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# NBS TECHNICAL NOTE 765

## Low Temperature Thermometry: Interim Report

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# Low Temperature Thermometry: Interim Report

George Cataland and Harmon H. Plumb

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F O R E W O R D

In the interest of informing the public on program developments and technical advances within the Division, we are increasing our publications activities, beyond the customary formal publication of complete pieces of work, through the media of NBS Technical Notes and NBS Reports. The former provide the means to deal with the subject matter at greater depth than normally tolerated by today's journal editors and the latter afford an opportunity to describe informally the progress toward current goals where the work is incomplete.

This is a progress report. The work is incomplete and is continuing. Results and conclusions are not necessarily those that will be included in the final, formal publications. The period covered herein is the calendar year 1972.

H. H. Plumb, Chief  
Temperature Section  
National Bureau of Standards

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George Cataland and Harmon H. Plumb

Earlier acoustical thermometer 'speed of sound' isotherms are compared with recent measurements. Where systematic deviations had appeared in "smoothing" through acoustically derived temperatures via germanium thermometers, a possible solution has emerged; more precise temperature control of the acoustical thermometer is desirable and has been realized. A cursory examination of the applicability of the Helmholtz-Kirchhoff equation was made with a modified sonic chamber. Measurements with the modified instrument have shown no changes in the experimental results.

The maintenance of the NBS P2-20 (1965) scale is described. A new calibration apparatus was constructed to accommodate more germanium thermometers thus reducing the calibration cost per thermometer during a calibration run.

Key words: Acoustical thermometer; calibrator; germanium thermometers; heat conduction; NBS P2-20 (1965) scale; 'speed of sound' isotherms; viscosity.

### 1. Introduction

A number of years ago the National Bureau of Standards Provisional Temperature Scale 2-20 (1965) was established; it was based upon acoustical thermometry and described in the literature [1]. Since that time the International Practical Temperature Scale of 1968 [2] was defined down to 13.8 kelvins and the NBS P2-20 (1965) has been used extensively not only within the United States but also by external research laboratories. The scale's widespread use has certainly been enhanced by the existence of a calibration facility [3] that could meet the requested calibration needs.

Although the low temperature scale met an immediate need for temperature determinations in the 2 to 20 K range, it was recognized that there were deficiencies in the acoustical thermometry from which the temperature determinations were derived and it was hoped that improvements could be made in the measurements and techniques of acoustical thermometry. This report will describe most of the continuing work, describe some of the problems that have been encountered, and indicate what may be the remaining program of measurements.

### 2. Speed of Sound in a Gas and Absolute Temperature

For clarity it is desirable to repeat some of the discussion that was previously presented [1].

When sound is propagated through a gas, if the frequency of the sound is not too high relative to the mean collision frequency of the gas molecules, the propagation is nearly adiabatic, and speed of sound [4,5] is

$$W = \sqrt{E/\rho} = \sqrt{\frac{-v^2}{M}} \left( \frac{\partial p}{\partial v} \right)_S = \sqrt{-\frac{C_p v^2}{C_v M}} \left( \frac{\partial p}{\partial v} \right)_T \quad (1)$$

where  $\rho$  is the density of the medium of molecular weight  $M$  and  $E$  is the adiabatic bulk modulus defined by the relation

$$E = -v \left( \frac{\partial p}{\partial v} \right)_S = -\frac{C_p}{C_v} v \left( \frac{\partial p}{\partial v} \right)_T. \quad (2)$$

The symbols  $v$  and  $p$  refer to the volume per mole and pressure of the medium, and the partial derivative is at constant entropy or temperature, as indicated;  $C_p$  and  $C_v$  are specific heats at constant pressure and volume, respectively. If the conducting medium were an ideal gas, its equation of state would be expressed by

$$pv = RT \quad (3)$$

and there would result

$$\left(\frac{\partial p}{\partial v}\right)_T = -\frac{RT}{v^2} \quad (4)$$

Additionally, the ratio of specific heats would be that of an ideal gas,

$$\left(\frac{\frac{C}{p}}{C_v}\right)_{\text{ideal gas}} = 5/3$$

(if the gas is assumed to be monatomic), and eq. (1) becomes

$$W_o^2 = \frac{5}{3} \frac{RT}{M}. \quad (5)$$

Equation 5 indicates the simple relationship between the absolute thermodynamic temperature  $T$  and the speed of sound in an ideal gas  $W$ . (The gas constant  $R$  and the molecular weight  $M$  are considered to be accurately known constants.)

In actuality an ideal gas does not exist, so eq. (5) must be modified for application to real gases. Of the two gases,  $^3\text{He}$  and  $^4\text{He}$ , which can be used for sound measurements at temperatures between 2 and 20 K,  $^4\text{He}$  has been selected for experimental measurements. Its equation of state can be assumed to be represented by the empirical relationship [6]

$$pv = RT\left(1 + \frac{B}{v} + \frac{C}{v^2} \dots\right) \quad (6)$$

where  $B$  and  $C$  are functions of the temperature called the second and third virial coefficients, respectively. If the quantity

$$\left(\frac{\partial p}{\partial v}\right)_T$$

is evaluated from eq. (6) and the ratio  $C_p/C_v$  is evaluated for the real gas, eq. (1) can be written

$$W^2 = \left(\frac{\frac{C}{p}}{C_v}\right)_{\text{ideal}} \frac{RT}{M} (1 + \alpha p + \beta p^2 + \dots) \quad (7)$$

where

$$\alpha = \frac{1}{RT} [2B + \frac{4T}{3} \frac{dB}{dT} + (4/15) T^2 \frac{d^2B}{dT^2}]. \quad (8)$$

$\beta$  is a term that includes higher orders of the virial coefficients  $B$  and  $C$ . From eqs. (5) and (7) we obtain

$$W^2 = W_o^2 (1 + \alpha p + \beta p^2 + \dots). \quad (9)$$

It is now apparent that, if one determines an isotherm of  $W^2$  as a function of pressure, the intercept  $W_o^2$  of the extrapolated (to  $p = 0$ ) isotherm readily affords a means of calculating the isotherm temperature on the absolute scale.  $W_o^2$  is the slope of the isotherm and there is no reason to assume that the slopes are not a smooth or continuous function of temperature.

In order to obtain values of the speed of sound as a function of pressure, one must determine the wavelength and the frequency. Several methods are possible but our work is based upon the creation of standing waves in a gas by means of an apparatus with a fixed frequency and a variable path. A quartz crystal, operating at its resonant frequency, radiates through  $^4\text{He}$  gas to a reflector surface which is parallel to the quartz crystal; radiation is reflected back through the gas to the quartz crystal. If the spacing between the crystal and the reflecting surface is an integral number of half-wavelengths the helium gas will be in resonance and a peak voltage will be measured across the quartz crystal. The reflecting surface is displaced through detected integral numbers of half-wavelengths and its displacement measured. Thus  $\lambda$  is determined, the frequency of the quartz crystal is measured and, consequently,  $W$  can be calculated. This value of  $W$  is, of

course, the phase speed corresponding to whatever mode exists in the tube of gas. It is supposed that this mode is sufficiently like the plane wave mode to allow identification of  $W$ , for our purpose, as the free-space value. The mode may not be pure but, if this is the case, its components are spaced so closely that the ultrasonic interferometer does not resolve them. The cavity that contains the  $^4\text{He}$  gas is a right circular cylinder with an X-cut quartz crystal covering one end of the cavity and the reflecting surface of a piston, which can slide freely within the cylinder, nearly fills the other end of the cylinder. Provision is made for the gas space to connect to an external gas ballast volume and manometer. The resonating gas volume is maintained at the cryogenic temperature of interest but displacements of the piston's reflecting surface are measured at room temperature. A fused quartz rod extends from the piston through temperature gradients of a Dewar to ambient conditions and reliance has been placed upon the small value of the coefficient of expansion for fused quartz. More extensive details of the instrument and its operation appear in *Metrologia* [1].

