seems likely that bridge errors influence the results as a function of frequency and it is probable that the actual dielectric loss is independent of frequency, and that the actual value is lower than the value measured at 2 KC. Despite the lack of accuracy, it is likely that the precision of the measurements is sufficient to permit valid comparisons between the dielectric loss of the different cryogenic liquids and to study the effect of temperature.

It seems reasonable, therefore, to accept a value of dissipation factor for liquid hydrogen lower than .0000001 at the boiling point (a Q greater than ten million). The dissipation factor of liquid helium at the boiling point appears to be about 10 times that of liquid hydrogen. It is interesting to consider the mechanism of dielectric loss in cryogenic liquids. For this reason, measurements of dissipation factor were made on the liquids at temperatures below the normal boiling point by decreasing the pressure over the boiling liqud. (Care was taken to avoid build up of cryogenic "ice" between the plates of the measuring cell.) The tan δ of both liquid nitrogen and hydrogen decreased significantly by a factor of about two between the normal boiling temperature and the temperature at the triple point. The dissipation factor of liquid helium decreased also down to the λ temperature, 2.19^{o}K. Then a sudden decrease in loss occurred followed by an increase as the temperature was decreased still further.

The temperature dependence in the dissipation factor of liquid helium calls to mind the studies of induced ion conductivity made by Carreri at the University of Rome and reviewed in the 9th quarterly report of this series. He found that ionic conductivity in liquid helium increased as the temperature was decreased below the λ point. It is interesting to note also that Professor Blaisse of the Technical Hochschule in Delft, Holland has found a discontinuity in the electric strength of liquid helium above and below the λ temperature. (These results will be reported at a symposium in London on October 16th, which the author will attend.) Dr. A.H. Sharbaugh, Jr., of the General Electric Research Laboratory has calculated, in an unpublished report, that cosmic radiation could not induce enough ions to explain the dielectric losses observed in this program on cryogenic liquids. However, no indication of an increase in loss, with a decrease in frequency, has been observed experimentally which would be characteristics of ionic conduction. While it may be tempting to ascribe the dielectric loss to ionic conductivity, it must be concluded on balance that the loss mechanism in dielectric liquids is not yet understood.

From the practical point of view, the studies described in the foregoing, indicate that electrical devices with selected dielectrics might be operated at cryogenic temperatures with extremely low dielectric losses (i.e., Q's of 10 million or even higher).

Cryogenic Connectors - the Problems

Problems likely to be encountered with cryogenic connectors for wire and cable can be summarized as follows:

1. Cracking of plastic parts in thermal cycling.
   a. Due to shrinkage mismatch between metal inserts and plastic.
   b. Due to different shrinkage with uneven cooling.
   c. Due to freezing of entrapped moisture.
2. Surface electrical failure as frost melts with warming.
   a. On exposed, unprotected leakage surfaces.
   b. At interfaces between plastic parts or metal and plastic.
   c. Where frost can "bridge" live parts.

For the purposes of the subject program, any marked, or sudden decrease in DC resistivity between separate conductors or between conductors and the grounded shell of a connector during or after moisture condensation is considered to constitute failure. It is recognized that a small decrease in resistance for a very short time might be tolerated in service. However, such a decrease in test is evidence of weakness which might in service be worsened by repeated temperature cycles, by contamination, or by other environmental factors.

Surface Leakage Paths

The experimental results indicate that the DC resistance (and presumably other electrical properties) is always markedly decreased when a coherent film of water forms over an insulation surface as frost melts. In fact, in thick frost layers, the surface melting alone may produce a coherent water path without even melting down to the insulation surface. Several factors influence the likelihood of forming a coherent water path.

a. The length of the path.
b. The amount of available water - the thickness of the forst and the rate of melting.
c. The nature of the insulation surface and the contact angle of water with it.

Even with Teflon surfaces, continuous, coherent water paths can form over short distances as frost melts. It is concluded that surface leakage paths between electrical parts in connectors must be eliminated unless long creepage distances can be utilized. Obviously, long creepage paths are not consistent with the desire for small and lightweight connectors.

Interfacial Leakage Paths

It may be possible to eliminate surface leakage paths by utilizing plastic parts and insulated wire with insulating surfaces in contact. However, water may penetrate or "wick" between such surfaces to produce an electrical leakage path. Experimental results indicate that several factors are involved.

a. The degree of conformity between the "mating" surfaces.
b. The pressure between the surfaces.
c. The tendency for change in internal air pressure with temperature change to drive water into the interface.
d. The nature of the insulation surface, the contact angle of water with it and the presence of oil or grease.

It seems possible to produce satisfactory, leakage free interfaces between silicone rubber faces or between a silicone rubber and a rigid plastic, if a slight pressure can be maintained between the two surfaces throughout the temperature cycle. A very small amount of silicone oil and possible silicone grease on the surfaces in pressure contact appears to reduce the pressure needed between the surfaces to prevent the entrance of moisture. The quantitative aspects of this situation have not been determined and may perhaps be best developed by empirical approaches in each particular design.

Mechanical Failure

A number of trials have indicated that it will be difficult to find or develop a potting compound which will have the necessary resiliency and sufficient match in thermal contraction to prevent cracking when used to encapsulate connectors for cryogenic applications. Hence, at least for the present, the encapsulation approach is not being followed.

The likelihood that plastic parts with interfaces under slight pressure might fail at cryogenic temperatures has been investigated. So far, failure has not occurred even from thermal shock by direct immersion in liquid nitrogen. On the other hand, tight metal inserts in plastic parts can cause shattering at low temperatures. The tendency for such shattering is decreased by using very small, round metal inserts and the optimum material. In example, a room temperature vulcanizing silicone rubber (GE RTV-11) can incorporate even relative large, sharp edged, metal inserts without shattering upon immersion in liquid nitrogen. Further work is needed tooptimize the selection of materials and to determine the design limitations.

Connector Design and Evaluation

A multiple conductor MS connector has been evaluated in which the principal faces between the male and female portions are held under pressure when mechanically locked together. This connector has been further modified so that the insulated conductors entering the "back" faces of the connector are held in contact with a slight pressure. In test, so far, this connector gives promise of achieving the desired goal of high leakage resistance between conductors when exposed to thermal shock in liquid nitrogen followed by melting frost and even after water immersion. Electrical "shorts" and mechanical cracking occurred, however, when the wires entering the back faces of the connector were not held under slight pressure.

In the MS connector the interfacial leakage path between pins is very short. A design is being developed by E. J. McGowan in which such leakage paths can be considerably increased without unduly increasing the size of the connector. Prototypes are under construction.

It has been recognized from the start that the design experience and model making capabilities of a connector manufacturer could be of great value in the subject program. In consequence, representatives from five connector manufacturers were interviewed to determine their interest and capabilities in connection with the subject program. Modular Electronics of Osseo, Minnesota has agreed to furnish at cost models of special connectors for this program.

Mr. W. Gammel, President of Modular Electronics, has visited the Advanced Technology Laboratories in Schenectady. Just as soon as possible, sketches of proposed cryogenic connectors will be submitted to him. His suggestions and proposals will also be sought.

Insulation Removal

The development of cryogenic ML coated round wire and ribbon cable with FEP laminated H-film has led directly to the problem of removing these insulations where connections and terminations must be made. The toughness, heat and solvent resistance of these insulations makes their removal difficult. "Brute force" techniques of mechanical scraping and delamination can be utilized with test specimens, but such approaches are not suitable for either manufacturing or field use.

Particularly for fine round wires, a chemical means of removing ML enamel is needed. To date, only a eutectic of sodium and potassium hydroxide at 200C and long exposure to concentrated sulfuric acid at room temperature have been successful in removing ML enamel. These approaches are not considered to be suitable.

The situation with H-film, flat ribbon cable is more promising. The laminations at the cut end of the flat cable can be loosened rapidly by heating with a gas-oxygen flame. The cable can then be stripped and the FEP remaining on the conductors easily removed with the flame.

VISIT TO NASA, HUNTSVILLE

A visit was made to Marshall Space Sciences Laboratory, Huntsville, Alabama (E.C. McKannon) on September 16 and 17. A technical review of the subject program was presented. The proposed technical program was discussed with the technical officer. One thousand feet each of 19 strand and 7 strand shielded cable (items 1 and 3 as described in the 12th Quarterly Report) and the required amount of unshielded and shielded flat H-film cable (items 1 and 6 as described in the 12th Quarterly Report) were delivered during the visit.

PROGRAM FOR THE MONTH OF OCTOBER AND THE FOURTEENTH QUARTER

During the month of October and for the next quarter the following activities will be underway:

1. Construction of additional connector models.

2. Submission of proposed model designs to Modular Electronics for consideration, quote and procurement.

3. Cryogenic shock, condensed moisture and immersion test on connector models.

4. Study of methods for removal of ML enamel from round wire and more suitable techniques for field stripping of flat, ribbon cable.

OBJECTIVES AND SUMMARY OF TEST RESULTS

Dielectric Loss at Low Temperatures - Ice, H-film and FEP

In this program it has been suspected repeatedly that moisture absorbed in plastic films or occluded ice have influenced the dielectric loss measurements. It was decided that dielectric loss measurements on ice itself, as a function of temperature, were essential to the understanding of the performance of materials like H-film and ML enamel. Moreover, it was recognized that the dielectric properties of ice will be important in respect to the performance of cryogenic connectors upon which frost may collect.

A literature search did not at first uncover any information on the dielectric properties of ice at very low temperatures, so an experimental program was started.((Additional literature research did reveal some useful information as listed in Appendix A.)

