Argon Triple Point

www.isotech.co.uk

Isothermal Technology Innovation

John Tavener

President and Head of Laboratories

jpt@isotech.co.uk

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

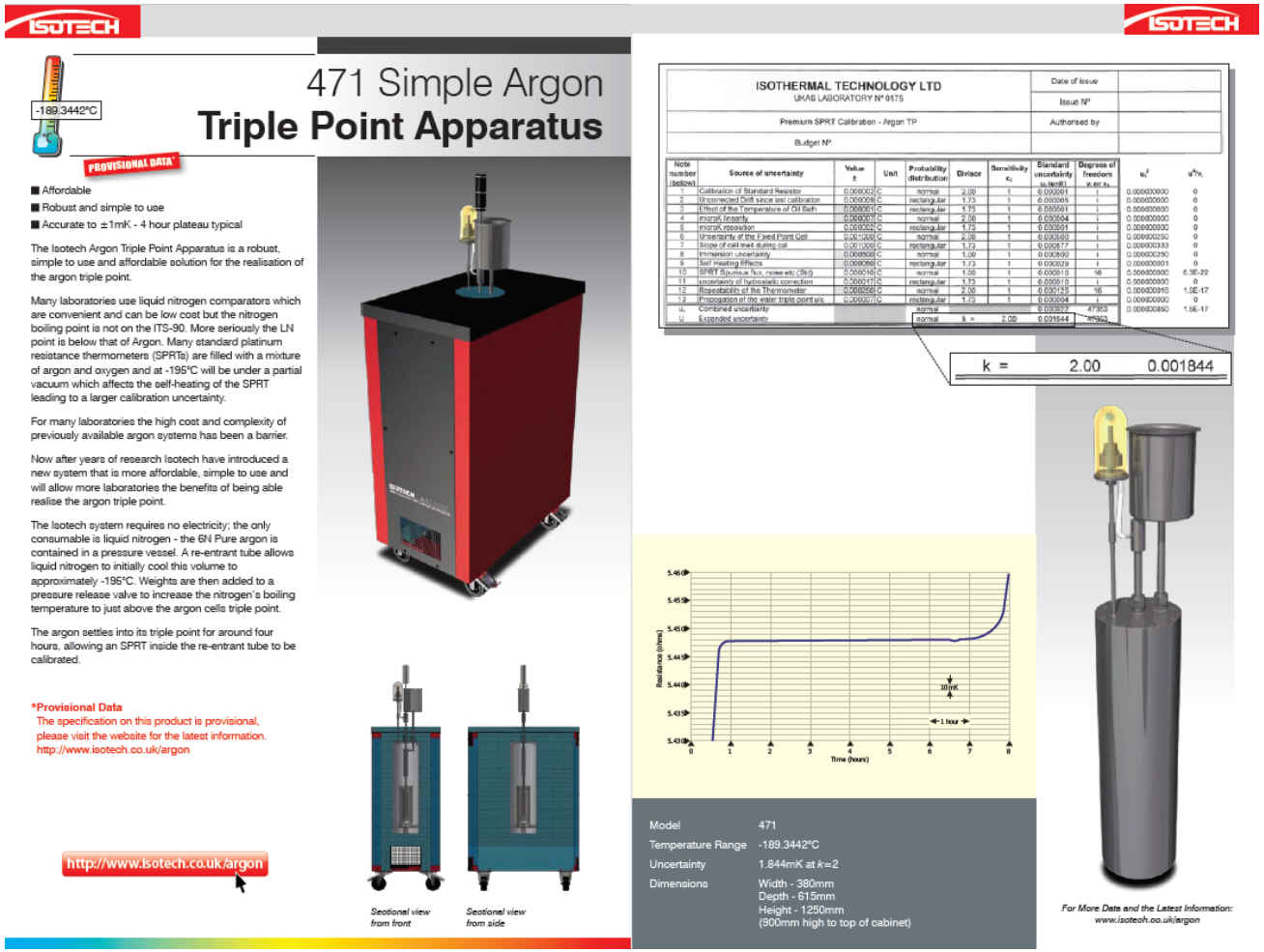
Can affect the self-heating of the SPRT leading to a larger calibration uncertainty

