

# CERTIFICATE OF CALIBRATION

ISSUED BY: ISOTHERMAL TECHNOLOGY LIMITED.

DATE OF ISSUE: 14<sup>th</sup> October 2009

CERTIFICATE NO:



0175

## ISOTECH

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Page 1 of 7

Approved Signatory

Name:

Signature:

CUSTOMER:

CUSTOMER ORDER:

ITL REFERENCE:

DESCRIPTION: Isotech Model 493 Isothermal Tower Aluminium Fixed Point Cell.

IDENTIFICATION:

DATE OF CALIBRATION: From 6<sup>th</sup> October 2009 to 10<sup>th</sup> October 2009

BASIS OF CALIBRATION: The Isothermal Tower has been inter-compared to one of the Laboratory Reference cells.

The temperature scale in use in this Laboratory is the International Temperature Scale of 1990 (ITS 90).

This laboratory is accredited in accordance with the recognised International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer joint ISO-ILAC-IAF Communiqué dated January 2009).

The Joint Communiqué is available on the ILAC website at [www.ilac.org](http://www.ilac.org) on the publications and resources page.

**The Northern Temperature Primary Laboratory is the Calibration Laboratory of Isothermal Technology Limited**

This certificate is issued in accordance with the laboratory accreditation requirements of the United Kingdom Accreditation Service. It provides traceability of measurement to recognized national standards and to units of measurement realized at the National Physical Laboratory or other recognized national standards laboratories. This certificate may not be reproduced other than in full, except with the prior written approval of the issuing laboratory.

# CERTIFICATE OF CALIBRATION

Description: Isotech Model 493 Isothermal Tower Aluminium Fixed Point Cell

Identification:

UKAS Accredited Calibration Laboratory No. 0175

N.T.P.L. Northern Temperature Primary Laboratory, Southport, England

Tel. +44 (0) 1704 543830 / 544611 Fax. +44 (0) 1704 544799

CERTIFICATE NO:

Page 2 of 7

## MEASUREMENTS:

1. The fixed point cell within the Isothermal Tower comprises an ingot of 99.9999 (6N) pure metal in a very high purity graphite crucible, all sealed in metal.

Melt and freeze plateaux were observed, using a Standard Platinum Resistance Thermometer (SPRT) to investigate the slopes of the melt and freeze curves (ref. Isotech Journal of Thermometry Volumes 7.1 and 8.1).

2. The cell was also compared to one of the Laboratories reference cells.

For this, a monitor SPRT was placed in each cell.

The reference cell was melted and held at 5°C above the melt temperature overnight whilst the Isothermal Tower was melted and held at 2°C above the melt temperature overnight.

The following day both cells were put onto their freeze plateau in the prescribed manner at the same time. The plateau temperatures of the test and reference cells were recorded for a period of 20 minutes after stabilisation.

The SPRT's were then exchanged and the readings of temperature difference between the test cell and the reference cell recorded for 20 minutes.

The SPRT's were exchanged once more and readings of temperature recorded as before.

Finally the remainder of the freeze plateau for both the test and reference cells was recorded to establish that the measurements of temperature difference had been made on the first 25% of the freeze plateau. Each freeze plateau lasted in excess of 10 hours.

The inter-comparison was repeated again on subsequent days using separate freezes.

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# CERTIFICATE OF CALIBRATION

Description: Isotech Model 493 Isothermal Tower Aluminium Fixed Point Cell

Identification:

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## RESULTS:

The temperature of the test cell has been compared to that of the Isotech Open Primary Reference Aluminium cell which has traceability to the reference cell of NIST, and for which an uncertainty of  $\pm 0.80$  mK (2s) has been established.

The equilibrium temperature of the calibrated cell differed from that of the reference cell as follows;

INTER-COMPARISON	TEST CELL – REFERENCE CELL (mK)
1	-1.622
2	-1.749

Mean Difference = -1.686 mK

Standard Deviation = +/- 0.127 mK (2s)

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CERTIFICATE NO:

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## UNCERTAINTIES:

The principle components of uncertainty in the inter-comparison of the cells were:

### TYPE A: (statistical)

1. The short term reproducibility of the SPRT in the cells and the effects caused by thermal shock during transfer from one cell to the other.
2. Statistical uncertainties in the resistance measurements, particularly those due to electrical interference from the furnace heater and controller.

### TYPE B:

3. The effects of impurities.
4. The effects of different thermal conditions in the furnaces used (ie. temperature gradients)
5. Uncertainties resulting from the bridge measurements.

Reproducibility of the SPRT and the measurements, and interference effects (1 & 2 above) cause Type A (statistical) uncertainties.

Impurities contribute to the Type B uncertainty in the freezing point and are estimated from the quality of the freezing plateau obtained.

The uncertainty estimates at approximately 95 % confidence (2 standard deviations) for the inter-comparison are given in Table 1. The uncertainty of the inter-comparison was obtained by combining the component uncertainties in quadrature (ie. by calculating the square root of the sum of the squares), and is also at 2 standard deviations.

Table 1.

Type A	1 & 2	$\pm 0.421$ mK
Type B	3, 4 & 5	$\pm 1.775$ mK
Uncertainty of Inter-comparison (2s)		$\pm 1.824$ mK

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The uncertainty of the equilibrium temperature of the calibrated cell is  $\pm 2$  mK. (2s), this does not include the mean temperature difference of the calibrated cell to the reference cell.

The reported expanded uncertainty is based on a standard uncertainty multiplied by a coverage factor  $k = 2$ , providing a level of confidence of approximately 95%.

The uncertainty evaluation has been carried out in accordance with UKAS requirements.

