IMPROVEMENTS IN TEMPERATURE MEASUREMENT

by

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A thesis submitted to the University of Birmingham for the degree of

DOCTOR OF SCIENCE

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December 2014

ABSTRACT

This submission for the degree of Doctor of Science describes work initiated, led or contributed to by Dr Graham Machin, a graduate of the University of Birmingham, over the last 25 years.

The submission comprises of six main parts, illustrating the different contributions made to the area of temperature measurement. In brief these are:

- Development and improvement of non-contact thermometry standards
- Comparisons of non-contact thermometry scales, including the development of new methods of comparison
- Contributions to the development of high temperature fixed points as new temperature references above the freezing point of copper (1084.62 °C)
- Development of methods for the mitigation of temperature sensor drift at high temperatures
- Improvements and innovations in medical thermometry – in a variety of settings
- Leadership in international temperature measurement research

The thesis begins with an introduction to the subject of the research, including a brief overview of the presented papers. Then a list of the presented papers is given followed by copies of the manuscripts. The thesis ends with an Appendix containing an up to date publication record.

ACKNOWLEDGEMENTS

I am very pleased to acknowledge the unfailing support of my National Physical Laboratory (NPL) and other colleagues from the many other National Measurement Institutes around the world with whom I have had the privilege of working over the past 25 years. I thank them for their constant enthusiasm, encouragement and willingness to collaborate.

I particularly want to thank my mentor Dr Richard Rusby; former Head of Temperature at NPL, whose patient and wise council has pointed me in the right direction on numerous occasions.

I also thank the National Measurement Office of the Department of Business, Innovation and Skills who has funded much of the work that is presented here.

Last but by no means least I gratefully acknowledge the love and support of my wife and family without which I would not have achieved all that I have done.

CONTENTS

1. INTRODUCTION

1.1 Preamble

A reliable global infrastructure for measurement is essential in our interdependent interconnected world. This is true for all the seven base quantities of the international system of units (the SI[1]); mass, length, time, electric current, temperature, amount of substance, and luminous intensity as well as the numerous associated derived quantities. The requirement for continually improving measurement arises from a broad spectrum of human endeavour for example in industry, medicine, defence and research.

The foundation of temperature measurement is primary thermometry. Primary thermometry utilises a fundamental equation of physics that relates a temperature dependent measureable quantity to thermodynamic temperature; for example; gas pressure is proportional to thermodynamic temperature or the speed of sound in a gas is proportional to the square root of thermodynamic temperature. Since primary thermometry is generally slow and difficult to perform a range of practical thermometers are used, the three main types being thermocouples, resistance thermometers and radiation thermometers[2]. The output of these practical thermometers is related to temperature by a process of calibration to the current temperature scale, the International Temperature Scale of 1990 (ITS-90)[3].

As temperature measurement needs are continually evolving there is a vibrant level of research activity in this field both improving current measurement methods, the development of new approaches as well as establishing their traceability to ITS-90.

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[1] Background information about the SI can be found at: http://www.bipm.org/en/si/

[2] Good descriptions of these thermometers are found in a number of text books such as Quinn, T.J., Temperature, 2nd Edition, Academic Press and Nicholas, J.V., White, D.R., "Traceable temperatures", 2nd edition, Wiley

[3] Preston-Thomas H., "The international temperature scale of 1990 (ITS-90)", *Metrologia*, **27(1)**, 3-10, (1990)

The activities described in this thesis cover a wide range of temperature measurement research that I have performed, initiated or led; this includes development of fundamental standards and assuring their veracity through comparisons, innovating new high temperature standards, solving industrial measurement problems (e.g. low uncertainty calibrations at high temperatures and sensor drift), innovations in medical thermometry and demonstrating leadership in world thermometry research.

1.2 Improving non-contact thermometry standards

Low uncertainty standards for the realisation and dissemination of the ITS-90 are the essential foundation for reliable temperature measurement.

