CCT-K10

PROTOCOL FOR CCT COMPARISON OF ITS-90 REALISATIONS ABOVE THE SILVER POINT USING TWO TRANSFER RADIATION THERMOMETERS AND A SET OF HIGH TEMPERATURE FIXED-POINT BLACKBODY CELLS

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PROTOCOL FOR HIGH TEMPERATURE KEY COMPARISON

1 INTRODUCTION

There is a strong requirement for a CCT Key Comparison of the ITS-90 above the silver point (961.78 °C). Over this temperature range the ITS-90 is realised using a characterised radiation thermometer calibrated using a blackbody at one of the defining fixed points (i.e. Ag, Au (1064.18 °C) or Cu (1084.62 °C)) and extrapolated upwards from the reference fixed point using Planck's law in ratio form. The definition can result in undetected scale realisation uncertainties which generally increase as T^2. In addition there are various ways in which the scale can be realised leading to different sources of uncertainty, dependent upon the method used. It is essential to substantiate claimed Calibration and Measurement Capabilities (CMCs) for the ITS-90 above the silver point by undertaking a key comparison led from CCT-WG5 involving leading NMIs in different regions.

The results of the previous CCT-led key comparison (CCT K5) for the range up to 1700 °C, which took place around 15 years ago, were unsatisfactory for the following reasons:

1. the comparison was carried out using tungsten ribbon lamps which inherently have a relatively large associated uncertainty and also the radiance temperature of the ribbon is strongly dependent on the wavelength;
2. the comparison was only up to a temperature of 1700 °C whereas NMIs can routinely realise the ITS-90 up to much higher temperatures, up to 3000 °C;
3. there was evidence of some artefact instability in some of the results obtained;
4. the comparison results showed differences which were thought not to be representative of participants' actual ability to realise the high temperature scale.

As a consequence CCT K5 cannot satisfactorily substantiate NMIs' claimed CMCs, either to the level of uncertainty or over the required temperature range. This new CCT Key Comparison (KC) will address this issue, and will support CMCs for ITS-90 calibration of radiation thermometers and future CMCs for fixed point calibration above the Ag point.

The proposed KC will consist of two parts:

1. a comparison of ITS-90 scale realisations over the range from the Ag point to 3000 °C (or the highest temperature participants realise) using two transfer radiation thermometers (an IKE Linear Pyrometer LP3 and a Chino radiation thermometer IR-RST65). The transfer thermometers will be supplied along with a transportable copper fixed-point blackbody source which will be used to confirm the stability of the radiation thermometers throughout the comparison;
2. Three high temperature fixed-point (HTFP) blackbody cells (Ru-C and WC-C along with doped Ni-C (Ni-C-X)) will also be measured to probe scale realisation uncertainties at these particular temperatures with better precision.

The instruments and artefacts will be circulated in the form of a semi-collapsed star or 'flower' as described below. This will enable the performance of the instruments to be regularly checked throughout the comparison by the pilot laboratory and should help to minimise issues due to incorrect operation or drift.

2 PARTICIPANTS

The participants are drawn from a number of regions, namely EURAMET, SIM, COOMET and APMP. The details of NMI and contact person are given in Table 1 below.

Region	NMI	Details of contact person
EURAMET	NPL (pilot)	Helen McEvoy/Graham Machin National Physical Laboratory Hampton Road, Teddington, Middlesex, United Kingdom TW11 0LW E-mail: helen.mcevoy@npl.co.uk
EURAMET	CEM	Maria José Martín Centro Español de Metrologia Temperature Division C/ del Alfar 2. 28760 Tres Cantos. Madrid. Spain E-mail: mjmartinh@cem.minetur.es
EURAMET	PTB	Klaus Anhalt and Jörg Hollandt Physikalisch-Technische Bundesanstalt FB 7.3 Detektorradiometrie und Strahlungsthermometrie Abbestraße 2-12 10587 Berlin E-mail: klaus.anhalt@ptb.de/ joerg.hollandt@ptb.de
EURAMET	LNE-Cnam	Mohamed Sadli Lab. Commun de Métrologie 61, rue du Landy 93210 Saint-Denis, La Plaine France E-mail: mohamed.sadli@cnam.fr

Table 1: Participants and contact details for the CCT high temperature KC

APMP	NMIJ	Yoshiro Yamada National Metrology Institute of Japan AIST Tsukuba Central 3-1 1-1-1 Tsukuba Ibaraki 305-8563 Japan E-mail: y.yamada@aist.go.jp
APMP	NIM	Lu Xiaofeng National Institute of Metrology 18 Bei San Huan Dong Lu Beijing, 100013 China E-mail: luxf@nim.ac.cn
APMP	KRISS	YongShim Yoo Korea Research Institute of Standards and Science 1 Doryong-dong Yuseong-gu Daejeon 305-340 Korea E-mail: ysyoo@kriss.re.kr
SIM	NIST	Howard Yoon National Institute of Standards and Technology 100 Bureau Dr. Stop 1070 Gaithersburg MD 20899-1070 United States E-mail: howard.yoon@nist.gov
SIM	NRC	Andrew Todd National Research Council of Canada M-36, room 1209 1200 Montreal Road Building M-58 Ottawa, Ontario K1A 0R6 Canada E-mail: andrew.todd@nrc-cnrc.gc.ca
COOMET	VNIIM	Mikhail Matveyev D. I. Mendeleyev Institute for Metrology (VNIIM) Moskovsky pr.19 St.Petersburg 190005 Russia E-mail: M.S.Matveyev@vniim.ru

Table 1: Participants and contact details for the CCT high temperature KC - continued

3 COMPARISON SCHEME

The comparison will be in the form of a 'semi-collapsed star' or 'flower'. In other words, the comparison artefacts will be sent from the pilot laboratory to one of the regional NMIs, and measurements will then run in a loop around that region before the artefacts are sent back to the pilot laboratory. Following checks on the artefacts, the pilot will then send them to an NMI in the next region for a further loop. This will continue for each of the regions.

The measurements are due to be completed by the autumn of 2016 with the final project report to be completed by the end of Dec 2016. Refer to the Table in Appendix 1 for the full details of the circulation).

4 CIRCULATING INSTRUMENTS

The circulating instruments will be:

An IKE LP3 radiation thermometer supplied by PTB;
A Chino radiation thermometer IR-RST65 supplied by NMIJ;
A Chino copper fixed-point source (for checking the stability of the radiation thermometers prior to the measurements at each NMI) supplied by NRC.

Additionally HTFP cells of doped Ni-C (Ni-C-X), Ru-C and WC-C will be circulated to allow participants to probe the uncertainty of their primary ITS-90 scale realisation at the fixed point temperatures. One of each type of cell will be circulated.

The technical specifications of each of the instruments are given in the following tables. Operational guidelines for the use of each instrument are given in Appendix 2. Additionally, each instrument is supplied with an operating manual giving further details. Participants should familiarise themselves with the operating manuals for each instrument before starting measurements to minimise the risk of accidental damage or incorrect operation.

Chino radiation thermometer	
Model	IR-RST65
Serial number	IS143A001
Spectral range	650 nm, FWHM 12 nm
Measuring temperature range	960 °C to 3000 °C
Distance Ratio (Distance / Target size)	650 (400 mm/ 0.6 mm Ø)
Output	Radiance signal: 0 to 10 V Internal temperature stabilization monitor signal: 0 to 5 V (0 to 50 °C)
Measurement distance	400 mm to ∞
Warm up time	Half a day
Power requirements	24 VDC, DC power supply (for 100-240 VAC) accompanies the instrument

Table 2: Specifications for the Chino radiation thermometer

Working distance: Note: for the calibration the thermometer should be set at a **working distance of 700 mm**. The distance should be measured from **the front of the thermometer casing** to the defining point of the source (e.g. the defining aperture).