## NOTES:

1. There is a temperature variation with depth below the metal surface where the ITS 90 defined temperature applies.
  2. In the calibrated cell the distance from the metal surface to the bottom of the re entrant well is  $180 \text{ mm} \pm 5 \text{ mm}$ .
  3. The hydrostatic temperature variation of this cell is 0.0016 mK per mm.
  4. The correction should be calculated using the distance between the metal surface and the mean sensing position of the thermometer in the re entrant well.
  5. The ambient temperature was  $20 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$  throughout the calibration.
  6. Throughout the inter-comparison the Zone Controller of the Isothermal Tower was set to 12.0.
-

# CERTIFICATE OF CALIBRATION

Description: Isotech Model 493 Isothermal Tower Aluminium Fixed Point Cell

Identification:

UKAS Accredited Calibration Laboratory No. 0175

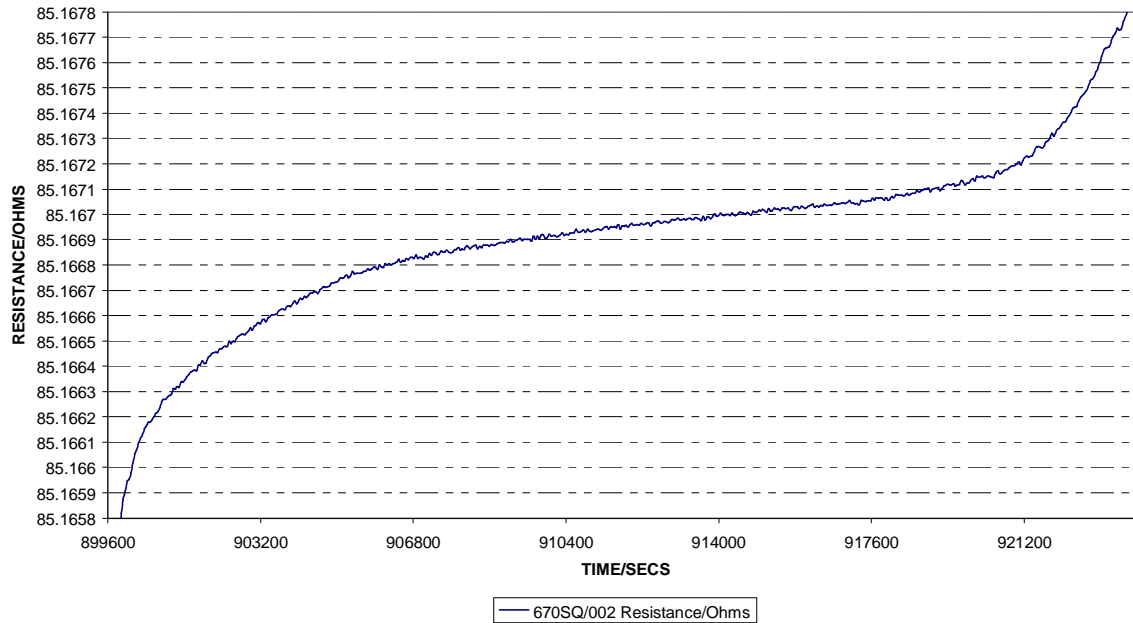
N.T.P.L. Northern Temperature Primary Laboratory, Southport, England

Tel. +44 (0) 1704 543830 / 544611 Fax. +44 (0) 1704 544799

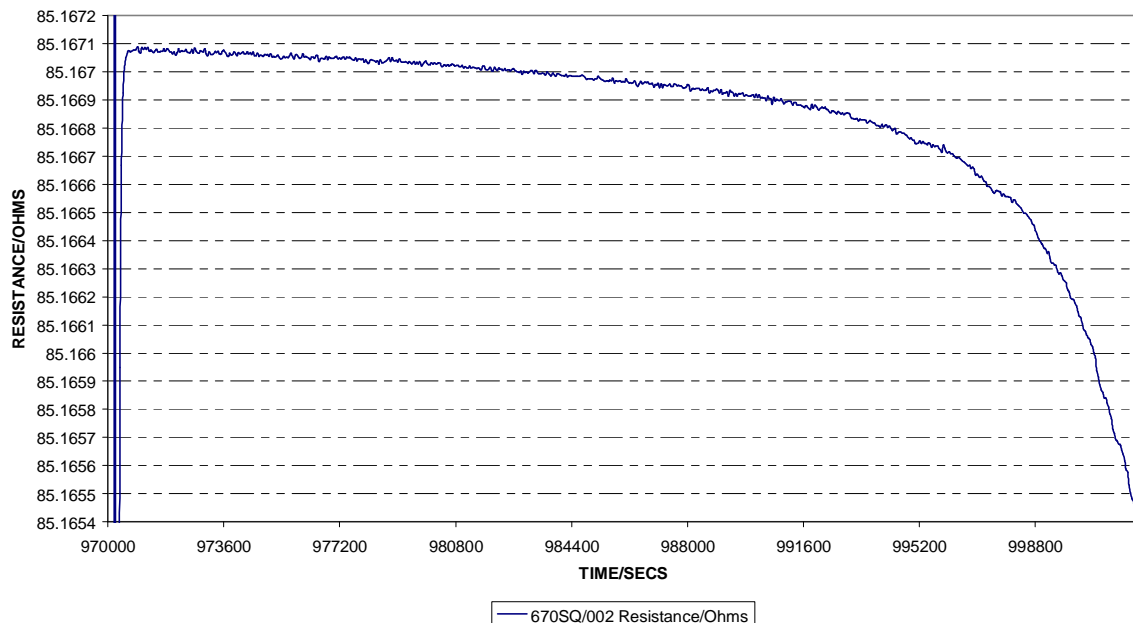
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## Isothermal Tower Aluminium Fixed Point Cell MELT PLATEAU 4 17th September 2009 (S.P. 660.8 Deg C, Zone 12.0)



## Isothermal Tower Aluminium Fixed Point Cell FREEZE PLATEAU 4 18th September 2009 (S.P. 660.2 Deg C, Zone 12.0)



# CERTIFICATE OF CALIBRATION

Description: Isotech Model 493 Isothermal Tower Aluminium Fixed Point Cell

Identification:

UKAS Accredited Calibration Laboratory No. 0175

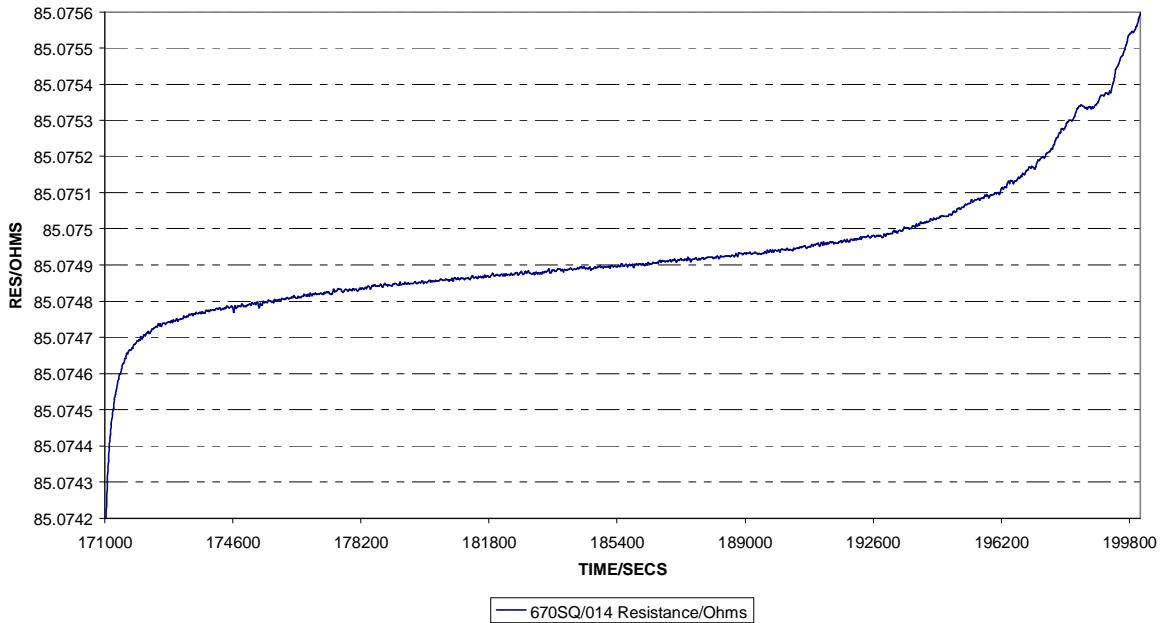
N.T.P.L. Northern Temperature Primary Laboratory, Southport, England

Tel. +44 (0) 1704 543830 / 544611 Fax. +44 (0) 1704 544799

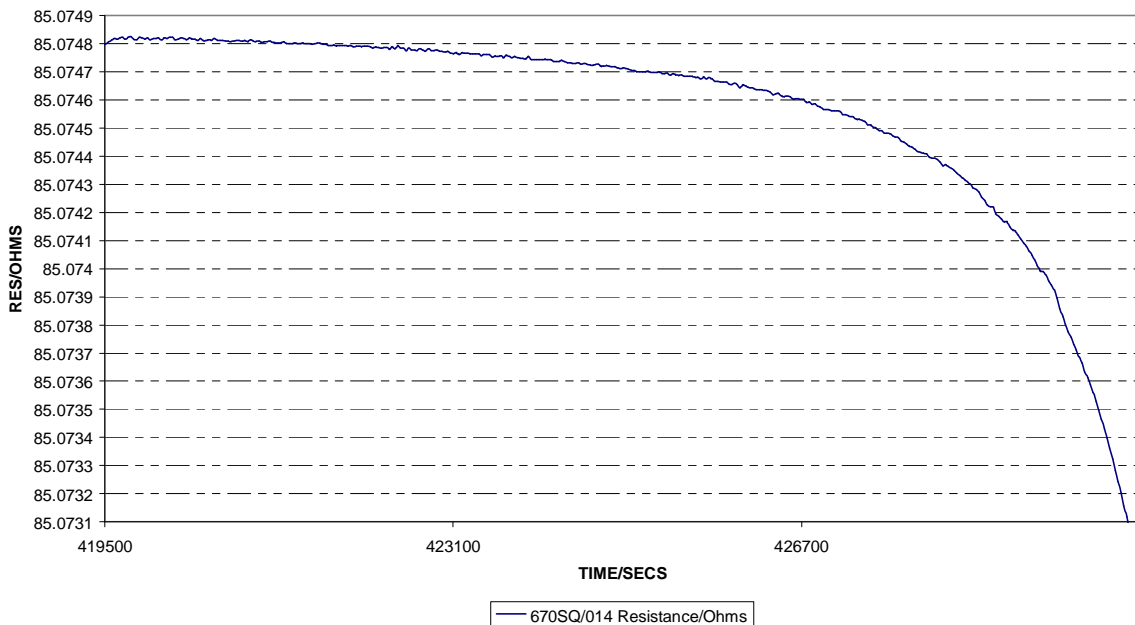
CERTIFICATE NO:

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## Isothermal Tower Aluminium Fixed Point Cell MELT PLATEAU 5 2nd October 2009 (S.P. 661.4 Deg C, Zone 12.0)



## Isothermal Tower Aluminium Fixed Point Cell FREEZE PLATEAU 5 5th October 2009 (S.P. 660.0 Deg C, Zone 12.0)



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Page 1 of 8

Approved Signatory

Name:

Signature:

CUSTOMER:

CUSTOMER ORDER:

ITL REFERENCE:

DESCRIPTION: Isotech Model 491 Isothermal Tower Tin Fixed Point Cell.

IDENTIFICATION:

DATE OF CALIBRATION: From 24<sup>th</sup> September 2009 to 1<sup>st</sup> October 2009

BASIS OF CALIBRATION: The Isothermal Tower has been inter-compared to one of the Laboratory Reference cells.

The temperature scale in use in this Laboratory is the International Temperature Scale of 1990 (ITS 90).

This laboratory is accredited in accordance with the recognised International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer joint ISO-ILAC-IAF Communiqué dated January 2009).

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Description: Isotech Model 491 Isothermal Tower Tin Fixed Point Cell

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## MEASUREMENTS:

1. The fixed point cell within the Isothermal Tower comprises an ingot of 99.9999 (6N) pure metal in a very high purity graphite crucible, all sealed in metal. Melt and freeze plateaux were observed, using a Standard Platinum Resistance Thermometer (SPRT) to investigate the slopes of the melt and freeze curves (ref. Isotech Journal of Thermometry Volumes 7.1 and 8.1).

2. The cell was also compared to one of the Laboratories reference cells.

For this, a monitor SPRT was placed in each cell.

The reference cell was melted and held at 5°C above the melt temperature overnight whilst the Isothermal Tower was melted and held at 2°C above the melt temperature overnight.

The following day both cells were put onto their freeze plateau in the prescribed manner at the same time. The plateau temperatures of the test and reference cells were recorded for a period of 20 minutes after stabilisation.

The SPRT's were then exchanged and the readings of temperature difference between the test cell and the reference cell recorded for 20 minutes.

The SPRT's were exchanged once more and readings of temperature recorded as before.

Finally the remainder of the freeze plateau for both the test and reference cells was recorded to establish that the measurements of temperature difference had been made on the first 25% of the freeze plateau. Each freeze plateau lasted in excess of 10 hours.

The inter-comparison was repeated again on subsequent days using separate freezes.

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## RESULTS:

The temperature of the test cell has been compared to that of the Isotech Open Primary Reference Tin cell which has traceability to the reference cell of NIST, and for which an uncertainty of  $\pm 0.29$  mK (2s) has been established.

