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A NEW STEADY STATE CALORIMETER FOR MEASURING HEAT TRANSFER THROUGH CRYOGENIC INSULATION

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ABSTRACT

A guarded cylindrical calorimeter, using evaporation techniques for heat flux measurement, is described. The apparatus can be used to measure the thermal conductivity of the same, undisturbed, sample over wide temperature limits to an accuracy of better than 3 percent. The conductivity of one sample of SiO_2 plus aluminum powder is reported.

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One of the most important problems in the continuing effort to understand the heat transfer mechanism in cryogenic insulation and to develop more effective insulating materials is the accurate determination of the effective thermal conductivity of insulating materials. Although a number of calorimeters[1, 2, 3, 4, 5] have been designed and built to date almost all of them suffer from certain limitations which restrict their accuracy and limit the temperature range over which they can yield satisfactory data. Among the best known efforts in the construction and use of calorimeters are the flat plate calorimeters described in Refs. 1 to 4, and the cylindrical calorimeter described in Ref. 5. The flat plate calorimeters can potentially yield good accuracy with a number of materials but they require extensive guards to shield against extraneous heat transfer, are difficult to operate, expensive to build[4], require a long time before reaching steady state and may necessitate the use of expensive electronic feed-back equipment to satisfy the demands of high accuracy[2]. The available cylindrical calorimeters are simple to operate, cheaper to build, but can only operate over limited temperature ranges and are subject to errors due to conduction along the fill and vent piping, and uncertainty as to whether or not they actually achieve steady state.

This paper describes the construction and operation of a novel, highly accurate, but at the same time relatively inexpensive type of calorimeter suitable for measuring cryogenic insulation materials under steady state conditions over a wide range of temperatures. The criteria established for the design included spanning the entire cryogenic range down to 4°K, allowing both large and small temperature differentials, and measurement of conductivity with high precision. The resulting apparatus has the following characteristics.

1. The calorimeter will measure heat transfer in the steady state through a sample while maintaining one wall at temperatures of 76°K, 194°K, or any desired temperature between 273 and 373°K while the other wall can be maintained at 4°K, 20°K, or 76°K.
2. Measurements with various temperature differences can be made on a given sample without the need to remove, change, or alter the sample in any way. This makes possible the determination of the temperature variation of an average effective thermal conductivity over wide ranges of temperature.
3. The calorimeter will maintain steady state conditions for a sufficient length of time to enable good thermal equilibrium. It reduces to a minimum any error due to conduction and thermal radiation along the fill and vent tube of the measuring vessel and due to condensation of gas leaving the measuring vessel on the wall of the guard assembly.
4. The apparatus was built to maintain high vacuum and allows measurement of the interstitial gas pressure of the cryogenic insulation within the sample space.

5. It is accurate in maintaining the annular insulation space geometrically and will permit changing insulation samples with ease.

A schematic diagram of this calorimeter and its supporting apparatus is shown in Fig. 1 and a photograph of the calorimeter is shown in Fig. 2. The rate of heat transfer through an insulation sample is determined by measuring the mass rate of flow of saturated vapor leaving the inner measuring vessel as described in Refs. 5 and 6. Temperatures of the inner and outer wall surface are determined in the usual manner from the pressure measurements and the thermodynamic properties of the liquid gas used to fill the inner and outer containers respectively.

The measuring vessel is guarded on one end and hemispherically capped on the other end, leaving a 7/8" annular insulation sample space. The inner vessel has an o. d. of 8" and the outer vessel has i. d. of 9-3/4". The guard and measuring vessels are 8" and 28. 3" in depth respectively. The guard vessel is isolated effectively from the fill and vent tube of the measuring vessel by means of a stand-off. Micarta bumpers are placed on the guard vessel to maintain the dimensional accuracy of the annular insulation sample space.

The effective area and geometric configuration for the heat transfer through the insulation correspond to two hollow concentric cylinders attached to hemispherical ends. The advantage of this type of configuration is that isotherms are smooth and continuous throughout the insulation sample and thus aid in the interpretation of data where conduction and radiation modes of heat transfer are sought independently.

Any calorimeter designed for cryogenic work is subject to thermal contraction of one or both of the insulation boundary surfaces. For the calorimeter at hand the geometric modification due to thermal expansion and contraction was calculated using data of Corruccini and

Gniewek[7]. Calculations show an error as high as 4.6 percent can be induced in the results unless the expansion and contraction phenomena are taken into account in the evaluation of the data[8].