Two experimental approaches have been taken. In the first, distilled water was slowly frozen in a solid nickel "Berberich" cell (see ASTM D-150) and measured over the range of 50 to 10,000 cps with the GR bridge. Such measurements provided a basis for determining the dielectric constant without appreciable error for ice close to the freezing temperature. However, as the temperature of the ice was decreased to very low values, it was considered likely that errors might be introduced if the ice tended to crack and perhaps pull away from the walls of the cell. In consequence, a measuring "cell" was made by using concentric cylinders of .001 inch stainless steel foil spaced about .010 apart. This "cell" was insulated with Teflon and supported in a coffee can which was connected to guard potential. Water was added slowly to the can and frozen from the bottom up. In this way cracking between the foil electrodes was largely avoided and no voids developed even at liquid helium temperature. The dielectric constant, of course, could not be determined directly but was calculated with reasonable accuracy by making comparisons with results close to $0^{o}C$ using the

Berberich cell. The foil electrodes provided a relative high capacitance which made the measurement of low losses easier. On the other hand, the relatively high resistivity of the stainless steel foil may have contributed to errors in the measurement of very low losses at the higher frequencies.

Dissipation factor values for ice at different temperatures are given in Figures 1 and 2. From these results it is apparent that ice has a dissipation factor peak due to its polar character which occurs at a frequency above 10 KC at $0^{o}C$, at about 2 KC at -30C, at about 300 cps at -49C and below 50 cps at -100C. As the temperature is decreased still further, the characteristic slope of the dissipation factor is maintained over the whole frequency range at -157C and is still apparent at -196C. At -196 and -223C the results in the dashed portion may be subject to series measurement error, although it is possible, but unlikely, that another dissipation factor peak at a frequency above 10 KC is indicated. At liquid helium temperature (269C) the losses of ice were too low to be measured.

In Figure 2 it is apparent that no abrupt change in dielectric characteristics occur at the melting point as might have been suspected. The increase in tan δ at the lower frequencies for ice near the melting point is undoubtedly due to ionic conductance. In this range the loss will be affected by the amount of dissolved ionic contaminant.

The dielectric constant of ice versus frequency is plotted for the different test temperatures in Figure 3. These results are consistent with the dissipation factor values. The results, however, have not been corrected for changes in dimension due to shrinkage at low temperatues, but errors from this source would be relatively small. An apparent error, probably introduced by high conductivity at -3C, is plotted as a dashed line in Figure 3.

The dielectric characteristics of ice have been plotted as a function of temperature in Figures 4 and 5. This method of plotting gives a better engineering idea of the very low values of loss at low temperatures. However, the fundamental characteristics may be observed better in the plots as a function of frequency. Once the very low loss characteristics of ice at low temperatures had been established, it was possible to analyze the influence of moisture absorption in such plastic materials as H-film. Specimens of H-film with evaporated gold electrodes were dried for 2 days at 200C. Part of the sample was kept dry in a dissector and the other part was immersed for 2 days in water at 80C - "wet" film. The dielectric values versus temperature are shown in

Figures 6 and 7.

From Figure 6 it is apparent that absorbed moisture has made a considerable effect on tan δ even at very low temperatures. The results at -269C in liquid helium are given in the table below for three frequencies.

Range in Tan δ at -269C

	10^{0} cps	1 KC	10 KC
Dry H-film	.00008 (range ?)	.000086 to .000101	.000141 to .000142
"Wet" H-film	.00022 to .00031	.000300 to .000317	.000357 to .000363

It is apparent that absorbed moisture has contributed a surprisingly large effect even at temperatures as low as -269C. At those temperatures the dielectric loss of ice is too low to be measured. When the specimens exposed to moisture were dried, the dissipation factor was still at least 50% higher than for dry specimens. (Results of limited test are not plotted). It is probably that hydrolytic instability in H-film is indicated.

In addition to the moisture effects, the curves for H-film in Figure 6 indicate that two dielectric absorption peaks occur in H-film below room temperature. Undoubtedly, two molecular mechanisms are indicated. It is probable that these peaks can be used to study the polymeric character of H-film.

Since the moisture effects were so marked with H-film, attempts were made to determine if similar effects could be noted in Teflon. Unfortunately, satisfactory electrodes could not be applied to TFE Teflon, but adequate test specimens were obtained with FEP Teflon. The results are also plotted in Figures 6 and 7. Absolutely no difference could be detected between dry and "wet" FEP as was expected. However, two loss peaks were found below room temperature. Incomplete results indicate that these peaks shift with quenching. A powerful tool for the study of branching and crystallinity in such linear polymers is indicated.

Dielectric Loss in Cryogenic Liquids

In view of the extremely low dielectric loss of many insulating materials at cryogenic temperatures, it is useful to know the loss characteristics of the cryogenic liquids in which they may be immersed. Earlier in this program attempts were made to measure the cryogenic liquids. Unfortunately, it was discovered, after the conclusion of the work, that errors had been introduced by excessive series resistance in the measuring leads. The work to be described was undertaken to overcome these errors so far as possible.

Since the 1000 pf vacuum capacitor used in the original work had been broken, another was obtained and the cell, as originally described, was duplicated. Thus, a fully shielded construction with shielded leads was used which could be evacuated to provide a known standard upon which to base comparative results when the cell was filled with the cryogenic liquid. Careful dielectric measurements indicated that small mechanical changes occurred in the cell under vacuum which could produce an error of perhaps .01% in the capacitance measurement, but should not affect the measurement of tan δ.

After careful flushing and evacuation to about 10^{-6} torr, nitrogen, hydrogen or helium gas was condensed in the cell which was placed in a dewar containing the same cryogenic liquid. The gases had a guaranteed purity of 1 part in a million or better and were run through a charcoal filter and a nitrogen trap before entering the dielectric cell. Thus, every precaution was taken to achieve high purity and to prevent contamination. After the cell reached the liquid temperature, the dissipation factor was measured at atmospheric pressure. Next, the pressure over the cryogenic liquid in the dewar surrounding the dielectric cell was lowered and held at the pressure needed to achieve the desired temperature. With hydrogen and nitrogen, the triple point temperature was chosen. Test results for tan δ are reported in Figures 8 and 9. The values for dielectric constant are given in Table I and Figure 10.

In Figure 8 the change in tan δ with frequency is approximately the change which would be expected from a series resistance error. A series error in electrodes and leads would have been essentially eliminated by the comparison technique used. However, series error could occur in the bridge circuit itself. The values of dielectric constant are so independent of frequency that dielectric absorption must be ruled out as the explanation for the change in tan δ. Even so, a significant difference exists between the values of tan δ for the different cryogenic liquids. This difference was maintained with repeated test. It seems likely that the absolute accuracy of the measurements has no exact meaning, but that the relative values do. It seems reasonable also to believe that the actual value for tan δ in each case is below that measured at 2 KC. (At lower frequencies no measurements were possible).

The results for liquid helium as a function of temperature in Figure 9 are fascinating. At all three frequencies a discontinuity seems to occur at the lambda point (below which helium is super fluid). It is admitted that the accuracy of these measurements is not nearly sufficient to guarantee that the discontinuity really occurs. The change in the slope of the tan δ curves above and below the λ point is interesting also. The results for dielectric constant in Figure 10 are consistent with the tan δ results except perhaps that no discontinuity occurs at the λ point. The changes in dielectric constant for liquid helium are similar to those in density over the same temperature range. A reciprocal temperature scale has been used in Figures 9 and 10, since the change in density is known to give a reasonable set of linear plots with this relationship.

A very thorough literature search revealed no information on the dielectric loss of cryogenic liquids, although values of dielectric constant have been reported, as indicated in Table I. In order to interpret the results with cryogenic liquids, measurements were made on a non-polar hydrocarbon liquid - hexane - as shown in Figure 11. The very limited evidence in the literature indicated that the loss in hexane would be primarily due to ionic conductivity resulting from moisture as an impurity. The results obtained indicate that the loss is, in fact, entirely due to ionic mobility even when the hexane was thoroughly dried with calcium hydride. When a potential of 135 volts* was applied for about 18 hours to sweep ions out of the liquid, the loss was reduced still further, as shown, but was still attributable to remaining ionic conduction.

It is interesting that the dielectric loss of undried hexane at -22C is almost the same as the dried hexane at room temperature and is still completely ionic in character. However, at -87C (just above the freezing temperature) the dielectric loss in hexane is relatively high, but apparently no longer due to ionic conductivity. It is immediately apparent that the loss characteristics of hexane are completely unlike those of the cryogenic liquids, which, by structure, should also be non-polar.

*The dissipation factor measurements were made without removing the DC potential.

Despite the shape of the tan δ curves versus frequency for the cryogenic liquids, it is tempting to propose an ionic conduction mechanism in this case. The source of the ions must be considered. Dr. A.H. Sharbaugh, Jr. of the General Electric Research Laboratory considered the possible effect of cosmic radiation in producing ions in liquid nitrogen. He calculated on this basis a tan δ of about 10^{-9} - much lower than that detected in these results. Hence, the loss mechanism cannot yet be explained.

Cryogenic Connectors

Considerable attention was given to the special problems which would occur in connectors operating under cryogenic conditions. The conclusions from a combination of analysis and experimental work have been given earlier in this report. The discussion here will be limited to outlining the experimental approach. So many individual tests have been made that no attempt will be made to describe them all in detail, but rather the orderly experimental justification for the conclusions reached will be presented.

Mechanical failure of plastic parts with metal inserts, due to differential thermal contraction from room to cryogenic temperatures, was expected and obtained. A rubbery polyurethane, a rigid epoxy without and with several types of filler, including Zerifac, micro-balloons and silica were all unsatisfactory. However, at room temperature vulcanized, silicone rubber (GE RTV-11 with 1% of T-12 accelerator) did withstand repeated thermal shock in liquid nitrogen even with sharp edges brass inserts ¼" x ½" in cross section. Small commercial connectors with small pins molded in filled diallyl phthalate resin were also subjected to thermal shock in liquid nitrogen and helium without mechanical failure. Consequehtly, it was apparent that molded-in metal inserts could be considered for use in cryogenic connectors even though the problem of a final potting or encapsulating compound might remain unsolved.