The equilibrium temperature of the calibrated cell differed from that of the reference cell as follows;

INTER-COMPARISON	TEST CELL – REFERENCE CELL (mK)
1	-0.448
2	-0.426

Mean Difference = -0.437 mK

Standard Deviation = +/- 0.022 mK (2s)

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CERTIFICATE NO:

Page 4 of 8

## UNCERTAINTIES:

The principle components of uncertainty in the inter-comparison of the cells were:

### TYPE A: (statistical)

1. The short term reproducibility of the SPRT in the cells and the effects caused by thermal shock during transfer from one cell to the other.
2. Statistical uncertainties in the resistance measurements, particularly those due to electrical interference from the furnace heater and controller.

### TYPE B:

3. The effects of impurities.
4. The effects of different thermal conditions in the furnaces used (ie. temperature gradients)
5. Uncertainties resulting from the bridge measurements.

Reproducibility of the SPRT and the measurements, and interference effects (1 & 2 above) cause Type A (statistical) uncertainties.

Impurities contribute to the Type B uncertainty in the freezing point and are estimated from the quality of the freezing plateau obtained.

The uncertainty estimates at approximately 95 % confidence (2 standard deviations) for the inter-comparison are given in Table 1. The uncertainty of the inter-comparison was obtained by combining the component uncertainties in quadrature (ie. by calculating the square root of the sum of the squares), and is also at 2 standard deviations.

Table 1.

Type A	1 & 2	$\pm 0.317$ mK
Type B	3, 4 & 5	$\pm 0.673$ mK
Uncertainty of Inter-comparison (2s)		$\pm 0.744$ mK

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CERTIFICATE NO:

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The uncertainty of the equilibrium temperature of the calibrated cell is  $\pm 0.80$  mK. (2s), this does not include the mean temperature difference of the calibrated cell to the reference cell.

The reported expanded uncertainty is based on a standard uncertainty multiplied by a coverage factor  $k = 2$ , providing a level of confidence of approximately 95%.

The uncertainty evaluation has been carried out in accordance with UKAS requirements.

## NOTES:

1. There is a temperature variation with depth below the metal surface where the ITS 90 defined temperature applies.
  2. In the calibrated cell the distance from the metal surface to the bottom of the re entrant well is  $180 \text{ mm} \pm 5 \text{ mm}$ .
  3. The hydrostatic temperature variation of this cell is  $0.0022 \text{ mK per mm}$ .
  4. The correction should be calculated using the distance between the metal surface and the mean sensing position of the thermometer in the re entrant well.
  5. The ambient temperature was  $20 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$  throughout the calibration.
  6. Throughout the inter-comparison the Zone Controller of the Isothermal Tower was set to 4.0.
-

# CERTIFICATE OF CALIBRATION

Description: Isotech Model 491 Isothermal Tower Tin Fixed Point Cell

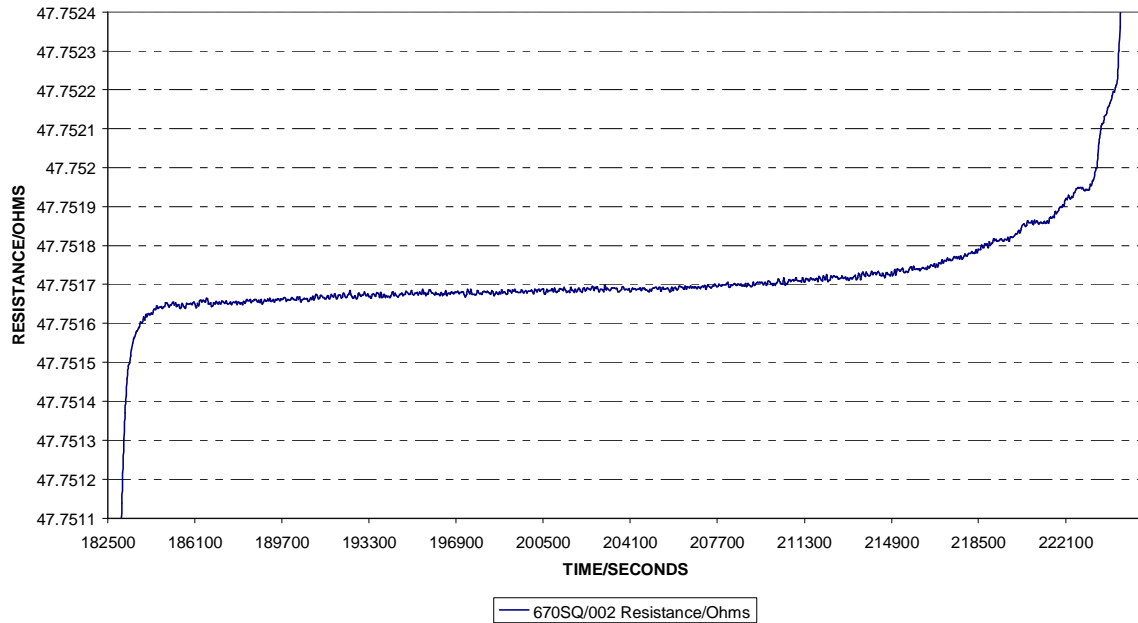
## Identification:

UKAS Accredited Calibration Laboratory No. 0175  
N.T.P.L. Northern Temperature Primary Laboratory, Southport, England  
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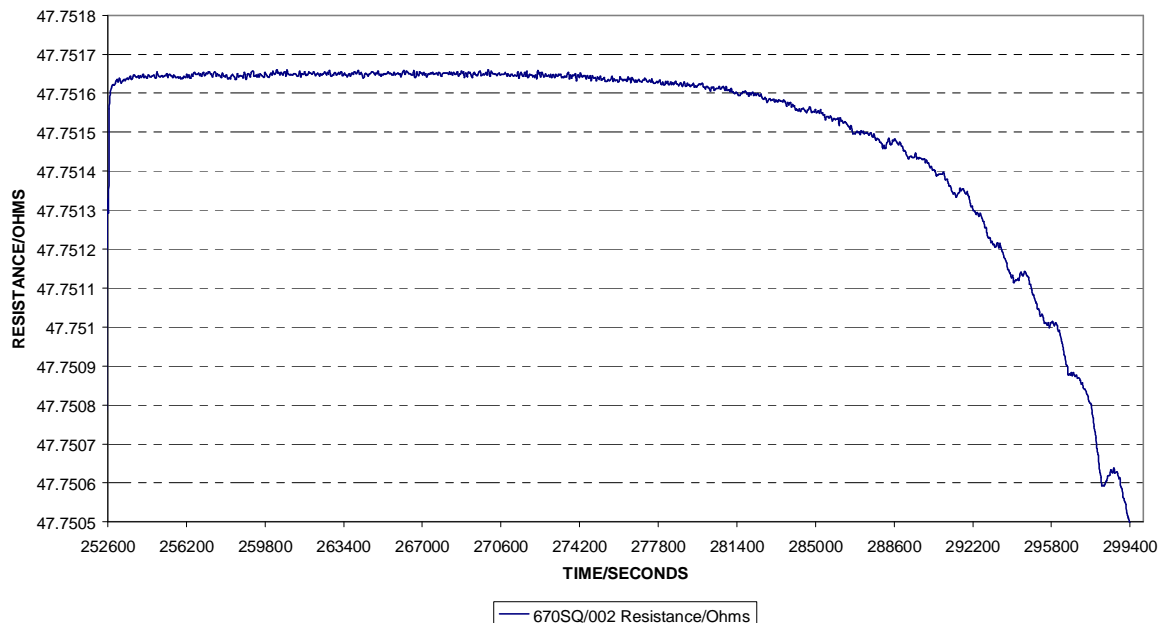
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### Isothermal Tower Tin Fixed Point Cell MELT PLATEAU 2 (S.P. 231.6 Deg C, Zone 4.0) 14th August 2009



### Isothermal Tower Tin Fixed Point Cell FREEZE PLATEAU 2 (S.P. 231.0 Deg C, Zone 4.0) 15th August 2009



# CERTIFICATE OF CALIBRATION

Description: Isotech Model 491 Isothermal Tower Tin Fixed Point Cell

Identification:

UKAS Accredited Calibration Laboratory No. 0175

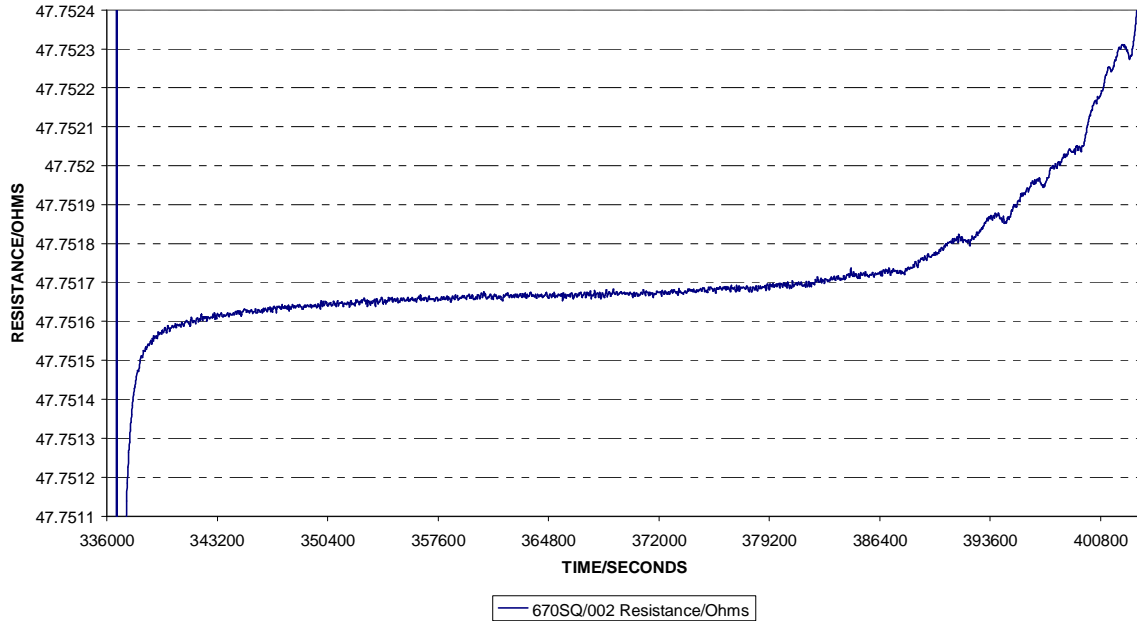
N.T.P.L. Northern Temperature Primary Laboratory, Southport, England

Tel. +44 (0) 1704 543830 / 544611 Fax. +44 (0) 1704 544799

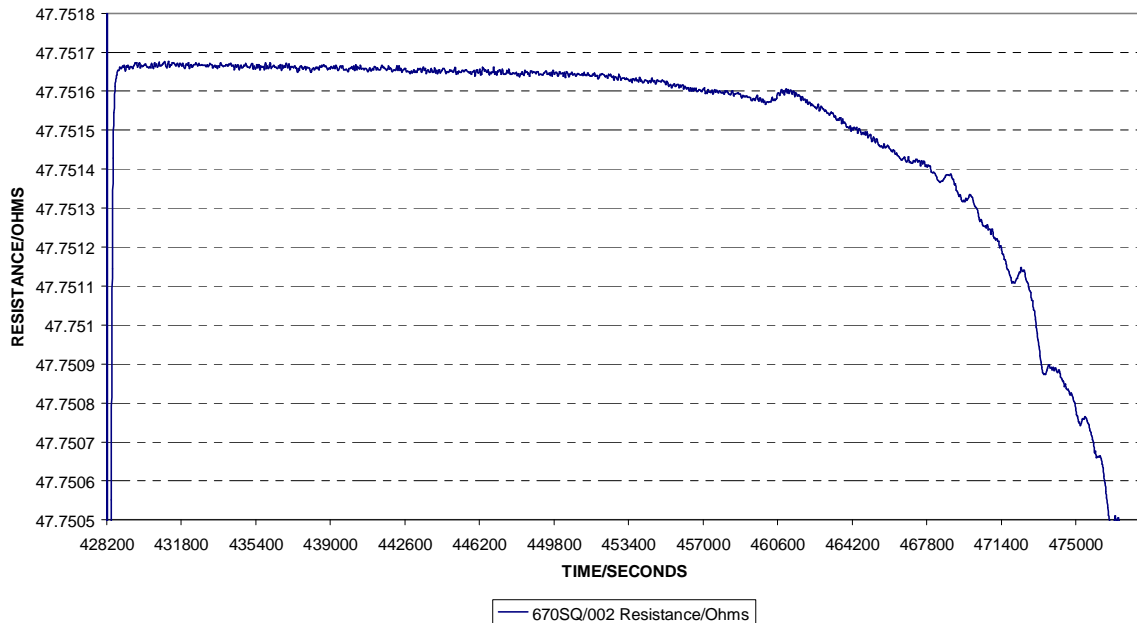
CERTIFICATE NO:

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## Isothermal Tower Tin Fixed Point Cell MELT PLATEAU 3 (S.P. 231.6 Deg C, Zone 4.0) 16th August 2009



## Isothermal Tower Tin Fixed Point Cell FREEZE PLATEAU 3 (S.P. 231.0 Deg C, Zone 4.0) 17th August 2009



# CERTIFICATE OF CALIBRATION

Description: Isotech Model 491 Isothermal Tower Tin Fixed Point Cell

Identification:

UKAS Accredited Calibration Laboratory No. 0175

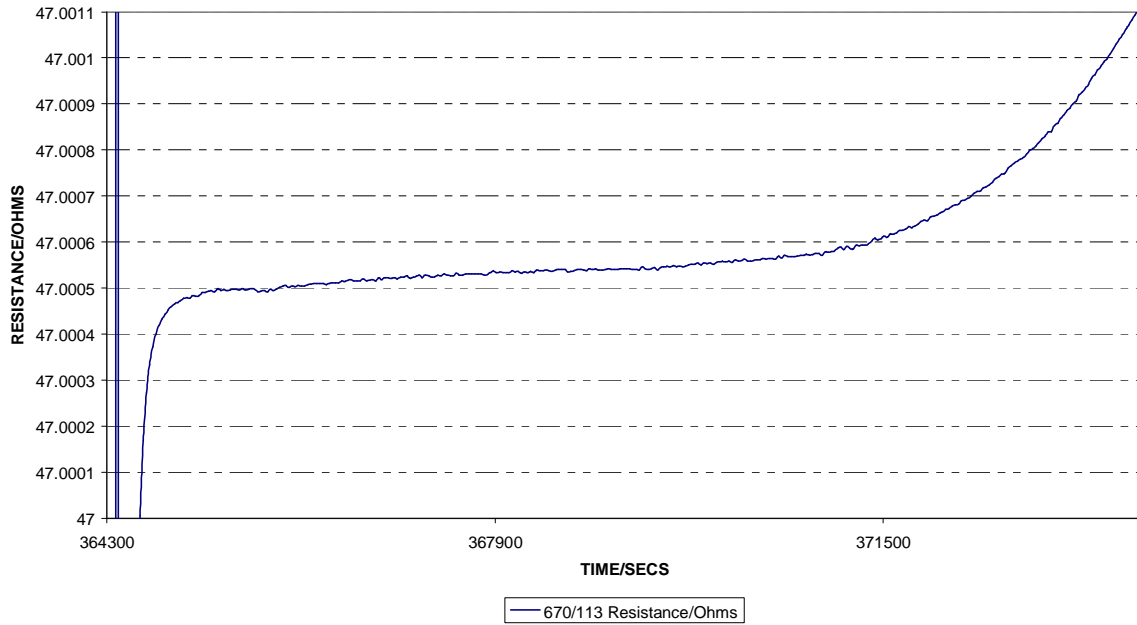
N.T.P.L. Northern Temperature Primary Laboratory, Southport, England

Tel. +44 (0) 1704 543830 / 544611 Fax. +44 (0) 1704 544799

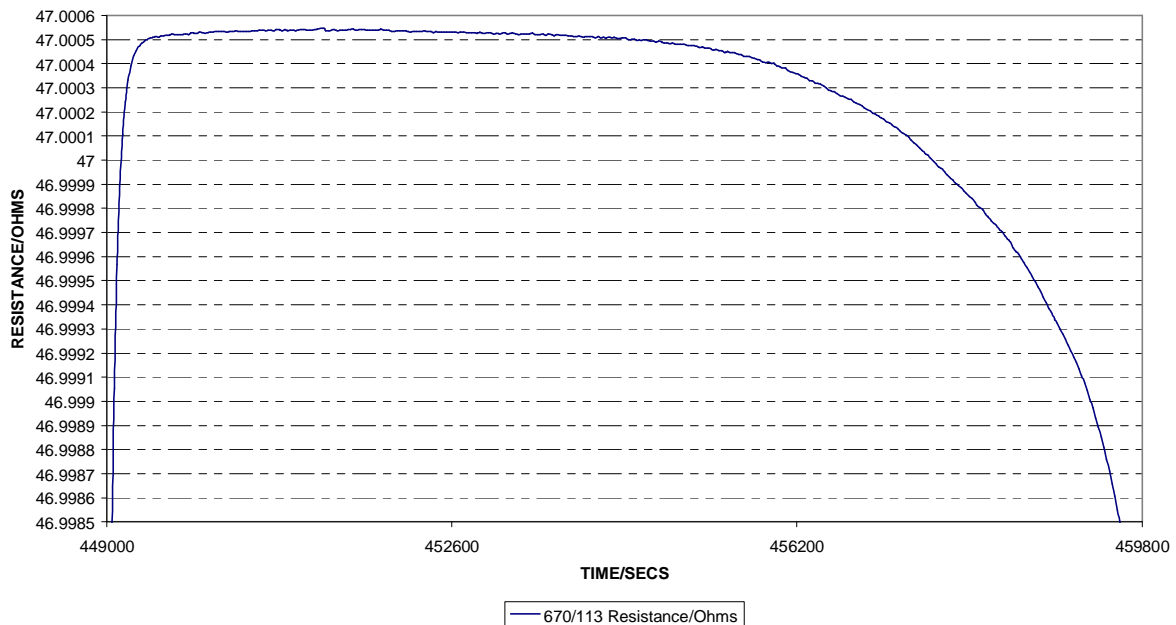
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## Isothermal Tower Tin Fixed Point Cell MELT PLATEAU 4 21st September 2009 (S.P. 232.8 Deg C, Zone 4.0).



## Isothermal Tower Tin Fixed Point Cell FREEZE PLATEAU 4 22nd September 2009 (S.P. 230.0 Deg C, Zone 4.0).



# CERTIFICATE OF CALIBRATION

ISSUED BY: ISOTHERMAL TECHNOLOGY LIMITED.

DATE OF ISSUE: 20<sup>th</sup> October 2009

CERTIFICATE NO:



0175

## ISOTECH

# N. T. P. L.

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Email [info@isotech.co.uk](mailto:info@isotech.co.uk)

Page 1 of 7

Approved Signatory

Name:

Signature:

CUSTOMER

CUSTOMER ORDER:

ITL REFERENCE:

DESCRIPTION: Isotech Model 492 Isothermal Tower Zinc Fixed Point Cell.

