

PROPERTIES OF WATER. PART IV. PRESSURE-VOLUME-TEMPERATURE RELATIONS OF WATER IN THE RANGE 100-400 C AND 100-1400 BARS*

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ABSTRACT. New precision measurements of the specific volume of water in the range 100-400 C and 100-1400 bars are generally close to earlier published measurements. Recent measurements in the critical region by Kirillin, Rumiantsev, and Zubarev are used to complete a table of specific volumes for this range.

INTRODUCTION

The specific volume of water was measured in the range 100-400 C, 100-1400 bars during 1957, as part of the general program of precision measurements of the pressure-volume-temperature relations of water. Measurements of apparently high precision have long been available from the work of Smith and Keyes (1934) for the liquid water region to about 350 bars. Kirillin, Rumiantsev, and Zubarev (1956) provide a large number of recent measurements near the critical temperature, extending for some temperatures to 900 bars. The previous measurements of Kennedy (1950), at temperatures above 200 C, extended to 2500 bars. Kennedy accepted the data of Smith and Keyes, which were in substantial agreements with his own. It was not expected that the measurements in this lower temperature region would be subject to appreciable errors by reaction with the bomb wall as was encountered at high temperature and low pressure (Kennedy, 1957). However, plots of isothermal compressibility and isobaric thermal expansion indicated some inconsistency and lack of continuity in Kennedy's table; these seem to be most pronounced at about 400 bars, where these measurements replace those of Smith and Keyes.

Precision equipment assembled for the purpose of measurements in a higher temperature range (Holser and Kennedy, in press) made it possible to check the earlier measurements to 1400 bars, and to extend them down to 100 C to meet the new measurements in the range 0-100 C (Kennedy, Knight, and Holser, 1958). The present measurements are principally concerned with the liquid water region. The numerous data by Kirillin were already available for the critical region, and an improvement or correction of these values would have required much more precise control and measurement of temperature than was immediately available. Therefore the Kirillin data have been used extensively in completing a smooth table through the critical region.

APPARATUS

A schematic diagram of the apparatus used is shown in figure 1. The arrangement is similar to that used earlier (Kennedy, 1954, p. 230), with the addition of a dead-weight piston gauge in the oil system for precise pressure measurement.

Piston gauge corrections for temperature and pressure were made as described by Johnson and Newhall (1953, p. 303). The sensitivity of the gauge

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was about .05 percent. In order to effectively use this sensitivity with the capillary lines used for connections, it was necessary to use a very low viscosity transmission fluid, obtained by mixing equal proportions of kerosene and a light oil (Gargoyle Vacuoline type E). The Bourdon gauges are 15-inch Heise gauges with a range of 1400 bars.

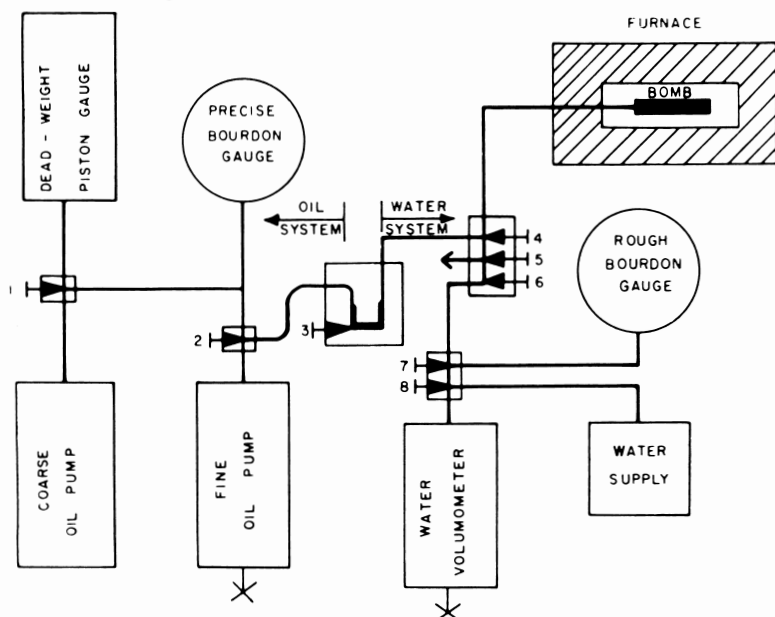


Fig. 1. Schematic diagram of apparatus.

