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JRP full title	Implementing the new Kelvin
Version numbers of latest contracted Annex Ia and Annex Ib against which the assessment will be made	Annex Ia: V1.2 Annex Ib: V1.0
Period covered (dates)	From 1st October 2012 To 30th September 2015
JRP-Coordinator	
Name, title, organisation	Graham Machin, Dr, NPL
Tel:	+44 20 8943 6742
Email:	graham.machin@npl.co.uk
JRP website address	http://projects.npl.co.uk/ink/
Other JRP-Partners	

Short name, country	CEM, Spain	CNAM, France
	CSIC, Spain	INRIM, Italy
	LNE, France	VTT, Finland
	PTB, Germany	TUBITAK, Turkey
	DIISRTE, Australia	NIM, China
	UVa, Spain	VNIIOFI, Russian Federation

REG-Researcher (associated Home Organisation)		
REG-Researcher SIB01(REG1)	Aya Shibahara	Start date: 01 Oct 2012
(associated Home Organisation):	RHUL, United Kingdom	Duration: 36 months
REG-Researcher SIB01(REG2)	Matthias Meschke	Start date: 01 Jan 2013
(associated Home Organisation):	AALTO, Finland	Duration: 24 months
REG-Researcher SIB01(REG3)	Livio Gianfrani	Start date: 01 Nov 2012
(associated Home Organisation):	UniNA2, Italy	Duration: 24 months
REG-Researcher SIB01(REG4)	Livio Gianfrani	Start date: 01 Dec 2013
(associated Home Organisation):	UniNA2, Italy	Duration: 22 months
ESRMG-Researcher	Jose Manuel Mantilla Amor	Start date: 01 Mar 2013

Report Status: PU Public

SIB01(ESRMG1)   CEM, Spain   Duration: 4 months

(associated Home Organisation):

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1   Executive Summary

Introduction
This project addressed outstanding high-priority issues in temperature measurement for the international metrology (measurement science) community. In particular the methods and data generated here are being used to prepare the international measurement community for the forthcoming redefinition of the kelvin in 2018. The project developed National Measurement Institute capabilities for making and disseminating high-temperature (> 1000 °C) and low-temperature measurements (< 1 K) directly linked to the forthcoming new kelvin definition, and in addition, generated the lowest-uncertainty data ever achieved for the international temperature scale of 1990 (ITS-90).

The Problem
The kelvin (K), the SI unit of temperature in the International System of Units, will be redefined in 2018 as part of the Committee for Weights and Measures (CIPM) program to redefine all SI base units in terms of fundamental physical constants. The kelvin will be redefined in terms of a fixed value of the Boltzmann's constant.

To ensure the redefinition is successful, the transition must be supported by accurate primary thermometry methods and a robust and documented *mise en pratique* (*MeP*-K) that allows the new definition to be realised in practice at National Measurement Institutes (NMIs). Ahead of the redefinition the BIPM's Consultative Committee for Thermometry (CCT) identified the following high-priority requirements in primary thermometry:

- The determination of transition temperatures for the high temperature fixed points to be used for realising and disseminating high-temperatures.

- A trial of the methods proposed in the *mise en pratique* for the dissemination of high-temperatures.

- More reliable, low-uncertainty values of the differences between the ITS-90 and thermodynamic temperature for the *MeP*-K, and potential future temperature scale.

- New measurements of the differences between PLTS-2000 and thermodynamic temperature for the *MeP*-K and improved calibration of temperature sensors below 1 K.

From the outset of the project it was realised these steps were a long-term process and would require a follow-on project. The InK 2 project was started in June 2016 to complete the work begun in InK 1.

The Solution
The assignment of thermodynamic temperatures to the new HTFPs will make available *a completely new generation of fixed points for temperature dissemination and scale comparison*
Comparison of two completely different methods of radiometric measurements at high temperatures will enabling the temperature community to choose the most appropriate dissemination and comparison method
New reliable values of $T - T_{90}$ with the potential to *reduce the uncertainties in the current best estimates.*
 The current PLTS-2000 is based on discrepant data (of order of 6 %) at the lowest temperatures; This very unsatisfactory will begin to be resolved through developing three independent reliable thermodynamic thermometer measurements to determine $T - T_{2000.}$

Impact

The project has made advances in temperature measurement which will be particularly important in supporting the international thermometry community in the transition to the new definition of the kelvin in 2018.

Dissemination of results
The results of the project have been shared widely throught the thermometry community. 63 papers have been published in journals (listed in the next section), and it is likely that further papers will be published incorporating work from this project with the InK 2 project. A Royal Society workshop, *Towards implementing the new kelvin*, was held in May 2015 and proved a fitting climax to the project, with many new and important results presented. All presentations, linked to InK 1, from the event have been published in a special edition

of the *Philosophical Transactions of the Royal Society A.,* Vol. 374, March 2016. This volume will serve as the most important method of dissemination and will prove to be an enduring record of the InK 1 project. Other dissemination activities include annual reports to the CCT and Regional Metrology Organisation's Technical Committees for Thermometry, and invited presentations at international conferences.

Immediate impact

The project has achieved immediate impact in four technical areas:

1. For the first time, definitive transition temperatures for a set of high-temperature fixed points have been determined, and the Cu freezing point has been re-determined. These new values have been published and will, when further work to assign final uncertainty components for furnace and impurity effects, be recommended to the CCT for inclusion in the next version of the *mise en pratique* (*MeP*-K).

2. For the first time, a comparison of two *MeP*-K recommended approaches for disseminating high-temperatures have been trialled and compared. The work has been published, and the outcomes have been discussed at the CCT Working Group for non-contact thermometry. Recommendations to the CCT have been made on the basis of these findings, and will help the thermometry community to objectively decide which is the most appropriate dissemination approach for high-temperatures.

3. The world's lowest uncertainty determination of ITS-90 has been undertaken in the range of 25 K to 303 K, some of which was performed by two independent temperature measurement methods. This data has been considered by the CCT Working Group for Contact Thermometry for inclusion in a revision of the thermodynamic temperature data annex of the *MeP*-K.

4. For the first time since its inception, PLTS-2000 has been comprehensively investigated using independent methods in the temperature range of 0.02 K to 1 K. The approaches developed here will be used in the follow-on InK 2 project to evaluate PLTS-2000 over its complete range (i.e. down to 0.9 mK above absolute zero).

Potential future impact

The results of this project, when combined with those of the InK 2 project, will make a major and enduring contribution to the thermometry community through the recommendations made to the CCT, and through the provision of new and more accurate temperature data to refine the current temperature scales. The methods and data developed here will support the successful realisation of the redefined kelvin through the development of an effective *mise en pratique*, and in the longer term potentially inform a future temperature scale.

2   Project context, rationale and objectives

The need for the project

Background

Nearly all temperature measurement around the world is currently based on traceability to a defined scale either the ITS-90[1] or, below 1 K, the PLTS-2000[2]. Defined scales are used because, whilst more fundamental, primary methods, based on an equation of state such as the gas law or Planck's law *have been* intrinsically less reliable, with higher uncertainties. However recent technical innovations at the highest level of temperature metrology, the developing *mise en pratique* for the definition of the kelvin (*MeP*-K) and the proposed kelvin redefinition (targeted for around 2018) provide a unique opportunity to fundamentally change the practice of temperature measurement.

The major objective of this project was to perform the necessary background research to help prepare the temperature metrology community for a smooth transition to the new kelvin definition. It was clear as the project progressed that a follow on project was required to complete this task and lead into the formal redefinition at the end of 2018, this has been agreed, is known as InK-2, and will start in June 2016.

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[1] http://www.bipm.org/en/publications/its-90.html
[2] http://www.bipm.org/utils/en/pdf/PLTS-2000.pdf

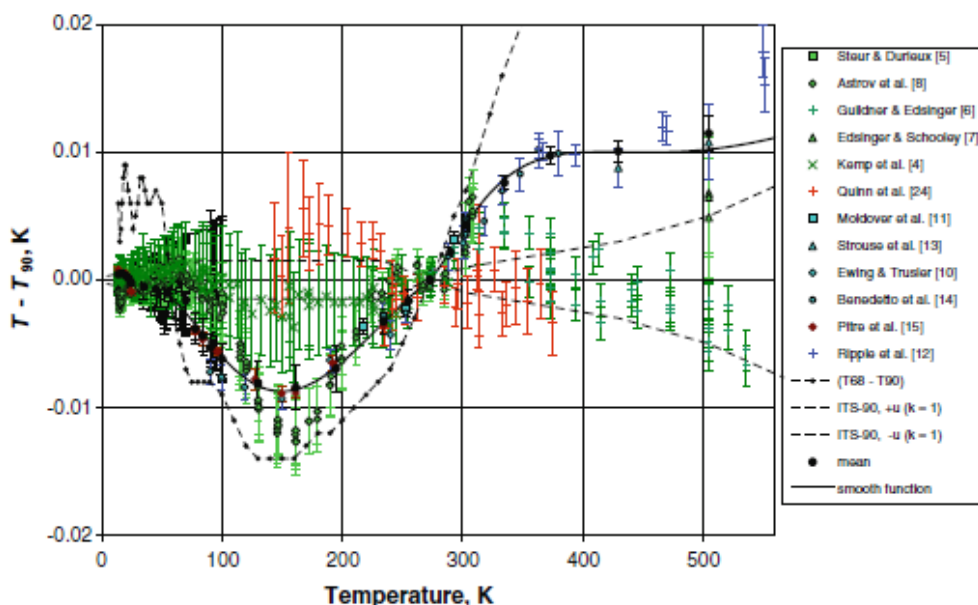Examples of the current problems faced by the thermometry community are given in Figure 2.1 and 2.2.

Figure 2.1 shows a summary of the data $T\text{-}T_{90}$ prior to this project, with many discrepancies larger than the uncertainties. InK aimed to significantly increase the number of these data sets, and reduce the uncertainties in the best estimates.

The current PLTS-2000 is based on discrepant data (of order of 6 %) at the lowest temperatures; this can be seen in Figure 2.2 below. This is very unsatisfactory so in this project $T - T_{2000}$ will begin to be re-measured to address this discrepancy.
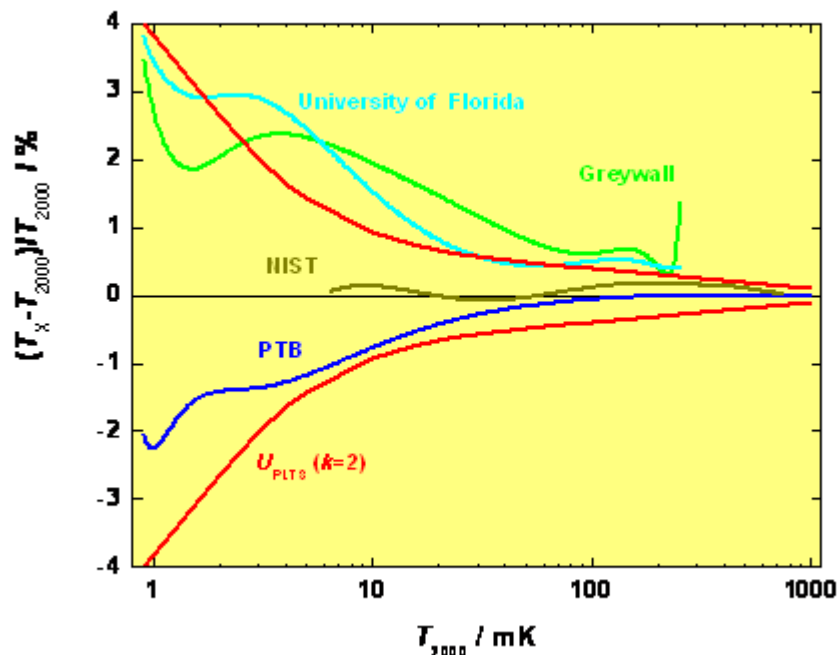
Figure 2.2: The background data to PLTS-2000 ($T_X \equiv T$). The discrepancy in the different data sets at the lower temperatures grows rapidly to 6% at ~0.0001 K leading to a large uncertainty increase in the PLTS-2000

A final high level objective was to develop the means to improve high temperature metrology above the silver freezing point (i.e. the high temperature part of the ITS-90). This involved research into two areas:

- Determination for the first time, by primary radiometry, the thermodynamic temperature of a selected set of high temperature fixed points (HTFPs; Co-C, Pt-C and Re-C). The temperature and radiometry community has been calling for such high temperature fixed points to be developed since 1996. These fixed points could then be used to reliably realise and disseminate T rather than T_{90}, with similar or even lower uncertainties than the current scale, thus demonstrating that a direct and reliable linkage to the new kelvin could be realised.
- A comparative study of disseminating the thermodynamic temperature, T, using both radiometers and high temperature fixed points. This was to develop the techniques for future dissemination of T instead of a defined scale and to show that it was possible with sufficiently low uncertainties.

In summary the research needs are:

- The assignment of thermodynamic temperatures to the new HTFPs will make a completely new generation of fixed points available for temperature dissemination and scale comparison

- Comparing two completely different methods of radiometric measurements at high temperatures will enable the whole temperature community chose the most appropriate dissemination and comparison method

- New low uncertainty values of $T - T_{90}$ will begin to provide a definitive set of data for any users requiring thermodynamic temperatures and provide the reliable background data essential for any high integrity replacement scale to ITS-90

- The new measurements of $T - T_{2000}$ will begin to solve the long standing discrepancy in the background data of the PLTS-2000

- New high performance sensors will be developed to facilitate the dissemination of thermodynamic temperature at low temperatures

Stakeholder Need:

The high level stakeholders for the research described in this project are a) the world metrology community, the international committee of weights and measures (CIPM) and its consultative committees for thermometry (CCT) and units (CCU) and b) the regional metrology organisations (RMOs) technical committees for thermometry (TC-T). All these stakeholders recognise the problems with the current defined temperature scales and also recognise the challenges that the redefined kelvin will bring. A priority for the international temperature metrology community is to maintain a consistent realisation of the SI unit for temperature and practical means of its dissemination in this changing environment.

Industrial users whose processes are reliant on high accuracy thermometry need direct traceability to the redefined kelvin in order to guarantee long term confidence in their temperature measurement, ideally without recourse to a defined scale – which can change over time. In addition, a consistent approach to thermometry (i.e. a definition of temperature that is based on a fundamental quantity) will mean that Industry need not worry about any future changes in temperature scales (which are costly to implement).

Links to standards

The links to standards is through the CIPM CCT providing substantial input into the developing *mise en pratique* for the definition of the kelvin (*MeP*-K) and also through development of primary thermometry methods that facilitate a direct realisation and dissemination of the redefined kelvin.

Need for a collaborative approach:

This project brought together primary thermometry activity from around Europe, and the world. A collaborative approach was required because:

- Improvement to primary thermometry is required on a broad front; that is in six orders of magnitude of temperature.

- Individual primary thermometry methods are expensive and more than one or at most two methods was likely to be unaffordable by a single organisation

- A large number of data sets are required, and in order to achieve this is a timely manner many organisations must be involved

- The uptake of the results from the InK project requires wide acceptance of results gathered from many organisations, This is best achieved through a collaborative approach involving all the main institutes involved in primary thermometry, not only those in Europe.

