

**ISOTECH**

# Argon Triple Point



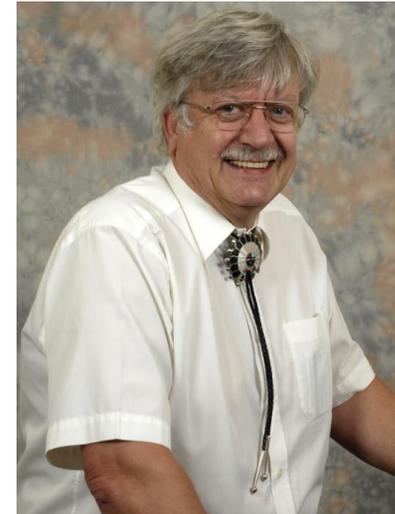
[www.isotech.co.uk](http://www.isotech.co.uk)

# Isothermal Technology Innovation

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# Isotech Model 471: Further Refinement

## Simple Argon Triple Point Apparatus



# Isotech Model 471: Further Refinement

Before looking at the changes we will review the apparatus



# What is the Argon Triple Point?

ITS-90 Point  
-189.3442 °C

*3.3.1.3. The Triple Point Of Argon (83.8058 K) to the Triple Point of Water (273.16K). The thermometer is calibrated at the triple points of argon (83.8058 K), mercury (234.3156 K) and water (273.16 K).*



# Benefits over liquid nitrogen calibration

Many laboratories use liquid nitrogen comparators

convenient  
can be low cost

but the nitrogen boiling point is not on the ITS-90



# Benefits over liquid nitrogen calibration

More seriously the LN point is below that of Argon -183 vs -195°C

Many SPRTs are filled with a mixture of argon and oxygen and at -195°C will be under a partial vacuum

Can affect the self-heating of the SPRT leading to a larger calibration uncertainty



# A New Simple Solution

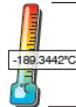
For many laboratories the high cost and complexity of previously available argon systems has been a barrier

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



# Isotech Model 471



**PROVISIONAL DATA**

- Affordable
- Robust and simple to use
- Accurate to  $\pm 1\text{mK}$  - 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  $-195^\circ\text{C}$  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 re-entrant tube allows liquid nitrogen to initially cool this volume to approximately  $-195^\circ\text{C}$ . 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.

**\*Provisional Data**  
The specification on this product is provisional, please visit the website for the latest information.  
<http://www.isotech.co.uk/argon>

<http://www.isotech.co.uk/argon>

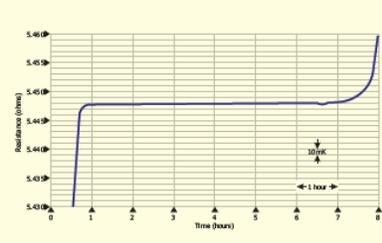
## 471 Simple Argon Triple Point Apparatus






Note number (below)	Source of uncertainty	Value $\pm$	Unit	Probability distribution	Divisor	Sensitivity $c_i$	Standard uncertainty $u_i$ (k=1)	Degrees of freedom $\nu_i$	$u_i^2$	$u_i^2/\nu_i$
1	Calibration of Standard Resistor	0.000012	C	normal	2.00	1	0.000001	1	0.00000000	0
2	Unconnected DRT since last calibration	0.000006	C	rectangular	1.73	1	0.000005	1	0.00000000	0
3	Effect of the Temperature of Oil Bath	0.000010	C	rectangular	1.73	1	0.000006	1	0.00000000	0
4	Argon Purity	0.000010	C	normal	2.00	1	0.000005	1	0.00000000	0
5	Physical Equations	0.000004	C	rectangular	1.73	1	0.000003	1	0.00000000	0
6	Uncertainty of the Fixed Point Cell	0.001000	C	normal	2.00	1	0.000500	1	0.00000250	0
7	Stoichiometry of the Fixed Point Cell	0.001000	C	rectangular	1.73	1	0.000577	1	0.00000333	0
8	Impression Uncertainty	0.000006	C	normal	1.00	1	0.000006	1	0.00000036	0
9	Self-heating Effects	0.000006	C	rectangular	1.73	1	0.000039	1	0.00000031	0
10	SPRT Structure Non-resistance	0.000010	C	normal	1.00	1	0.000010	16	0.00000006	6.1E-03
11	Uncertainty of Interchange Connections	0.000171	C	rectangular	1.73	1	0.000100	1	0.00000000	0
12	Repeatability of the Thermometer	0.000006	C	normal	2.00	1	0.000003	16	0.00000016	1.6E-17
13	Propagation of the error propagation	0.000010	C	rectangular	1.73	1	0.000006	1	0.00000000	0
U	Combined uncertainty			normal	k = 2.00		0.001844	27.853	0.00000350	1.5E-17
U	Expanded uncertainty			normal	k = 2.00		0.001844	27.853		

