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Accurate and precise lattice parameters of H2O and D2O ice I*h* **between 1.6 and 270 K from high-resolution time-of-flight neutron powder diffraction data**

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Electronic Supplementary Text and Figures

Accurate and precise lattice parameters of H_2O and D_2O ice I*h* between 1.6 and 270 K from high-resolution time-of-flight neutron powder diffraction data.

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<center>Electronic Supplementary Text and Figures</center>

1. Use of infrared thermography to evaluate thermal gradients in the samples.

As described in the main text, and shown in Figure S1, a slab can was fitted with a RhFe resistance thermometer inside the sample cavity (introduced through the back window). The purpose of this was to measure the temperature difference between the centre of the sample and that measured by the RhFe sensor that is routinely inserted into the aluminium frame of the sample holder. As the thermal diffusivity of the sample (particularly in its powdered state) is typically much smaller than that of the aluminium frame, there will be a lag between the sample temperature that requires a period of equilibration once the temperature of the holder reaches and controls at the set point.
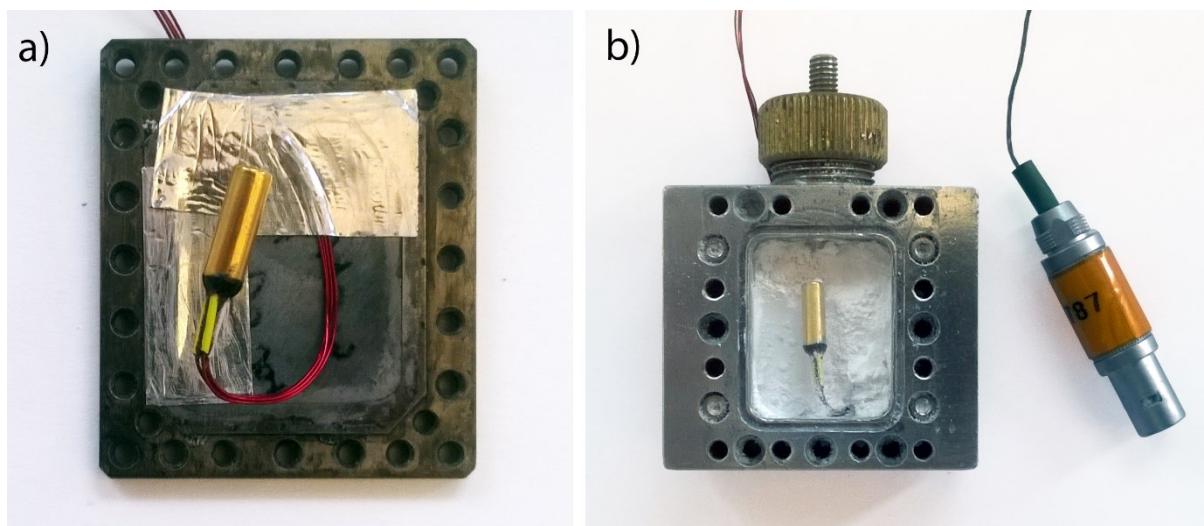

Figure S1. (a) The internal sensor wired through a standard vanadium window. The package is 10 mm long and 3.1 mm in diameter. (b) The assembled can, half filled with MgO powder.

Since it is considerably more convenient to load, and since I wished to carry out *ex situ* thermal imaging at and above room temperature, I chose to perform these tests with MgO powder instead of ice. MgO is a reasonable proxy in terms of its thermal diffusivity for a mixture of ice and silicon. At 295 K the thermal diffusivity, α, of MgO is 15.5 x 10^{-6} m^2 s^{-1} (Hoffmeister, 2014). A crude estimate of the thermal diffusivity of the ice-silicon mixture may be obtained from scaling by their volume abundances; thus for an estimated 75:25 mix of ice (15.4 x10^{-7} m^2 s^{-1}: e.g., Slack, 1980) and silicon (8.5 x 10^{-5} m^2 s^{-1}: Glassenbrenner & Slack, 1964) by volume, I find $\alpha \approx 22$ x 10^{-6} m^2 s^{-1}. These quantities are, of course, representative of the <u>bulk</u> crystalline solid and will be considerably smaller in the powder.

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For snow with a bulk density comparable to that of my powdered ice specimens (~0.7 g cm^{-3}), the formulas given by Sakatume & Seki (1978) and Fukusako (1990) indicate a thermal *conductivity* ~ 0.75 of the bulk crystalline value. Hence, the thermal diffusivity of MgO is probably very similar to a mixture of ice and silicon, at least at higher temperatures. It is evident, furthermore, that adding silicon does far more than ensure accuracy of the lattice parameters, it also ensures faster equilibration and hence more accurate temperatures by increasing the thermal diffusivity of the sample.

Both the frame temperature and the embedded sample temperature were logged with the MgO sample mounted in a cryostat as a variety of different thermal cycles were performed. These included free cooling (i.e., following the CCR down in temperature without internal heating), controlled cooling (with PID-controlled halts every 20 K), and several cycles of controlled warming.

These data reveal a near uniform offset between the frame and sample temperatures when the system is at room temperature and not being actively heated, as well as when the system is being actively controlled below room temperature, of 110 ± 140 mK.

Since the two resistance thermometers only sample the temperature at two locations, infrared thermography was used to identify any significant spatial temperature variations, resulting from packing inhomogeneity for example. Mid-range thermal imaging equipment allows images with a spatial resolution of better than 1 mm per pixel to be obtained from a distance of 30 cm and with a precision of < 0.1 K. Inevitably, imaging must be done outside the cryostat and with one of the vanadium windows removed so that the sample may be viewed directly. Consequently, tests were done in the open air to evaluate the cell performance on heating and cooling in the range 295 – 340 K, both with and without one of the windows attached. Unsurprisingly, thermal control is very poor with one window removed, and so images could only be acquired during brief exposures of the sample.

Images were acquired with a FLIR E8 camera, supplied by Red Current Ltd., UK. The camera captures images on an uncooled micro-bolometer with 320 x 240 pixels, through a fixed lens of 45 x 34° field of view, and with a stated precision of 0.06 K.

Figure S2 shows representative thermal images collected during the warming from 295 to 340 K (a) and (b) and once the 340 K setpoint was reached and both the frame and embedded sensors were in equilibrium (c). Images were post-processed in FLIR Tools; an emissivity of 0.78 was applied in order to obtain accurate temperatures on the sample.

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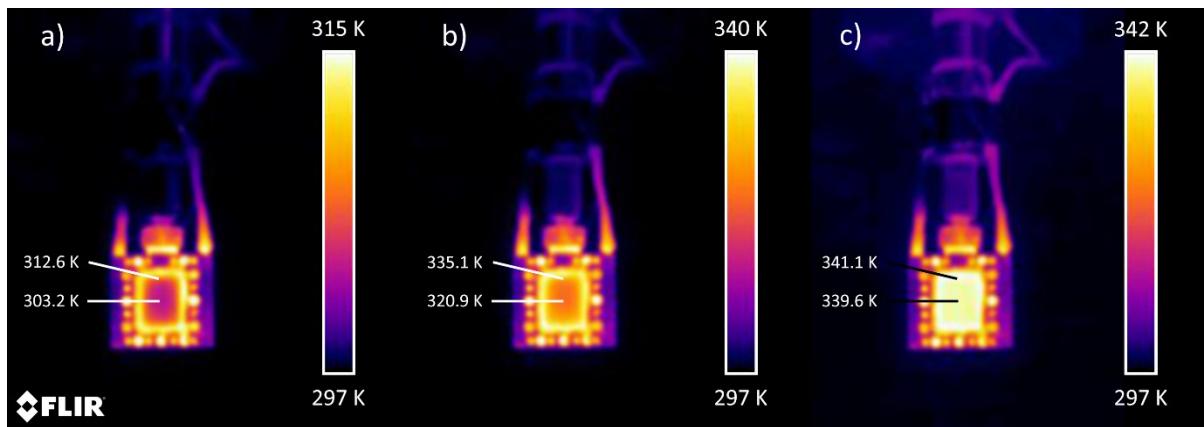
Figure S2. Thermal images, captured using a FLIR E8 camera, of the heated sample holder and the exposed sample. The cartridge heater is on the right and the RhFe sensor mounted in the frame is on the left of each image. Since proper heating of the sample requires the window to be attached, this was removed momentarily for the images to be captured.