Detailed overview of the scientific and technical objectives

1) Assign thermodynamic temperatures to high temperature fixed points above 1000 °C (>1300 K).

   The aim of this objective was to assign definitive low uncertainty temperatures to a set of high temperature fixed points, namely the Cu point, the Co-C (1597 K), Pt-C (2011 K) and Re-C (2748 K). The Cu point was included because of unresolved problems with the current measurements as specified by CCT-WG4, and to retain linkage with ITS-90. High quality high temperature fixed points are one of the most sought after artefacts in high temperature measurement as identified by a joint working group of CCT and CCPR. The selection of the Co-C, Pt-C and Re-C points was pragmatic in that they are the most developed of the HTFPs, reliably showing the required level of reproducibility (<0.1°C even at the Re-C point) and are far enough spread in temperature to be used to determine low uncertainty temperatures, when required, of other HTFPs in the future.

2) Realisation and dissemination of *T* instead of ITS-90 at high temperatures.

   The aim of this objective was to perform a detailed evaluation of two potential schemes for the realisation and dissemination of *thermodynamic temperature* above 1000 °C. The two possible alternatives were:

   a) Dissemination by absolutely calibrated radiation thermometers, directly traceable to the electrical watt, the metre and the second

   b) Dissemination mediated through *a priori* calibrated or known temperature references; namely high temperature fixed points.

These alternative dissemination routes for thermodynamic temperature were *for the first time* evaluated with the involvement of all the European NMIs capable of such measurements. Both approaches have strengths and weaknesses and these will be identified and recommendations made to CCT.

3) Determination of $T - T_{90}$ with lowest ever uncertainties.

The aim of this objective was to effect a significant reduction of the uncertainty (a factor two) currently associated with ($T - T_{90}$) over the extended temperature range 2.5 K to ~650 K. The selected technical work has been guided by the recent definitive critical revision of the existing $T - T_{90}$ values. The scientific activities of this objective involved the use of various primary thermometry methods (acoustic, dielectric, noise, refractive index and spectral radiometry) for a low uncertainty determination of T. The comparison of results obtained by the different methods will significantly reduce the contribution of systematic uncertainties that affect each particular technique.

4) Develop primary methods of realising and disseminating T below 1 K and resolve the long standing discrepancy in the low temperature part of PLTS-2000.

The aim of this objective was to develop and improve primary thermometers for measurement of thermodynamic temperature with low uncertainties. The comparison of thermometers based on different primary methods will facilitate the identification of systematic effects in thermodynamic temperature measurement which wouldn't be possible if only one technique was used. This research could also lead to the direct dissemination of the redefined kelvin in the low-temperature range.

3   Research results

Objective 1- Assign thermodynamic temperatures to HTFPs above 1000 °C (>1300 K)

Introduction

The purpose of this objective was to use primary radiometry to determine for the first time definitive thermodynamic temperatures to a selected set of high temperature fixed points (HTFPs). A secondary objective was to re-determine the copper point whose current value was based on discrepant data.

This objective was attained, the Cu point was re-determined and the world's most reliable values for the melting temperatures of Co-C, Pt-C and Re-C measured. These measurements were performed by ten NMIs, some of whom were funded partners of the InK project, some of whom were unfunded partners and some of whom were collaborators.

The results were presented at a Royal Society workshop held on 18-19 May 2015 and have been submitted to CCT WG NCTh to inform the emerging *MeP*-K-19.

Research undertaken

Introduction

The project involved the circulation between the participating laboratories of four HTFP cells of each type (i.e. four Re-C, four Pt-C, four Co-C and four Cu). The cells had been preselected to be the best available cells from a larger batch of cells made following best practice techniques established through earlier international collaboration. Each participant measured two or four of these cells using a primary radiometric thermometry technique. The measured values were then combined to obtain a single best estimate of the thermodynamic temperature.

Measurements

Participants made measurements by determining the absolute radiance of a source (in this case the blackbody cavity surrounded by the HTFP material) in a narrow wavelength band. This absolute radiance is then used to determine the thermodynamic temperature of the blackbody via Planck's Law.

The measurement of absolute radiance is usually done with a filter radiometer – an instrument with an optical system comprising two apertures, a filter and a detector. In some cases the primary filter radiometer included a lens, and here the HTFP cells could be measured directly. In other cases, where the primary instrument did not include a lens, the filter radiometer was used to measure the temperature of a variable temperature blackbody with a large aperture, and this was, in turn, used to calibrate a radiation thermometer that could be used to measure the HTFP cells. Note that one participant used a monochromator-based system rather than a filter radiometer and another participant used a photometer, a specific type of filter radiometer that mimics the human eye response and which has a different calibration methodology.

There are four calibration methods (radiance, irradiance, power and hybrid) that have been proposed for the future edition of the *mise en pratique* for the definition of the kelvin. These methods differ in where in the calibration chain two apertures are introduced to define the geometry of measurement. All four calibration techniques were used by different participants in this measurement campaign. Measurements were also made at different wavelengths (from 520 nm to 808 nm) by different participants. Some participants had broadband filter radiometers (with bandwidths of 50 nm or more) and others had narrowband filter radiometers (with bandwidths of 20 nm or less), one used a monochromator as the filtering system.

Despite this variety of methodology, and the fact that some participants used new facilities and others used well-established capability, the uncertainties achieved by the different laboratories were very similar, for example for the measurement of Re-C (2747.84 K) uncertainties ranged from 0.28 K to 0.57 K (see figure 3.1 – the graph of uncertainties). Furthermore the analysis showed a high level of consistency, both for the overall uncertainty budget and between the model and results (which suggested that participants had appropriately estimated correlation). This shows the maturity of filter radiometry as a method for determining thermodynamic temperature and was achieved through thorough analysis and review of sources of uncertainty at all participants and an open sharing of best practice, experience and challenges overcome. It also built on a previous "comparison of filter radiometry" that had been organised by the CCT[3] in 2009-2011, and which about half of the participants of this project had taken part in.
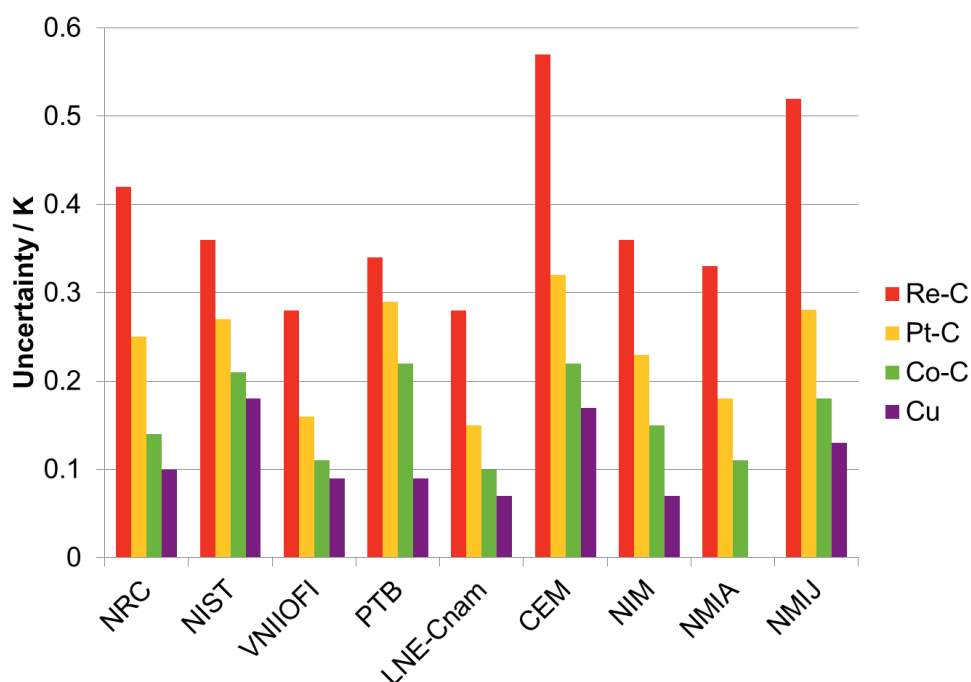

Figure 3.1 - Standard uncertainties (not expanded) associated with thermodynamic temperature measurements by the different participants for the different fixed points.

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[3] Consultative Committee on Thermometry and in particular WG5, which has been renamed as the working group for non-contact thermometry

Data analysis and results

The results of the measurement campaign were combined in a mathematically robust manner that took into account the full covariance of the measurements. This included analysis of the effects such as impurities within the fixed point materials, emissivity of the cavity, the temperature drop (the difference in temperature between the fixed point material and the inside of the cavity due to the thickness of the cavity wall) and structural effects due to the rate of the previous freeze. This analysis took into account the work of many other scientists, including collaborators to the project and unfunded partners.

Within this project what was determined was a pragmatic quantity, namely the point of inflection of the melting curve of the eutectic fixed points. The measured melting curves (temperature as a function of time) take the form shown in the red dotted line in Figure 3.2. The determined point of inflection (the purple diamond) was calculated from fitting a cubic to the central part of this curve.

There is a more fundamental quantity – the liquidus temperature obtained under equilibrium conditions. The consortium is currently carrying out further analysis to understand what uncertainty to include to estimate the liquidus temperature. This is possible to calculate from the data obtained during this project and will be presented in Tempmeko 2016 (see section on future impacts).
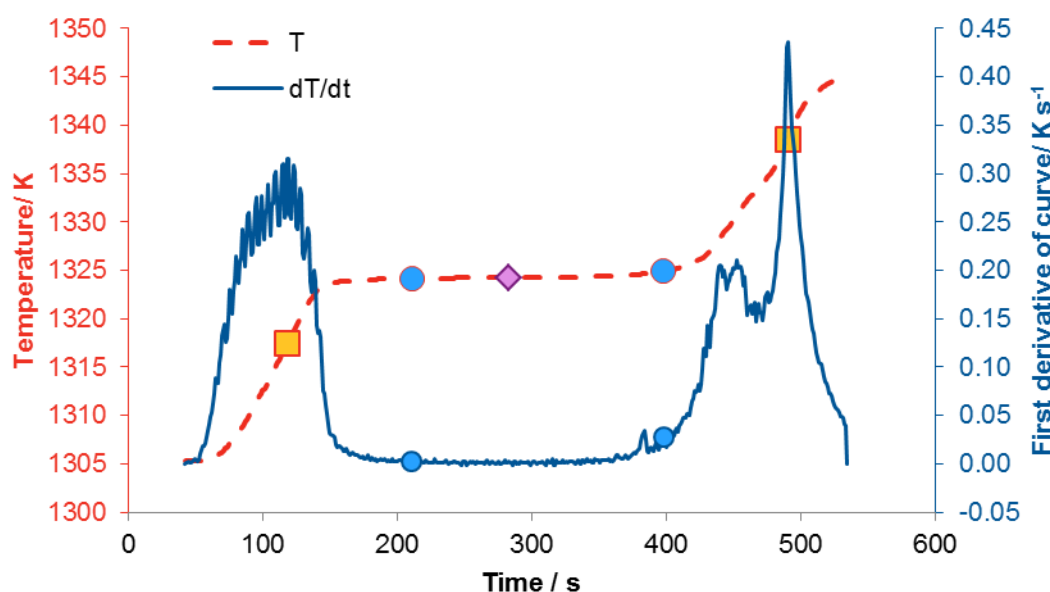

Figure 3.2 - A typical melting curve (dashed red line) and its first derivative (solid blue line), showing the point of inflection (purple diamond) calculated from a fit between the blue circles, determined from the start of melt and end of melt (yellow squares).

The main outcome of the InK project was the publication of thermodynamic temperature values for the four HTFP transitions (the point of inflection of the melting curve for the eutectic fixed points and the freezing point for copper). These were presented at the Royal Society's meeting on implementing the new kelvin, which was held in May 2015. A paper, with 40 co-authors, was prepared, submitted to and accepted for publication by Philosophical Transactions of the Royal Society. It is anticipated that this will come into print in February 2016.

The temperature values given in that paper were:

	Value /K	Associated standard	Expanded uncertainty

HTFP		uncertainty / K	$(k=2)$ / K
Re-C	2747.84	0.18	0.35
Pt-C	2011.43	0.09	0.18
Co-C	1597.39	0.06	0.13
Cu	1357.802	0.041	0.081

This paper has extensive "supplementary material" including an additional document describing the analysis method, providing the uncertainty budgets for each participant and giving more information about impurity analysis, amongst other things. The raw data is also presented in spreadsheets to allow further analysis to take place.

More fundamentally, this project has shown that primary radiometric thermometry is now sufficiently mature to be used as a method for realising, directly, thermodynamic temperature. This is key enabling evidence to support the expected next edition of the *mise en pratique* for the definition of the kelvin, which is expected to encourage primary thermometry techniques.

The project has also shown that it is possible to circulate HTFP cells and for these to hold a thermodynamic scale placed on them by filter radiometry. Only one, Re-C cell, showed any noticeable change during the measurement campaign (this conclusion is currently being verified by a post-campaign re-assessment of cell-to-cell differences). This supports relative primary thermometry – thermodynamic temperature mediated by fixed points.

The project has also come a long way to establishing the thermodynamic transition temperature for these fixed points. It has determined the point of inflection of the melting transition and has collated, and begun to analyse, the data needed to estimate by how much the more fundamental liquidus temperature may vary from this. Some further work is needed to bring this to a conclusion, and it is expected this to be presented at Tempmeko 2016, along with a recommendation to the CCT for implementing these temperatures into the temperature scales and into the evolving *mise en pratique*.

Summary of key research outputs and conclusions

The key research outputs are:

- The thermodynamic temperature of the point of inflection of the melting transitions of Re-C, Pt-C and Co-C and of the freezing transition of Cu has been determined with the lowest ever uncertainties.

- This temperature is a true consensus temperature of all the expert laboratories measuring using primary radiometric thermometry. The different laboratories achieved very similar uncertainties using differing methodologies and the results were consistent, both with overall uncertainties and with the analysis of correlation. This suggests that primary radiometric thermometry is a mature technique that can be used for realising thermodynamic temperature.

- A paper has been presented at the Royal Society, and published in a high level paper (see footnote 4), summarising these results, with extensive supportive material explaining the methodology.

- This measurement campaign was analysed in a more robust manner than is commonly used for this type of project.

Objective 2 - Realisation and dissemination of T instead of ITS-90 at high temperatures.

Introduction

This objective was devoted to the assessment of two different methods of dissemination of the thermodynamic temperature. These two methods are the dissemination via high temperature fixed points

(HTFPs) with assigned thermodynamic temperatures[4] and the dissemination by radiometers or radiation thermometers calibrated in terms of thermodynamic temperature.

In order to evaluate these two distinct dissemination methods two comparisons were organised. In the first one, the circulating artefacts were off-the-shelf high-temperature fixed point cells with transition temperatures ranging from 1597 K to 2747 K. In the second comparison, absolutely-calibrated pyrometers and filter radiometers were compared in the temperature range 1273 K to 2773 K in front of a variable temperature blackbody radiator.

This collaborative work allowed the determination of the level of uncertainties achievable in both cases as far as the participants to this project were concerned. It is however possible to generalise the findings to foresee and choose the future possibilities of dissemination artefacts and methodologies.