Considerable attention has been given to the problem of electrical leakage over wet dielectric surfaces. It is inevitable that frost will collect on a connector as it returns to room condition from cryogenic temperatures. As the connector warms still more, the frost will melt to produce a wet surface. Frost and water will also collect along a cable if one end is held at low temperature. In order to achieve a reasonable uniformity for test, the cryogenic - moisture condensing test array, sketched in Figure 12, has been developed and built. In this device liquid nitrogen can be admitted to a 4" hollow brass cube with ¼" thick walls supported on plastic rods. The specimen under test is placed on the top surface of the brass cube. The insulation under test is held in place by clamps. In the case of test of the interface between two insulations (as shown in Figure 12), the whole assembly can be held in contact with a dead weight. After the top surface and specimen have been cooled to a low temperature, room temperature air is blown into the cube at a controlled rate to remove the liquid nitrogen and increase the temperature. To increase the amount of frost and water condensed on the surface, the assembly is mounted in a humidity cabinet at 90% RH and 23°C.

Several varieties of inserted and surface electrodes have been evaluated. For surface measurements, a silver paint, ring electrode space around a central circular electrode is convenient. For measurement at the interface between insulating materials, two brass strip electrodes are set flush in the surface of one of the insulating materials (in rectangular shape). The spacing between the ends of the electrodes and the distance to the edge of the insulation (the overlap) are controlled. DC resistance with 500 volts between electrodes is measured continuously as frost forms and subsequently is melted.

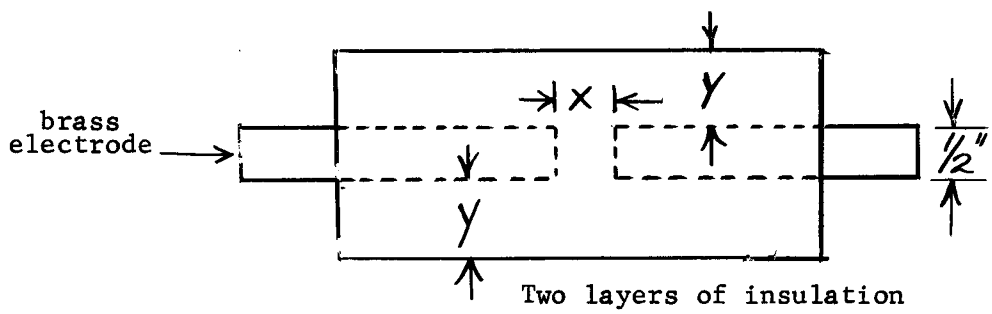
In making the measurements of resistance over surfaces on which frost had melted, it was hoped that an insulation material could be found which would maintain good electrical properties. Hope for such a situation existed, since atmospheric moisture condensing on Teflon usually does not cause a drastic drop in surface resistance. However, with a quarter inch spacing between electrodes, melting frost decreased the resistance to about 10^4 ohms even with the best materials such as Teflon, silicone rubber, and silicone oil or grease coated surfaces. In fact, as a thick layer of frost melts, the resistance of the wet layer at the surface decreases markedly even before the surface of the insulating material involved. Without attempting to obtain exact quantiative results, it became apparent that several factors affected the electrical resistance of melting frost:

1. The thickness and the density of the frost.
2. The humidity and temperature of the surrounding air.
3. The rate of melting (overall heat input).
4. The nature of the insulation surface.
5. The distance between electrodes.

Of these factors, the kind of insulation is perhaps the least important although moisture sensitive surfaces do tend to retain a water film for relatively long periods as contrasted to Teflon. It became obvious that exposed dielectric surfaces (i.e., across the connector back face) could not be tolerated in a connector designed to operate in or close to a cryogenic enivornment.

The remaining approach involves the mating of two insulations in contact so as to prevent the entrance of moisture and thereby maintain good electrical properties across the interface. To some extent this problem is similar to that with cryogenic seals except that effective sealing need not be maintained at cryogenic temperatures, but only at and above $0^{o}C$.

The test setup shown in Figure 12 was used to make a series of quantiative measurements of insulation resistance between electrodes at the interface of two insulation surfaces while a heavy layer of frost melted. The electrode and insulation dimensions are sketched below:



The spacing, X, between the electrodes and the overlap, y, from the edge of the electrodes along the interface between the two sheets of insulation to the outer edge of the specimen were varied. The pressure between the layers of insulation was also varied. Two layers of RTV-11 rubber or a layer of RTV-11 in contact with a layer of filled epoxy resin were tested. In some tests a coating of silicone oil or of silicone grease was applied to the mating surfaces.

In qualitative summary of the test results, the following comments can be made:

1. A rubber to rubber contact is superior to rubber in contact with a rigid material.
2. Silicone oil and silicone grease both decrease the pressure needed to seal the interface.

3. The length of the overlap, y, is the most important factor in the maintenance of good electrical properties across the interface.

With $y = \frac{3}{8}''$ and silicone grease applied to the interface, no failure occurred at the interface between sheets of silicone rubber at 0.2 psi. With $y = \frac{1}{8}''$ and no silicone grease applied to the interface, electrical failure occurred even with a force of 1.5 psi between two sheets of silicone rubber. Failure under these conditions was eliminated when silicone grease was applied to the interface.

The foregoing test series provided some "feel" for the quantitative aspects of the design needed. However, it is recognized that the geometry of a cryogenic connector will also influence the spacings and pressures needed to produce an electrically reliable interface.

Connector Design

Discussions were held with representatives from 5 manufacturers of hook-up wire connectors and the products of each were examined. In addition, catalogs were obtained from several additional manufacturers. An MS connector made by the "The Pyle-National Company" (Cat. No. ZZB-R-1022-55) was particularly interesting because it utilized a rigid and a silicone rubber face which were held in contact under pressure when the connector was mechanically locked together. The connecting wires enter the back face through holes in the silicone rubber. Test under frost conditions showed that leakage could occur at the interface between the wire insulation and the silicone rubber in the back faces. However, the back faces were easily modified to provide compression between the rubber and wire insulation as sketched in Figure 13. The back faces of the modified connector are pictured in Photo 2. For these trials, compression was applied to the back face by wrapping wire around it and twisting it tight. In an actual model a spring or snap on clamp would be used. In the picture four leads are shown leaving the back face, but for most of the tests, the remaining holes were filled with short lengths of dummy wires. However, tests were made between the two wires at the edge, between the two wires in the center and between all wires connected together and the shell. To complete the picture, Photo 3 shows the front faces of the connector before they were clamped together for test. It should be noted that the spacings between the pins in this connector is short.

Nine tests were made with the "Pyle" connector under a variety of conditions which involved thermal shock by immersion in liquid nitrogen followed by the development of frost when mounted on the device shown in Figure 12. As the work progressed, it became apparent that adequate frost build-up could be obtained by simply placing the connector taken from the nitrogen bath in the 90% RH humidity cabinet since the shell had considerable heat capacity. However, even this test sequence did not produce failure in the modified connector. The final test involved alternate immersion in liquid nitrogen and room temperature water followed by overnight soaking in the water!

In one of the tests the clamping pressure was not applied around the back faces and two tiny cracks developed in the silicone rubber between the wires when the connector was taken from the water bath and immersed in liquid nitrogen. It is not certain whether or not trapped water aided the formation of these cracks. At any rate, failure did occur between one set of wires when the connector was again immersed in water.

Finally, the re-dried connector was soaked in silicone oil under slight vacuum for about 6 hours, recycled between water and liquid nitrogen and then immersed in liquid water for 23 hours without developing failure even along the cracks. The test in water is being continued.

While the results with the modified "Pyle" connector are very promising, it is recognized that advantages might accrue if the interfacial leakage path between the pins at the mating surfaces could be increased. E. J. McGowan has made several suggestions in this connection. One version is sketched in Figure 14.and a picture of a fabricated model is shown in Photo 4. This model was made to illustrate the concept and is not intended for test. In the functional model, silicone rubber will be used and the wires connecting to the end plate will incorporate the ideas developed in the modified Pyle connector.

Table I

Dielectric Constant

Cryogenic Liquids

Liquid	Temp. ^{o}K	Measured Value	Literature Values*	Measured % ΔC 100 cps to 10 KC
Helium	4.21	1.0469^{5} $^{+.00001}_{-.00002}$	1.048[1]	$2 - 3 \times 10^{-4}$
Helium	2.23^{5}	1.0563 $^{+.00008}_{-.00007}$	1.055^{6}	1×10^{-4}
Helium	2.19	$1.0563^{5} \pm$?	1.055^{5}	1×10^{-4}
Helium	2.15	1.0565 $^{+.00008}_{-.00008}$	1.055^{4}	1×10^{-4}
Helium	1.83^{4}	1.0562^{5} $^{+.00009}_{-.00007}$	1.055^{2}	0.5×10^{-4}
Nitrogen	77.3	$1.431^{1} \pm$?	1.433[2]	$< 1 \times 10^{-4}$
Nitrogen	63.1	$1.467 \pm$?	1.475	2×10^{-4}
Hydrogen	20	1.231^{4-5}	1.227[3]	2×10^{-4}
Hydrogen	14	$1.259^{4} \pm .0025$	1.248	?

*Extrapolated or estimated from published values.