**k = 2.00    0.001844**

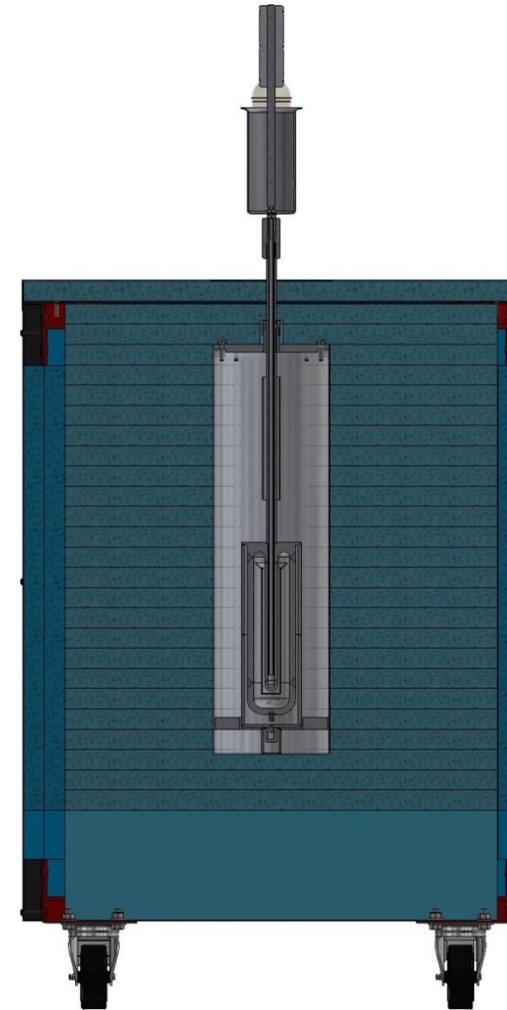


Model 471  
Temperature Range  $-189.3442^\circ\text{C}$   
Uncertainty  $1.844\text{mK}$  at  $k=2$   
Dimensions  
Width - 380mm  
Depth - 615mm  
Height - 1250mm  
(900mm high to top of cabinet)

For More Data and the Latest Information:  
[www.isotech.co.uk/argon](http://www.isotech.co.uk/argon)

# Cross Section

The cross section shows the insulation inside the tank – it is rated to -200°C



# Inner Container

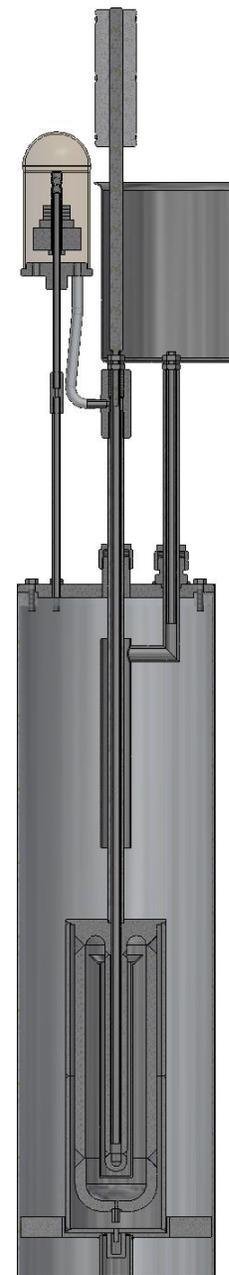
There is an inner 8 litre stainless steel container 500m deep from the lid