Thermal gradients of $10 - 15$ K between the edge and the centre of the sample are typical of the heating phase, and accurately reflect the magnitude of the thermal lags seen in the resistance thermometers. Once controlling at 340 K, however, the gradients observed are similar to 1 K.

Closer examination of 'hot' spots around the edge of the sample (Figure S3) shows that they are spatially correlated with loose and disturbed powder (compare the gouges in the visible-light image with the warm regions in the thermal image). This disturbed powder is the result of applying a soft brush during loading to clean up excess powder and then removal of the window to capture thermal images.

Where the powder is well packed in the middle of the exposed face, thermal gradients are small. In particular, the oval region where the relative neutron flux is > 85 % of its peak value (indicated on the intensity map in Fig. S3) has a temperature of 340.0 ± 0.2 K.

To summarise, thermal imaging of well consolidated specimens reinforces the observations from the embedded resistance thermometer that there no significant thermal gradients in the region sampled by the most intense portion of the incident neutron beam.
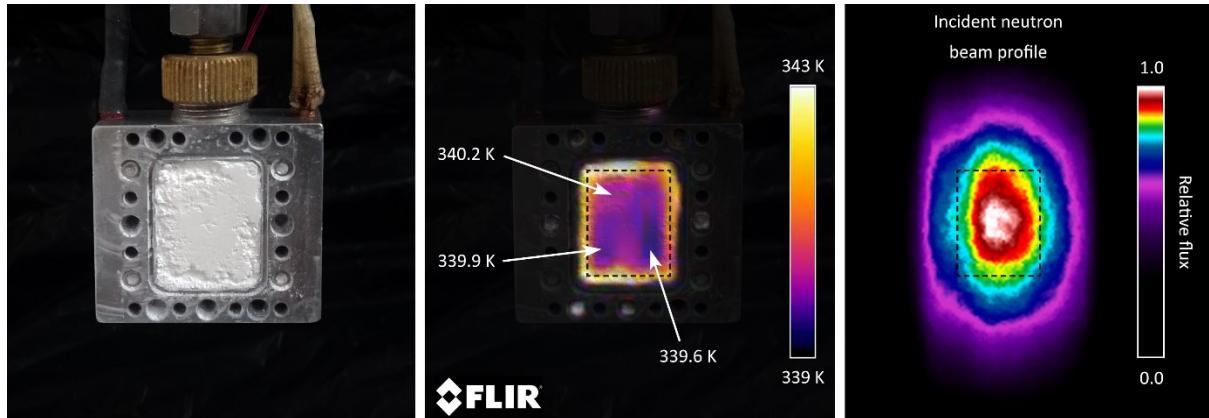
Figure S3. The central thermal image is from Figure S2c, with the thermal contrast stretched to highlight variations across the surface of the exposed sample. Spatial resolution is 0.6 mm per pixel in this image acquired from a distance of < 25 cm. The thermal image is superimposed on a visible-light image of the sample can (also shown on the left for clarity, with a pixel resolution of 0.06 mm). The black dashed line shows the region of the sample that is actually illuminated by the neutron beam. The panel on the right shows a direct image of the actual neutron flux distribution in HRPD's standard 30 – 130 ms time-of-flight and with the standard vertical and horizontal jaw gaps equal to 20 and 15 mm, respectively (Neutron camera provided courtesy of Dr Craig Bull, ISIS).

