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UNITED STATES ATOMIC ENERGY COMMISSION

DENSITY OF SOLID HEX NEAR THE TRIPLE
POINT, AND THE EXPANSIVITY OF THE
LIQUID FROM THE TRIPLE POINT TO 92°C

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Apparatus:

The material was contained in a calibrated glass tube of approximately three millimeters inside diameter and 123 mm. in length which was used as a pycnometer.

The pycnometer was suspended in an unsilvered dewar flask by means of a wire attached to a lowering device above the dewar. The lower half of the dewar was filled with water which was stirred and could be thermostated to $\pm .02$ degrees. The temperature of the air space above the water could also be controlled.

Expansivity of the Liquid:

The lowering device was adjusted so that the liquid meniscus in the pycnometer was approximately one centimeter below the surface of the water. The height of the liquid in the pycnometer at various temperatures was obtained by measuring the height of the meniscus above a fiducial scratch on the pycnometer with a travelling microscope. The meniscus and vapor corrections that were made were of the order of .2 mm equivalent height in the tube. Approximately eighty millimeters of the tube were filled with the liquid.

Two sets of measurements of the expansivity were made on different days. In all, fifteen determinations of the volume were made. The results are expressed by the equation

$$V/V_f = 1 + 1.727 \times 10^{-3}(T-T_f) + 3.59 \times 10^{-6}(T-T_f)^2$$

Where V is the volume of the liquid under its own vapor pressure, T is the temperature, and the subscript "f" refers

to values at the triple point. T_f was measured in the Low Temperature Laboratory of the National Bureau of Standards in connection with determinations of the heat capacity of the material. It is this value, $337.212^\circ\text{K} = 64.052^\circ\text{C}$, that was used in deriving the equation.

The agreement of equation and observations is shown in the accompanying table.

For the expansivity of the liquid at the triple point we may take $\alpha_f = (1.727 \pm .008) \times 10^{-3}$.

Though the experimental values of V/V_f definitely showed a curvature upward, the value of dd/dT , namely 7.18×10^{-6} , should be regarded as reliable only as to order of magnitude.

Density of the Solid Just Below the Triple Point:

The material was frozen by lowering the pycnometer from the hot air space into the cooler water bath. This lowering was done very slowly; successful freezing, without the trapping of gas bubbles in the solid, took from four to five hours.

The liquid was frozen with the temperature of the water bath from five to ten degrees below the triple point. After the freezing was completed the temperature of the bath was raised to the temperature of measurement. The volume of the solid was not appreciably affected by the temperature of the water bath during freezing.

The final measurements showed that the ratio of the volume of the liquid at 65.1° to the solid at 62.5° is $1.343 \pm .002$ (probable error). Taking Priest's value of 3.667 g/cm^3 for the density of the liquid at 65.1° the density of the solid is 4.93 g/cm^3 . The calculated density of the liquid at the triple point is 3.674 g/cm^3 .

No abnormal expansion of the solid just below the triple point was observed. In fact, it was found possible to take the solid from room temperature to the triple point in the space of a few minutes without breaking the pycnometer. This was possible whether the material was frozen slowly as

described above or was frozen by rapid lowering into the water at room temperature.

Supercooling of the Liquid:

It was also found possible to keep the liquid supercooled 1.6° below the triple for several hours. In addition, the liquid in the tip of the pycnometer could be cooled gradually to nearly seven degrees below the triple point before solidification set in.

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Table of V/V_f , the ratio of the volume V of the liquid at T degrees above the triple point (64.052°C) to the volume V_f at the triple point. Calculated values were derived from the equation:

$$V/V_f = 1 + 1.727 \times 10^{-3} T + 3.59 \times 10^{-6} T^2$$

Date of run	T	V/V_f observed	Observed-Calculated
Feb. 6	-1.60	.997	.000
	+7.26	1.013	.000
	9.59	1.017	.000
	9.59	1.017	.000
	11.93	1.022	.001
	18.95	1.034	.000
	21.29	1.038	.000
	25.98	1.048	.001
	28.33	1.053	.001
Feb. 9	-1.60	.997	.000
	- .02	1.000	.000
	+ .92	1.001	-.001
	9.59	1.016	-.001
	18.95	1.033	-.001
	28.33	1.051	-.001