# Assembly

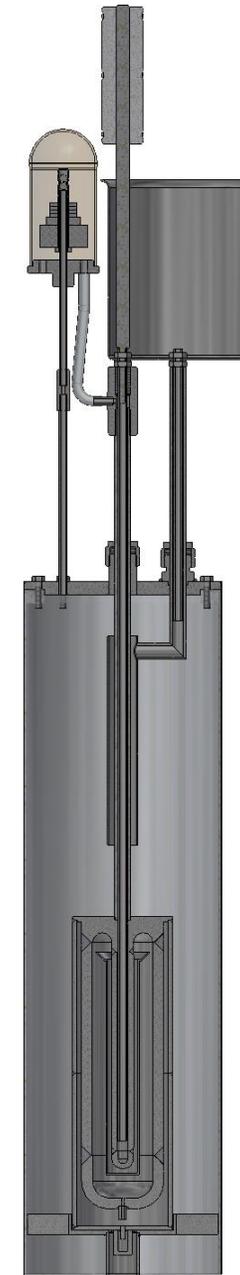
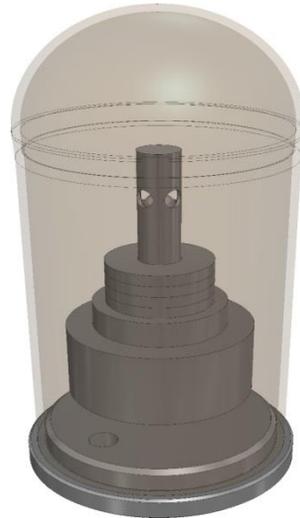
The lid has three tubes protruding – the central one goes into the argon triple point cell

To one side is the filling tube – with a funnel. This can be removed and then the tube can be sealed



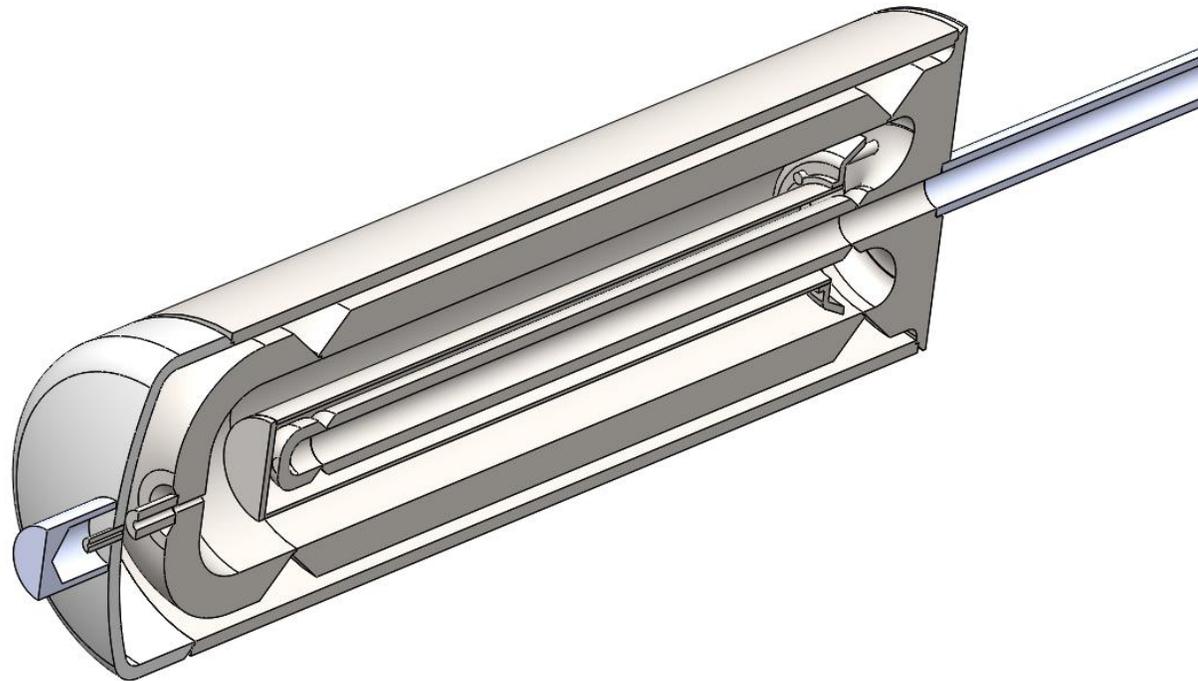
# Inner Assembly

To the other side is a pressure relief valve – it controls the pressure and hence the nitrogen boiling point temperature



# Inner Assembly

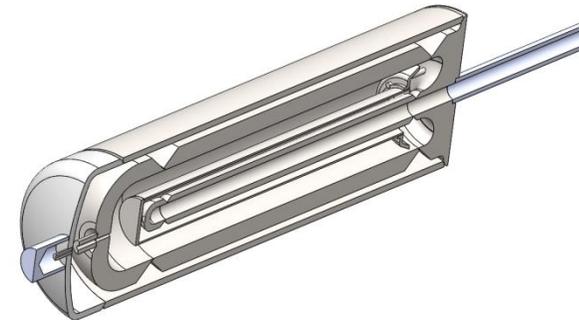
The argon triple point cell is a pressurised vessel containing pure argon.