2. Additional supplementary plots
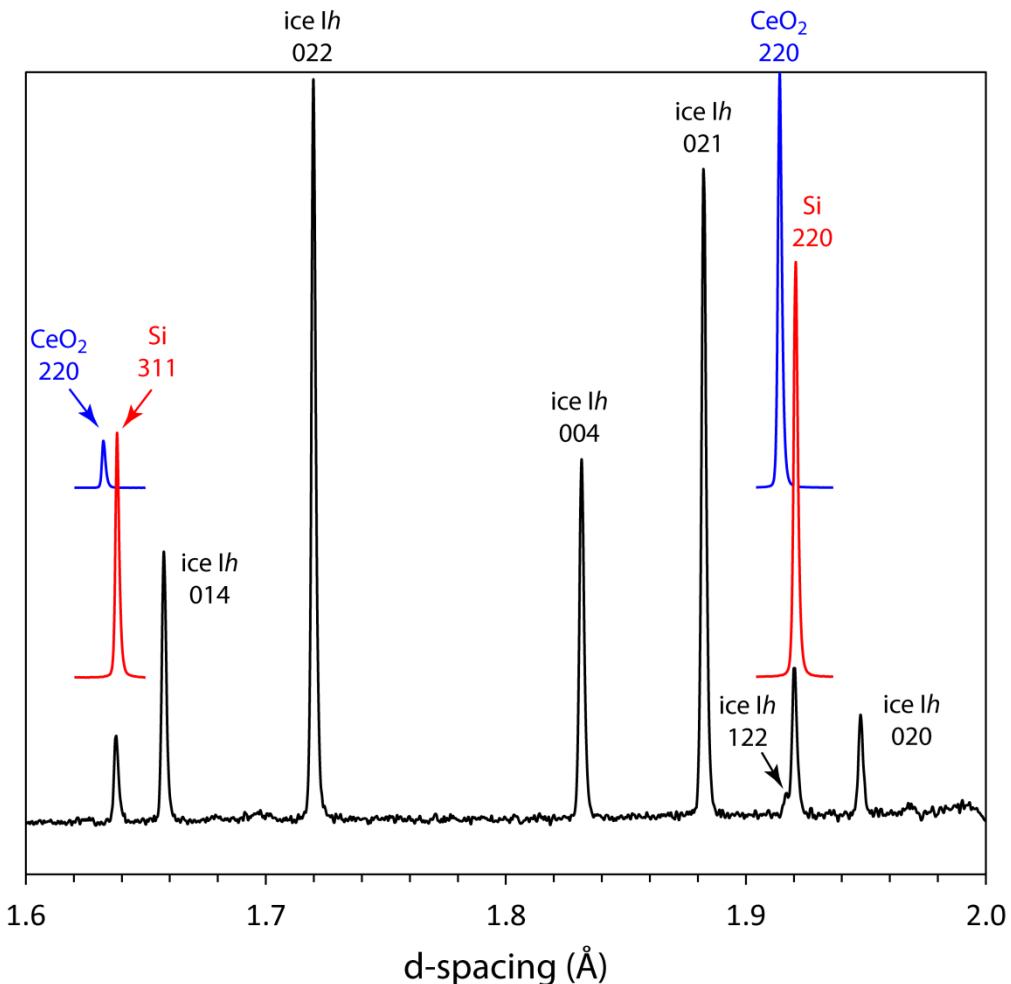
Figure S4: Segment of powder diffraction data collected from D_2O ice Ih at 2 K (D_2O series 2), measured in HRPD's backscattering detectors. Shown for comparison in red and blue are calibration measurements collected from NIST silicon and from a ceria pellet (note that only two reflections from each are present in this range), the latter being used on HRPD as a line-shape standard to define the instrumental resolution. This clearly shows that the ice Bragg peaks are of comparable width to the sharp line standards and exhibit no *hkl*-dependent broadening or asymmetry.
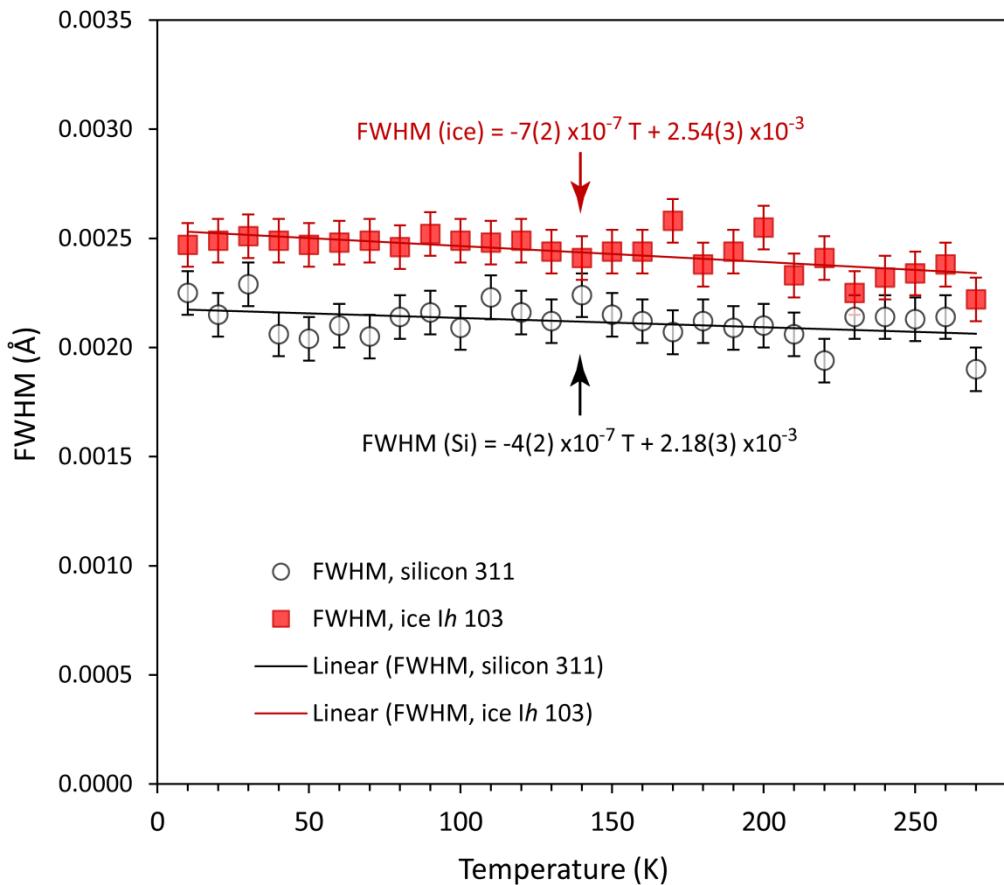
Figure S5: Extracted full width at half maximum (FWHM) for Bragg peaks from ice and silicon in the backscattering data for D_2O series 1. Note that the FWHM of the ice reflection is only slightly larger than that of the silicon reflection, both exhibit essentially the same temperature dependence and neither exhibits any anomalous kinks or changes in slope. This is good evidence that the ice does not preserve any significant strain from the cooling used to prepare it, does not undergo any obvious relaxation (such as might occur by annealing out of defects) and shows no correlation with anomalies observed in the axial ratios.
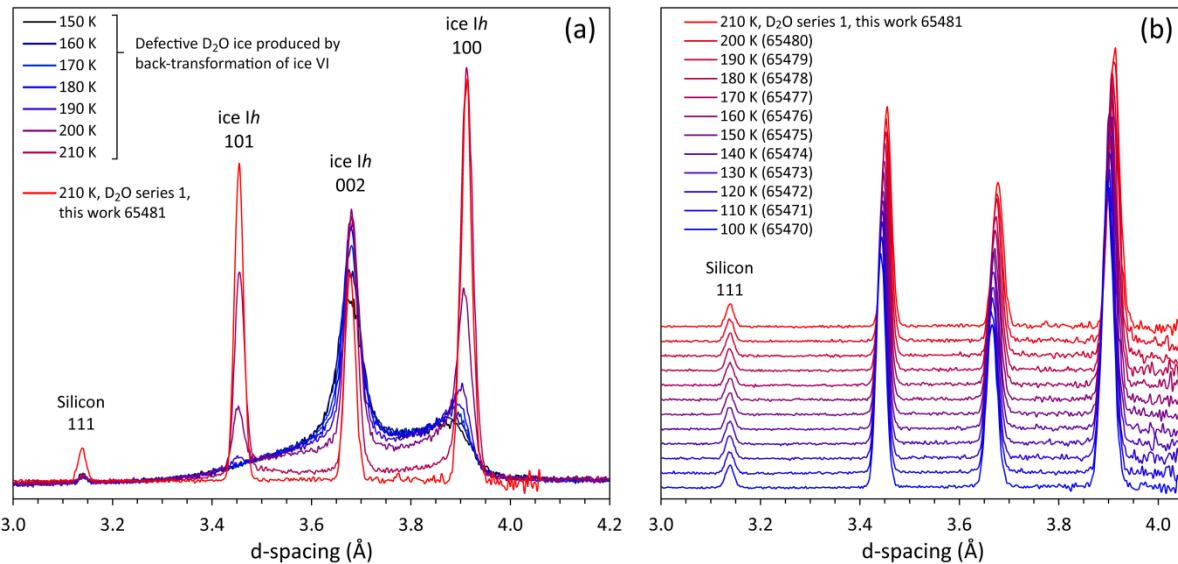
Figure S6: (a) Multiplot showing powder diffraction data acquired in HRPD's 90° detector banks (thus at lower resolution than the backscattering data shown elsewhere) during the back-transformation of recovered D_2O ice VI. The data, beginning at 150 K, reveal the formation of high-cubicity stacking-defective ice that anneals on warming to form progressively less defective material. At 210 K, where this experiment was ended, the ice is substantially hexagonal in nature and the manifestation of some residual cubicity is apparent only in a slight broadening of 002 and in the meniscuses of diffuse scattering between the Bragg peaks. For comparison, a dataset measured at 210 K in this work is overlaid, which shows that the peaks are sharper, 101 is more intense and the diffuse scattering is absent. This shows that the ice measured in this work, from which all of the analysis follows, was wholly or substantially hexagonal in character and did not possess stacking faults at the level where they would be detectable. (b) Multiplot depicting the diffraction data from this work (D_2O series 1) collected on warming from 100 – 210 K; the uppermost diffraction pattern is thus the same one shown in the panel on the left. Apart from a shift to longer d-spacings due to thermal expansion, the Bragg peaks exhibit no variation in intensity or width.

