

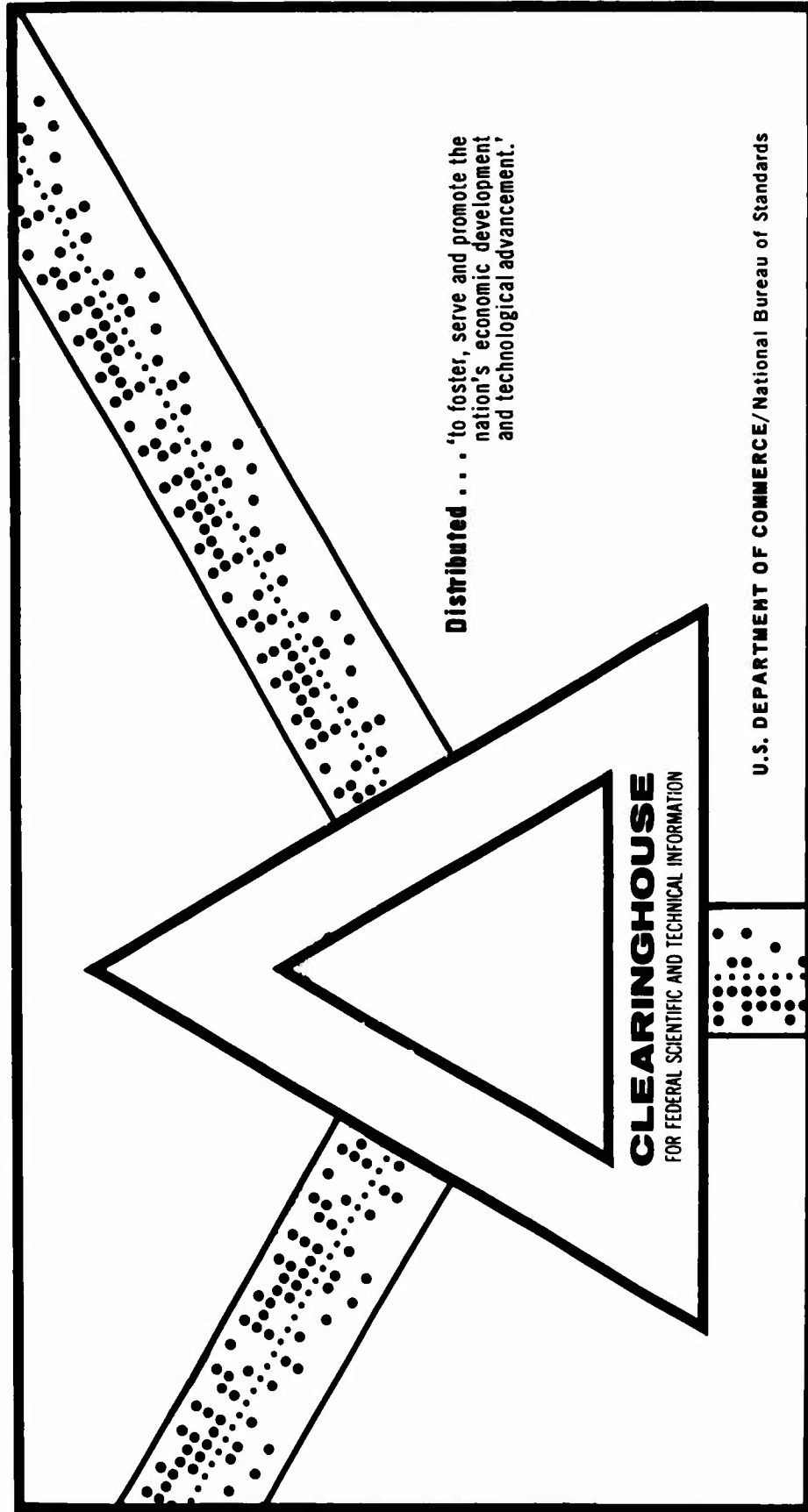
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MEASURING 14 PROPERTIES WITH THE MULTI-PURPOSE INSTRUMENT AND
A TEMPERATURE FREE METHOD OF MEASURING THERMAL TRANSPORT
PROPERTIES

Wolfgang Leidenfrost

Purdue University
Lafayette, Indiana

August 1969



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Air Force Contract
AF 49(638)-1574

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ERRATA

Page	Line	
2	end of Abstract	add: Whenever possible comparisons with reference data were made. The agreement is in practically all cases excellent.
12	14	Change "vacuum" to "vacuum"
14	18	Should read: case of capacitance
16	6 & 7	Should read: ceramic does not necessarily have the
37	12	Should read: In the case
45	4	Should read: identical to the
94	Abstract line 8	Should read: method, electrical conductivity and dissipation factor of C_6H_6 -liquid, thermal

Foreword

This program had as its objective the development of a new instrument to accurately measure electrical, thermal and optical properties of fluids over wide ranges of pressure and temperature and of important properties of the instrument and its material. The cell was designed and constructed and brought into operational readiness under contract AF 49(638)-1574. The research described in the report demonstrates that fourteen of the properties that the instrument is capable of measuring can be determined accurately. For the continuation of the work properties of selected fluids such as simple monoatomic gases will be measured to permit construction of a theoretical model for use with more complex molecules. Influences of the effects of dissociation will also be studied. Selected liquids such as freons will be investigated for their suitability as heat transfer substances especially in compact electronic devices as used in computers and guidance systems of high speed aircraft and rockets.

It is further anticipated that the feasibility of converting the multi-purpose instrument to semi- or fully automatic operation will be investigated and that the unsteady technique described in the report for remote observations of heat transfer and properties will be developed.

Table of Contents

	Page
Foreword	i
Abstract	1
1. Introduction	1
2. Short Description of the Instrument and the Procedure for Measuring the Various Properties	4
Fig. 1 Multi-Purpose Instrument [MPI]	5
Fig. 2 Wiring Diagram and Switching Arrangement for Measurements of the Different Properties	8
Fig. 3 Wiring Diagram for Power Measurements	8
Fig. 4 Photographic Picture of Laboratory	10
3. Experimental Results Under Steady State Conditions	11
3A Determination of the Properties of the Instrument	11
3A1 Measurements of the Geometric Constant B	11
Fig. 5 Capacitance as a Function of Vertical Displacement of Hot Body at 25°C Under Vacuum	14
Fig. 6 Capacitance of Geometrical Arrangement Under Vacuum as a Function of Tempera- ture	15

	Page
3A2 Determination of Thermal Expansion Coefficient of Instrument Material and Centering Rod	15
Fig. 7 Capacitance as a Function of Temperature for Hot Body Displaced Off Center	17
3A3 Determination of the Volume of the Instrument Accommodating the Test Fluid	19
Fig. 8 Arrangement For Measuring Volume of MPI	21
3B Determination of Various Properties of Selected Substances	27
3B1 Thermal Conductivity of Helium at 20 and 30°C in a Range of Pressure	27
Fig. 9 Thermal Conductivity of Helium Gas vs. Pressure	28
3B2 Measurements of Dielectric Constant, Thermal Conductivity and p-v-T Data of Nitrogen at Three Constant Densities in a Temperature Range 0-100°C. And Evaluation of Susceptibility, Polarizability and Index of Refraction	29
Fig. 10 Pressure and Dielectric Constant of Nitrogen vs. Temperature at Constant Density $\rho = 0.36265 \times 10^{-3} \text{ gm/cm}^3$	30
Fig. 11 Pressure and Dielectric Constant of Nitrogen vs. Temperature at Constant Density $\rho = 1.02723 \times 10^{-3} \text{ gm/cm}^3$	31

Fig. 12 Pressure and Dielectric Constant of Nitrogen vs. Temperature at Constant Density $\rho = 1.7334 \times 10^{-3} \text{ gm/cm}^3$. . .	32
Fig. 13 Thermal Conductivity of Nitrogen vs. Temperature at Three Constant Densities	33
3B3 Determination of Thermal Conductivity and Dielectric Constant of Octafluorocyclobutane C_4F_8 - Vapor at 1 atm in the Range of Temperature 0-100°C	35
Fig. 14 Thermal Conductivity and Dielectric Constant of C_4F_8 at 1 atm vs. Temperature	35
3B4 Vapor Pressure Measurements of Methanol in the Temperature Range 0-100°C	36
Fig. 15 Vapor Pressure of Methanol vs. Temperature	38
3B5 Determination of Thermal Conductivity, Dielectric Constant, Electrical Conductivity and Dissipation Factor of C_4F_8 Liquid at 15 atm in the Temperature Range 0-80°C	39
Fig. 16 Thermal Conductivity and Dielectric Constant of C_4F_8 at 15 atm vs. Temperature	40

Fig. 17 d.c. Electrical Conductivity and Dissipation Factor of $C_4 F_8$ at 15 atm vs. Temperature	41
3B6 Determination of Thermal Expansion Coefficient of Benzene at 1 atm at Various Temperatures	42
Fig. 18 Thermal Expansion Coefficient of Benzene at 1 atm vs. Temperature	43
4. Unsteady State Experiments. A Technique For Precise Determination of Heat Transfer in the Absence of Temperature Measurements and Its Extended Applica- tion to Properties	43
Fig. 19 Multi-Purpose Instrument Used in Transient Conditions to Determine Thermal Diffusivity of Test Fluids	44
Fig. 20 Schematics of $T = f(t)$ for Hot and Cold Bodies and $C = f(T, t)$ for Case a, Test Fluid is a Perfect Insulator	45
Fig. 21 Same but for Case b, Test Fluid is a Perfect Conductor	45
Fig. 22 Same but for Case c, Test Fluid is a Finite Conductor	46
4.1 Determination of Thermal Transport Properties of Helium, Nitrogen, and Argon at 1 atm and Vacuum in a Small Temperature Range	46

**Fig. 23 Change of Capacitance with Time for
the Determination of Thermal**

Diffusivity of Fluids	48
5. Summary and Future Work	49
6. Acknowledgment	50
7. References	51

MEASURING 14 PROPERTIES WITH THE MULTI-PURPOSE
INSTRUMENT AND A TEMPERATURE FREE METHOD
OF MEASURING THERMAL TRANSPORT PROPERTIES

Abstract

The multi-purpose instrument originally designed to determine seven properties was extended in its capabilities to measure additional properties. The paper describes briefly the instrument and the operational procedures and brings the results of measurements of some of the properties, namely: four properties of the instrument or its wall material, thermal conductivity of He, N₂ and C₄F₈ (in vapor and liquid state), p-v-T data of N₂, dielectric constant of N₂, He, A and C₄F₈-vapor and liquid, vapor pressure of methanol, electrical conductivity and dissipation factor of C₄F₈-liquid, thermal expansion coefficient of Benzene index of refraction and polarizability of N₂. A new unsteady method is introduced for the determination of heat transfer and thermal transport properties without measuring temperature and its technique is demonstrated by heat transport observations in He, A, N₂ and vacuum.

1. Introduction

Much effort is currently expended to measure and/or calculate the properties of materials over continuously widening ranges of pressure, temperature and, in the case of mixtures, composition. The user of such data has the task of

reconciling different types and quality of information and often estimating new values for a needed condition. This is true for a single property. Often more than one property is needed and the necessary information cannot be found. Even if it can be found the values usually are not physically concordant. In order to overcome these difficulties the concept of an instrument able to measure many properties of substances was introduced and followed up by the construction of the apparatus which, when properly functioning, should (a) eliminate variations in sample in different tests, (b) secure identical conditions for all different properties and (c) greatly reduce the time and expenses otherwise needed for separately measuring all the properties. The instrument further should allow most of the data to be observed absolutely, with high precision and over as wide a range of temperature and pressure as feasible for accurate measurements. The data therefore should be usable for theoretical studies, to verify and check models for prediction of properties by statistical mechanics especially for those ranges of temperature where it is impossible to carry out measurements. The theory and design of the development of the instrument have been described in detail in reports (1)* seminars (2,3) and presentations at national and international meetings (4,5,6) and so were the facilities of the laboratory and the experimental setup necessary for operating the instrument (7). This paper

* () refers to References.

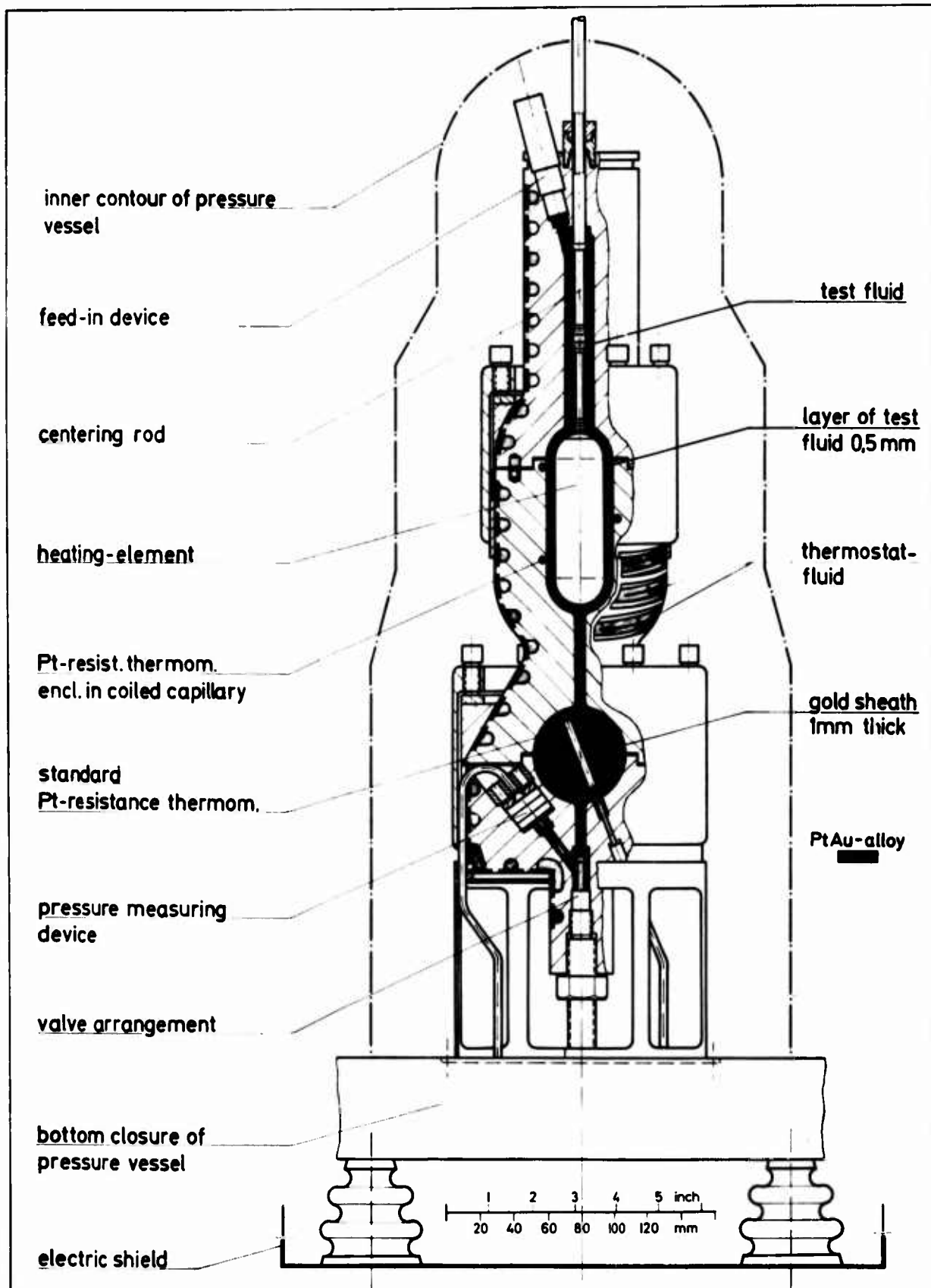
discusses first the procedure to obtain the properties of the instrument and then the results of measurements of properties of various test fluids. It was not attempted to measure for each of the test fluids all the properties the instrument can determine but to demonstrate that the apparatus can be used to determine with high accuracy as many properties as was proposed.