IKE Linear Pyrometer	
Model	LP3
Serial number	80-13
Spectral range	650 nm (and 950 nm†)
Measuring temperature range (at 0.65 μm)	800 °C to 3030 °C Neutral density filter (filter A2) to be used above 2400 °C
Measuring temperature range (at 0.95 μm)	600 °C to 2500 °C Neutral density filter (filter A2) to be used above 2000 °C
Measurement ranges	R1 for photocurrents from 100 pA to 8 nA; R2 for photocurrents from 500 pA to 800 nA
Target size versus distance	1 mm at 700 mm; 1.7 mm at 1000 mm
Output	Photocurrent and temperature* via serial RS232 interface and PC using supplied LP3DE.exe software‡
Measurement distance	730 mm to 1000 mm
Warm up time	2 hours (until cell is stabilised at 29.5 °C)
Power requirements	110 V or 220 V, 50/60 Hz, 2 x supplied power cables (one for 110 V, one for 220 V) and plug in accordance with CEE 7/7

Table 3: Specifications for the LP3 radiation thermometer

† For the comparison only the 650 nm filter should be used (filter B1)
* For the comparison only the photocurrent reading should be used
‡ Participants are not required to use the supplied software. Alternative (participant's own) software can be used to record the LP3 photocurrent provided that the software doesn't apply any data manipulation to the raw photocurrent values.

Working distance: Note: for the calibration the thermometer should be set at a **working distance of 750 mm**. The distance should be measured from the **front of the thermometer casing** to the defining point of the source (e.g. defining aperture).

Transportable copper fixed-point blackbody source	
Furnace model	IR-R0A
Serial number	RA12YB002
Fixed-point blackbody	Cu point
Blackbody cavity	8 mm Ø× ~53 mm, with 120 °conical end and 6 mm Ø aperture
Effective emissivity	~0.9998
Plateau duration	Approximately 10 min
Heat-up time	Approx. 120 min from room temperature to Cu point
Cool-down time	Approx. 120 min from Cu point to 150 °C
Power	110-120/ 220-240VAC, 50 – 60 Hz, 750 VA max
Gas requirements	Pure argon, 0.7 l/min
Temperature control	PID control by a pre-programmed controller

Table 4: Specifications for the transfer Cu point source

High temperature fixed point cells						
HTFP cell	Identification number	Supplied by	Length/ mm	Outer diameter/ mm	Aperture diameter/ mm	Emissivity
Ru-C cell	6Ru1	LNE-Cnam	44	24	3	0.9997
Ru-C cell	6Ru2	LNE-Cnam	44	24	3	0.9997
Ru-C cell	Ru-C-4	Tubitak	24	24	3	0.9990
Ni-C cell	NiC #11	INMETRO	40	24	3	0.9997
Ni-C cell	NiC #12	INMETRO	40	24	3	0.9997
WC-C cell	W2 6SSC-2	NIM	45	24	3	0.9997
WC-C cell	WC-C#1	NPL	44	24	3	0.9996
WC-C cell	WC-C#2	NPL	44	24	3	0.9996

Table 5: Dimensions and emissivity values for the high temperature fixed-point cells

The two/ three cells of each of the fixed point types will be cross-compared prior to the start of the comparison to determine the temperature difference between the cells. One of each type of cell (i.e. one Ni-C cell, one Ru-C cell and one WC-C cell) will be chosen at random for circulation among the participants. The remaining cell(s) of each type will be kept at NPL in case of breakage of any transfer cell during the comparison.

5 SHIPPING

The NMI performing the measurements should arrange for the thermometers and cells to be transferred to the next NMI by the date given in the schedule in Appendix 1. Wherever possible, door-to-door transportation should be used to minimise the risk of damage to the instruments. The NMI performing the measurements is required to arrange the safe transportation of the instruments to the next NMI and for paying the transport costs. Therefore it is strongly recommended that the NMI arranging the shipping takes out insurance in case of loss or damage of the instruments and cells during the transportation. The value of the goods, for insurance purposes, should be taken to be in the region of £115000. The NMI receiving the instruments should carry out the checks described in Section 9.1 and confirm to the pilot laboratory by e-mail that the instruments have arrived safely.

NPL will provide all necessary documentation including customs documentation for shipping the instruments from NPL to the particular region and then back to NPL. For customs purposes, during the comparison the instruments will be considered to be a temporary export out of the UK. It is important that all the items within the shipment are kept together and transported as one consignment with all the accompanying paperwork.

The approximate dimensions and weights of each of the items are given in Table 6.

Item	Packaging	Dimensions*/ mm	Gross weight*/ kg	Number of units
LP3 radiation thermometer	Aluminium box placed inside wooden box on EUR pallet	700 x 800 x 1200	50	1
Chino radiation thermometer	Duralumin carrying case in corrugated cardboard box reinforced on the inside with ply wood boards	520 × 460 × 330	12.3	1
Transfer Cu fixed point	Pelican™ 500 transport case	1015 x 600 x 730	40	1
Ni-C cell	Carrying case/ box	300 x 300 x 300	3	1
Ru-C cell				1
WC-C cell				1

Table 6: Approximate dimensions and weights of items in consignment

6 PROBLEMS

During the measurement process participants should endeavour to make sure that all the instruments are safely and correctly handled while they are at their laboratory. If desired, insurance may be taken out to cover against loss or damage. The NMI arranging shipping to the next participant will be responsible should anything go wrong during the shipping process resulting in any damage to any of the instruments. It is therefore strongly recommended that insurance is taken out in case of loss or damage of the instruments or the cells during transportation.

Should the results of the copper fixed point check (Section 9.1.5) indicate that there is a problem with either of the radiation thermometers then it might be necessary to return that thermometer to the pilot laboratory so that a further assessment can be carried out.

Should any problems arise with the operation of the instruments, both the coordinator and the NMI supplying the equipment should be contacted.

7 PREPARATION FOR MEASUREMENTS

Prior to receiving the instruments each participant should prepare for the measurements. Please ensure sufficient time is given for this activity so the measurement schedule is not delayed.

Preparation for measurements includes:

- Ensuring that the radiation thermometer used for primary ITS-90 scale realisation is fully characterised/ calibrated according to the local methodology (i.e. in terms of spectral response, linearity, size-of-source effect, range ratios, ITS-90 fixed point reference calibration etc.);
- The high temperature furnace to be used for the calibration of the radiation thermometers is operational;
- The furnace to be used for the HTFP measurements has been prepared for these measurements, including: baking out to minimise the risk of cell contamination; determination of the optimum cell position within the furnace; ensuring furnace insulation, cell holders are available etc.. Further guidance and details of this process are given in

Appendix 3. This guidance has been adapted from the protocol for the InK Workpackage 1 HTFP measurement campaign.

8 MEASUREMENTS AT THE PILOT LABORATORY (NPL)

The pilot laboratory will fully calibrate and characterise the two radiation thermometers prior to the start of the comparison and at the end of the comparison, and will also validate the transfer Cu fixed point and the HTFP cells. The calibration/ validation will consist of:

- Size-of-source effect measurements;
- Gain or range ratio measurements as appropriate;
- Non-linearity;
- Spectral response of the LP3 (the spectral response of the Chino thermometer will be carried out by NMIJ prior to sending the thermometer to NPL);
- Validation of the transfer Cu fixed point source using the NPL primary Cu point and radiation thermometer (the NPL IKE LP3);
- Measurement of the KC radiation thermometer outputs using the transfer Cu point (to provide a baseline reference signal);
- Calibration of the KC radiation thermometers at the comparison temperatures, namely 960, 1100, 1300, 1500, 1700, 1800, 2000, 2200, 2400, 2500, 2600, 2800, 2900, 3000 °C, using the NPL high temperature blackbody source and primary reference thermometer (the NPL LP3);
- Assignment of the NPL ITS-90 temperatures to a set of HTFP cells, consisting of one of each of Ni-C-X, Ru-C and WC-C, using the usual NPL method for determining the point of inflection (poi) temperature of the melt of the HTFPs using the NPL LP3. This set of cells will be circulated among participants. The remaining cells will be held at NPL, to serve as a reference to test for any possible drift of the HTFPs during the comparison and to provide a spare in case of breakage during the comparison. (Note that the temperature difference between the cells of each type will already have been measured prior to the start of the comparison.)