The ice I_{SD} powder data are reproduced with the kind permission of Prof. Ian Wood, University College London.
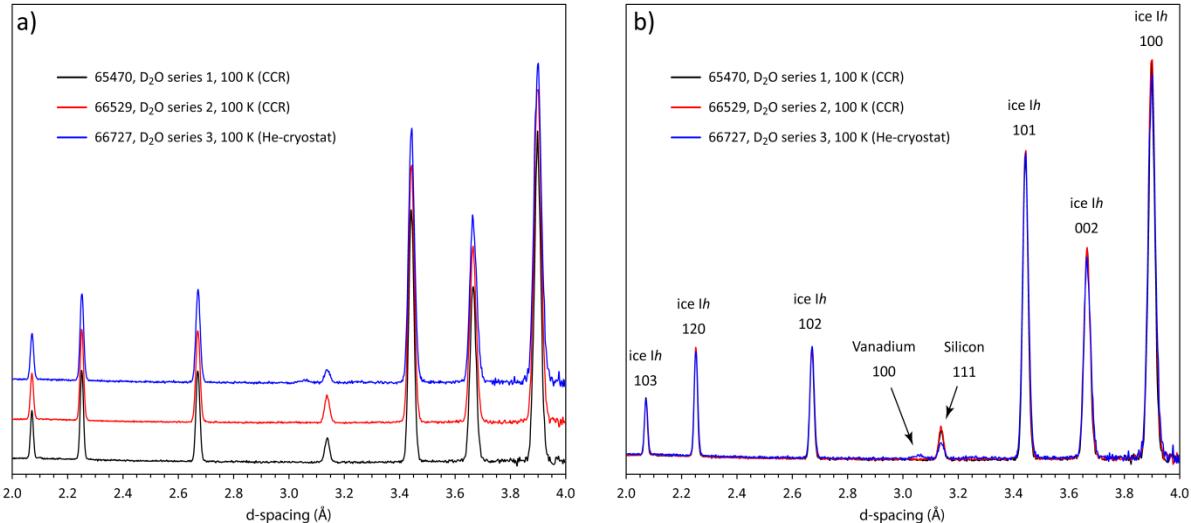
Figure S7: Data measured at 100 K in HRPD's 90° detectors from the three different D_2O ice I*h* specimens, shown separated on the left and collapsed on the right. This reinforces the observation from Figure S6 that the peaks are sharp and have no residual diffuse intensity and further illustrates that the same is true of all three D_2O samples. Indeed the peak widths, intensities and backgrounds of all three specimens are nearly identical. Small differences in the data are restricted to the silicon 111 peak (due simply to different amounts of silicon powder being mixed with the ice) and a very weak vanadium peak in the specimen measured using a helium cryostat due to the presence of additional V-foil windows compared with the closed-cycle refrigerator.
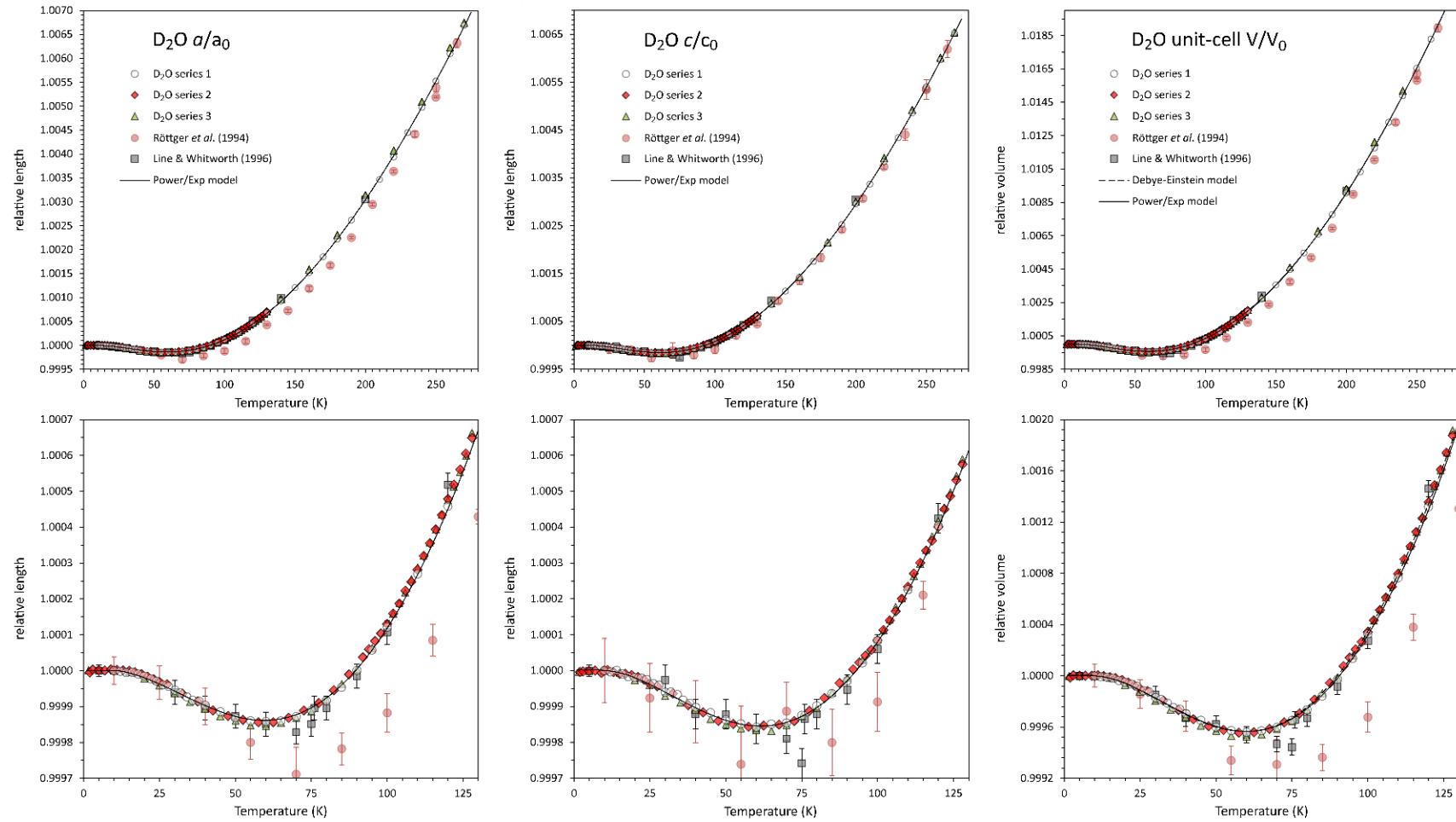
Figure S8: Lattice parameters of D_2O ice Ih referenced to their 10 K values (since this was only low-T datum in common between all three samples), although described as a/a_0 etc. There is very good agreement between the relative values of the three new series, as well as with the dataset measured on HRPD twenty-two years ago (Line & Whitworth, 1996). Agreement with Röttger *et al*. (1994, 2012) is poorer.
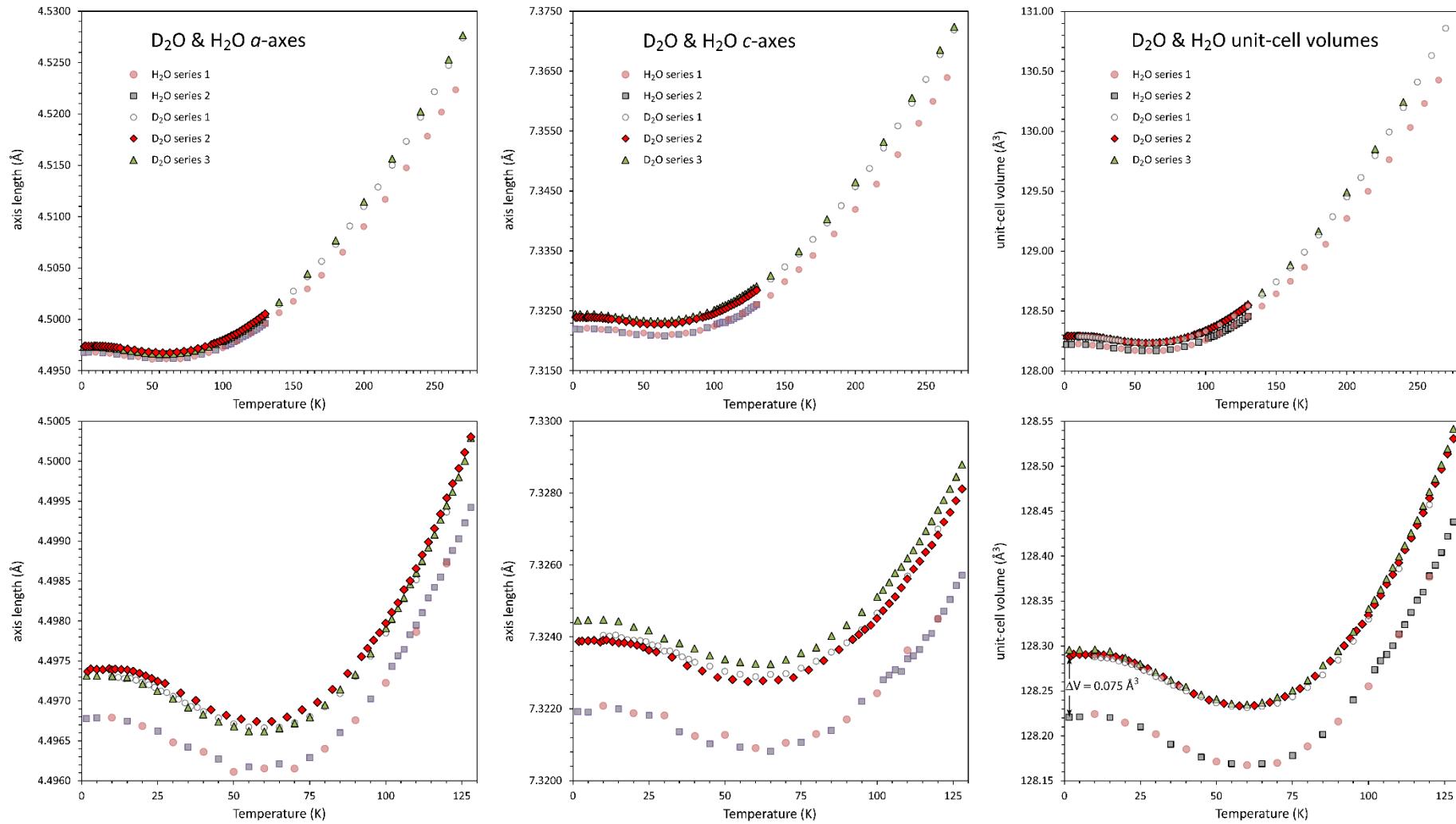