For comparison reasons such test fluids in most instances were chosen for which the property or properties to be determined were well known. The ranges of pressure and temperature were selected accordingly. In some other cases samples were selected for which only one or two properties were known accurately but not the others--or given only for single conditions of pressure and temperature. For safety reasons the measurements were carried out at pressures and temperatures which are far from the extremes of the design capability of the instrument. Some of the measurements are used for theoretical studies but those are beyond the scope of this paper and therefore not included.

For the sake of easier reading a short description of the instrument and its operation is felt necessary despite the fact that detailed information is given in the above references.

2. Short Description of the Instrument and the Procedure to Measure the Various Properties

The instrument shown schematically in Figure 1 consists of four major parts. A heating element (the so called "hot body") of cylindrical shape with hemispherical ends enclosed in a similarly formed but slightly larger upper cavity of the cold body, which is made out of three parts. The lower part encloses a pressure measuring device, valve arrangements and a standard Pt-resistance thermometer which is located in the lower spherical cavity formed between the lower and middle parts of the cold body. The upper part suspends and centers the hot body by means of a centering rod. It furthermore houses a feed-in device. The test fluid is filled into the system either from below or through the feed-in device. The instrument can be sealed off completely for constant volume measurements or possibly connected with outside instrumentation for other types of measurements. The temperature of the instrument is regulated by means of thermostat fluids channeled bifilarly through passages provided as indicated in the figure. The temperature of the hot body is determined by a Pt-resistance thermometer placed in a coiled capillary hard soldered in the wall at a well known distance from the surface. The temperature of the cold body is also measured by a Pt-resistance thermometer similarly mounted in the wall at a location where it surrounds the hot body. The range of temperature is from -190°C to $+650^{\circ}\text{C}$, the pressure range from vacuum to 500 atm.



Multipurpose instrument

Fig. 1

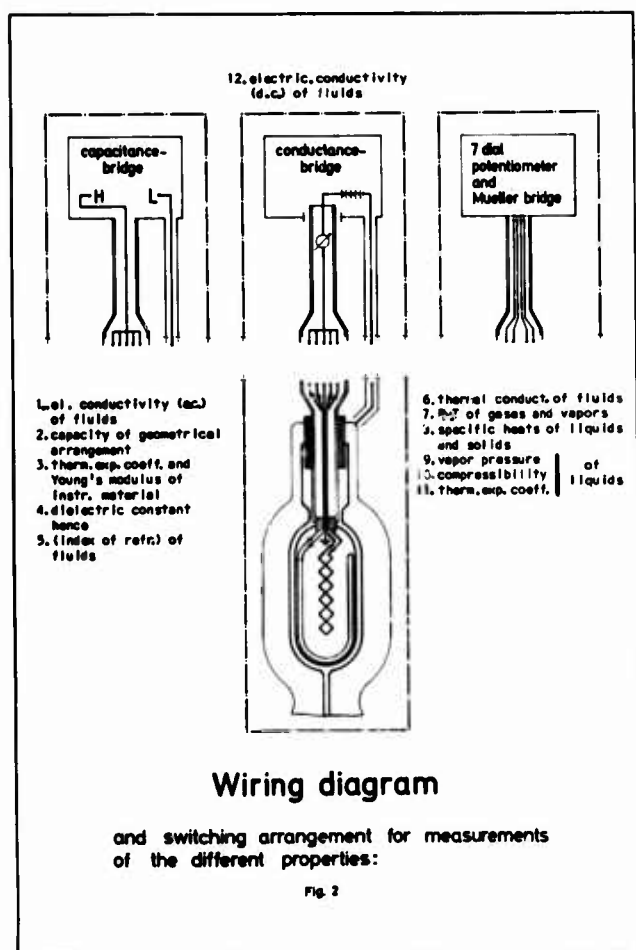
The hot body contains a heater element and serves to generate a thermal potential between it and the cold body when thermal conductivity is to be determined. Likewise by establishing an electrical potential difference between the hot and cold bodies the fluid electrical conductivity can be measured. In the first case Fourier's Law is applied, in the latter case Ohm's Law. The geometric constant, i.e., the ratio of overall area and average width of the gap between hot and cold body for both cases, is determined by capacitance measurements under vacuum and the value of permittivity of free space. Measuring the capacitance of the arrangement as a function of temperature will yield the change of geometry with temperature which allows the thermal expansion coefficient of the instrument material to be computed. It will be shown below that repeating these measurements under slightly eccentric positions of the hot body within the cold body cavity will yield the thermal expansion coefficient of the centering rod. The change of the instrument with pressure can be determined by observing pressure changes of capacitance with a test fluid for which the dielectric constant is known as a function of pressure. Measuring the capacitance with a test fluid in the system allows evaluation of the dielectric constant of the sample when the capacitance value is divided by the vacuum value observed at identical temperature and corrected for possible change of geometry due to pressure. In many cases the index of refraction can simply be computed from the dielectric constant.

The measurements of capacitance are carried out by a three lead technique with the aid of a capacitance bridge (General Radio: Type 1615A) which for highest sensitivity must be balanced in respect to dissipation. This yields together with the frequency of operation the a.c. electrical conductivity.

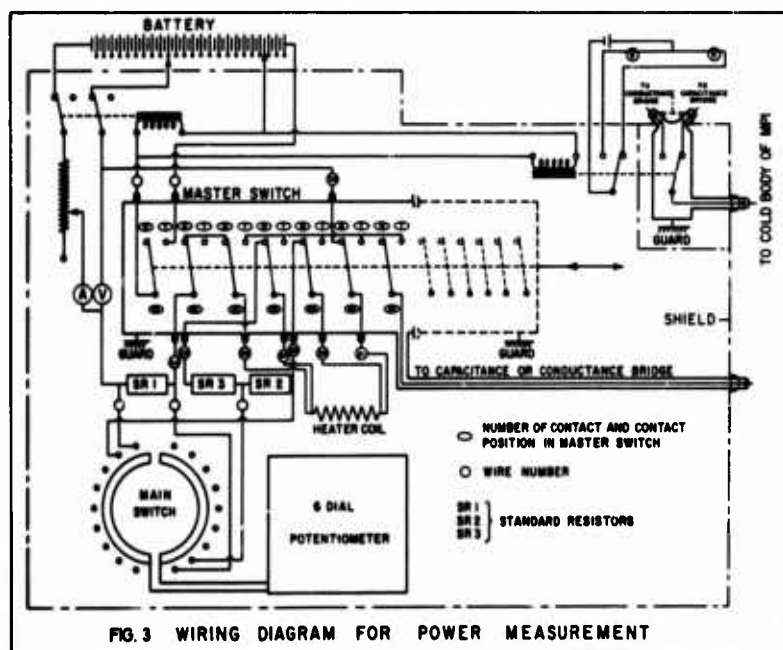
The volume of the instrument accommodating the test fluid can be determined as shown below precisely at a given condition of temperature and pressure. From the properties of the instrument known the volume value for other conditions can be computed. Therefore p-v-T properties of vapors and gases can be determined by observing under various specific volume conditions the change of pressure with temperature.

Likewise vapor pressure can be measured when the instrument is filled only with a certain amount of liquid. If the instrument is filled completely with a liquid which is in contact with outside instrumentation then its volume change with pressure or temperature can be observed and its compressibility and thermal expansion coefficients can be computed.

There are other steady state measuring possibilities of other properties which are in part described in (1)--also some others under quasi-steady state. These are not utilized in the present paper. In addition the instrument can be used under unsteady state conditions for the observation of six other properties. Only one of these, namely the determination of thermal diffusivity (and conductivity), will be demonstrated and discussed in detail below.



The measurements of most of the properties listed are carried out and made possible by altering only outside connections as shown schematically in Figure 2 for the electrical observations. A more detailed wiring is given in Figure 3 showing the arrangements for measuring power input to the hot body by voltage and current measurements with the aid of standard resistors and a potentiometer. The current and potential leads of the heater coil are connected by means of a master and main switch to the potentiometer and the standard resistors. In



another position of the master switch the four leads of the heater coil (and also those of the hot body thermometer--not shown) are combined and connected to the ground wire of the hot body.

The so formed single lead is connected in one position of the master switch to the low side of the capacitance bridge. In another similar position of the master switch* the single lead is connected to a conductance bridge. The cold body is connected to the high sides of the two bridges by means of an antenna switch activated accordingly by the masterswitch. (The antenna switch is shown in the upper right hand corner of Figure 3.)

A considerable effort was spent to assure in the wiring least possible dissipation in respect to capacitance measurements and highest possible d.c. insulation in respect to conductance measurements. The conductance bridge (Honeywell Model 1700) is a Wheatstone type bridge utilizing in their arms two simultaneously changeable standard resistors which are of 33 Megohm value when extremely low conductivity values are to be observed. The wiring of the instrument represents many parallel resistors to these bridges resistors. For the sake of high accuracy in the measurements it was therefore necessary to insulate the wiring from the shield as well as possible. This was achieved by selecting wires with high grade insulation and placing them in metallic and insulated tubes which are connected to a metallic box enclosing the masterswitch. This box is on shield or guard potential and the metallic tubes represent shield or guard continuation.

*The master switch is a rotary switch with 60 positions circumferentially and 50 positions axially. From the 3000 possible switch combinations only 300 are used for the wiring. Other positions of master switch refers to a different position circumferentially--and activation of other contacts.

The box is maintained continuously under a slight positive pressure of dry nitrogen which leaks out at the open ends of the tubes. Moist air therefore never can enter the system. Dissipation losses could be disregarded completely (at least in the most common range of frequency used) and a total d.c. resistance between wires and shield of better than 10^{12} Ohm could be established and maintained.

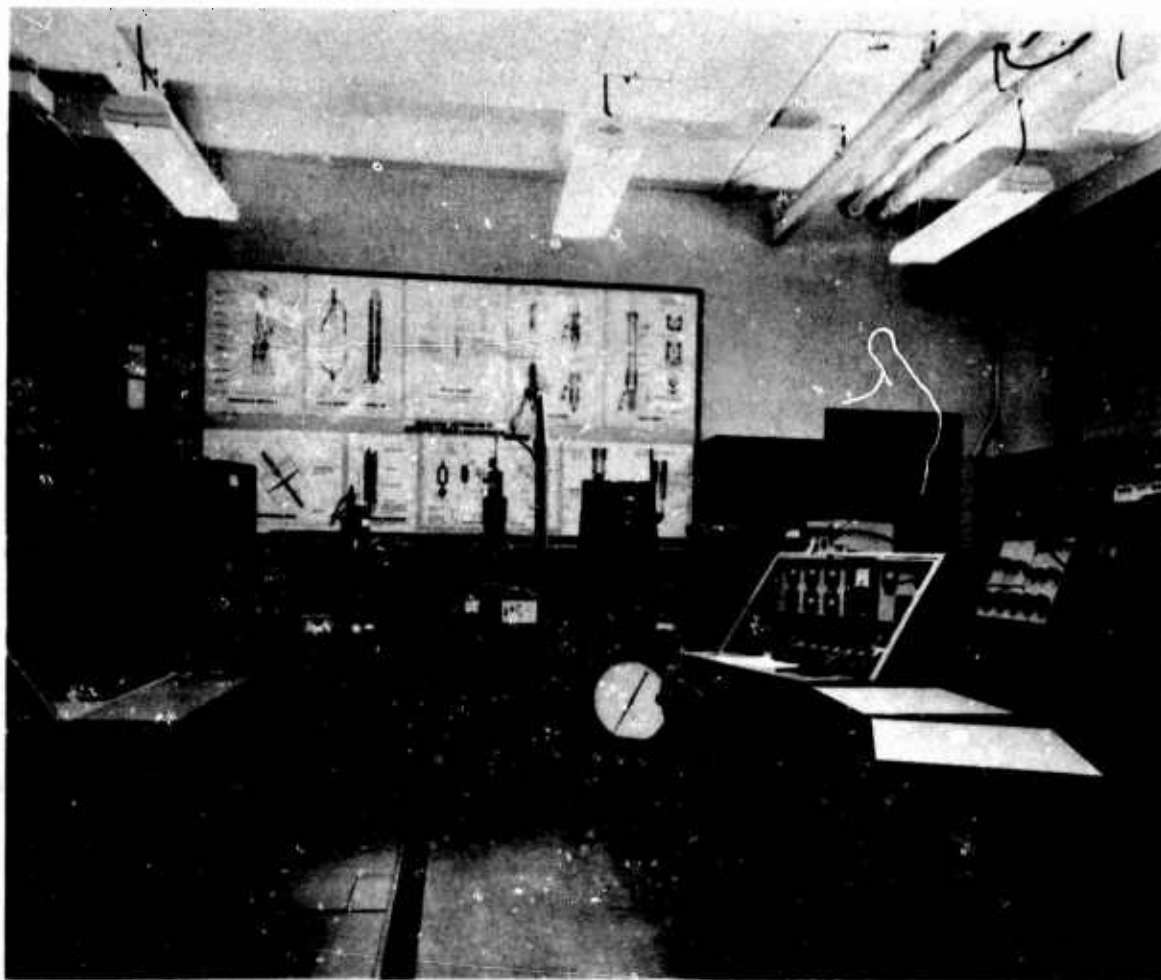


Fig. 4 Laboratory

Figure 4 shows a photograph of the experimental setup of the laboratory and the arrangements of the operational facilities. Starting at the right hand side Figure 4 shows

first the capacitance bridge, next to it the conductance bridge and the masterswitch. Further to the left can be seen the potentiometer and the mainswitch. All these bridges are mounted in consoles which are insulated from ground and represent shield respectively guards which are connected to the mounting platform of the multi-purpose instrument shown in the center of the photograph. The console on the right hand side of the instrument houses devices for filling and emptying the test fluid, further pressure measuring equipment and devices to observe volume changes of test liquids. On the other side of the instrument are located vacuum pumps and thermostats. Further to the left is an automatic control unit needed to heat or cool the cold body to the temperature of the calorimeter container when specific heats are measured. (See Ref. 1-specific heat measurements are not included in the present work.) Next to this unit is a Mueller bridge used to calibrate with the aid of a NBS standard the Pt-resistance thermometers built into the cell. The equipment is arranged in such a way that it can be run by a single observer.