### 3. Speed of Sound Isotherms 9 K to 21 K

A number of the isotherms upon which the scale NBS P2-20 (1965) were based did not appear to have been adequately characterized by fitting a linear function to the isotherm data. This is seen in table 1 where the experimental  $W^2$  and  $p$  are listed for the 7 K, 8.1 K and 9 K isotherms. The  $\Delta$ 's for each isotherm indicate that there are systematic discrepancies between the actual data points and the assumed linear representation.

Table 1

T	$W^2(\text{m/s})^2$	P (Atmos)	$\Delta^a$	$\sigma^b$
9.0 K	30,980	.2017	5.0	
	30,818	.4076	- 3.0	
	30,665	.6068	- 6.9	
	30,524	.8096	2.8	
	30,380	1.0005	2.2	5.6
8.1 K	27,792	.13 <sup>b</sup> 3	5.1	
	27,642	.2680	- 4.4	
	27,466	.4380	- 2.0	
	27,287	.6090	- 2.3	
	27,120	.7740	3.5	4.8
7.0 K	23,988	.1326	9.5	
	23,770	.2680	-10.1	
	23,532	.4370	- 1.8	
	23,287	.6040	- 2.5	
	23,069	.7580	4.9	8.7

a. The columns under  $\Delta$  contain the difference between the experimental  $W^2$  at the indicated pressure and the  $W^2$  of the linear function determined by the computer least squares fitting.

b.  $\sigma$  is the standard deviation in the least squares fitting of an isotherm.

Additionally, continued experimental experience indicated that some of the deviations, and the standard deviation  $\sigma$  (3.4 is approximately equivalent to 1 mK), were larger than might be associated with the inherent limitations of the measurements and the apparatus. The most probable causes of the observed systematic deviations appeared to be: that the linear representation covered a larger range of pressure than was applicable (the experimental data were not properly represented by just the linear term of eq. 9); or that the lowest pressure points were inaccurate because of experimental difficulties which one encounters in attempting to conduct measurements at low gas densities; operational problems involving the control of the experimental parameters (germanium resistance thermometry, frequency control, thermal equilibrium) might be improved upon.

It appeared that the range of linear representation might be the most significant cause of data misrepresentation so experimentation was initiated to determine acoustical isotherms at each kelvin from 9 to 21 kelvins and for pressure ranges that extended up to about two atmospheres. By these measurements we hoped to extend the pressure range for each isotherm to such an extent that significant curvatures would appear at the higher pressures; this would then be a guide that would be informative in determining the proper value of pressure for truncating the upper limit of pressure. The reason for conducting the experimentation between 9 and 21 kelvins, as opposed to below 9 kelvins, was that meetings of the Comité Consultatif de Thermométrie were to take place in the immediate future and we were desirous of making a contribution to the selection of thermometry fixed points in the liquid hydrogen temperature range. (At about the same time that these new acoustical measurements were being made, effort was diverted to conducting liquid hydrogen vapor pressure measurements). Another reason for initiating the higher temperature measurements was that the slope of the acoustical isotherms passes through zero at about 13.5 K and there was some question about the adequacy of previously determined isotherms in the vicinity of this temperature.

After identifying the pressure range for which the isotherms (at nearly integral temperature intervals) could be represented linearly, isotherms at half kelvin values, between 9 and 21 K, and at the triple and normal boiling points of equilibrium liquid hydrogen were measured. The experimental data and appropriate statistical information are given in table 2 for the triple-point-of-hydrogen isotherm.

Table 2

T	$W^2$ (m/s) <sup>2</sup>	P (Atmos)	$\Delta$	$\sigma$
13.8057	47808.70	.3743	-.208	
	47811.76	.4416	.191	
	47813.07	.5216	-1.66	
	47818.75	.5820	1.63	
	47820.94	.6624	.641	
	47823.13	.7325	.055	
	47825.32	.8029	-.544	
	47829.25	.8806	.319	
	47831.44	.9542	-.405	0.96

It is to be noted that the deviations,  $\Delta$ , in table 2 are much more random than those of table 1. In addition, the standard deviation of the least squares fitting is much less than those of table 1. The standard deviations of the 13 and 14 kelvin isotherms that were reported earlier [1] were 5.4 and 8.3, respectively, and the  $\Delta$  deviations exhibited systematic departures from linearity. The pressure ranges of the previously reported 13 and 14 kelvin isotherms extended from 0.4 to 1.4 atmospheres - obviously, this was too great a pressure range.

When the new isotherms from 9 to 21 kelvins had been determined it was appropriate to check them for consistency and smoothness. This was done in two ways. It was assumed that the resistance of a germanium resistance thermometer was a smooth function of temperature and subsequently the resistor was calibrated. Values of resistance were associated with the values of temperature that had been calculated for the new isotherms and the resistance-temperature values of the thermometer were computer fitted to a polynomial of the form

$$\log R = \sum_{m=0}^n A_n (\log T)^n$$

The results are given in table 3.

Table 3

## Calibration of Germanium Thermometer 561

$T_{\text{acoustic}}$ (K)	$R_{\text{Ge}}$ ( $\Omega$ )	$T_{\text{acoustic fitted}}$ (K)	$\delta T$ (mK)	$\frac{dR}{dT}$ ( $\Omega/K$ )
8.9917	444.470	8.9914	-0.3	-133.2
9.4956	383.300	9.4964	0.8	-109.9
9.9951	333.239	9.9955	0.4	-91.4
10.4968	291.467	10.4954	-1.4	-76.3
10.8989	262.810	10.8983	-0.6	-66.2
11.4948	227.069	11.4958	0.8	-54.0
12.0127	201.432	12.0128	0.1	-45.0
12.4956	181.063	12.4967	1.1	-38.9
12.9593	164.277	12.9600	0.7	-33.7
13.4989	147.533	13.4985	-0.4	-28.7
13.8057	139.140	13.8047	-1.0	-26.2
14.0484	132.976	14.0482	-0.2	-24.4
14.5019	122.593	15.5004	-1.5	-21.5
14.9893	112.710	14.9900	0.7	-18.90
15.5002	103.664	15.5014	1.2	-16.56
16.0462	95.255	16.0449	-1.3	-14.45
16.5012	89.004	16.5014	0.2	-12.96
16.9676	83.281	16.9673	-0.3	-11.64
17.4980	77.430	17.5006	2.6	-10.34
18.0112	72.451	18.0090	-2.2	-9.27
18.4994	68.123	18.4999	0.5	-8.38
18.9407	64.578	18.9417	1.0	-7.68
19.5009	60.514	19.5002	-0.7	-6.90
20.0436	56.927	20.0472	3.6	-6.24
20.2840	56.512	20.2789	-5.1	-5.98
20.4998	54.194	20.5037	3.9	-5.75
21.0102	51.432	21.0056	-4.6	-5.27
22.8236	43.136	22.826	2.4	-3.94

$T_{\text{acoustic fitted}}$  are the values of temperature that would be obtained in a smooth relationship of  $R-T$  for the germanium thermometer.  $\delta T$  is the difference between  $T_{\text{acoustic fitted}}$  and  $T_{\text{acoustic}}$ . Although the  $\delta T$ 's show some systematic deviations, particularly at the lower temperatures, the most noteworthy point is the magnitude of the  $\delta T$ 's from 20 to 21 kelvins. Also, the values of  $\delta T$  at 17.5 and 18 K are significant. One can probably consider that the values of  $T_{\text{acoustic fitted}}$  may yield more consistent and better values of temperature than those associated with the temperature of individual isotherms but the approximately  $\pm 5$  mK for  $\delta T$  in the vicinity of 20 kelvins is distressing. It appears to indicate that errors may exist in the acoustical isotherms. Later in this report specific causes of a possible systematic error will be discussed.