# Inner Assembly

It has re-entrant tube to accommodate either a funnel of liquid nitrogen for cooling or an SPRT for calibration

There are no electrical connections



# Operation of the Apparatus



# Running costs

In England liquid nitrogen costs less than £2.00 per litre plus a fixed delivery charge, so a day's calibration costs between £25.00 and £50.00.

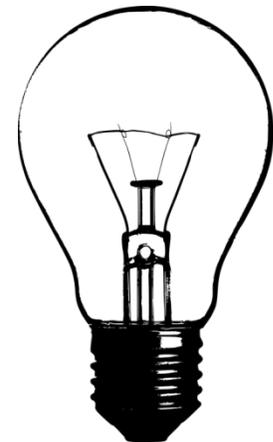
This may vary from country to country.



# Results

Thomas Edison tried some 2000 times before he produced a successful tungsten filament lamp.

Asked about his failures he replied that he had not failed, but found 2000 ways not to produce the lamp.



# Results

Four years, 300 experiments, 5000 litres of liquid nitrogen produced many ways not make a successful Argon Triple Point Apparatus, however eventually we were at a point where we could evaluate



# Results

But I realised we could do even better by

Using a higher purity gas

Creating more internal nucleation points

Reducing the immersion depth to allow calibration of shorter thermometers



# Results

So *even more testing*.... with a higher purity gas

## CERTIFICATE OF ANALYSIS Grade of Product: RESEARCH PLUS

Part Number:	AR RP300	Reference Number:	82-401006514-1
Cylinder Number:	4652608Y	Cylinder Volume:	336.0 CF
Laboratory:	124 - Riverton (SAP) - NJ	Cylinder Pressure:	2640 PSIG
Analysis Date:	Oct 12, 2017	Valve Outlet:	580
Lot Number:	82-401006514-1		

### ANALYTICAL RESULTS

Component	Requested Purity	Certified Concentration
ARGON	99.9999 %	99.9999 %
CARBON DIOXIDE	< 0.1 PPM	<LDL 0.10 PPM
CARBON MONOXIDE	< 0.1 PPM	<LDL 0.10 PPM
OXYGEN	< 0.1 PPM	<LDL 0.03 PPM
THC	< 0.1 PPM	<LDL 0.03 PPM
NITROGEN	< 2.0 PPM	<LDL 0.04 PPM
MOISTURE	< 0.2 PPM	<LDL 0.20 PPM

Notes: Analysis by SPC data

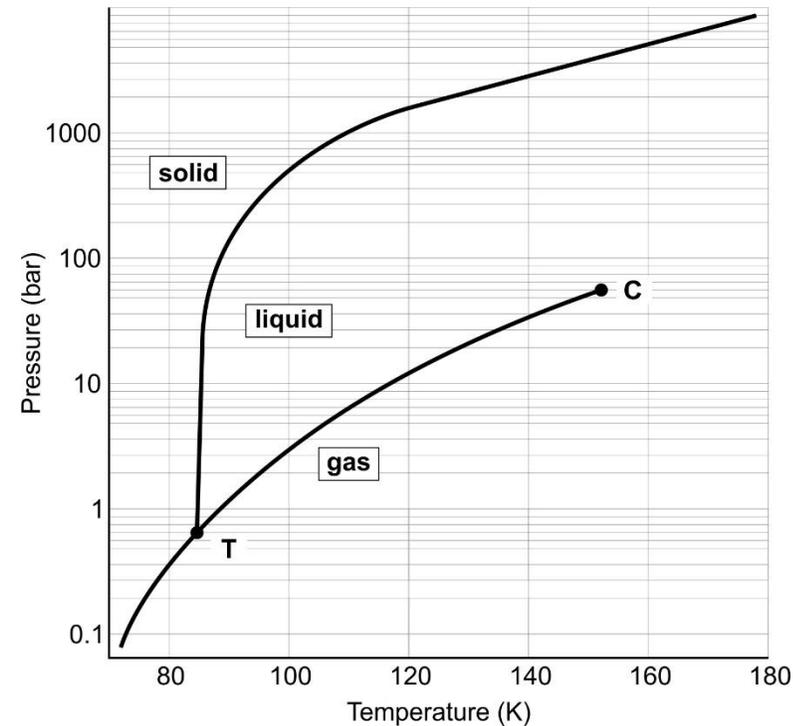
Impurities verified against analytical standards traceable to NIST by weight and/or analysis.