The design criteria for this calorimeter demanded that not more than 10 percent of the cryogenic fluid evaporate from the measuring vessel during the time required for steady state. Maximum evaporation will occur when liquid hydrogen is used in the inner measuring vessel and the outer wall is maintained near room temperature. Table 1 shows the results of calculations indicating the time required to evaporate liquid hydrogen under these conditions for various calorimeter geometries and insulating materials. Using this table as a guide, standard available materials were utilized to keep the cost of constructing the calorimeter to a minimum. Table 2 gives the time required to evaporate 10 percent of the liquid from the measuring vessel of the calorimeter at steady state for various heat transfer rates. It shows that for most cryogenic insulating materials which have thermal conductivities of the order of 5μ w/cm°K the calorimeter will be able to operate in the steady state with nitrogen and hydrogen and even with helium for a sufficiently long time to permit accurate measurements.

One possible source of error in a calorimeter of this design occurs when the inner and outer cylinders are not axially aligned. Fig. 3 shows the percentage error of the measured thermal conductivity due to the cylinders being axially displaced (see Ref. 9 for the theoretical background and Ref. 8 for actual calculations). In order to reduce errors due to non-concentric alignment an alignment pin was fastened to the bottom of the measuring vessel through a special alignment port in the bottom of the outer hemispherical cap during the time the calorimeter was being filled. After the sample had been placed in the calorimeter the alignment pin was removed and the port was closed with a highly

compressed O-ring[10]. This seal remained high vacuum tight when cycled many times from 76°K to 393°K. During actual tests concentricity of the sample space was maintained by means of three conically shaped micarta bumpers which were glued to the guard vessel. With this arrangement the axial displacement did not exceed 1/16" so that the maximum possible error due to that factor was less than one-quarter of one percent.

Test Results

In order to test the performance of this calorimeter the apparent mean thermal conductivity of a powder insulation sample composed of 50% Cabosil, and 50% aluminum powder by weight was determined with various wall temperatures. The aluminum powder used for these tests was 5 micron diameter spheres. It should be noted that 50%, by weight, of spherical aluminum powder does not yield as low an apparent mean thermal conductivity as reported earlier for flat platlets[11]. In addition these results agree well within the limits of accuracy with results obtained on an earlier calorimeter[5]. The results of these tests are summarized in Table 3 and the apparent mean thermal conductivity is plotted as a function of the mean insulation boundary surface temperature in Fig. 4. Fig. 5 shows a typical heat flux vs. time plot which gives an indication of the manner in which the calorimeter approached equilibrium after refilling. (In this instance the liquid used for filling was slightly subcooled.) As shown in detail in Ref. 8 the total error incurred in any measurement of apparent mean thermal conductivity will not exceed three percent. In passing it should be noted that the heat anomaly discussed by Cline and Kropschot[12] is still present although less apparent than in the original reference.

In summary this paper describes the construction, method of operation, and accuracy of a new and very accurate calorimeter suitable for measuring apparent thermal conductivities of cryogenic insulating materials over wide ranges of temperature. The major disadvantage of this device is the limited temperature ranges available using conventional cryogenic fluids. With the availability of this instrument however it is hoped that in the future accurate measurements of a variety of cryogenic insulating materials can be made and that a satisfactory understanding of the complete heat transfer mechanism through such materials can be obtained. In fact it may be possible to make relatively accurate measurements on certain multiple layer samples if they can be adequately contoured to the hemispherical ends of the calorimeter.

