

The Water Triple Point - A Reference Cell Close to the ITS-90 Value

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Summary

A brief history of the Water Triple Point Cell is given leading up to the ITS-90 definition, and an explanation of the 3 main sources of error in a water triple point cell.

Cells filled with water from a still designed to produce pure, air-free water of the isotopic composition of ocean water are analysed by comparing them to 2 cells of unconventional design based on H. F. Stimpson's Cell described in 1943.

The results suggest a way to select reference cells which are much closer to the ITS-90 value than has previously been possible.

The Triple Point of Water; A Reference Cell Close to the ITS-90 Value

Background

The first written references to the ice point, which was the forerunner to the triple point, involve Hooke and Fahrenheit [1]:-

Daniel Gabriel Fahrenheit born in 1686 in Danzig wrote in 1724 that he slipped on the stairs while carrying a flask of supercooled water and found that it suddenly became full of flakes of ice.

However before this in 1663 the under-valued Robert Hooke, describing how he calibrated thermometers wrote:- *“Then, for graduating the stem, I fix that for the beginning of my division where the surface of the liquor in the stem remains when the ball is placed in common distilled water, that is so cold that it just begins to freeze and shoot into flakes; and that mark I fix at a convenient place on the stem, to make it capable of exhibiting very many degrees of cold, below that which is requisite to freeze water”.*

Around 1800 Sir Humphry Davy showed by rubbing two blocks of ice together, and melting them that heat was not a substance but a form of energy, which was defined at first in terms of the properties of water. The unit of heat was taken as the energy required to heat a unit mass of water 1°. This was convenient because the specific heat of water varies little with temperature.

During the 1920's complex measurements were made using Calorimeters at the Massachusetts Institute for Technology on behalf of the National Bureau of Standards to investigate the thermal properties of water more accurately. About this time it was realized ice point determinations were also subject to uncertainties of $\pm 0.001^{\circ}\text{C}$. The ice point is defined as the temperature of equilibrium between ice and air-saturated water at normal atmospheric pressure.

When an accuracy of $\pm 0.0001^{\circ}\text{C}$ was desired it was recognised that the temperature of pure water at the triple point was simpler to define precisely, but it was considered that the apparatus for using it was too complicated for routine testing [2].

The temperature of equilibrium of ice and water is lowered as the pressure above them is increased from its vapour pressure to 1 Bar. The value is around $0.00747^{\circ}\text{C}/\text{atmosphere}$. Foreign substances in solution also lower the temperature. Normal outdoor air lowers the freezing temperature by 0.0024°C at 1 atmosphere. The pressure and dissolved air together lower the freezing temperature by 0.00991°C which means the triple point is nearly 0.0100°C above 0°C .

During the early 1940's a practical triple point apparatus was designed by Stimpson (see figure 1). When in use these cells were completely immersed in an ice bath. The cells were of Pyrex glass about 2" diameter, 16" long and $\frac{1}{2}$ " internal diameter.

Each cell was filled nearly to the top with ordinary distilled water which had been air-freed, re-purified, and then distilled into the cell before sealing off. Once sealed into the apparatus any non-volatile contamination such as dissolved glass could be eliminated at will by pouring the liquid into its 1 litre flask and allowing it to redistill quietly into the cell by keeping the cell in an Ice bath for about a day.

This design was too fragile to be commercially transported and so the flask was removed and the connecting arm bent to hook conveniently over the dewar flask. The cell became known as the Jarrett A-11 and is still in production.

