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Novel Techniques for Traceable Temperature Dissemination

Overview

In 2018 the kelvin, the international base unit of temperature, will be redefined. The ITS-90 temperature scale will continue to be the main way in which the redefined kelvin will be disseminated, and against which temperature measurement devices will be calibrated, but issues with the scale need to be addressed to ensure the scale can be used accurately and that it will adequately support the redefinition. This project improved the accuracy of regions of the ITS-90 scale, and developed alternative methods to disseminate the redefined kelvin.

Need for the project

Temperature is one of the most frequently measured physical quantities in science and industry. Over two thirds of industrial processes are monitored and controlled by temperature measurements, and the accurate and consistent measurement of temperature underpins global manufacturing, health and safety, and the development of new sciences and technologies. The International Temperature Scale of 1990 (ITS-90) is used world-wide to calibrate temperature measurement instruments, and is one of the main mechanisms by which temperature measurement is standardised and disseminated. In 2018 the international base unit of temperature, the kelvin, will be redefined, and although ITS-90 will continue to be used to disseminate the kelvin and temperature measurement, the scale has a number of limitations that need to be addressed.

ITS-90 uses 'fixed points', materials that undergo phase changes at known temperatures (e.g. the freezing point of tin is 505.078 K / 961.78 °C) against which measurement devices can be calibrated. Standard platinum resistance thermometers (SPRTs) are used to interpolate temperatures between these points. However, measurements in some regions of the ITS-90 scale have larger uncertainties than others, limiting the accuracy of temperature measurements. The exact transition temperatures of some fixed points are not known as accurately as others, due to impurities in the fixed point materials, non-uniformity of temperatures during their heating, and difficulties in modelling their phase transitions. Furthermore SPRTs can introduce larger uncertainties due to limitations in their use, such as the influence of environmental factors.

Lower uncertainty measurements are required in regions of ITS-90, particularly from -218 ºC up to 1000 ºC, to ensure the temperature scale can support sufficiently high accuracy measurements in science and industry. In addition alternative, simpler methods are also needed to disseminate the temperature scale, including providing a practical direct link to thermodynamic temperature.

Scientific and technical objectives

The goal of this project was to improve the dissemination of the temperature scale, through improving the accuracy of regions of ITS-90, and by developing new techniques for traceable temperature measurements back to the kelvin definition.

1. <u>To optimise the realisation of the ITS-90. This will be achieved through:</u>
   - Resolving the key issues in the defining fixed points, in particular thermal and impurity effects, and phase transition modelling.
   - Study of the temperature scale uniqueness.
   - Optimising calibration processes for capsule type SPRTs and long stem SPRTs.
2. <u>To develop and validate new methods to provide traceability to the kelvin, including the development of alternative instruments and procedures. This will be achieved through:</u>
   - Developing and validating new fixed points in temperature ranges where large temperature gaps exist between established fixed points.

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- Establishing and validating an approximation to the kelvin in the range from 500 ºC to 1000 ºC; specifically by a) the design and construction of a new near infrared (NIR) thermometer and the necessary facilities to enable the radiometric characterisation of NIR wavelength range radiation thermometers b) the use of vapour pressure temperature scales by means of temperature amplifiers.
- Developing and validating methods for direct calibration to the thermodynamic temperature and alternatives to SPRTs by the determination of a new reference function for Au/Pt thermocouples and the development of practical acoustic thermometers. In addition new dedicated electronics for improved temperature controls will be developed.

Results

1. Optimising the realisation of the ITS-90

1a. Defining fixed points: resolving key issues related to impurities, thermal flux and phase transition modelling

The fixed points used in ITS–90 are based on phase transitions of pure chemical elements. Impurities in these elements alter the temperature of their phase transitions, whilst temperature variations inside the apparatus in which they are heated (thermal flux), and difficulties in modelling the transitions, obscure the exact point at which the transition occurs. Addressing these issues will allow fixed points and ITS-90 to be used with greater accuracy.

For the first time, this project determined values to correct transition temperatures for low-concentration metallic impurities (correction coefficients) in mercury. Phase diagrams were developed for the alloy of mercury-silver-cadmium using the ‘sum of individual estimates’ (SIE) method, which demonstrated the applicability of this approach for estimating uncertainties due to impurities.