The volumeters and mercury U-block have been previously described. The volumeter was calibrated by weighing water ejected at various pressures, with a result precise to 0.03 percent. The bombs used in these experiments were similar to those previously described, made of Inconel-X, or 100-NT2 alloy. The volumes of about 70 and 170 cc were measured by repeated filling with water at about 200 C and 200 bars and ejecting and weighing the water; the precision was .03 percent. Volumes at other temperatures and pressures were calculated from the known thermal expansion and elastic properties of the materials. Water was supplied from a flask connected with a reflux condenser in such a manner that steam at one bar was always in contact with the water surface, in order to keep gases out of solution.

Temperature was measured with a Pt, Pt-10 percent Rh thermocouple inserted in a well in the bomb. The thermocouple was calibrated at various times against two thermocouples that had previously been calibrated at the National Bureau of Standards. The accuracy of temperature measurement was about ± 0.2 C.

METHOD

Most of the measurements of specific volume were made along isotherms, by measuring the compressibility with respect to a low-pressure (300 bar) base. The base line was established by measuring the isobaric thermal expan-

sion at 300 bars, starting with the previously measured value at 100 C, 300 bars (Kennedy, Knight, and Holser, 1958), and supplemented by the earlier measurements of Smith and Keyes (1934) and Kirillin, Rumiantsev, and Zubarev (1956).

A typical isotherm measurement started with loading of distilled, de-aerated water into the bomb at the highest pressure, along with a little CuO to eliminate errors from hydrogen generated by reaction with the bomb wall. Valve 6 was then cut off, so that if the mercury remained at a constant level, the mass of water in the bomb was very nearly constant. A correction for the change of mass of water in the bomb, due to changes of compression of water in the capillary line, was usually insignificant. The bomb with its contained water was brought to thermal equilibrium close to the even "isothermal" temperature. After a preliminary determination of the pressure using the Bourdon gauge, the final measurement was made with valves 1, 2, and 4 open to the dead-weight piston gauge, which was balanced for no motion of the mercury in the U-tube. Valve 4 was then closed and valve 6 opened, and the volumeter backed off, removing sufficient water to reduce the pressure by 50 to 100 bars. The volumeter was read at a calibrated pressure before and after the removal, with valve 6 closed. After thermal equilibrium had been re-established, the new pressure was determined, and the process repeated down to a pressure of about 100 bars. Small departures from the even isotherm temperature were corrected by approximate values of the thermal expansion of water and of the bomb material.

A slightly different arrangement was used for measurements of the isobaric thermal expansion. The volumeter was used to measure the mass of water that had to be removed from the bomb, in order to keep the pressure constant as the temperature was raised to successively higher values. A very long time would have been required at each temperature, to obtain constant temperature over the 15-30 minutes required for a reading of the piston gauge. Therefore the pressures were read with the precision Heise Bourdon gauge, which in turn was calibrated against the piston gauge before and after the runs. Readings were made with the temperature stepping both upward and downward.

An independent check on the measurements of temperature and pressure was given by measurements along the saturation line. The saturation pressure at a given temperature is known to high precision from the work of Osborne and Meyers (1934); at 365 C the measured pressure of about 200 bars matched their value within the 1 part in a 1000 corresponding to the temperature uncertainty of 0.1 C. A further check on the system of measurement was provided by two isotherms run at 100 C. These measurements correlated closely with those obtained previously on similar equipment, but by another operator in another laboratory (Kennedy, Knight, and Holser, 1958).

