M(C)-C eutectic research plan – the next steps

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SUMMARY

This document contains the research priorities for the next five to six years for progressing metal-carbon eutectic fixed-point technology from an interesting research topic to a mainstream metrology tool. The main goal is the replacement of the current definition of ITS-90 above the silver point with a new definition that is

- Wholly thermodynamic
- Has significantly lower uncertainties than the current definition
- Simpler and more flexible to implement than the current definition
- Is accessible to the whole metrology community

We, the authors, strongly encourage that National Measurement Institutes and other relevant researchers working in this field allow their work to be guided by the contents of this document so that research resources are best utilised and progress to the desired goals can be achieved in the specified timeframe.

We further propose under the auspices of CCT-WG5:

- That, within the bounds of national metrology priorities, the world research effort in this field be coordinated to minimise duplication of research i.e. to achieve the best utilization of resources
- An annual workshop of eutectic research be held, organised by the CCT-WG5 research coordinator, where current research can be described, where researchers in the field can meet and discuss their work and where progress towards the objectives be reviewed and plans adjusted. Ideally this would involve the wider user community including industrialists. The first of these will be held at LNE, Paris 6-7 Jun 2006

1.0 Introduction

There has been a long-standing requirement for high temperature fixed-points, above the copper point, in both thermometry and radiometry. With the advent of metal-carbon eutectics [1] it is now clear that this requirement will be met. Since that time research into metal-carbon/metal-carbide-carbon (generically specified as M(C)-C) fixed-point cells has progressed rapidly. There are now many groups around the world in contact thermometry, non-contact thermometry and radiometry active in M(C)-C research. It is clear that these cells have huge metrological potential, and whilst not being the same as pure metal fixed-points they can nevertheless fulfil the same function, in the higher temperature regime.

The aim of this discussion document is to spell out the required research areas, with a particular emphasis (but not exclusively) in non-contact thermometry. It will propose a framework for co-ordinated research in this field and give an indicative timetable for the research priorities whose aim will be the implementation of the technology into mainstream metrology.

1.1 Current world research activities into M(C)-C eutectic systems

The aim here is to give a comprehensive brief summary of the current research undertaken in different research groups around the world. The data in Tables 1 and 2 are taken from the reviews written by Yamada [2] and Woolliams *et al* [3] and captures the current state of world research, as published, to early 2005. Other work that remains as yet unpublished has not been included – e.g. NIST, NIM and VNIIM M(C)-C cell construction for non-contact thermometry or NPL's cell construction for contact thermometry.

Table 1: Non-contact thermometry/radiometry

Organisation	Activity
NMIJ	M(C)-C cell construction, modelling, impurity studies, ITS-90 temperature measurement, comparisons, repeatability studies, filling studies, robustness studies
NPL	M(C)-C cell construction, modelling, impurity studies, ITS-90 temperature measurement, T measurement, comparisons, repeatability studies, filling studies, robustness studies
CNAM-INM	M-C cell construction, ITS-90 temperature measurement, comparisons, repeatability studies
PTB	ITS-90 temperature measurement, T measurement, comparisons, repeatability studies
VNIIOFI	M(C)-C cell construction (particularly for radiometry and above Re-C), T measurement, repeatability studies, comparisons
NIST	ITS-90 temperature measurement, T measurement, comparisons
KRISS	M-C cell construction, repeatability studies

Table 2: Contact thermometry

Organisation	Activity
NMIJ	M-C construction, ITS-90 temperature measurement, comparisons, repeatability studies, filling studies, robustness studies
LNE	M-C cell construction, modelling, ITS-90 temperature measurement, comparisons, repeatability studies, filling studies, robustness studies
PTB	M-C cell construction, ITS-90 temperature measurement, comparisons, repeatability studies, filling studies, robustness studies

1.2 Applying CCT recommendation CCT T2 (2005)

CCT-23 recommendation T2 to the CIPM states that “national laboratories initiate and continue experiments to determine values of thermodynamic temperature and the Boltzmann constant”. One of the underlying reasons for the recommendation was “that values of T are needed for the freezing temperatures of zinc, aluminium, silver, gold and copper, **and transition temperatures in eutectic** and other materials at higher temperatures, **to reduce uncertainties in thermometry and radiometry**” (emphasis added by authors).