A New Simple Solution

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

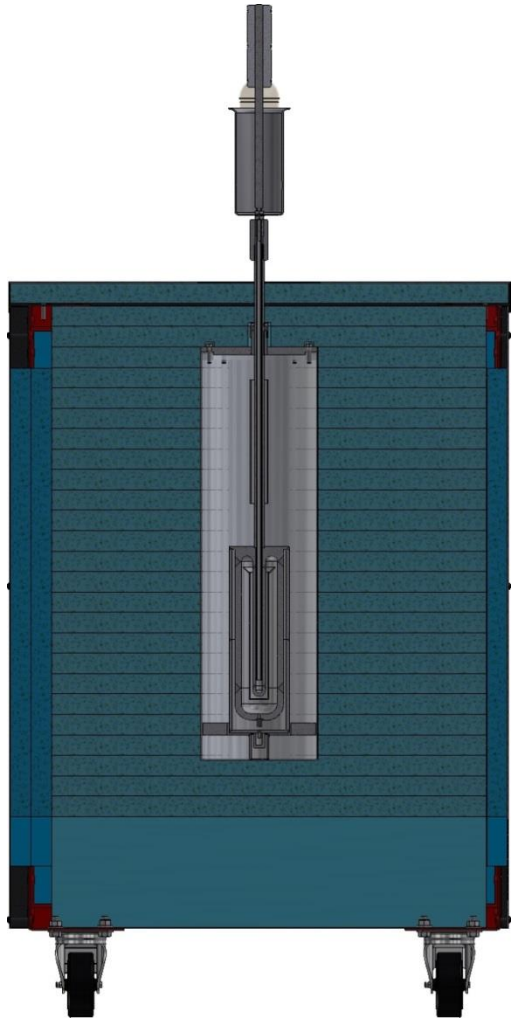
Now after years of research I have developed a new system that is more affordable, simple to use and will allow more laboratories the benefits of being able to realise the argon triple point

Isotech Model 471



Cross Section

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



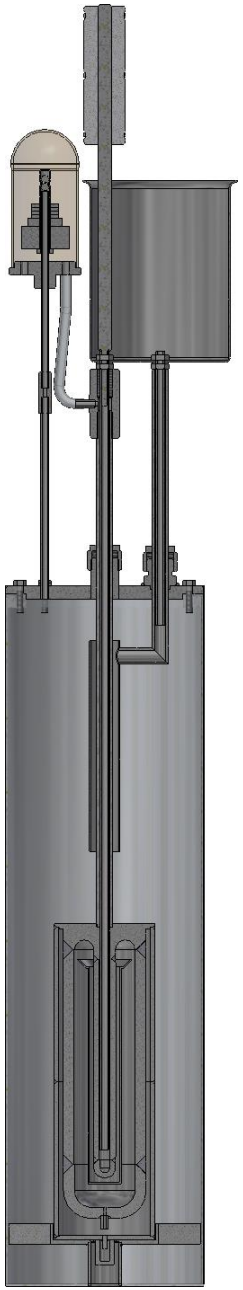
Inner Container

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

Assembly

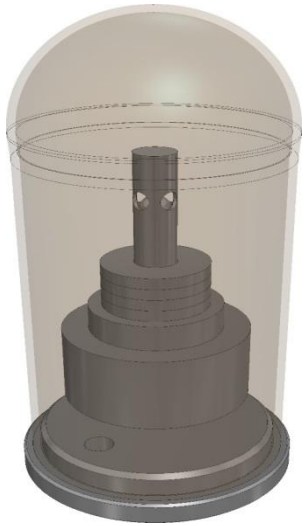
The lid has three tubes protruding – the central one goes into the argon triple point cell