1. W.M. Keesom, "Helium", ElSevier, Princeton, N.J., 1942.

2. Robert Guillien, J. Physique (8) 1, 29 (1940)

3. A. Van Itterbeck and J. Spoeden, Physica (9), 339 (1942)

Values for dielectric constant are given also in Nat. Bur. of Stds., Circ. 514

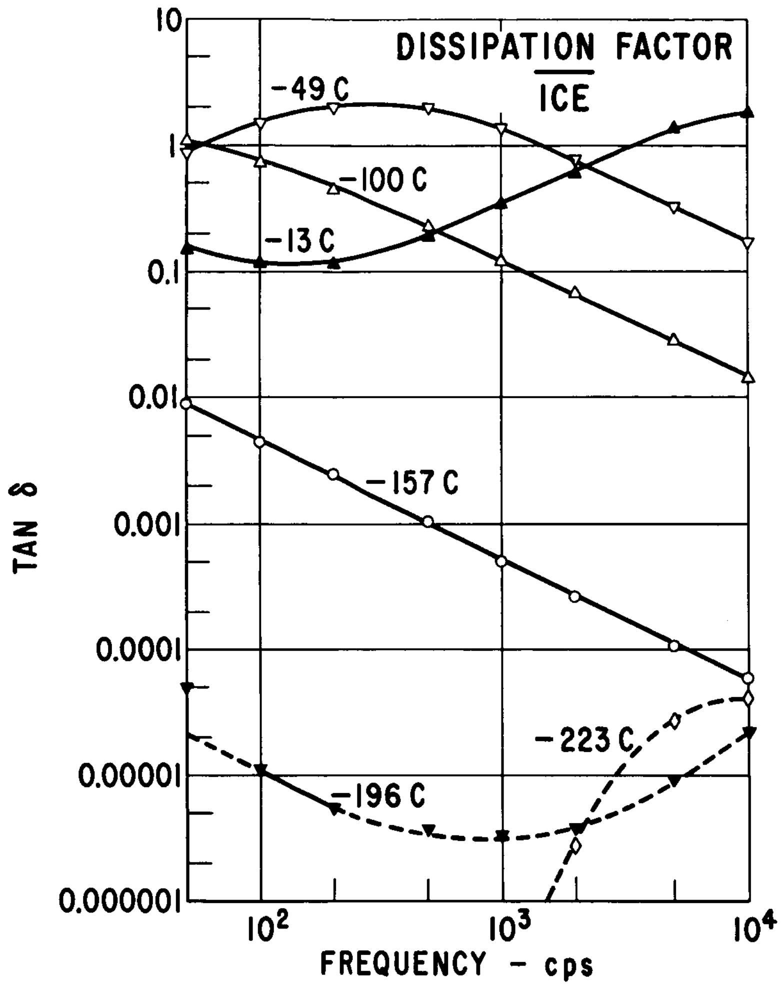


Figure 1 - Dissipation Factor of Ice versus Frequency at -13 to -223C

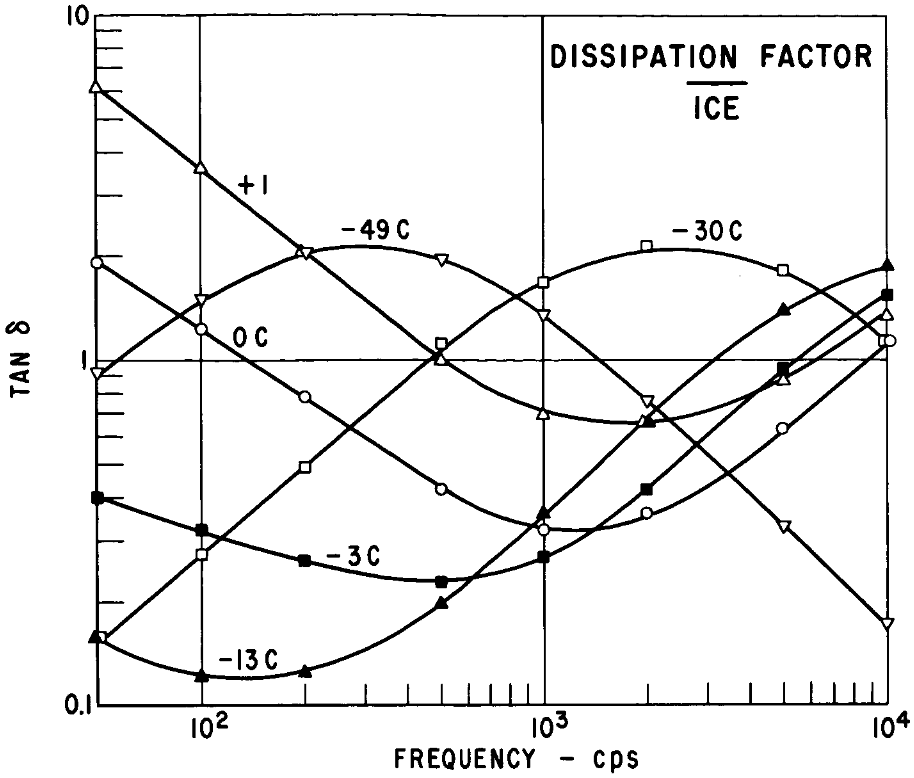


Figure 2 - Dissipation Factor of Ice versus Frequency at 0 to -49C and for Water at 1C

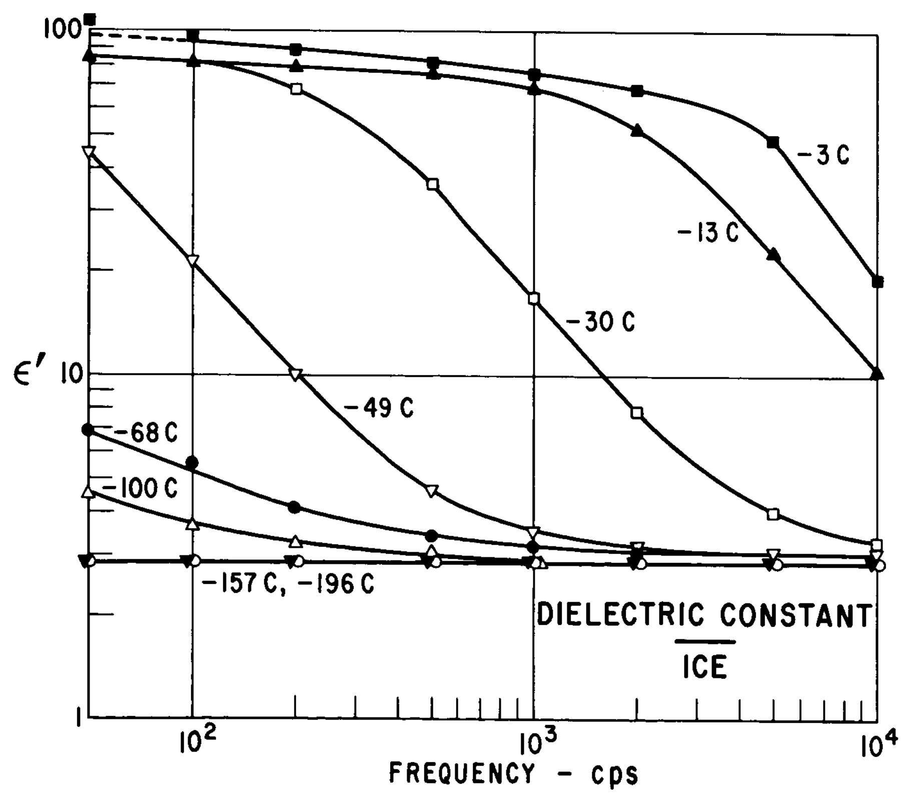


Figure 3 - Dielectric Constant of Ice versus Frequency at -3 to -196C

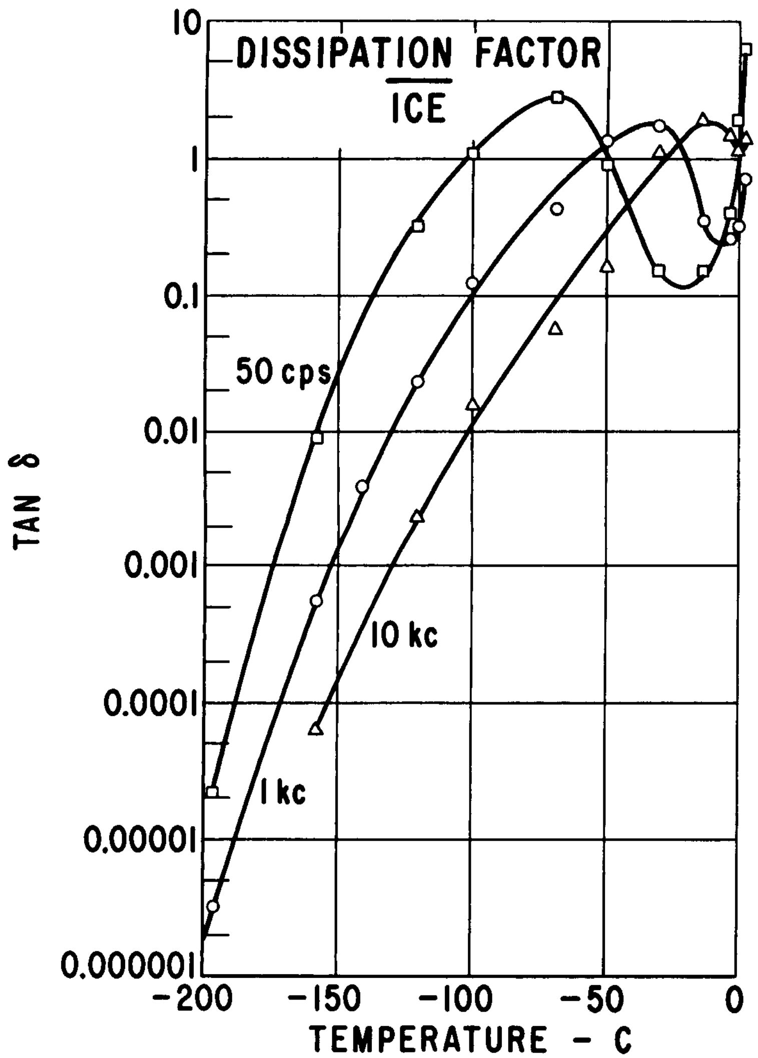


Figure 4 - Dissipation Factor of Ice versus Temperature at 50 cps, 1 KC and 10 KC