# We now need to know the following:-

1. How close is our Argon Triple Point to the ideal temperature of  $-189.3442\text{ }^{\circ}\text{C}$ ?
2. How long can the plateau be maintained?
3. How flat is the plateau?
4. What is the reproducibility of the plateau?
5. What is the combined uncertainty of the apparatus?

# 1: How close is our Argon Triple Point to the ideal temperature?

The Triple Point of Argon-189.3442 °C



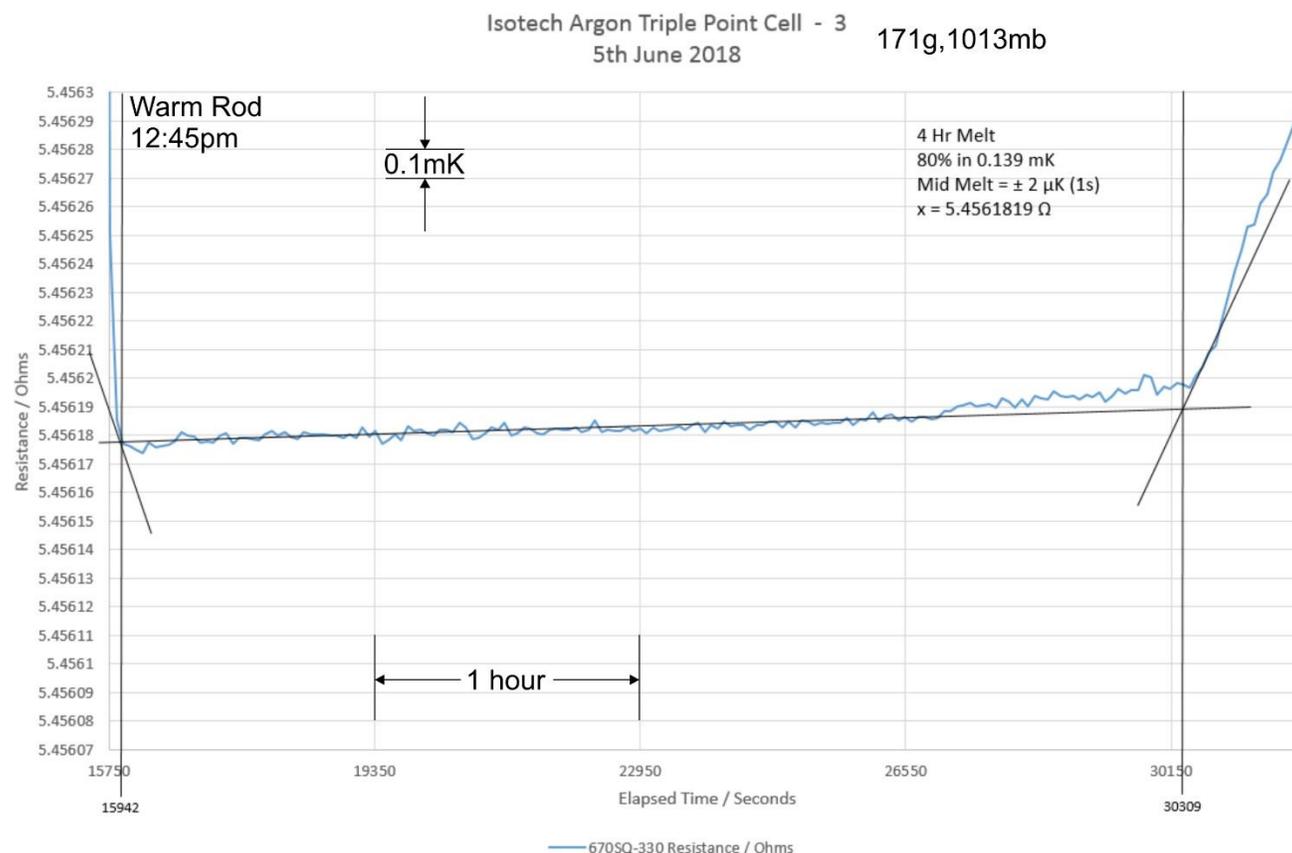
# Argon Triple Point Results

*Inter-comparisons over 3 days with an argon apparatus certified at NIST showed our realisation to be approximately **0.5mK below ITS-90***