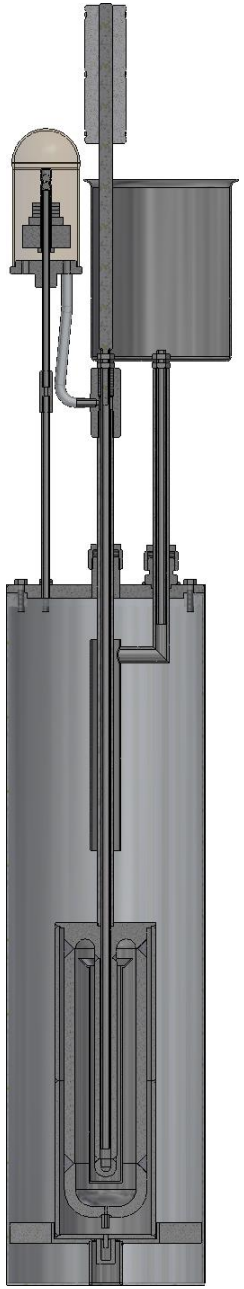
To one side is the filling tube – with a funnel. This can be removed and then the tube can is sealed



Inner Assembly

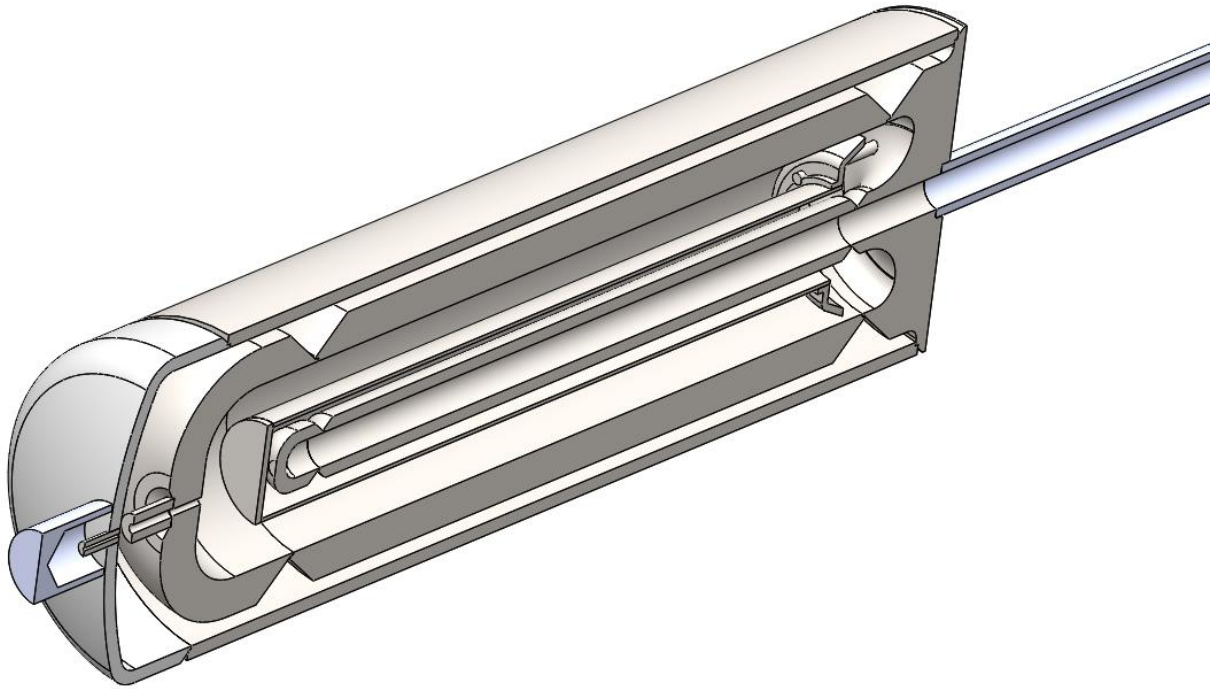
To the other side is a pressure relief valve – it controls the pressure and hence the nitrogen boiling point temperature





Inner Assembly

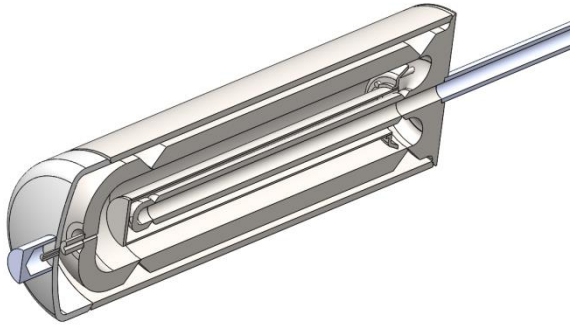
The argon triple point cell is a pressurised vessel containing pure argon.