IDENTIFICATION:

DATE OF CALIBRATION: From 14<sup>th</sup> October 2009 to 20<sup>th</sup> October 2009

BASIS OF CALIBRATION: The Isothermal Tower has been inter-compared to one of the Laboratory Reference cells.

The temperature scale in use in this Laboratory is the International Temperature Scale of 1990 (ITS 90).

This laboratory is accredited in accordance with the recognised International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer joint ISO-ILAC-IAF Communiqué dated January 2009).

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Page 2 of 7

## MEASUREMENTS:

1. The fixed point cell within the Isothermal Tower comprises an ingot of 99.9999 (6N) pure metal in a very high purity graphite crucible, all sealed in metal.

Melt and freeze plateaux were observed, using a Standard Platinum Resistance Thermometer (SPRT) to investigate the slopes of the melt and freeze curves (ref. Isotech Journal of Thermometry Volumes 7.1 and 8.1).

2. The cell was also compared to one of the Laboratories reference cells.

For this, a monitor SPRT was placed in each cell.

The reference cell was melted and held at 5°C above the melt temperature overnight whilst the Isothermal Tower was melted and held at 2°C above the melt temperature overnight.

The following day both cells were put onto their freeze plateau in the prescribed manner at the same time. The plateau temperatures of the test and reference cells were recorded for a period of 20 minutes after stabilisation.

The SPRT's were then exchanged and the readings of temperature difference between the test cell and the reference cell recorded for 20 minutes.

The SPRT's were exchanged once more and readings of temperature recorded as before.

Finally the remainder of the freeze plateau for both the test and reference cells was recorded to establish that the measurements of temperature difference had been made on the first 25% of the freeze plateau. Each freeze plateau lasted in excess of 12 hours.

The inter-comparison was repeated again on subsequent days using separate freezes.

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## RESULTS:

The temperature of the test cell has been compared to that of the Isotech Open Primary Reference Zinc cell which has traceability to the reference cell of NIST, and for which an uncertainty of  $\pm 0.52$  mK (2s) has been established.

The equilibrium temperature of the calibrated cell differed from that of the reference cell as follows;

INTER-COMPARISON	TEST CELL – REFERENCE CELL (mK)
1	0.767
2	0.808

Mean Difference = 0.788 mK

Standard Deviation = +/- 0.041 mK (2s)

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## UNCERTAINTIES:

The principle components of uncertainty in the inter-comparison of the cells were:

### TYPE A: (statistical)

1. The short term reproducibility of the SPRT in the cells and the effects caused by thermal shock during transfer from one cell to the other.
2. Statistical uncertainties in the resistance measurements, particularly those due to electrical interference from the furnace heater and controller.

### TYPE B:

3. The effects of impurities.
4. The effects of different thermal conditions in the furnaces used (ie. temperature gradients)
5. Uncertainties resulting from the bridge measurements.

Reproducibility of the SPRT and the measurements, and interference effects (1 & 2 above) cause Type A (statistical) uncertainties.

Impurities contribute to the Type B uncertainty in the freezing point and are estimated from the quality of the freezing plateau obtained.

The uncertainty estimates at approximately 95 % confidence (2 standard deviations) for the inter-comparison are given in Table 1. The uncertainty of the inter-comparison was obtained by combining the component uncertainties in quadrature (ie. by calculating the square root of the sum of the squares), and is also at 2 standard deviations.

Table 1.

Type A	1 & 2	$\pm 0.360$ mK
Type B	3, 4 & 5	$\pm 0.773$ mK
Uncertainty of Inter-comparison (2s)		$\pm 0.853$ mK

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The uncertainty of the equilibrium temperature of the calibrated cell is  $\pm 1$  mK. (2s), this does not include the mean temperature difference of the calibrated cell to the reference cell.

The reported expanded uncertainty is based on a standard uncertainty multiplied by a coverage factor  $k = 2$ , providing a level of confidence of approximately 95%.

The uncertainty evaluation has been carried out in accordance with UKAS requirements.