When the instruments are returned to the pilot during the comparison a limited number of the above measurements will be carried out in order to check the correct operation and stability of the instruments. These measurements will be:

- check of the operation of the transfer Cu fixed point using the NPL LP3 and the NPL primary Cu point;
- check of the output of each of the two KC thermometers using the melt/ freeze transition of the transfer Cu fixed point source;
- size-of-source effect of the two transfer thermometers;
- re-calibration of the two KC radiation thermometers at the temperature points listed above, including check of gain and range ratios and LP3 ND filter transmission;
- measurement of the circulating HTFP cells, including a cross-comparison of the each of the transfer cells with the appropriate reference cell(s) being retained at NPL to check the stability of the circulating HTFPs.

9 MEASUREMENTS AT THE PARTICIPATING NMIS

9.1 CHECK ON ARRIVAL AT EACH NMI

A visual inspection of the packaging and all the instruments and cells should be made on arrival of the equipment at each NMI. Any evidence of damage should be reported immediately both to the pilot and the NMI responsible for the shipment using the form in Appendix 4.

In the case of the HTFP cells they should be carefully inspected both before and following transportation for any obvious damage and for any sign of metal diffusing through the cell walls. Both on receipt of the cells and before shipping them, each participant should report the appearance of the cells, if appropriate with a photograph, by sending an email to the pilot and the previous participant (on receipt) or next participant (before posting).

As a further check of the KC transfer thermometers, to make sure that they have not been damaged during transport, within one week of their receipt the output of each radiation thermometer should be measured using two melt/ freeze cycles of the supplied Cu fixed-point blackbody source (refer to Section 9.1.5) and the result reported to the pilot.

9.1.1 Stabilisation/ warm up

The stabilisation (warm up) times for the thermometers are given in Tables 2 and 3 and must be adhered to. The thermometers should subsequently be left switched on, if possible, for the duration of all the measurements.

9.1.2 Lens cleaning

Superficial dust should be blown off the front lens of the thermometer using clean air or other means but otherwise the lens should NOT be cleaned. The protective lens cap should be placed on the front of the thermometer between measurements and great care should be taken not to touch the front lens. The LP3 is provided with an objective extension ring as a further protection of the front lens. This should not be removed.

9.1.3 Positioning

The thermometers should be set up and aligned at the prescribed working distance, namely 700 mm (Chino thermometer) or 750 mm (LP3) from the front of the thermometer casing to the source, according to the local procedure, with reference to any specific instructions supplied with the thermometers.

9.1.4 Background (dark reading) measurements

Background measurements should be carried out by placing the protective lens cap on the front of the thermometer.

9.1.5 Check using transfer Cu fixed point source

Within one week of their receipt the output of each transfer radiation thermometer should be measured using two melt/ freeze cycles of the supplied Cu fixed-point blackbody source, using the supplied instructions for using the source (refer to Appendix 2 and the instruction manual supplied with source).

The fixed point aperture is located at a distance of 155 mm behind the front panel of the furnace. The measurements should be made at the prescribed working distance (i.e. 700 mm for the Chino thermometer and 750 mm for the LP3, measured from the front of the thermometer casing to the fixed point aperture), with the thermometer focused and aligned on the centre of the Cu fixed point aperture. This can be achieved by focusing and aligning on the centre of the white alumina aperture, which is located 4 mm in front of the fixed point aperture, and then linearly moving the thermometer towards the furnace by 4 mm.

The measurements of the Cu point with the Chino thermometer should be made with the thermometer set on gain L; the measurements for the LP3 should be made with the thermometer set on Range R1.

Measurements over the central half of the Cu freezing plateau will be used for the check. The results of the check (i.e. average output signals of the thermometers during the central half of the two Cu freezing plateaux, corrected for dark reading (background)), along with the internal temperature of the thermometer and the laboratory ambient conditions, should be e-mailed to the pilot laboratory using the template form in Appendix 5, within one week of receipt of the thermometers.

9.2 COMPARISON MEASUREMENTS

This is a Key Comparison of ITS-90 temperature scales.

Each NMI will:

i. calibrate each transfer radiation thermometer using their realisation of the ITS-90 scale (i.e. usually via a high temperature blackbody source and a reference thermometer);
ii. determine the ITS-90 temperature of the point of inflection (poi) of the melt of the supplied HTFPs using their own primary radiation thermometer used for high temperature ITS-90 scale realisation.

9.2.1 Calibration of the transfer thermometers

The calibration of the transfer (KC) thermometers should be carried out according to the local procedure for the calibration of such devices using the participant's high temperature blackbody (HTBB) source, the temperature of which should be assigned according to the usual method at the NMI[†]. The calibration should be carried out with the thermometers set at the prescribed working distance from the blackbody source, i.e. 700 mm (Chino thermometer) or 750 mm (LP3) from the front of the thermometer casing to the source.

The calibration of the KC thermometers is to be carried out at all of the following temperatures, although the maximum calibration temperature can be reduced to fit the particular capability within an NMI.

960, 1100, 1300, 1500, 1700, 1800, 2000, 2200, 2400, 2500, 2600, 2800, 2900, 3000 °C.

The calibration at all temperatures should be carried out using an HTBB. The HTBB should be set to be within 0.5 °C of the specified temperatures. The gain/ range settings for each instrument should be set appropriately for each temperature as specified in Appendix 6. For the LP3 the neutral

density (ND) filter, filter A2, needs to be used for temperatures above 2400 °C, again as specified in Appendix 6.

Participants should carry out repeat measurements at a minimum of three of the above calibration temperatures spread over the range; e.g. 960 °C, 1800 °C and 2800 °C.

The reference source size for the comparison is 25 mm diameter.

([†]Note: as an alternative, participants can instead realise the ITS-90 directly using the transfer radiation thermometers, by fully characterising them in terms of the spectral response, linearity etc. and performing an ITS-90 reference fixed point measurement. In this case measurements using a high temperature blackbody source are not carried out. Instead the thermometer signal output for each of the calibration temperatures will be derived by calculation.)

Participants should measure the size-of-source effect (SSE) of the two transfer thermometers so that the calibration results can be corrected to a source size of 25 mm, if the size of the HTBB used in the NMI is different from 25 mm. The measurement of the SSE will also allow the NMI to calculate any uncertainty to take into account the thermal uniformity of their HTBB (see Section 9.2.3). Alternatively, if an NMI does not wish to measure the SSE, the pilot laboratory will provide the SSE results measured by the pilot at the start of the comparison for the NMI to calculate its own correction. (An additional uncertainty component might subsequently need to be included for that NMI if it is found that the SSE of the transfer thermometers changes during the comparison, for example as a result of contamination of the front lens).

Participants should also measure the transmission of the LP3 neutral density (ND) filter using a stable blackbody source at an appropriate temperature so that any changes in transmission with time can be taken into account in the results analysis.

Additionally participants should measure the range/ gain ratios of the transfer thermometers using a stable blackbody source maintained at a temperature appropriate for the range/ gain ratio being measured. Again this is so that any changes in the ratios with time can be monitored and can be taken into account in the results analysis if appropriate.

Any additional supplementary measurements should also be carried out if these form part of the usual necessary calibration procedure for a high temperature radiation thermometer (for example if they are required to fully assess the calibration uncertainties).

The report giving measurement method, details of equipment used, calibration results for each thermometer and estimated calibration uncertainties for each thermometer should be sent to the pilot within one month of completion of the measurements. For further details of the reporting format refer to Section 10.