A re-sealable triple point of water cell (the 0.01 °C fixed point) was constructed that allowed the water to be replaced multiple times. The cell exhibited good stability over one month, with variations of the order of 20 μK (20 billionths of a kelvin). Investigations into the influence of oxygen isotope content on the triple point temperature of water has enabled a reduction in the uncertainty in the isotopic correction, and in the overall uncertainty in the realisation of the triple point temperature.

A phase-field model was developed to study the influence of thermal fluxes on fixed points. The model handles the physics associated with solidification of very pure metals, and explained some phenomena observed, including behaviour under different freeze nucleation regimes, and the range of conditions under which the analytical Scheil correction methodology is applicable. It has been established that the hybrid SIE/modified-OME (overall maximum estimates) method is the most consistent across all the different fixed point cells. This research highlighted the strengths and weaknesses of all the available techniques, and ultimately enables the uncertainty associated with the correction to be better characterised.

A new experimental method based on the difference of the electrical conductivity of solid and liquid metals was developed, which enables the solid fraction and melt fraction along the phase transformation in high temperature fixed points to be studied. To enable these studies a two-zone impedance measurement system, a targeted furnace-controller and an analytical model for furnace optimisation were developed, increasing the duration of the temperature plateau from a few hours to an unlimited amount of time. A transient heat conduction model and spatial distribution model of eddy currents were also developed. This new measurement technique will help increase theoretical knowledge of the phase transitions of the fixed points, ultimately providing an accurate theoretical model. As part of the microscopic modelling of solidification of highly pure metals, a survey of distribution coefficients was performed and published, which for the first time provides a clear picture of the overall trends in distribution coefficient for impurities across the periodic table.

1b. Study of ITS-90 non-uniqueness effects

SPRTs are used in the ITS-90 scale for temperatures between the fixed points, and use mathematical functions that relate temperature to electrical resistance. However, these interpolating equations do not take into account physical effects associated with real thermometers, and hence the equations are always subject to an interpolation error of the order of at least 0.5 mK. Different thermometers of the same type, calibrated at the same fixed points, and using the same interpolating equation, may give different results for the same temperature. This affects all of the ITS-90 range, but is particularly severe between the aluminium point

(660.32 °C) and the silver point (961.78 °C), where there is very limited data due to the drift and oxidation of the SPRTs.

The project consortium investigated this issue using high-quality SPRTs, a number of the new fixed points (see 2a), and gas controlled heat pipes, together with a statistical study on historical calibration data between fixed points and SPRTs from different manufacturers. The ITS-90 non-uniqueness calculated from these data showed temperature differences within ±15 mK. For the first time information is available in a peer reviewed journal on the magnitude of the non-uniqueness at high temperatures, however further investigations are required and the new mise en pratique for the kelvin will need to include techniques to address or reduce this effect. (The mise en pratique is set of instructions that allows the definition of the new Kelvin to be realized in practice at the highest level).

1c. Improved calibration procedures for capsule type SPRTs and long stem SPRTs

Capsule type SPRTs (CSPRTs) are used as ITS-90 interpolation instruments at low temperatures, and due to their compact form, are also often employed with thermodynamic thermometers to determine the difference between the thermodynamic temperature and ITS-90 temperature. The calibration of these thermometers, using fixed point cells intended for long stem SPRTs, is often performed in a very different environment to that intended for CSPRTs or in which the CSPRTs are used.

A range of adapters were developed to enable CSPRTs to be used with conventional fixed point cells. The effects of environmental influences, such as gas, oil, or vacuum on the performance of CSPRTs was also investigated, and it was found that none significantly increased the calibration uncertainty provided good thermal contact is assured.

A number of factors, including self-heating effects, the use of bushings, oxidation and annealing effects were investigated, with the aim of improving calibration procedures for SPRTs. A new system to measure the current of the AC and DC resistance bridges was designed, which enabled the uncertainty associated with the self-heating of thermometers to be evaluated for calibrations at the highest level of accuracy. A study on the benefits of different types of bushings (brass, copper and aluminium) and two types of thermometers encapsulated in silica and Inconel were conducted. The study identified the optimum materials for bushings and guidance on the use of metal bushings and the estimation of the decrease in self-heating or increased heat-loss has been published in the journal Measurement.

2. New methods and instruments for an improved and simpler traceability to the kelvin

2a. New fixed points to address gaps in ITS-90

As the ITS-90 fixed points rely on phase transitions of specific materials (usually elements), the fixed points are not evenly distributed across the temperature scale and gaps of over 200 °C exist between some points.