## NOTES:

1. There is a temperature variation with depth below the metal surface where the ITS 90 defined temperature applies.
  2. In the calibrated cell the distance from the metal surface to the bottom of the re entrant well is  $180 \text{ mm} \pm 5 \text{ mm}$ .
  3. The hydrostatic temperature variation of this cell is  $0.0027 \text{ mK per mm}$ .
  4. The correction should be calculated using the distance between the metal surface and the mean sensing position of the thermometer in the re entrant well.
  5. The ambient temperature was  $20 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$  throughout the calibration.
  6. Throughout the inter-comparison the Zone Controller of the Isothermal Tower was set to 13.0.
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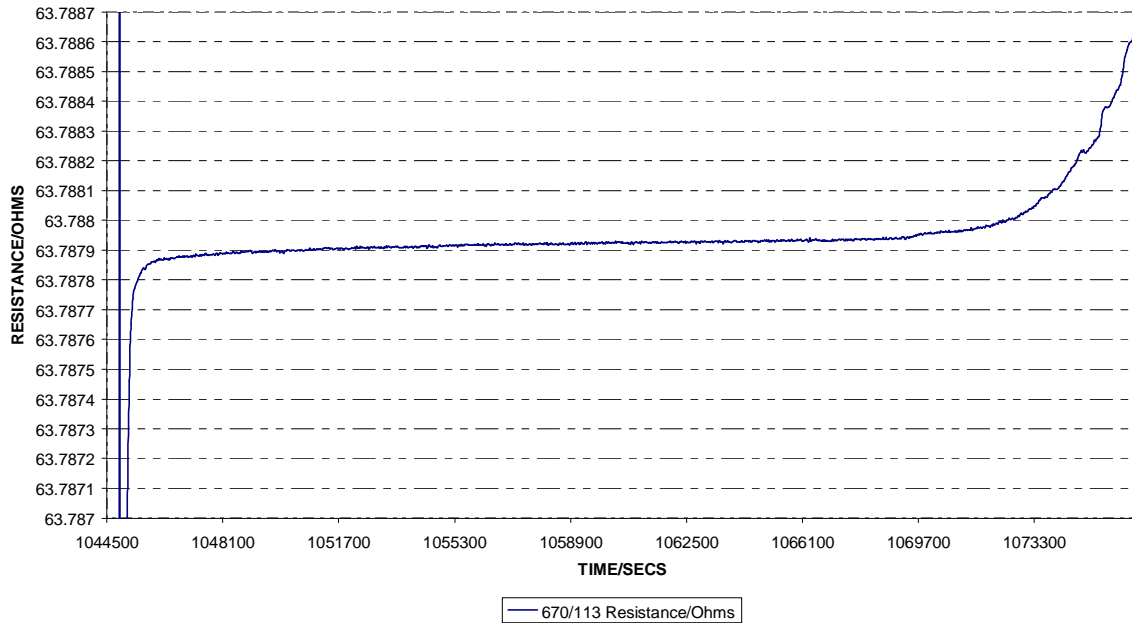
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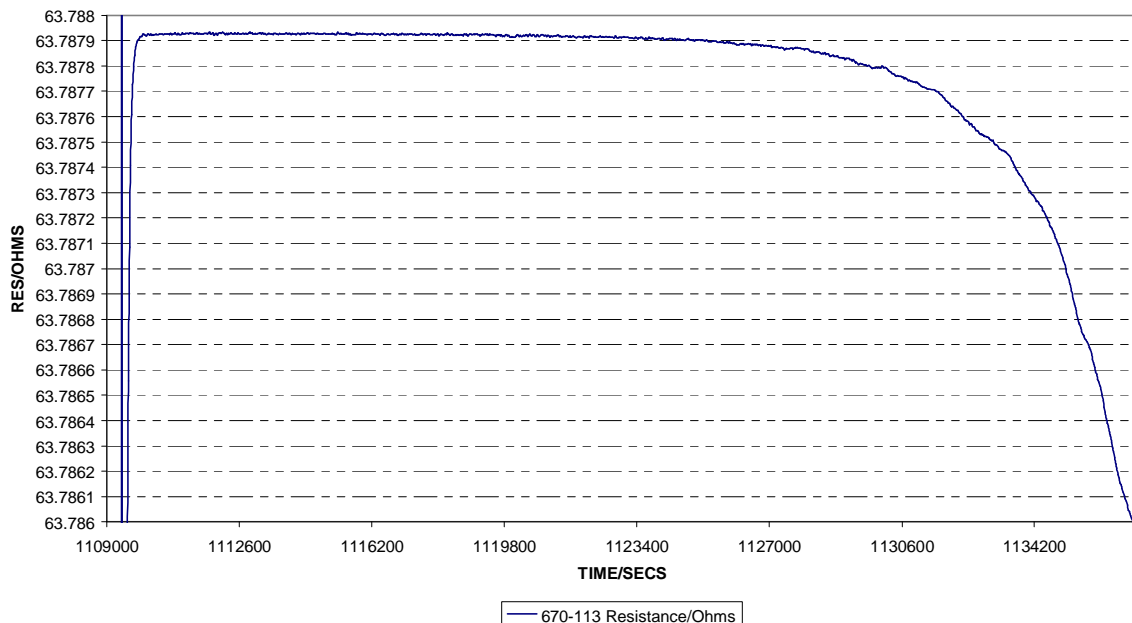
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### Isothermal Tower Zinc Fixed Point Cell MELT PLATEAU 12th October 2009 (S.P. 420.0 Deg C, Zone 13.0)



### Isothermal Tower Zinc Fixed Point Cell FREEZE PLATEAU 13th October 2009 (S.P. 419.0 Deg C, Zone 13.0)



# CERTIFICATE OF CALIBRATION

Description: Isotech Model 492 Isothermal Tower Zinc Fixed Point Cell

Identification:

UKAS Accredited Calibration Laboratory No. 0175

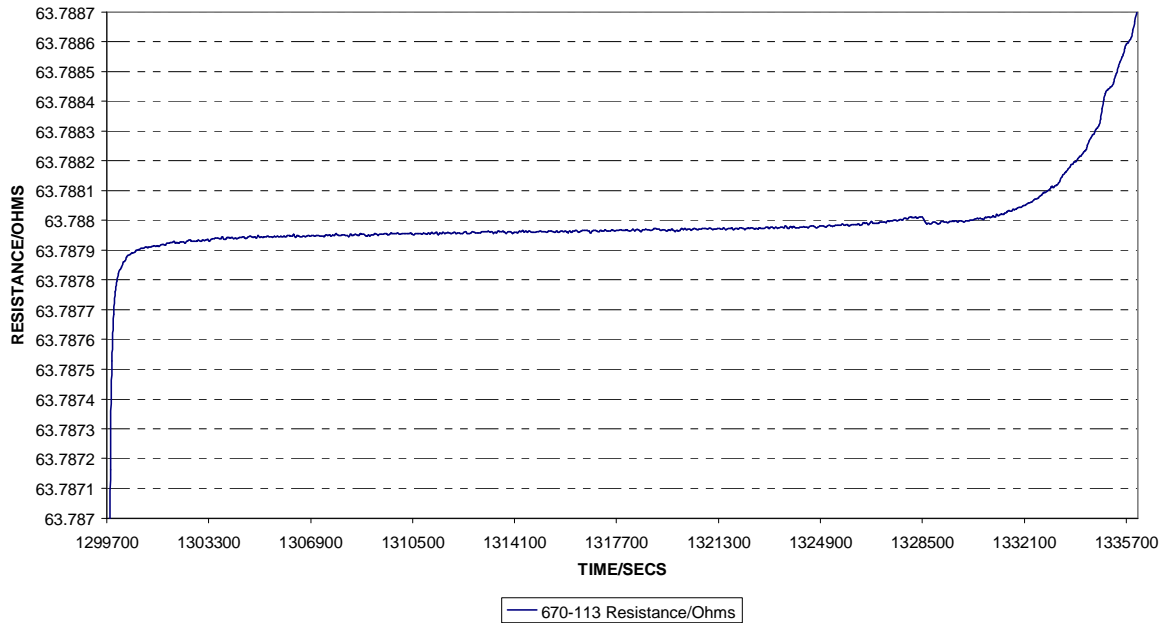
N.T.P.L. Northern Temperature Primary Laboratory, Southport, England

Tel. +44 (0) 1704 543830 / 544611 Fax. +44 (0) 1704 544799

CERTIFICATE NO:

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## Isothermal Tower Zinc Fixed Point Cell MELT PLATEAU 2 15th October 2009 (S.P. 420.0 Deg C, Zone 13.0)



## Isothermal Tower Zinc Fixed Point Cell FREEZE PLATEAU 2 16th October 2009 (S.P. 419.0 Deg C, Zone 13.0)