Figure S9: Comparison of the absolute lattice parameters of H_2O and D_2O ice Ih.
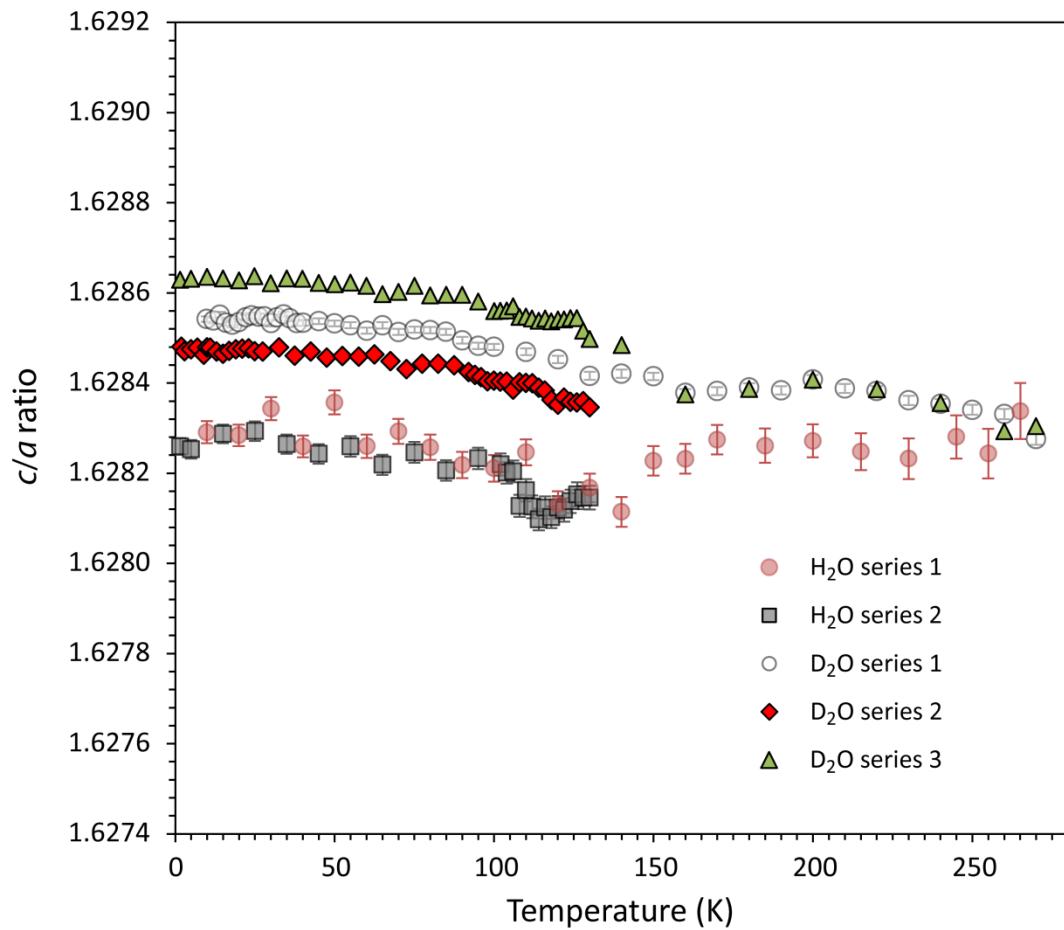
Figure S10: Comparison of the c/a ratios of H_2O and D_2O ice Ih.
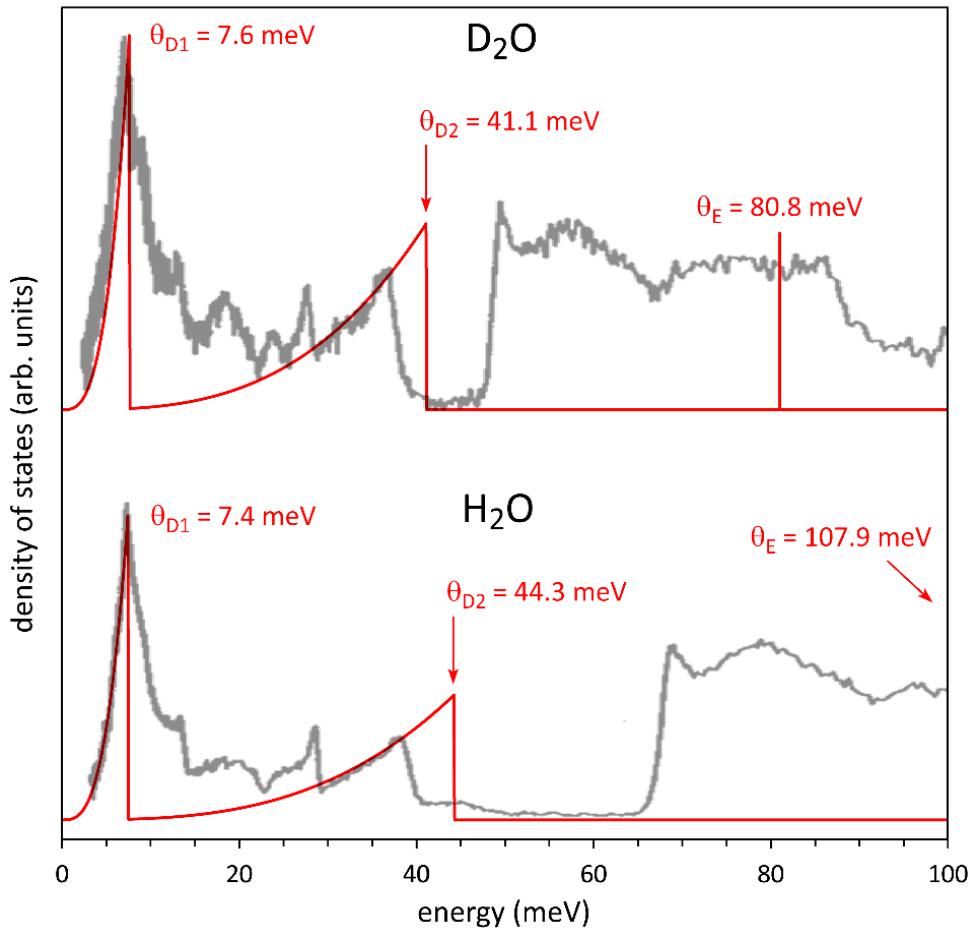
Figure S11: Comparison of measured and model phonon densities of states. The grey lines show the vibrational densities of states obtained by inelastic neutron spectroscopy (Li, 1996). The solid red lines a representative of the double-Debye + Einstein oscillator model used to fit the volume thermal expansion. The two Debye terms at lower frequencies, θ_{D1} and θ_{D2}, correspond rather well with the gross features of the measured spectrum, particularly in relation to the transverse acoustic peak at ~ 7 meV that controls the negative expansion at low temperatures. Conversely, the delta function that represents the Einstein term (θ_E) is a much poorer (and entirely *ad hoc*) representation of the higher frequency portion of the spectrum.
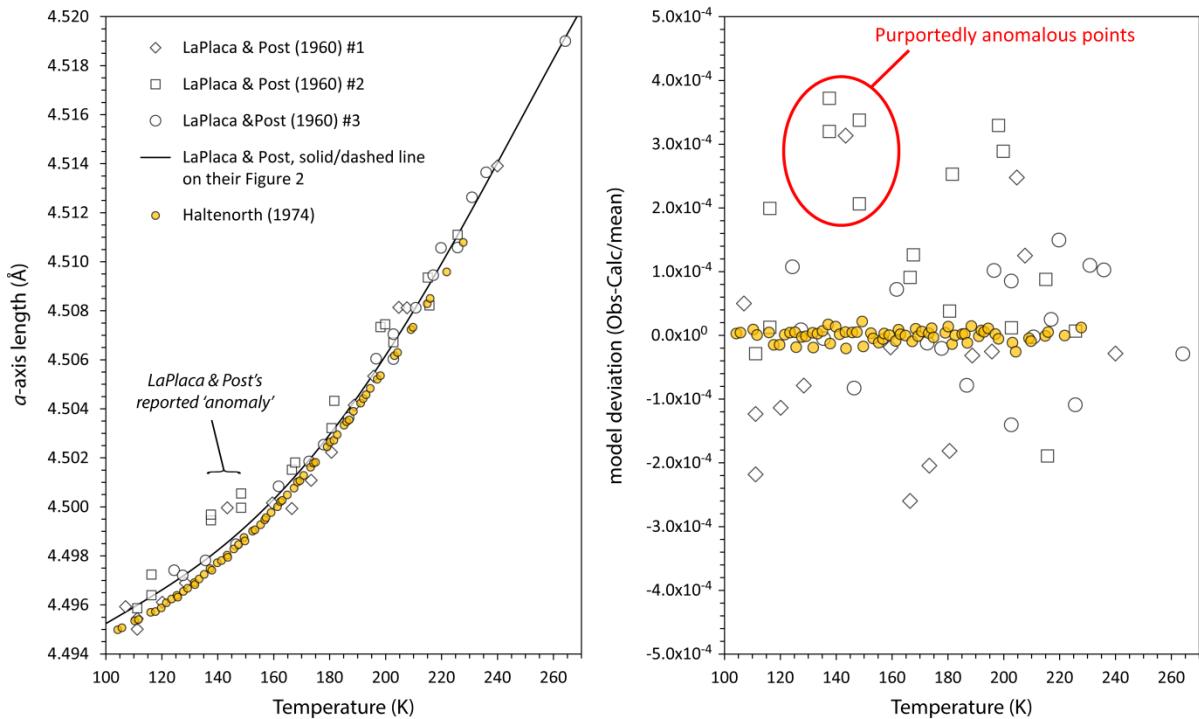
Figure S12: Left, plot of 'raw' *a*-axis lengths from the paper of LaPlaca & Post (1960) and the thesis of Haltenorth (1974). The symbols from LaPlaca & Post's work correspond to the three reported data series that they measured over a period of several months and the solid line corresponds with the solid/dashed line in their Figure 2. Highlighted are the points that are described as a reproducible plateau or anomaly.