A second method of smoothing the isotherms to detect possible systematic variations was achieved by fitting the isotherm slopes to expressions of temperature that were similar to experimentally observed dependencies of the second virial coefficient. The three expressions

$$\begin{aligned}f(T) &= a_1 + b_1/T + c_1/T^2 \\f(T) &= a_2 + b_2/\sqrt{T} + c_2/T \\f(T) &= a_3 + b_3 \ln T + c_3/T\end{aligned}$$

yielded residuals of the same sign (there was one exception).

Table 4

Temperatures and Slopes of the Speed of Sound Isotherms

T (K)	Slope <sup>a</sup>	Upper Pressure Limit (Atm)	Residual
9.4956	-14.892 (.16)	0.8	-.172
9.9951	-12.098 (.17)	1.0	.106
10.4968	-10.204 (.10)	0.8	-.276
10.9002	- 8.458 (.12)	0.9	-.204
11.4948	- 5.904 (.14)	0.8	.108
12.0127	- 4.117 (.13)	1.0	.130
12.4961	- 2.573 (.08)	0.9	.165
12.9596	- 1.238 (.09)	0.9	.164
13.4989	.149 (.09)	0.8	.117
13.8057	.952 (.04)	1.0	.157
14.0484	1.528 (.13)	0.9	.155
14.5019	2.356 (.05)	1.0	-.044
14.9893	3.540 (.10)	0.9	.110
15.5002	4.634 (.06)	1.0	.198
16.0473	5.259 (.08)	1.0	-.179
16.5012	6.158 (.21)	1.0	-.059
16.9676	6.870 (.09)	0.8	-.100
17.4980	8.178 (.09)	0.9	.404
18.0112	8.121 (.06)	0.8	-.383
18.4994	9.187 (.09)	0.9	.029
18.9407	9.888 (.06)	1.1	.170
19.5009	10.384 (.10)	1.0	-.006
20.0436	11.073 (.20)	0.9	.071
20.2840	10.850 (.19)	0.9	-.411
20.4998	11.589 (.10)	1.0	.101
21.0107	11.535 (.20)	0.6	-.472
22.8236	13.458 (.09)	1.0	-.186
27.1076	16.556 (.11)	1.0	.010
29.4130	17.832 (.09)	0.7	.082
33.9758	19.798 (.22)	0.8	.235

a) The standard deviations of the slopes are in parenthesis.

If the isotherm slopes in table 4 are smooth functions of temperature, then the values of the slopes (and residuals) at 17.5 and 18 kelvins and between 19.5 and 21 kelvins clearly indicate the presence of peculiarities. This substantially supports the suggested errors in acoustical isotherms that were noted in the germanium thermometer "smoothing" of temperatures.

The preceding discrepancies and indeterminacy have prevented us until now from

replacing or up-dating the NBS P2-20 (1965) scale, and presenting a liquid hydrogen vapor pressure scale, or values of  $^4\text{He}$  virial coefficients.

#### 4. Recent Acoustical Isotherm Measurements

A systematic error has been ascertained in recent acoustical thermometer experimentation. It was observed that not all data points lay on a straight line within the apparent reproducibility of the speed of sound-pressure measurements. Efforts to determine the cause of this discrepancy were concentrated in measurements at 19 K and 9 K and the cause of the data variability appears to be related to heat flow problems within the acoustical thermometer low-temperature chamber.

In order to increase the efficiency of acquiring experimental data certain set procedures have been followed. In general, the vacuum space of the acoustical thermometer was filled with some helium gas to enhance its cooling from room temperature down to the approximate temperature of measurements. Then the helium gas was evacuated by an oil diffusion pump and vacua of  $1 \times 10^{-7}$  mm of Hg were commonly achieved and maintained during the acoustical measurements. The vacuum gauge was in the pumping line at room temperature and the pumping tube within the Dewar system was of fused quartz—it was desirable that it be of the same material that was employed in other lines (the displacement measuring tube and rod that are used in connection with the piston-displacement measurements). Customarily, heat was applied to the acoustical thermometer through a heater coil that was wound around the copper top of the low-temperature portion of the instrument. A copper radiation shield surrounded the apparatus. The instrument was brought into temperature control, as indicated by several germanium resistors that were mounted in the cylinder housing which contained the helium gas, the quartz crystal and the reflecting surface. A temperature controller supplied the required heat for the apparatus and the germanium thermometers generally indicated a temperature control and temperature differential that were within one millikelvin. In the customary measurement techniques the acoustical thermometer would be at low temperatures for weeks or even months and experimental data points would be measured in a systematic manner. That is, measurements would be taken at say 19 K, the apparatus would be permitted to cool to 18 K, and measurements would be taken 18 K and so forth. At particular times the acoustical thermometer was heated to higher temperatures, e.g., 27 K, considerable outgassing of helium gas would be observed, and the instrument's temperature would again be lowered to obtain additional measurements at the regular isotherm temperatures, e.g., 19 K. After some time it was noted that the isotherm data points which were achieved after a 27 K outgassing were slightly below the "customarily" measured isotherms. The difference appeared to be greater than the expected reproducibility of the isotherm. This is supported by the data presented in figure 1 and figure 2 for the 9 K and 19 K isotherms, respectively. It was decided that, in spite of the indications of the vacuum gauge, problems of heat flow and temperature gradients existed with the acoustical thermometer chamber.

Subsequently, the radiation shielding of the instrument was modified. The innermost shield of figure 3 was incorporated in the apparatus, a heater coil was wound around the outer radiation shield and additional germanium resistors (for monitoring and controlling the outer radiation shield temperatures) were installed. The apparatus was then operated so that sufficient heating power was applied to the outer shield to cause the outer shield temperature to be a few millikelvins warmer than the cylinder of the acoustical thermometer. After some experience had been acquired in taking measurements under these modified conditions, it was ascertained that the germanium resistors which were mounted within the acoustical thermometer walls indicated stabilities within a few tenths of a millikelvin. Also the acoustical resonance peaks were sharper and the instrument could be operated at somewhat lower helium gas pressures than had been possible previously. The vertical posts that are attached to the lower portion of the inner radiation shield were used for additional thermal anchoring of the inner germanium thermometers' current leads. The use of these additional thermal anchors does not seem to noticeably affect the thermometers' stability or the instrument's operation.