9.2.2 Measurement of the HTFPs

The measurements of the HTFPs should be carried out according to the local procedures using the radiation thermometer usually used for primary ITS-90 scale realisation at the participant's NMI. Participants are required to measure the ITS-90 radiance temperature of the point of inflection (poi) of the melt of each cell (see for example [1]) using the usual method at their NMI.

The measurements of the HTFPs should be carried out at a wavelength of 650 nm. Where this is not possible (the radiation thermometer operates at a different wavelength for example) measurements may be carried out at a different wavelength which should be clearly reported.

The reference source size for the HTFP measurements is 3 mm diameter.

It is recommended that measurements be performed in the sequence WC-C, Ru-C, Ni-C-X. As a minimum four melt/ freeze cycles should be carried out for each cell, over one day. It is recommended that a second set of four melt/ freeze cycles is carried out on a subsequent day, with the cell removed and replaced within the furnace in-between the two days, in order to assess the reproducibility of the measurements due to (re-)positioning of the cell within the furnace. The first melt and freeze cycle of each day is **not** to be included in the analysis [i.e. only the second, third and fourth cycles are analysed] and so can be used for checking the alignment of the radiation thermometer on the cell aperture, temperature profile checks etc..

The melt and freeze transitions should be initiated using step sizes of, respectively, +20 K and -20 K from the melt temperature. (Note that these are approximate temperature steps for guidance only. If it is not possible to set the furnace to exactly these steps then the step size can be adapted.)

If the first cycle (the one not analysed) takes less than 30 minutes, then the furnace should be held at the below-melt temperature (the end point of the first cycle) for additional time to ensure that at least 30 minutes have passed between *the start of* the first cycle and *the start of* the second cycle (i.e. if the first cycle takes 20 minutes, this temperature should be held for 10 minutes before the start of the second cycle). Subsequent cycles can be closer together. However, it is necessary to wait until the cells have reached the offset temperatures (+20 K) following the melt to ensure that they are fully molten before starting the freeze (but see comment below about WC-C[†]), and then that they reach the offset temperature (-20K) following the freeze to ensure that they are fully frozen before attempting the next melt.

[†]Note: WC-C cells can experience a very deep undercool and can become difficult to freeze. To avoid problems with WC-C, the cell should be frozen soon after it completes the melt. It is important to ensure the melt is completed, but also to ensure that it does not remain molten for long.

If a cell is damaged while measurements are being carried out, then the participant should contact the pilot laboratory immediately with a description of the damage or poor performance. Damage includes, but is not limited to:

- Structural damage following accident (e.g. dropping cell)
- Structural damage following a heating/cooling cycle (e.g. cracking)
- Excess metal diffusing through cell wall as a result of cell ageing

9.2.3 Supplementary information

i) Furnace lateral uniformity for SSE correction

The lateral uniformity of the furnace should be measured by scanning horizontally and, if feasible, vertically across the furnace aperture. This scan should be performed for both the high temperature furnace used for the calibration of the KC radiation thermometers and the furnace used for the HTFP cell measurements. This will enable corrections and/ or uncertainty contributions to be estimated taking into consideration the different thermal profiles of each of the furnaces used by the participants and the size-of-source effect of the radiation thermometers, and enable the results to be

corrected, if applicable, to the appropriate reference source diameter (25 mm for the KC radiation thermometer calibration and 3 mm for the HTFP measurements; see Sections 9.2.1 and 9.2.2 above). Participants can choose whether to measure the uniformity at one temperature or all temperatures according to their usual local procedure.

ii) Furnace thermal inertia

Participants are requested to measure the thermal inertia of their furnace, as described in Appendix 3, Section 4 , in case a correction and/ or uncertainty needs to be applied to the HTFP results to allow for any effect of differing thermal inertias on the poi of the melt.

iii) Uncertainty components due to furnace performance

For the measurements of the HTFPs an uncertainty component will be included by the pilot for each participant's results to account for emissivity, temperature drop and other furnace effects. For this, the participant must provide a detailed description of the furnace geometry (diameters and positioning of baffles, furnace wall etc.). Uncertainty estimates will be taken from [2].

iv) Uncertainty component due to the emissivity of the cells and the operating wavelength of the radiation thermometer

For those HTFP measurements which were carried out at the specified wavelength of 650 nm, no correction to the participant's results is necessary to take into account the emissivity of the cell, and therefore there is also no additional uncertainty due to a correction. For those participants who carry out the HTFP measurements at a wavelength other than 650 nm the pilot laboratory will apply a correction to the results to take into account the difference between the participant's wavelength and the specified wavelength and the calculated estimated emissivity of the cell. An additional component to allow for the uncertainty in the correction due to the uncertainty in the estimated emissivity of the HTFP cell will be included in the overall uncertainty budget for that participant.

10 REPORTING

The following information should be provided to the pilot laboratory within one month of completion of the measurements.

10.1 GENERAL INFORMATION

This should take the form of a Word document and should contain a description of the equipment and the measurement method used at the NMI, including:

- make, model, serial number and geometry of the high temperature blackbody source used for the calibration of the KC thermometers (if applicable[†]);
- details about the reference thermometer used if applicable[†] (e.g. type (radiation thermometer or thermocouple), make, model, serial number, spot (target) size, operational wavelength(s) etc.);
- methodology used in the calibration of the transfer KC thermometers (procedure followed; working distance from thermometer to blackbody if different from that prescribed; additional checks or measurements performed etc.);
- make, model, serial number and geometry of the high temperature furnace used for the measurements of the HTFP cells, if different from above;
- steps taken to prepare the furnace for the HTFP measurements;

- details of the primary reference radiation thermometer used for the HTFP measurements (make, model, serial number, spot (target) size, operational wavelength(s) etc.) if different from above;
- methodology used in the measurement of the HTFP cells (procedure followed, additional checks or measurements performed, including any additional melt/ freeze cycles carried out for e.g. optimising the position of the cell in the furnace etc.);

(† In the case where the transfer (KC) radiation thermometers are fully characterised so that they can be used for direct realisation of the ITS-90 then no measurements of a high temperature blackbody will be carried out and this information is not applicable.)

10.2 RESULTS

The results should be provided in the form of an Excel spreadsheet and should include the following information. Templates for reporting the results and uncertainties are given in Appendices 6 and 7.

10.2.1 For the calibration of the KC radiation thermometers:

- Results of the stability check using the transfer Cu fixed-point;

and, for each calibration point†:

- The ITS-90 temperature of the blackbody source;
- Thermometer output/ reading, after correction for the dark reading (background), measured with the appropriate gain or range, and ND filter setting for the LP3, as specified in Appendix 6;
- The internal temperature of the thermometer;
- The laboratory ambient temperature;

The following additional information must be given:

- The size-of-source effect of the KC radiation thermometers (if measured);
- The transmission of the LP3 ND filter;
- The results of the range/ gain ratio measurements for the comparison thermometers;
- Uncertainty budget identifying all components of uncertainty and their values, and the total expanded uncertainty. The budget should take the form of the template supplied in Appendix 7 and should include:
  - Uncertainty in the ITS-90 scale realisation, with all components identified;
  - Uncertainty due to HTBB window transmittance (if applicable);
  - Radiance temperature uniformity across the blackbody aperture;
  - Temperature stability of the blackbody source;
  - Effect of emissivity of the blackbody source (e.g. due to difference in wavelength between the participant reference thermometer and the wavelength of operation of the KC radiation thermometer);
  - Uncertainty due to alignment on the blackbody source;
  - Repeatability / reproducibility of the calibration measurements;
  - SSE of the KC thermometer (if measured);

- Correction of the results to the reference source size of 25 mm diameter;
- Resolution of the KC thermometer;
- Any additional uncertainty components not listed above.