3. Experimental Results Under Steady State Conditions

3A Determination of the Properties of the Instrument

3A1 Measurements of the Geometric Constant B

The geometry of layer of test fluid bounded by the walls of hot and cold bodies is given by

$$\frac{A}{L} = B = 2\pi \left[\frac{h}{\ln D/d} + \frac{D \cdot d}{D-d} \right] \quad (1)$$

where h is the cylindrical length of hot body and of cold body's upper cavity, d and D are their respective diameters.

This relationship is correct only for perfect geometry, i.e., homogeneous field everywhere. Influences of surface roughness and interruption of the layer by the bore at the bottom and at the top and at the location where the upper and middle part of the cold body join together are neglected.

It is impossible to correct the relationship analytically but it is possible to measure its true value which includes all the effects of these disturbances by determining the capacitance of the arrangement according to the equation

$$C = B \epsilon_0 \epsilon_f \quad (2)$$

where ϵ_0 is the permittivity of free space, a physical constant precisely known and ϵ_f the dielectric constant of the test fluid. Measuring under vacuum makes ϵ_f equal unity and therefore

$$B = \frac{C}{\epsilon_0} \quad (3)$$

This value of B is correct for the measurements of the transport properties only if in those cases identical conditions exist, i.e., isopotential surfaces and /or homogeneity of the field can be assumed or assured. For electrical measurements isopotential surfaces normally can be achieved more easily than in thermal cases. In order to aid the conditions for the latter measurements the cold body's sink represented by

the channels for the thermostat fluid has been given the geometrical shape of the hot body. (The influence of non uniform heat flow due to disturbance in its path will be discussed below.)

The value of the capacitance measured and therefore of B will have a minimum at a position of hot body within cold body's cavity where the most homogeneous field is present. In case of perfect geometry this position will be at a perfectly centered one. Due to the disturbances mentioned above (especially caused by the bores) a minimum value will occur at a slightly eccentric position in axial direction as was proven in (8). It is of advantage to measure properties under homogeneous field conditions and therefore it was necessary to determine this position. This was achieved by measuring the capacitance of the arrangement under vacuum and constant temperature as a function of axial displacement of the hot body in the cold body's cavity. The results of the measurements are given in Figure 5. The almost parabolic curve was observed by moving the hot body by means of a screw arrangement from a lowest possible position to the upper most position. The latter occurs at a lesser displacement off center because of the sealing arrangement between the centering rod and the upper cold body. The measurements were carried out at 25°C. In order to establish the geometric constant for other temperatures of interest to the measurement of the transport properties and the dielectric constant it was necessary to determine the capacitance of

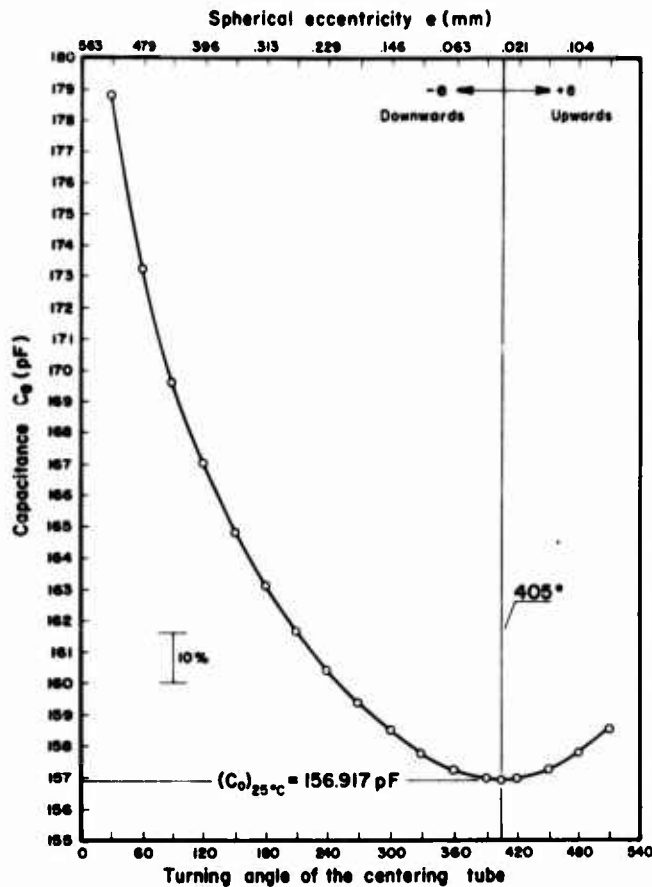


Fig.5 Capacitance as a function of vertical displacement of hot body at 25°C under vacuum

the arrangement at minimum setting and under vacuum as a function of temperature. The results of these measurements are shown in Figure 6. The data observed under increasing and decreasing temperatures scatter only in a few cases and then only by thousandths of a per cent around the smooth curve.

During the measurements of each point it was observed that the capaci-

tance values given were not established at the instant thermodynamic equilibrium was reached but with a certain time lag of the order of 1/2 hour. In the case capacitance measurements under increasing temperatures the data increased by a few thousandths of a per cent to the final value. The opposite was observed for measurements under decreasing temperature. This observation indicates that the change of dimensions due to thermal expansion does not occur simultaneously with a change in temperature.

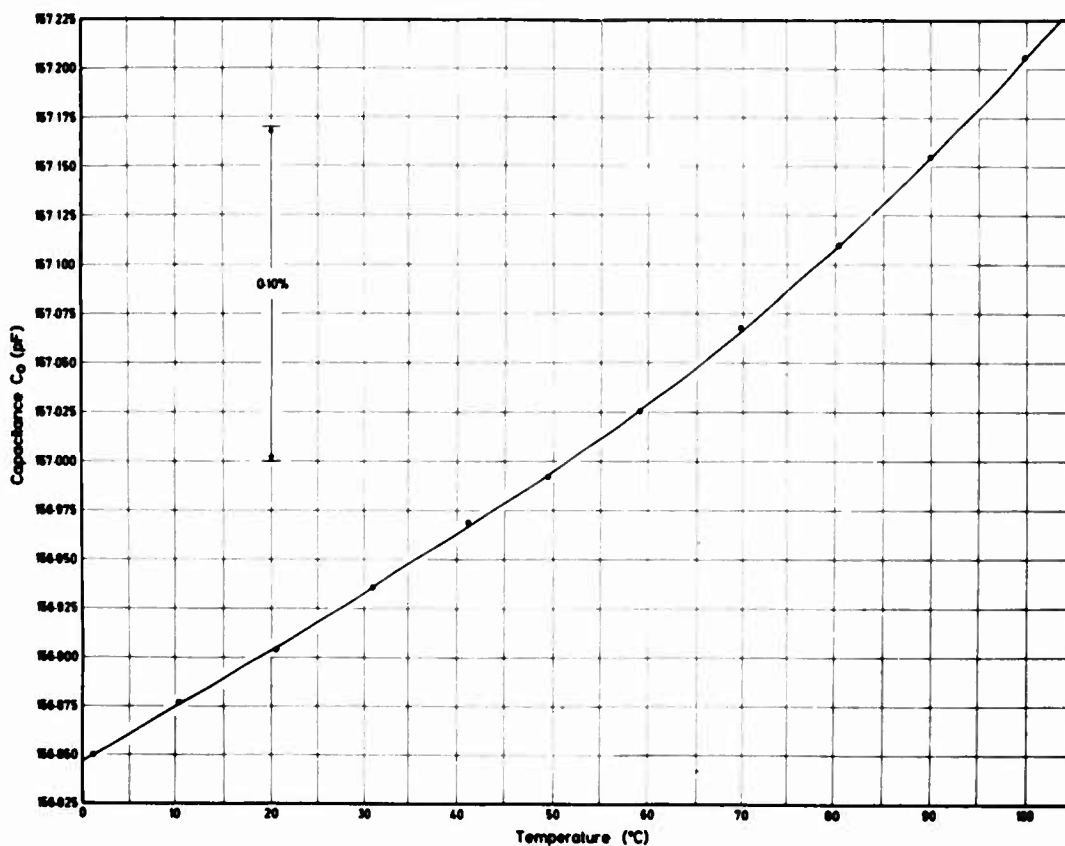


Fig. 6 Capacitance Of Geometrical Arrangement Under Vacuum As A Function Of Temperature

3A2 Determination of Thermal Expansion Coefficient of Instrument Material and Centering Rod

The capacitance curve of Figure 6 shows an increase in slope with temperature. Inspection of equation 1 demonstrates that the geometric constant will increase linearly with temperature for constant thermal expansion coefficient. The curvature therefore could be explained by an increase in thermal expansion coefficient with temperature. An analysis of the curve yields values of the thermal expansion coefficient and its temperature dependence which are both considerably higher than literature data. Therefore an

additional temperature dependent influence on the capacitance measurements must exist. Those changes are possible only when changing temperature will cause displacement of the hot body relative to the cold body. An axial displacement is likely to occur for reasons that the centering rod being a structure composed of three different metals and ceramic not necessarily has the same thermal expansion as the material of the cold body--despite the fact that during the design and construction a matching of thermal expansion coefficients of the two bodies was attempted.

Figure 5 demonstrates that an axial displacement in either direction from a minimum setting will increase the capacitance. The axial displacement due to a temperature change from 25°C (at this temperature hot body was placed such to achieve minimum capacitance of the arrangement) is

$$e = L_{R_T} - L_{N_T} \quad (4)$$

with length of centering rod at $T \neq 25^\circ\text{C}$

$$L_{R_T} = L_{R_{25}} (1 + \alpha_R \Delta T)$$

and length of cold body surrounding centering rod

$$L_{N_T} = L_{N_{25}} (1 + \alpha_N \Delta T)$$

for

$$L_{R_{25}} = L_{N_{25}} = L_{25}$$

$$e = L_{25} (\alpha_R - \alpha_N) \quad (5)$$

Increasing temperature will cause a motion of the hot body relative to the cold body in a downward direction when the thermal expansion coefficient of the rod α_R is larger than that of the cold body α_N . For $\alpha_R < \alpha_N$ motion in the opposite direction will occur.

Repeating the measurements of capacitance change with temperature will determine if $\alpha_R > \alpha_N$ or vice versa when in the arrangement the hot body is placed eccentric at 25°C.

The capacitance curve of the arrangement obtained with hot body originally displaced downward is given in Figure 7.

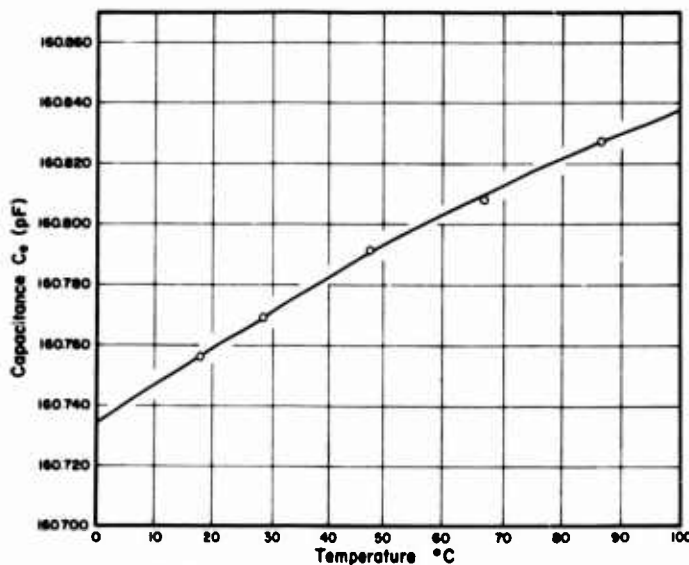


Fig.7 Capacitance as a function of temperature for hot body displaced off center

The curve deviates from linear but now by a slope decreasing with temperature. This indicates that axial displacement was in an upward direction and therefore $\alpha_R < \alpha_N$.

The curves of Figures 6 and 7 allow the values of α_R and

α_N to be evaluated as follows.