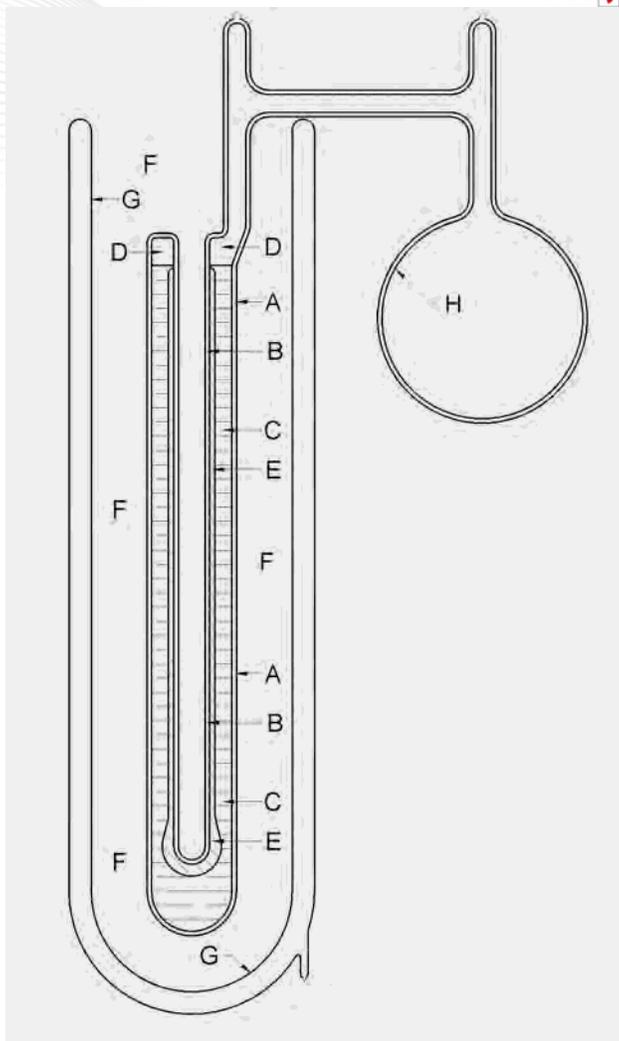


Figure 1 - Triple Point Apparatus Described by E. H. Stimpson in 1943 [2]

A, triple-point cell; B, thermometer well; C, liquid water; D, water vapour; E, ice mantle; F, ice bath; G, vacuum vessel; H, flask for redistillation.

The first suggestion that the water triple point be made a fixed point of the temperature scale was made in 1928 by Moser [2]. By the 1950's it was realized that as well as water purity and vapour pressure, the composition of the water i.e. its isotopic composition was a fundamental variant of water's triple point. Research showed that water from any ocean had a very consistent Isotopic composition, where as variations of lake, and tap water were much larger. The recent temperature scales all specify that the water in a triple point cell shall be of the Isotopic composition of ocean water. There is a Standard Mean Ocean Water (SMOW) analysis which gives an equivalent uncertainty of $\pm 3\mu\text{K}$ of ocean water around the world.

The currently available sample is held in Vienna and is referred to as V-SMOW.

Current Practice

All traceable temperature measurement after 1990 is based on the International Temperature Scale which became legal in January that year (acronym ITS-90). With the definitions of the Scale comes a useful 170 page book called 'Supplementary Information for ITS-90'. Beginning on page 29 is a section describing ways of realizing the defined fixed points of the ITS-90 [4].

It begins by describing the water triple point and immediately in the first paragraph introduces a confusion into our knowledge about water, it says:-

“An operating triple-point cell contains ice, water, and water vapour, all of high purity and of substantially the isotopic condition of ocean water.” Here is a contradiction indeed, how can we have pure water on the one hand, and specify a ‘typical’ content of isotopes?

Our author continues:-

“Variations in the isotopic content of naturally occurring water can give rise to detectable differences in the triple point temperature. Ocean water contains about 0,16mmol of ^2H , per mole of ^1H , and 0,4mmol of ^{17}O and 2mmol of ^{18}O per mole of ^{16}O ; water coming from polar snow or glacial ice may occasionally contain as little as 0,1mmol of ^2H per mole of ^1H .

The purifying of the water may slightly modify its isotopic composition (distillation normally entails a decrease in the ^2H content), and the isotopic composition at an ice-water interface is very slightly dependent on the freezing technique.