This section outlines the contributions I have made to non-contact thermometry standards. These are; the establishment of a low uncertainty realisation of ITS-90 in the temperature range -40 °C to 3000 °C, characterisation of radiation thermometers for the size of source effect (SSE)[4] and the development of reliable uncertainty budgets for non-contact temperature scale realisation.

Above the silver freezing point (961.78 °C) the ITS-90 is realised by radiation thermometry. This requires a high performance radiation thermometer and a reference blackbody at one of the defining fixed points of the ITS-90[5]. A radiation thermometer[6] is used to measure both the emitted spectral radiance of the reference blackbody and that of an unknown radiance source

[4] See footnote 14 for a description of the size of source effect

[5] Blackbody cavities at the freezing points of Ag (961.78 °C), Au (1064.18 °C) or Cu (1084.62 °C) are all allowable starting points for a realisation of the ITS-90 above the silver point

[6] The radiation thermometer used for ITS-90 scale realisation generally has a silicon photodiode detector, a narrow band interference filter to define the wavelength of operation (usually at 650 nm or around 900 nm), a small target size with well characterised linearity.

(usually a variable temperature blackbody). Through use of Planck's law[7] the temperature of the unknown radiance source is determined from radiance ratios. I established the NPL primary realisation of the ITS-90 above the silver point described in [1].

Below the silver point the ITS-90 is defined in terms of contact thermometry. Platinum resistance thermometers (PRTs)[8], of specified quality, are calibrated against defined fixed points[9] to establish the scale. PRTs[10] were used to establish traceability to the ITS-90 for a series of high performance blackbody reference sources, with low uncertainties, for the calibration of radiation thermometers and thermal imagers from -40 °C to 1000 °C [2, 3].

An alternate form of realising the scale below the silver point is through using the ITS-90 defining fixed points as blackbody sources. A set of lower temperature fixed point blackbody cavities based on the fixed point materials of tin, lead (~327.5 °C)[11], zinc, aluminium (660.323 °C) and silver were constructed. A radiation thermometer equipped with an InGaAs[12] detector [4] was constructed and calibrated against these fixed point blackbodies. The measured signal was used to establish an interpolation function based on a parameterised form of Planck's law known as the Sakuma-Hattori equation[13]. The thermometer was then used to calibrate unknown temperature blackbody cavities in terms of ITS-90. The thermometer was subsequently used to evaluate the scale established by variable temperature blackbodies [2, 3] and satisfactory agreement found between both methods [5].

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[7] Planck's law in ratio form is used in the definition and realisation of the ITS-90. The formal definition can be found in the reference given in footnote 3.

[8] Platinum resistance thermometers are the specified interpolation thermometers of the ITS-90 below the silver freezing point to the triple point of equilibrium hydrogen (~13.8 K). They act as thermometers through utilising the variation of resistance of platinum wire (of specific purity) with temperature.

[9] Examples of fixed points used for ITS-90 scale realisation are the freezing points of tin [231.928 °C] or zinc [419.527 °C] and the triple points of water [0.01 °C] and mercury [-38.8344 °C]

[10] Or other temperature sensors whose calibration is directly traceable to ITS-90 calibrated PRTs

[11] Not formally an ITS-90 reference point. It was used because it was a convenient temperature reference for calibrating the radiation thermometer

[12] Indium gallium arsenide

[13] Sakuma F., Hattori S., In *Temperature: Its Measurement and Control in Science and Industry*, Vol. **5**, Pt. 1, New York, American Institute of Physics, 421-427, (1982)

For radiation thermometers a number of parameters (for example linearity, wavelength) need to be accurately quantified to facilitate reliable temperature scale realisation and dissemination. One parameter which is particularly difficult to quantify is the SSE[14]. The temperature uncertainty due to SSE for a range of different radiation thermometers was determined and described in [6, 7].