†In the case where the transfer thermometers are fully characterised for direct ITS-90 realisation then the following should instead be reported:

- Thermometer output/ reading after correction for the dark reading (background) for each of the comparison temperatures at the appropriate gain or range, and ND filter setting for the LP3, as specified in Appendix 6;
- The gain or range setting of the thermometer;
- The size-of-source effect of the KC radiation thermometers (if measured);
- The transmission of the LP3 ND filter;
- The results of the range/ gain ratio measurements;
- Uncertainty budget identifying all components of uncertainty and their values, and the total expanded uncertainty. The budget should take the form of the template supplied in Appendix 7 and should include
  - Uncertainty in the ITS-90 scale realisation, with all components identified;
  - SSE of the KC thermometer (if measured);
  - Resolution of the KC thermometer;
  - Any additional uncertainty components not listed above.

10.2.2 For the HTFP measurements the following information must be supplied:

- Cell identification;
- Measured ITS-90 radiance temperature of the point of inflection of the melt transition;
- The temperature profile of the high temperature furnace (if measured);
- Uncertainty budget identifying all components of uncertainty and their values, and the total expanded uncertainty. The budget should take the form of the template supplied in Appendix 7 and should include:

  - Uncertainty in the ITS-90 scale realisation, with all components identified;
  - Uncertainty in the determination of the point of inflection;
  - Repeatability of HTFP melt transition;
  - Uncertainty due to HTBB window transmittance (if applicable);
  - Uncertainty due to alignment;
  - Uncertainty due to lateral temperature uniformity/ effective source diameter, including correction of the results to the reference diameter of 3 mm;
  - Any additional uncertainty components not listed above;

(Note that it is not necessary to include uncertainty components to account for emissivity, temperature drop and other furnace effects as this will be done by the pilot laboratory – see Section 9.2.3.)

11 ANALYSIS OF FINAL RESULTS

The pilot laboratory will analyse the overall final results taking into account the measurements made at the pilot laboratory before, during and after the comparison and the results from the individual participants.

The analysis method will be as follows.

11.1 Analysis of the results of the two KC thermometers

For each of the two transfer thermometers the result (background corrected output versus the ITS-90 temperature) at each nominal comparison temperature will first be normalised [corrected] to a common comparison temperature, t_{nom}, using the sensitivity of the thermometer using Equation (1) (e.g. suppose a participant provides results for t_{90} = 1699.9 °C then the thermometer output will be corrected to give the equivalent output at t_{nom} = 1700 °C).

$$\Delta S = \Delta T \frac{c_2}{\lambda T^2} S \qquad (1)$$

where ΔS is correction to be applied to the thermometer output signal S, ΔT is the difference between the participant measurement temperature T (= t_{90} in K) and the comparison temperature T_{nom} (= t_{nom} in K), c_2 is the second radiation constant and λ is the thermometer wavelength.

The results of the measurements using the transfer Cu fixed-point blackbody will be used to assess whether or not there has been any drift of the transfer thermometers over the course of the comparison. If any drift is evident for either of the thermometers then the thermometer output signal will be corrected to take into account the drift, using the change in output signal at the Cu point from the start of the comparison (the first set of measurements by the pilot). The results at each t_{nom} will be corrected using the Cu point drift and the sensitivity of the thermometer at that t_{nom} (i.e. the drift will be scaled according to t_{nom}). An additional component of uncertainty will be included to take into account either the thermometer drift or the correction due to the thermometer drift, depending on the magnitude of the drift.

Similarly the results of the LP3 ND filter transmission and the KC thermometer range and gain ratio measurements will be analysed and if there is any evidence of drift during the course of the comparison, a further uncertainty component might need to be included and/ or the results corrected to minimise the effect of the drift.

The proposed analysis method follows that given in Section 5 of [3].

For each transfer thermometer the results of all the pilot laboratory measurements over the entire period of the comparison will be averaged for each t_{nom}, after correction of the results to allow for drift of the thermometer if necessary, to give one pilot result for each thermometer for each t_{nom}. The final uncertainty associated with the pilot results will include the standard deviation of the pilot measurements (after correction of results for any drift).

Where a participant has more than one result for a particular t_{nom}, as a result of carrying out repeat measurements, then the results will be averaged to give one result for that t_{nom} for that participant and this will be used in the subsequent analysis.

For each transfer radiation thermometer a reference value will be calculated for each t_{nom} using the weighted mean with cut-off of all the participant results, including the average pilot results, at that $t_{nom.}$ The weight for each participant, including the pilot, is the inverse of the square of the standard measurement uncertainty for that participant at that t_{nom}. The cut-off values for the weights will be the average of the uncertainty values of those participants that reported uncertainties smaller than or equal to the median uncertainty of all the participants.

The weighted mean, *y*, will be calculated according to Equation (2):

$$y = \frac{x_1/u^2(x_1) + \cdots + x_N/u^2(x_N)}{1/u^2(x_1) + \cdots + 1/u^2(x_N)} \quad (2)$$

where x_1 through to x_N are the results from participants 1 through to *N* and $u(x_1)$ through to $u(x_N)$ are the associated standard uncertainties for participants 1 through to *N*, which will include any additional uncertainty components such as that due to the thermometer drift (if any).

If, as is likely, the participant uncertainties are expressed in terms of temperature, then the equivalent uncertainty in terms of thermometer output signal will be calculated using the thermometer sensitivity at that temperature (Equation (1)) before calculating *y* using Equation (2).

The standard deviation $u(y)$ associated with *y* will be calculated according to Equation (3):

$$\frac{1}{u^2(y)} = \frac{1}{u^2(x_1)} + \cdots + \frac{1}{u^2(x_N)} \quad (3)$$

A consistency check of the results will be carried out in the form of a chi-squared test by calculating the observed chi-squared value, χ^2_{obs}, according to Equation (4) and assigning the degrees of freedom, *v*, according to Equation (5)

$$\chi^2_{obs} = \frac{(x_1 - y)^2}{u^2(x_1)} + \cdots + \frac{(x_N - y)^2}{u^2(x_N)} \quad (4)$$

$$v = N - 1 \quad (5)$$

The consistency check will be regarded as failing if

$$Pr\{\chi^2(v) > \chi^2_{obs}\} < 0.05 \quad (6)$$

where *Pr* denotes ‘probability of’.

Provided that the consistency check does not fail then *y* will be accepted as the KCRV for that particular transfer thermometer with an associated standard uncertainty of $u(y)$. If the consistency check fails then an alternative method for calculating the KCRV, such as the median of all the participant results, will be considered following the procedure given in Section 6 of [3].

The degrees of equivalence for each participant for each thermometer and for each t_{nom} will be calculated using the difference between the result of that participant and the KCRV for that particular thermometer at that t_{nom} and the uncertainty of the difference, which will be the participant uncertainty combined with the uncertainty of the KCRV. The degrees of equivalence between each pair of participants will also be calculated in a similar way.

Finally the degrees of equivalence for each participant, both in relation to the KCRV and between each pair of participants, will be converted to be in terms of equivalent temperature differences along with the associated uncertainties, again in terms of temperature.

The final result for each participant will therefore be, for each thermometer, the difference from the KCRV for that thermometer and the other participants, along with the overall combined expanded (k = 2) uncertainty of the comparison at that temperature, which will include the participant uncertainty, the uncertainty in the KCRV value and any other associated uncertainties of the comparison, for example due to thermometer drift. All the differences and the associated uncertainties will be expressed in terms of temperature.

The use of two KCRVs, one for each transfer thermometer, will avoid potential difficulties that could arise should an average KCRV be specified but one of the transfer thermometers performs less well than the other during the comparison, for example due to drift. If appropriate, the degrees of equivalence for each participant for each transfer thermometer can subsequently either be combined by taking the average or the larger values used to provide a single degree of equivalence for future regional comparison linkage.

11.2 Analysis of the results of the HTFP cells

For each type of cell (Ni-C, Ru-C, WC-C) the results of all the pilot laboratory measurements over the entire period of the comparison will be averaged to give one pilot result for each cell type. The final uncertainty associated with the pilot results will include the standard deviation of the pilot measurements.