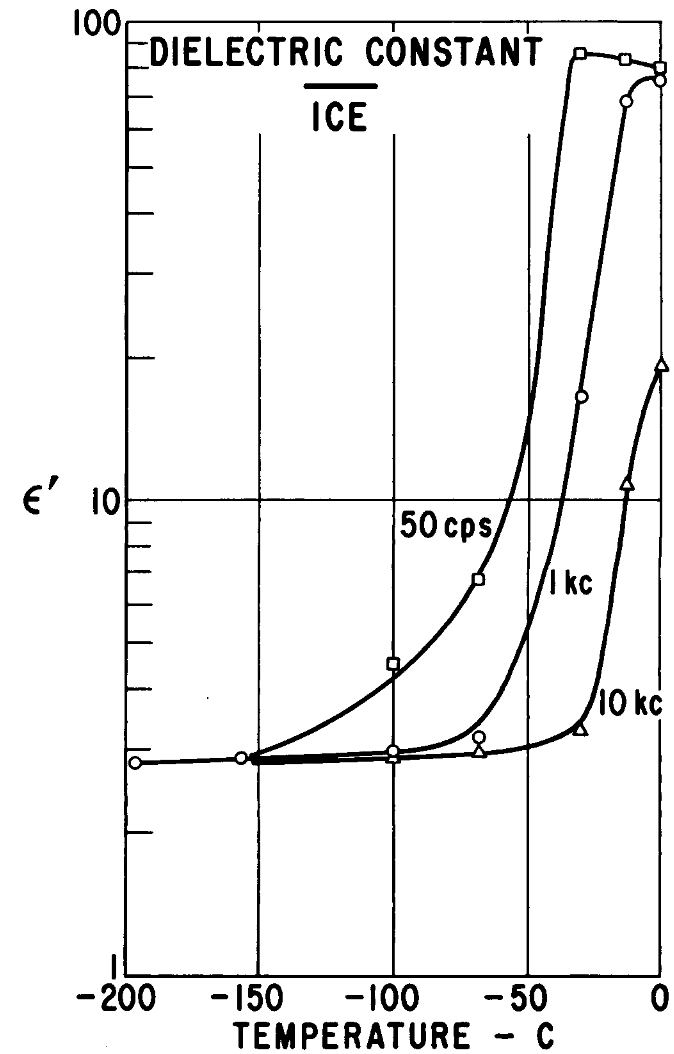


Figure 5 - Dielectric Constant of Ice versus Temperature at 50 cps, 1 KC and 10 KC

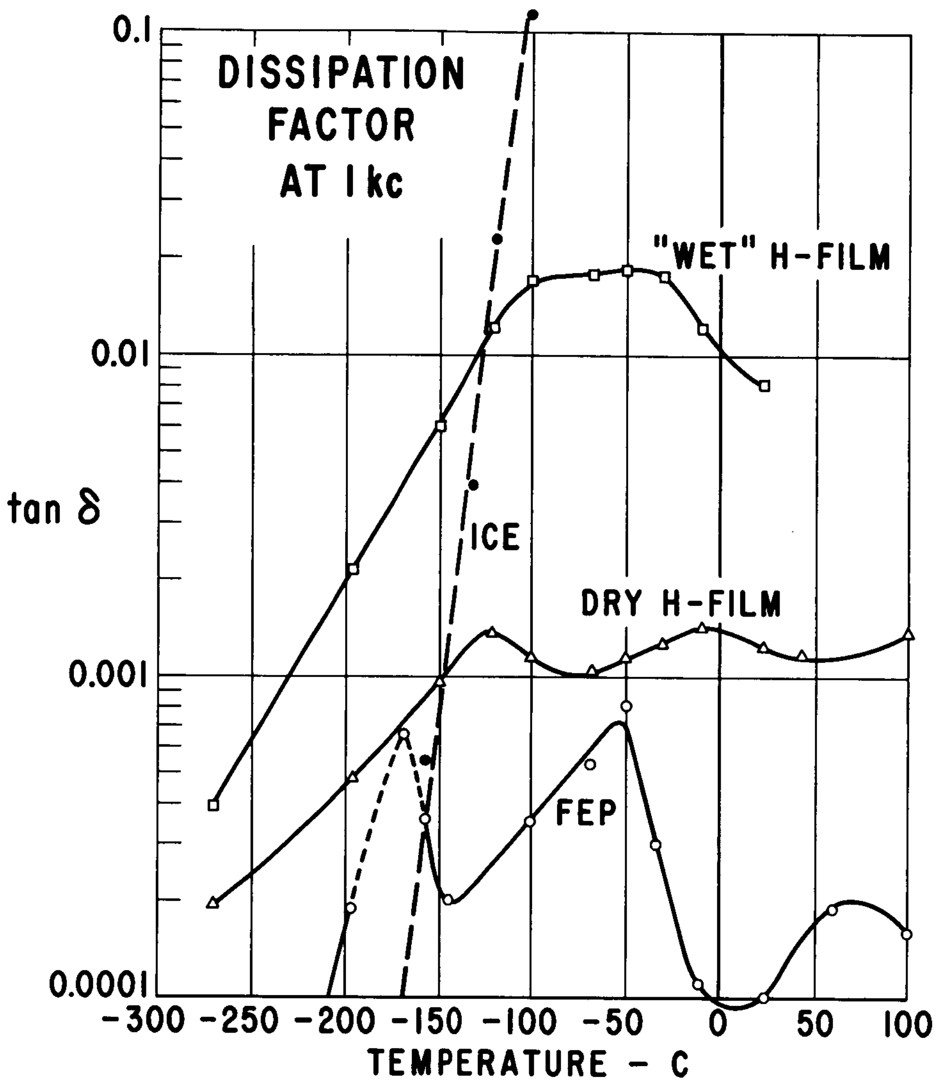


Figure 6 - Dissipation Factor versus Temperature
Dry H-film (heated 2 days at 200C)
"Wet" H-film (as above plus 2 days in water at 80C)
FEP Teflon - both "wet" and dry as above (no difference)

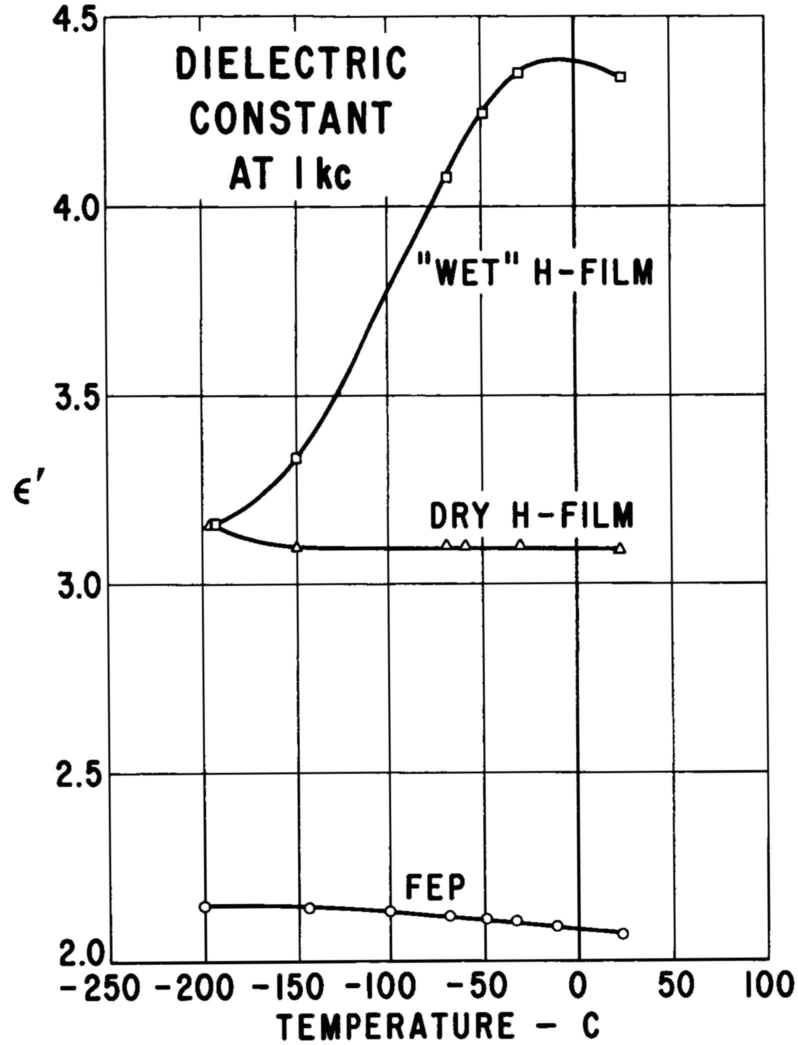


Figure 7 - Dielectric Constant versus Temperature
Dry H-film (heated 2 days at 200C)
"Wet" H-film (as above plus 2 days in water at 80C)
FEP Teflon - both "wet" and dry as above (no difference)