To establish a reliable temperature scale it is essential to understand and quantify all the uncertainty sources in its realisation. Work in the Consultative Committee for Thermometry (CCT)[15] led to the development of a uniform way of assessing and expressing uncertainties in the scale realisation of ITS-90 above the silver point. The outcome of that work is described in [8]. As a member of CCT-WG5[16] I contributed to writing the text and quantifying particular uncertainty components elaborated in that document. I also contributed through a critical review of the whole document before publication.

1.3 Precision comparisons of temperature scales and fixed points for non-contact thermometry

1.3.1 *Precise comparison of non-contact temperature scales*

The establishment of national scales for any measurement quantity is essential for industry and regulation. However to achieve the highest confidence in these scales it is necessary to

[14] The Size of Source Effect (SSE) arises as follows. In principle radiation thermometers, if they had perfect optical systems, would generate 100% of their signal within their nominal target spot. However due to imperfections in their optics thermal radiation from outside of the target spot is scattered into the measurement region and thermal radiation from the target spot is scattered out of the measurement region leading to errors in temperature measurement. This is a subtle effect and leads to radiation thermometers reading too low (or too high) a temperature depending upon whether the temperature of the surroundings is cooler (or hotter) than the target being viewed

[15] The Consultative Committee for Thermometry is a consultative committee of the International Committee of Weights and Measures http://www.bipm.org/en/committees/cc/cct/

[16] CCT-WG5 is a working group of the CCT responsible for all matters pertaining to radiation thermometry, now known as CCT WG-NCTh (non-contact thermometry). I have chaired this group since 2004.

perform comparisons with those of other national measurement institutes (NMIs) to ensure worldwide equivalence of scale realisation.

In the past high temperature scale comparisons were performed using high stability tungsten ribbon lamps[17]. However from the early 1990s it became increasingly clear, because of technological advances, that this artefact was no longer fit for purpose[18]. A new method of comparing scales was urgently required. Two different approaches were pioneered over the years, with work I performed in the vanguard of these developments.

The *first approach* to scale comparison was to use compact radiation thermometers. These were silicon photodiode, lens based devices. Comparisons within the EU [9] and with NIST[19] (USA) [10] showed that the approach was superior to the use of lamps and more technologically appropriate. Since that time the use of high stability lamps for scale realisation, comparison and dissemination has almost ceased.

However radiation thermometers are prone to stochastic changes in output and so the final attainable comparison uncertainty is restricted to a few degrees Celsius at the highest temperatures, which does not probe the *claimed* uncertainties of national scale realisations.

To facilitate comparison of high temperature scales at those claimed uncertainties I pioneered a *second approach*. In this a number of highly stable high temperature fixed points[20] (HTFPs) [see Section 1.4], were constructed and circulated. These artefacts, because they are fixed points, are driftless and well suited to the low uncertainty comparison of scales. I showed for

[17] For example see Lee, R.D., Kostkowski, H.J., Quinn, T.J., Chandler, P.R., Jones, T.P., Tapping, J., Kunz, H., In: *Temperature: Its Measurement and Control in Science and Industry*, Vol. **4** (Edited by H.H. Plumb), Pittsburgh, Instrument Society of America, 377-393, (1972)

[18] The limitations of the high stability tungsten ribbon lamp (HSL) are; fragility (having a quartz envelope), a small target size (1.5 mm), the radiating area was not a blackbody and the highest temperature an evacuated HSL could attain was 1700 °C. Whereas increasingly temperature scales were being realised on blackbody cavities, with large target sizes (typically 25 mm diameter) and operated to temperatures around 3000 °C.

[19] The National Institute of Standards and Technology, Gaithersburg, Washington D.C.