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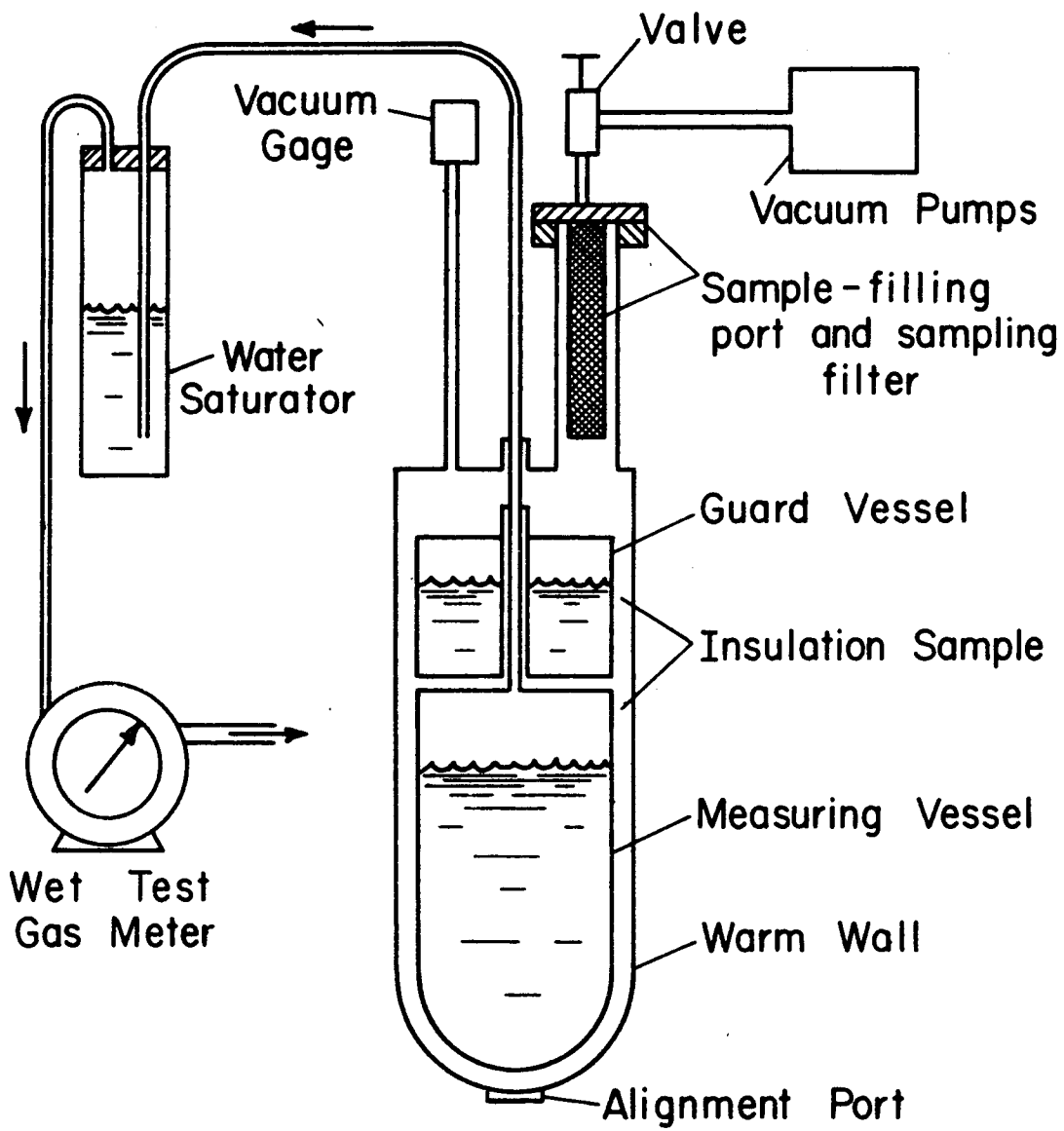

Fig. 1 Schematic diagram of the calorimeter and associated apparatus.