# 2: How Long is the Plateau?

**The melt lasted some 4 hours**

The length of the plateau is long enough for premium calibrations and inter-comparisons to take place

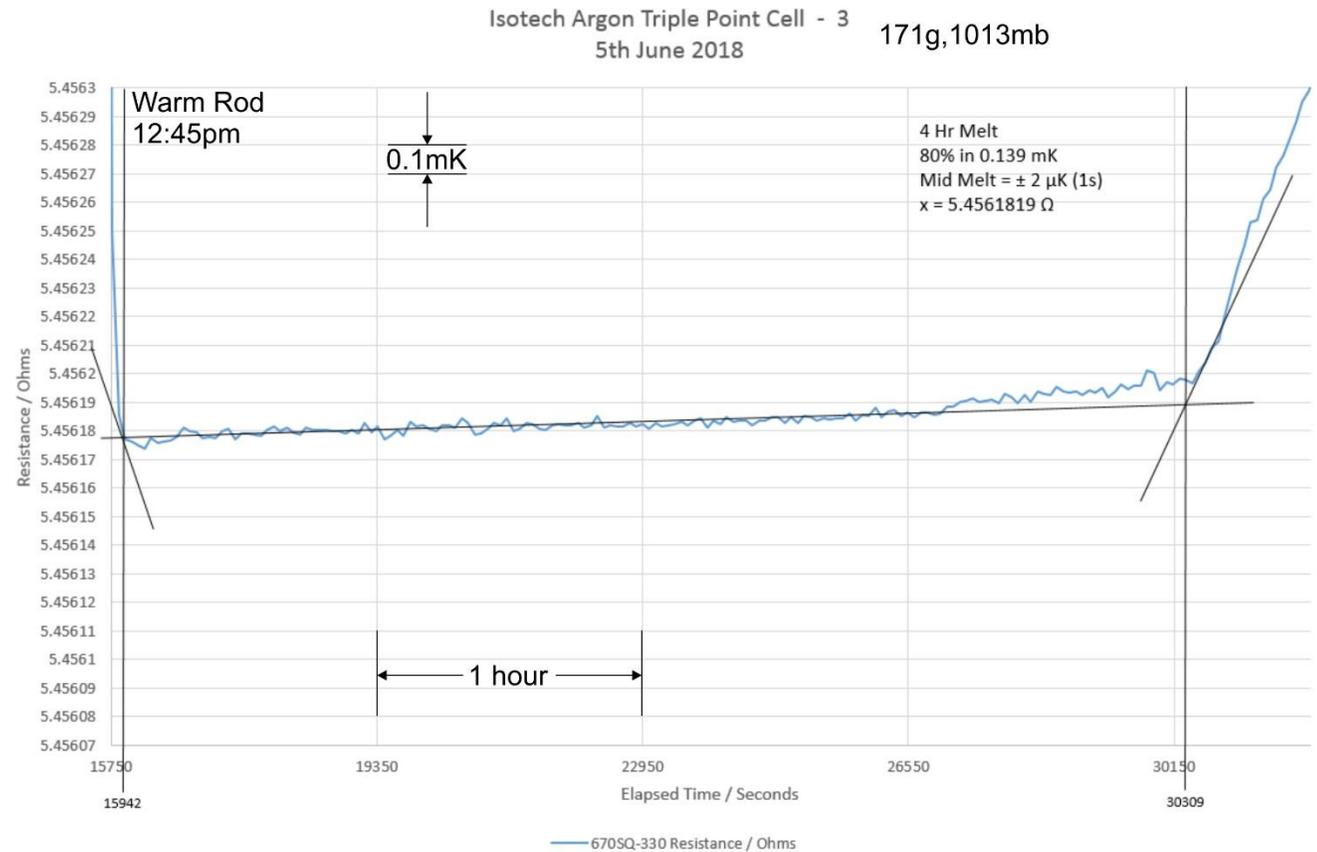


The plateau was initiated by withdrawing the thermometer monitoring the cell temperature for 20 seconds and reinserting it  
External pressure was 1013mb Weight of pressure release valve 171g