[20] High temperature fixed points based on the metal carbon eutectic reaction. Typical fixed points are: Co-C (1324°C), Pd-C (1492°C), Pt-C (1738°C), Ru-C (1954°C), Re-C (2474°C) with the approximate melting transition temperature indicated in brackets.

example in [11], in a comparison between NIST and NPL, that it was possible to use HTFPs to compare national scales at selected temperatures to <0.5 °C. This was the first time such reliable comparisons had been performed over such a wide range of temperatures. The process has been refined over the years until scale equivalence with very low uncertainties have been demonstrated. For example a comparison using HTFPs between NPL and the NMIs of China and Spain[21] showed extremely good levels of scale agreement ~0.1 °C at Pt-C (1738 °C) and ~0.4 °C at Re-C (2474 °C) [12].

Although still novel the temperatures of some HTFPs have become relatively well known. This knowledge potentially undermines the objectivity of any comparison because the temperature of the comparison artefact is known *a priori*. To obviate this I led work[22] showing that by adding small amounts of impurity to known HTFPs their phase transition temperature can be changed without degrading performance. A trial comparison using such artefacts was performed between INMETRO[23] (Brazil) and NPL at one nominal temperature with scale agreement of <0.1 °C at around 1330 °C [13][24].

Comparison of scales below the silver point is difficult because they are usually realised on variable temperature blackbody cavities [2, 3]. However a portable InGaAs detector based radiation thermometer was constructed for this purpose by the Italian NMI[25]. This was calibrated against fixed point blackbodies between the In (156.5985 °C) and the Ag freezing

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[21] National Institute of Metrology (China), Centro Español de Metrologia (Spain) respectively

[22] PhD Student Renato Nunes Teixeira, "Development of High Temperature Comparison Artifacts for Radiation Thermometry", Pontifica Universidade Catolica, Do Rio de Janeiro, 2013, PUC-Rio, cosupervisors, Prof Graham Machin (NPL), Prof. Alcir de Faro Orlando (PUC-Rio)

[23] INMETRO is the Brazilian NMI. INMETRO stands for National Institute of Metrology, Quality and Metrology, located near Rio de Janeiro

[24] Building on this work a global comparison of high temperatures scales involving NMIs in Europe, the Asia-Pacific, the Americas and Russia is currently under way, led from NPL, and it will use both radiation thermometers and objective HTFPs – this is anticipated to be completed in 2017.

[25] The Italian NMI was then known as Istituto di Metrologia "G. Colonnetti" (IMGC) subsequently renamed Istituto Nazionale di Ricerca Metrologica (INRIM)

points and used to compare scales with NPL and the Turkish NMI[26]. I led the NPL contribution to this comparison, which demonstrated excellent scale agreement of ~0.1 °C throughout the range [14].

1.3.2 *Precise comparison of non-contact thermometry fixed points*

The foundation of the ITS-90 above the silver freezing point is the reference blackbody. It is essential that the spectral radiance emitted by that blackbody closely represents the temperature of the fixed point. These blackbodies are carefully designed to attain this condition. However comparison of primary reference fixed points is desirable to give assurance as to the quality of the realisation. Such a comparison was performed with the temperature group of the Mexican NMI (CENAM[27]). The Mexican national standard silver point blackbody was transported to NPL and compared with the UK national reference silver point. Agreement between the two blackbody sources was demonstrated to be <0.010 °C confirming the veracity of both silver fixed point blackbodies [15].

1.4 Development of high temperature fixed points to above 3000 K

When ITS-90 was formulated only precise thermodynamic temperatures were known for pure materials. This meant that the scale at high temperatures was based on relatively low temperature fixed points[28]. This definition, although an improvement on previous scales (IPTS-68, ITS-48, ITS-27[29]), still had the problem that the scale realisation uncertainty at high temperatures generally increased as T^2.

[26] The Turkish NMI is known as Ulusal Metroloji Enstitusu (UME)
[27] CENAM stands for Centro Nacional de Metrologia, located at Queretaro, Mexico
[28] See footnote 5.
[29] The International Practical Temperature Scale of 1968, the International Temperature Scale of 1948 and the International Temperature Scale of 1927 respectively.