A decrease of $10\mu\text{mol}$ of ^2H per mole of ^1H corresponds to a decrease of temperature of the triple point of $40\mu\text{K}$; this is the difference between the triple points for ocean water and the normally occurring continental surface water. The extreme, and quite atypical, difference in the triple-point temperatures of naturally-occurring water is about 0,25mK and is that between sea water and water obtained from melting old polar ice”.

The Isotopic content is crucial to our understanding of water because its triple point is, as we have seen, the fulcrum of the temperature scale. The ITS-90 document itself makes no reference to the Isotopic content, but refers back to 1967-8.

The International Practical Temperature Scale IPTS-68 read [3]:-

“To define the unit of temperature with greater certainty the ice point value can be fixed for all time, thus eliminating the experimental errors of the subdivision of the ice-steam interval. This principle was adopted by the CIPM in 1954 but the definition was expressed in terms of the triple point of water because it is more reproducible than the ice point. After agreement that the triple point temperature was 0.01 K higher than the ice point, the unit was defined by fixing the triple point of water at 273.16 K exactly. At the time the unit was called the ‘degree Kelvin’, symbol °K. The wording of the definition and the name of the unit were changed in 1967 so that it is now given as ‘the Kelvin, unit of thermodynamic temperature of the triple point of water’. (Again, no mention of the Isotopic composition).

Dr. J. V. Nichols and D. R. White have spent much research time on this subject. In an article [5] they say:- “As the definitions stand at the moment we may assume the water defined in ITS-90 has the composition of Standard Mean Ocean Water (SMOW)”.

“The natural variation in the isotopic composition of ocean water is small compared to other natural waters and it corresponds to $\pm 3\mu\text{K}$ ”.

Table 1:

<i>Component</i>	<i>Mole Fraction</i>
¹ H	0.999 842
² H (deuterium)	0.000 158
³ H (tritium)	0
¹⁶ O	0.997 640
¹⁷ O	0.000 371
¹⁸ O	0.001 989

From Table 1 it can be easily seen that there are only four substances of significant abundance in ‘pure’ natural water, ¹H₂¹⁶O, ¹H₂¹⁷O, ¹H₂¹⁸O and ²H ¹H ¹⁶O. In practice ¹H₂¹⁷O can be ignored. Water can therefore be considered a mixture of three substances, ¹H₂¹⁶O, ¹H₂¹⁸O and ²H₂¹⁶O that when mixed form a variety of combinations. It is assumed that SMOW is the intended defining composition of the ITS-90 recommendations. The chemical and thermo-chemical properties of each of the substances is markedly different. For example, heavy water has undesirable physiological effects as compared to natural water.

Table 2:

<i>Substance</i>	<i>Triple Point / K</i>
SMOW	273.160 (defined)
$^1\text{H}_2\text{}^{16}\text{O}$	273.158
$^1\text{H}_2\text{}^{17}\text{O}$	273.31 (estimated)
$^1\text{H}_2\text{}^{18}\text{O}$	273.46
$^2\text{H}_2\text{}^{16}\text{O}$	276.95
$^2\text{H}_2\text{}^{17}\text{O}$	277.06 (estimated)
$^2\text{H}_2\text{}^{18}\text{O}$	277.16

Table 2. The triple point temperature for various water substances. The values for the triple points or the melting points are based on thermo-chemical measurements and calculations. The ^{17}O values are estimated from the ^{16}O and ^{18}O values.

From the above it can be seen that water with isotopes does not have a single triple point, and that the so called triple point will vary with isotopic content.