# 3: How Flat is the Plateau?

**80% of slope in 0.14mK**

The melt curve shows that the Argon is of very high purity and is uncontaminated by the contact with the cells inner surface



# 4: What is the reproducibility of the plateau?

Over the two weeks of measurements the reproducibility was  $\pm 0.1$  mK

# 5: What is the combined uncertainty of the apparatus?

It is possible to calculate the accuracy of calibration for an SPRT in our apparatus by

Analysing melt curves

Self-heating of a typical SPRT (model 670) was measured as was the immersion characteristics over the bottom 30mm

(Self-heating was typically 1mK and the immersion profile over the bottom 30mm was within the measurement uncertainty of  $\pm 17\mu\text{K}$ )

Lastly, our apparatus was compared on 3 separate days to a reference apparatus having a NIST certificate

# Argon Triple Point: Premium Calibration: 0.5 mK

An uncertainty budget for the calibration of SPRT's showing an uncertainty of  $\pm 0.5$  mK at  $k=2$

This is four times better than the original apparatus

ISOTHERMAL TECHNOLOGY LTD UKAS LABORATORY N° 0175		Date of issue	
		Issue N°	
Argon T.P. Cell Premium Calibration - $\pm 0.5$ mK UCT		Authorised by	
Budget N°.			

Note number (below)	Source of uncertainty	Value $\pm$	Unit	Probability distribution	Divisor	Sensitivity $c_i$	Standard uncertainty $u_i$ (unit)	Degrees of freedom $\nu_i$ or $\nu_r$	$u_i^2$	$u_i^4/\nu_i$
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	i	0.000000000	0
5	micro K resolution	0.000002	C	rectangular	1.73	1	0.000001	i	0.000000000	0
6	Test and Std cell slope differences	0.000183	C	rectangular	1.73	1	0.000106	i	0.000000011	0
7	Measured H/H uncertainty in Std Cell	0.000178	C	rectangular	1.73	1	0.000103	i	0.000000011	0
8	Measured H/H uncertainty in Test Cell	0.000017	C	rectangular	1.73	1	0.000010	i	0.000000000	0
9	Estimated H/H uncertainty in Std Cell	0.000017	C	rectangular	1.73	1	0.000010	i	0.000000000	0
10	Estimated H/H uncertainty in Test Cell	0.000017	C	rectangular	1.73	1	0.000010	i	0.000000000	0
11	Sprt self heating max $\Delta t$ in Std cell	0.000141	C	rectangular	1.73	1	0.000082	i	0.000000007	0
12	Sprt self heating max $\Delta t$ in Test cell	0.000167	C	rectangular	1.73	1	0.000097	i	0.000000009	0
13	Lead moisture effects	0.000010	C	rectangular	1.73	1	0.000006	i	0.000000000	0
14	Temp effect of oil bath on Std resistor	0.000001	C	rectangular	1.73	1	0.000001	i	0.000000000	0
15	Calibration of Std resistor	0.000002	C	normal	2.00	1	0.000001	i	0.000000000	0
16	Uncorrected Drift of Std Resistor	0.000000	C	rectangular	1.73	1	0.000000	i	0.000000000	0
17	Uncertainty Of standard cell	0.000280	C	normal	2.00	1	0.000140	i	0.000000020	0
$u_c$	Combined uncertainty			normal			0.000250	1895	0.000000062	2.1E-18
U	Expanded uncertainty			normal	k for $\nu_r$	2.00	0.000500	1895		

**k = 2.00      0.000500**

# Conclusion

The results above show that the 3rd version of the Isotech-Bonnier Argon Triple Point Apparatus enables long stemmed SPRT's to be calibrated to an uncertainty of  $\pm 0.5\text{mK}$  at the argon triple point at  $k=2$  confidence level. The cell in this report was  $0.5\text{mK}$  below the ITS-90 temperature of the argon triple point.

It is simple to use, cannot drift and costs considerably less than existing alternatives. Results show that it is as accurate as the best alternatives