When the acoustical thermometer was operated under these new conditions of thermal control, we measured isotherm data points, ("two shields" in figure 1 and figure 2) that are in close agreement with those earlier data which were acquired after the thermometer

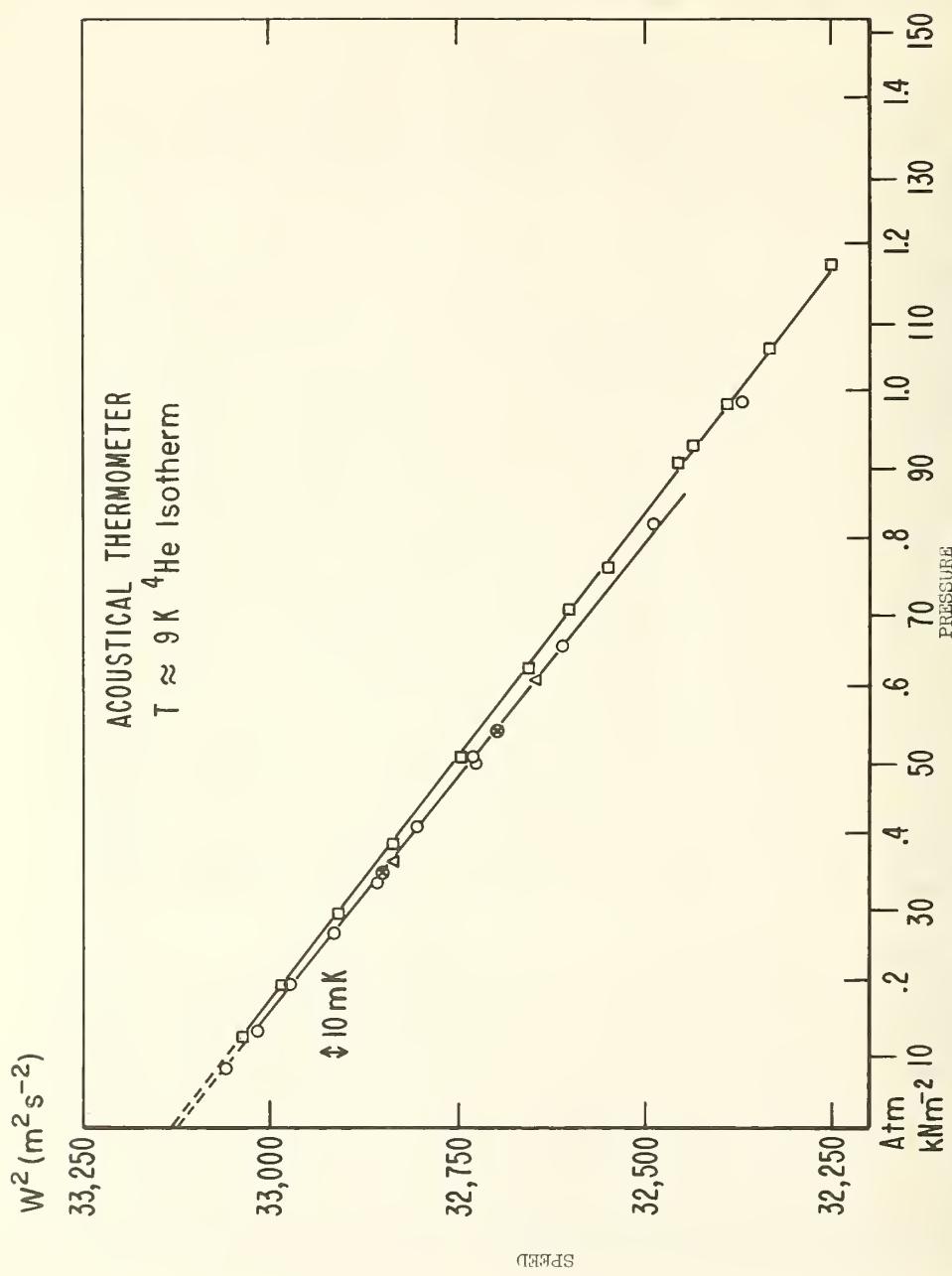


Figure 1.  $8.990\text{ K}$  Isotherm -  $W^2$  as a function of pressure. A comparison between data derived by different experimental procedures: preliminary measurements (single shield-not outgassed)  $\square$  1968. Recent (1972) with two shields  $\circ$ . Recent (1972) with single shield-outgassed prior to measurements  $\otimes$ . Sonic cavity diameter doubled to investigate if H-K corrections applicable  $\blacktriangle$ .

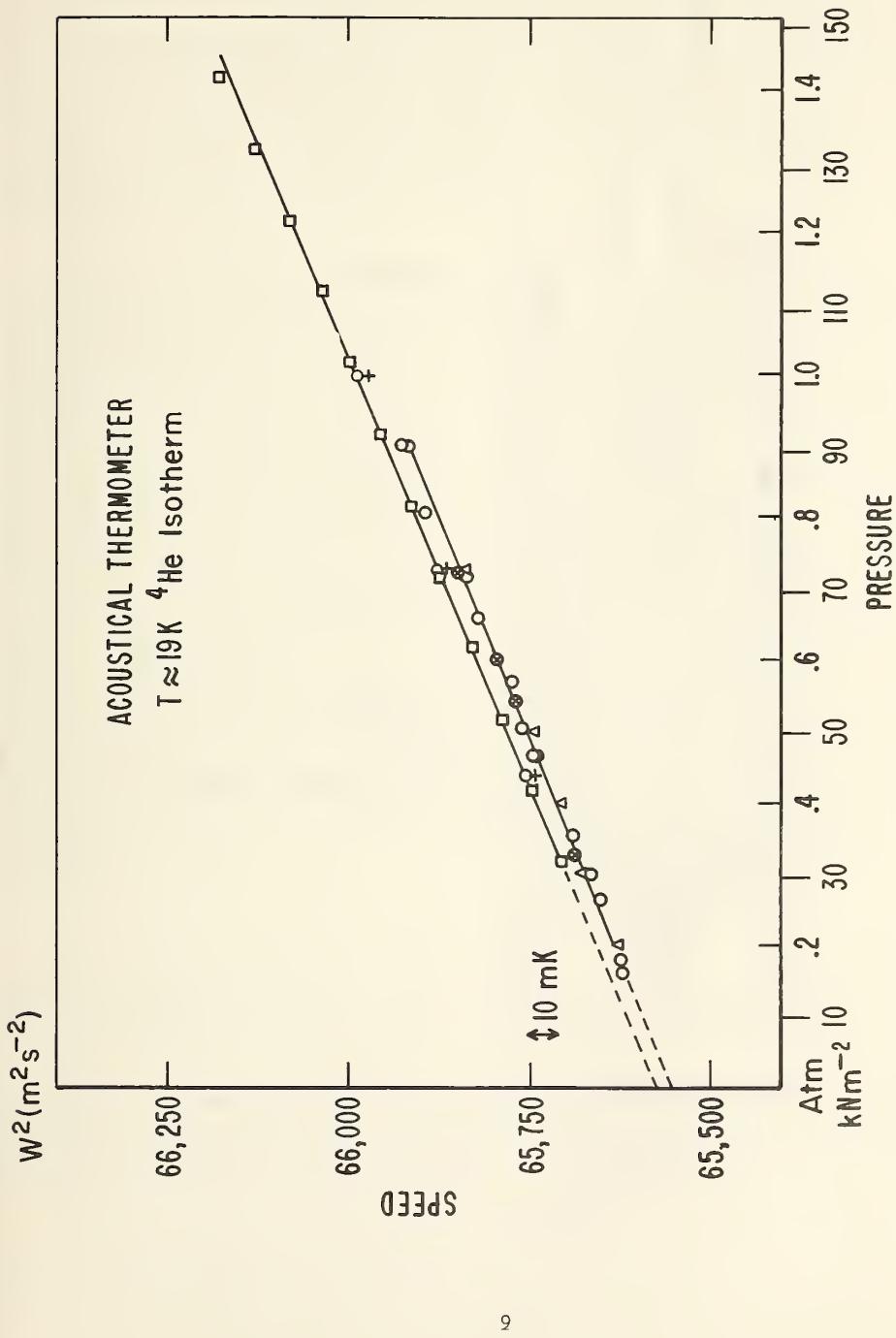


Figure 2. 18.935 K Isotherm -  $W^2$  as a function of pressure. A comparison between data derived by different experimental procedures: preliminary measurements (single shield-not outgassed)  $\square$  1968,  $\oplus$  1972. Recent (1972) with two shields  $\circ$  . Recent (1972) with single shield-outgassed prior to measurements  $\Delta$  . Sonic Cavity diameter doubled to investigate if K-H corrections applicable  $\Delta$  .