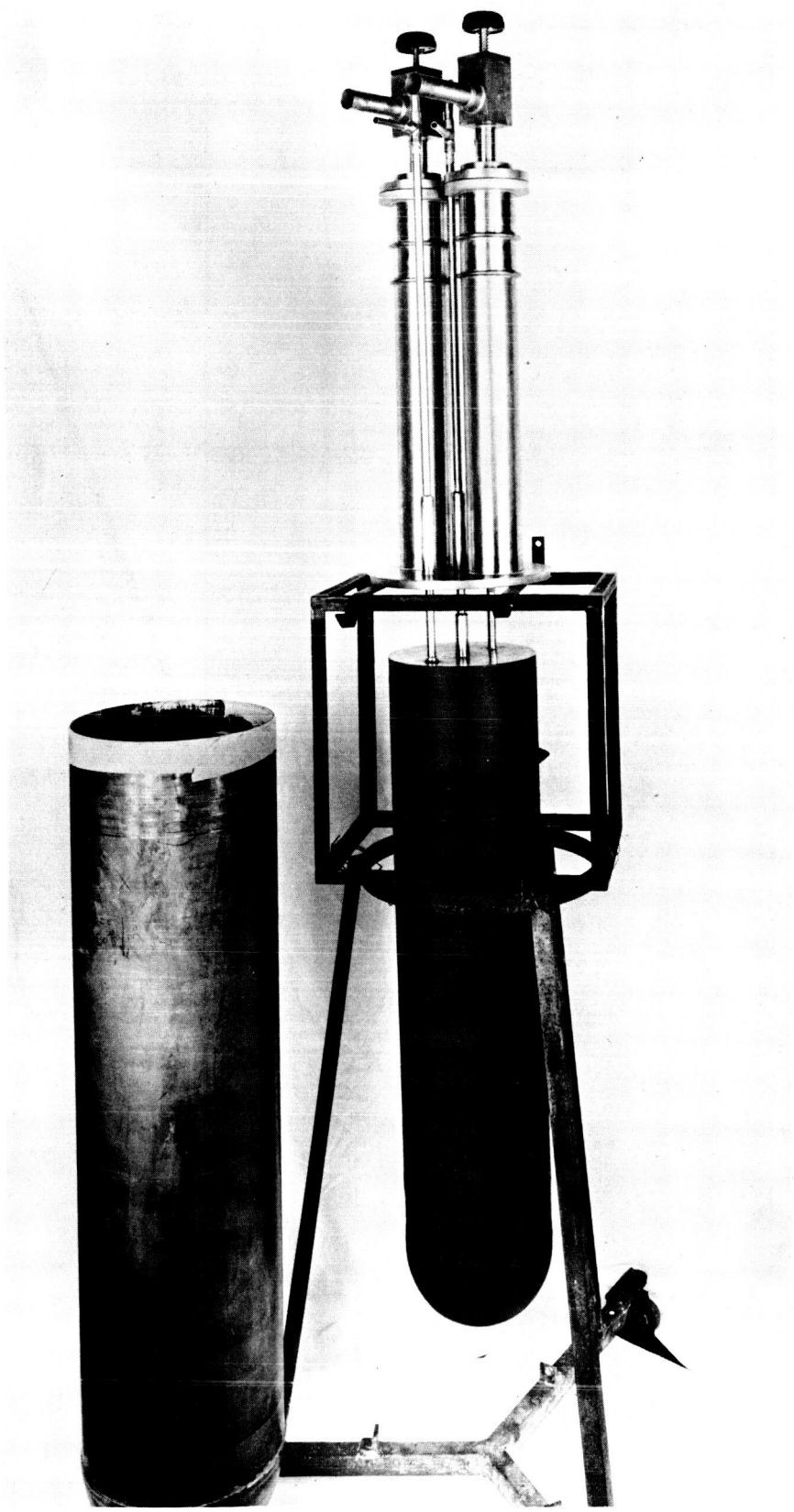

FIGURE 2. Photograph showing the disassembled calorimeter.