Table 3: Composition and triple point temperatures of terrestrial water samples

<i>Source</i>	<i>Composition</i>	<i>t (°C)</i>
Ocean Water	^{17}O ; 0.04 mol/100 mol ^{16}O ^{18}O ; 0.2 mol/100 mol ^{16}O	0.0099
Continental Water	^2H ; 0.016 mol/100 mol ^1H ^2H ; 0.015 mol/100 mol ^1H	0.0099-0.00004
Polar Water	^2H ; 0.010 mol/100 mol ^1H	0.0099-0.00025

The water gets even muddier when consideration is given to the changes in isotopic content that occur when water turns to ice. These mean that the temperature of the so called triple point of water varies depending both on the % of ice in the cell and the age of the ice mantle. These variations total up to 70 or 80 μK depression [5].

The best water triple point cells are traditionally selected from a group by choosing those with the highest absolute temperature. This means that the cells also have the highest isotopic content.

Nicholas & White recommend that the isotopic content of reference water triple point cells be measured.

All this leads to the conclusion that the three main sources of uncertainty within a water triple point cell are:-

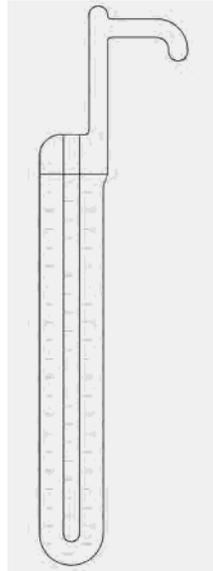
1. Entrapped Air.

A water triple point cell should be at the vapour pressure of the water it contains.

Entrapped air within the cell increases the pressure and has the effect of decreasing the water triple point temperature. 1 atmosphere causes a depression of approximately 7.5mK.

There are two commonly available designs of water triple point cell. The preferred design illustrated in *figure 2a* includes a McLeod gauge which can be used to measure the volume of trapped air. From this, the cell depression, if any, can be calculated, as shown in *figure 2b*.

Sostmann suggests that a bubble 4mm in diameter will cause a depression of 0.3 μ K for the Jarrett-Isotech Cell type A-11 [6] and this is typically the size of bubble in such a cell.



Type A

*Figure 2a: (a) The configuration of Jarrett-Isotech Type A cells.
Note the handle, which is a McLeod gauge.*



Figure 2b: Procedure for entrapping a gas bubble in Type A cells: (a) position the cell as shown (b) gently invert the cell to trap and compress the bubble.

2. Impurities in Water.

All impurities depress the triple point of water. Fortunately when water is solidified in a triple point cell the ice rejects the impurities, concentrating them in the remaining water. By turning 50% of the water into ice around the re-entrant tube of the cell, the impurities within the water are approximately doubled.

To measure the temperature within the made up water triple point cell, a rod of copper at ambient temperature is placed in the water filled re-entrant tube and as it cools a thin film of ice is melted around the tube. This thin film is very pure, being water melted from the ice.

After measuring the cell's temperature the cell is inverted three times, allowing the ice mantle to float and suck in impure water from the cell to replace the clean water from the melted ice.

The cell's temperature is re-measured and the depression is a measure of the effect of impurities. ASTM 1750-95 [7] suggests a good cell is within $30\mu\text{K}$ of its pre-inverted temperature (*also see Isotech's Journal of Thermometry Volume 7.2 (1996) page 121*).

3. Isotopic Composition.

We think of water as H_2O , more precisely we should write $^1\text{H}_2\text{}^{16}\text{O}$. This has been termed 'light' water and is unobtainable.

If it were obtainable its triple point would be approximately $+0.008^\circ\text{C}$.

In practice what we drink from the tap, and what is in the ocean is a soup of ^1H , ^2H with ^{16}O , ^{17}O and ^{18}O .

This means real water does not have a single triple point temperature.

Its 'combination triple point' will vary as the isotopic composition varies.

This was understood more than 50 years ago and after much analysis of water from different sources recent temperature scales state that the water in a triple point cell shall have the isotopic composition of ocean water.

Although not explicitly stated, there is a Standard Mean Ocean Water (SMOW) and it is assumed that the isotopic content of SMOW fulfils the ITS-90 requirements.