New fixed point cells (Xe, CO_2, I_2 and Al-Cu eutectic) were developed addressing the temperatures -111.75 °C, -56.56 °C, 113.7 °C and 548.16 °C respectively, and the uncertainties associated with the realisation of these fixed points ranged from 1 mK for Xe and CO_2, up to 10 mK for the Al-Cu eutectic. These new cells were used for the study of non-uniqueness of ITS-90 (see 1b) and will enable studies to be undertaken aimed at possible improvements to the ITS-90 equation to make it more robust.

2b. Methods and instrumentation for establishing an approximation to the kelvin from 500 °C to 1000 °C

The temperature range between 500 °C and 1000 °C is especially problematic for ITS-90 due to the instability of high temperature SPRTs. Three different methods (near infra-red radiation thermometry, vapour pressure temperatures, and acoustics) were tested and implemented as the basis for new thermodynamic temperature standards.

A near infrared radiation thermometer (NI-RT) with tuneable operating wavelength was constructed, and, for the first time, was used to calibrate and validate an imaging type, single detector radiation thermometer in the near infrared range. The relative standard uncertainty achieved was comparable to the uncertainty that can be attained with the highest level of non-imaging filter radiometers within this wavelength range. This instrument, together with the calibration procedure developed, allows a direct measurement of thermodynamic temperature, bypassing the use of the ITS-90 scale, and thus provides an alternative means of disseminating the kelvin definition.

A new ‘Temperature Amplifier’ was developed based on coupled gas-controlled heat pipes. The instrument was used to relate two different temperature ranges, and can be used to redefine one range in terms of the other. This represents a key achievement, and improvement on the present technology for thermal metrology between the aluminium and silver fixed points.

2c. Potential alternative thermometers to SPRTs

High temperature SPRTs are the standard interpolating instruments for ITS-90 between 660.32 °C and 961.78 °C, however they are expensive and can exhibit instabilities due to intrinsic mechanical stress, contamination, and mechanical problems caused by damage to their quartz sheath, and hence are not commonly used in industry.

Gold-platinum thermocouples (temperature sensors) are much more robust, easier to use, and are much cheaper than SPRTs. Performance comparisons between gold-platinum thermocouples and high-temperature SPRTs indicated that the SPRTs gave more stable/repeatable measurements when not moved, but if the measurement sensor has to be moved, then the thermocouples were more accurate and are a suitable alternative in this situation. These findings have been published in the International Journal of Metrology and Quality Engineering.

Thermometers based on a sapphire whispering-gallery-mode resonator are also good candidates to overcome the mechanical instabilities of SPRTs in industrial applications where measurement uncertainties below 10 mK are required. A new prototype whispering gallery thermometer, operating in the temperature range from -80 ºC to 180 ºC, with a temperature resolution better than 1 mK and a temperature stability at 0 °C better than 3 mK was constructed. The results show potential for considerable improvements in electromagnetic thermometry methods and have been published.

In addition, two different concepts of acoustic thermometer prototypes were designed, constructed and characterised, with the aim of enabling calibration of long stem standard platinum resistance thermometers directly to thermodynamic temperature for the first time. The prototype built by INRiM, the Italian National Measurement Institute, operating from the mercury fixed point (-38.83 ºC) up to the indium point (156.60 ºC), maintains the high accuracy already demonstrated by primary acoustic thermometers, though it is drastically simplified in its design, easier and quicker to operate, and cheaper. The prototype built by NPL, the UK’s National Measurement Institute, retains many of the advantages of acoustic thermometry i.e. sensitivity and a simple operation, and is suitable for calibrating/comparing conventional contact thermometers over the range 0 °C to 100 °C.

Actual and potential impact

This project addressed some of the limitations of the ITS-90, particularly in the temperature range from -218 ºC up to 1000 ºC, and developed a range of alternative methods to calibrate directly to thermodynamic temperature. The new techniques, instruments and data developed by this project will directly impact on National Measurement Institutes and the laboratories that disseminate the kelvin through the calibration of high level thermometers, and on industrial and scientific users who require high-accuracy temperature measurements, through improving the robustness and reducing the uncertainties of the temperature scale.

Impact on the metrological and scientific communities

The evaluation of the influence of impurities, thermal flux and phase transitions on the ITS-90 fixed point cells has led to improved corrections and models, and a reduction in the associated uncertainties. The models enable optimal furnace conditions, fixed point cells, and initiation techniques to be designed more quickly and cheaply, allowing the experimental procedures required to realise the fixed points to be reduced to the minimum.