For each type of cell a reference value will be calculated using the weighted mean with cut-off of all the participant results (namely the ITS-90 radiance temperatures of the point of inflection of the melt transitions), including the average pilot results. The weight for each participant, including the pilot, is the inverse of the square of the standard measurement uncertainty for that participant at the cell melt temperature. The cut-off values for the weights will be as described in Section 11.1 above for the radiation thermometer measurements. The reference value for each cell will be calculated according to Equation (2). The uncertainty of the reference value will be calculated according to Equation (3). A consistency check will be carried out as per Equations (4), (5) and (6) and assuming that the check does not fail then the calculated reference value (weighted mean with cut-off) will be taken as the final reference value for that cell, along with its associated uncertainty.

It is assumed that the cells will not drift during the comparison. However, if the results of the repeat measurements at the pilot laboratory show drift (as determined from the cross-comparison measurements between the circulating cell and the reference cell held at the pilot) then a correction will need to be applied to the affected participant results with the corresponding uncertainty of the correction being included in the overall uncertainty of the reference value(s)

The degrees of equivalence for each participant for each cell will be calculated using the difference between the result of that participant and the reference value for that particular cell and the uncertainty of the difference, which will be the participant uncertainty combined with the uncertainty of the reference value. The degrees of equivalence between each pair of participants will also be calculated in a similar way.

12 REFERENCES

[1] Lowe, D and Machin, G, Evaluation of methods for characterizing the melting curves of a high temperature cobalt–carbon fixed point to define and determine its melting temperature, *Metrologia*, 2012, **49(3)**, pp 189-199

[2] Castro, P, Machin, G, Bloembergen, P, Lowe, D, Thermodynamic Temperatures of High Temperature Fixed Points: Uncertainties due to Temperature Drop and Emissivity, to be published in the *International Journal of Thermophysics*

[3] Cox, M G, The evaluation of key comparison data, *Metrologia*, 2002, **39**, pp 580 – 595.

[4] Fischer, J et al, Uncertainty budgets for realisation of scales by radiation thermometry, CCT-WG5 document, CCT/03-03, 2003

APPENDIX 1 – CIRCULATION TIMETABLE

NMI	Measurement period	Instruments to be received at next lab by
NPL (pilot)	to end Sept 14	mid-Oct 2014
NMIJ	mid-Oct 14 to end Nov 14	mid-Dec 2014*
NIM	mid-Dec 14 to mid-Feb 15	end Feb 2015*
KRISS	Mar 15 to mid-Apr 15	end Apr 2015*
NPL (pilot)	May 15	mid-June 2015
NIST	mid-June 15 to mid-July 15	end July 2015
NRC	Aug 15	mid-Sept 2015
NPL (pilot)	mid-Sept to mid-Oct 15	end Oct 2015
VNIIM	Nov 15 to mid-Dec 15	end Dec 2015
NPL (pilot)	Jan 16	mid-Feb 2016
LNE-Cnam	mid-Feb 16 to mid-Mar 16	end Mar 2016
PTB	Apr 16	mid-May 2016
CEM	mid-May to mid-June	end June 2016
NPL (pilot)	July 16 to Aug 16	-

Table 7: The timetable of the comparison

* If possible the consignment should be sent to NIM earlier, and NMIs immediately following should anticipate some variation (slippage) in the schedule dates to try and accommodate potential delays with getting the goods through Chinese customs.

APPENDIX 2 – OPERATIONAL GUIDELINES FOR THE TRANSFER INSTRUMENTS

A summary of the steps necessary for the correct setting-up, use and re-packing for each of the two transfer radiation thermometers and the transfer copper fixed point is given below. Note that this includes key points only. For full operating instructions please refer to the instruction manuals supplied with the instruments. Additionally videos, prepared by NMIJ, for the Chino radiation thermometer and the Chino transfer copper fixed point, are available on-line; see

https://skydrive.live.com/redir.aspx?cid=0e97d241fa436fea&page=self&resid=E97D241FA436FEA!105&parid=E97D241FA436FEA!103&authkey=!Au5ltrffW4NfXmA&Bpub=SDX.SkyDrive&Bsrc=Share

Note however that the assembly/ disassembly instructions for the NRC Cu fixed point are different to those described in the video – see Section 3.1 in this Appendix.

1. User instructions for the LP3

1.1 On receipt:

1. Wait for instrument to acclimatise to room temperature before starting to unpack;
2. Check that all items and accessories are present. Note how the items are packed into the crate so that they can be repacked in the same manner;
3. Insert the eyepiece into the rear of the thermometer, removing the plastic protection cover on the eyepiece holder first, and lock into position;
4. Remove the locking screw from just under the objective tube on the front of the casing and replace with the smaller screw supplied to block to hole;
5. Unlock the objective lens adjustment control on the side of the casing and adjust the position of the lens so that a target at 750 mm from the front of the thermometer casing appears sharply in focus. Lock the lens tube in position;
6. Plug the power supply unit appropriate to the laboratory mains voltage (230V or 110 V) into the back of the thermometer and plug the power supply unit into the mains;
7. Turn the power on using the switch on the power supply unit. Allow the thermometer to warm up for at least 2 hours (until the cell is stabilised at 29.5 °C) before starting measurements.

1.2 Shipment to the next participant:

1. Replace the lens cap on the thermometer objective;
2. Unplug, switch off and disconnect the power cable;
3. Unlock the objective lens tube using the locking screw at the side of the thermometer case and slide it completely back towards the front plate, then lock in position;
4. Remove the small screw from just under the objective lens tube on the front of the case and replace with the larger locking screw to keep the objective lens firmly in place during shipping;
5. Unlock and remove the eyepiece and replace the plastic protection cover onto the eyepiece holder;
6. Pack everything carefully into the packing crate in the same manner in which the instrument arrived.

2. User instructions for the Chino radiation thermometer

2.1 On receipt and setting up for use:

1. Wait for instrument to acclimatise to room temperature before starting to unpack;
2. Check that all items and accessories are present. Note how the items are packed into the crate so that they can be repacked in the same manner;
3. Connect the signal cable to the back of the thermometer. Connect the output terminals to two digital voltmeters, one for reading the thermometer radiance signal output, and the other for monitoring the instrument internal temperature;
4. Connect an AC power cable to the DC power supply unit of the thermometer then connect the DC power supply cable to the back of the thermometer;
5. Plug the power supply into the mains. The LED on the rear of the thermometer will light up red, and will change to green when the thermometer has warmed up;
6. Select the appropriate gain using the knob at the back of the instrument.
7. Allow the thermometer to warm up for at least half a day before starting measurements.

2.2 Shipment to the next participant:

1. Replace the lens cap on the thermometer objective;
2. Unplug and disconnect the power cable from the back of the thermometer;
3. Disconnect the signal cable from the back of the thermometer;
4. Pack everything carefully into the packing crate in the same manner in which the instrument arrived.

3. User instructions for the Chino copper fixed point source

3.1 On receipt and setting up for use:

1. Wait for instrument to acclimatise to room temperature before starting to unpack;
2. Check that all items and accessories are present. Note how the items are packed into the crate, and how the fixed point components are packed in the inner cardboard box, so that they can be repacked in the same manner;
3. Unscrew and remove the ‘top plate’ cover of the furnace;
4. Unscrew and remove the metal protection plate at the rear of the furnace;
5. Remove the rear alumina insulation block;
6. Remove the plastic packing material;
7. Wearing gloves assemble the fixed point unit within the soaking tube according to the diagram and photograph in the accompanying documentation; namely insert the components in the following order from back to front within the tube:

   Insulation block 3; buffer; crucible; alumina aperture; graphite ring; insulation block 2

   Notes: The mark on the crucible assembly should face upwards. Hold the fixed-point unit carefully with both hands and do not hold just by the thin tube as this could result in breakage.