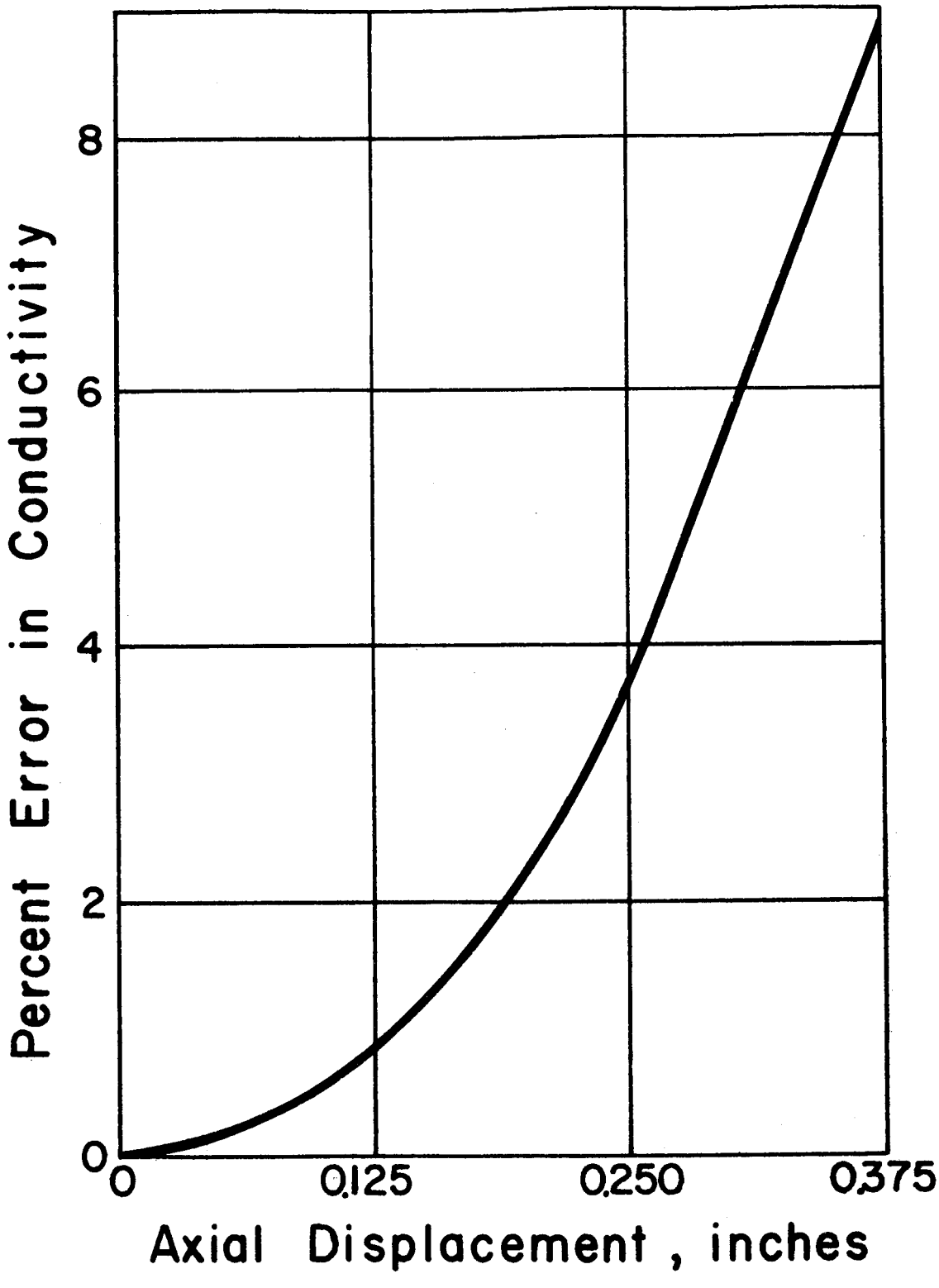

Fig. 3 Error in thermal conductivity due to axial displacement of the two co-axial cylinders. r_i is 4" and r_o is 4-7/8".