To the author's knowledge no actual cell has this combination and so the deviation of an actual cell from the prescribed ideal (SMOW) will give the elevation, or depression of it's water triple point temperature from +0.01C.

By analysing a sample of water from a water triple point cell it's deviation from the ideal (SMOW) can be calculated approximately and allowed for as an offset.

Cells Closely Associated to ITS-90

The purpose of this article is to present data from analysis of a group of cells from a still designed to produce pure, air-free water of isotopic composition close to V-SMOW.

For this, a reference cell is required, not selected from a group as having the highest temperature, but made from water with negligible impurities, no trapped air, and of V-SMOW composition.

A cell was designed and made based on Stimpson's construction, but with the addition of a Mcloed gauge (figure 3).

Two cells (KT001 and KT002) were filled with water form the still and samples of water from the cells were sent for isotopic analysis to a facility with the ability to analyse its deviation from V-SMOW to an equivalent uncertainty of $\pm 2\mu\text{K}$.

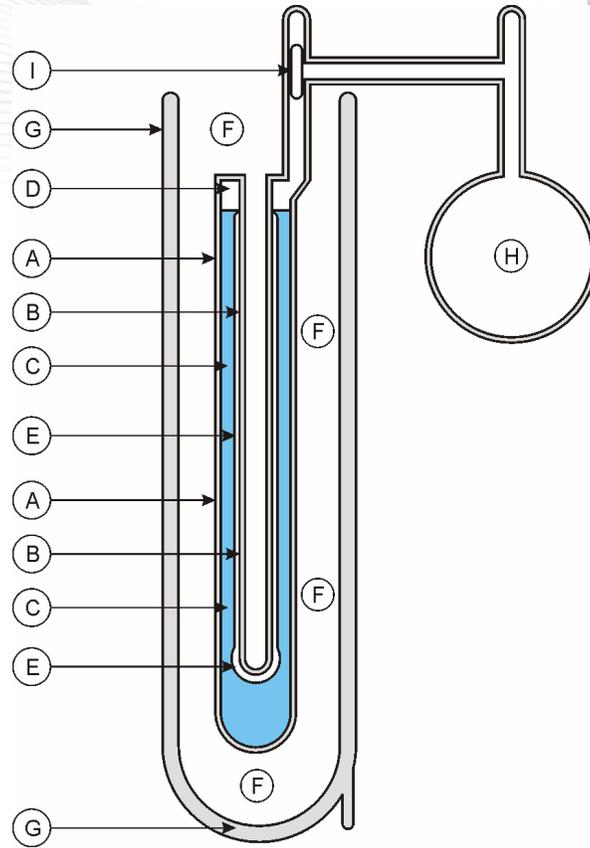


Figure 3 – A Triple Point Apparatus with Mcleod Gauge

A, triple point cell; **B**, thermometer well; **C**, liquid water; **D**, water vapour; **E**, ice mantle; **F**, ice bath; **G**, vacuum vessel; **H**, flask for redistillation; **I**, Mcleod gauge.

PHYSICAL FEATURES

Type A cells (fig. 4a) were designed by Dr. H. F. Stimson at NBS. A tubular glass extension at the top of the cell serves as a convenient handle for lifting and carrying the cell, as a hook for supporting it in an ice bath, and as an indicator of partial pressure of air in the cell.

Type B cells (figs. 4b & 4c) were designed at NRC of Canada. The thermometer well extends 100mm above the top of the cell. Heat transfer to the ice mantle may be essentially eliminated by keeping these cells packed in ice to the top of the well extension, or by immersing them sufficiently in a water-triple-point-maintenance bath.