8. Put the two soaking tube holders on to the front and back of the soaking tube, then slide all the components into the furnace in the order: insulation block 1-1; insulation block 1-2; fixed point unit; insulation block 4;
9. Put back and screw on the metal protection plate;
10. Connect the rubber hose on to the centre tube of the metal protecting plate;
11. Screw into place the ‘top plate’ cover of the furnace

12. IMPORTANT: Check that the correct fuse is installed for the mains supply voltage: 5 A for 230 VAC and 10 A for 110 VAC;
13. Connect the argon gas supply to the inlet on the bottom rear of the furnace and connect the power supply cable
14. Ensure that the 'fixed point pin' is placed in the Cu fixed point position.

3.2 Fixed point realisation

1. Set the argon gas flow rate to maximum on the regulator and let it flow for a few minutes, then set the flow rate to 0.7 litres/ minute on the flow regulator;
2. Turn on the mains power supply switch;
3. Set the correct pattern (program) number for the Cu fixed point (refer to the instruction manual and/ or the video for full details of how to do this, and how to subsequently run the program). The correct pattern is number 1;
4. Press the heater on/ off switch and then run the program. The argon gas will start to flow automatically when the furnace reaches the appropriate temperature (230 to 250 °C); check that the gas flow light is green and also check the flow periodically;
5. The melt is realised automatically using the pre-set program. When this is complete the freeze should be initiated (again refer to the full operating instructions and/ or video);
6. Once the freeze is complete the cycle can be repeated if necessary;
7. At the end of the measurements switch the heater on/ off switch off;
8. DO NOT turn off the mains power supply switch until the argon gas automatically shuts off, and DO NOT turn off the argon gas supply until the furnace reaches the temperature for automatic gas shut-off.

3.3 Shipment to the next participant:

Notes: Do not ship the furnace with the fixed point cell inside the furnace, and make sure that the power cable is disconnected from the furnace and the furnace is cold before removing the top cover.

1. Disconnect the argon gas supply tube;
2. Unscrew and open the 'top plate' cover of the furnace;
3. Disconnect the rubber tube from the inlet of the protection plate at the rear of the furnace;
4. Unscrew and remove the back protection plate;
5. Remove the alumina insulation block and the fixed point cell assembly;
6. Replace the plastic packing material into the furnace tube;
7. Re- insert the alumina insulation block and replace and screw on the back protection plate;
8. Screw the 'top plate' into place;
9. Carefully disassemble the fixed point unit (the reverse of Section 3.1 points 6) and 7)) and re-pack all the components in bubble wrap within the cardboard box;
10. Replace all the items into the shipping crate in the same manner as they arrived.

APPENDIX 3 – HTFP MEASUREMENTS

1 Preparing the furnace for the HTFP measurements

Prior to the start of the measurement campaign, in order to minimise the risk of contamination of the HTFPs, the furnace and its component parts should be baked out.

By way of precaution, the furnace should be considered **heavily contaminated,** if it has ever been used for filling of a fixed point cell, or if it has been used extensively for realising fixed points of high vapour pressure, such as Pd-C, Cu, Cr_3C_2-C or TiC-C. In this situation participants should consider more extensive replacement of furnace parts and/or extra baking to ensure these metals cannot possibly contaminate the HTFP.

Contamination of a cell by a polluted heater or furnace insulator can happen not only during the filling process, but also during the normal implementation of the cell. This is why the bake-out procedure described below must be followed at the start of the measurement campaign.

In addition, participants will need to have a set of cell holders (if applicable for their furnace type) and insulation material dedicated to each *type* of fixed-point. All components that touch the cell directly should be replaced when the cell *type* is changed. Any new materials need baking out (according to the procedure below) before use. As long as they are used only with one type of fixed-point material, this can happen prior to the start of the measurement campaign. Such materials may be used for other cells (i.e. cells not participating in this measurement campaign) prior to the measurement campaign – but only cells of the same type. So, taking Ru-C as an example, materials that touch the cell (insulation, cell holders, etc.) used for Ru-C cells can only be used with other Ru-C cells. Such materials need baking out according to the procedure for Ru-C (below) before their first use, which may be before the measurement campaign, or before the participant's own measurements with different Ru-C cells. The same is true for the other fixed-point materials.

If all materials have been purified before the start of the measurement campaign, and measurements are made in the order WC-C, Ru-C, Ni-C-X, then during the measurement campaign only a single bake-out is required at the start at the WC-C temperature. Participants may choose to bake-out their furnace between cell types.

The following sections describe the bake-out procedure, if needed, for each furnace and at different temperatures. If only one bake-out is used, then this should be the bake-out for WC-C at the start of the measurements.

For *VNIIOFI/Vega HTBB* type furnaces:

To prevent contamination of the fixed-point cells the following steps should be followed. Note, however, that due to the high temperature of the WC-C melting point no final baking temperature is prescribed. Instead participants should take whatever steps they deem necessary for their particular furnace to minimise the risk of cross-contamination of the cells.

For Ru-C, Ni-C-X

a. The furnace, equipped with a quartz window or solid plate, must be baked at 2000 °C for approximately half an hour under vacuum and then at 2100 °C under pure argon purge (obtained by slightly unfastening the window) for a minimum time of 30 min.

b. If any deposition is observed on the quartz window/solid plate that cannot be removed by just a blast of dry air then the window/solid plate should be cleaned using an alcohol soaked wipe and the procedure in a. above, should be continued until no more deposition is observed. The furnace is then ready for installation of the cell.

For WC-C

a. The furnace, equipped with a quartz window, must be baked at 2000 °C for approximately half an hour under vacuum. Participants should then take whatever additional steps (e.g. baking) they deem appropriate to ensure the chance of contamination is minimised.

For *Nagano/Chino* type furnaces:

To prevent contamination of the fixed-point cells the following steps should be followed:

a. The furnace must be baked at 2000 °C under vacuum for at least three hours for all cell types. In the case of WC-C participants should take whatever additional steps (e.g. baking) they deem appropriate to ensure that the chance of cross-contamination is minimised.
b. If any contamination is observed on the quartz window that cannot be removed by air blow but requires wiping with an alcohol-humidified towel, the procedure in a. above should be continued until no more deposition is observed.

For *Thermo Gauge* type furnaces:

To prevent contamination of the fixed point cells the following steps should be followed:

a. the heating tube must be replaced by a new one for each cell type.
b. The furnace is baked under argon purge for at least three hours at 2000 °C before using the Ni-C –X and Ru-C cells. For the WC-C cells participants should take whatever steps (e.g. baking) they deem appropriate to ensure that the chance of cross-contamination is minimised.

IMPORTANT : Information on the use of the furnace prior to the comparison measurements (such as use for filling of cells), the cleaning procedures performed, and any indication as to the cleanliness of the furnace as a result of cleaning (e.g. observation of deposits and their disappearance) should be included in the measurement report.

2 Furnace insulation, cell holder and optimum cell position

The furnace should be made as uniform in temperature as possible around the cell. This may require the use of appropriate insulation (graphite felt, or possibly CC-sheet) around the cell, along with baffles placed both before and after the cell in the furnace tube.

It is recommended that participants test their furnace to determine the optimum arrangement of insulating materials and baffles. The HTFP cell should be placed in the most uniform part of the furnace to ensure even heating of the entire ingot. Determining the optimum position requires some preparative measurements. Initial tests should be performed **using an alternative (in-house) cell** by 'trial-and-error'. However it is recognised that a few measurements may be needed with the cells actually used for the comparison, as the optimum position may differ from cell to cell. These measurements should be kept to an absolute minimum and included in the total number of cycles for which the cell is used (see also Section 10.1).

The best location is identified where the melting plateau is flattest, and longest, with a clean entrance into and exit from the melt, neither of which should be 'rounded'.

There are some guidance papers concerned with setting up furnaces for HTFP measurements in the literature, including [A1 - A3].

