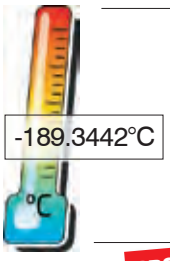


471 Simple Argon **Triple Point Apparatus**

PROVISIONAL DATA*

- Affordable
- Robust and simple to use
- Accurate to ±0.5mK - 4 hour plateau typical

The Isotech Argon Triple Point Apparatus is a robust, simple to use and affordable solution for the realisation of the argon triple point.

Many laboratories use liquid nitrogen comparators which are convenient and can be low cost but the nitrogen boiling point is not on the ITS-90. More seriously the LN point is below that of Argon. Many standard platinum resistance thermometers (SPRTs) are filled with a mixture of argon and oxygen and at -195°C will be under a partial vacuum which affects the self-heating of the SPRT leading to a larger calibration uncertainty.

For many laboratories the high cost and complexity of previously available argon systems has been a barrier.

Now after years of research Isotech have introduced a new system that is more affordable, simple to use and will allow more laboratories the benefits of being able realise the argon triple point.

The Isotech system requires no electricity; the only consumable is liquid nitrogen - the 6N Pure argon is contained in a pressure vessel. A filling tube allows liquid nitrogen to initially cool this volume to approximately -195°C. Weights are then added to a pressure release valve to increase the nitrogen's boiling temperature to just above the argon cells triple point.

The argon settles into its triple point for around four hours, allowing an SPRT inside the re-entrant tube to be calibrated. At the argon T.P. to an accuracy of ±0.5 mK, K = 2

SCAN ME

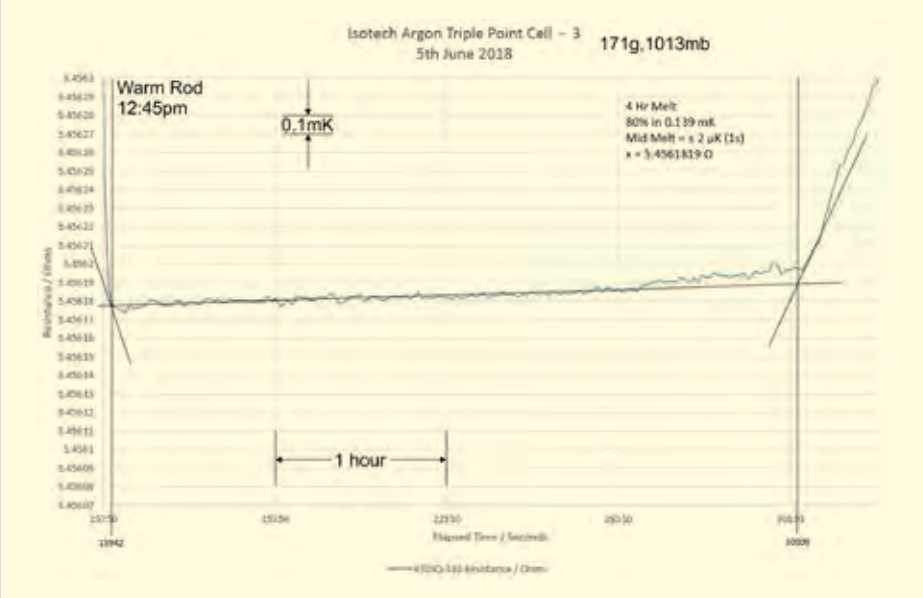
Sectional view from front

Sectional view from side

ISOTHERMAL TECHNOLOGY LTD UKAS LABORATORY Nº 0175	Date of issue	13th March 2017
	Issue Nº	01
Argon T.P. Cell Premium Calibration - ±0.5 mK UCT	Authorised by	I. Dixon
Budget Nº. BT116		