LOW-PRESSURE BASE LINE

Originally, we contemplated measurement of only the isothermal compressibility of water to check Kennedy's earlier determinations. The specific volumes were to be calculated from the base values at low pressures established by Smith and Keyes (1934). It became apparent, however, that the Smith and

Keyes data should be checked for the following reasons:

- (1) Specific volumes given by formulae 1-6 of Smith and Keyes (1934, p. 287-289) depart by as much as 0.003 cc/g from their measured values, as shown by their figure 1 and table II; this deviation is much greater than the internal consistency of the individual isotherms. The formulae were used in making their final tabulation, and the latter has been copied into many "steam tables" published since.
- (2) Osborne, Stimson, and Ginnings (1937, p. 427-430) found that above 330 C, the calorimetric determinations of the saturation volume of liquid water differed from those obtained by extrapolation according to the Smith and Keyes formulae, although the experimental data were shown to be reasonably consistent.
- (3) In the range 0-100 C Kennedy, Knight, and Holser (1958) also noted the disagreements of the formulae with previous measurements at one bar and with their own measurements at high pressures.
- (4) Recent measurements of Kirillin, Rumiantsev, and Zubarev (1956) did not seem concordant with the measurements of Smith and Keyes in the range 340-380 C.

The question therefore arose as to whether interpolations among the *measured* values of Smith and Keyes are the better values for other temperatures and pressures, or whether their *formulae* provided a real smoothing among the isotherms.

Measurements were made on two runs, at intervals of 5-20 C. The measurements were continued to 405 C, although the rapid change of thermal expansion above 360 C reduced the precision considerably.

Because of the troubles previously experienced with analytical formulations, the data were reduced by a graphical smoothing of $\Delta v/\Delta T$. The results of this smoothing are presented in table 1, along with the derived values for isobaric thermal expansion. Deviations of the experimental points from the smooth curve are shown in figure 2; for the most part they are less than deviations that might be due to a temperature uncertainty of ± 0.1 C. At high temperatures the deviations of volume caused by a small temperature change, increase so fast, that it was found most useful to plot the deviations relative to the thermal expansion itself.

The figure also shows deviations of the values of other investigators from our smooth curve. The "measured" values of others represent an interpolation (or extrapolation) to 300 bars from nearby actual measurements. To the necessary or accidental extent that these interpolations may be inaccurate, as they certainly are in the critical region, such a comparison does not do justice to the data. However, some of the deviations are so large that they cannot be ascribed to errors of this sort.

In general, our smoothed curve agrees more closely with the measurements of Smith and Keyes, than it does with their formulation. An exception occurs in the range 300-330 C, where the Smith and Keyes formulation lies closer to our curve. One other exception is evident in the Smith and Keyes measurement at 200 C, which seems out of line with their other measurements and differs appreciably from their smoothed value and from our experimental and observed values.

TABLE 1
Specific Volume and Isobaric Thermal Expansion of Water at 300 Bars
Smoothed Measurements of Holser and Kennedy

T deg C	v cc/g	$\partial v / \partial T$ (cc/g·deg C)·10 ⁴
100	1.0290	7.3
110	1.0366	7.8
120	1.0447	8.4
130	1.0533	8.9
140	1.0626	9.5
150	1.0724	10.1
160	1.0828	10.7
170	1.0938	11.4
180	1.1055	12.1
190	1.1179	12.8
200	1.1310	13.6
210	1.1449	14.5
220	1.1597	15.4
230	1.1756	16.5
240	1.1927	17.8
250	1.2111	19.2
260	1.2310	20.8
270	1.2526	22.6
280	1.2762	24.7
290	1.3023	27.4
300	1.3313	30.5
305	1.3471	32.5
310	1.3638	34.5
315	1.3815	36.9
320	1.4004	39.4
325	1.4208	42.3
330	1.4428	45.5
335	1.4667	49.6
340	1.4928	54.5
345	1.5214	59.6
350	1.5528	65.7
355	1.5877	66.
360	1.6273	75.
365	1.673	99.
370	1.727	119.
375	1.792	147.
380	1.875	188.
385	1.983	254.
390	2.135	370.
395	2.381	680.
400	2.815	1010.

It is not claimed that our measurements are definitive. In the critical region particularly the equipment used by the Keyes and Kirillin groups, involving platinum resistance thermometers and large thermal baths, could be expected to afford much more accurate data than our equipment, which was essentially designed for operation at higher temperatures. Our new measure-

ments may throw some light on the disagreements among previously published values. In any event their use as a base for our tables makes our tabulation completely independent of all but the well established low temperature, 1 bar values used as a base by Kennedy, Knight, and Holser (1958).