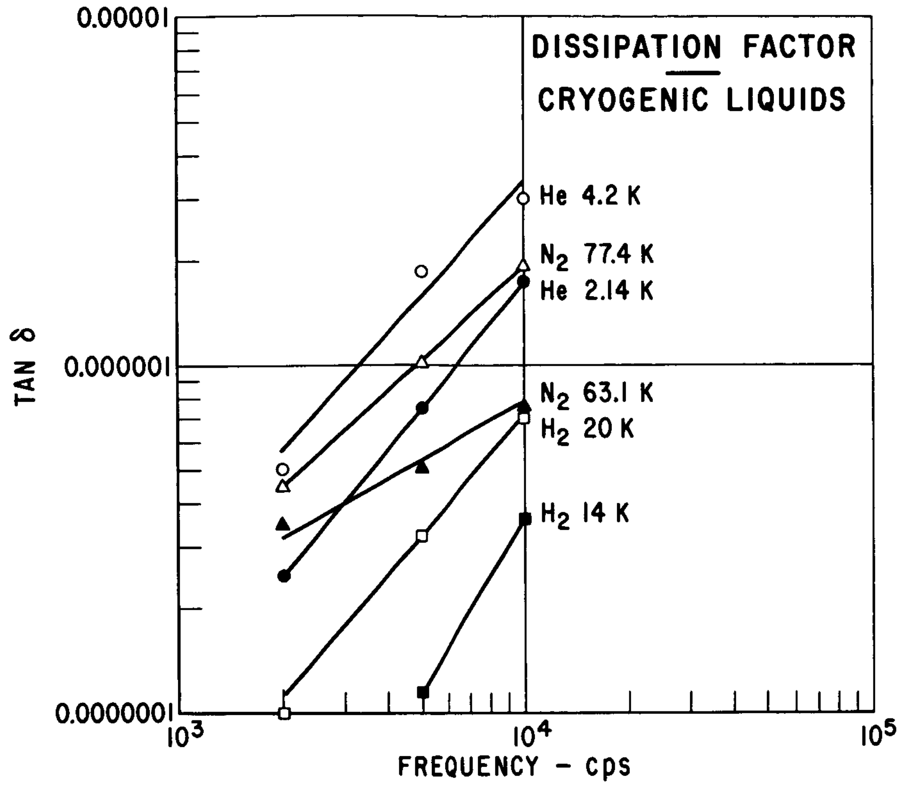


Figure 8 - Measured Values of Dissipation Factor for Cryogenic Liquids Plotted as a Function of Frequency, (see text)

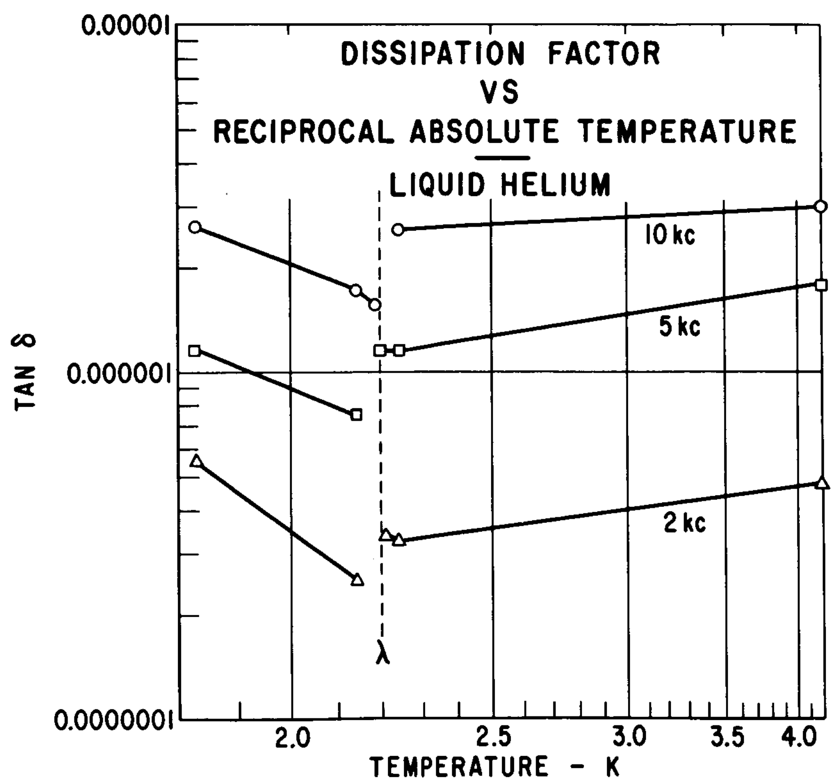


Figure 9 - Dissipation Factor of Liquid Helium versus Reciprocal of the Absolute Temperatures (plotted at 3 frequencies - see text)

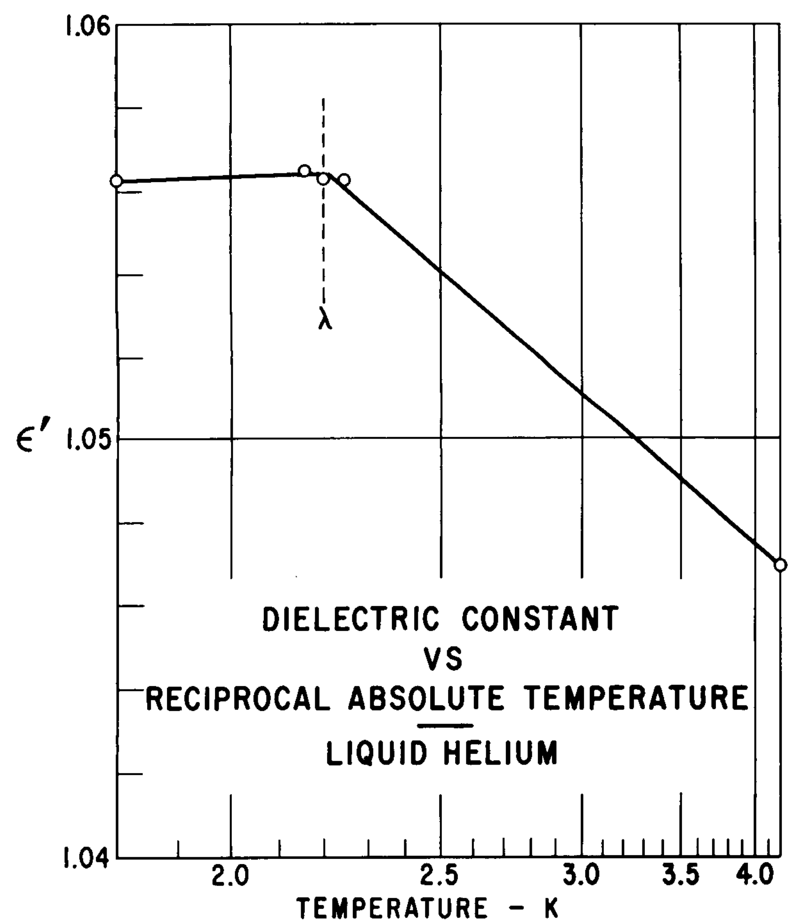


Figure 10 - Dielectric Constant of Liquid Helium versus Reciprocal of Absolute Temperature

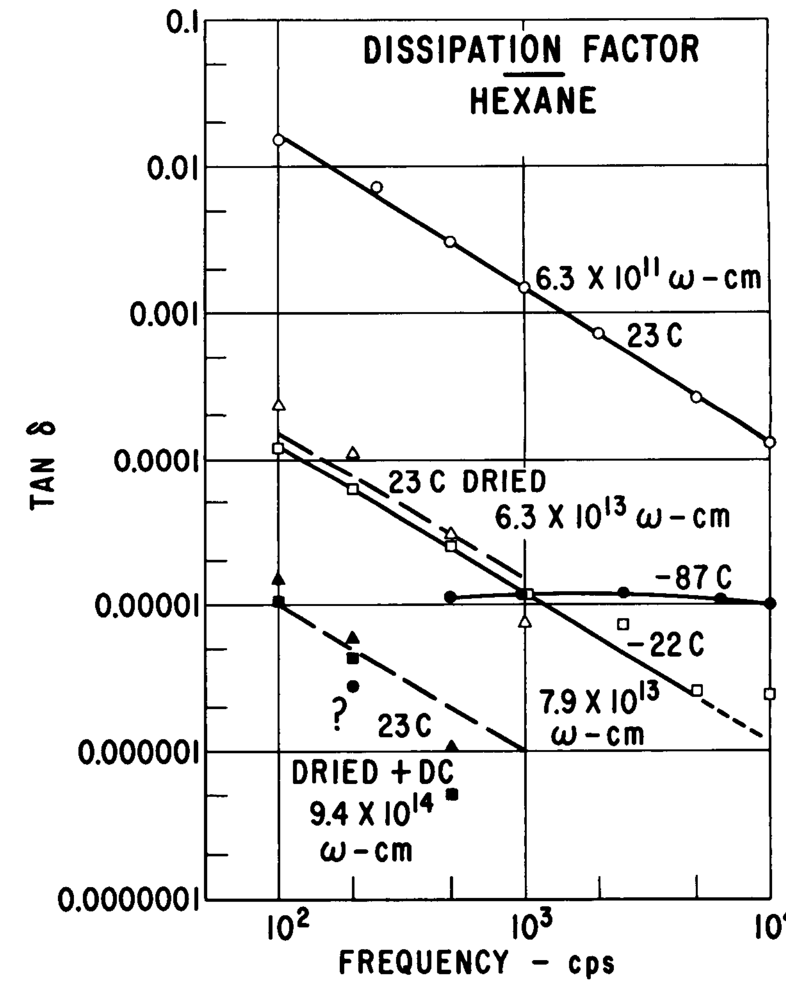


Figure 11 - Dissipation Factor of Liquid Hexane versus Temperature as a Function of Drying and Temperature