Note number (below)	Source of uncertainty	Value ±	Unit	Probability distribution	Divisor	Sensitivity c_i	Standard uncertainty u_i (unit)	Degrees of freedom v_i or v_i	u_i^2	u^4/v_i
1	Standard deviation	0.000069	C	normal	1.00	1	0.000069	11	0.000000005	2.06E-18
2	SPRT Spurious heat flux, noise etc (Std cell)	0.000005	C	normal	1.00	1	0.000005	22	0.000000000	2.8E-23
3	SPRT Spurious heat flux, noise etc (Test cell)	0.000006	C	normal	1.00	1	0.000006	22	0.000000000	5.9E-23
4	micro K linearity	0.000007	C	normal	2.00	1	0.000004	1	0.000000000	0
5	micro K resolution	0.000002	C	rectangular	1.73	1	0.000001	1	0.000000000	0
6	Test and Std cell slope differences	0.000183	C	rectangular	1.73	1	0.000106	1	0.000000011	0
7	Measured H/H uncertainty in Std Cell	0.000178	C	rectangular	1.73	1	0.000103	1	0.000000011	0
8	Measured H/H uncertainty in Test Cell	0.000017	C	rectangular	1.73	1	0.000010	1	0.000000000	0
9	Estimated H/H uncertainty in Std Cell	0.000017	C	rectangular	1.73	1	0.000010	1	0.000000000	0
10	Estimated H/H uncertainty in Test Cell	0.000017	C	rectangular	1.73	1	0.000010	1	0.000000000	0
11	Sprt self heating max Δt in Std cell	0.000141	C	rectangular	1.73	1	0.000082	1	0.000000007	0
12	Sprt self heating max Δt in Test cell	0.000167	C	rectangular	1.73	1	0.000097	1	0.000000009	0
13	Lead moisture effects	0.000010	C	rectangular	1.73	1	0.000006	1	0.000000000	0
14	Temp effect of oil bath on Std resistor	0.000001	C	rectangular	1.73	1	0.000001	1	0.000000000	0
15	Calibration of Std resistor	0.000002	C	normal	2.00	1	0.000001	1	0.000000000	0
16	Uncorrected Drift of Std Resistor	0.000000	C	rectangular	1.73	1	0.000000	1	0.000000000	0
17	Uncertainty Of standard cell	0.000280	C	normal	2.00	1	0.000140	1	0.000000020	0
u_c	Combined uncertainty			normal			0.000250	1895	0.000000062	2.1E-16
U	Expanded uncertainty			normal	k for v_i	2.00	0.000500	1895		

k for v	2.00	0.000500	1895



Model	471
Temperature Range	-189.3442°C
Uncertainty	+0.5mK at k=2
Dimensions	Width - 380mm Depth - 615mm Height - 1250mm (900mm high to top of cabinet)

For More Data and the Latest Information:
https://www.isotechna.com/product-p/471.htm

The Best Primary Standards for your Laboratory

The key factor is that of purity. ITS-90 specifies that the purity of the ITS-90 fixed points should be 99.9999% (6N).

The performance of an optimal 6N pure cell has been best described in CCT/2000-13 "Optimal Realization of the Defining Points of the ITS-90…"

All Isotech's cells for primary laboratories conform to the ideals set out in this document. To prove to ourselves, and you, our customer, that this is so we compared some 160 UKAS certificates over 10 years using a variety of metal samples to CCT/2000-13. The results tabulated below show that we equal or exceed the values given in that document.

CCT/2000-13 Optimal Realizations of ITS-90

Comparison of 160 Isotech UKAS Certified Cells to CCT/2000-13

	CCT/2000-13	Large (Optimal) Cells mK
Hg	0.2mK	0.12
Ga	0.1mK	0.05
In	0.5mK	0.17
Sn	0.3mK	0.18
Zn	0.5mK	0.21
Al	0.7mK	0.66
		0.3*
Ag	1.1mK	1.1

*6N5 pure

With each delivery of metal the supplier furnishes us with a certificate detailing the impurities detected in ppm.

At Isotech we go one step further, samples of the metal are sent to NRCC in Canada who analyse the sample to parts per billion and look for 60 elements rather than the 20 that the supplier analyses. This independent analysis increases confidence in the metal of the cell.

The metal of the cell is contained in a graphite crucible. Our graphite is the densest available having an average grain size of just 7 μm.

No metal has ever penetrated this graphite. It is supplied with a purity better than 99.9995% and at Isotech temperature and vacuum processing further reduced the impurities.

Whether you choose a sealed, or resealable cell we need pure argon to surround the cell, our argon is 99.9999% pure.

Before we make cells commercially with a new delivery of metal we make a cell for evaluation. It goes through the same 5 step process as is used by National Laboratories for international intercomparisons.

The cell is melted and frozen three times and the coincidence between melt and freeze point measured. The impurities are used to calculate the expected depression of the metal from ITS-90 and the cell is intercompared on 2 separate occasions with a reference cell directly traceable to NIST's realization. This process takes 15 working days.

ITS-90 specifies that the melting or freezing should take place at 101,325 Pa.