To apply this recommendation for M(C)-C eutectic systems implies the following:

That NMIs strive to develop improved facilities for the determination of thermodynamic temperature of small aperture-size[1] M(C)-C eutectic fixed-point blackbody systems with low standard uncertainties (e.g. ≤200 mK (*k*=1) at 2800 K)

It is proposed in Section 2 of this document that multi-lateral comparisons be undertaken of M(C)-C eutectic systems by the radiometry community to aid progress towards this goal.

[1] The small aperture size (typically 3.0 mm, but ultimately dependent on cavity geometry) is to reduce the influence of the furnace on the temperature drop across the back-wall of the cavity and on the effective emissivity of the cavity-furnace combination.

2.0 Near/mid term research opportunities and challenges – 2006-2010

2.1. Introduction

The aim is to advance the work on M(C)-C eutectics sufficiently to measure temperatures with much lower uncertainties than those achievable by ITS-90 above the silver point by 2011. Before this can happen a set of primary research requirements has to be addressed. In addition a further programme of research, whilst not essential to the primary research objectives, would significantly deepen our understanding of M(C)-C eutectic systems.

The following bullet points indicate the areas where more research is needed; they are elaborated in more detail in sections 2.2 to 2.4.

The research requirements are:

- Firmly identify minimum purity requirement for cell construction (currently specified as 5N) and identify reliable sources of ultra-pure materials
- Understanding how to construct M(C)-C eutectics to guaranteed level of reproducibility (ultimate target ±50 mK) and produce recommended construction procedures
- Undertake long-term stability studies to definitively establish the suitability of these sources as fixed-point references
- Establish technical requirements for the construction of robust cells
- A clear understanding of the minimum furnace requirements and how to identify these
- Through modelling and backup measurements clear understanding of the temperature drop and furnace effect on the temperature drop
- Quantifying the influence of the furnace on the effective emissivity of the cavity-furnace combination.
- Constructing an uncertainty budget for the fixed-point cells – mirroring that currently used for contact thermometry cells, covering thermometer, furnace and cell effects
- Perform a two step comparison of radiometric (T) scales, using selected M(C)-C cells to ascertain, step1, the current status of radiometry, provide indications for further improvements and, step 2, assign definitive values of T to a small number of critically selected fixed points.

A further programme of research could be undertaken in parallel with the above to deepen understanding of M(C)-C eutectic systems

- Justifying, from a physical basis, why the melt inflection point is the best temperature
- Clear understanding of the role of impurities and their contribution to the uncertainty
- Understanding the role of eutectic structure

2.2. The primary research requirements

The research required to achieving the desired objectives is described in detail in this section.

2.2.1. Cell reproducibility

Identify sources of ultra-pure materials

The supply of ultra-pure filling materials, and the unreliability of assay, remains an important issue. The current recommended minimum purity is 5N (mass basis). However the same 5N material but from different batches, even from the same supplier, can have very different residual impurities, therefore, besides the requirement for 5N materials, limits *may* have to be specified in the level of specific impurities to ensure that the goals of 2.2 can be achieved. This is because intra-cell reproducibility appears to be mainly governed by the differing impurity levels [and distributions] between individual cells.

One short-term approach to guaranteeing the required purity of samples is to establish good working relationships with materials suppliers whereby they reserve a batch of material, the user has a sample analysed by GDMS or other reliable method, and on the basis of the analysis accepts or rejects the batch.

In the longer term work needs to be done with suppliers of materials, and chemical analytical institutes (e.g. BAM), so that our communities requirements are clearly understood and work instigated by them to ensure materials of the required purity can be routinely and reliably supplied.