Research undertaken - Dissemination using HTFPs

Dissemination of thermodynamic temperatures using HTFPs was part of this objective. This work was performed with ITS-90 temperatures assigned to the HTFPs because definitive thermodynamic temperatures were only available at the end of the project. This task will however help assessing the methods required for dissemination when thermodynamic temperatures become available for the HTFPs.

To achieve this dissemination exercise, NPL and CNAM supplied two sets of HTFP cells at the Co-C, Pt-C, Ru-C and Re-C high-temperature fixed points. The cells were either off-the-shelf, used cells or had been constructed for this particular project.

Two batches of cells were constituted with one cell from each fixed point: Co-C, Pt-C, Ru-C and Re-C. One batch was meant to be kept in CNAM as a reference batch and the other batch was circulated among the participants. Before the start of the circulation of the cells, the circulating batch was compared to the reference batch. This initial comparison was intended to make a link between the cells and to be able to replace a cell in case of breakage without completely losing information between the cells. This initial comparison was performed by CNAM.

Table 3.1 summarises the cells used in the circulating batch and in the reference batch. Cells 7CO1, 7CO2, 7CO4, 5PT2, 6PT2, 4RE1 and 4RE2 were supplied by CNAM. Cells INKRU1, INKRU and PT2006-2 were supplied by NPL.

HTFP	REF	CIRC
Co-C	7CO2	7CO1 then 7CO6
Pt-C	5PT2	PT2006-2 then 6PT2
Ru-C	INKRU	INKRU1
Re-C	4RE2	4RE1

Table 3.1 Identification of the cells participating to InK in the circulating (CIRC) batch and in the reference (REF) batch.

Cells 7CO1 and PT2006-2 broke during the circulation process and were replaced by new cells (7CO6 and 6PT2 respectively). This showed in particular that the early designs of cells is not suitable (the case of Pt-C cell from NPL based on the C/C sheet design) and that the Co-C point is a difficult point for which robustness remains an issue even with the hybrid design (consisting of C/C sheets and a graphite sleeve). The new cells were constructed by CNAM in the hybrid design and compared to the reference batch of cells before being put back to circulation.

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[4] Woolliams, E., Anhalt, K., Ballico, M., Bloembergen, P., Bourson, F., Briaudeau, S., Campos, J., Cox, M. G., del Campo, D., Dury, M.R., Gavrilov, V., Grigoryeva, I., Hernandez, M.L., Jahan, F., Khlevnoy, B., Khromchenko, V., Lowe, D.H., Lu, X., Machin, G., Mantilla, J.M., Martin, M.J., McEvoy, H.C., Rougié, B., Sadli, M., Salim, S.G., Sasajima, N., Taubert, D.R., Todd, A., Van den Bossche, R., van der Ham, E., Wang, T., Wei, D., Whittam, A., Wilthan, B., Woods, D., Woodward, J., Yamada, Y., Yamaguchi, Y., Yoon, H., Yuan, Z., "Thermodynamic temperature assignment to the point of inflection of the melting curve of high temperature fixed points", Phil. Trans R. Soc. A. **374**: 20150044 (2016) http://dx.doi.org/10.1098/rsta.2015.0044

A further comparison of the circulating and reference cells was performed at the end of the dissemination trial and allowed the determination of the drift of the cells. Table 3.2 summarises the level of this drift for each of the cells. Of course, the two broken cells could not be assessed for drift.

Cell	Drift (unc k=2) mK
7CO6	-25 (22)
7CO1	Unknown
6PT2	- 41 (18)
PT2006-2	Unknown
INKRU1	27 (150)
4RE1	28 (54)

Table 3.2 Drift of the circulating cells during the dissemination trial (over a period of more than one year). Uncertainties (k=2) are given between brackets.

The drift can in all cases be considered as negligible in comparison with the uncertainties of temperature determination. Earlier studies have already shown that the stability of HTFPs in the long term is ensured in case of the hybrid cell design[5, 6] and this study shows clearly that, passed the robustness issues with early designs, HTFP cells can be considered as reliable and efficient artefacts for comparisons.

Each participant (CEM, PTB, NPL, UME and CNAM) reported the assigned ITS-90 temperature of the melting transition realised with the circulating HTFP cells in their facilities.

The uncertainties reported by the participants were mainly related to the ITS-90 scale and to the repeatability of the realisation of the melt. The uncertainties and temperature differences between the participating NMIs can be seen in figure 3.3. The discrepancy between the determined temperatures is within 1 K even at the highest temperatures.

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[5] Sadli, M., Bloembergen, P., Khlevnoy, B., Wang, T., Yamada, Y., et al. "An International Study of the Long-Term Stability of Metal-Carbon Eutectic Cells" International Journal of Thermophysics **32**, 1786-1799 (2011).
[6] Sadli, M., Yamada, Y., Wang, T., Yoon, H.W., Bloembergen, P., et al. "Stability and robustness tests on Co-C, Pt-C and Re-C cells: the first results" Acta Metrologica Sinica **29**, 59-64 (2008).
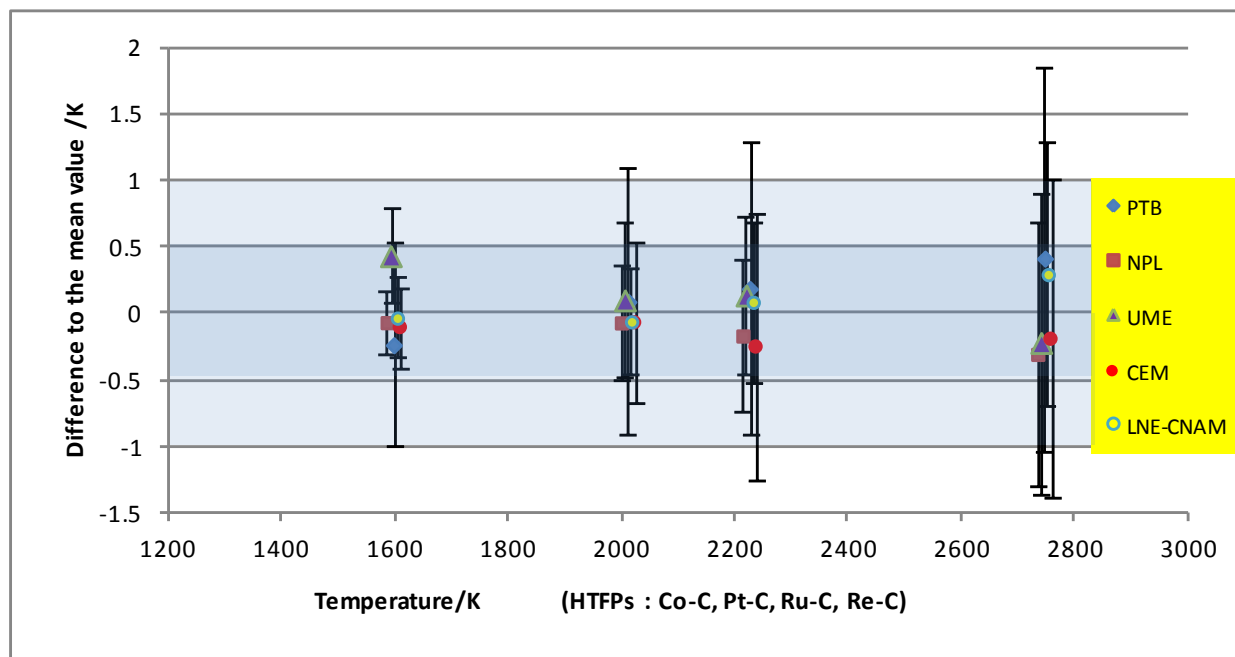
Figure 3.3: Differences between the reported temperatures by each participant for the four HTFPs. Uncertainty bars are expanded uncertainties (*k=2*).

To analyse the results of this comparison during which HTFP cells have been circulated among five partners over a period of more than one year, the consortium can already state that HTFPs can be considered as suitable means to probe and compare the capabilities between different realisations of the scale or of thermodynamic temperatures as the work to assign thermodynamic temperatures to a selected set of HTFPs[4] has already shown.

These results also show that it is already possible to use HTFPs to disseminate the scale with relatively low uncertainties. This would be a relatively simple process with two or more HTFPs having their temperature assigned by a national measurement institute and then sent to the user who would only need a suitable high temperature furnace and radiation thermometer for the interpolation. To reduce further the uncertainties in this approach to dissemination some sources of uncertainty need more detailed investigation. The two main remaining ones are associated with the interaction of the HTFP cell with the furnace. Both the furnace thermal inertia (probably the source of the so-called furnace effect) and the temperature profile are both likely to yield slightly different melting temperatures contributing to additional uncertainties of the order 0.1 K to a few 0.1 K. The latter may be optimised by ensuring the HTFP cell is placed in the region of most uniform temperature as an earlier study has shown[7].

An additional significant source of uncertainty would originate from the determination of the fit coefficients (exp. Sakuma-Hattori fit) when a radiation thermometer is calibrated in thermodynamic temperature using these dissemination artefacts. However this source of uncertainty can be made small provided the approach was restricted to the use of only narrow-band radiation thermometers (10-20 nm maximum) ensuring small residuals to the fit of temperature and photocurrent.

<u>Research undertaken - Dissemination using absolutely calibrated filter radiometers or pyrometers</u>

The second dissemination scheme was performed using radiation thermometers, calibrated in terms of thermodynamic temperature and a filter radiometer of known absolute spectral responsivity. Different methods were applied by the participants (PTB, CEM, MIKES and CNAM) to calibrate a transportable radiation thermometer or the filter radiometer for the measurement of the thermodynamic temperature.

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[7] Bourson, F., Briaudeau, S., Rougié, B. & Sadli, M. "Determination of the furnace effect of two high-temperature furnaces on metal-carbon eutectic points". AIP Conference Proceedings **1552**, 380-385 (2013).

- PTB calibrated an IKE LP3 pyrometer in terms of thermodynamic temperature by comparison to a filter radiometer via a large area high-temperature blackbody. This method is well established but it requires good temperature uniformity across the blackbody aperture to achieve a low uncertainty.

- MIKES used a filter radiometer (FR800) which consists of a single Si photodiode, an interference filter with nominal central wavelength of 800 nm, and a 4-mm precision aperture. The relative and absolute responsivity of the diode and filter were measured with a reference spectrometer of Aalto University[8], by comparing the output of the detector with a trap detector traceable to the cryogenic radiometer of SP, Sweden. This filter radiometer showed a large drift between the calibrations performed before and after the comparison which resulted in large uncertainties at the level of 0.4 %.

- CEM calibrated an IKE LP4 pyrometer using their HTFP cells of Cu, Co-C, Pt-C and Re-C as references for thermodynamic temperatures. The thermodynamic temperatures assigned to these HTFPs were the ones assigned to these HTFPs in the first objective of the InK project[4]. A Sakuma-Hattori fit was determined to derive the relation between the photocurrent delivered by the radiation thermometer and the thermodynamic temperature over the whole range (1000 °C to 2500 °C).

- CNAM applied a similar scheme as CEM, which involved the calibration of the CNAM LP3 pyrometer, except that the HTFP cells used were directly measured in terms of thermodynamic temperature using the CNAM radiance method put in application in the first objective of InK[4]. The cells used for the calibration of CNAM LP3 pyrometer had the local assigned thermodynamic temperatures. A Sakuma-Hattori fit was determined using these four temperatures to provide a relation between photocurrent and thermodynamic temperature.

Table 3.3 summarises the instruments which participated to this dissemination trial. The four instruments were gathered at PTB and put, alternatively, in front of a high-emissivity high-temperature furnace, the VNIIOFI-made HTBB 3200PG. The furnace temperatures were varied in steps of 200 °C between 1000 °C and 2500 °C.

CEM	PTB
Radiation thermometer LP4 (650 nm)	Radiation thermometer LP3 (658 nm)
Calibrated against 4 HTFPs Cu, Co-C, Pt-C, Re-C + Sakuma-Hattori fit	Calibrated with respect to thermodynamic temperatures against filter radiometer via HTBB furnace

CNAM	MIKES
Radiation thermometer LP3 (850 nm)	Filter radiometer (800 nm)
Calibrated against 4 HTFP with T values assigned at LNE-Cnam + Sakuma-Hattori fit	Absolute spectral responsivity

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[8] F. Manoochehri and E Ikonen: High-accuracy spectrometer for measurement of regular spectral transmittance, Appl. Opt. **34**, 3686-3692 (1995).

Table 3.3 Instruments calibrated in terms of thermodynamic temperature at the participants laboratories and compared at PTB

The uncertainties and temperature differences between the participating instruments can be seen in figure 3.4 below. The overall agreement seems to lie within 2 K at the highest temperatures.
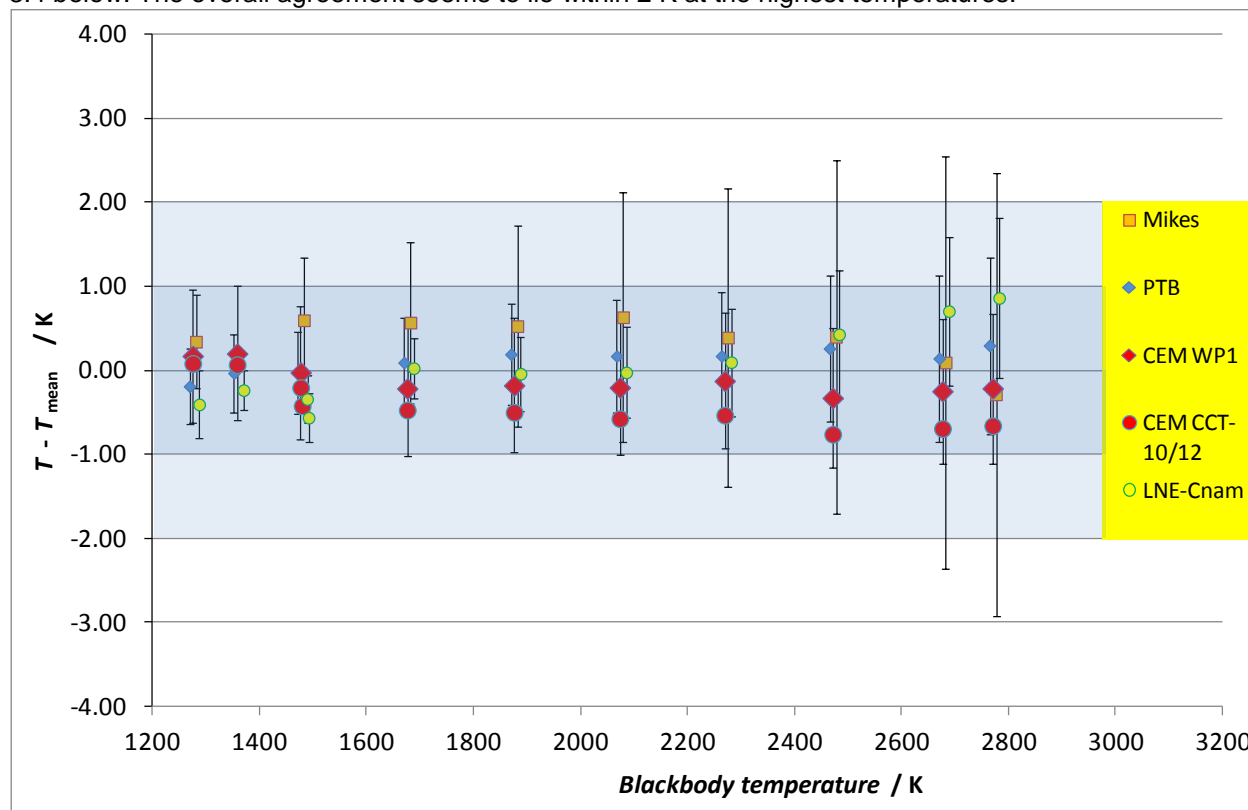

Figure 3.4: Results of the comparison of absolutely calibrated instruments. CEM radiation thermometer LP4 was calibrated using HTFPs and different reported temperature values for the HTFPs are given here: either thermodynamic temperatures according to EMRP InK or ITS-90 temperatures (given in[9]).