The axis of the centering rod is perfectly aligned with the axis of the cold body. Similarly well aligned is the bore accommodating the rod in the upper part of the cold body with the axis of the upper cavity. Therefore it can be assumed that eccentricity in a radial direction does not exist and

will not be introduced by temperature changes. The curve of Figure 5 therefore gives only the change of capacitance with axial displacement--and the curves of Figures 6 and 7 are influenced by eccentricity in axial direction only. In the neighborhood of the minimum value the curve of Figure 5 can be represented by an equation which results from eccentricity in the spherical part of the arrangement only as was proved in Ref. (8).

$$(C_e)_{\text{total}} = (C_e)_{\text{sphere}} + (C_o)_{\text{cyl.}} \quad (6)$$

where the subscript e refers to axial eccentricity and the subscript o to zero eccentricity and

$$(C_e)_{\text{sphere}} = (C_o)_{\text{sphere}} \left[1 + 2 \left(\frac{d}{D(D-d)} \right)^2 e^2 \right] \quad (7)$$

is the capacitance of the spherical arrangement influenced by eccentricity, neglecting higher order terms. $(C_o)_{\text{sphere}}$ is the capacitance value of the spherical part of $(C_o)_{\text{total}}$ of Figure 5.

The C_e value observed at 25°C in Figure 7 yields from Figure 5 or eq. 6 and 7 the initial axial displacement. The capacitance values observed in the temperature range of Figures 6 and 7 introduced into equations 1, 5, 6 and 7 allow the thermal expansion coefficients of the centering rod and of the Nimonic 80A wall material of the instrument to be determined--at any temperature. Since the computation is quite involved only averaged values (0-100°C) were calculated with the results.

$$\alpha_N = 12.56 \times 10^{-6} (\text{°K})^{-1}$$

$$\alpha_R = 12.02 \times 10^{-6} (\text{°K})^{-1}$$

Of these two values only α_N is of interest to the measurements with the multi-purpose instrument mainly for reasons of correcting the volume of the test fluid enclosed within the cell when p-v-T, thermal expansion and compressibility are determined as function of temperature.

A literature value of thermal expansion coefficient of Nimonic 80A (9) is

$$\alpha_N = 11.9 \times 10^{-6} (\text{°K})^{-1}$$

and deviates from the value given above by approximately 5%. The walls of the multi-purpose instrument as shown in Figure 1 are lined at its wetted surfaces by a 1 mm thick gold layer. Taking the thermal expansion coefficient of gold $\alpha_g = 14 \times 10^{-6} (\text{°K})^{-1}$ into account will yield for the combined structure a higher coefficient which is in very good agreement with the value computed from the measurement.

The averaging procedure followed for the computation introduces a certain degree of uncertainty. But this error is small enough to be neglected.

3A3 Determination of the Volume of the Instrument Accommodating the Test Fluids

The multi-purpose instrument as discussed in section 2 is operated either with the test fluid connected to outside

instrumentation or hermetically sealed. This is accomplished by using two different valve arrangements seated differently into the lower part of the cold body. The valve shown in Figure 1 is seated at a location close to the lower spherical cavity. In this condition the test fluid is connected with the outside. In case p-v-T data will be observed or other properties under constant volume conditions another valve is used which is then seated at a somewhat lower location and at a wider rim. The instrument can be evacuated by means of this valve and test fluid either can be charged into the cell through this valve or by means of the feed-in device. After charging both devices will be sealed off. The pressure of the sample is then measured by means of the pressure measuring device which is at the temperature of the test fluid and its readings therefore cannot be influenced by condensation and/or evaporation.

The volume occupied by the test fluid within the instrument under these different sealing conditions must be precisely known. They were determined by measuring the volume of mercury necessary to replace a gas which fills the instrument from vacuum to a preset pressure. The arrangement is shown in Figure 8. The two self-explanatory steps of measurements yield the unknown volume according to the equation

$$V = \frac{M_1 - M_2}{\rho} \quad (8)$$

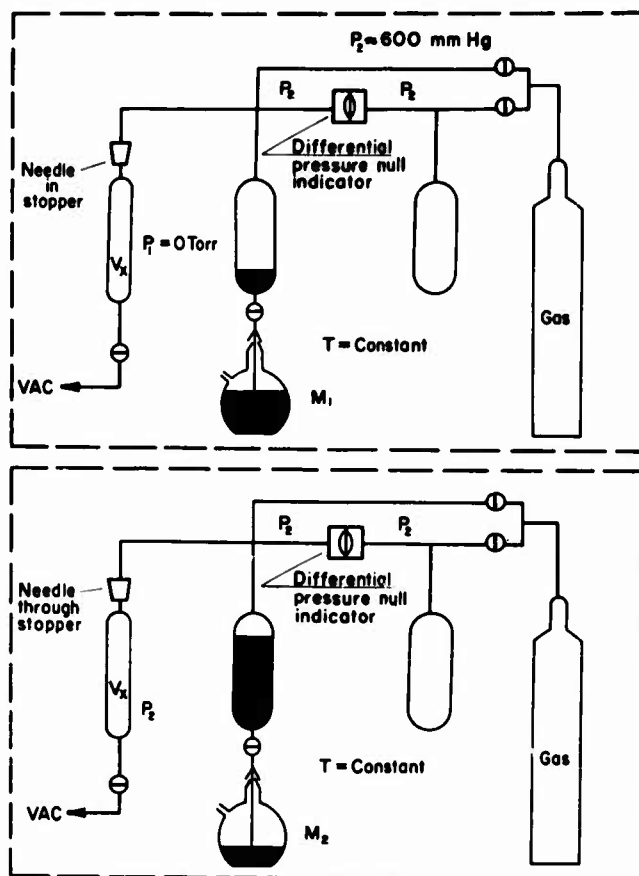


FIG.8 ARRANGEMENT FOR MEASURING VOLUME OF MPI

where M_1 is the mass of mercury of density ρ observed in the reservoir at step 1 and M_2 the mass remaining in the reservoir after the mercury piston compressed the gas charging the instrument to the original pressure p_2 . M_1 and M_2 were determined with the aid of a precision balance which readings were corrected for buoyance.

The dotted lines surrounding the experimental

set up represent an isothermal envelope. In the present case the laboratory itself was maintained by controlled heating and cooling at a temperature of 28.15°C . The volumes observed at this temperature are:

a., Valve seated at upper rim: $V_L = 83.8730 \text{ cm}^3 \pm 0.0018 \text{ cm}^3$

b., Valve seated at lower rim: $V_V = 84.2284 \text{ cm}^3 \pm 0.0007 \text{ cm}^3$

These values can be corrected for other temperatures by means of α_N .

Together with the geometric constant the most important properties of the instrument itself are therefore known and properties of test samples can be determined absolutely and with high accuracy. Obviously in addition it is necessary to

determine changes of volume and of geometric constant with pressure. Due to the complexity of the geometry only an estimate can be made analytically.

For this it was assumed that the volume of the cell is enclosed in a cylinder of inside bore equal in diameter to the bore of the upper cavity and of identical wall thickness.

For an infinitely long cylinder (inside pressure $p_i = p$ outside pressure $p_o = 0$)

$$\frac{\Delta r}{r_i} = \frac{1}{E} (\sigma_t - \nu \sigma_r) = \frac{p}{E} \left[\frac{(r_o/r_i)^2 + 1}{(r_o/r_i)^2 - 1} + \nu \right] \quad (9)$$

where r_i = inside radius = 17.5 mm
 r_o = outside radius = 45 mm
 E = module of elasticity = 1.9×10^6 kp/cm²
 ν = Poisson's ratio = 0.3

For $p = 500$ atm

$$\frac{\Delta r}{r} = 4.36 \times 10^{-4} \text{ and}$$

$$\frac{\Delta V}{V} = 8.72 \times 10^{-4}$$

The change of volume is less than .1% and will be approximately .1% when the change of volume of the hot body is included.

The real volume change with pressure will be smaller for reasons of the heavier walls of the spherical cavity which occupies approximately 75% of the total volume.

The remaining uncertainties in determining the volume as a function of internal pressure therefore might be of the

order of a few hundredths of a per cent at highest pressure and negligibly small at lower pressures.

The situation is much more critical in respect to the geometric constant because Δr is relatively large compared to the layer of test fluid. Together with the decrease of diameter of hot body the total increase in width will be 2.6% at 500 atm. This large influence necessitates a more careful evaluation as follows.

Equation 1 can be rewritten

$$B_2 = 2\pi \left[\frac{\sqrt{h_{c2}} \cdot \sqrt{h_{h2}}}{\ln D_2/d_2} + \frac{D_2 \cdot d_2}{D_2 \cdot d_2} \right] \quad (10)$$

the subscript 2 refers to the dimensions of the hot and cold bodies under a different pressure than when B was determined. B_2 cannot be analyzed properly but it can be determined by measurements of capacitance with a test fluid of known dielectric constant at two (or more) different pressures.

$$C_1 = B_1 \epsilon_0 \epsilon_1 \quad \text{observed at pressure } p_1$$

(Preferably 1 atm--since ϵ_1 is known)

$$C_2 = B_2 \epsilon_0 \epsilon_1 \quad \text{observed at pressure } p_2$$

Introducing $B_2 = \beta B_1$ yields

$$\beta = \frac{C_2 \epsilon_1}{C_1 \epsilon_2} \quad (11)$$

Measurements with helium at 1 and 15 atm and 20°C resulted in

$$\beta = \frac{156.878 \times 1.000065}{156.913 \times 1.000650} = .99919$$

where ϵ_2 was evaluated from ϵ_1 by assuming perfect gas behavior. β indicates a decrease in geometric constant by .08%. Linear extrapolation to 500 atm yields 2.69% a slightly higher value than estimated above which is understandable because the analysis regarded only changes in radial direction.

If the change of dimensions of each of the terms in equation (10) could be evaluated properly in terms of p and E the latter could be evaluated. But only when the pressure does not introduce eccentricity in the arrangement.

The measurements of β will include possible eccentricities which are only a function of geometry and properties of the instrument and the pressure forces. β itself therefore is a property of the instrument and must be remeasurable under identical pressures independent of the test substance, as long as the respective fluid is ideal and ϵ_2 can be computed properly. If measurements with a gas do not reproduce β then the gas is not ideal and the deviation in β is a measure of the deviation from perfect gas conditions for this sample at the respective pressure.

Since E is practically temperature independent β will remain constant with temperature when corrected for thermal expansion. The change of geometry with pressure observed above can be used to determine a pressure coefficient of B by introducing

$$B_2 = B_1 (1 - \alpha_p \Delta P) \quad (12)$$

which yields with β

$$\alpha_p = 57.9 \times 10^{-6}/\text{atm}$$

Changing pressures from 1 atm to vacuum will result in an increase of B. Whenever a gas of susceptibility = 57.9×10^{-6} is in the system then changing the pressure from 1 atm to vacuum will not change the capacitance despite the fact that the dielectric constant of the gas decreases to unity. For gases having a susceptibility smaller than 57.9×10^{-6} the capacitance of the evacuated system will be larger than at 1 atm. This effect was observed with the multi-purpose instrument at elevated temperatures and helium as the test gas.

The temperatures as already stated above are measured by means of resistance thermometers made out of standard grade platinum. These thermometers mounted and sealed securely into the instrument were calibrated in place. The properties observed were used to compute tables with one thousandths of a degree Kelvin interval.

The properties of the thermometers are:

	$R_0 = 25.6168 \text{ Ohm}$
	$\alpha = 0.003924846$
Standard	$\delta = 1.55367$
	$\beta = 0.110$

Hot Body

$$R_o = 1.62968 \text{ Ohm}$$

$$\alpha = 0.003919673$$

$$\delta = 1.61647$$

$$\beta = 0.110$$

Cold Body

$$R_o = 1.73489 \text{ Ohm}$$

$$\alpha = 0.003912994$$

$$\delta = 1.59571$$

$$\beta = 0.110$$

The pressure is measured by means of mercury or water manometers, by calibrated Heise gauges and by a pressure balance. Unfortunately the pressure balance only covers the range 250 to 500 atm. The measurements of this report were restricted for small pressure ranges whenever precision in the readings were necessary. It is hoped that funds become available to cover the total range of pressure measurements with more sensitive and reliable instruments. The pressure measuring device mentioned above observes the pressure indirectly by means of a diaphragm opening and closing an electrical contact. At contact position the diaphragm is very sensitive but due to the fact that it was not perfectly flat when mounted into the device a slight pressure differential must be overcome first. This value has been determined and is known to a fraction of .1 mm Hg for the temperature range of the measurements.