Cell construction

Much progress has been made in this regard. Comparisons of independently constructed M(C)-C eutectics at NPL [4] and at PTB [5] have indicated that, while some construction problems remain (particularly controlling contamination during manufacture and subsequent use), it should be possible to attain reproducibility between cells from different sources of ±100 mK above 2300 K. Beyond this more work will need to be done to move to the ultimate aim of ±50 mK [6].

The remaining work to be done is

1) Develop a guideline for the reliable filling of cells to ensure that contamination can be eliminated during manufacture
2) Document an outline guide for the construction of reproducible and reliable M(C)-C cells. Reliability is addressed in 2.2.2.

2.2.2. Cell reliability

Long-term stability

One urgent research requirement that has generally been missing from current research in this field is systematic effort in establishing the long-term stability of M(C)-C eutectic systems, which may be affected e.g. by contamination during prolonged use. This is essential to demonstrate their utility as genuine fixed points. This can be relatively easily ascertained by constructing several cells from one batch of material, using quasi-identical filling procedures. One or more of the cells is then reserved as a reference, the other cells are used and a note kept of their usage. Periodic comparison with the reference cell would ascertain the long-term stability of such systems.

Robustness.

Robustness refers to the lifetime of a cell which is terminated by breakage due to extensive use. The issue of robustness is thought to have been largely addressed through the matching of graphite and metal expansivities [7], or by utilizing an internal isolation of highly purified carbon composite sheet material [8] though these will still need further verification.

2.2.3. Methods for specifying the operational characteristics of M(C)-C eutectic fixed points

Furnace effects on the measured M(C)-C eutectic temperature
It is clear that the quality of the furnace has a significant impact on the quality of the realised M(C)-C eutectic plateau, and in extreme cases of the realised temperature itself. It is required that minimum furnace conditions for good eutectic measurements be established. Pragmatically this may simply be determined by testing the performance of any furnace through the use of a known good M(C)-C eutectic cell. However more fundamental studies should be performed to facilitate understanding, in particular, the role of temperature gradients on plateau shape and duration. These studies may be greatly augmented by thermal modelling. For instance [9] has shown that simple changes to insulation can have a profound effect on furnace gradients and the duration of melting plateaux in contact thermometry.

The temperature drop across the backwall of a blackbody cavity
This is a clearly understood issue, but poorly characterised. For lower temperature fixed points (Cu and below) a simple model was employed suggesting that reasonable uncertainty estimates could be obtained whilst assuming that all radiant exchange was extra-furnace, neglecting that between furnace and cavity (and within the cavity). However for higher temperature, and in particular, larger aperture systems this approach is not adequate.

Pioneering modelling in [10] with ANSYS has indicated that at high temperatures radiance exchange within the cavity and between the cavity and the hot furnace surroundings has a large impact on reducing the perceived temperature drop at the backwall. More modelling work needs to be performed, with realistic furnace conditions, to ensure that the temperature drop is properly understood and accounted for.

The effective emissivity of the cavity-furnace combination.
The furnace can influence the effective emissivity of the cavity-furnace combination; its influence increases with increasing cavity aperture for a given cavity geometry. It has been shown that at the eutectic temperature of Re-C the furnace contribution to the spectral emissivity -depending on the cavity geometry- can be virtually eliminated for an aperture diameter of 3.0 mm over the whole spectral range from 200 nm to 2.5 µm, whereas it remains substantial for an aperture of 8.0 mm [11]. Care must be exercised that this effect is properly accounted for when absolute temperature determinations are undertaken, or fixed-points are compared in different furnaces, or fixed-points of different design are compared.
In addition to the above there is a basic requirement to measure the emissivity of graphite at high temperatures.