It should be noticed that the stability of the radiation thermometers used in this study was considered as satisfactory (i.e. drift within 0.5 K). This was not the case for the filter radiometer which has shown temperature equivalent drifts of 0.4 K to 1.8 K over the range 1000 °C – 2500 °C. This drift was probably due to the repair of the instrument after an open circuit was detected upon arrival at PTB.

Therefore the stability and fragility of the instruments is probably the main drawback of this method. It is to be doubted that if the radiometers/radiation thermometers had been circulated as much by post as had been the HTFPs whether the drift would have been as small as was observed. Also in the case of the irradiance mode being used (ie. with a straightforward filter radiometer) the method requires a uniform and high-emissivity furnace for the transfer to a radiation thermometer. In the case of poor temperature uniformity, the corrections and related uncertainties would have represented a large part of the final dissemination uncertainty.

Key research outputs and conclusions

To summarise, the results of this study have shown that both schemes can, with care, be applied successfully with comparable achievable uncertainties. However, the advantage of the HTFP dissemination scheme is the stability of the HTFPs which are thought to be much more achievable than the stability of radiation thermometers or filter radiometers. On the other hand, using radiation thermometers or filter

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[9] G. Machin et al. "Realisation and dissemination of thermodynamic temperature above the silver point (1234.93 K)" CCT document 10/12 June 2010 http://www.bipm.org/cc/CCT/Allowed/25/D12r_MeP-HT_v8.pdf

radiometers as thermodynamic temperature dissemination means does not require high-temperature fixed points and specially adapted furnaces, which could be an advantage for some NMIs. However if the latter was used great care would have to be taken to allow for the possibility of unknown drifts in the output of the radiometer. If the latter case is followed then a HTFP should be used periodically to check the stability of the transfer radiometer.

On the whole, while the dissemination of thermodynamic temperature is more direct by a radiometer or absolute calibrated radiation thermometer its dissemination by HTFPs is more secure because they are effectively driftless artefacts.

The key research outputs are as follows;

- A comparison of high-temperature fixed points used as dissemination artefacts was successfully performed among five participating laboratories. This comparison proved that the use of this kind of artefacts is a suitable means for disseminating thermodynamic temperatures with an uncertainty below 1 K over the temperature range from 1324 °C and 2474 °C.

- For the first time, a comparison of 3 radiation thermometers calibrated in thermodynamic temperature and 1 filter radiometer calibrated in absolute spectral responsivity was organised among 4 participants to this project. The overall agreement between instruments was remarkably good and the discrepancy in the determined temperatures in front of a high-temperature black-body were all with 1.5 K over a temperature range from 1000 °C to 2500 °C.

These results were published in the following high impact paper: Sadli, M., Machin, G., Anhalt, K., Bourson, F., Briaudeau, S., del Campo, D., Diril, A., Lowe, D., Mantilla Amor, J. M., Martin, J.M., McEvoy, H., Ojanen, M., Pehlivan, O., Rougié, B., Salim S. G. R.., "Realisation and dissemination of thermodynamic temperatures above the silver point", Phil. Trans R. Soc. A. **374**: 20150043 (2016) http://dx.doi.org/10.1098/rsta.2015.0043

Objective 3 - Determination of ($T-T_{90}$) with ultra-low uncertainties

Introduction

The purpose of this research is to develop a reliable set of T-T_{90} data for inclusion in the annex of the *MeP*-K and, in the longer term, if required, to form the backbone of any future temperature scale.

This work built on participants developed whilst performing extremely precise experiments for the determination of the Boltzmann constant with different methods and techniques. These experiments required complex apparatus designed for achieving the most accurate performance working at a single specific temperature, i.e. the triple point of water T_w = 273.16 K.

Design, implementation and testing of several significant modifications to the pre-existing experimental facilities to extend their capabilities to work over a temperature interval, rather than at a single reference temperature, without significantly reducing the measurement accuracy, was the main initial activity of the project partners, as described in section 3.1. The determination of the differences (T-T_{90}) with these new measurement capabilities continued throughout the duration of the project, finally providing a new set of results spanning the overall temperature range between 29 K and 303 K, and a revised set of results between 2.5 K and 36 K. These results and their relevance in view of the unprecedented low uncertainty achieved, which makes them, collectively, the most accurate temperature measurements ever reported, are described and discussed in section 3.2. The research effort towards the further extension of the working range of primary thermometers beyond that previously explored by each method is described in section 3.3. This has been a complex technical area which continued throughout the duration of the project, in parallel with the acquisition and the analysis of measurement data. The new measurement capabilities finally span the range between 1 K and 1350 K, i.e. beyond the initial (1 K to 1000 K) target. However because the technical challenges were greater than originally envisaged differently determinations of (T-T_{90}) over this wider operating range could not be achieved by the project deadline, these will be undertaken in the InK-2 follow on project.

Research undertaken - Preparation of experimental capabilities for primary thermometry

These activities were:

- The construction and assembly, or the modification of basic apparatus or artefacts, as are the newly-designed acoustic and/or microwave resonant cavities used to implement acoustic gas thermometry (AGT) at INRiM, LNE-CNAM and UVa. Namely: i) the assembly of a 3-litre volume spherical copper resonator (Fig. 3.5) used at INRiM for determinations of (T-T_{90}) between 234 K and 303 K[10]; ii) the assembly of a 3-litre volume quasi-spherical copper resonator used at LNE-CNAM for determinations of (T-T_{90}) between 220 K and 290 K[10]; iii) the preparation of an AGT apparatus based on a small volume stainless steel spherical acoustic resonator for AGT at UVa[11].

- The newly-designed construction, or the upgrade of several thermostatic systems, including: i) successive modifications of a NPL isothermal cryostat to minimize thermal gradients and extend the working range of their AGT apparatus to progressively lower temperature ranges down to 120 K[12]; ii) at LNE-CNAM, the upgrade and repair of an adiabatic cryostat for AGT measurements in the working range between ambient temperature and 77 K and the novel design and construction of a pulsed-tube cryostat for AGT measurements down to 4 K (Fig. 3.5); iii) the realization at NIM of a double thermostat working in the range between 250 K and 420 K to implement a RIGT experiment (Fig. 3.6).
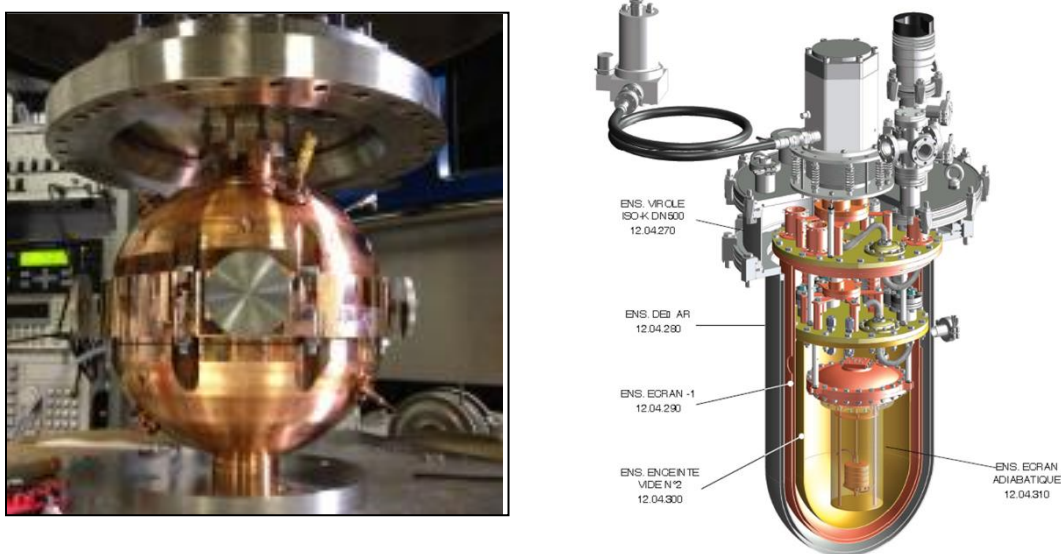

Figure 3.5 (Left) Assembled 3-litre volume copper spherical resonator used at INRiM for AGT determinations of the thermodynamic temperature between 234 K and 303 K. (Right) Sketch of a pulse-tube cryostat designed at LNE-CNAM for an AGT experiment working between 4 K and 290 K.

[10] Gavioso, R.M., Ripa, D.M., Steur, P.P.M., Gaiser, C., Zandt, T., Fellmuth, B., de Podesta, M., Underwood, R., Sutton, G., Pitre, L., Sparasci, F., Risegari, L., Gianfrani, L., Castrillo, A., Machin, G., "Progress towards the determination of the thermodynamic temperature with ultra-low uncertainty", Phil. Trans R. Soc. A. **374**: 20150046 (2016) http://dx.doi.org/10.1098/rsta.2015.0046
[11] F. J. Pérez-Sanz, J. J. Segovia, M. C. Martín, M. A. Villamañán, D. del Campo, C. García "Progress towards an acoustic determination of the Boltzmann constant at CEM-UVa" *Metrologia*, **52**, S257-S262 (2015)
[12] R. J. Underwood, M. de Podesta, G. Sutton, L. Stanger, R. Rusby, P. Harris, P. Morantz, G. Machin "Estimates of the difference between thermodynamic temperature and the ITS-90 in the range 118 K to 303 K" *Phil. Trans. Roy. Soc. A* (submitted 2015)

Figure 3.6 Experimental apparatus realized at NIM for implementing a RIGT between 250 K and 420 K.

- The characterization of the thermoelastic properties of materials and artefacts used in the construction of primary thermometers. Particularly, an accurate determination of the compressibility of the capacitors used to implement dielectric constant gas thermometry (DCGT) and of the compressibility of microwave resonators used to implement RIGT are crucial pre-requisites which may set the limit of accuracy of these techniques. An additional difficulty was represented by the need to determine the variation of the compressibility as a function of temperature over the entire working range spanned by these primary thermometers. This issue required a dedicated experimental effort, based on adaptations of a resonant ultrasound spectroscopy (RUS) technique both at PTB, where a successful reduction of the uncertainty of the effective compressibility k_{eff} of the material used for the construction of the capacitors (CuBe) was obtained (Fig. 3.7) to extend the measurement capabilities of DCGT above 80 K.
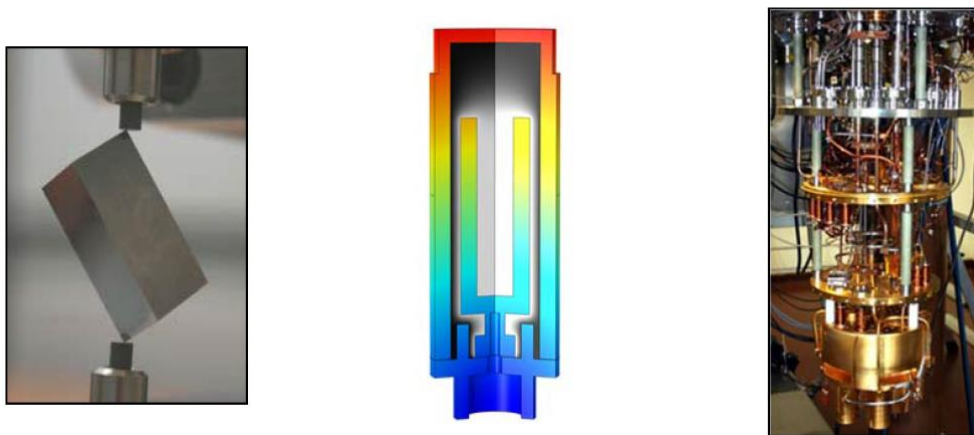

Figure 3.7 (Left) Resonant ultrasound spectroscopy was used at PTB to estimate the temperature dependence of the bulk modulus of a newly-designed cylindrical CuBe capacitor (central sketch) and (right) cryostat used for DCGT measurements between 2 K and 300 K.

- Improvements of the accuracy by which relevant state variables and experimental quantities can be determined. Among these are: i) the remarkable improvements achieved by PTB in the measurement of the experimental pressure[13] for DCGT; ii) several improvements of the main

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[13] T. Zandt, W. Sabuga, C. Gaiser, B. Fellmuth "Measurement of pressures up to 7 MPa applying pressure balances for dielectric-constant gas thermometry" *Metrologia*, **52**, S305-S313, (2015)

electronic components used in the realization of Johnson Noise Thermometry (JNT) as achieved by the cooperation between NIST and NIM[14].

- The design and the construction of dedicated instrumentation and tools to extend over a wider temperature range the measurement capabilities of particular techniques, as for instance: i) the high temperature microwave probes (Fig. 3.8) designed and tested by Tubitak-UME[15] for use up to 830 K in the AGT experiment being developed at INRiM; ii) the high temperature microwave and acoustic waveguides developed in cooperation by NIST and NIM, successfully tested respectively up to 1350 K and 800 K[16].
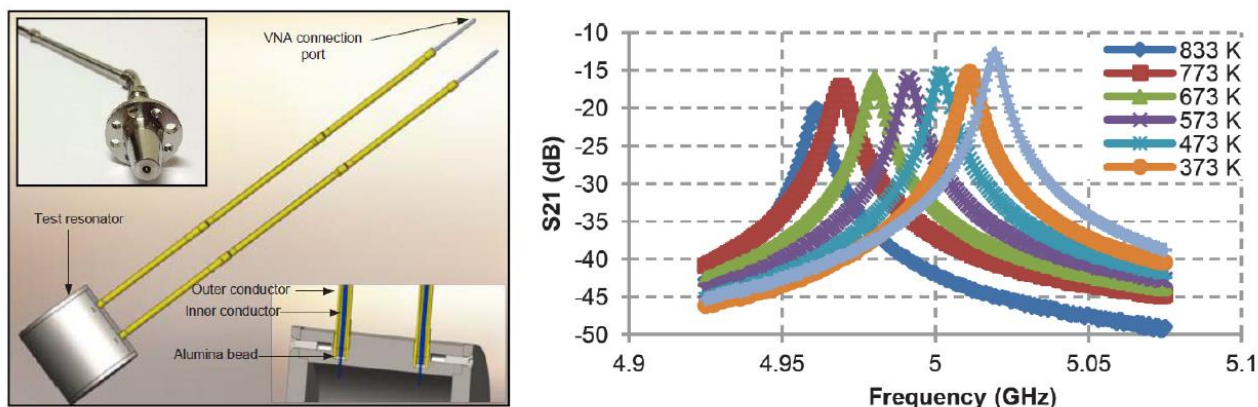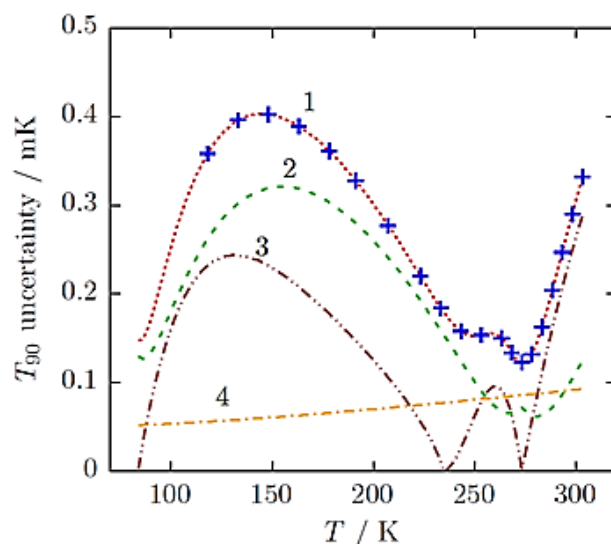

Figure 3.8 (Left) Sketch of the custom high-temperature (800 K) microwave waveguides designed and realized at Tubitak-UME. The inset picture shows a bent version of the waveguides as custom designed for high-temperature AGT developed at INRiM. (Right) TE111 mode measured up to 833 K at Tubitak-UME in a stainless steel cylindrical cavity.