Ideally the scale would be realised through using higher temperature fixed points. Then using Planck's law in ratio form to extrapolate downwards the systematic uncertainties would scale to smaller values as T^2. Unfortunately such fixed points were not available at the inception of the ITS-90. However in 1999 the feasibility of exploiting binary metal-carbon eutectics as high temperature fixed points (HTFPs) was demonstrated[30]. It was subsequently shown that a wide variety of binary alloy fixed points were possible, even to >3000 °C [see Table 1 in [16] for a complete list].

From the inception of HTFPs I have played a leading role in their development. Through leading a consortium of EU NMIs in a Framework 5 GROWTH programme project (known as HIMERT[31]) I established major EU capability for HTFP research [17].

One of the concerns of early researchers was to establish whether the eutectic phase transition was repeatable[32] and reproducible[33]. Repeatability of individual HTFPs was quickly established to be <0.02 °C. However initial results regarding reproducibility were mixed with variations of ~1 °C between HTFPs of nominally the same material being reported [18]. If this had been a limiting factor, it would have severely restricted the usefulness of HTFPs as temperature reference artefacts. However it was shown [19] that provided residual impurities within the base material were carefully controlled, then HTFP temperature reproducibility of <0.1 °C was possible, even at the highest temperatures.

Once reproducibility was established the next step was to determine the HTFP thermodynamic temperatures. In pursuit of that objective I launched an international initiative

[30] Yamada, Y., Sakate, H., Sakuma, H., Ono, A., In: *Proceedings of Tempmeko '99*, edited by J.F. Dubbeldam and M.J. de Groot, Nmi Van Swinden Laboratorium, Delft, 535-540, (1999)

[31] HIMERT stands for High temperature, Metal-carbon Eutectic fixed points for Radiation Thermometry, Radiometry and Thermocouples

[32] That is for a given fixed point under quasi-identical laboratory conditions was the same temperature attained.

[33] That is if different HTFPs were made by different researchers from different lots of nominally the same material do they give the same temperatures.

to determine the thermodynamic temperature of a selected set of HTFPs. That project began in 2007 [20] with a progress report in 2012 [21].

One of the difficulties with assigning accurate thermodynamic temperatures to HTFPs is quantifying the magnitude of the temperature drop across the finite back-wall thickness of the blackbody. In addition the blackbodies are made out of graphite which has a natural variation in emissivity which affects the radiance temperature of the fixed point. These two sources of uncertainty are not easily investigated experimentally so have been quantified in detail through thermal modelling [22].

Nearly all early research into HTFPs was performed by radiation thermometry. However HTFPs can also be used to calibrate high temperature thermocouples. In particular the Pt-Pd thermocouple[34], which was thought to have superior performance to the standard Pt-Pt/Rh thermocouples (type R, S or B[35]), could not be calibrated by usual methods (i.e. the Pd wire bridge method[36]) because the Pd thermoelement would melt during the procedure. However the Pd-C eutectic melting temperature is 1492 °C[37] and so ideally suited to calibrate such thermocouples. I, with my French and German colleagues, launched a collaboration where Co-C (1324 °C) and Pd-C HTFPs were independently constructed and compared. The results of this study are reported in [23] to which I contributed significant leadership in the NPL activities and guidance to the overall activity. NPL has become the first EU NMI to offer accredited noble metal thermocouple calibrations using HTFPs and through these

[34] A thermocouple whose thermoelements were made of pure Pt and pure Pd wire

[35] Type R, S and B thermocouples are noble metal thermocouples that have different composition thermoelements, these are: type R Pt vs Pt13%Rh, type S Pt vs Pt10%Rh, type B Pt30%Rh vs Pt6%Rh

[36] The wire bridge method is an established method of calibrating high temperature thermocouples. In the method the measurement junction is cut and a short piece of wire of pure material, usually Au or Pd (and for type B, Pt), welded between the thermoelements, forming a "bridge". This is then heated and the thermovoltage monitored. On melting of the "bridge" material the thermovoltage does not increase. As the melting temperature of the "bridge" material is known a calibration point (i.e. the thermovoltage) is obtained.