The establishment of an uncertainty budget for M(C)-C eutectic fixed-point cells
It is highly desirable to be able to calculate the uncertainty budget for a specific fixed-point cell. That is given materials and graphite of specified purity and a blackbody design that yielded a particular emissivity it should be possible from first principles to calculate what the associated uncertainty should be. Influence parameters pertaining to the cell and furnace need to be included as well as those of the thermometer such as stability at high radiance, non-linearity, interpolation, SSE and extending the temperature range above 3000 K

2.2.4. Assignment of thermodynamic temperatures to M(C)-C eutectic fixed-points

One limitation to the progress in assigning thermodynamic temperatures to M(C)-C eutectic fixed points is the lack of absolute radiometry capability that can routinely achieve 200 mK standard uncertainties at 2800 K, hence the top-level recommendation of this document. For the full potential of metal-carbon eutectic fixed-points to be realised throughout the temperature standards community their thermodynamic temperature, T, must be assigned reliably by absolute radiometry. In addition a defining fixed point of the ITS-90 should be included in this assignment. Establishing T_{90}-T should be an important secondary objective of these measurements.

The absolute thermometry capability of the participating laboratories should be assessed via a multi-lateral comparison before starting the definitive assignments of thermodynamic temperature.

2.3. Implementation

To realize the targets formulated in 2.2 we propose a campaign to be set up involving six - partly concurrent - workpackages (WP's). A lead laboratory needs to be agreed on by participants and a protocol needs to be written for each WP.

WP 1: *Cell reliability*
Establishing methods of construction for stable and robust cells. Undertaking "long-term" thermometry/radiometric reliability studies. Proposed time frame 2006-2008.

WP 2: *Cell reproducibility*
The primary cells (at least two per M(C)-C eutectic) used for thermodynamic temperature assignment (in WP 5), will be constructed by at least three different laboratories. Their melt temperatures will need to be measured to assign reproducibility values before WP 5 is initiated. This assignment does not need to be performed using absolute radiometry, it is only a relative measurement. These measurements should be performed in at least one laboratory capable of undertaking measurements to the required precision to determine the said reproducibility. The proposed time frame for this WP is 2007-2009, to run in parallel with WP 4, but to be finished prior to WP 5.

WP 3: *Methods for specifying the operational characteristics of M(C)-C eutectic fixed points.*
The operational characteristics in question are reviewed in section 2.2.3. This WP should run in parallel with the WP's 1, 2 and 4, so the proposed time frame would be 2006 to 2009. Keeping track of the developments by the coordinator and exchange of information between the partners involved and coordinator would be the main features of this WP. An interim survey (mid 2007) and a final report (end 2009) are to be drafted by the coordinator.

WP 4: *Assessing absolute thermometry capability of participating laboratories.*
Using currently available eutectics a comparison of absolute radiometry capabilities is to be undertaken, using at most three eutectic fixed-points, of which stability and robustness have been checked in WP 1. Three possible candidates are Re-C, Pt-C and Pd-C. Ti(C)-C was considered but rejected for this exercise as being to early in its development phase for inclusion. The proposed timeframe for this comparison is 2007-2008, with up to 6 laboratories participating. A preliminary comparison of this type has already been performed between PTB and NIST using NPL cells with promising results [12]. This type of comparison would clearly identify where limitations in current radiometry capability existed and where further work was required to improve facilities.

In this workpackage, where validating the performance of institutes in measuring the thermodynamic temperature is the prime target, repeatability of the transfer cells is the main criterion. So only the requirements as regards cell reliability i.e. long-term stability and robustness need to be met.

WP 5: *Assigning thermodynamic temperatures within the context of a multi-lateral comparison.*

Before entering WP 5 it is important that the full research requirements specified in sections 2.2.1 to 2.2.3 be properly addressed.