- The fixed-point calibration of long-stem or capsule-type standard platinum resistance thermometers (SPRT) used in the primary thermometry experiments for the sake of the later comparison of the determined thermodynamic temperature T with temperatures on the International Temperature Scale of 1990 (ITS-90). As accomplished at NPL, this part of the work also included a careful, updated assessment of the inherent uncertainty associated with the determination of T_{90} (Fig. 3.9);

Figure 3.9 Estimated standard uncertainty in NPL measurements of T_{90}. Curves are: 1. Total uncertainty; 2. SPRT calibration uncertainty; 3. Non-uniqueness; 4. SPRT drift and resistance measurement.

Research undertaken - Thermodynamic temperature measurements

To reduce the influence of those uncertainty contributions which do not vary with temperature, most of the primary thermometric methods are best operated in relative mode, which implies repeated measurements at the temperature of interest T compared to some reference temperature T_{ref}, with the latter typically chosen to be the temperature of the triple point of water T_w. Many of the primary thermometers developed in the initial course of this project were modifications of those used to determine the Boltzmann constant but then modified to determine T-T_{90} by relative means, the results are summarised in[10]. At this stage of the project, cooperative work among partners was developed to assess the composition of the monoatomic gas samples used in the absolute experiments being conducted at T_w, including: i) the characterization of the isotopic composition of commercial argon samples used at NPL, LNE-CNAM and NIM, with a mostly relevant contribution from the Korean National Metrological Institute (KRISS) who made available to the project its experimental facilities and expertise in mass-spectrometry; ii) the determination, using the mass spectroscopy facilities available at PTB of helium samples being used at INRiM.

Following these initial achievements and the completion of the preparative work described in section 3.1, the primary thermometers developed at various laboratories were operated to cover increasingly wide temperature ranges finally leading, by the end of the project, to the determinations of ($T - T_{90}$) summarized in Figure 3.10 These include, namely, a subset of DCGT measurements obtained at PTB in the temperature range between 29 K and 140 K, and three subsets of AGT measurements respectively obtained at NPL, LNE-CNAM, INRIM, in the overall temperature range between 78 K and 303 K.
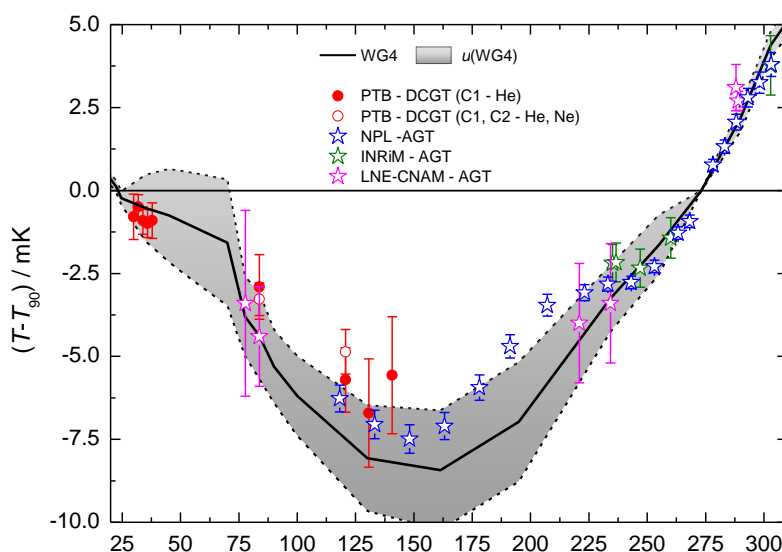

Figure 3.10 Determinations of the differences ($T - T_{90}$) achieved at various laboratories with two different primary thermometry methods in the course of this project. These are, namely, DCGT at PTB, and by AGT at NPL, LNE-CNAM and INRiM. The black line and the shaded area display the best fit and the uncertainty of an interpolation by CCT Working Group 4 of ($T - T_{90}$) data which were available before the start of the InK project.

Details of the notable features of each of these subsets are listed below:

- Figure 3.11 displays the determinations of ($T - T_{90}$) obtained at PTB using two different capacitors and two different test gases (helium and neon). The redundancy of this careful procedure, which includes repeated isotherms at 84 K and 121 K, and the mutual consistency of the results, significantly reduces the possible influence of several gas-dependent and apparatus-dependent systematic sources of uncertainty. Remarkably, the new DCGT data demonstrate that the useful operating range of the DCGT method can be extended to higher temperatures than previously reported, enhancing the significance of the comparison with AGT ($T - T_{90}$) data obtained from other laboratories in the overlapping temperature range. Finally, the new determinations of the effective

compressibility of the capacitors as a function of temperature lead to a correction of previously published results in a lower cryogenic temperature range down to 2.5 K and 36 K.
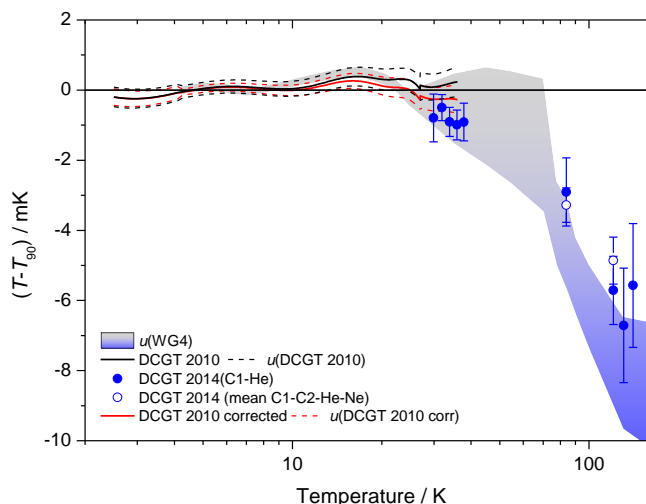

Figure 3.11 Determinations of the differences ($T - T_{90}$) achieved by DCGT at PTB between 29 K and 140 K (hollow and filled circles) using two different capacitors (C1, C2) and two different test gases (He, Ne). The new determinations of the effective compressibility of the capacitors provide a correction for previously published results between 2.5 K and 36 K (continuous lines).

- The AGT apparatus used at NPL was operated, in relative mode, to measure the speed of sound in Ar as a function of pressure along several (20) isotherms, including $T_{\rm w}$, between 118 K and 303 K, leading to a closely-spaced set of ($T-T_{90}$) determinations[12]. Most notably, these determinations are characterized by unprecedented low uncertainties, improving the state of the art of primary thermometry with any method in this temperature range. In fact, the uncertainty associated to the thermodynamic temperature T resulted sensibly lower than the inherent uncertainty of best-practice T_{90} measurements using standard platinum resistance thermometers. The extremely high precision and accuracy of the dense NPL thermometry dataset also reveals some subtle, previously unnoticed features of the differences ($T - T_{90}$), which are likely to be inherent in the definition of ITS-90. Finally, the analysis of NPL data close to $T_{\rm TPW}$, sheds new light on the previously discussed discontinuity of the slope d($T - T_{90}$)/dT_{90} appears to be slightly negative or possibly zero rather than positive, as previously reported.

- The determinations of ($T-T_{90}$) by AGT at LNE-CNAM were reported at six temperatures between 77 K and 290 K and were obtained by measurements of the speed of sound in argon using three different resonators, including a large volume (3-litre) copper quasi-spherical cavity which had been carefully characterized to achieve an extremely precise determination of $k_{\rm B}$. The values and the rather large uncertainties displayed by these results in Figure 3.10 should be considered provisional and are likely to be significantly reduced when the calibration of the standard thermometers used in the experiments will become available.

- The AGT apparatus, previously used at INRiM to achieve a precise determination of $k_{\rm B}$, without any relevant modifications, was operated in the temperature range between 235 K and 303 K, leading to the measurements of ($T - T_{90}$) displayed in Figure 3.10 using helium as the test gas.

The comparison of ($T-T_{90}$) results obtained by different laboratories is the founding value and the main motivation of joint research as it was addressed in the original organization of this project. Such a comparison shows that the results displayed in Figure 3.10 are consistent with each other over the intervals where the temperature ranges covered by various experiments overlap. Particularly, the agreement of various AGT data at the temperature of the melting point of gallium $T_{\rm Ga}$ is indeed remarkable, considered that these data were obtained with weakly correlated experiments and motivates the further future development of AGT at a higher temperature range. Below 100 K, the new DCGT results of PTB are in satisfactory agreement with the new and previous AGT results of LNE-CNAM. A similarly satisfactory agreement of new DCGT results is found between 120 K and 140 K with the new extremely accurate AGT

results obtained at NPL. This satisfactory overall agreement for data obtained by four different laboratories with two different experimental methods indicates that the possible influence of unknown or undetected systematic effects was not underestimated.

As a whole, the new thermodynamic temperature determinations are found to be mostly consistent with the CCT-WG4 interpolation of (T-T_{90}) data which were available before the start of the project. A minor exception is represented by the temperature interval between 175 K and 225 K where the accuracy of the NPL dataset indicates that the WG4 interpolating function slightly underestimates the difference (T-T_{90}).

<u>Research undertaken - Further extension of experimental capabilities for primary thermometry</u>

In addition to the results described in the previous section, various laboratories continued the development of apparatus and facilities to extend the working temperature range of their primary thermometry methods throughout the course of this project. Among the notable achievements are:

- The design and realization at NPL of an apparatus for AGT primary thermometry up to 1000 K (Fig. 3.12). The AGT is based on an aluminium alloy resonator of cylindrical shape inserted within an Inconel vessel capable of withstanding a maximum pressure of 1 MPa. The vessel is equipped with thermometer wells to allow the quick insertion and removal of long-stem SPRTs. The high temperature furnaces is a custom modified fan circulating oven. Preliminary tests up to 100 °C demonstrated the possibility to achieve an accurate determination of the thermal expansion of the cylinder using microwave measurements[17].
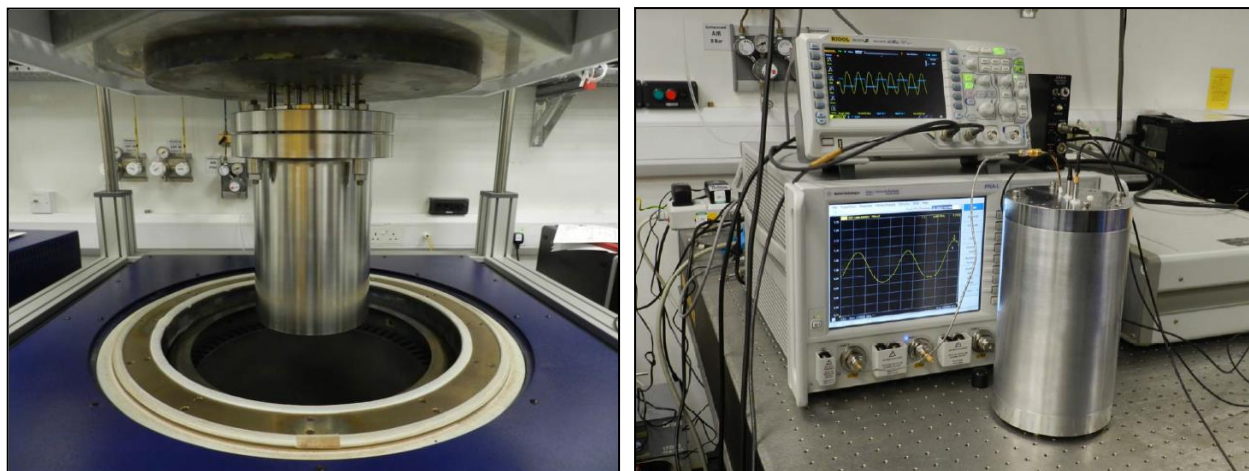

Figure 3.12 (Left) The NPL cylindrical resonator inside its pressure vessel shown above the recirculating air bath which will heat it to temperatures between 30 °C and 730 °C. (Right) The NPL cylindrical resonator undergoing microwave and acoustic tests.

- The design and realization at INRiM of an apparatus for AGT primary thermometry up to 1000 K as sketched in Fig. 3.13. This AGT is based on a spherical stainless steel resonator with an internal radius of 8 cm equipped with suitable acoustic and microwave waveguides (Fig. 3.8). The furnace comprises an external insulating vacuum vessel, infrared heated isothermal copper shields, an internal cylindrical pressure vessel designed to withstand a maximum pressure of 1 MPa at the highest working temperature.

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[17] R. J. Underwood, G. J. Edwards "Microwave-Dimensional Measurements of Cylindrical Resonators for Primary Acoustic Thermometry" *Int. J. Thermophys.* **35**, 971–984 (2014)
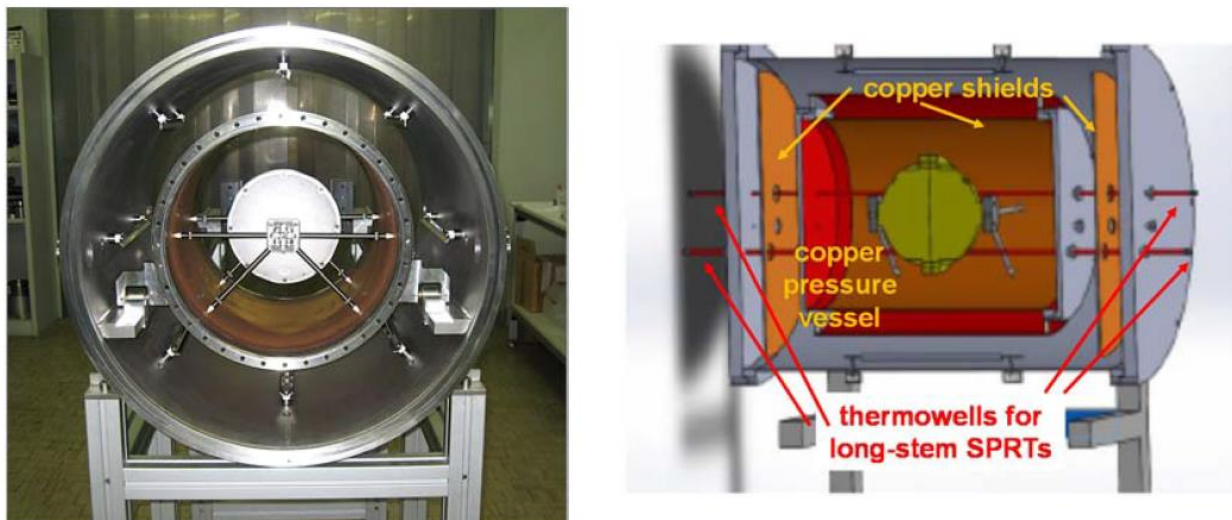
Figure 3.13 High-temperature apparatus designed and realized at INRiM for AGT measurements up to 1000 K.