3B Determination of Various Properties of Selected Substances3B1 Thermal Conductivity of Helium at 20 and 30°C in aRange of Pressure

The measurements of thermal conductivity were the most critical ones for reasons that they not only check a considerable part of the total system but most of all the quality of the gold bond. Values observed too low would indicate a faulty bond for reasons of apparently too high values of temperature difference. An insufficient and/or non-uniform bond obviously would in addition influence or possibly even prevent the measurements of most of the other properties. It was therefore a great relief when the first value of thermal conductivity ever measured with the multi-purpose instrument was identical to the data given in Ref. (10). Further measurements under increasing and decreasing pressure agreed very satisfactorily with the well established pressure dependence of helium for the two isotherms. The hump in the curve observed first in (10) seems to be verified by the measurements. The data given in Figure 9 was computed from the measurements of ΔT between hot and cold body and the power input q and the geometric constant at the respective temperatures from Fourier's Law.

$$k = \frac{q}{B\Delta T} \quad (13)$$

B was corrected for change of pressure and for the influence of $T_h > T_c$. Other corrections were not applied for reasons

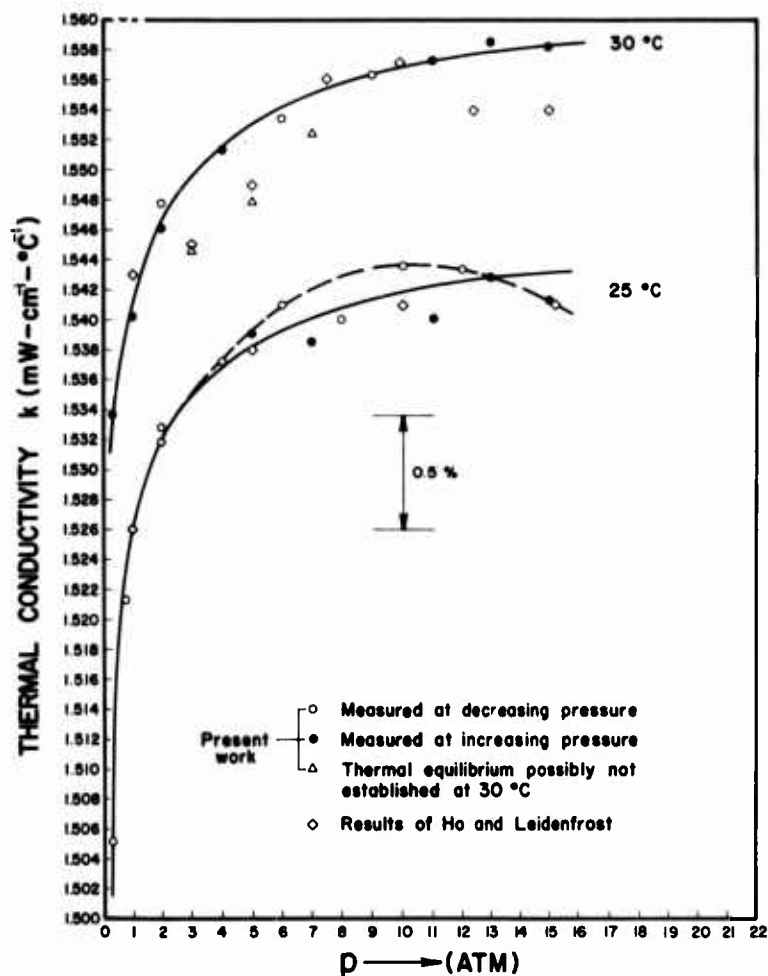


FIG.9 THERMAL CONDUCTIVITY OF HELIUM GAS vs. PRESSURE

that radiant heat transfer between the highly polished gold surfaces is very small in this temperature range. Free convection practically does not exist because of very low Rayleigh number, and lead-in losses also are very small in accordance with Ref. (11).

To the best knowledge of the author it is the first time that a correction for a temperature drop in the walls was also not necessary. Due to the proper mounting of the thermometers, the true surface temperature existing at the regions outside the ranges of unhomogeneous heat flow in the wall is measured. This was proved in (1 and 12). In (12) it was also shown that the disturbances of the heat flow by the sensing elements in the walls does influence the geometric constant by only a negligibly small amount.

The data in Figure 9 scatter by .2% around the smooth curve. This scatter possibly results from the fact that after each pressure change not enough time was allowed to reestablish thermodynamic equilibrium. This was proved by measuring the time needed by the gas to reach its original temperature after a pressure change. The instrument itself was kept at a constant temperature. The time observed was approximately equal to the time normally allowed between the readings taken at different pressures. Providing more time resulted in a lesser scatter as demonstrated for the 30°C isotherm of Figure 9.

3B2 Measurements of Dielectric Constant, Thermal Conductivity and p-v-T Data of Nitrogen at Three Constant Densities in a Temperature Range 0-100°C. And Evaluation of Susceptibility, Polarizability and Index of Refraction.

The instrument was evacuated and pressurized several times with high purity nitrogen gas and finally sealed hermetically after enough mass had entered to establish a certain pressure at ambient temperature. After equilibrium was established pressure and temperature were recorded and the capacitance measured. Power was then provided for the heater coil in order to establish a steady state temperature difference for the measurements of thermal conductivity. This procedure was repeated stepwise under increasing temperatures up to 100°C and then under decreasing temperatures until 0°C was reached. Finally the measurements taken

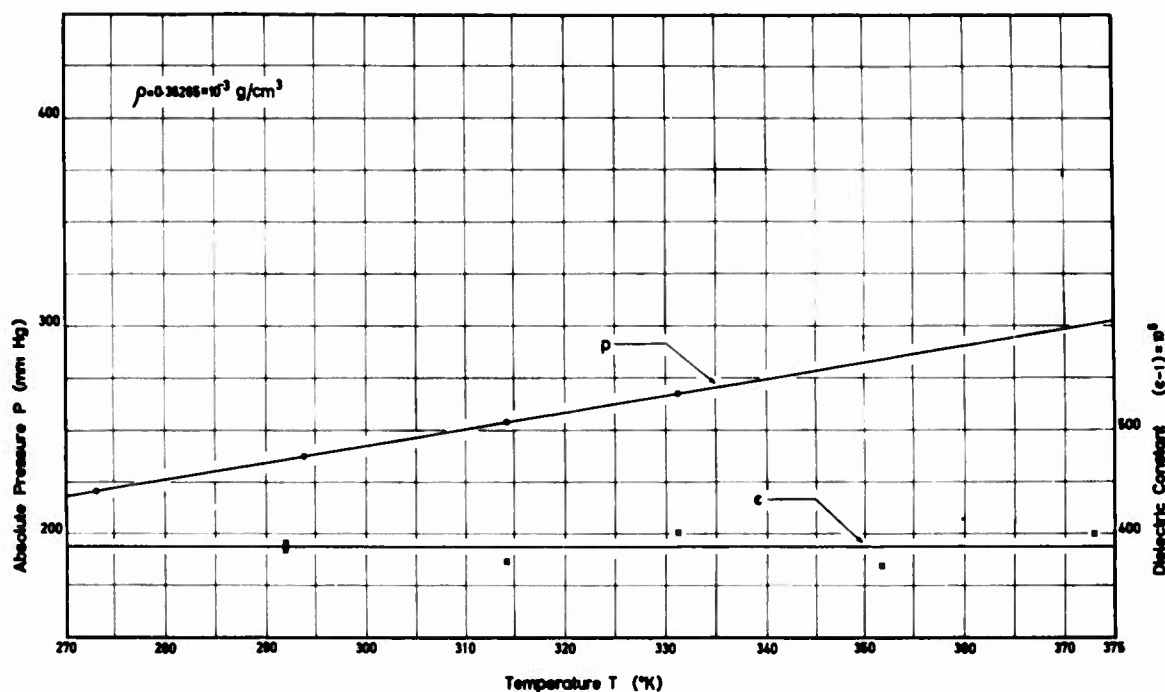


Fig.10 Pressure & Dielectric Constant Of Nitrogen Vs. Temperature At Constant Density

at ambient conditions were repeated. Identical results of the readings especially in respect to pressure gave assurance that the system had been sealed perfectly. All measurements therefore were taken at constant mass conditions. The valve was then opened and the instrument was charged with an additional mass of nitrogen and a second set of data taken in a similar way as demonstrated above.

The pressures observed were corrected for the volume change of the instrument due to thermal expansion. Therefore the curves given in Figure 10, 11, and 12 refer to constant density conditions. The density values were calculated by means of thermodynamic relationships. Normally it is anticipated to determine the mass introduced into the system before and/or after the test.

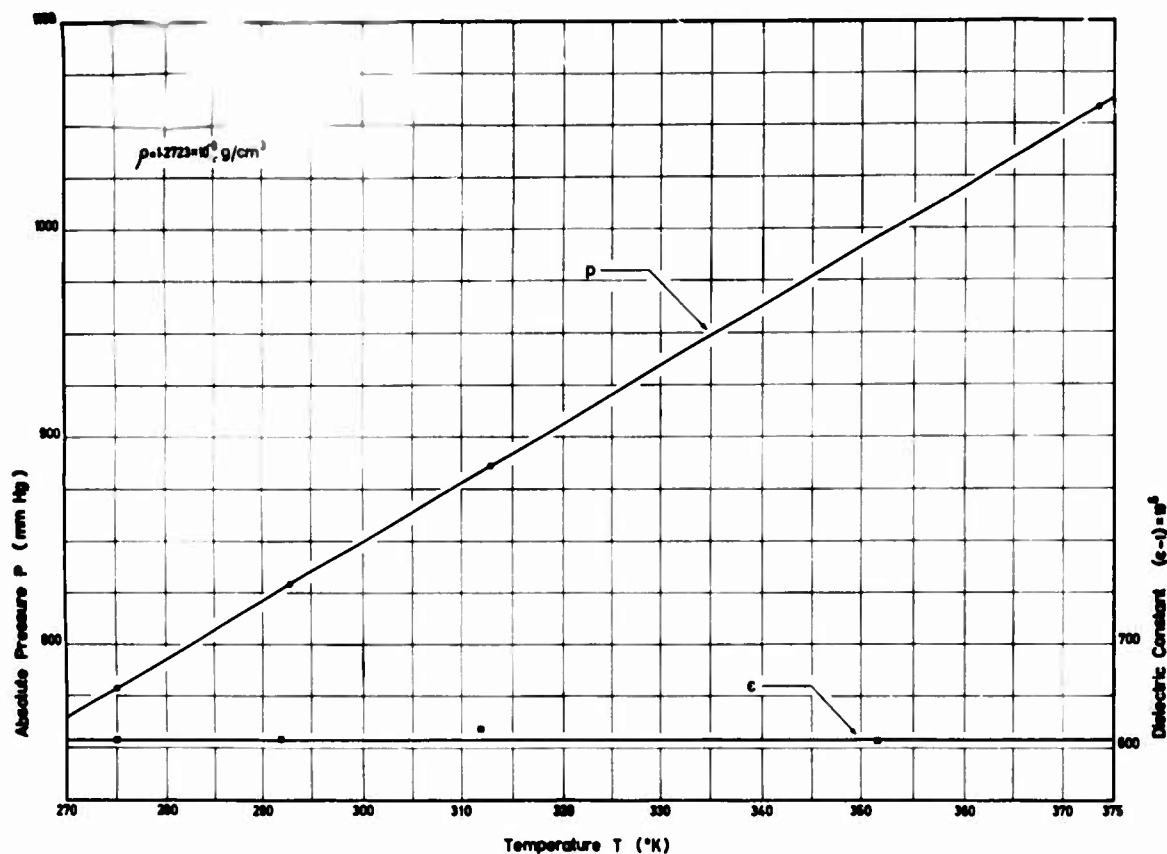


Fig.11 Pressure & Dielectric Constant Of Nitrogen Vs. Temperature At Constant Density

The pressure curves appear to be linear in the graphs but the data demonstrate for each plot a different curvature which will be used later and when more measurements have been completed to evaluate compressibility factors and virial coefficients. The same figures show also the measurements of the dielectric constant. These values remain practically constant with temperature as was expected. The scatter of the values is very small and mainly caused by small deviation from thermodynamic equilibrium conditions; i.e., the temperatures of the hot and cold bodies were not identical. Temperature differences of the order of one hundredth of a degree will influence the readings of ϵ by one thousandth of one per cent. For the hot body warmer than the cold body ϵ apparently will be higher.

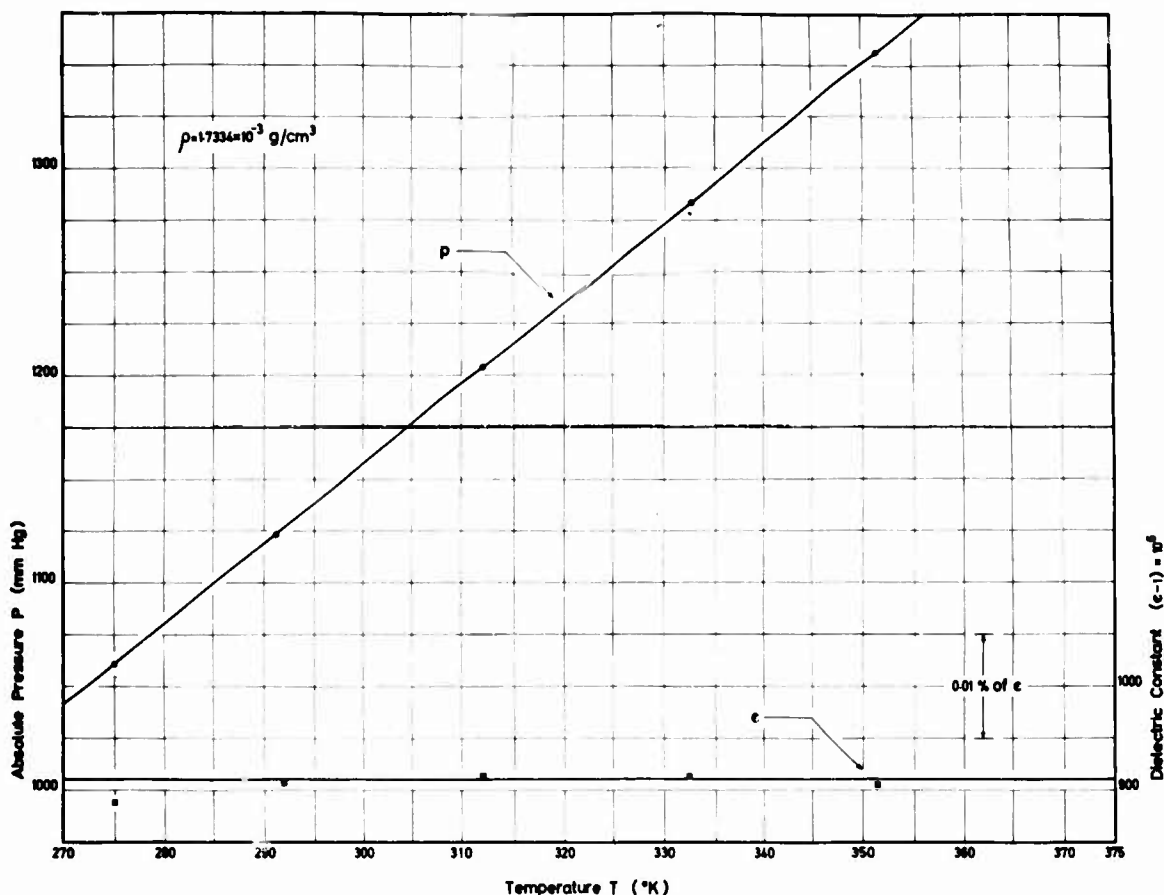


Fig.12 Pressure & Dielectric Constant Of Nitrogen Vs. Temperature At Constant Density

A similarly small temperature deviation of the temperature difference will cause errors in thermal conductivity of the order of 1 per cent. Which demonstrates again that those measurements are much more difficult to achieve and much more attention is necessary. The thermal conductivity data observed at the three different values of density are represented in Figure 13 by a single curve. Each point on the curve represents an averaged value of the data measured under the three density conditions and at the respective temperatures. The deviation between the readings were small and of the order of a few tenths of a per cent. The thermal conductivity of nitrogen therefore can be reported as

pressure independent in the small pressure range of the measurements and within the accuracy of the determination. The values agree very well with NBS data (13) but are approximately one per cent higher than recommended by TPRC (14).