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

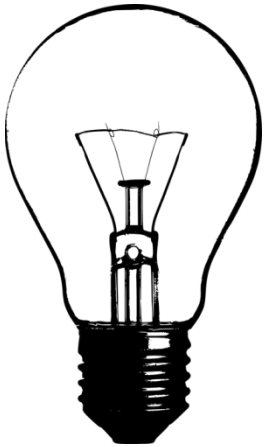
In England liquid nitrogen costs less then £2.00 per litre plus a fixed delivery charge, so a day's calibration costs between £25.00 and £50.00.

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.



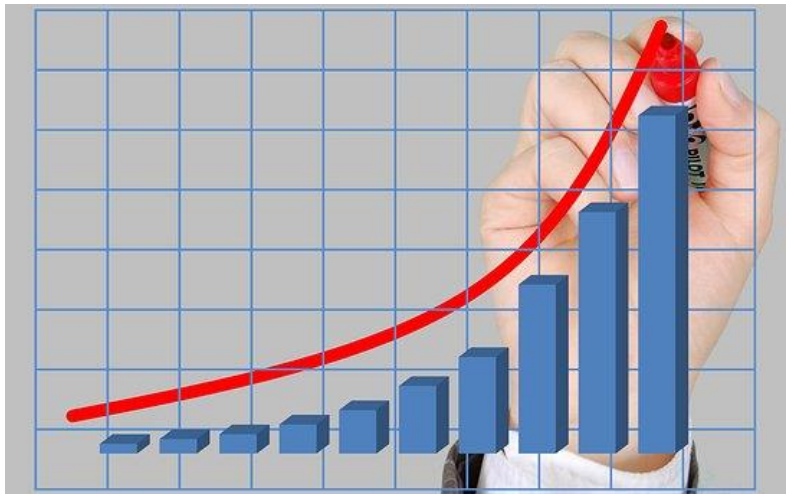
Results

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

Airgas
an Air Liquide company

Airgas Specialty Gases
Airgas USA, LLC
600 Union Landing Road
Cinnaminson, NJ 08077-0000
Airgas.com

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

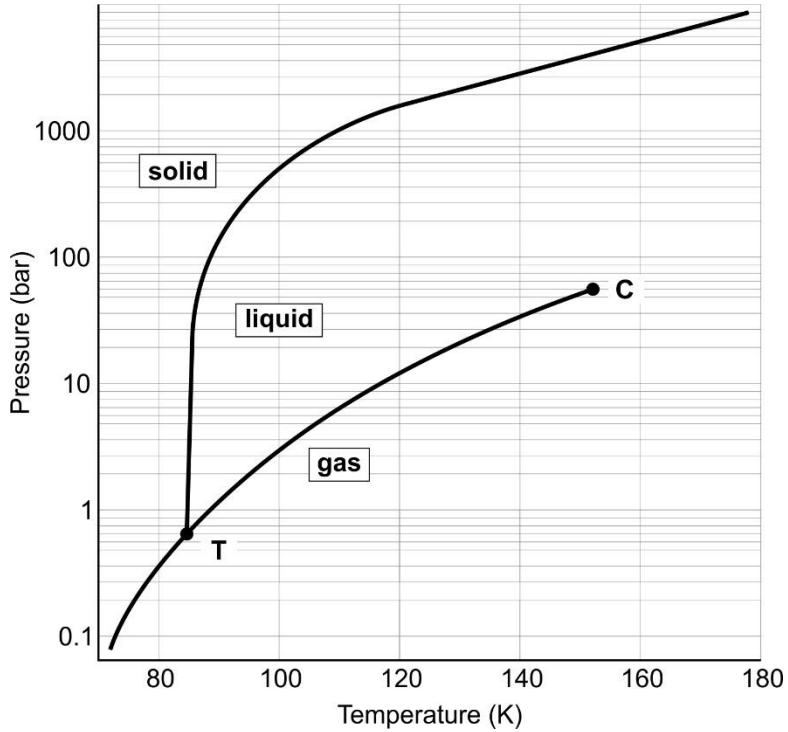
Impurities verified against analytical standards traceable to NIST by weight and/or analysis.