- the realization at LNE-CNAM of a miniature copper cavity and custom instrumentation, including a cryogenic valve entirely realized in copper and a shielded cryogenic current amplifier for the realization of an AGT working below 4 K (Fig. 3.14);

Figure 3.14 (Left) Miniature copper acoustic resonator designed and realized for AGT measurements below 4 K at LNE-CNAM with accessories: (Centre) cryogenic valve entirely realized in copper; (Right) cryogenic current comparator amplifier for enhancing the S/N ratio of acoustic signals at low pressure.

Key research outputs and conclusions

This workpackage has significantly progressed the stat of the art of primary thermometry by acoustic thermometry, dielectric constant gas thermometry and Johnson noise thermometry. These facilities and techniques will be used in the successor InK 2 project to perform a complete assessment of T-T_{90}.

The results already taken are impressive having the world's lowest ever uncertainties and identifying previously unknown features in the T-T_{90} consensus curve.

These results are summarised in the paper by Gavioso et al Phil Trans A 374, which is in the proceedings of the Royal Society Workshop held to discuss progress "towards the new kelvin".

The achievements are summarised as follows:

o (T-T_{90}) determined by DCGT at PTB with unprecedented low (first in the world) uncertainty between 29 K and 140 K;

o (T-T_{90}) determined by AGT at NPL with unprecedented low (first in the world) uncertainty between 118 K and 303 K;

o Absolute primary thermometry at T_w (k_B determination) by JNT at NIM/NIST with unprecedented low (first in the world) uncertainty;

o New measurement capability (first in the world) for AGT up to 1350 K demonstrated at NIM/NIST;

o New measurement capability (first in the world) for AGT down to 1 K realized at LNE-CNAM;

o New measurement capabilities for high-temperature AGT up to 1000 K realized at NPL, INRiM/Tubitak-UME (first in Europe);

o (T-T_{90}) determined by AGT down to 77 K at LNE-CNAM (first in Europe);

o (T-T_{90}) determined by AGT between 234 K and 303 K at INRiM;

Objective 4 - Develop primary methods of realising and disseminating T below 1 K and resolve the long standing discrepancy in the low temperature part of PLTS-2000

Introduction: Primary thermometry for low temperatures

The aim of this objective was to develop and improve primary thermometers for measurement of thermodynamic temperature in the cryogenic low-temperature range below 1 K with low uncertainties.

The activities of this objective aimed at:

• Building and testing several novel designs of primary thermometers for the direct realisation of the kelvin to simplify the dissemination of the kelvin and for practical thermometry at temperatures below 1 K.

• Resolving the long-standing discrepancy between the background data of the Provisional Low Temperature Scale 2000 (PLTS-2000) through low-uncertainty primary thermometry.

Reliable and traceable thermometry is still a demanding task at low temperatures even though a great variety of thermometers is available for temperature measurements. Most kinds of those thermometers exhibit very non-linear characteristics restricting the useful range of application. For thermometry with sufficient resolution and uncertainty the application of different thermometers in parallel is necessary to cover the working range of widely used dilution refrigerators. Especially in the millikelvin region problems arise from the dissipation of heat by the measurement process in the thermometer and the growing thermal resistances between the sensing element and the object, the temperature of which is to be measured. In order to prevent overheating of the temperature sensors, the excitation power of the measurement has to be kept very small (below nanowatts or even picowatts). In addition, the devices may suffer from instabilities and high sensitivity to external influences as thermal cycling during use, electromagnetic fields and others. Therefore, relative uncertainties of temperature measurement of the order of 1% and lower are challenging when going down in temperature below 1 K. In the range below 1 K, the dissemination of the kelvin is usually realised using practical thermometers and superconductive reference points. The calibration of these thermometers requires the expensive and time-consuming realisation of the PLTS-2000 traceable to SI units. For an individual practical thermometer the expenses for a calibration are at the same level or even higher than the costs of the thermometer itself. In addition, after reasonable time a recalibration is advisable to eliminate long-term drifts.

Research undertaken – Development of new primary thermometers

Three types of primary thermometers were developed, which are based on different working principles allowing the identification of systematic effects in thermodynamic temperature measurement. The thermometers developed are the Current Sensing Noise Thermometer (CSNT), the Coulomb Blockade

Thermometer (CBT) and the primary Magnetic Field Fluctuation Thermometer (pMFFT). Even though first implementations of these thermometers have been made several years ago significant improvements were made during the project.
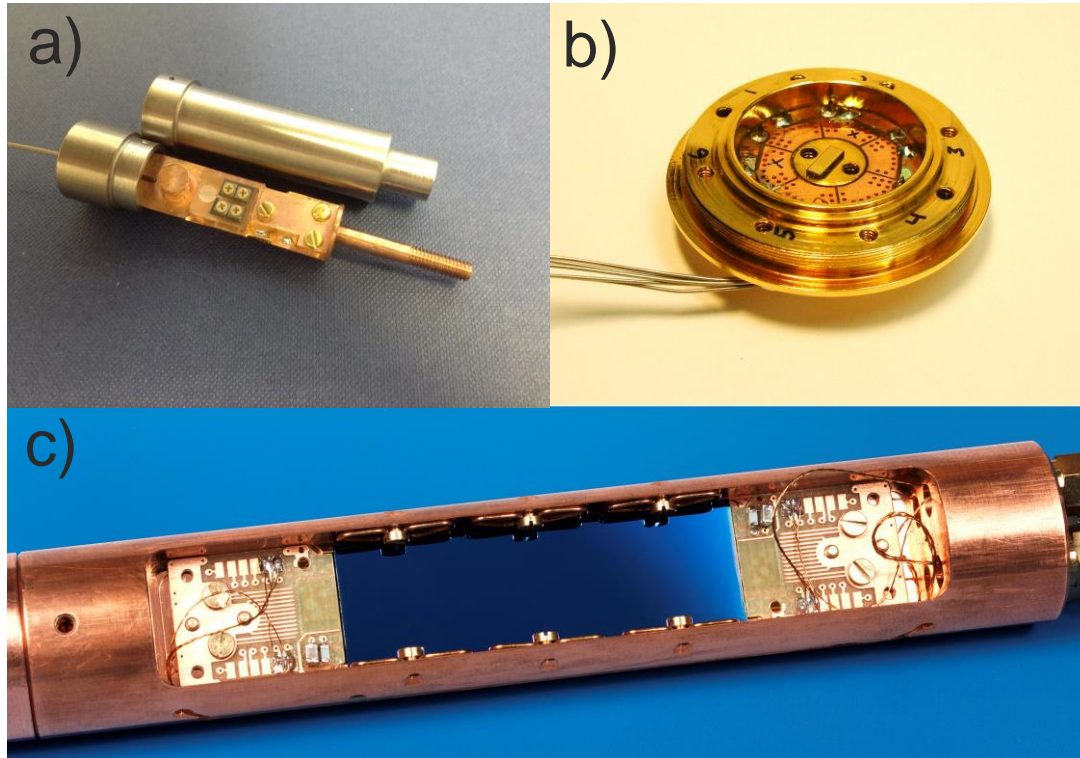

Figure 3.15 Pictures of the noise sensor of the current sensing noise thermometer (a), the Coulomb blockade thermometer (b) and the primary magnetic field fluctuation thermometer (c).

The CSNT and MFFT are two different variants of noise thermometers, which rely on a fundamental law of physics, the so-called Nyquist relation connecting the voltage fluctuations of thermally excited electrons in a (noise) resistor to absolute temperature. Such noise thermometers have found application at high as well as low temperatures. But the lower the temperature is the lower are the noise signals, which finally become so tiny that special equipment is necessary to measure them.

To overcome this limitation for the application at low temperatures, dc Superconducting Quantum Interference Devices (SQUIDs) are used as the basic component in the measurement chain of CSNT and pMFFT. The SQUIDs are also located at low temperature and their intrinsic noise properties determine the performance of the whole thermometer set-up. SQUIDs show intrinsic frequency dependent noise contributions other than Nyquist noise. If SQUIDs are used for noise thermometry, this excess noise must be much lower than the measured Nyquist noise, from which the temperature information is obtained. One main parasitic noise source of the SQUID itself comes from the shunt resistors necessary for a non-hysteretic operation of the SQUID. At the first stage of the project, PTB has developed a novel design for the main SQUID components, i.e. a new layout for the shunt resistors, the cooling fins and the stripline connections between SQUID and distant shunt resistors (see figure 3.16)[18].

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[18] Drung, D. Et al. IEEE Transactions on Applied Superconductivity **21**, Issue: 3, 340-344 (2011)
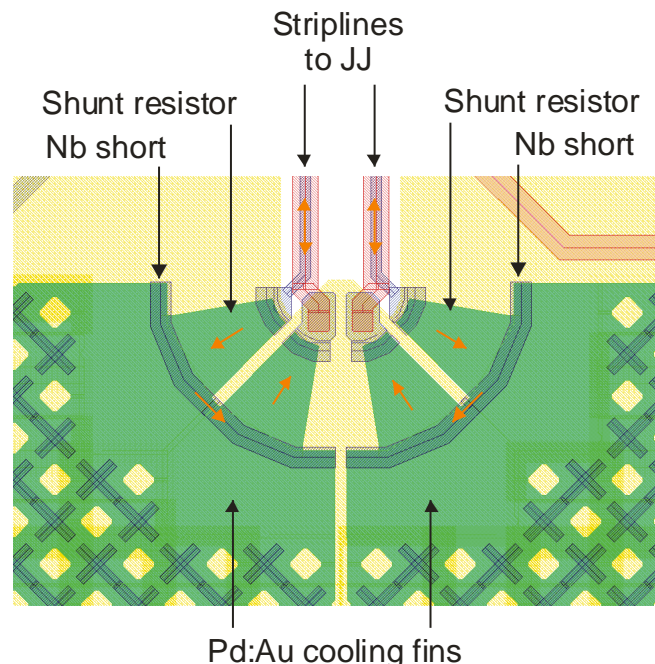
Figure 3.16 New SQUID design with layout of the shunt resistors and the cooling fins.

The new design provides better cooling of the shunt resistors by an enlarged volume of the resistor material to prevent overheating of the electrons and enlarged cross sections for enhanced heat conduction to reduce temperature gradients. At RHUL, the noise characteristics of the new PTB SQUIDs were investigated in a broad temperature range from 1.7 K down to 13 mK. It appeared that the noise level at high frequencies decreased with decreasing temperature down to about 300 mK, whereas at low frequencies the noise level increased with decreasing temperature. In the frequency range important for operating the CSNT a significant reduction of the parasitic noise contributions from the shunt resistors was achieved. At 1 Hz the negligible contribution of the SQUID noise to the temperature reading of the CSNT amounted to only 15 µK for the 2 mΩ thermometer and 1.5 µK for the 0.2 mΩ thermometer. The new SQUIDs were an essential precondition for the improvement of the CSNT and the development of the pMFFT noise thermometers. Such new SQUID sensors were fabricated and provided for the partner RHUL for use with the CSNT.

The development of the CSNT was first focused on the improvement of the design of the resistive noise sensors. The value of the noise sensor is a trade-off between the device noise temperature, which should be kept small, and the measurement time for a given precision, which should be small for reasonably fast measurements[19]. Higher resistance values make the thermometer faster but reduce the noise power, which must be much higher than the SQUID noise, and cause problems with thermalisation as the thermal conductivity is reduced and thermal gradients across the noise sensor increase. The choice of the noise resistor was determined by three intended applications: (i) primary thermometry from 4 K down to 5 mK (R_N = 1.29 Ω, material: PtW), (ii) primary thermometry in high magnetic fields (R_N = 0.14 Ω, material: PtW), and (iii) high-precision primary thermometry between 1 K and 0.9 mK (R_N = 0.2 mΩ, material: Cu foil)19[19]. For the corresponding CSNTs the noise spectra are shown in figure 3.17[19]. Special precautions were taken to minimize the heat leak to the resistor, which may result from conduction along the superconducting wires from the SQUID sensor operated at higher temperature. In cases (i) and (ii), where a 50 µm diameter PtW wire is hard soldered to two pieces of copper foil, one foil is electrically grounded and therefore heat sunk to a large copper holder with contact to the sample plate. The other foil is electrically isolated from the holder. In case (iii), the resistor is a copper foil, one end of which is electrically grounded to the holder using a copper clamp, while the other (floating) end was insulated by a thin layer of varnished cigarette paper and connected via a thin strip of niobium foil, heat sunk to the holder by varnished cigarette paper. In addition, the superconducting twisted pair of wire (NbTi without cladding) from the SQUID sensor is heat sunk at a post extruded from the copper holder before connecting the resistor. One advantage of the split CSNT design, where the resistor is separated from the SQUID sensor is that the latter can be located and operated at the optimum temperature, where the energy sensivity of the SQUID is as low as possible and

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[19] Casey A et al. J. Low Temp. Phys. 175, 764-775 (2014)

consequently also the noise contribution from the SQUID. Cooling it to 300 mK (present at the cold plate of the dilution refrigerator) is sufficient because its effective temperature (shunt resistors of the SQUID) will not decrease further.

Results of temperature measurements with the CSNT, equipped with different noise resistors, are shown in figure 3.18. There is excellent agreement between the thermometers over a wide temperature range, and deviations from the reference temperature become visible below an individual lower temperature. The decoupling of the CSNT at the lowest temperatures can be described assuming a constant heat leak into the CSNT from higher temperature stages via the wiring. The CSNT with 1.29 Ω sensor is two orders of magnitude faster than previous noise thermometers, reaching 1.2% precision in 0.1 s. The CSNT with 0.14 Ω sensor is in agreement with the ^{3}He melting curve thermometer to within less than 1% over the operating temperature range of a dilution refrigerator obtained in less than 2 s. Reasonable agreement with a pulsed platinum NMR thermometer down to below 200 μK was reached by the CSNT with the 0.2 mΩ sensor, which takes about 200 s to obtain 1% precision.