For the isotherms tabulated (table 2) at 380 and 400 C, a lack of any nearby data from our measurements (except at 300 bars), has led us to rely on Kirillin's extensive data in deriving these values. Any of the rest of our tabulation can be adjusted easily by anyone who prefers different base values at 300 bars.

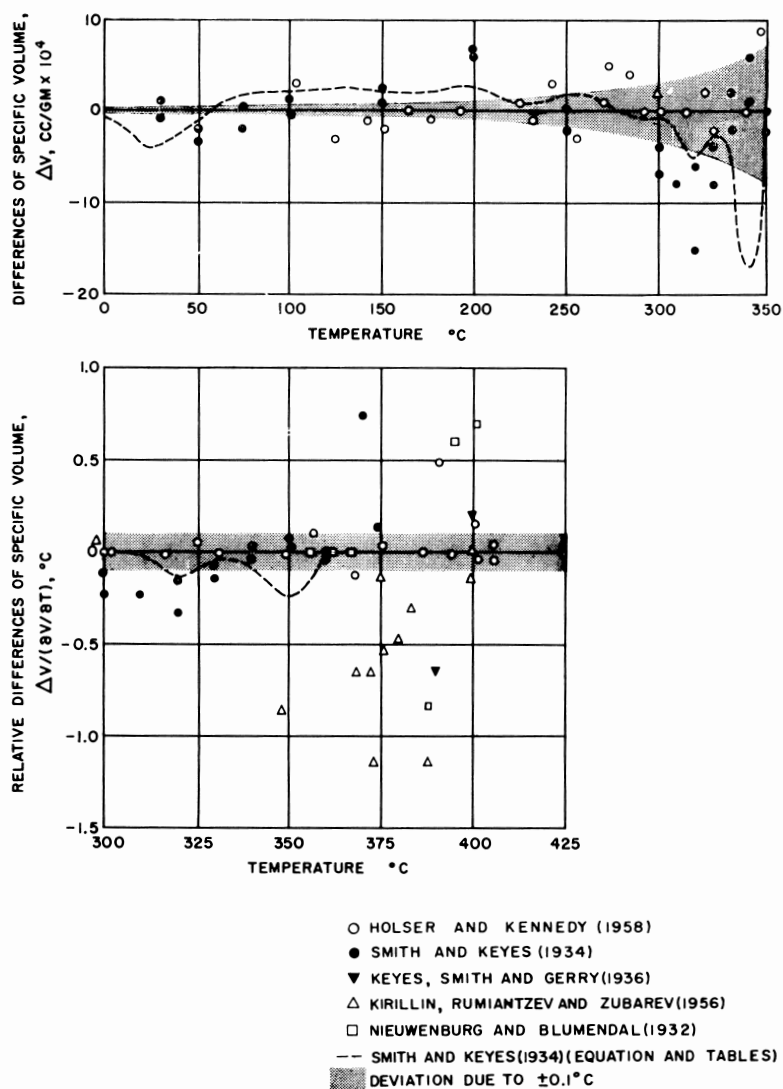


Fig. 2. Differences of specific volume at 300 bars—smoothed values of Holser and Kennedy (table 1) less other values.