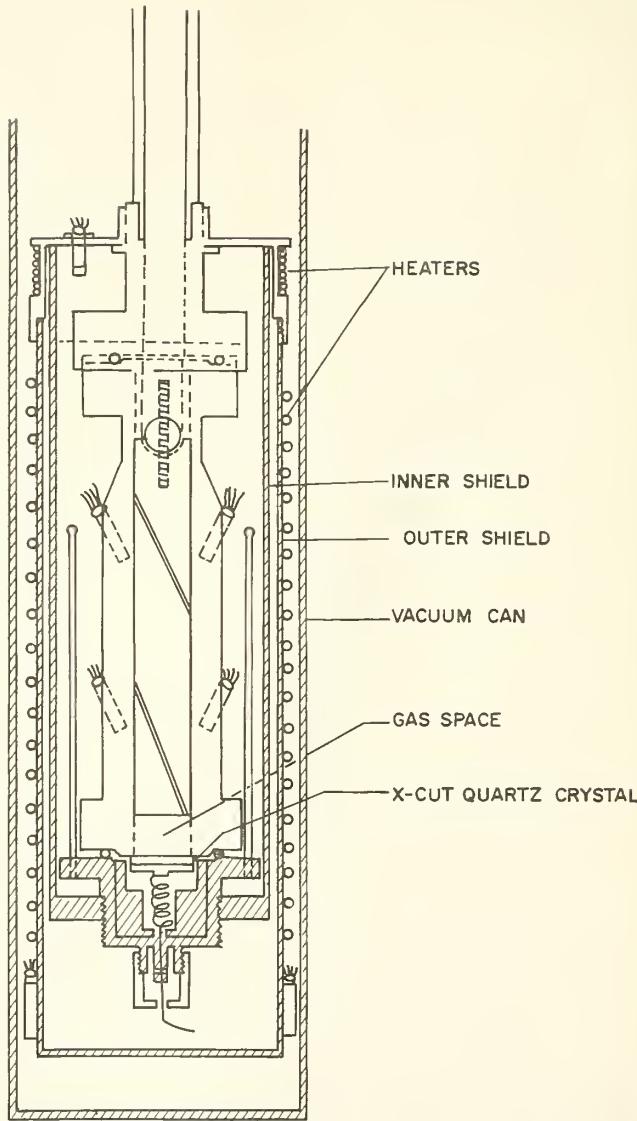


FIGURE 3

had been outgassed at 27 kelvins. It appears that during prolonged experimentation there may be an accumulation of helium gas in the acoustical thermometer chamber. This could result from helium penetrating the fused quartz evacuation line (within the liquid helium Dewar) and its subsequent condensation, adsorption or accumulation around the lower temperature portion of the acoustical thermometer. This in turn could cause thermal gradients in the resonating helium gas and between the monitoring germanium resistors. The improved thermal stability and improved resolution of helium gas resonances which are achieved during the two shield operating conditions strongly suggest the attainment of better operational procedures. It is to be noted in figure 1 and figure 2 that this technique has resulted in isotherm determinations for 9 and 19 K which yield slightly lower temperatures and slightly different slopes than were obtained when the measurements involved the use of a single radiation shield and no special high temperature outgassing of the acoustical thermometer.

The redefinition of isotherms between 9 and 21 K has not been continued because it was desirable to eliminate another possible source of systematic error. It was mentioned earlier that the measurement of the reflecting piston's displacement is effected by measuring the displacement at room temperature of the end of a fused quartz rod which extends downward approximately one meter and is connected to the copper piston by a ball joint. The fused quartz rod, and a surrounding fused quartz tube that supports the low temperature components of the acoustical thermometer, pass through temperature gradients which exist in the liquid helium Dewar. It is possible that, despite the relatively low coefficient of expansion for fused quartz, the temperature gradients may change or the fused quartz rod does not attain adequate thermal equilibrium with its surroundings during the course of the experimental measurements. To eliminate this possible source of error it has been decided to replace the existing piston-displacement measuring system with a laser interferometer. It will essentially measure the displacement of the upper end of the reflecting piston relative to a surface in the acoustical thermometer which is maintained at low temperatures -- more specifically, a reference corner cube will be mounted on the horizontal surface, figure 3, of the component that accommodates the main heater coil. This design, in addition, to providing a more accurate displacement measurement will permit the use of a thin wall stainless steel tubing for suspending the acoustical thermometer. Fused-quartz and metal epoxy joints can be replaced by solder joints, the instrument will not be so fragile and the fused quartz evacuation line, which may have been a contributing factor to the thermal instability (described in the previous paragraphs), will be replaced by a stainless steel line. Some of the rebuilding of the acoustical thermometer, to accommodate the laser interferometer, and preliminary operational checking of the laser system have already been performed. It is also hoped that the detection of gas resonance in the acoustical thermometer can be discerned digitally -- it has been detected in the past by the visual observation of a voltmeter. These apparatus changes should lead to the elimination of possible sources of systematic error and permit the determination of better isotherms.

## 5. Helmholtz-Kirchhoff Equation

It was stated earlier in this report that the value of  $W$  is, of course, the phase speed corresponding to whatever mode exists in the tube of gas and it is supposed that this mode is sufficiently like the plane wave mode to allow the identification of  $W$ , for our purpose, as the free-space value.

The equations derived by Helmholtz [7] and Kirchhoff [8] are based upon the effects of viscosity and heat conduction at the boundary walls for a gas that travels along the length of a cylinder. The equations assume that the amplitude of the sound waves are uniform over the whole cross-section of the cylinder and predict that both the speed and the absorption differ from free space values. For the speed

$$W = W_0 \left[ 1 - \frac{1}{2b} \sqrt{\frac{\eta_e}{\rho \pi f}} \right]$$

where  $W$  is the speed within a cylinder;

$W_0$  is the free space speed  
 $b$  is the radius of the tube  
 $\rho_0$  is the density of the gas  
 $f$  is the frequency of the sound

and  $\eta_e$  is an effective viscosity that is related to  $\eta$ , the real viscosity of a gas by

$$\eta_e = \eta [1 + \{(\gamma - 1) \frac{\sqrt{\frac{K}{C_p \eta \gamma}}}{\eta}\}]^2$$

$\gamma$  is the ratio of specific heats

$K$  is the thermal conductivity of the gas

and  $C_p$  is the specific heat at constant pressure.