For primary operation, the effective transresistance of the CSNT (including the SQUID electronics), which converts the current measured in the input coil to the SQUID output voltage, must be determined[20]. For this three quantities have been measured independently: (a) the resistance R_N, (b) the mutual inductance between SQUID and input coil and (c) the mutual inductance between SQUID and feedback coil. The current set-up for the CSNT used in the comparison measurements allows measurements of thermodynamic temperature with a relative combined standard uncertainty of 1.5%.
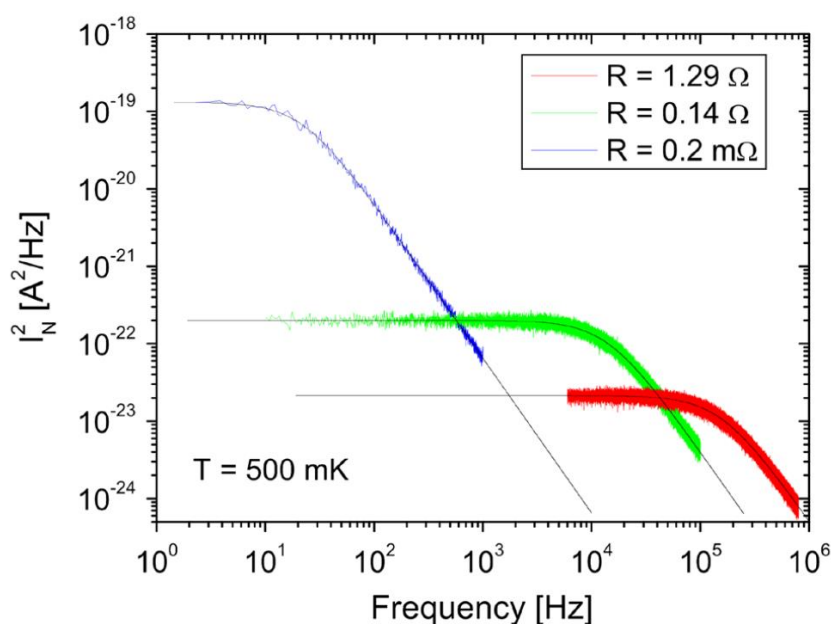

Figure 3.17 Current noise spectra at 500 mK for CSNTs with different noise resistors[19]. The solid lines are fits to the low-pass like shape of the power spectral density determined by the noise resistor value and the total inductance of the SQUID input circuit.

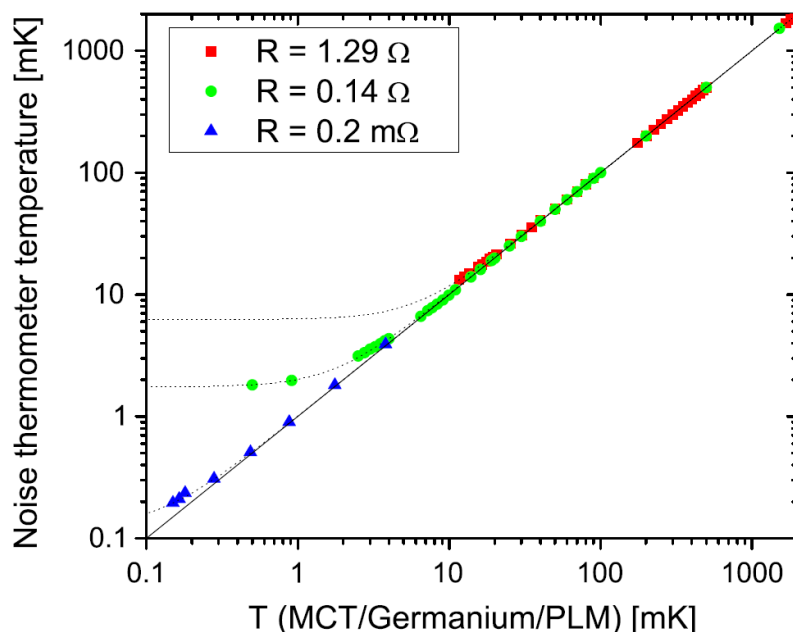[20] Shibahara A et al. Phil. Trans. Royal Soc. A. to appear in 2016

Figure 3.18 Noise thermometer temperatures measured using CSNTs with different noise resistors in comparison with reference temperatures provided by a [3]He melting curve thermometer, a Pt nuclear magnetic resonance thermometer and a calibrated germanium resistance thermometer[19]. The dotted lines model the thermal decoupling of the thermometers from the reference temperatures assuming a constant heat leak into the noise thermometer.

For the other noise thermometer developed in this project, the pMFFT[21], the essential advantage is the large volume of the bulk metallic temperature sensor, in which the electrons can interact (thermalize) with the phonons, i.e. with the lattice of the sensor's copper body. As a result, the hot electron effect that may occur at low temperatures and affect the measured (electronic) temperature is very small. The main disadvantage of the pMFFT, however, is a potentially lower thermal noise power coupled into the SQUID sensor, as the detection coils are at a certain distance above the surface of the temperature sensor and the noise power diminishes quickly with growing distance. The consequence is an effectively increased noise temperature.

The pMFFT design overcomes this problem by introducing the cross-correlation technique using two independent signal channels. The evaluated signal is the correlated thermal noise power in the two channels, where each channel may have individual, uncorrelated excess noise components, which do not contribute to the measured (correlated) signal. In the pMFFT, the readout scheme of the SQUID gradiometers, which detect the thermal noise, is the input-current lock (instead of the usual flux-locked loop). This is realized by applying the feedback into the SQUID input circuit (and not into the SQUID loop as is done in case of the usual flux-locked loop). This together with a special multi-gradiometric design of the detection coils decouples the two signal channels. Nevertheless, there is an almost complete correlation of the detected thermal noise. The second advantage of this feedback scheme is the virtual absence of interaction with the temperature sensor, which would otherwise affect the frequency behavior of the measured noise spectrum.

The new design of the pMFFT enables primary thermometry by making all relevant aspects of the pMFFT calculable. The design of temperature sensor and detection coils is made simple so that it can be mathematically easily modeled and all necessary geometrical parameters can be precisely determined. This means that all necessary calculations are based on analytically solvable models, and demanding FEM calculations are not required. The planar thin-film detection coils are defined by photolithography, having typical line widths of 2.5 µm. Their distance to the surface of the temperature sensor of about 100 µm is measured optically in the infrared by means of low-coherence interferometry.

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[21] Kirste A. Engert J. "A SQUID-based primary noise thermometer for low temperature metrology" Phil. Trans. Royal Soc. A. **374**:20150050 (2016) http://dx.doi.org/10.1098/rssta2015.0050

In order to calibrate the SQUID gradiometers including the SQUID electronics (in terms of output voltage per magnetic flux) a calibration coil is integrated into the MFFT. The according calibration is done using known dc or quasi-dc currents. The second purpose of the calibration coil is to enable an independent measurement of the electrical conductivity of the temperature sensor, which must be known for the pMFFT. This measurement is based on the strong frequency dependence of the magnetic field (flux) permeating the temperature sensor. Thus, the conductivity can be determined by applying known AC currents into the calibration coil. Finally, having determined all parameters for operating the pMFFT the relative combined standard uncertainty of temperature measurements was estimated to be 0.6%.
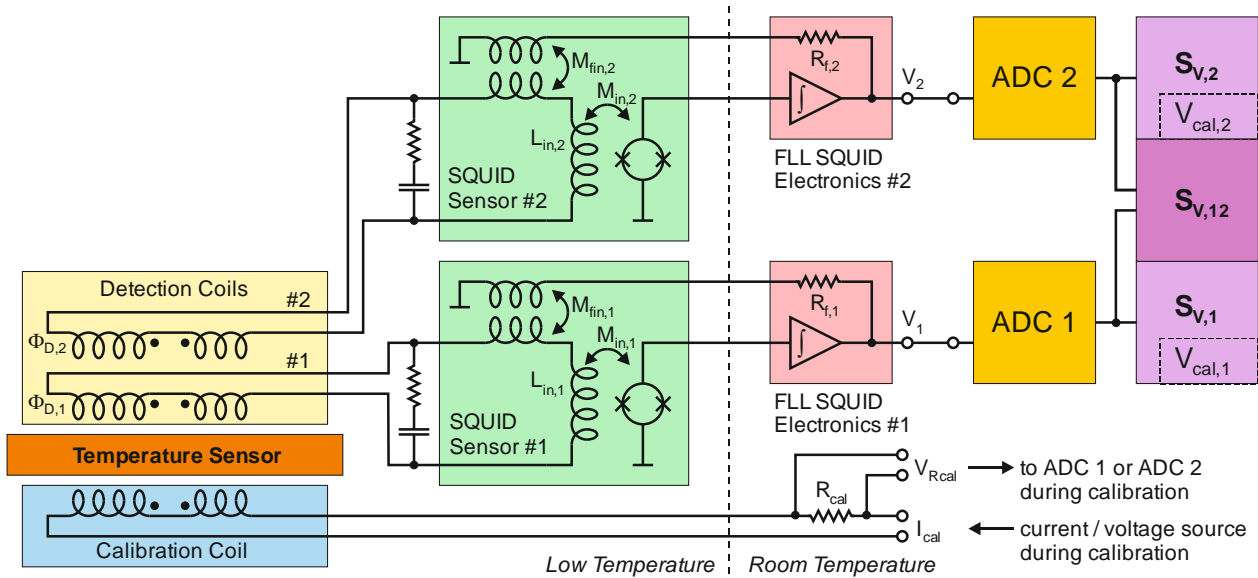

Figure 3.19 Block diagram of the primary MFFT with two independent signal channels to apply the cross- correlation technique. The calibration coil is used to calibrate both signal channels and to determine the electrical conductivity of the temperature sensor.

The Coulomb blockade thermometer developed and used in this project is a result of joint effort of VTT-MIKES and Aalto University Finland. In contrast to the noise thermometers, the CBT is based on a different physical phenomenon. It is based on single electron tunneling and relates the measured voltage width at half minimum of the normalized differential conductance dip around zero bias of an array of normal metal tunnel junctions to temperature by means of the Boltzmann constant k_B and the charge of the electron e. The typical working regime of the CBT, in which it can be operated as primary thermometer, is the weak Coulomb blockade regime $E_c << k_B T$, where $Ec \equiv ((N\text{-}1)/N)(e^2/C)$ is the charging energy of the system with N junctions in series and C is the total capacitance of an island between the junctions. Since this capacitance depends on the physical size of the contact and the self-capacitance of the island, the junctions can be tailored for a specific temperature range. Since the width of the conductance dip has a universal temperature dependence, sample parameters do not play a role, otherwise. If the condition of the weak Coulomb blockade regime is not (fully) met, universal corrections can be applied without affecting the primary nature of the CBT[22].

Extending the temperature range of the CBT to the lower end of the mK range means that the regime of the weak Coulomb blockade is left towards the intermediate Coulomb blockade, $E_c \sim k_B T$. Therefore, corrections to the half width and to the depth of the measured conductance dip were necessary and have been applied[23]. It was experimentally shown that corrections up to the third order in the series expansion of the half width and the normalized differential conductance are sufficient in the intermediate regime. If thermometer operation is limited to $k_B T/E_c > 0.4$, the error in temperature is less than 2.5% from either of the

[22] Feshenko A.V. et al. J. Low Temp. Phys. 173, 36-44 (2013)
[23] Prance J. R. et al. arXiv:1505.07244 [cond-mat.mes-hall]

opposite extremes (i.e. maximum or minimum blockade) when using the third-order approximation. Ultimately, conductance curves are precisely calculated at any ratio of $k_B T/E_c$ without approximations, allowing the inclusion of overheating effects of the tunneling electrons.

The CBT sensors used consist of twenty parallel arrays with 33 or 99 junctions in series. Overheating effects are relatively small down to 55 mK due to cooling fingers, which provide a good thermalisation between the array and the substrate. This effect and the influence of background charge distribution limit the accuracy of the CBT at low temperatures.

The fabrication scheme for the tunnel junctions was improved to significantly reduce the inhomogeneity in the parameters of the tunnel junctions in the array. On the one hand, this allows precisely operating CBTs above about 10 K. On the other hand, CBTs in the classical range between 50 mK and 1 K can take advantage of the same process with its high fabrication accuracy. The resulting variation of the total junction area will be negligible so that other effects (e.g. variations in the oxide layer thickness) become the dominating uncertainty component in the homogeneity of the array. In addition to optimized cooling fins for thermalisation of the electrons also a new filter technique with on-chip electronic filters was introduced to allow measurements in the low mK region [6]. Figure 3.20 shows a CBT mounted on the setup for comparison measurements in a cryogen-free dilution refrigerator. Here, the indium sealed double RF shield is removed to show the CBT chip. The connection to the room temperature electronics is realized via Thermocoax cables for additional filtering of parasitic disturbances. Finally, a relative uncertainty for temperature measurements of about 1% and less was achieved for the CBTs.
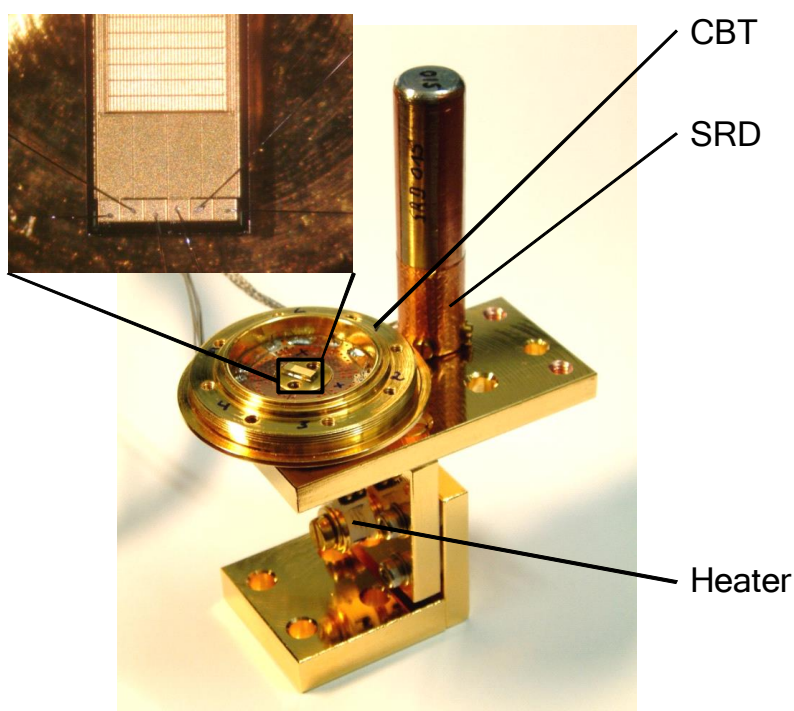

Figure 3.20 Experimental stage for comparison measurements between CBT, SRD and other thermometers. The CBT is open with the cover removed. The insert shows the chip with tunneling structures forming the CBT sensor.

Research undertaken – Low temperature measurements using new primary thermometers

The second part of activities of this objective was focused on the problems connected with the PLTS-2000. The PLTS-2000 is now nearly fifteen years old. The scale itself is based on data which are discrepant at the lowest temperatures by a factor 6 compared to their individual uncertainties. As such it was only meant to be a provisional arrangement to be superseded when more consistent reliable data was available. For the first time since 2000, in this project research was started to resolve this discrepancy in the background data. To

tackle the challenge four European low-temperature laboratories from national metrological institutes and universities jointly conducted comparison measurements using the primary thermometers developed during the first project stage.