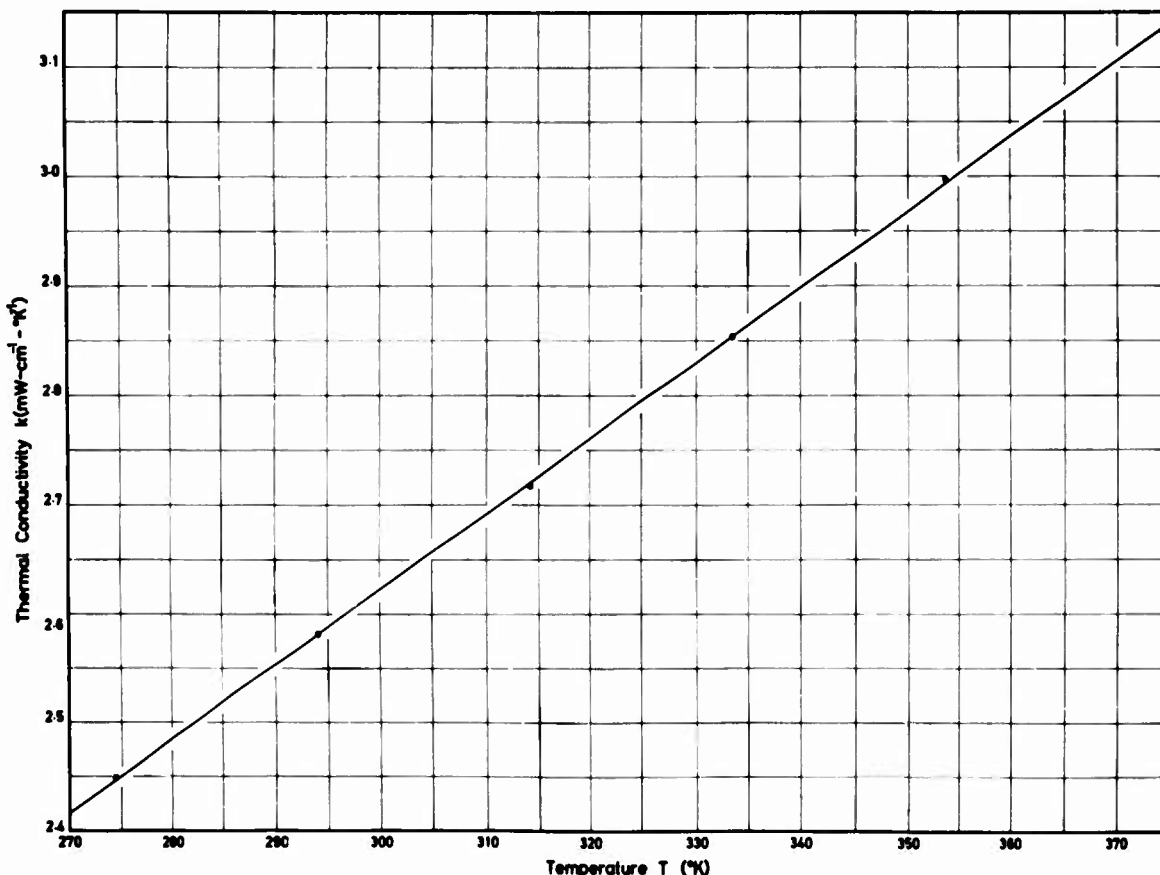


Fig. 13 Thermal Conductivity Of Nitrogen At Pressures Of 0.2 To 14 Atm. V.S. Temperature

From the values of dielectric constant and density the specific susceptibility, the polarizability and index of refraction can be computed.

The constant density measurements of p and ϵ taken close to atmospheric conditions as shown in Figure 11 resulted in a value of susceptibility

$$\epsilon - 1 = 605 \times 10^{-6}$$

Applying the relationship

$$\frac{\epsilon - 1}{\epsilon + 2} \frac{M}{\rho} = \frac{4\pi}{3} N_L \alpha \quad \text{allows} \quad (14)$$

evaluation of the polarizability of nitrogen. With $M = 28$ the molecular weight of nitrogen $\rho = 1.2723 \text{ kg/m}^3$ the density and $N_L = 2.68719 \times 10^{25}/\text{m}^3$ Loschmidt's number one obtains

$$\alpha = 17.58 \times 10^{-25} \text{ cm}^3$$

which is practically identical to the value of $17.6 \times 10^{-25} \text{ cm}^3$ given in literature.

The pressure observed under constant density conditions at 20°C was 829.4 mm Hg. Reducing the value for comparison reasons to 760 mm Hg yields

$$(\epsilon)_{20^\circ\text{C}, 760} = 1.000554$$

which deviates only by 6 ppm from the value given in NBS circular 537. According to Maxwell

$$n^2 = \epsilon \quad (15)$$

and

$$(n)_{20, 760} = 1.000277$$

This value reduced for comparison reasons to 0°C yields

$$(n)_{0, 760} = 1.000297$$

which again is in agreement with data listed in Smithsonian tables.

3B3 Determination of Thermal Conductivity and Dielectric Constant of Octafluorocyclobutane C_4F_8 - Vapor at 1 atm in the Range of Temperature 0-100°C

The vapor thermal conductivity of this substance is known only for a single point which is in large disagreement with data estimated later. The test material used for the investigation is of "Food-grade" purity and was furnished by DuPont under the trade name Freon C-138. The measurements were carried out under increasing and decreasing temperatures by holding the pressure constant at 760 Torr.

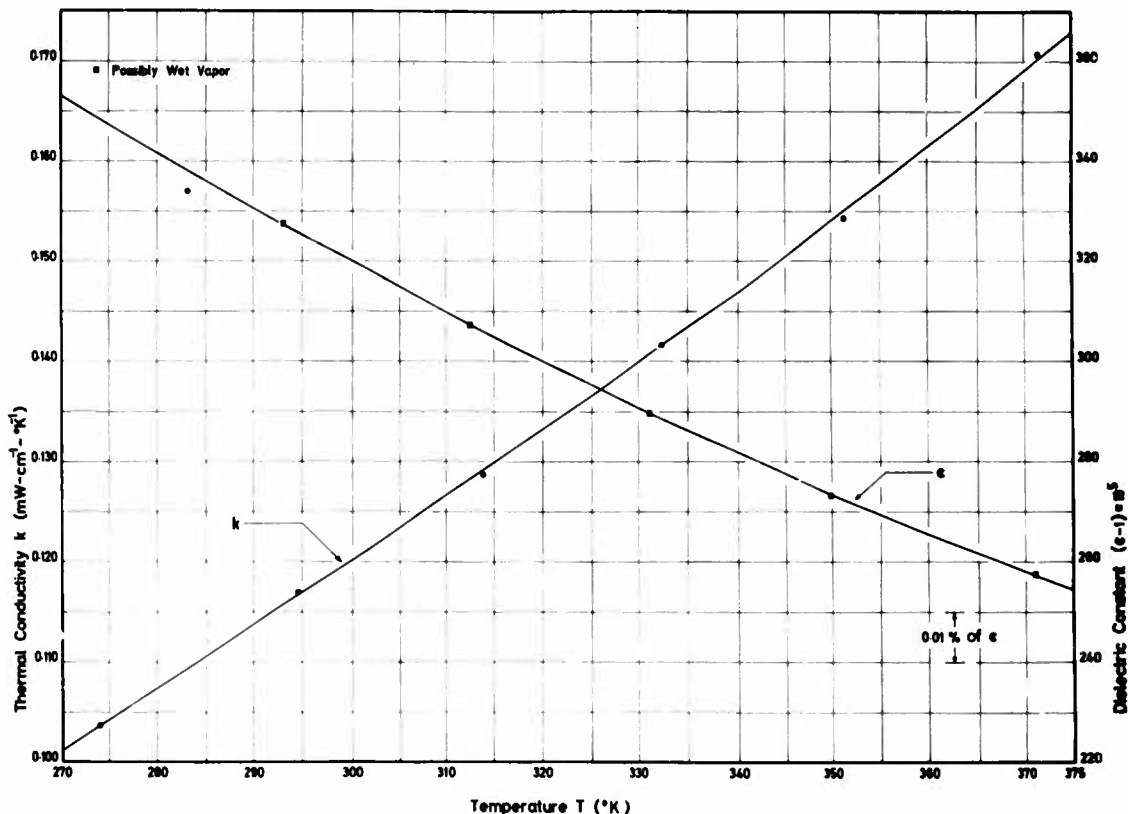


Fig. 14 Thermal Conductivity & Dielectric Constant Of C_4F_8 Vapor At 1 Atm. As A Function Of Temperature

The values of thermal conductivity and dielectric constant observed are given in Figure 14. The thermal conductivity curve agrees in curvature and slope with the estimated

i.e., low pressure conditions. But this was ruled out by remeasuring exactly the value which had been taken at low pressure and before the temperature had been raised. Since the literature data were regarded as correct and because the measurements of pressure cannot be faulty by the amount observed only the test fluid itself must have provided the conditions for the different readings.

The correctness of this reasoning easily could be checked by measuring other properties of the vapor known at certain conditions. The measurements of thermal conductivity and dielectric constant at these respective conditions yielded results which did not agree with literature data. In case of thermal conductivity the value measured was too high; the dielectric constant was too low. Both results indicate that not pure vapor but a mixture possibly with moist air was present. (This demonstrates one of the features of the multi-purpose instrument; namely that the measurements of more than one property allow checking if anomalies or deviations observed in one of the properties really exist.)

The methanol was delivered in a bottle not sealed hermetically and it must be assumed that moist air and/or other gases were absorbed by the fluid in time. Boiling of the sample at low temperature yielded better results of the vapor pressure which are still slightly higher than the table values represented by the solid curve in Figure 15 but they are in a very good agreement with newer data (17).

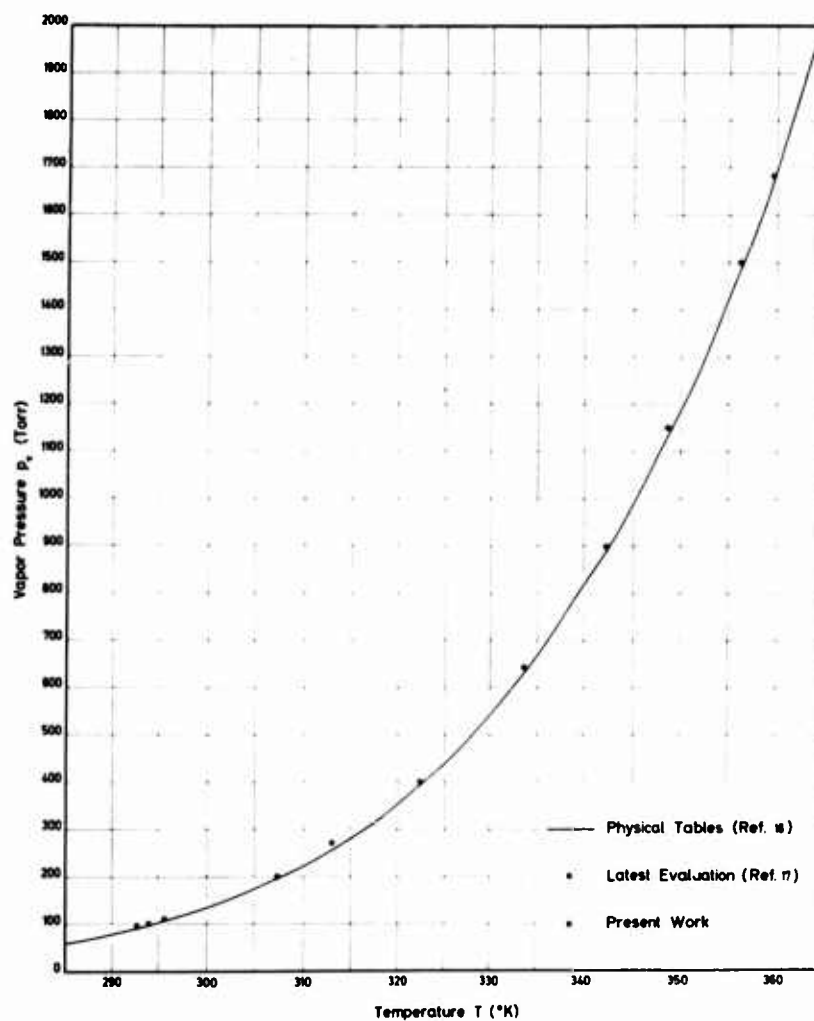


Fig. 15 Vapor Pressure Of Methanol

The solid curve is represented in accordance with (16) by an equation based on four references.

$$\log p = - \frac{0.05223a}{T[^\circ\text{K}]} + b \quad (16)$$

where the constants are

$$a = 38.324$$

$$b = 8.8017$$

The data of (17) are based on more information (thirty two references) which resulted in

$$\log p = - \frac{a}{c + t[^\circ\text{C}]} + b \quad (17)$$

when

$$a = 1474.08$$

$$b = 7.89750$$

$$c = 229.13$$

3B5 Determination of Thermal Conductivity, Dielectric Constant, Electrical Conductivity and Dissipation Factor of C₄F₈ Liquid at 15 atm in the Temperature Range 0-80°C.

The instrument first was filled with pure vapor under vacuum and then connected by means of a valve to a reservoir containing the test substance under its vapor pressure--which forced the liquid to enter the system. The decrease of weight of the reservoir was observed and when the conditions of complete charging was assured the liquid in the reservoir was pressurized further to 15 atm with nitrogen. All the measurements were carried out under constant pressure at 15 atm assuring that boiling could not occur at the highest test temperature.