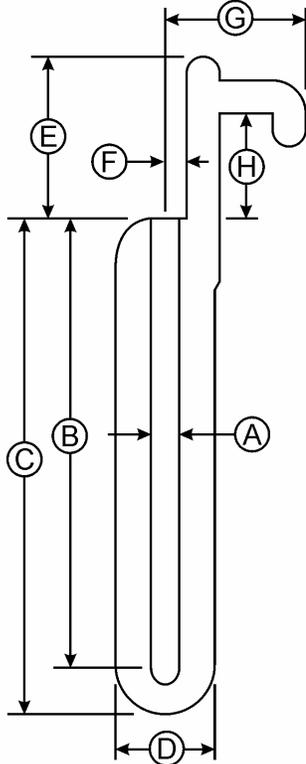


Fig. 4a

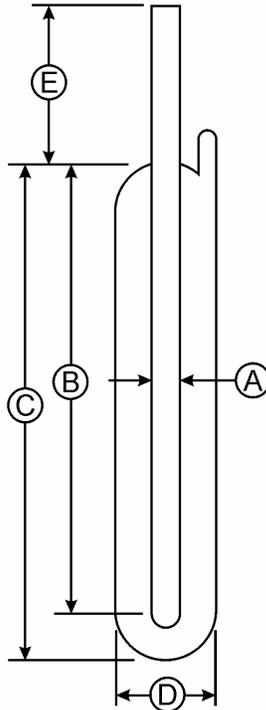


Fig. 4b

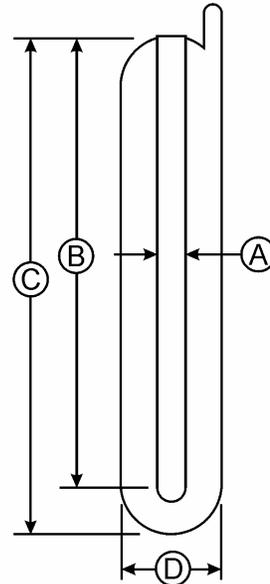


Fig. 4c

Fig.	Model		Nominal Dimensions in Millimetres							
	Number	A*	B	C	D	E	F	G	H	
4a	A-11	11	317	348	51	104	14	93	70	
4a	A-13	13								
4b	B-11	11	317	348	64	100				
4b	B-13	13								
4b	B-16	16								
4b	B-19	19								
4b	B-22	22								
4b	C-12	12.5	240	281	46	70				
4c	D-8	8.5	134	154	30					

N.B. Type C Cells are the equivalent of the type B Jarret Cells and Type E Cells are the equivalent of the type A Jarrett Cells.

Figure 4

Table 4:

Isotopic Analysis and association with SMOW of a Variety of Cells

Serial Number	$\Delta^{18}\text{O}$	$\Delta^2\text{H}$	$\mu\text{K from SMOW}$ $\pm 2\mu\text{K}$
E11 - 057	+7	+36	+26
E11 - 058	+0.7	+3	+2.4
E11 - 059	+0.08	+5	+3.5
C12 - 061	+2.1	+4	+4
E11 - 063	-1.5	-3.9	-4
E11 - 067	-0.3	-12	-7
KT - 001	+0.2	-12	-7
KT - 002	+0.8	+0.6	+1
E11 - 080	-4.2	-25	-18
B16 - 1593	-0.2	-6	-4
KT - 004	-4	-35	-24
KT - 005	-4	-34	-23
E11 - 140	-2	-21	-14
E11 - 139	-0.3	-18	-11
E11 - 145	+0.8	-6	-5
E11 - 183	+0.8	+14	+9
B11 - 2083	+0.4	+10	+6
E11 - 166	+1.7	+4	+4
E11 - 170	0	-0.8	-1
E13 - 195	-1.5	-12	-8
B11 - 2093	-1.4	-16	-10
E11 - 200/201	-0.7	-13	-8
E11 - 203/202	-1	-3	-2
E11 - 206/207	+0.4	-0.6	0
E11 - 212	+2.5	+9	+7
E11 - 213	+3.4	+23	+16

To obtain Microdegrees the deviations from SMOW are multiplied by the mole fraction and by the triple point (ref. 5).