The aim of these comparison measurements was twofold. Firstly, a common basis and reference for temperature measurements was necessary to ensure comparability and equality of the measurement results. To this end, a high-precision superconductive reference point device (SRD) was calibrated by the PTB and provided for the partners. The reference points of the SRD ensured the realisation of highly stable reference temperatures for the comparison measurements in the partner's laboratories and allowed a first direct comparison of temperature realisations by European low-temperature labs. The comparison measurements were carried out following a before agreed protocol. The results confirm, for the first time, that the transfer of high-precision temperature realisations between different laboratories is possible on a level of a relative uncertainty of few 10^{-4} in the low-temperature region below 1 K.

As an example, figure 3.21 shows the relative deviations of temperature measurements using the CSNT, from T_{2000} temperatures realised using the SRD. The agreement between the thermodynamic temperature determinations and T_{2000} is within 1% based on the combined standard uncertainties (coverage factor $k = 1$). Similar results were obtained for the CBT and the pMFFT. For all thermometers the target uncertainty of thermodynamic temperature determinations of about 1% and less was met.
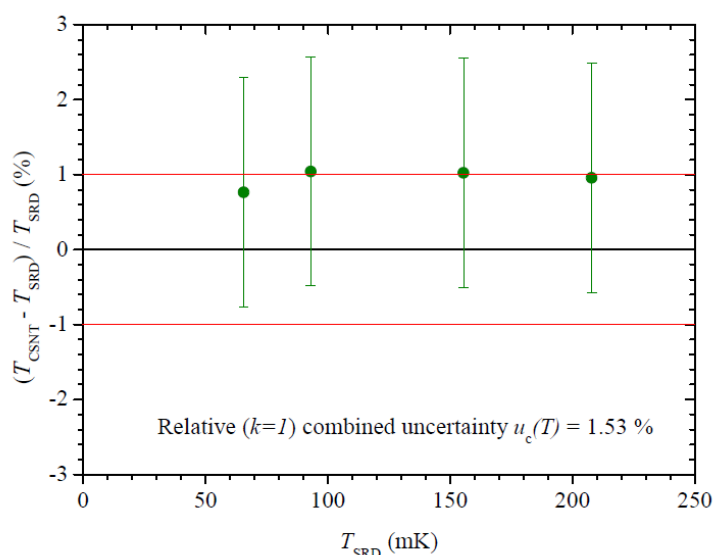

Figure 3.21 Relative deviations of temperature measurements with the CSNT from reference temperatures T_{2000} provided by a superconductive reference point device calibrated according to the PLTS-2000[20].

To highlight the progress achieved in investigating the background data discrepancy of the PLTS-2000 in more detail, figure 3.22 shows the relative deviations of thermodynamic temperatures measured with the pMFFT from the PLTS-2000. Also shown are the deviations of the background data, on which the PLTS-2000 is based (NIST, University of Florida and PTB[24]). Over a broad range of temperatures from 700 mK down to 15 mK the new data obtained within this project supports the PLTS-2000 within 1% as is marked in figure 3.22 by the light green band. The tendency of the pMFFT data to higher deviations from the PLTS-2000 at temperatures below 15 mK has to be investigated further by extending the experiments down to 1 mK.

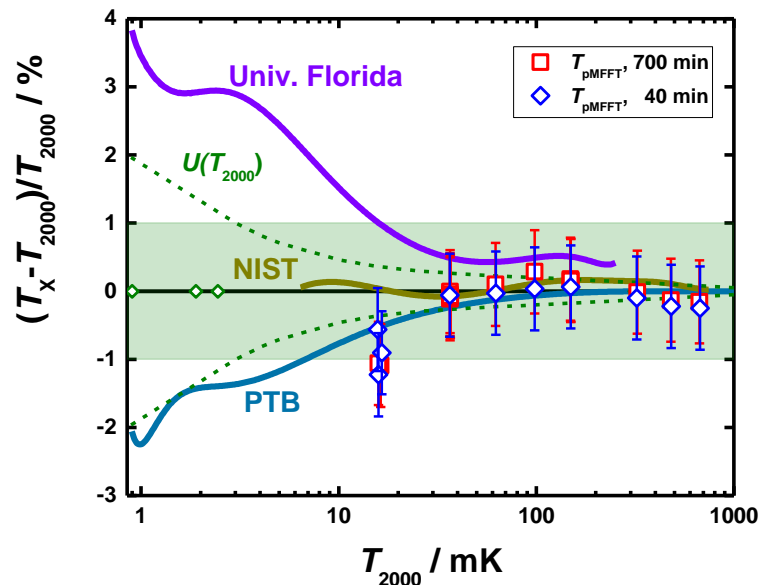[24] Rusby R. et al. J. Low Temp. Phys. 126 N°1-2, 633-642 (2002)

Figure 3.22 Relative deviations from the PLTS-2000 of thermodynamic temperature determinations with the pMFFT[21]. Further details are given in the text.

Still, the problem of the discrepant background data of the PLTS-2000 and the correct relation to thermodynamic temperature is not finally resolved for the lowest temperatures. Nevertheless, all preparative steps have been made and the means developed within the InK project are now available to successfully tackle this problem in near future before the awaited redefinition of the kelvin in 2018. Importantly for all users and producers of cryogenic low-temperature equipment, the PLTS-2000 agrees well with thermodynamic temperature in most of the working range of dilution refrigerators.

Key research outputs and conclusions:

- Three new primary thermometers were developed for the low-temperature region below 1 K: a Current Sensing Noise Thermometer, a Coulomb Blockade Thermometer and a primary Magnetic Field Fluctuation Thermometer. For all low-temperature primary thermometers a detailed uncertainty budget was established in accordance with metrological requirements.

- Agreement within 1% between the new low-temperature primary thermometers was experimentally verified. Thus, the primary thermometers developed in this objective of the project are well suited for a direct realisation of the kelvin in practical applications.

- The results of this objective of the project confirm that in practically most of the application range of standard dilution refrigerators from 20 mK up to 700 mK the PLTS-2000 agrees well with thermodynamic temperature within 1% and can further be used as a reliable and accurate temperature scale.

4   Actual and potential impact

Objective 1

Establishment of High Temperature Fixed Points and Validation of Filter Radiometry for thermodynamic temperature realisation and dissemination

Dissemination activities undertaken

During the project the following dissemination activities were undertaken to develop primary radiometric thermometry into a "mature discipline" and to capture and disseminate our best practices:

- An open workshop has been held (September 2012 in INRIM) on best practice in filter radiometry which included extensive technical discussion on methodologies, lessons learnt, techniques, potential problems and how to develop a robust uncertainty budget.

- This was followed by a publication on uncertainty analysis in filter radiometry, published at Tempmeko 2013, along with publications on the methodologies of the different participants (also at Tempmeko 2013). This provides a set of reference material for this field.

- Significant progress was made by the participants, and a few other members of CCT WG NCTh, to develop a CCT guideline document on uncertainties in primary radiometric thermometry.

It is hoped that this provides a body of reference for supporting others developing these techniques. These documents will be referenced in the recommendation to the CCT that will be made in 2016 after the Tempmeko 2016 conference.

The results of the fixed point measurements have been published in:

- A presentation to the Royal Society at a Theo Murphy scientific meeting at the Kavli centre – the meeting title was "Towards implementing the new kelvin"

- A paper has been submitted to and accepted by Philosophical Transactions of the Royal Society, A.

Anticipated future impacts and dissemination plan

The main early impact of this work will be the incorporation of the re-determined Cu point and low uncertainty values of HTFPs into the *MeP*-K. Recommendations will be sent to CCT after the meeting of CCT-NCTherm in July 2016. This will directly feed into the establishment of these as formal fixed points, both for a temperature scale and for the direct dissemination of thermodynamic temperature with appropriate uncertainty.

It is anticipated that this will both lower the uncertainty associated with high temperature measurement, with benefit for high temperature manufacturing. In addition it will be the first part of the temperature scale which could be superseded with a true thermodynamic approach (either by direct or indirect through the HTFPs).

Objective 2

From the results obtained in Objective 2 the participants have issued recommendations to the community of radiation thermometry through the CCT "Non-Contact Thermometry" working group:

- HTFPs can be used to disseminate either the ITS-90 scale or thermodynamic temperature from National Measurement Institutes to users with uncertainties at least comparable with current approaches.

- However considering that this study (along with others) have shown that there are still some ill understood effects due to the interplay of the furnace and HTFP cell, namely thermal inertia and furnace uniformity, it is clear that if the lowest uncertainties are to be obtained in the dissemination of temperature by this route these effects need further study and quantification.

- Filter radiometers and radiation thermometers, directly calibrated to the radiometric references, can be used directly to disseminate thermodynamic temperature to users with uncertainties comparable to current methods.

- However it should be noted that unknown radiometer drift remains a problem and it is recommended that if this approach of dissemination if adopted that a HTFP be used in the institute to periodically assess the stability of the radiometer, or that at least two radiometers be used as the basis of the transfer and periodic cross comparisons be performed to confirm stability.

- A detailed study should be performed to reliably quantify the corrections and uncertainties for the non-uniformity of high-temperature furnaces used as radiance sources to transfer the calibration of a reference filter radiometer to a radiation thermometer.

The dissemination thermodynamic temperatures in the high-temperature range will benefit from the findings of this study. There are still important points to be considered in the future studies especially towards a better understanding of the thermal effects and their related uncertainties. These will be discussed and a way forward agreed at the July meeting of CCT-NCTherm in July 2016. The results of this research will form the important basis for those discussions.

Objective 3

The output from the research activities related to the determination of the thermodynamic temperature will have a short-term impact by providing relevant information to the CCT, i.e. the International Committee who is responsible for the realisation and dissemination of the kelvin and for the temperature scales. This information will be in the form of an agreed set of accurate and consistent determinations of the differences $(T - T_{90})$, as they have been obtained through the course of the project by different primary thermometry methods.

In the medium-term, the new $(T - T_{90})$ determinations will have substantial impact on dissemination, providing the fundamental input data which are needed to work out a revised International Temperature Scale to provide a new, significantly more accurate, formal approximation of the thermodynamic temperature scale.

In addition the work performed in this InK project directly informed the research that will be performed in the InK-2 project, which when combined with the work undertaken here, will provide a complete thermodynamic data set over the whole range of ITS-90, for incorporation into the *MeP*-K annex in 2019.

The improvement of primary thermometry methods obtained in this project will favour, on a longer time-scale, the realization of simplified and economic versions of the same methods. When this will happen, the direct dissemination of the thermodynamic temperature may become an attractive competitor to the current dissemination practice.

Objective 4

The results of the work carried out in Objective 4 directly address the needs of users as well as producers of cryogenic equipment. The problems of thermometry in the low- and ultralow-temperature region are caused by the fact that the thermometers available have restricted working ranges with insufficient resolution and precision, are prone to external influences and instabilities and may lose the thermal contact to the object measured with decreasing temperature. For calibrating these thermometers the actual reference, the PLTS-2000, is not accessible for the majority of users because of the high costs and the special knowledge, which is required for its realisation. The situation is nicely described by citing a quote attributed to Prof. Lounasmaa, Helsinki University of Technology (now Aalto University)[19]: "A person with one thermometer knows what the temperature is; a person with two will forever have doubt".

A first visible and direct impact of Objective 4 is the fact that for the realisation and dissemination of the kelvin in the cryogenic region three new low-temperature primary thermometers are now available, which comply with metrological requirements. The equivalence of the temperature measurement using these thermometers has been directly demonstrated removing the ambiguity in low-temperature thermometry mentioned before. For manufacturers of cryostats and cryogenic equipment this opens the opportunity to rely now on approved thermometers to demonstrate the parameters of their products such as the base temperature of dilution refrigerator or the cooling power. In addition, these thermometers are user friendly and can replace thermometers such as the nuclear orientation thermometer used before, which requires radioactive materials for operation, a real obstacle for trade to outside Europe. This underpins the worldwide leadership of European cryogenic companies in the current situation of still increasing sales of cryogen-free dilution cryostats for the sub-kelvin range. In this context it is worth to mention that for PTB the number of calibration requests for a commercial variant of the MFFT has significantly increased over the time of the project, for the last year at least by a factor of three.

Another important outcome of the project is that via the joint comparison measurements the partners from national metrological institutes and from leading low-temperature labs in Europe now have established and confirmed a direct and common reference for their thermometry at low and ultralow temperatures. This

enables them to support a high-level infrastructure in European temperature metrology. In addition, through their numerous scientific contacts the partners from the universities involved in this project act as promoters of the results not only in the low-temperature community in Europe but also worldwide.

In addition there is a clearly increasing demand from nanoscience, quantum computing and other quantum research in ultralow-temperature environments[25]. Such ultralow-temperature environment is crucial because quantum effects become visible only at low temperatures when the thermal energy of the investigated quantum systems is smaller than the internal interaction energies. This requires the creation, control and maintenance of ultralow temperatures. The CSNT developed by the RHUL has already found a successful application in such an ultralow temperature cryostat[26]. The system built together with a major supplier of cryostats has combined a cryogen-free dilution refrigerator with a superconducting magnet and an attached adiabatic demagnetisation stage, all precooled by a pulse tube cooler. The system has reached temperatures as low as 0.6 mK as measured with the CSNT. A similar approach was realised in[27], where a precursor of the pMFFT developed here, a commercial MFFT, has been used for temperature measurements below 1 mK. Therefore, cryogen-free ultralow-temperature platforms for new, rapidly growing research and application areas seem to be a realistic perspective and the thermometry necessary for their operation has been developed within this project.

It is noteworthy that a patent is pending, which covers the special implementation of the measurement of fluctuating magnetic field signals using SQUIDs for noise thermometry with the pMFFT.

5   Website address and contact details

http://projects.npl.co.uk/ink/

Contact - Dr Graham Machin

Tel – +44 20 8943 6742

Email - graham.machin@npl.co.uk

6   List of publications

- L. Moretti, A. Castrillo, E. Fasci, M. D. De Vizia, G. Casa, G. Galzerano, A. Merlone, P. Laporta, and L. Gianfrani "Determination of the Boltzmann Constant by Means of Precision Measurements of H218O Line Shapes at 1.39 μm" *Physical Review Letters* PRL 111, 060803 (2013) - 9 August 2013

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- M. R. Moldover, R. M. Gavioso, J. B. Mehl, L. Pitre, M. de Podesta and J. T. Zhang "Acoustic Gas Thermometry" *Metrologia*, **51**, R1-R19 (2014)

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[25] Siora G. et al. Cryogenics Impact Report, Science & Technology Facilities Council (2015), http://www.stfc.ac.uk/research/engineering-and-enabling-technologies/the-uk-impact-of-cryogenics/
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- E R Woolliams "Uncertainty analysis for filter radiometry based on the uncertainty associated with integrated quantities" *International Journal of Thermophysics*, **35,** 1353-1365 (2014)

- J. M. Mantilla, M. Hernanz, J. Campos, M. J. Martín, A. Pons, D. del Campo "Monochromator Based Absolute Calibration of a Standard Radiation Thermometer" *International Journal of Thermophysics*, **35,** Issue 3, 475-484 (2014)

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