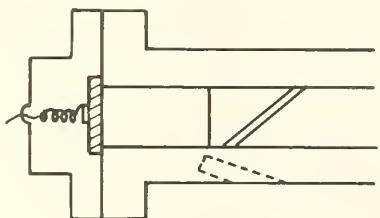
While these equations [9] have been experimentally investigated at frequencies up to 120 kHz and for tube diameters of a few millimeters, information is lacking at 1 MHz and higher. There is some verification of the equations at the relatively low frequencies but, at the very least, questions have been raised concerning the effect of the engineering finish of the boundary surface.

We thought that it would be both desirable and interesting to determine if the Helmholtz-Kirchhoff (H-K) equations were applicable to the NBS acoustical thermometer. In the correction term

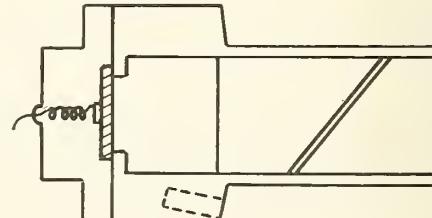
$$\frac{1}{2b} \sqrt{\frac{\eta_e}{\rho \pi f}},$$

increases in both the cylinder diameter and the frequency should increase the value of the measured sound speed within the cylinder. For an acoustical isotherm of  $W^2$  versus pressure or density there should be a noticeable bending of the isotherm with decreasing pressure. The temperature dependency of  $\eta$  is such that these effects should be greater at higher temperatures. For our acoustical thermometer  $b$  is close to 0.5 cm and it operates at 1 MHz. At an operating temperature of about 19 K, see figure 1, the H-K equations would predict that the data point at 0.25 atmospheres would depart from the isotherm linearity by the equivalent of about 8 millikelvin -- there would be a systematic decrease of  $W^2$  from linearity as  $p$  becomes smaller. As yet, both in the plotting of isotherm data and in its analysis by computer, it has not been possible to detect systematic curvatures at low pressures.

Another experiment was conducted to check the applicability of the H-K equation for our acoustical thermometry. Another gas chamber was constructed and its principal diameter was close to 2 cm. Figure 4a indicates the usual design of the containing gas space; figure 4b shows the design of the chamber of larger diameter.



(a)



(b)

FIGURE 4

We should have preferred that the entire cylindrical gas volume, to the right of the quartz crystal, in figure 4b, be of the 2 cm dimension but we desired to use the same quartz crystal that had been employed in previous experiments. One may assume that the larger diameter gas volume would cause a higher value for the measured speed of sound because the H-K correction term would be smaller; in figure 2, at 0.25 atmospheres, the measured speed of sound would be the equivalent of about 4 millikelvins higher than that which was measured with a cavity of customary design. The measured data points for the 2 cm cavity do not indicate that this is the case in figure 1 and figure 2.

As an additional check on the applicability of the H-K equations for our measurements it is highly desirable that the acoustical thermometer be operated at higher frequencies. While it was not convenient to do this during the past investigations of the H-K equations, it must and will be done when the new instrument that is being developed is finished. Additionally, if the new thermometer can be operated at yet lower pressures, it will yield greater sensitivity for detecting a possible low pressure curvature of the isotherms.

## 6. Calibration of Germanium Thermometers

Since 1965 the National Bureau of Standards has provided a calibration service for germanium thermometers on the NBS P2-20 (1965) scale for the general public on a reimbursable fee basis. Since the major cost associated with the calibration facility was for personnel, it was considered advantageous to increase the number of thermometers that could be calibrated during a calibration run, in order to reduce the unit cost per thermometer. Within the first calibration apparatus [3], the total number of germanium thermometers that could be accommodated was limited to four and two of the thermometers were calibration reference standards. Therefore, it was decided to construct a calibrator that could accommodate at least six thermometers and to update the existing support equipment.

Below will be discussed the improvements that have been incorporated in the new calibration apparatus and the stability of the two reference germanium thermometer standards over the last seven years. These two resistors are of a group of six that maintain the NBS P2-20 (1965) temperature scale.

For the new apparatus a vacuum seal, fabricated from stycast resin, was employed to admit electric lead lines into the evacuated chamber of the calibrator. Twice the necessary number were cast within the seal to meet possible future needs. In the former apparatus the absence of an adequate number of leads was a factor that limited the number of thermometers that could be calibrated at a time. Normally, when measurements of resistance are to be made potentiometrically, the thermometers current leads are wired in series and if one of the thermometers should become open circuited, the complete calibration would have to be terminated. To avoid the necessity of such a shut-down, individual leads were wired to the current anchors within the calibrator and terminated at one external multiple switch box; thus an open circuited germanium resistor could be by-passed and the calibration continued. A secondary radiation shield was added to the vacuum environment and was attached to the main calibrator core at its base. Another selector switch box was acquired that contained more terminals and the apparatus can now accommodate seven thermometers and with a few minor changes in the wiring, nine.

During a calibration run it is desired to attain a temperature (via resistance) that is as close as possible to the "assigned" resistance values of the calibrated germanium thermometer standards. If the values of the standards are within  $\pm 2$  mK of their assigned temperature, measurements of resistance are taken for all of the thermometers within the calibrator. Since the first derivative ( $dR/dT$ ) of each of the calibration standards is well defined, it is relatively simple to assign normalized values of temperature to a resistance value. Then the average of temperature of both standard thermometers at each calibration point (temperature) is assigned to each measured resistance value for the other thermometers that are to be calibrated.