One the right is shown the relative deviation of the symbols from the solid line in LaPlaca & Post (1960); it is thus apparent that the anomalous points are not statistically significantly different from the other measurements and that the purported anomaly is not real. The greater *perceived* deviation of the points around 140 K compared with those around 200 K in the left-hand figure is probably due to the difference in slope of the trend line, resulting in an optical illusion.

For comparison, the scatter in Haltenorth's data about a fitted third-order polynomial are an order of magnitude smaller, being a few x 10^{-5}. As Haltenorth states at multiple points in the text of his thesis, there is no *a*-axis anomaly comparable with that reported by LaPlaca and Post.
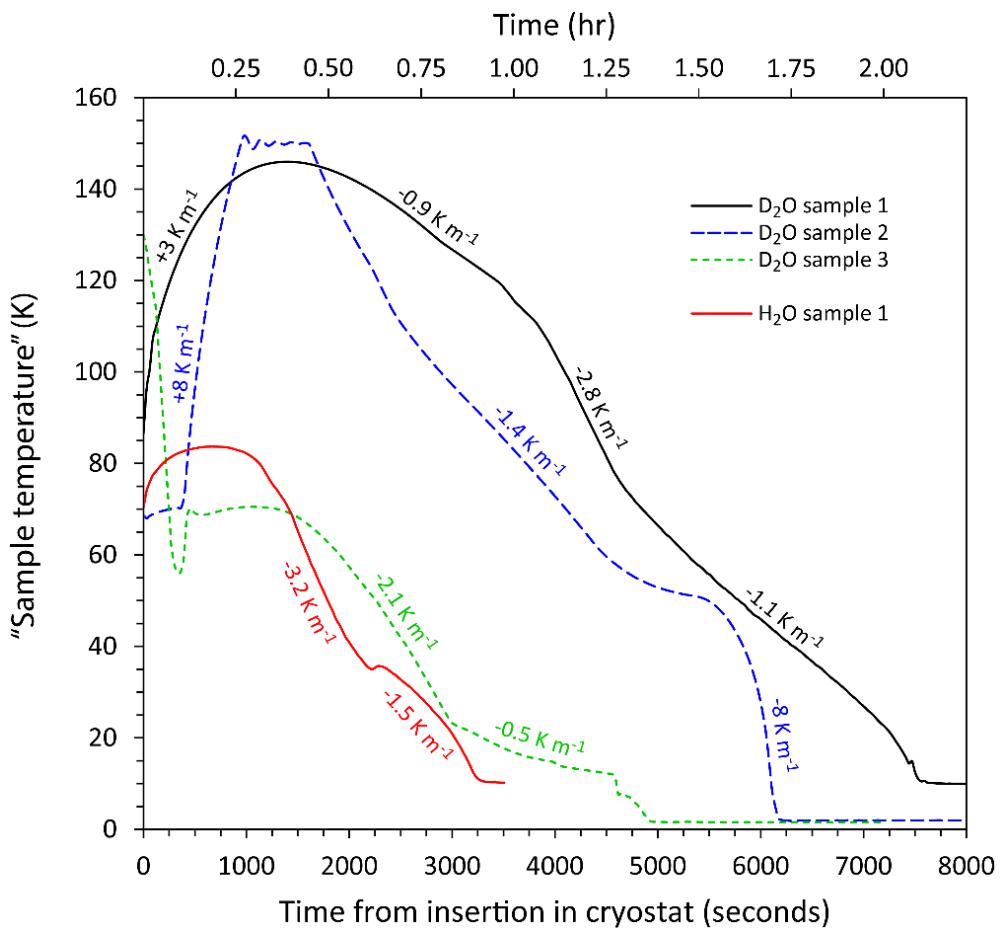
Figure S13: Plot of the logged sample temperatures after insertion in the cryostat. Variations in cooling rates reflect manual adjustments in exchange-gas pressure (CCR) or helium flow rates ("Orange" cryostat) as well as the intrinsic variations in the sample holder's heat capacity.