After the lessons learnt from the comparison exercise in WP 4 have been implemented a second comparison should be performed to definitively assign thermodynamic temperatures to a set of selected M(C)-C eutectics. With the completion of WP 2 definitive high quality reliable and reproducible metal-carbon eutectic fixed-point cells should have been constructed by the temperature researchers. Provided the necessary level of consistency in results has been achieved these absolute radiometry measurements would produce the baseline thermodynamic temperatures for metal-carbon eutectic cells for years to come. In addition a defining fixed-point of the ITS-90 [Ag, Au or Cu] should be included in this comparison. The proposed time frame for this WP is 2009-2010, with again up to 6 laboratories participating.

WP 6: *Redefining temperature above the silver point*

Analysis of the results obtained in WP5. Preparing a proposal to the CCT on redefining temperature above the silver point (through mise-en-pratique) to formally allow dissemination of T mediated through M-C eutectics (2011).

The background to this workpackage is given in Section 3.0

2.4. Supplementary basic studies

In addition the following research is proposed, whilst probably not essential to achieving the primary objectives formulated in 2.2, is nevertheless important to understanding the physics and materials science background to metal-carbon eutectics.

Justifying the use of the melting temperature.

In thermometry, apart from the Ga point the freeze is universally used as the reference point, even for the fixed-points of Ag, Au and Cu used in radiation thermometry. It has been proposed that users of M(C)-C eutectics ought to follow the same well-established method and use the freeze to determine the temperature of the cells. However common practice, and the experience of researchers in the field is that the point of inflection of the melt is a highly repeatable quantity, much better than the freeze. Some studies have been undertaken to establish why this is the case but more work needs to be done to establish the physical basis why, in the case of M(C)-C eutectics, the melt inflection point is preferable to the freeze.

Understanding the role of impurities

The role of impurities in M(C)-C cells is still not clearly understood. However recent doping studies of Co-C with Fe indicate that it may be possible to characterize ternary systems satisfactorily on the basis of thermo-chemical software such as e.g. MTDATA [9] and Thermo-calc [13].

It may be possible, provided reliable assays are established, to calculate the effect of impurities, apply a correction and build reliable estimates for their effect into the uncertainty budget. Much more work needs to be performed before the case for using modelling to reliably estimate this effect is established such as doping studies for other systems in conjunction with thermochemical modelling.

This work should also address the feasibility of defining the melting temperature by the liquidus point of the *pure* M(C)-C eutectic system, which is an invariant quantity uniquely associated with the M(C)-C eutectic system in question.

Assessing the impact of the eutectic structure

The effect of the thermal history e.g. freezing rate or annealing on the eutectic structure, the possible interplay between structure and impurities and the impact on the melting behaviour should be clarified. From this it may be clear that close cooperation with materials science institutes is indispensable [14].

3.0 Redefining temperature above the silver point (2011)

The longer-term goal of this research would be that of providing a way of redefining temperature **above the silver point through absolute radiometry mediated by the use of M(C)-C eutectics**. The advantages of doing this are

a) Thermodynamic temperature can be directly disseminated to the user community via short traceability lines
b) The uncertainty in scale realisation and dissemination would be ~5-10x less than current scale
c) A methodology of scale realisation is set up that is a direct analogue of contact thermometry, giving much greater flexibility than the current ITS-90 – though some knowledge of interpolating instruments waveband is required when utilizing two-point interpolation schemes
d) This would enable most NMIs throughout the world to realise the new scale, which would be fully thermodynamic, without having to incur the cost of establishing absolute radiometry facilities.
e) Easy to disseminate the scale to user community through the supply, or self-construction to a prescribed recipe, of a set of M(C)-C eutectic cells.

This goal may be achieved through the research activities outlined in Sections 2.2 and 2.3. This work will converge on being able to construct metal-carbon fixed point cells from first principles, that will yield a known thermodynamic temperature, with a specified uncertainty, if used in a furnace of prescribed quality.

These cells could then be used to disseminate the scale to the user community through the calibration of secondary M(C)-C eutectic cells as transfer standards with only a $\sqrt{2}$ increase in uncertainty with respect to the primary laboratory.