[37] This is >60 °C below the melting point of pure Pd

developments thermometry in the heat treatment of turbine blades for aerospace applications was improved [24].

In recognition of my leadership in this field I gave an invited keynote address at the decennial International Temperature Symposium in 2012, Los Angeles, USA. That paper presented a comprehensive review of world HTFP research [16].

1.5 Development of self-validation of temperature sensors to 2300 °C

Above 1000 °C typical contact thermometry sensors used in industry are thermocouples. A thermocouple is a very simple device made of two dissimilar wires joined at the measurement junction, the thermovoltage being generated in the temperature gradient region of the wires.[38] Although all thermocouple types drift the work I led focused on the refractory metal thermocouple; partly because they have a large uncertainties even when new (±20 °C at 2000 °C), they show large in-service drifts, with 50 -100 °C not being uncommon, and the thermoelements embrittle when used at high temperatures precluding removal for drift investigation. The papers presented here mainly arose out of a PhD[39] I instigated and supervised, and a project I conceived and led for the European Space Agency (ESA) into the development of self-calibrating thermocouples[40]. Self-validation, above 1000 °C, using HTFPs, had not been attempted before this work.

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[38] In industry, at temperatures up to about 1300 °C, mineral insulated metal sheathed (MIMS) nickel based thermocouples (type K or N) are typically used. Above 1300 °C to about 1600 °C, noble metal Pt-Rh alloy thermocouples[35] are used. Above that temperature to about 2300 °C refractory metal tungsten rhenium (W-Re) thermocouples are generally used with either molybdenum or tantalum sheaths.

[39] Oijai Ongrai, "New Approaches to Improve Thermocouple Thermometry to 2000 °C", March 2012, University of Surrey, UK, (co-supervisors, Prof Graham Machin, NPL Prof Steve Sweeney University of Surrey)

[40]The principle of self-calibration is straightforward. One or more known temperature reference points are incorporated around the measurement junction of the thermocouple. When this reference point is heated to its melting point the fixed point material melts, at its known temperature, and the thermovoltage of the

The investigation began by making miniature fixed points of HTFP material (Co-C). These were placed in mini graphite crucibles and attached directly to the measurement junction of a noble metal thermocouple [25]. These measurements successfully demonstrated the concept of self-validation but the method could not be used at temperatures above the Co-C point because the Pt thermocouple wires became embrittled due to physical contact with the graphite crucibles.

After these preliminary investigations tantalum sheathed W-Re thermocouples were selected for subsequent use. Tantalum was selected to ensure compatibility with graphite to 2300 °C. No access to the measurement junction was possible so very small crucibles with a re-entrant well were constructed, filled with HTFP material and placed around the measurement junction. This approach was superior to the first because the measurement junction was completely surrounded by the ingot material rendering the thermocouple less sensitive to surrounding thermal conditions [26]. The research progressed successfully to the highest temperatures (the Ir-C point [2292 °C]), and the results were summarised at the decennial International Temperature Symposium in 2012 [27].

ESA, on learning of the possibility of self-validating thermocouples, funded research into such sensors for space applications[41]. That work showed the importance of self-validation with the refractory metal thermocouple drifting by 80 °C at the Ir-C melting point in only 10 hours of operation [28].

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thermocouple is recorded. The measured thermovoltage is then compared to that of the reference table for that type of thermocouple and the observed difference allows a correction to be made so that the thermocouple reads the correct temperature. This correction process can be performed whenever the fixed point undergoes a phase transition.

[41] For example thermal protection system development and validation for temperature sensors used "in-flight".