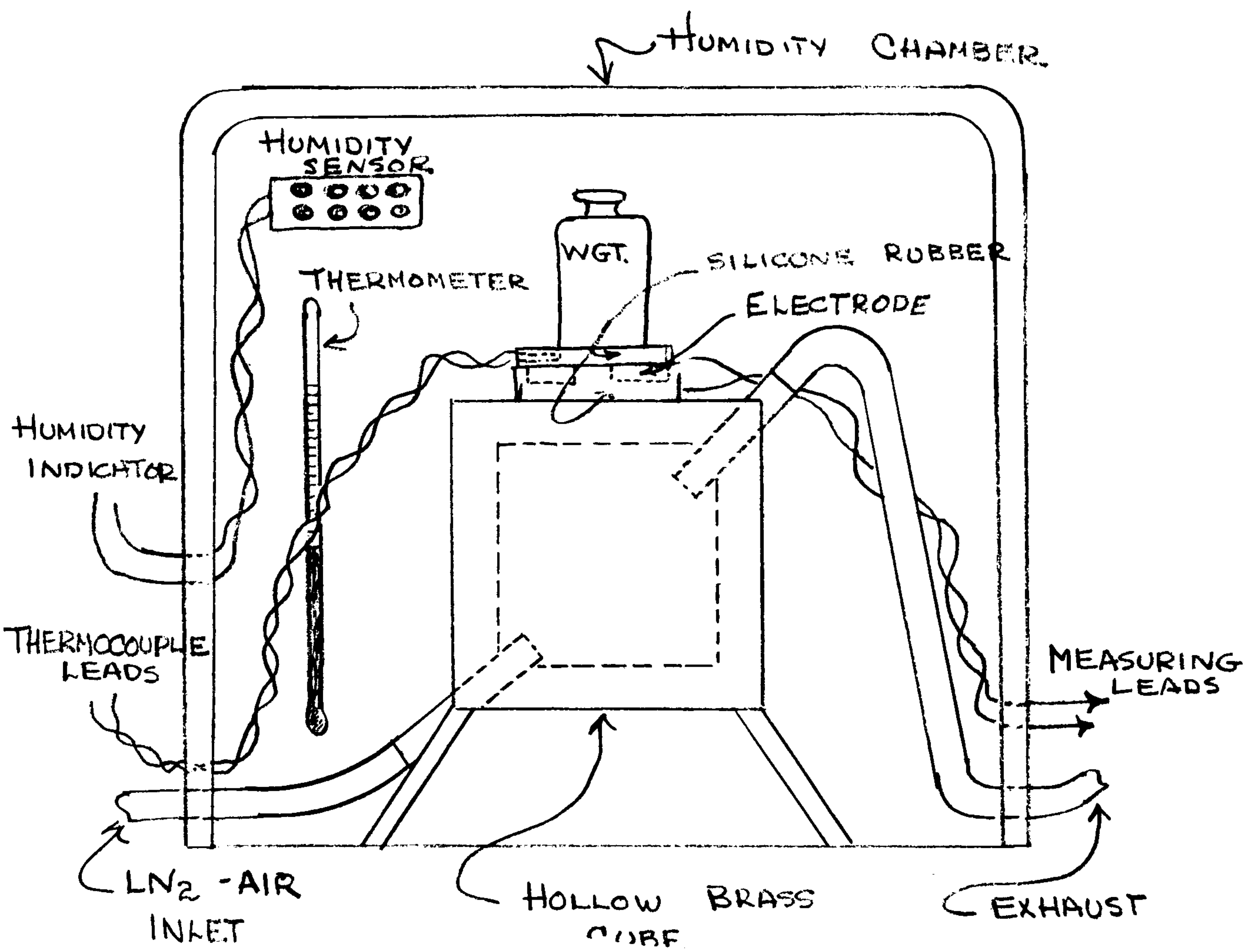


Figure 12 - Test Setup for Producing and Melting Frost on Insulation Surfaces and Interfaces

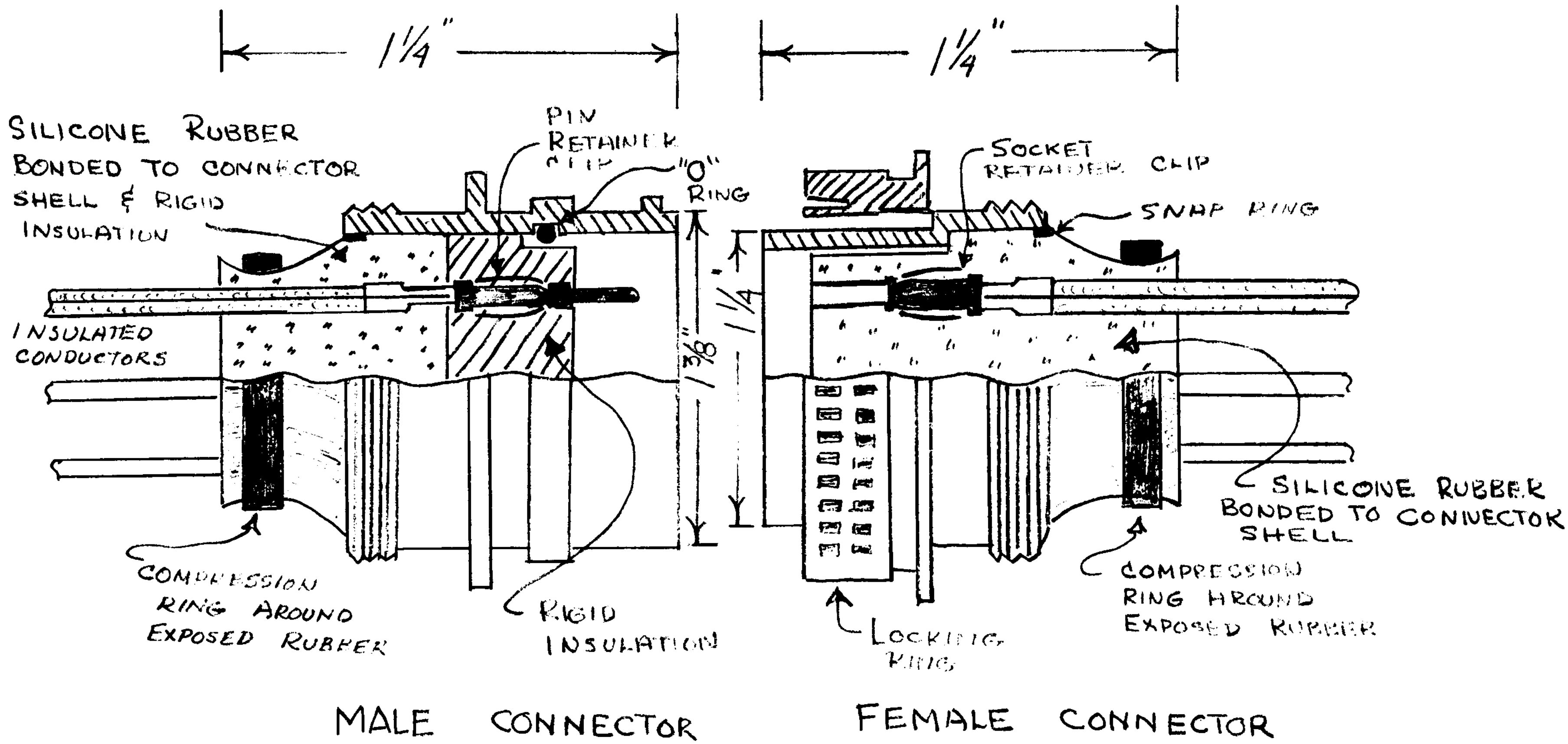


Figure 13 - Cross-section of "Pyle" MS Connector Modified to Hold Wire Insulation Surfaces in Contact (see text)

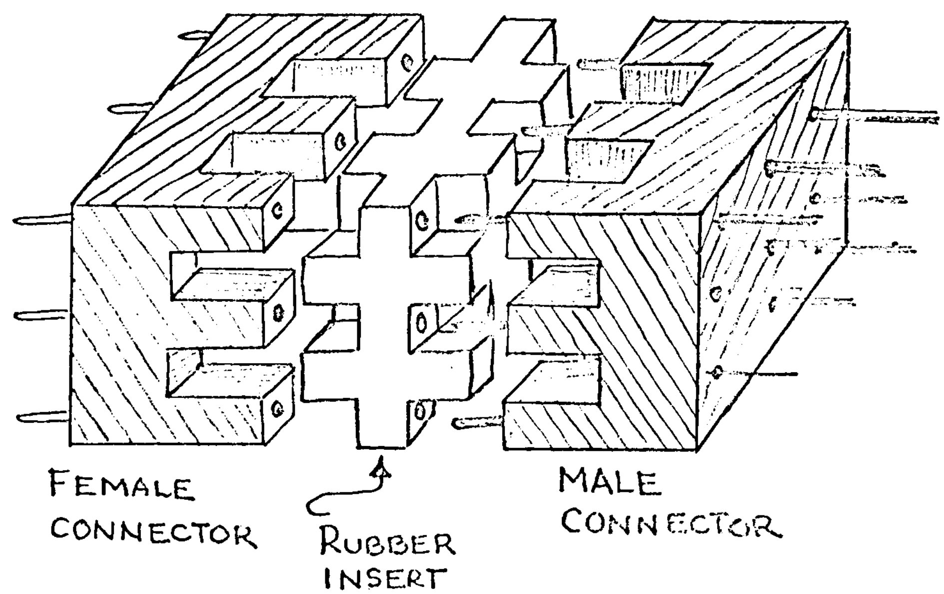


Figure 14 - Model of Connector Designed to Increase Leakage Paths along Insulation Interfaces (not a functional model)