TABLE 2
Specific Volume of Water, cc/g
Temperature, deg C

Pressure bars	120	140	160	180	200	220	240	260	280	300	320	340	360	380	400
100	1.0549	1.0741	1.0960	1.1208	1.1490	1.1814	1.2196	1.2655	1.3227	1.3978	19.26	21.49	23.32	24.94	26.41
150	1.0523	1.0711	1.0925	1.1168	1.1442	1.1756	1.2121	1.2559	1.3093	1.3779	1.4725	1.629	12.58	14.32	15.68
200	1.0497	1.0682	1.0892	1.1129	1.1396	1.1701	1.2054	1.2471	1.2973	1.3602	1.443	1.5689	1.825	8.259	9.969
250	1.0472	1.0654	1.0860	1.1092	1.1352	1.1648	1.1989	1.2388	1.2864	1.3449	1.4207	1.5261	1.697	2.254	6.056
300	1.0447	1.0626	1.0828	1.1055	1.1310	1.1597	1.1927	1.2310	1.2762	1.3313	1.4004	1.4928	1.6273	1.891	2.814
350	1.0422	1.0598	1.0797	1.1019	1.1268	1.1547	1.1868	1.2237	1.2668	1.3187	1.3826	1.4653	1.5790	1.772	2.130
400	1.0398	1.0571	1.0766	1.0984	1.1228	1.1500	1.1811	1.2167	1.2581	1.3072	1.3669	1.4410	1.5420	1.690	1.923
450	1.0374	1.0544	1.0736	1.0949	1.1188	1.1454	1.1756	1.2100	1.2498	1.2966	1.3526	1.4212	1.5116	1.635	1.812
500	1.0350	1.0518	1.0707	1.0916	1.1150	1.1410	1.1704	1.2037	1.2420	1.2867	1.3396	1.4036	1.4852	1.589	1.735
600	1.0304	1.0467	1.0650	1.0851	1.1076	1.1326	1.1604	1.1918	1.2276	1.2688	1.3166	1.3732	1.4415	1.520	1.628
700	1.0259	1.0417	1.0595	1.0790	1.1007	1.1246	1.1512	1.1809	1.2145	1.2526	1.2966	1.3475	1.4074	1.477	1.564
800	1.0215	1.0370	1.0542	1.0731	1.0941	1.1171	1.1426	1.1708	1.2025	1.2380	1.2787	1.3250	1.3790	1.439	1.516
900	1.0173	1.0324	1.0492	1.0675	1.0877	1.1099	1.1344	1.1614	1.1914	1.2248	1.2626	1.3054	1.3544	1.410	1.477
1000	1.0132	1.0280	1.0443	1.0621	1.0816	1.1031	1.1266	1.1525	1.1811	1.2129	1.2482	1.2881	1.3331	1.385	1.444
1100	1.0093	1.0237	1.0396	1.0569	1.0758	1.0966	1.1193	1.1441	1.1715	1.2017	1.2351	1.2725	1.3142	1.362	1.416
1200	1.0055	1.0195	1.0350	1.0519	1.0703	1.0904	1.1124	1.1362	1.1625	1.1912	1.2230	1.2582	1.2972	1.342	1.392
1300	1.0018	1.0155	1.0306	1.0471	1.0650	1.0845	1.1057	1.1287	1.1539	1.1814	1.2117	1.2449	1.2817	1.324	1.370
1400	.9982	1.0117	1.0264	1.0424	1.0598	1.0787	1.0992	1.1215	1.1458	1.1723	1.2012	1.2326	1.2674	1.307	1.352

ISOTHERMS

Isotherms were determined in the above-described manner at 140 (2), 180, 220, 260, and 298 C. An isotherm at 355 and two at 420 (described in Holser and Kennedy, in press) were run all the way down to low pressure where the specific volume of the small amount of remaining steam was well known from previous measurements (Kennedy, 1957). Therefore these higher temperature isotherms were more or less independent of the 300 bar base line.

It was desirable to smooth all isotherms by graphing the data in a manner that would emphasize its small irregularities. Therefore the method of item differences was employed, by which, in effect, the slopes given by the differences between succeeding determinations are smoothed, and the smooth values integrated back. This procedure is facilitated if the differential (or difference) function that is graphed lies on approximately a straight line. If the specific volume of water on an isotherm were represented by equations of either the Tumlirz (Eckart, 1957) or van der Waals types,

$$(p + p_0)(v - v_0) = K \text{ (or } -RT) \quad (1)$$

then the function $(-dp/dv)^{1/2}$ should plot as a straight line against pressure:

$$K^{1/2} - \frac{dp}{dv}^{1/2} - p_0 = p \quad (2)$$

On the other hand, if the data for volume is more precisely represented by an equation like that of Tait (Hirschfelder, Curtiss, and Bird, 1954, p. 261):

$$\frac{v_0 - v}{v_0} = A \ln \frac{B + p}{B} \quad (3)$$

then the slope (dp/dv) , rather than its square root, plots as a straight line:

$$Av_0 \frac{dp}{dv} + B = -p \quad (4)$$

Several comparisons in the range 300-360 C indicated that $(dp/dv)^{1/2}$ was less straight than (dp/dv) , so the latter function was used in smoothing all the isotherms. These curves are slightly concave toward the p axis at high temperature, but below 300 C no curvature could be detected within the precision of the data. A detailed comparison of the various equations of state for liquid water will be made in a subsequent publication of this series.