1.6 Improvements and innovations in medical thermometry

Temperature measurement is widely used in medicine, from patient temperature monitoring to more novel approaches to aid disease diagnosis and treatment. I instigated and led research activities aimed at improving temperature measurement in a number of areas of medical thermometry.

Thermal imaging is used in medicine to identify and track a variety of medical conditions[42]. However the performance of thermal imagers is relatively poor[43] presenting a barrier to their deployment in medicine. I initiated research to develop blackbody sources specifically aimed at improving clinical thermal imaging. A travelling standard blackbody that could test imager performance *in-situ* was developed (see [3]). This showed the performance of thermal imagers that were in use by clinical centres differed greatly and they often departed from manufacturers specifications. This finding led to the development of a set of completely novel fixed point blackbody temperature references based on ethylene carbonate (36.3 °C), gallium (29.8 °C) and gallium-zinc eutectic (25.2 °C). These sources were small enough be inserted into the measurement field at the time of patient examination. This facilitated real time calibration of thermal imagers maximising reliability of use for clinical thermometry [29][44].

Mercury in glass thermometers were, until recently, the thermometer routinely used in medicine. However such devices have been replaced with electronic thermometers and in particular tympanic membrane or "ear" thermometers. These new thermometers required

[42] Examples of use of thermal imagers in medicine are in the diagnosis of Raynaud's Phenomena where, in response to a moderate cold challenge, circulation of blood to the fingers becomes very low (leading to temporary loss of finger use and pain), or Scleroderma which is a chronic systemic autoimmune disease characterised by hardening (sclero) of the skin (derma).

[43] Device manufacturers quote typical uncertainties of 2% or 2 °C whichever is greater

[44] Note that the work concerning fixed point blackbodies for clinical thermal imager assessment is briefly described in [3] full details are given in [29].

traceable calibration so I led the development of two blackbody sources for this purpose, one of variable temperature the other based on the fixed point material of ethylene carbonate.

To confirm the performance of the developed variable temperature ear thermometer calibrator a comparison of the different calibrators was performed. The ear thermometer calibrators of Japan and Germany were transported to NPL and their performance compared using modified ear thermometers with 0.01 °C resolution. The results of this comparison showed excellent performance for the three calibrators over the temperature range of interest [30].

The fixed point ear thermometer validator was based on ethylene carbonate and constructed of PTFE (which has a high emissivity in the infrared region where ear thermometers operate). Such a device could rapidly validate the performance of a large number of ear thermometers in a clinical setting and its performance is described in [31][45].

In critical care settings, particularly in situations where trauma[46] has occurred the temperature of the brain is closely monitored using small contact thermometers. In use these thermometers displayed small variations (of a few tenths of a degree Celsius) and there were questions about the clinical significance of such variations. Collaboration was established with the University of Manchester[47] and the performance of a number of these thermometers was investigated with two fixed point temperature references diphenol ether (25.3 °C) and ethylene carbonate. The thermometers performed well over the temperature range of interest, implying that the observed small temperature variations were due to real temperature variations within the brain [32, 33].

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[45] The work on establishing temperature traceability in a clinical setting formed part of PhD studies performed under my direction by Dr Rob Simpson at NPL; PhD title "Medical Infrared radiation thermometry calibration and traceability", 2008. This was co-supervised by Prof. Graham Machin (NPL) and Dr. Peter McCarthy at the University of Glamorgan (now University of South Wales)

[46] Such as stroke or severe head injury

[47] School of Translational Medicine, Brain Injury Research Group, Salford Royal Hospital

The above method of measuring brain temperature is invasive and therefore not desirable. A non-contact method (nuclear magnetic resonance spectroscopy) had been used, in a research setting, for a number of years but without traceability to the ITS-90. Traceability was established through the development of stable temperature artefacts which contained a solution, at the appropriate concentration, of a molecule typically found in the brain (in this study n-acetyl-aspartate). To be compatible with the high magnetic fields of NMR machines these references were first made first of PTFE and then glass. This work developed, for the first time, a calibration curve of proton resonance frequency shift with temperature that was directly traceable to the ITS-90 [34][48].