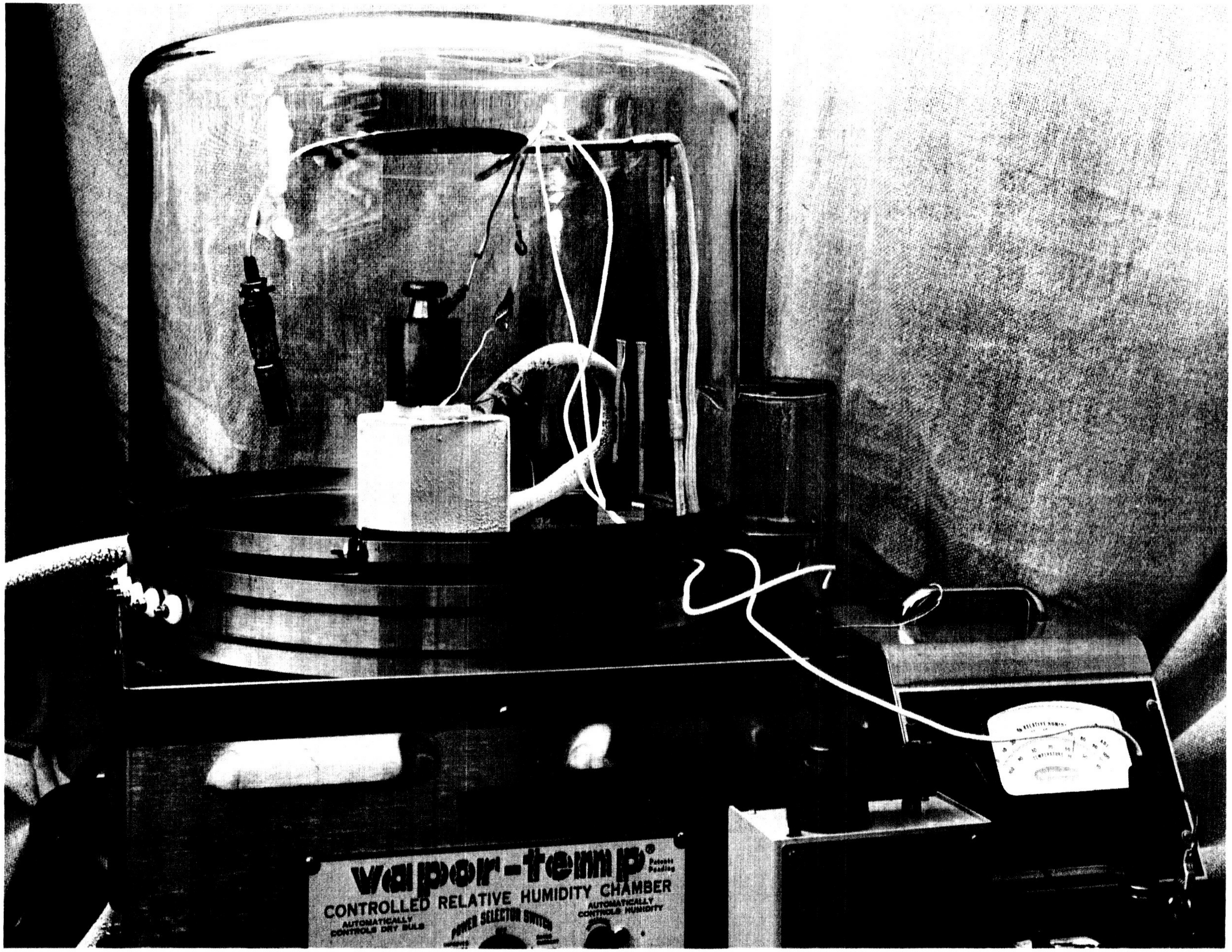


Photo 1 - Test Setup for Producing and Melting Frost on Insulation Surfaces and Interfaces
ATL Photo No. 830701

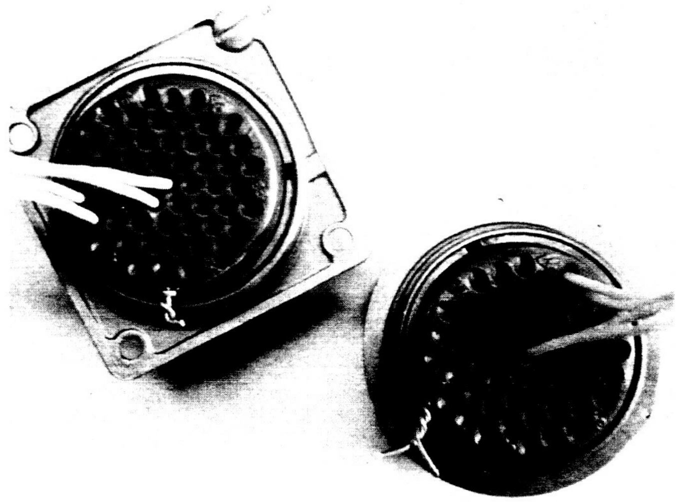


Photo 2 - Back Faces of "Pyle" MS Connector Modified so as to Produce Pressure on Insulation of Entering Wires (dummy wires not involved in test have been removed)

Photo 3 - Front Faces of "Pyle" MS Connector Which Lock Together Under Pressure

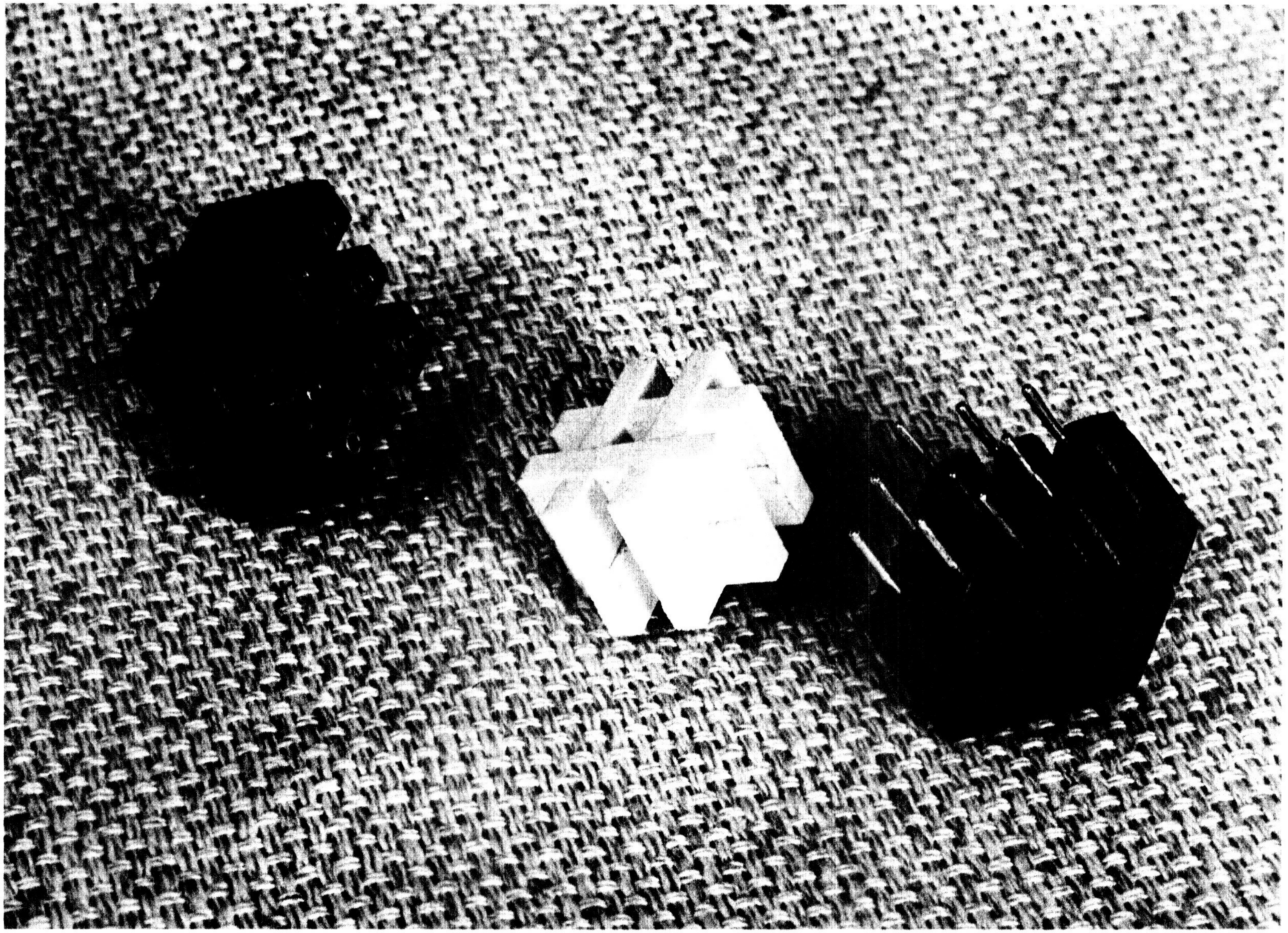


Photo 4 - Model of Connector Designed to Increase Leakage Paths Along Insulation Interfaces (not a functional model) ATL Photo No. 830702

Appendix A

Selected Literature References on the Dielectric Properties of Ice

"The Temperature Dependence of the Relaxation Time of Polarization in Ice", E. J. Murphy, Trans. of ElecChem. Soc. Vol. LXV (1934) pp 133 to 142.

Measurements were made between 20 cps and 100 KC and with ballistic measurements to periods of 270 sec. at temperatures down to -139C. The results are consistent in theory with the subject work with some quantitative differences.

"Properties of Ordinary Water Substance" (book) Dorsey (1940), Dielectric Properties of Ice, pp 495-510.

A collection of data from many sources which includes conductivity data and some information on voltage breakdown.

"The Concentration of Molecules on Internal Surfaces in Ice", E.J. Murphy, J. of Chem. Phys. Vol. 19, No. 12 (December 1951) pp 1516-1518.

A theoretical paper on "local" conductivity in ice.

"Dielectric Losses of Ice", F.K. Eder, Funk und Ton, No. 1, pp 21-29. (In German).

Measurements were made between 100 cps and 10 mc at temperatures down to -50C. Results are in substantially better quantitative agreement with the subject work than are Murphy's (see above). At -5C and 14 cm wavelength values of $\varepsilon' = 2$ and $\tan \delta = 0.1$ were obtained.