The reservoir is connected with the cell by high pressure tubing of small inside bore and of such a length that the liquid within the instrument will not be contaminated by diffusion of N₂ even in a very long time.

The observations of the properties were carried out by measuring heat input ΔT and/or capacitance and computing the properties with the respective equations and the geometric

constant corrected for pressure and temperature. The results are given in Figure 16. It can be seen that ϵ and k decrease with temperature but the curves have opposite curvature.

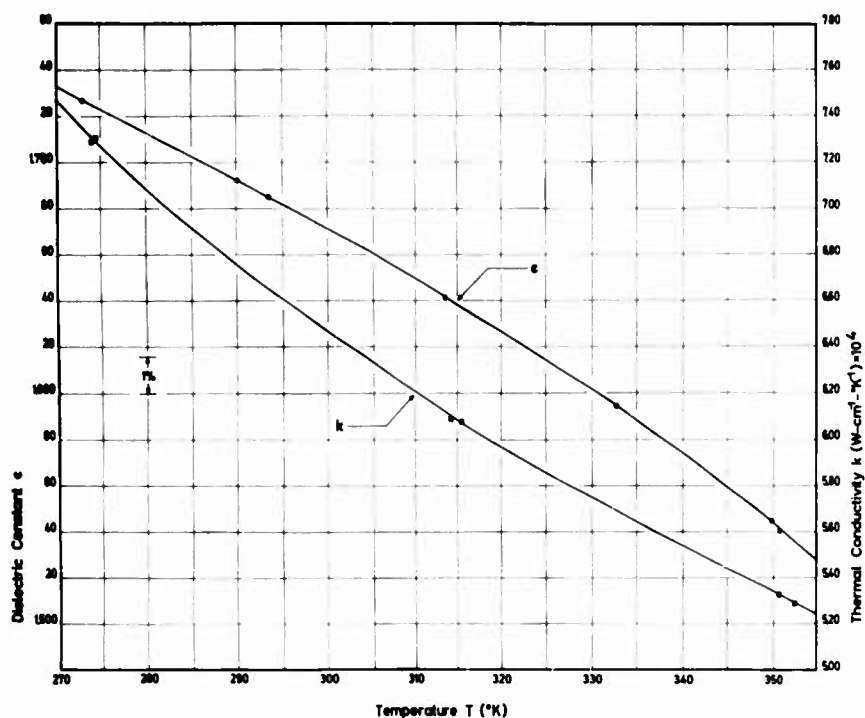


Fig. 16 Thermal Conductivity & Dielectric Constant Of C_2F_6 Liquid At 15 Atm.

The thermal conductivity data are in very good agreement with values observed at lower pressures and listed in (18,19). The single observation by (18) is identical to the data of the curve at the respective temperature. The values of (19) are a few tenths of a per cent higher than the present data.

Measuring the capacitance necessitates balancing the bridge for dissipation. The dissipation factor is observed by direct read-out of the bridge.

The conductance bridge connected by means of the master-switch to the system measured directly the conductance

$$G = \lambda \frac{A}{L} = \lambda B$$

From this the d.c. electrical conductivity λ is obtained.

Dissipation (observed at 1 kHz) and electrical conductivity are of very low value as can be seen in Figure 17. Dissipation and electrical conductivity remain practically constant in the low temperature range.

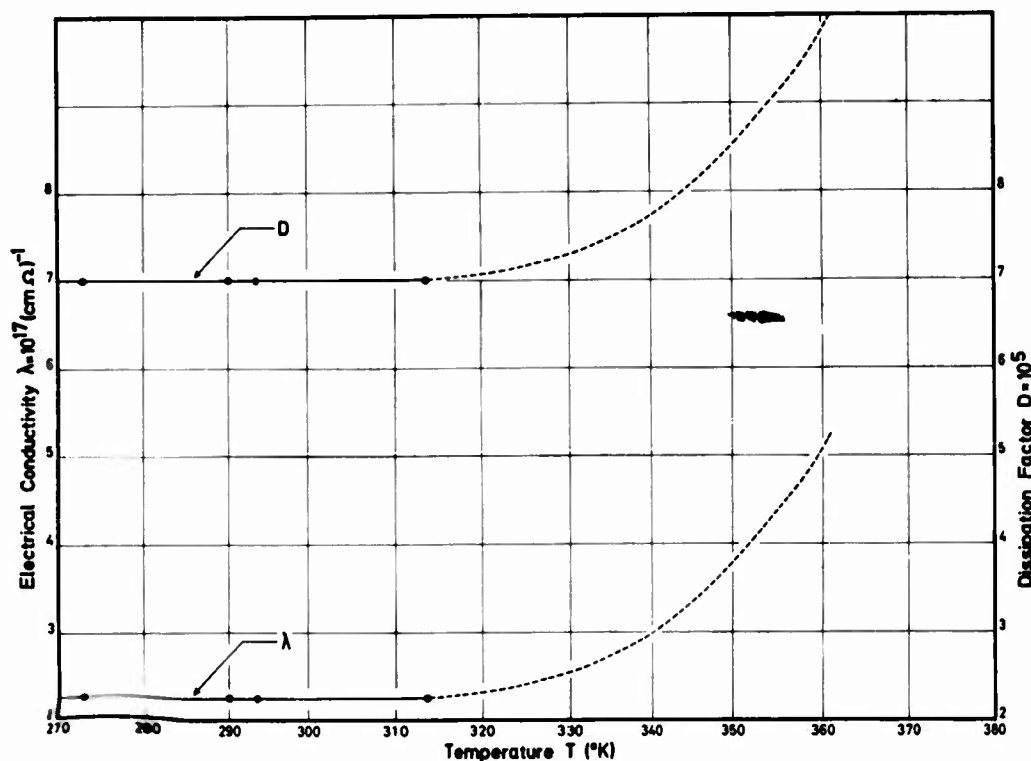


Fig.17 Electrical Conductivity And Dissipation Factor Of C_4F_8 At 15 Atm. V.S. Temperature

At temperatures above $40^\circ C$ measurements were difficult to obtain for reasons of fluctuations in the readings. The values of the properties increase as indicated by the dotted lines. These lines represent readings averaged over the range of fluctuation and must be regarded as inaccurate to a certain degree.

The unstable measurements could be a result of impurities introduced into the liquid. The impurities might be minute residues of previous test samples which had remained on the walls. They became dissolved by the Freon in time and the

particles started to move under the influence of the electrical field at higher temperature when the viscosity became low enough.

3B6 Determination of Thermal Expansion Coefficient of Liquid Benzene at 1 atm at Various Temperatures

The instrument was filled completely with the liquid in the same way as described above. The cell was then prepared for the measurements simply by closing the valve at the reservoir. The liquid within the cell is now only connected with a pressure balance arrangement. The free piston properly loaded maintains the pressure in the test fluid. Measuring the motion of the piston when the temperature of the instrument is changed by a value of ΔT allows evaluation of the thermal expansion coefficient from the volume displaced and the volume of the instrument V_L at the respective pressure and temperature.

The measurements were carried out at several temperature levels.

Since the motion of the piston is limited to approximately 2 cm and because the volume displaced is rather small it is necessary to bleed some of the liquid back into the reservoir (at a slightly lower pressure) in order to bring and hold the piston in proper starting position. Obviously the smaller the amount of liquid released the smaller the temperature increments between the measurements.

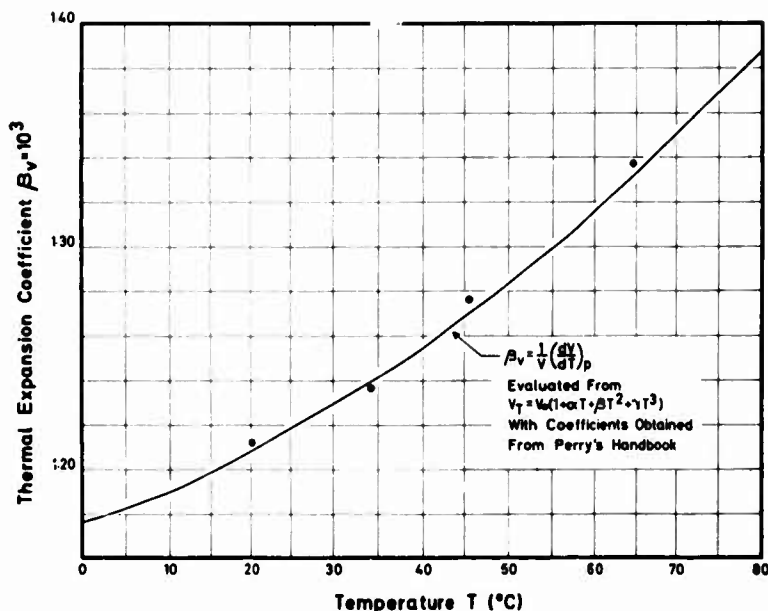


Fig.18 Thermal Expansion Coefficient Of C_6H_6 V.S. Temperature

The values measured at different temperatures are given in Figure 18. The solid curve represents data of physical tables. The measured points scatter around this curve

by a few tenths of a per cent.

4. Unsteady State Experiments. A Technique for Precise Determination of Heat Transfer in the Absence of Temperature Measurements and its Extended Application to Properties

Under section 2 of this paper it was pointed out that the multi-purpose instrument can also be used under conditions other than steady state. In this paper only the procedure for the unsteady observation of one property is included.

The procedures followed to determine thermal diffusivity (and conductivity) represents a novelty in measuring thermal properties and heat flow (i.e., heat transfer) insofar as the observations are carried out in the absence of temperature measurements. But by very sensitive observation of other quantities depending on true surface conditions which otherwise can be determined inaccurately or indirectly only.

A short description of the technique is included for the sake of demonstration. A detailed analysis will be given elsewhere (20).

The technique easily can be followed as demonstrated in Figures 19, 20, 21 and 22. Figure 19 shows schematically a

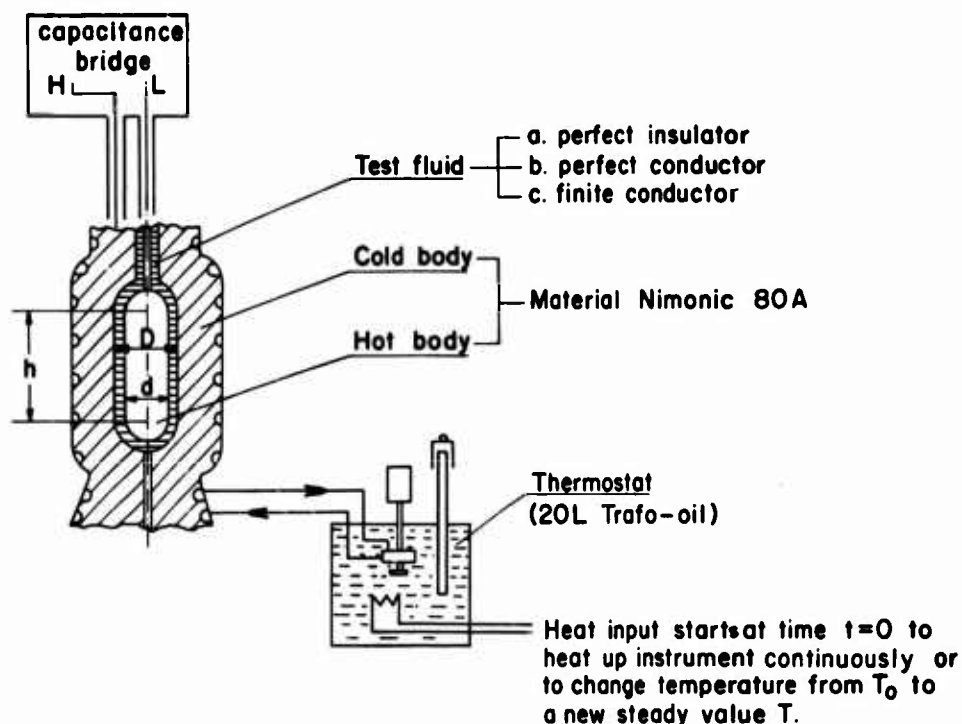


Fig. 19 Multipurpose instrument used in transient conditions to determine thermal diffusivity of test fluids

cross-section through the multi-purpose instrument. Hot body and cold body are properly connected to the capacitance bridge. The cold body originally is kept at a constant temperature T_0 by means of the thermostat. At time $t = 0$ the thermostat begins to heat (or cool) the cold body continuously or to a new steady value T . The test fluid enclosed in the gap between hot and cold body is considered to be:

- a. a perfect insulator
- b. a perfect conductor
- c. a finite conductor.

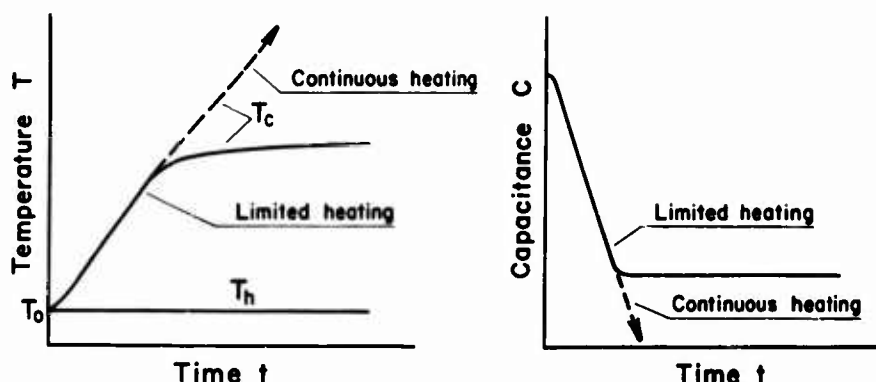


Fig.20 Schematics of $T=f(t)$ for hot and cold bodies and $C=f(T,t)$ for case a, test fluid is a perfect insulator

The heat input to the cold body obviously never will be noticed by the hot body when the test fluid is a perfect insulator. For a perfect conductor it will change its temperature simultaneously with the cold body. In case of a real substance the hot body will follow after a certain time lag the temperature change of the cold body.