1.7 Leadership in world thermometry research

The kelvin along with the kilogram, ampere and mole is to be redefined in terms of fundamental constants[49] possibly as soon as 2018. A programme of research, to determine low uncertainty values of the Boltzmann constant, for the kelvin redefinition, was coordinated through a working group of the CCT [35], by a colleague from the German NMI (PTB[50]). Within this international setting I initiated NPL's activities to re-determine the Boltzmann constant[51].

To identify future thermometry research requirements I led two technical workshops. These consisted of European and world thermometry experts whose objective was to identify the research requirements for primary thermometry [36] and also the thermometry needed to

[48] I provided overall leadership for the NPL part of this project. The experimental work at NPL was performed by my colleague Andrew Levick. Collaborative investigation for different molecules and solute concentrations are ongoing, with a research group based at the University Hospitals Birmingham, Imaging & Medical Physics.

[49] Mills, I.M., Mohr, P.J., Quinn, T.J., Taylor, B.N., and Williams, E.R., 2011 *Phil. Trans. R. Soc. A,* **369**, 3907-3924 (doi: 10.1098/rsta.2011.0180) (2011)

[50] PTB stands for Physikalisch-Technische Bundesanstalt, the Temperature Group is based in Berlin

[51] NPL subsequently went on to determine the world's lowest reported uncertainty value (to date) for the Boltzmann constant with the experimental work led my colleague Michael de Podesta; reported in: de Podesta, M., Underwood, R., Sutton, G., Morantz, P., Harris, P., Mark, F. D., Stuart, F. M., , Vargha, G. & Machin, G., "A low-uncertainty measurement of the Boltzmann constant", *Metrologia*, **50**, p. 354 – 376 (2013)

address societal grand challenges [37]. The latter workshop produced a roadmap which has influenced the thermometry research content of the evolving Euramet[52] Strategic Research Agenda. The outcome of the former workshop led to my initiating the European Metrology Research Programme (EMRP) "Implementing the new Kelvin" (InK) project [38]. This has >15 international institutes as partners and addresses challenges in primary thermometry from 0.0009 °C to 3000 °C. Primary thermometry will be developed at the extremes of temperature (<1 K, >1300 K) to supplant defined scales and low uncertainty values of $T - T_{90}$ and $T - T_{2000}$ will be determined for the *mise en pratique* for the definition of the kelvin[53] (*MeP*-K) and the future temperature scale, the International Temperature Scale-20xx (ITS-xx). In addition through leading a sub group of CCT-WG5 the text for the *MeP*-K for high temperatures was elaborated [39].

Many industrial temperature measurement challenges remain such as variable transmission through contaminating optical windows and unknown and unquantifiable sensor drift. I initiated and led a major collaborative EMRP project, High temperature measurement solutions for industry [HiTeMS]), involving >15 EU institutes, whose objective was to solve a number of these problems [40].

[52] Euramet is the European Association of National Metrology Institutes. For more information http://www.euramet.org

[53] Ripple, D.C., Davis, R., Fellmuth, B., Fischer, J., Machin, G., Quinn, T., Steur, P., Tamura, O. & White, D. R., **"The roles of the mise en pratique for the definition of the kelvin"**, *Int. J. Thermophys.*, **31**, p. 1795-1808, (2010)

2. LIST OF SELECTED PUBLICATIONS IN TEMPERATURE METROLOGY

The numbers enclosed in the brackets {p. xx} denote location of manuscript in this thesis.

[1] Machin, G., "**The National Physical Laboratory (NPL) high temperature calibration facility (1200 K to 3100 K)**", In: *Metrologie 99, 9th International Metrology Congress*, Bordeaux, France, Published: Mouvement Francais pour la Qualite, 556-559, (1999) {p. 21}

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