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Table S1: Lattice parameters of H_2O ice Ih as a function of temperature.

H₂O series 1			H₂O series 2		
T (K)	a (Å)	c (Å)	T (K)	a (Å)	c (Å)
9.94(2)	4.49679(3)	7.32208(10)	1.559(3)	4.49678(3)	7.32192(7)
19.94(3)	4.49668(3)	7.32188(10)	4.99(4)	4.49679(3)	7.32191(8)
30.08(3)	4.49648(3)	7.32181(10)	14.97(4)	4.49675(3)	7.32200(8)
40.09(2)	4.49636(3)	7.32124(10)	25.03(4)	4.49662(3)	7.32182(9)
49.98(1)	4.49611(3)	7.32127(11)	34.95(2)	4.49642(3)	7.32136(8)
60.06(1)	4.49615(3)	7.32091(10)	45.01(1)	4.49627(3)	7.32102(9)
70.00(1)	4.49615(3)	7.32105(11)	54.97(1)	4.49617(3)	7.32094(9)
79.97(2)	4.49640(4)	7.32130(11)	64.98(1)	4.49621(3)	7.32081(9)
90.02(1)	4.49676(4)	7.32170(12)	75.03(1)	4.49629(3)	7.32107(9)
100.00(1)	4.49722(4)	7.32243(12)	84.95(1)	4.49660(3)	7.32139(9)
110.03(1)	4.49786(4)	7.32363(12)	95.00(1)	4.49703(3)	7.32220(9)
120.02(2)	4.49872(4)	7.32450(12)	102.04(1)	4.49743(3)	7.32281(10)
130.02(1)	4.49958(4)	7.32608(13)	103.99(1)	4.49757(3)	7.32294(9)
139.96(2)	4.50065(4)	7.32757(13)	106.03(3)	4.49765(3)	7.32308(10)
149.98(2)	4.50175(4)	7.32987(13)	107.98(1)	4.49783(3)	7.32304(10)
159.98(1)	4.50296(4)	7.33186(13)	110.03(1)	4.49795(3)	7.32339(10)
169.95(1)	4.50428(4)	7.33421(13)	111.97(1)	4.49810(3)	7.32348(10)
184.99(2)	4.50653(5)	7.33781(15)	114.01(2)	4.49829(3)	7.32365(9)
199.96(3)	4.50903(5)	7.34192(15)	116.05(2)	4.49842(3)	7.32399(10)
215.04(1)	4.51169(5)	7.34614(16)	117.99(1)	4.49855(3)	7.32410(10)
230.04(2)	4.51475(6)	7.35106(18)	120.02(1)	4.49874(3)	7.32450(10)
244.96(1)	4.51783(6)	7.35630(19)	121.97(1)	4.49888(4)	7.32472(10)
254.99(2)	4.52018(7)	7.35995(22)	124.00(2)	4.49903(4)	7.32504(10)
265.03(2)	4.52235(8)	7.36391(25)	126.03(1)	4.49923(4)	7.32543(11)
			127.98(1)	4.49942(4)	7.32571(10)
			130.01(2)	4.49963(4)	7.32605(10)

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Table S2: Lattice parameters of D$_2$O ice Ih as a function of temperature.

D$_2$O series 1			D$_2$O series 2			D$_2$O series 3		
T (K)	a (Å)	c (Å)	T (K)	a (Å)	c (Å)	T (K)	a (Å)	c (Å)
9.93(2)	4.497301(9)	7.324044(23)	1.99(2)	4.497366(8)	7.323872(22)	1.601(0)	4.497313(7)	7.324454(19)
12.02(2)	4.497293(10)	7.324017(24)	2.97(3)	4.497401(8)	7.323884(21)	5.00(4)	4.497313(10)	7.324465(26)
14.03(3)	4.497278(10)	7.324049(24)	4.98(3)	4.497393(8)	7.323892(22)	10.02(4)	4.497310(10)	7.324478(26)
15.94(2)	4.497282(9)	7.323976(24)	6.97(2)	4.497389(8)	7.323902(22)	14.97(6)	4.497294(10)	7.324435(26)
17.91(2)	4.497259(10)	7.323919(25)	9.02(2)	4.497403(8)	7.323854(22)	20.04(5)	4.497210(10)	7.324279(27)
19.95(3)	4.497233(9)	7.323902(23)	9.98(5)	4.497388(8)	7.323901(22)	24.89(3)	4.497124(10)	7.324183(28)
22.12(2)	4.497200(9)	7.323896(23)	11.03(2)	4.497396(8)	7.323913(21)	29.99(2)	4.497028(10)	7.323956(26)
23.97(2)	4.497170(9)	7.323865(24)	12.99(5)	4.497392(8)	7.323868(21)	35.03(4)	4.496918(10)	7.323822(26)
26.12(2)	4.497122(9)	7.323777(24)	15.05(2)	4.497386(8)	7.323835(22)	39.95(3)	4.496831(10)	7.323679(26)
27.99(3)	4.497104(9)	7.323749(24)	16.91(2)	4.497370(8)	7.323837(21)	45.00(4)	4.496739(10)	7.323486(27)
30.08(3)	4.497055(9)	7.323601(25)	19.02(2)	4.497343(8)	7.323809(21)	50.06(3)	4.496681(10)	7.323379(27)
31.87(2)	4.497021(9)	7.323606(24)	21.06(2)	4.497313(8)	7.323768(22)	54.98(4)	4.496621(10)	7.323297(26)
34.00(3)	4.496973(9)	7.323554(24)	23.08(1)	4.497280(8)	7.323718(22)	60.04(5)	4.496615(11)	7.323256(27)
35.91(3)	4.496929(9)	7.323444(24)	24.99(2)	4.497244(8)	7.323630(22)	65.02(5)	4.496657(10)	7.323242(27)
38.06(1)	4.496918(9)	7.323378(24)	27.42(2)	4.497218(8)	7.323583(21)	70.02(4)	4.496719(10)	7.323364(27)
40.08(2)	4.496865(9)	7.323296(24)	32.56(2)	4.497099(8)	7.323431(22)	75.03(5)	4.496795(10)	7.323551(28)
45.00(2)	4.496786(10)	7.323184(25)	37.55(1)	4.497005(8)	7.323196(22)	80.00(3)	4.496949(10)	7.323706(27)
49.97(2)	4.496712(9)	7.323040(25)	42.54(1)	4.496889(8)	7.323049(22)	85.05(1)	4.497143(10)	7.324027(27)
54.97(1)	4.496673(10)	7.322959(25)	47.57(2)	4.496820(9)	7.322874(23)	90.00(1)	4.497328(11)	7.324327(28)
60.07(2)	4.496664(10)	7.322890(26)	52.45(1)	4.496771(8)	7.322812(22)	95.019(6)	4.497593(11)	7.324693(29)
64.98(1)	4.496671(10)	7.322955(26)	57.54(1)	4.496741(9)	7.322756(23)	100.04(1)	4.497910(11)	7.325113(28)
70.02(2)	4.496726(10)	7.322978(25)	62.49(1)	4.496741(9)	7.322779(23)	101.986(3)	4.498025(12)	7.325303(31)
75.03(1)	4.496804(10)	7.323129(26)	67.56(1)	4.496796(9)	7.322800(24)	104.019(4)	4.498158(11)	7.325522(29)