Most measurements were within ± 0.0002 cc/g of the smoothed isotherms, except at 355 C where deviations were as high as ± 0.0010 cc/g. The individual original measurements are listed and compared with the final tabulated values (see below) and deposited for reference.¹

The isotherms in the range 100-360 C were calculated on the basis of the 300 bar values that are described above and listed in table 1. A change in this base value would affect the calculated values at all other pressures by the same amount in cc/g, within the limits of precision of the measurements.

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TABULATION OF RESULTS

In order to facilitate interpolation between isotherms, and to bring out any errors or any necessity for smoothing, the approximate thermal expansions, $\Delta v/\Delta T$, were plotted against temperature for each isobar. In only one place, near the upper end of the 180 C isotherm, was any smoothing between isotherms indicated by these plots. Interpolations could be made with certainty to derive a table extending to 360 C. With the help of the data of Kirillin, Rumiantsev and Zubarev, the best values for the isotherms at 380 and 400 C could be deduced. The final result is shown in table 2.

COMPARISON WITH THE RESULTS OF OTHER INVESTIGATORS

The results along the 300 bar base line are compared with other measured values, and shown in figure 2. In this connection the values we list below 300 bars cannot be given much weight, inasmuch as we did not measure enough points at low pressure. In fact, it was necessary to depend upon the data of Smith and Keyes to indicate the detailed trend at low pressure above 300 C. At lower temperatures the values listed represent essentially a linear extrapolation of the $\Delta p/\Delta v$ plot. At all temperatures the values of $\Delta p/\Delta v$ derived from the measurements of Smith and Keyes are slightly but consistently above ours, and those of Kirillin, Rumiantsev, and Zubarev are too irregular to make any valid comparison. In the range 100-300 C our measurements of the volume at 300 bars (table 1) generally lie a little above those *tabulated* by Smith and Keyes, and therefore values of the saturation specific volume derived by our extrapolation would be even higher than those of Smith and Keyes. However, our data are still consistent with those derived by calorimetry. The calorimetric measurements by Osborne, Stimson, and Fiock (1930) and of Osborne, Stimson, and Ginnings (1937) give a function β , from which it is possible to calculate the specific volume of liquid water:

$$v_s = \frac{\beta}{T(dp/dT)_{sat}} \quad (5)$$

using only the well-established slope of the saturation line. These authors accepted the formulated and tabulated values of Smith and Keyes in making their final correlation (Osborne, Stimson, and Ginnings, 1939), but an evaluation of their original data gives specific volumes generally above those tabulated by Smith and Keyes. The difference amounts to about 1 or 2 standard deviations of the calorimetric data. Although this cannot be considered particularly significant, it is in the right direction to conform with our extrapolation.

At the lower end of the temperature range studied, the data are consistent in magnitude and trend with recently published data for the range 0-100 C (Kennedy, Knight, Holser, 1958).

The values reported earlier by Kennedy (1950) for temperatures up to 300 C are closely confirmed by the present measurements, even at 1400 bars. At 400 C the new values are somewhat lower at low pressures and higher at high pressures, in line with a trend measured on the isotherms at 420 C and above (Holser and Kennedy, 1958).

Although the specific volumes measured by Kiyama, Kinoshita and Kitahara (1955) at 300 bars (380 and 400 C) are so much greater than our smoothed values that they cannot be plotted on figure 2, agreement is better with us at high pressure. The single isotherm by Amagat (1893) at 198 C differs greatly from all other data, although his measurements below 100 C were generally concordant with ours. The measurements of Tamman and Ruhenbeck (1932) are very much smaller than ours at 200 C, and very much larger at high temperatures. It would appear unwise to give their data any weight.

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