The re-sealable triple point of water cell has proved the feasibility of this new concept and offers clear advantages with respect to permanently sealed cells, and cost-savings when doping experiments are required to investigate the effects of both impurities and isotopes.

The four new fixed point cells provide additional fixed points in temperature ranges where large temperature gaps currently exist, reducing interpolation uncertainties and contributing to a more robust scale. In addition, the CO_2 fixed point may eventually provide an alternative to the mercury fixed point, overcoming issues associated with its toxicity and transportation.

In 2014 the project's results were presented to international Consultative Committee for Thermometry (CCT), and the information related to the triple point of water has already been included in the CCT technical guide "Supplementary Information for the ITS-90" published by the BIPM. In addition the project outcomes related to impurity corrections are under discussion by CCT, and are likely to appear in an updated version of the document. New data was also provided and published on the ITS-90 non-uniqueness, where CCT had previously highlighted a lack of information, thus enabling a more reliable assessment of this source of uncertainty.

Impact on the industrial and end user communities

As part of optimising the realisation of the ITS-90, improved calibration procedures for SPRTs were developed and published. In addition, a dedicated instrument for integrated and non-intrusive direct measurement of the bridge current was developed and is available for purchase.

Dedicated electronics constructed for the temperature control of fixed point furnaces have, for the first time, provided the very accurate temperature control required for some innovative primary thermometry devices. A number of European manufacturers have already contacted the consortium, and information about the design and performance of the new electronics has been exchanged.

The outcomes of the study on gold-platinum thermocouples have been published in a journal paper and in a report sent to the CCT that will be taken into consideration at the 2017 CCT meeting. It is foreseen that these results will be included in the future update of the Guide on Secondary Thermometry published by the BIPM. This guide is widely used not only by NMIs but also by accredited laboratories and even in industry. In addition, discussions with European and non-European sensor manufacturers have taken place regarding the behaviour of the thermocouples versus high temperature SPRTs.

The practical acoustic thermometers and the sapphire whispering gallery model resonator thermometer developed within the project are the first step in enabling direct calibration to thermodynamic temperature without the use of the ITS-90 scale. The practical acoustic thermometers could be implemented not only in NMIs but also, in some cases, in accredited laboratories and specialised industries. The whispering gallery thermometer is a good candidate for industrial use as it could be calibrated with uncertainties comparable to those of the platinum resistance thermometers while offering better mechanical stability. A key benefit of measuring thermodynamic temperature directly is the elimination of the uncertainty arising from interpolation between the fixed points during ITS-90 calibration. Both instruments are of interest to users in the low-to-medium temperature range and will help to address difficulties experienced in the space industry, where accurate temperature measurements from -200 °C to +200 °C are required, particularly for earth observation satellites and large space simulators. Several European companies have already expressed interest in the practical acoustic thermometry technology. At higher temperatures the new Temperature Amplifier system is eventually intended to become a commercially available device for accurate temperature measurements and calibration, aimed at overcoming the lack of calibration points between 660 ºC and 960 ºC.

For the first time in radiation thermometry, an imaging type, single detector radiation thermometer has been calibrated directly to thermodynamic temperature in the near infrared range. These are widely used in industry, such as the glass and steel industries, for temperatures below 1000 °C, and could offer a calibration method that by-passes sources of measurement uncertainty introduced when using ITS-90.

The research into impurity corrections for fixed points generated a large database of results detailing the effects of a given impurity on the freezing temperature of the fixed point material. This database has been published and is already being used by some manufacturers and laboratories to inform/validate the corrections for the influence of impurities in the ITS-90 fixed points they develop.

Dissemination activities

The project's results have been disseminated widely through journal papers, presentations, workshops, articles on the web, and in trade journals. 25 papers have been published in leading measurement science journals, including the *International Journal of Thermophysics*, and *Metrologia* (see next section for details). 48 oral and poster presentations have been given at international conferences including the 16th International Congress of Metrology and the Symposium on Temperature and Thermal Measurements in Science and Industry, and at national conferences in Portugal, the Netherlands, Italy, Germany, Spain, Turkey and the Russian Federation. External interest in the project's outcomes extended well beyond Europe, with a further 7 presentations, including 2 invited talks, given at seminars and workshops in China, Slovenia, Brazil, Mexico and the Czech Republic. An initial stakeholder workshop was held at the start of the project, with a second workshop held in May 2015 attended by 50 delegates from 28 countries, which incorporated both high level training and a separate training course for new recruits.

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