79.97(1)	4.496927(10)	7.323324(27)	72.51(1)	4.496887(9)	7.322870(23)	105.96(1)	4.498288(11)	7.325777(28)
84.95(1)	4.497092(10)	7.323575(26)	77.52(1)	4.496981(9)	7.323077(24)	108.00(1)	4.498459(11)	7.325950(29)
90.02(2)	4.497304(11)	7.323836(28)	82.46(1)	4.497143(9)	7.323339(24)	110.04(1)	4.498602(11)	7.326186(28)
95.00(2)	4.497560(10)	7.324199(27)	87.47(1)	4.497341(9)	7.323647(24)	111.965(5)	4.498750(11)	7.326408(30)
100.00(2)	4.497848(10)	7.324656(27)	91.995(7)	4.497554(9)	7.323926(24)	114.01(1)	4.498917(11)	7.326657(30)
110.04(1)	4.498515(11)	7.325694(29)	93.999(7)	4.497658(9)	7.324066(25)	116.04(1)	4.499080(12)	7.326945(30)
120.03(1)	4.499362(12)	7.326998(31)	95.996(8)	4.497758(9)	7.324208(24)	117.98(1)	4.499267(11)	7.327220(29)
130.02(2)	4.500374(12)	7.328481(34)	97.974(5)	4.497854(9)	7.324323(24)	120.01(1)	4.499446(11)	7.327530(29)
139.96(3)	4.501466(13)	7.330281(35)	99.995(4)	4.497970(9)	7.324516(25)	121.954(6)	4.499615(11)	7.327808(29)
149.98(4)	4.502739(12)	7.332327(32)	101.957(4)	4.498105(9)	7.324729(25)	123.991(5)	4.499798(12)	7.328119(32)
159.98(2)	4.504151(12)	7.334461(32)	104.012(4)	4.498227(9)	7.324927(25)	126.02(1)	4.500004(11)	7.328452(30)
169.95(1)	4.505642(13)	7.336908(34)	105.968(5)	4.498390(9)	7.325113(25)	127.97(2)	4.500290(12)	7.328795(31)
180.00(1)	4.507303(13)	7.339648(35)	108.01(2)	4.498501(9)	7.325367(25)	130.00(2)	4.500506(11)	7.329063(30)
190.01(1)	4.509088(13)	7.342526(36)	109.96(1)	4.498655(9)	7.325612(25)	140.00(7)	4.501665(12)	7.330888(31)
199.96(1)	4.510977(14)	7.345711(38)	111.998(5)	4.498825(9)	7.325885(25)	159.99(2)	4.504431(13)	7.334900(32)
209.98(1)	4.512887(14)	7.348732(39)	114.036(5)	4.498986(9)	7.326100(26)	179.99(3)	4.507680(14)	7.340247(36)
219.97(1)	4.515012(15)	7.352165(41)	115.976(3)	4.499158(9)	7.326353(26)	200.00(3)	4.511433(14)	7.346450(38)
230.05(1)	4.517319(15)	7.355829(39)	118.010(4)	4.499337(10)	7.326552(27)	220.02(3)	4.515627(15)	7.353185(40)
240.05(1)	4.519682(15)	7.359641(41)	120.045(5)	4.499539(10)	7.326831(26)	240.07(3)	4.520216(16)	7.360518(42)
249.97(1)	4.522151(15)	7.363602(44)	121.984(6)	4.499719(10)	7.327195(26)	260.02(3)	4.525293(18)	7.368502(46)
260.01(2)	4.524728(16)	7.367756(46)	124.02(1)	4.499909(10)	7.327463(27)	269.99(3)	4.527646(18)	7.372385(48)
269.96(1)	4.527401(17)	7.371856(50)	125.96(1)	4.500110(10)	7.327785(28)			
			128.000(6)	4.500305(10)	7.328114(27)			
			130.032(4)	4.500536(10)	7.328430(26)			

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Table S3: Historical and contemporary data on the density of H$_2$O ice Ih at its melting point.

Specific gravity	Density (g cm^{-3})	Reference	Note
82 : 91⅛	~0.900	Boyle (1665)	
8 : 9	~ 0.888	Musschenbroek (1734)	
0.905	~ 0.905	Krafft (1746)	
11 : 12	~ 0.917	Mairan (1749)	
14 :15	~ 0.937	Irving, reported by Phipps (1774)	
0.945	0.945	Williams (1790)	(1)
243 : 272	0.8934	Heinrich (1806)	(2)
87 : 95	0.9158	— ″ —	
120 : 131	0.9160	— ″ —	
0.885	0.885	Meineke (1815)	(3)
14 : 15	~ 0.937	Scoresby (1818)	
0.920 ‡	0.919	Thomson (1818)	
0.915–0.925	0.915–0.925	Scoresby (1820)	
0.950 †	0.950	Le Royer & Dumas (1821)	
0.9268 *	0.9267	Osann (1830)	
0.9184 *	0.9183	Playfair & Joule (1845)	
0.9180 *	0.9179	Brunner (1845)	
0.91580 *	0.91565	Plücker & Geißler (1852)	
0.9078 *	0.9077	Kopp (1855)	
0.9175(7) *	0.9174(7)	Dufour (1860)	
0.9178(5) *	0.9177(5)	Dufour (1862)	
0.918–0.922 †	0.918–0.922	Duvernoy (1870)	
0.91674 †	0.91674	Bunsen (1870)	
0.91686	0.91686	Petterson (1883)	
0.916660	0.916660	Zakrzevski (1892)	
0.91614(2) *	0.91599(2)	Nicholls (1899)	(4)
0.91615(9) *	0.91600(9)	— ″ —	(5)
0.91807(4) *	0.91792(4)	— ″ —	(5)
0.91661(7) *	0.91646(7)	Barnes (1901)	
–	0.9160	Vincent (1902)	
0.91599**	0.91599	Dewar (1903)	
0.9176 *	0.91745	Leduc (1906)	
–	0.9157	Endo (1925)	(6)
–	0.91671(5)	Ginnings & Coruccini (1947)	
–	0.91680(4)	Dantl & Gregora (1968)	
–	0.9178(2)	Alimova *et al.* (1987)	
–	0.9159(5)	'Thermodynamic value'	(7)
–	0.91658(7)	This work (by extrapolation)	

* Relative to water at 0 °C

† Relative to water at 4 °C

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‡ Relative to water at 15.56 °C (specifically, 60 °F).

**Reference water temperature not stated, assumed to be 4 °C.

(1) The specific gravity is actually calculated and reported by Charles Hutton on the basis of other data given by Major Williams.

(2) Reports a mean value of 75 : 83.

(3) This cites Muschenbroek's value as 0.918, but questions whether this is referenced to the density of water at the correct temperature and calculates said correction.

(4) Nichol's determination based on a literature survey.

(5) Nichol's own values.

(6) Calculated by me using data in Endo's paper.

(7) calculated from dT_m/dP and ΔS_m and ρ_{water}.

Some of the earlier density determinations are discussed by Dorsey (1940). Note that many of the widely reported values prior to around 1840 are of somewhat opaque origin; for example, numerous textbooks (such as Filcher's *Physikalisches Wörterbuch*, Berzelius' *Lehrbuch der Chemie*, or Kastner's *Grundriß der Experimentalphysik*) attribute a value of 0.916 to Musschenbroek, who gives the specific gravity as 8:9 (or 0.888) in several of his publications. Even Meineke (1815) states that Musschenbroek's value is 0.918 before going on to apply a correction for the inferred water temperature and then reporting a value of 0.885. There is no correction that can shift the specific gravity from 0.918 to 0.885 but it is not difficult to shift from 0.888 to 0.885. Comparatively few writers tabulate Musschenbroek's determination as 0.888 (examples include von Gerstner's *Handbuch der Mechanik* and Böttger's *Tabellarische der Specifischen Gewichte der Körper*).

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