It should be noted that a formal redefinition may not need to be implemented to take full advantage of these developments. The device of a mise-en-pratique may be employed to allow this improved methodology to exist alongside the formal ITS-90 definition provided the differences between the two methods are sufficiently understood.

4.0 Radiometry

The active participation of the international radiometry community is essential to achieving the aims of this work. More work needs to be done by the M(C)-C eutectic research community to explain the objectives of this work and why its important to their colleagues in radiometry. Without the active participation of the radiometry community the goals of this research will not be achieved on the timescale laid out in this document.

Currently the focus of research in the radiometry community on metal-carbon eutectics is at higher temperatures the lowest temperature of interest being Re-C, with the higher temperature TiC-C, ZrC-C (2882 °C) even HfC-C (3185 °C).

The cells are seen as have utility in radiometry in the following areas

a) determining the calibration state of filter radiometers (which are prone to step changes in spectral responsivity)
b) improving the primary realisation of spectral irradiance scales by daily calibrating a filter radiometer using a M(C)-C eutectic
c) primary UV B scale realisation – because currently the stability and the uniformity of variable temperature blackbodies is generally the dominant uncertainty
d) allow smaller NMIs to realise a primary radiance scale without the need to have the detector based infrastructure required for a full realisation

The eutectics under development in radiation thermometry will already fulfil some of these functions – e.g. primary radiance scales.

However for a direct spectral irradiance realisation larger aperture cells, at the ultra-high temperatures will be required, these are currently the special focus of research of VNIIOFI [15]. For these cells it is necessary for furnace effects on the temperature drop across the backwall of the cavity and on the effective emissivity to estimated and corrected for.

5.0 Contact thermometry

The utility of M-C eutectics for high temperature contact thermometry was identified from their inception. Programmes of research have been pursued, often collaboratively in several centres including among others NMIJ, LNE and PTB, through the FP5 HIMERT project [16]. Studies of the metrological characteristics of Pt-Pd thermocouples, the utility of cells for the calibration of noble metal thermocouples and the use of higher temperature eutectics for the characterisation of W-Re type thermocouples have been undertaken. Metal-carbon eutectic cells of Co-C, Ni-C, Pd-C, Pt-C and Ru-C have all been constructed with contact thermometry applications in mind.

Work in the forthcoming years will centre on improving cell robustness, improving the calibration of noble metal thermocouples through replacing the wire bridge method with Pd-C ingot [with the aim of reducing the uncertainty to users by a factor of at least two], research into improving W-Re thermocouples and possible investigation of the Pt-Pd reference function. The temperature assignment of the cells will depend, initially at least, upon radiation thermometry.

Currently a Euromet project, led by LNE, the title of which is “High temperature fixed points for improved thermocouple calibrations”, involving LNE, PTB and NPL formally started at NPL on 11-12 July 2005. The aim of this project is to make robust M-C eutectic fixed points, Co-C and Pd-C for the routine calibration of noble metal thermocouples.

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8.0 Appendices

Appendix 1: Summary of the workpackages outlined in section 2.3

WP	Time frame	WP objective
1	2006-2008	To establish the long term stability and robustness of M-C eutectic cells
2	2007-2009	Selecting, constructing and comparing reproducible eutectic cells for T measurements
3	2006-2009	Methods for specifying the operational characteristics of M(C)-C eutectic fixed points.
4	2007-2008	To plan and perform a radiometric comparison of already extant M-C eutectic cells to identify any weaknesses in absolute thermometry and to implement improvements
5	2009-2010	To plan and perform T measurements of definitive eutectic cells
6	2011	To redefine temperature above the silver point (through mise-en-pratique) to formally allow dissemination of T mediated through M-C eutectics

Notes:

a) WP's 2 and 4 run mainly in parallel. WP 3 runs in parallel with WP's 1, 2 and 4.

Appendix 2: Time chart