Notes: E11-057 and E11-213 were both filled with water of known high isotopic content to illustrate that cells can be prepared above the V-SMOW analysis.

The two reference cells are not practical day-to-day working devices they were used to certify more practical designs such as All 2011 and NMI 119 shown in table 4. All subsequent cells were intercompared to these cells.

Method

Over a period of 6 months a method of intercomparison was developed as follows:-

Reference and referred cells were made up at the same time using ice mantle makers, heat pipes small enough to go down the triple point reentrant tubes and having a container of 400cc of liquid nitrogen at the emergent end. 3 fillings of this container and 20 to 30 minutes of cooling gave a good ice mantle in each cell. The cells were left 7 to 10 days in a water triple point maintenance bath model ITL-M-18233 and intercompared over the subsequent 3 to 4 days.

Furniture in the form of 2 foam buttons at the base of the reentrant tube, copper centralizing bushings and teflon guide tubes at the emergent end of the reentrant tube were used to guide and support the selected SPRT found to give most consistent results.

A software program was developed for a MII 6010 Bridge using 3 currents to ascertain the exact resistance of the thermometer as follows. The first current is selected and 60 measurements each of 20 seconds duration are made reversing the current after each measurement.

The first 24 measurements are discarded being the self-heating time of the thermometer and the mean and standard deviation of the last 36 measurements are made, the current is changed and the 20 minute process repeated. After the third current, the original current is reselected to check that the freed mantle had not reconnected itself to the reentrant tube, and the repeatability of the system.

The zero current resistance can be calculated in 3 ways and from the three resistances the mean is calculated. Correction is made for the exact hydrostatic head and the final resistance is obtained.

The thermometer is transferred to the second cell and after a 30 minute wait the process is repeated. 4 cells can be measured in 1 day, 1 reference and the 3 unknown using the above technique.

The cells are then repositioned in the maintenance bath and the measurements repeated again on a second and third day. The reason the cells are moved is that we do not repeat the measurements in the same sequence.

All measurements are eventually referred back to a specially selected Tinsley 25 Ω resistor with tempco of less than 0.1ppm/ $^{\circ}$ C and maintained at 20 $^{\circ}$ C \pm 0.007 $^{\circ}$ C in a stirred oil maintenance bath whose temperature is continuously monitored and recorded.

The still is used to fill a variety of cells with different immersion depths and diameters (figure 4) a selection of cell sizes have been included in the evaluation.

It has not been possible to isotopically analyse all the cells produced and the smaller cells (D8 type) produced do not have a large enough depth of immersion for the most accurate measurement and so only larger cells which have isotopic analysis are included in the major tables.

Early in the history of our Primary Laboratory and well before the still described here existed a reference cell was selected from our stock, and because of its long history it remains the cell to which other cells are referred. Its serial number is NMI 119 it is regularly intercompared to A11-2011 another reference cell.

Table 5 below shows the results of a large number of production cells which have been compared to NMI 119 using the 3 current measurement procedure detailed previously.

Table 5: Deviation from NMI 119 by intercomparison.

Serial Number	μK above (+) or below (-) NMI 119
A11 - 2011	+15
KT - 001	+10
KT - 002	+13
E11 - 083	0
E11 - 084	-34
E11 - 086	-7
E11 - 087	-7
C12 - 089	-29
C12 - 090	-15
C12 - 091	-15
D8 - 095	-17
C12 - 096	-18
C12 - 097	-17
C12 - 098	+6
C12 - 099	-38
C12 - 100	+3
C12 - 101	-10
C12 - 102	+8
C12 - 108	-11
A11 - 116	-6
B16 - 1593	-13
C12 - 123	+9
C12 - 125	+5
C12 - 127	-24
E13 - 139	-13
E11 - 145	-20
D8 - 160	-17
E11 - 163	-15
B11 - 2063	+1
E11 - 170	-29
E13 - 182	-13
E13 - 183	+2
E13 - 184	-12
C12 - 190	-28
D8 - 193	-43
E13 - 195	-18
E11 - 201	-28
E11 - 202	-16
B11 - 2064	+2.85mK (see note)
E11 - 212	-8
E11 - 213	-10

N.B. To illustrate an extreme case a drop of pure deuterium was added to B11-2064 it was possible to produce a water triple point cell of value 0.01285°C. it behaved in every way identically to the other cells.