3 Furnace operation

In order to avoid breakage of the cells during the comparison the ramp rates for heating the furnace from room temperature should be:

- ≤ 300 K/ hr for Ni-C-X
- <1200 K/hr for Ru-C and WC-C

The ramp rates for cooling the furnace back to room temperature after measurements should be:

- <400 K/hr for Ni-C-X
- <1000 K/hr for Ru-C and WC-C

4 Check of furnace thermal inertia

Participants are requested to measure the thermal inertia of the furnace used for the HTFP measurements, in case a correction and/ or uncertainty needs to be applied to the HTFP results to allow for any effect of differing thermal inertias on the poi of the melt. This check should be carried out close to the melting temperatures of each of the HTFP cells used in the comparison, with the cell installed in the furnace. This check can be carried out prior to the first melt/ freeze cycle for that particular cell.

The procedure is as follows:

- Set the furnace temperature to be sufficiently below the melting temperature of the cell (e.g. 30 K);
- Increase the furnace temperature by 10 K (step change) and monitor the temperature of the furnace using a suitable radiation thermometer (e.g. the standard radiation thermometer used for ITS-90 scale realisation within the laboratory). The cell should be kept in the solid state (i.e. do not melt the cell) during this check;
- The results of the check (measured furnace temperature versus time) should be made available to the pilot in the event that this information is needed during the analysis process.

4 References

[A1] Khlevnoy, B, Sakharov, M, Ogarev, S, Sapritsky, V, Yamada, Y and Anhalt, K 2008 Investigation of Furnace Uniformity and its Effect on High-Temperature Fixed-Point Performance *International Journal of Thermophysics* **29**(**1**) 271-284

[A2] Salim, S G R, Woolliams, E R, Dury, M, Lowe, D H, Pearce, J V, Machin, G, Fox, N P, Sun, T and Grattan, K T V 2009 Furnace uniformity effects on Re-C fixed-point melting plateaux *Metrologia* **46**(**1**) 33-42

[A3] Bourson, F, Briaudeau, S, Rougié, B and Sadli, M 2012 Determination of the furnace effect of two high-temperature furnaces on metal-carbon eutectic points. *Proceedings of ITS-9* AIP Conf. Proc. **1552** , 380 (20 AIP Conf. Proc. 1552 , 380 (2013) 13)

APPENDIX 4 – RECEIPT OF INSTRUMENTATION

Receiving NMI:

Date:

Artefact/ component	Received (yes/ no)	Damage (yes/ no)	Nature of damage
LP3			
Eyepiece			
Lens cap			
Objective extension ring			
110 V power unit			
230 V power unit			
RS232 cable			
Operating manual			
LP3DE.exe software			
Chino thermometer			
Lens cap			
DC power unit			
DC power cable			
Signal cable			
Lens blower			
Operating manual			
Power unit manual			
Cu fixed point			
Insulation block 1-1			
Insulation block 1-2			
Insulation block 4			
2 x Soaking tube holder			
Insulation block 2			
Graphite ring			
Alumina aperture			
Fixed-point crucible			
Buffer			
Insulation block 3			
Soaking tube			
Rubber gas tube			
AC power cables (3)			
Fuses (5A and 10A)			
Operating manual			
Ni-C-X fixed point			
Ru-C fixed point			
WC-C fixed point			

APPENDIX 5 – STABILITY MEASUREMENT CHECK USING THE TRANSFER COPPER FIXED POINT

Participant:

Date of check (day, month, year):

Thermometer identification	Gain/ range	Output signal† /V or /A	Ambient temperature/ °C	Thermometer internal temperature/ °C
LP3	R1			
Chino	L			

The working distance (distance from the thermometer to the fixed point aperture) should be as prescribed in Section 4, namely 700 mm (Chino thermometer) or 750 mm (LP3) from the fixed point aperture to the front of the thermometer casing.

† The reported output signal should be the average of the signals obtained during the central half of two freezing plateaux for each thermometer, and must be corrected for the dark reading (background).

APPENDIX 6 – CALIBRATION RESULTS FOR THE KC RADIATION THERMOMETERS

1) RESULTS FOR THE LP3

Nominal temperature / °C	ITS-90 temperature / °C	Output signal† / A	Range setting	ND filter	LP3 internal temperature / °C	Ambient temperature / °C	U (k = 2) / °C
960			R1	No			
1100			R1	No			
1300			R1	No			
1500			R2	No			
1700			R2	No			
1800			R2	No			
2000			R2	No			
2200			R2	No			
2400			R2	No			
2500			R2	Yes			
2600			R2	Yes			
2800			R2	Yes			
2900			R2	Yes			
3000			R2	Yes			
*							
*							
*							

* Repeat temperatures; fill in as appropriate

Working distance (distance from front of thermometer casing to blackbody)

= …….. mm

Aperture size of HTBB = …….. mm

† the output signals must be corrected for dark reading (background). For the measurements above 2400 °C the output signals should be those obtained with the filter in place, i.e. do not adjust the results to take into account the filter transmission.

2) RESULTS FOR THE CHINO THERMOMETER

Nominal temperature / °C	ITS-90 temperature / °C	Output signal† / V	Gain setting	Chino internal temperature / °C	Ambient temperature / °C	U(k = 2) / °C
960			L range			
1100			L range			
1300			L range			
1500			L range			
1700			L range			
1800			L range			
2000			M range			
2200			M range			
2400			M range			
2500			H range			
2600			H range			
2800			H range			
2900			H range			
3000			H range			
*						
*						
*						

* Repeat temperatures; fill in as appropriate

Working distance (distance from front of thermometer casing to blackbody)
= …….. mm

Aperture size of HTBB = …….. mm

† the output signals must be corrected for dark reading (background)

3) RESULTS FOR THE HTFP CELLS

Cell	Day	Melt number†	Melt transition step size / K	ITS-90 radiance temperature of poi of the melt / °C	U (k = 2) / °C
Ni-C-X	Day 1	2			
		3			
		4			
		Average day 1			
Ni-C-X	Day 2	2			
		3			
		4			
		Average day 2			
Ni-C-X		**Overall average**			
Ru-C	Day 1	2			
		3			
		4			
		Average day 1			
Ru-C	Day 2	2			
		3			
		4			
		Average day 2			
Ru-C		**Overall average**			
WC-C	Day 1	2			
		3			
		4			
		Average day 1			
WC-C	Day 2	2			
		3			
		4			
		Average day 2			
WC-C		**Overall average**			

† As stated in Section 9.2.2, the first melt of each day is not to be included in the analysis.

APPENDIX 7 – SAMPLE UNCERTAINTY COMPONENTS

1) Example of uncertainty components to be included for the radiation thermometer calibration

Uncertainty in the reference thermometer (uncertainty in ITS-90 realisation)	Reference fixed point uncertainty
	Reference fixed point measurement
	Spectral responsivity measurement
	SSE measurement and correction
	Nonlinearity
	Drift
	Ambient conditions
	Gain ratios
	Repeatability
Uncertainty in the comparison	HTBB window transmittance (if applicable)
	HTBB uniformity
	HTBB temperature stability
	HTBB emissivity
	HTBB effective source diameter and correction to reference diameter of 25 mm)
	Transfer device alignment on HTBB
	Transfer device short term stability
	Transfer device SSE
	Digital voltmeter
	Repeatability

2) Example of uncertainty components to be included for the HTFP measurements

Uncertainty in the reference thermometer (uncertainty in ITS-90 realisation)	Reference fixed point uncertainty
	Reference fixed point measurement
	Spectral responsivity measurement
	SSE measurement and correction
	Nonlinearity
	Drift
	Ambient conditions
	Gain ratios
	Repeatability
Uncertainty in the measurements	HTBB window transmittance (if window is used)
	HTBB effective source diameter and correction to reference diameter of 3 mm
	Alignment on HTFP aperture
	Determination of the point of inflection
	Repeatability