We now need to know the following:-

1. How close is our Argon Triple Point to the ideal temperature of -189.3442 °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



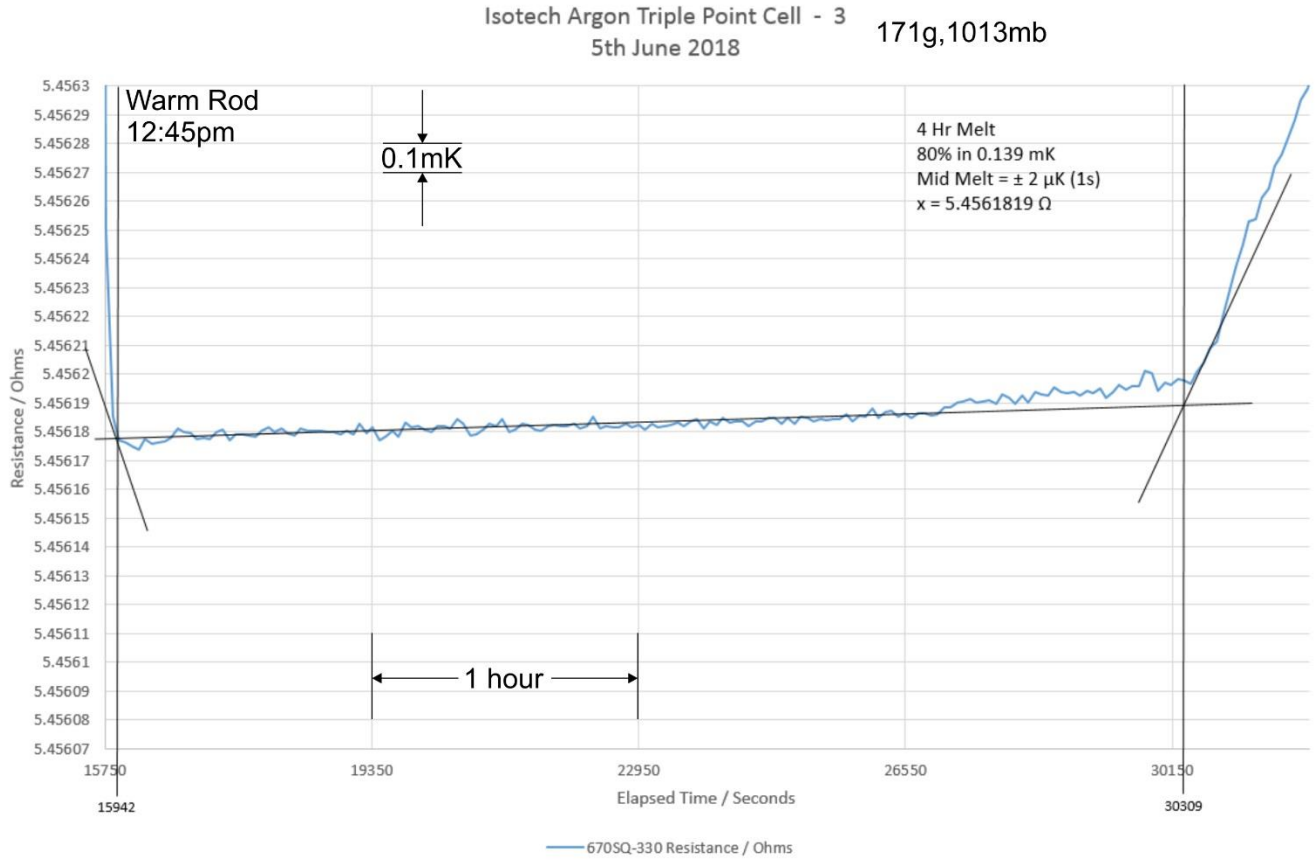
Argon Triple Point Results

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



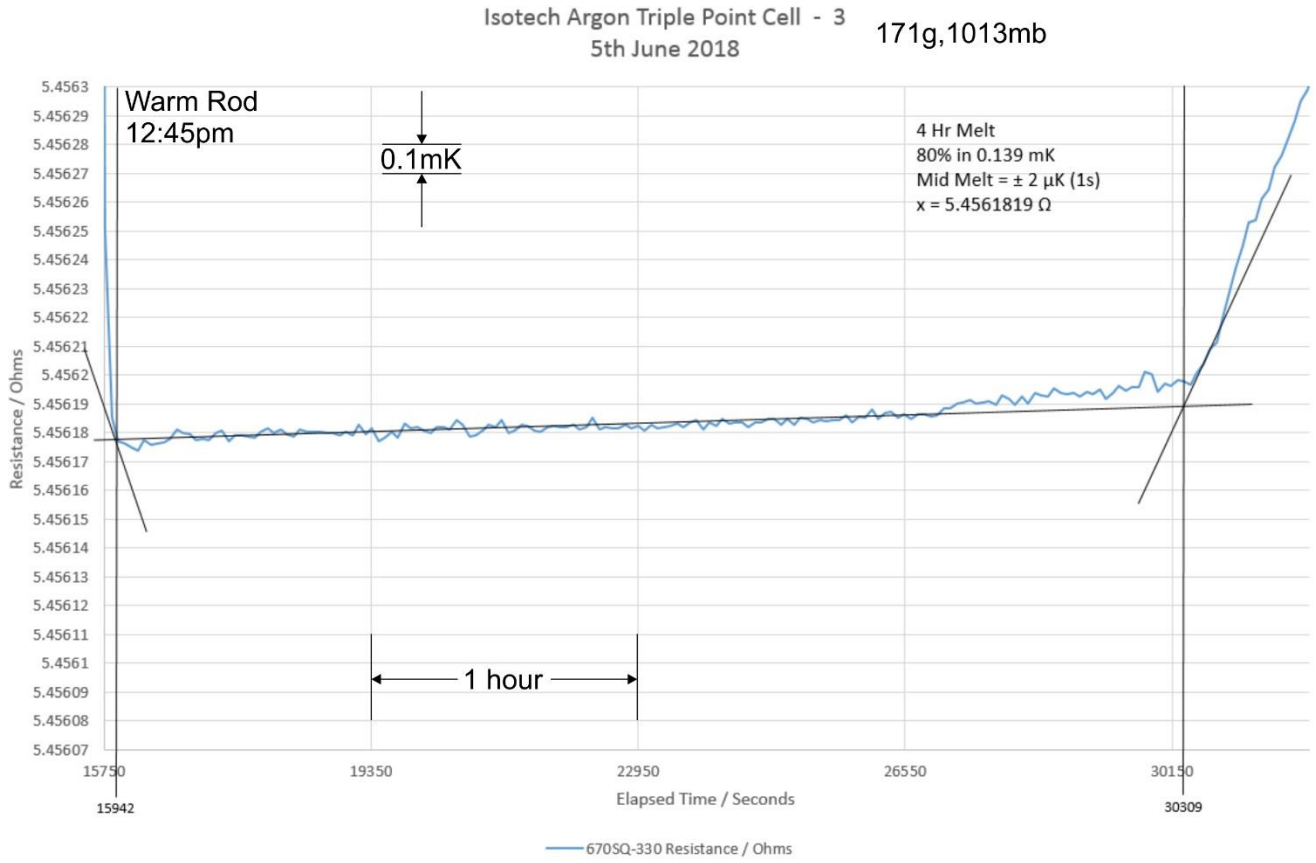
The plateau was initiated by withdrawing the thermometer monitoring the cell temperature for 20 seconds and reinserting it

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 ±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 ±17µK)

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 ±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 - ±0.5 mK UCT	Authorised by	
Budget N°.		

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_f	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	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 Δt in Std cell	0.000141	C	rectangular	1.73	1	0.000082	i	0.000000007	0
12	Sprt self heating max Δt 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 v_f	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 ±0.5mK at the argon triple point at *k*=2 confidence level. The cell in this report was 0.5mK 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