From the isotopic composition of KT002 it ought to be closest to ITS-90. This implies that NMI 119 is 10 to 15 μK below ITS-90 and A11-2011 is also very close to the ITS-90 value.

Table 6 below shows the cells that have been compared to NMI 119 and have their isotopic composition known. The standard deviations have been calculated individually for each cells results.

Table 6:

Serial Number	A $\Delta\mu\text{K vs NMI 119}$	B Isotopic Analysis $\mu\text{K vs SMOW}$	A-B $\Delta\mu\text{K}$	Standard Deviation S.D. μK
A11 - 2011	+15	-	-	4
KT - 001	+10	-7	+17	13
KT - 002	+13	+1	+12	14
E11 - 080	-46	-18	-28	12
B16 - 1593	-13	-4	-9	11
E11 - 139	-13	-11	-2	28
E11 - 145	-20	-5	-15	7
E11 - 163	-15	+9	-24	5
B11 - 2063	+1	+6	-5	3
E11 - 170	-29	-1	-29	4
E13 - 195	-18	-8	-10	3
E11 - 201	-28	-8	-20	18
E11 - 202	-16	-2	-14	17
E11 - 212	-8	+7	-15	24
E11 - 213	-10	+16	-26	16
			Mean -16	8 Standard Deviation

Notes: Excepting A11-2011, KT001 and KT002 all cells have a lower temperature than NMI 119 and are also below their calculated isotopic temperature on average by 16 μK . Based on KT001 & KT002. NMI-119 is 10 to 15 μK below ITS-90. All-2011 is the closest cell we have measured to ITS-90.

Discussion

In all cases the cells have a temperature lower than that calculated from the isotopic analysis. This difference is variable from 2 to 29 μK below NMI 119, and represents a combination of the dirtiness of the water residual air in the cell and our uncertainty in measuring the temperature.

The results of the isotopic analysis suggest that it is possible to have cells with triple points above, as well as below V-SMOW and so the present practice of selecting the cell with the highest temperature does not represent the cell closest to V-SMOW and hence ITS-90.

Conclusion

Analysing the cell contents for isotopic composition is a useful and valid addition to the usual techniques of intercomparing cells and gives an accurate indication of the cells ideal triple point value.

All cells except those with a flask are lower in temperature than predicted by the isotopic content, probably due to a very small amount of contamination caused by unavoidable glass deritrification during the final sealing process [8].

In the case of the cells with a flask the water can be repurified after sealing leaving any dissolved glass and other impurities in the flask.

Such cells, complete with isotopic analysis provide the closest association to the ITS-90 value of the water triple point and hence the KTTS Scale.

It is now possible to produce cells, based on Stimpson's original design which can be associated to V-SMOW within $\pm 3\mu\text{K}$ and this is a smaller uncertainty than can be provided by traditional measurements.

Conventional shaped cells can be produced on a commercial basis which are within $\pm 30\mu\text{K}$ of V-SMOW, and can be provided with a certificate of isotopic analysis.

Best Laboratory Practice

Based on the above, our two conventional reference cells NMI 119 and A11-2011 continue to be used on a daily basis and are intercompred annually to KT001 and KT002 just after their contents have been emptied into the flask and redistilled back into the cell.

Using the above method we feel more confident that our reference cells are closely related to the prescribed ITS-90 value.

In future our reference cells will have McLeod Gauges for measuring any entrapped air and an isotopic analysis of the water within the cells. Intercomparisons will be with NIST who offer the smallest intercomparison uncertainties.

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