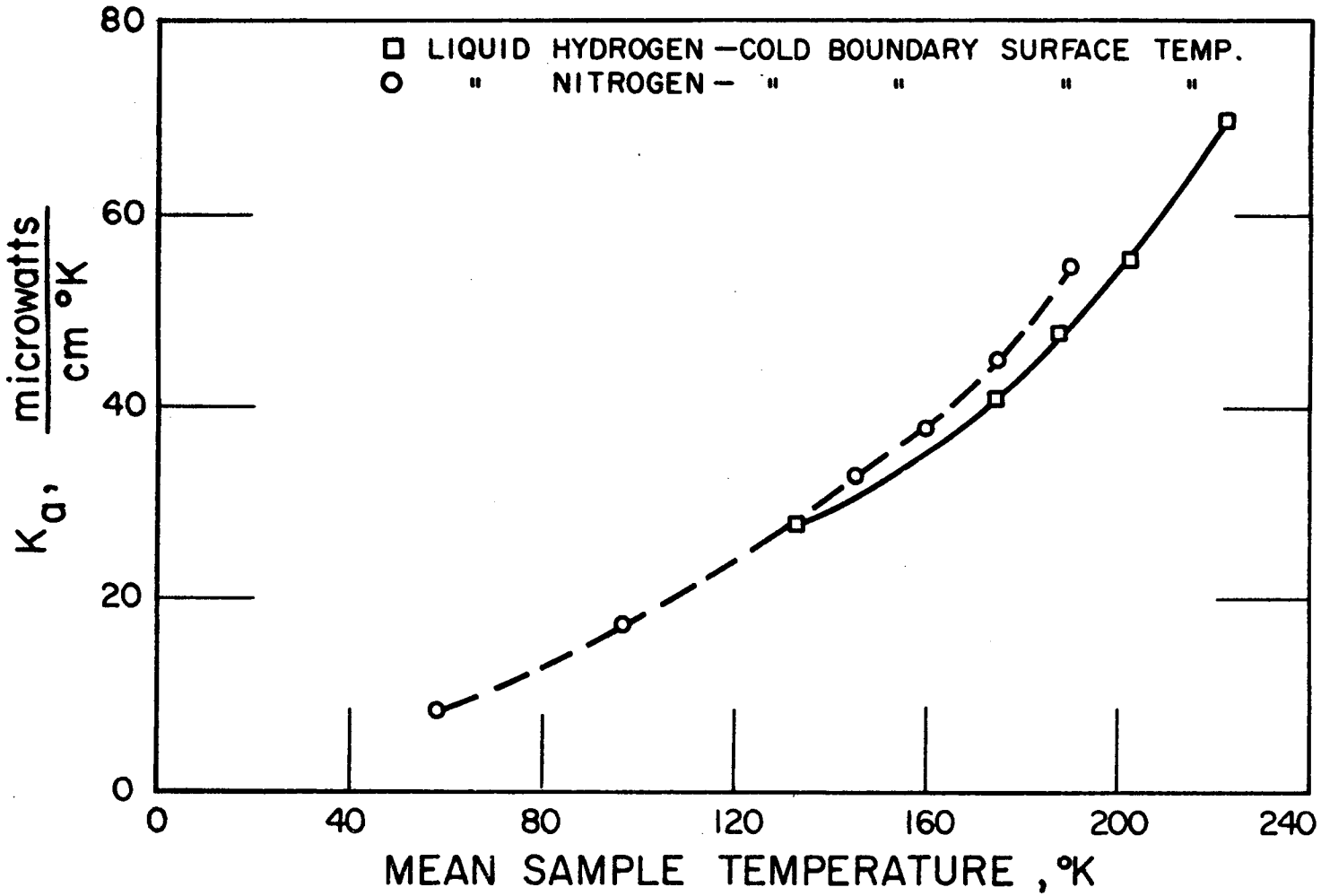

Fig. 4 Apparent mean thermal conductivity vs mean temperature for evacuated Cabosil + 50% Al powder.