Table 5

DATE*	SUBMITTED BY-(1)	T (CAL)	R565	T (CAL)	R540	T (CAL)	R565 (SCALE)	T (CAL)	R565 (SCALE)	T (CAL)	R540 (SCALE)	R540 (CAL)	QWNTTAB		NO. OF RESISTORS.....(3)	.....(4)	.....(5)	
													.....(2)	.....(6)				
7/2-2015													2.3240	2.3242	2.3244	2.3243	10821.0000	11472.0000
	2.8070	2.8071	2.8072	2.8073	2.8074	2.8075	2.8076	2.8077	2.8078	2.8079	2.8080	2.8081	6640.4000	6640.4000	6640.4000	6640.4000	7049.8000	11467.0000
	3.2110	3.2109	3.2113	3.2112	3.2111	3.2110	3.2109	3.2108	3.2107	3.2106	3.2105	3.2104	4818.9000	4819.1000	4819.1000	4819.1000	5129.8000	7049.5000
	4.2120	4.2120	4.2124	4.2122	4.2121	4.2120	4.2119	4.2118	4.2117	4.2116	4.2115	4.2114	2625.2000	2625.2000	2625.2000	2625.2000	2799.6000	5121.7000
	5.9240	5.9240	5.9241	5.9241	5.9241	5.9241	5.9241	5.9241	5.9241	5.9241	5.9241	5.9241	1792.3500	1792.3500	1792.3500	1792.3500	1916.4000	2799.0000
	6.0570	6.0568	6.0571	6.0570	6.0571	6.0570	6.0571	6.0570	6.0571	6.0570	6.0571	6.0570	1184.4200	1184.4200	1184.4200	1184.4200	1271.7400	1916.5000
	6.9320	6.9320	6.9323	6.9323	6.9323	6.9323	6.9323	6.9323	6.9323	6.9323	6.9323	6.9323	851.2900	851.2900	851.2900	851.2900	917.5200	917.4200
	9.0670	9.0666	9.0669	9.0669	9.0669	9.0669	9.0669	9.0669	9.0669	9.0669	9.0669	9.0669	595.9300	595.9300	595.9300	595.9300	644.9700	645.9100
	8.9910	8.9910	8.9910	8.9910	8.9910	8.9910	8.9910	8.9910	8.9910	8.9910	8.9910	8.9910	450.7500	450.7500	450.7500	450.7500	489.3100	489.3100
	9.8860	9.8860	9.8863	9.8863	9.8863	9.8863	9.8863	9.8863	9.8863	9.8863	9.8863	9.8863	351.5700	351.5700	351.5700	351.5700	382.5400	382.5090
	10.9010	10.9010	10.9013	10.9012	10.9013	10.9012	10.9013	10.9012	10.9013	10.9012	10.9013	10.9012	272.1600	272.1600	272.1600	272.1600	296.6900	296.6680
	12.0160	12.0160	12.0159	12.0159	12.0159	12.0159	12.0159	12.0159	12.0159	12.0159	12.0159	12.0159	211.6020	211.6020	211.6020	211.6020	230.9520	230.9310
	12.9610	12.9610	12.9614	12.9614	12.9612	12.9612	12.9614	12.9612	12.9614	12.9612	12.9614	12.9612	174.7370	174.7370	174.7370	174.7370	190.7990	190.7860
	14.0480	14.0480	14.0493	14.0493	14.0494	14.0494	14.0493	14.0494	14.0493	14.0494	14.0493	14.0494	143.4480	143.4480	143.4480	143.4480	156.6480	156.6280
	14.9920	14.9920	14.9923	14.9923	14.9922	14.9922	14.9923	14.9922	14.9923	14.9922	14.9923	14.9922	123.0180	123.0180	123.0180	123.0180	134.2940	134.2870
	16.0480	16.0480	16.0473	16.0473	16.0481	16.0481	16.0473	16.0481	16.0473	16.0481	16.0473	16.0481	105.2660	105.2660	105.2660	105.2660	114.6450	114.6470
	16.9680	16.9683	16.9683	16.9683	16.9683	16.9683	16.9683	16.9683	16.9683	16.9683	16.9683	16.9683	92.9640	92.9640	92.9640	92.9640	101.3670	101.3690
	18.0037	18.0037	18.0039	18.0039	18.0039	18.0039	18.0039	18.0039	18.0039	18.0039	18.0039	18.0039	81.7310	81.7310	81.7310	81.7310	89.0490	89.0500
	18.9410	18.9406	18.9411	18.9411	18.9409	18.9409	18.9409	18.9409	18.9409	18.9409	18.9409	18.9409	73.4680	73.4680	73.4680	73.4680	80.0060	80.0060
	20.0510	20.0506	20.0510	20.0510	20.0508	20.0508	20.0508	20.0508	20.0508	20.0508	20.0508	20.0508	65.3950	65.3950	65.3950	65.3950	71.1300	71.1300

Table 5 typifies the procedure for assigning temperatures to germanium resistors that have been calibrated. The temperature in the Column T (2-20) scale are the temperatures that are associated with the values of resistance, "R565 (scale)" and "R540 (scale)", for the standard resistors 565 and 540. The values under "R565 (cal)" and "R540 (cal)" are the resistance values at which the actual calibration of unknown germanium resistors were performed. Through the respective  $dR/dT$ 's, the "T (cal) R565" and "T (cal) R540" are calculated and from these "T (cal) avg" is obtained. "T (cal) avg" are the temperatures that correspond to respective resistance values for the "unknown" germanium thermometers.

When the new calibration apparatus became operational in September, 1971, resistor 561, which had been compared with 565 and 540 twice previously, was again installed to assess the maintenance of the provisional temperature scale. A comparison of the average value of the standards with resistor 561 and its previous calibration of 1967, indicated that the resistors 565 and 540 had maintained their calibration (relative resistance comparisons) to better than  $\pm 0.1$  mK.

Since the initiation of the calibration service for germanium thermometers, a record has been maintained for the two reference standards (565 and 540). Their reproducibility of assigned resistance values is evaluated by the differences ( $R_{540} - R_{565}$ ) between their observed values at each calibration point. Table 6 contains a compilation of the relative stability of the two standard thermometers and their relative reproducibilities over the last five years. The data involve 52 calibration runs and also include approximately 40 thermal cyclings of the calibrator from ambient to low temperatures. It also includes data that were taken after the installation of the monitor resistors within the new calibration apparatus since our primary concern is the retention and stability of the NBS P2-20 (1965) temperature scale.

Table 6

Statistical Review of the Two Reference Standards Within Calibration Apparatus

No. Runs 52	No. Cycles 40	Period Covered Nov 66 - Dec 71	
Temperature (K)	$\Delta T^a$ (mK)	$\pm 2\sigma$ (mK)	$dR/dT(561)$ ( $\Omega/mK$ )
2.3	1.0	.58	13.000
2.8	1.0	.56	5.800
3.2	1.0	.51	3.500
4.2	1.3	.63	1.350
5.0	0.5	.26	.770
6.0	0.5	.21	.440
7.0	0.6	.23	.290
8.0	0.5	.21	.190
9.0	0.4	.23	.130
10.0	0.6	.25	.093
11.0	0.5	.25	.065
12.0	0.6	.25	.045
13.0	0.7	.33	.035
14.0	0.8	.33	.025
15.0	0.6	.25	.019
16.0	0.9	.37	.015
17.0	0.7	.37	.012
18.0	0.8	.35	.010
18.9	0.9	.44	.008
20.0	1.0	.47	.007

a) Indicates maximum difference in "equivalent temperature" between the two reference standards ( $T_{540} - T_{565}$ ).

Below 4.2 and above 16.0 K there appears to be a larger spread between the assigned resistance values and their realization during a calibration run, i.e., both  $\Delta T$  and  $2\sigma$  appear to increase at both temperature extremes. At 4.2 K and below, the normal procedure is to admit  $^4\text{He}$  exchange gas to the vacuum can to hasten the cool down of the interior of the calibration apparatus and then to reduce the vapor pressure of the liquid helium bath to a pressure below the lambda point. Meanwhile the vacuum chamber is being pumped out and, when the automatically controlled bridge current approaches a steady-state current to the heater of the calibrator and the vacuum pressure remains at a steady value of  $10^{-7}$  mm, calibration measurements are made. In the former calibration apparatus there was a time limitation in which all the resistance values could be taken because of the gradual drift upwards in the temperature of the calibrator. This could be in part due to the continuing outgassing of the calibrator. In the present apparatus, temperature controlling at a selected low temperature is possible for hours and the problem of drift seems to have been eliminated. Above 16 K the  $dR/dT$ 's of the resistors become smaller and the uncertainty in the measured values of resistance is approaching our measurement capability. In our last comparison of resistance thermometer 561 with the two monitoring standards there were indications that resistance thermometer 565 may be experiencing a slight drift in its calibration at the higher temperatures (see table 6).

The new calibration apparatus has made it possible to reduce the germanium thermometer calibration costs from \$1,100 to \$600. This of course depends upon the receipt of enough thermometers so that at least four can be calibrated at one time.