An Isotech sealed cell is filled with 6N pure argon to 101,325 Pa ±0.04% as certified by a UKAS certificate of the vacuum gauge.

Nothing is left to chance with an Isotech Primary Standard.

Because we supply most of the worlds primary laboratories we need to be able to certify what we have made. Working with UKAS and NIST we have reduced our uncertainties of measurement to the smallest outside NIST. They are tabulated below for your information.

UKAS k=2	Isotech ±mK Quartz & Metal Clad (*)
Hg	0.22
H_2O	0.07
Ga	0.07
In	0.65
Sn	0.60
Zn	0.90
Al	1.10
Ag	2.00

*(*As November 2008 - The latest UKAS Schedule can be found from our website or at www.ukas.org)*

The above contains no fancy claims or unsubstantiated numbers, only independently verifiable facts.

Some 500 metrologists visit Isotech each year for discussions and training, you will be welcome.

The immersion of the cells from metal surface to the bottom of the re-entrant tube is 200mm ±5mm.

Each primary cell is accompanied by a conformity certificate which includes a copy of the impurities analysis, a copy of the metal of the cell evaluation freeze and melt curves.

At an extra cost we can issue a UKAS certificate to the uncertainty above. This takes 15 working days.

Cell Containment

■ Resealable cells

Traditionally our optimal cells have been assembled into resealable quartz tubes or crucibles whereby the cell can be vacuumed and refilled with pure argon to 1 atmosphere.

More recently Isotech have pioneered metal clad cells replacing the more fragile quartz with a pre-aged metallic alternative. These cells have a small metal tube which can be used for vacuuming and refilling the cell and because of less conduction from the cell; the cell is closer thermally to its ITS-90 value.

■ Sealed Cells

It maybe more convenient to have sealed cells - cells with a cladding whose internal pressure is preset to 1 atmosphere at the freeze temperature and then sealed. Isotech offers both quartz and metal clad sealed cells.

UKAS Calibration Service for SPRTs

SPRT Calibration with ITS-90 Fixed Points: Premium Service
ISOTECH UKAS Calibration Uncertainties (*k*=2)

Suitable only for Isotech 670SQ Models or other Primary Standard SPRTS of similar stability

Fixed Point	°C	Range 1	Range 2	Range 3	Range 4	Range 5	Range 6	Range 7[1]
				Typical Uncertainties ±				
TP Argon [2]	-189.3442	0.5mK		0.5mK	0.5mK	0.5mK	0.5mK	
TP Mercury	-38.8344	0.3mK	0.3mK	0.3mK	0.3mK	0.3mK	0.3mK	
TP Water	0.01	0.1mK	0.1mK	0.1mK	0.1mK	0.1mK	0.1mK	0.2mK
MP Gallium	29.7646		0.2mK					
FP Indium	156.5985			1mK	1mK			
FP Tin	231.928				1mK	1mK	1mK	1mK
FP Zinc	419.527					1.2mK	1.2mK	1.2mK
FP Aluminium	660.323						2mK	2mK
FP Silver	961.78							7mK

SPRT Calibration with ITS-90 Fixed Points: Standard Service
ISOTECH UKAS Calibration Uncertainties (*k*=2)

Suitable for Primary and Working SPRTS - Isotech 670 & 909 families and other SPRTS of similar stability

FIXED POINT Fixed Point	°C	Range 1	Range 2	Range 3	Range 4	Range 5	Range 6	Range 7[1]
				Typical Uncertainties ±				
BP Nitrogen	-195.798	10mK		10mK	10mK	10mK	10mK	
TP Mercury	-38.8344	2mK	2mK	2mK	2mK	2mK	2mK	
TP Water	0.01	1mK	1mK	1mK	1mK	1mK	1mK	10mK
MP Gallium	29.7646		1mK					
FP Indium	156.5985			3mK	3mK			
FP Tin	231.928				3mK	3mK	3mK	10mK
FP Zinc	419.527					3.5mK	3.5mK	10mK
FP Aluminium	660.323						10mK	25mK
FP Silver	961.78							40mK

Note 1: Model 96178 or other HTSPRTS of similar stability

Note 2: Alternatively in place of TP Argon the BP Nitrogen point can be used, the uncertainty increases to 5mK for Ranges 1, 3, 4 and 5 and 6mK for Range 6.

Note: TP = Triple Point MP = Melting Point
FP = Freezing Point BP = Boiling Point

The latest schedule can be found on the Isotech website or at www.ukas.org.

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