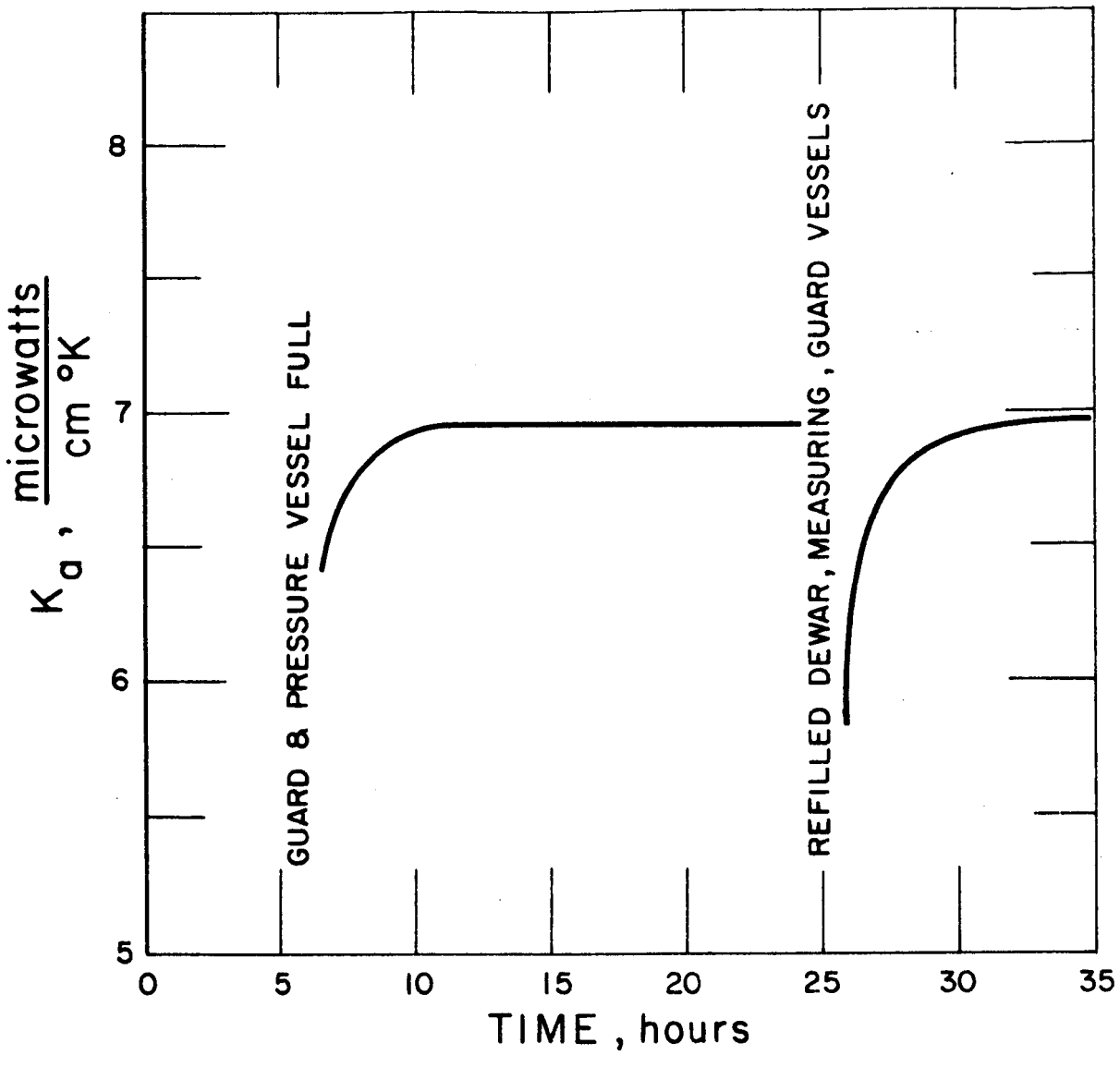

Fig. 5 Typical conductivity - time curve after refilling apparatus with liquid. Boundary temperatures 370 and 76°K.

TABLE 1

TIME REQUIRED TO EVAPORATE 10 PERCENT OF THE LIQUID HYDROGEN FROM THE CALORIMETER MEASURING VESSEL AND FOR TOTAL EVAPORATION FROM THE GUARD VESSEL (TEMPERATURE GRADIENT 300 → 20°K)

r_o	3.5"		4.0"		5.0"		Key
r_i	2.5"		3.0"		4.0"		
K_a $\frac{\mu \text{ watts}}{\text{cm°K}}$	A hrs.	B hrs.	A hrs.	B hrs.	A hrs.	B hrs.	A -- time for 10 percent evaporation from measuring vessel
8.0	2.52	23.4	3.13	28.9	4.34	39.1	
5.0	4.03	37.4	5.01	46.2	6.85	62.5	
2.0	10.1	94.0	12.5	115	17.1	156	B -- time for complete evaporation from guard vessel

TABLE 2

TIME REQUIRED TO EVAPORATE 10 PERCENT OF THE LIQUID FROM THE CALORIMETER MEASURING VESSEL AT STEADY STATE (THEORETICAL)

	$(T_h - T_c)$ °Kelvin			
	300 - 76	300 - 20	76 - 20	76 - 4
K_a $\frac{\text{microwatts}}{\text{cm°K}}$	Nitrogen	Hydrogen	Hydrogen	Helium
	hours	hours	hours	hours
0.5	-	-	299.0	21.5
2.0	96.5	14.9	74.8	5.37
5.0	38.6	6.0	29.9	2.15
8.0	24.1	3.7	18.7	1.34
15.0	12.8	2.0	10.0	.72

TABLE 3

APPARENT MEAN THERMAL CONDUCTIVITY AND THE TOTAL MEASURED HEAT TRANSPORT VERSUS THE TEMPERATURE GRADIENT FOR THIS CALORIMETER USING A POWDER INSULATION SAMPLE COMPOSED BY WEIGHT OF 50 PERCENT CABOSIL AND 50 PERCENT ALUMINUM POWDER (5 MICRON DIAMETER SPHERES)

Temperature Gradient °K	Apparent Mean Thermal Conductivity K_a (μ watts/cm°K)	Total Heat Flux Q_T (watts)
370 - 76	6.95	4.58
361 - 20	5.425	4.13
330 - 76	5.55	3.145
330 - 20	4.48	3.10
300 - 76	4.75	2.38
300 - 20	3.75	2.35
273 - 76	4.025	1.775
273 - 20	3.25	1.84
194 - 76	2.75	0.76
194 - 20	1.725	0.704
76 - 20	0.825	0.108

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