#### 7. Discussion

As was indicated previously in this report, the acoustical thermometry at NBS will be continued but a laser interferometer will be employed for wavelength determinations. When the final measurements have been made they may or may not lead to the revision of the temperature scale NBS P2-20 (1965). At the present time there is considerable activity concerning temperature determinations below 20 kelvins by several national laboratories. Under the auspices of the Comité Consultatif de Thermométrie a committee on thermometry below 90 kelvins has been formed and the Australian National Standards Laboratory (CSIRO) has undertaken the task of performing measurements to compare temperature scales below 20 or 30 kelvins. The NBS has calibrated three germanium thermometers, which will be sent to CSIRO, and similar contributions from PRMI, NPL, Leiden, Iowa State University and

CSIRO (and, possibly, other laboratories also) are expected. NBS will submit additional acoustical thermometry data as they become available. Although our new measurements will be reported to the public, it may be advantageous to await the extension of IPTS-68, rather than have the NBS present a modification of NBS P2-20 (1965) for calibration purposes.

The reader's attention is called to two papers that will shortly be appearing in *Metrologia*. The first is a contribution from Professor C. A. Swenson, of Iowa State University: "Relationship from 1 to 34 K Between a Paramagnetic Salt Temperature Scale and Other Scales; an Addendum". The second will concern the realization of low temperature fixed points by Dr. K. H. Berry of the National Physical Laboratory.

Table 7

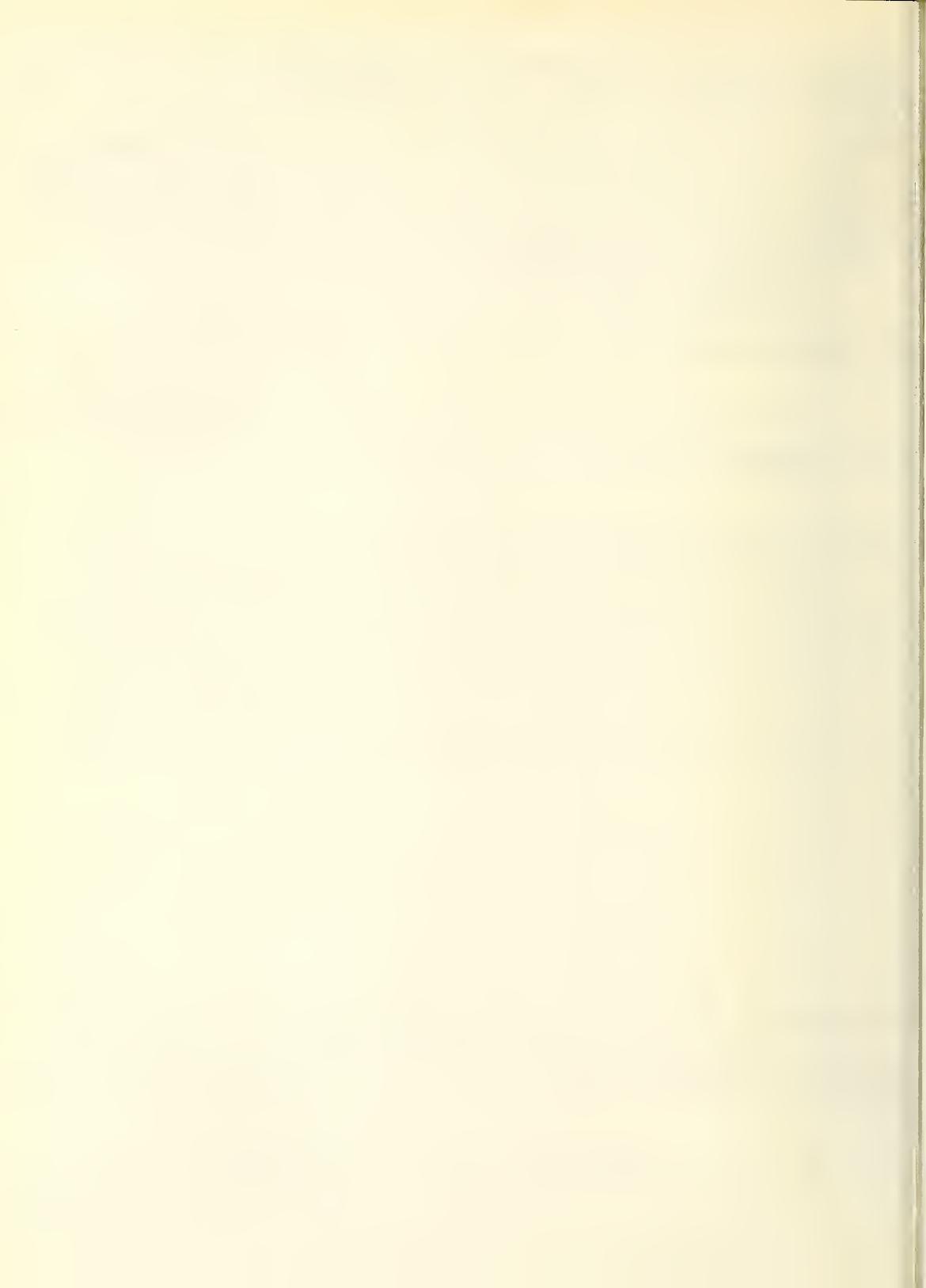
	NBS Acoustic Smooth	NPL Acoustic	NPL P-V Isotherms
$^4\text{He}$ NBP (K)	$4.225 \pm .001$	$4.2235 \pm .0018$	$4.2240 \pm .0007$
e- $\text{H}_2$ TP (K)	$13.8047 \pm .001$		
e- $\text{H}_2$ NBP (K)	$20.2789 \pm .005$	$20.2643 \pm .0049$	$20.2746 \pm .0013$

It is appropriate at this point to make reference to table 7, which includes values of temperature that have recently been reported from NPL for the normal boiling points of  $^4\text{He}$  and  $e\text{-H}_2$ . The "NPL acoustic" are values of temperature and their uncertainties that were obtained with the NPL low frequency acoustical thermometer [10]. The NPL p-v isotherm values were obtained by K. H. Berry and reported in the Annual Report 1971 of the Division of Quantum Metrology of the National Physical Laboratory. The "NBS acoustic smooth" values have also been cited earlier [11,12]. It should be repeated for clarity that the limits on 4.225 represent the scatter of the isotherm data points while the limits on 13.8047 and 20.2789 represent the variations in acoustically determined temperatures of the isotherms that were fitted with germanium thermometers in the vicinity of 13.8 and 20.27, respectively -- presented in table 3. Unpublished work [13] on  $^3\text{He}$  p-v isotherms by Dr. R. Sherman of the Los Alamos Scientific Laboratory also indicates that the NBP of  $^4\text{He}$  is about 10 mK greater than the temperature that is assigned to this fixed point on the  $T_{58}$  [14]  $^4\text{He}$  vapor-pressure scale.

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16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)  Earlier acoustical thermometer 'speed of sound' isotherms are compared with recent measurements. Where systematic deviations had appeared in "smoothing" through acoustically derived temperatures via germanium thermometers, a possible solution has emerged; more precise temperature control of the acoustical thermometer is desirable and has been realized. A cursory examination of the applicability of the Helmholtz-Kirchhoff equation was made with a modified sonic chamber. Measurements with the modified instrument have shown no changes in the experimental results.				
The maintenance of the NBS P2-20 (1965) scale is described. A new calibration apparatus was constructed to accommodate more germanium thermometers thus reducing the calibration cost per thermometer during a calibration run.				
17. KEY WORDS (Alphabetical order, separated by semicolons) Acoustical thermometer; calibrator; germanium thermometers; heat conduction; NBS P2-20 (1965) scale; 'speed of sound' isotherms; viscosity.				
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