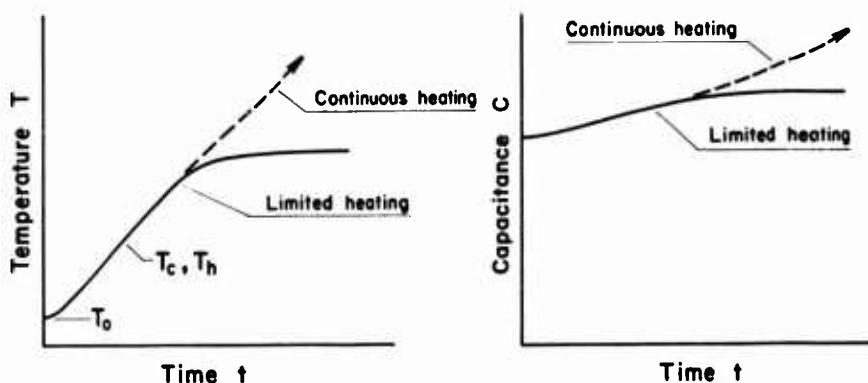


Fig.21 Schematics of $T=f(t)$ for hot and cold bodies and $C=f(T,t)$ for case b, test fluid is a perfect conductor

The temperature change experienced by the two bodies under constant or limited heating are given schematically in

Figure 20, 21 and 22. (Under cooling the effects will be similar.)

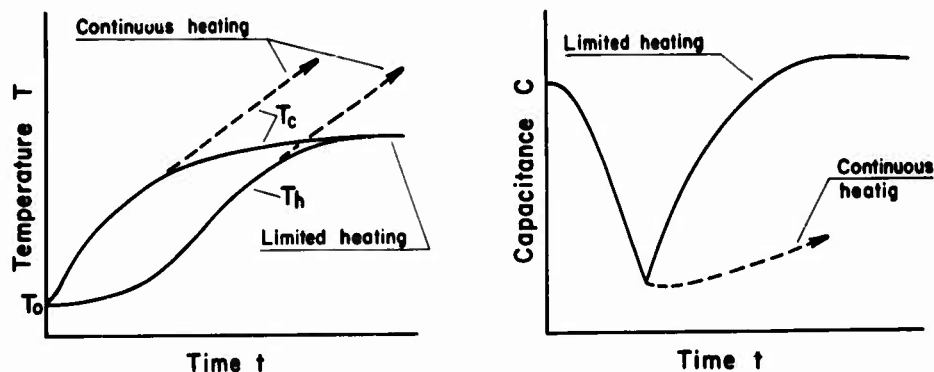


Fig.22 Schematics of $T = f(t)$ for hot and cold bodies and $C = (T, t)$ for case c, test fluid is a finite conductor

The temperature change within each of the two bodies will be uniform (therefore identical the change of surface temperature) for sufficiently small Bi-numbers. The poor heat transfer of the thermostat fluid guarantees those conditions. The dimensions of the bodies change therefore with temperature only due to thermal expansion. (Changes due to stress can be neglected.) Eq. (1) demonstrates that the change of dimension will cause a change of capacitance. These capacitance changes are given schematically also in Figures 20, 21 and 22 for the respective test fluids.

4.1 Determination of Thermal Transport Properties of Helium, Nitrogen, and Argon at 1 atm and Vacuum in a Small Temperature Range

For four different cases where the heat transfer between the two bodies varied from good to poor the method has been checked by using as test substances helium, nitrogen, argon

and vacuum. In the latter case heat transfer occurs only along the centering rod, by radiation and residual molecular conduction.

The results of the measurements are given in Figure 23. The change in capacitance observed verifies the change predicted in Figure 22. (The initial and final capacitance values of each curve divided by the C_0 -value of Figure 6 yield the dielectric constant of the test substance at the respective temperatures.) The maximum change in capacitance measured in helium is approximately one order of magnitude less than in the case of vacuum and new steady state conditions were established five times faster.

The maximum capacitance change observed in helium is of the order of .05%. This accounts for a similar change in gap width (nominal .5 mm) of 2.5×10^{-4} mm. If one assumes that the maximum change occurred at a time when the cold body only had changed in temperature then its excess temperature was approximately 1°C. (Free convection can be assumed negligibly small.)

This is the maximum temperature difference between cold and hot bodies. In accordance with Figure 22 it will decrease in time and finally become zero.

The sensitivity of the instrumentation is so high that change of capacitance of the order of .001% can be detected. This refers to a change in gap width of the order of 5×10^{-6} mm which in turn is identical to the change in diameter the cold body will experience when heated by 2×10^{-2} °C.

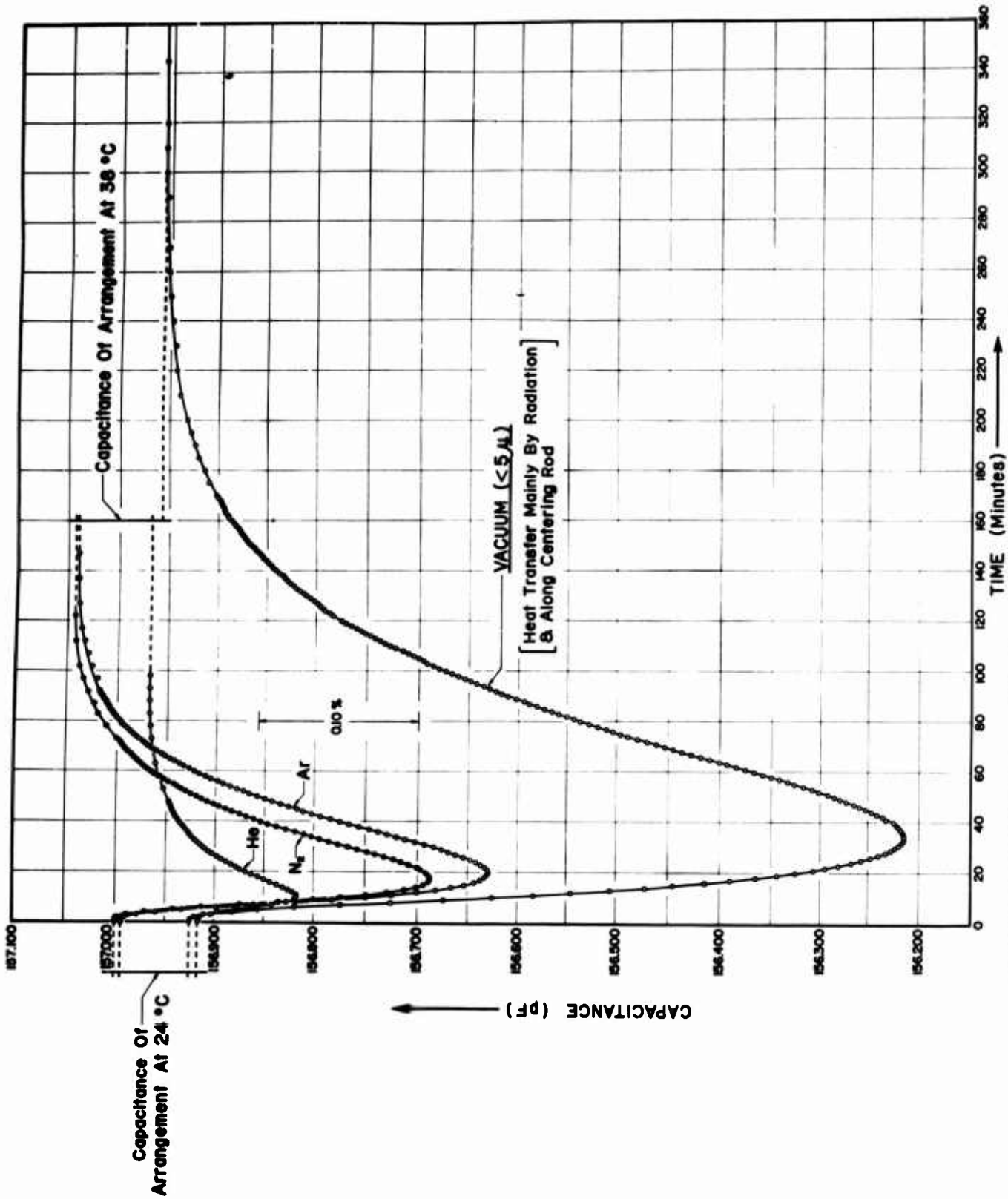


Fig. 23 Change Of Capacitance With Time Of MPI For The Measurement Of Thermal Diffusivity Of Fluids

The capacitance time curves observed are perfectly similar and can be made to form a single curve by proper reduction. Due to the complexity of the geometry an analysis is impossible. Therefore calibration becomes necessary and the measurements will be relative ones only.

Another system of simple geometry easily could be built which could be made of lesser heat capacity. Absolute and much faster measurements would then be possible.

The measurements by this technique* utilize electrical observations directly. This demonstrates the advantage of using it for remote control measurements of heat transfer and properties in many instances. Since diffusivity, thermal conductivity and dielectric constant are observed simultaneously the measurements also could be used for quality control and/or to determine the test substance in cases in which the sample is not known before hand.

The analysis of the technique and more detailed information of its use are outside the scope of this report--and will be given in another paper as already stated above.

5. Summary and Future Program

The measurements of the various properties carried out so far have indicated that the data observed compare favorably with literature values of separately measured properties. It is safe to assume that the determination of the properties of

*Patent disclosed by W. Leidenfrost.

the instrument and instrument material in all cases did aid the research on test fluids. In some of the cases it was demonstrated that the capability to measure more than one property under perfectly identical conditions is of advantage in determining other properties more reliably. It is hoped that the statements hold true in respect to the other properties which also can be determined with the instrument. This will be investigated shortly. Neglecting initial costs for the more complex and more involved experimental total set-up it also seems to be reasonable to state that measurements with a multi-purpose instrument will produce properties more cheaply.

For the future it is planned to determine properties of gases of different molecular structure and over wider ranges of pressure and temperature for theoretical studies. Selected liquids will be tested in respect to their heat transfer and electrical qualities in order to establish their suitability as coolants of powerful compact electronic devices. It is furthermore anticipated to develop instruments applying the transient technique described under section 4.

6. Acknowledgement

The work described in this paper was sponsored by the United States Air Force Office of Scientific Research, Office of Aerospace Research under Contract no. 49(638)-1574. The funds granted made possible the whole investigation and is gratefully acknowledged. The author also wishes to express

his sincere gratitude to Dr. A. Max former director of RCA Engineering Research and now with Purdue University's extension center in Indianapolis who performed the difficult task of lining the instrument with the thick layer of gold after the year long effort of expert companies had failed. Without Dr. Max's selflessly given efforts and help the gold possibly never could have been bonded properly--and the instrument could not have been made functioning under its original design conditions at least within the time interval in this contract. The author also wishes to extend his gratitude to Mr. G. Janicek a graduate assistant for helping to establish the laboratory and carry out the measurements and to our technician Mr. G. Urbanus who performed the task of fabricating delicate fine mechanics devices integrated in the measuring system and Mr. J. Taylor who helped to build up the laboratory to install the instrument and equipment. The author would like to thank also Mr. A. Clausen for the many helpful discussions and assistance in selecting the proper electrical instruments and the interference free wiring system, and Mr. W. Cameron who skillfully made the system work by painstaking attention to details. Mr. Clausen and Mr. Cameron in addition also deserve thanks for their always available help in troubleshooting.

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Unclassified

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Purdue University, School of Mechanical Engineering Lafayette, Indiana 47907		2a. REPORT SECURITY CLASSIFICATION Unclassified	
		2b. GROUP	
3. REPORT TITLE MEASURING 14 PROPERTIES WITH THE MULTI-PURPOSE INSTRUMENT AND A TEMPERATURE FREE METHOD OF MEASURING THERMAL TRANSPORT PROPERTIES			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Scientific Final			
5. AUTHOR(S) (First name, middle initial, last name) Wolfgang Leidenfrost			
6. REPORT DATE August 1969		7a. TOTAL NO. OF PAGES 63	7b. NO. OF REFS 20
8a. CONTRACT OR GRANT NO. AF 49(638)-1574		8a. ORIGINATOR'S REPORT NUMBER(S) WLRAF 6	
b. PROJECT NO. 9750-01		9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) AFOSR 69-2155TR	
c. 61102F 681308			
d.			
10. DISTRIBUTION STATEMENT This document has been approved for public release and sale; its distribution is unlimited			
11. SUPPLEMENTARY NOTES TECH, OTHER		12. SPONSORING MILITARY ACTIVITY AF Office of Scientific Research 1400 Wilson Boulevard (SREP) Arlington, Virginia 22209	
13. ABSTRACT The multi-purpose instrument originally designed to determine seven properties was extended in its capabilities to measure additional properties. The paper describes briefly the instrument and the operational procedures and brings the results of measurements of some of the properties, namely: four properties of the instrument or its wall material, thermal conductivity of He, N ₂ and C ₄ F ₈ (in vapor and liquid state), p-v-T data of N ₂ , dielectric constant of N ₂ , He, A, and C ₄ F ₈ - vapor and liquid, vapor pressure of methanol, thermal expansion coefficient of Benzene, index of refraction and polarizability of N ₂ . A new unsteady method is introduced for the determination of heat transfer and thermal transport properties without measuring temperature and its technique is demonstrated by heat transport observations in He, A, N ₂ and vacuum. Whenever possible comparison with reference data were made. The agreement is in practically all cases excellent. ()			

~~Unclassified~~

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Multi-Purpose Instrument						
Geometric Constant						
Nimonic 80A						
Lin. Therm. Expansion Coefficient						
Pressure Coefficient						
Helium						
Thermal Conductivity						
Nitrogen						
Dielectric Constant						
Index of Refraction						
Polarizability						
Susceptibility						
P-v-T Properties						
Octafluorocyclobutane						
Electrical Conductivity						
Dissipation Factor						
Methanol						
Vapor Pressure						
Benzene						
Volume Thermal Expansion Coefficient						
Argon						
Vacuum						
Heat transport						
Thermal Diffusivity						

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