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1 Executive Summary

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.

The Problem

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 °C up to 1000 °C, 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.

The Solution

The project set out solutions to the most pressing problems associated with high level practical temperature metrology, in the most widely used temperature range, from -218 °C up to 1000 °C by:

- development of new robust high performance sensors,
- development of practical primary thermometers, simpler, faster (in operation) and cheaper,
- reduction of the uncertainties in the measurement of temperature leading to, for example, improved tolerances in processes where temperature is a critical factor,
- new more accurate and faster calibration methods for contact thermometry.

Impact

The results of this project impact directly in the thermometry community formed by all the National Metrology Institutes and the laboratories (usually accredited) which carry out the dissemination of the kelvin by the calibration of different high level types of thermometers, it also has directly impacted in the scientific community and industry that need to perform high accuracy temperature measurements. The outputs of this project optimise the realisation of the ITS-90 and provide new methods, alternative instruments and procedures to provide traceability to the kelvin through:

The step change improvements obtained in this project have substantially improved the capabilities and reduce uncertainties of European National Metrology Institutes, many of which have substantial turnovers. This will enable enhanced competitiveness of these institutes with competitors outside Europe. This will benefit their external customers in turn, helping them to remain competitive.

Most industrial processes are monitored and controlled by measuring temperature and abnormal variations in temperature can be an early indicator of an impending failure of a device or process. These facts have resulted in temperature control and sensors (within the temperature range covered by this project) finding applications in many different industrial sectors such as automotive, consumer electronics, process industries, pharmaceuticals, food and beverage among others. The optimisation of temperature monitoring by the reduction of the uncertainties or the development of better equipment/sensors for temperature sensing facilitates the minimisation of energy use. This not only benefits the environment but also the company's finances by reducing production costs, minimising product rejection and improving product quality all of which will improve industrial competitiveness.

2 Project context, rationale and objectives

This project addressed some of the limitations of the ITS-90, particularly in the temperature range from 218 °C up to 1000 °C, 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 CO₂ 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 °C and 960 °C.

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.

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) To optimise the realisation of the ITS-90. This will be achieved through:
 - a) Resolving the key issues in the defining fixed points, in particular thermal and impurity effects, and phase transition modelling.
 - b) Study of the temperature scale uniqueness.
 - c) Optimising calibration processes for capsule type SPRTs and long stem SPRTs.
- 2) 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:
 - a) Developing and validating new fixed points in temperature ranges where large temperature gaps exist between established fixed points.
 - b) Establishing and validating an approximation to the kelvin in the range from 500 °C to 1000 °C; 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.

- c) 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.

3 Research results

In the following sections a selection of the more remarkable findings obtained in the project are presented. This is not an exhaustive list of all of them but a selection to demonstrate how the project has achieved the proposed objective. More detailed information of these and other results can be consulted in the papers already published or that will be published soon that are listed at the end of this report.

3.1 Objective 1: Optimisation of the ITS-90

Objective 1a: Defining fixed point: resolving key issues related to impurities and thermal fluxes

The CCT has proposed the SIE (sum of individual estimates) method for the correction and the estimation of uncertainties on the realization of the fixed points. But to use this method is necessary the previous knowledge of the of correction coefficients for impurities of the different fixed points. That is how the temperature of the fixed points changes with a determined concentration of a certain impurity. Within the project doping experiments with Hg, H₂O, Ga and Al have been carried out. Furthermore the impact of the oxygen isotopes in the triple point of water were also investigated.

For the first time correction coefficients for the influence of low concentration of relevant metallic impurities on the temperature of the triple point of mercury have been obtained, namely Pb, Cd and Ag. These results were obtained by the development of doping experiments. Furthermore, the phase diagrams of the ternary alloy Hg-Ag-Cd at low concentrations has been also obtained, this result has proven the applicability of the SIE method. In this activity CEM, GUM (in the frame of a Research Mobility Grant - RMG) and TUBITAK worked in collaboration to obtain more robust results and the validation of the doping procedure developed.

Figure 1 a) and b) present an example of the results obtained in which the variation of temperature with the increase of the concentration of impurities, Cd and Ag respectively can be observed.

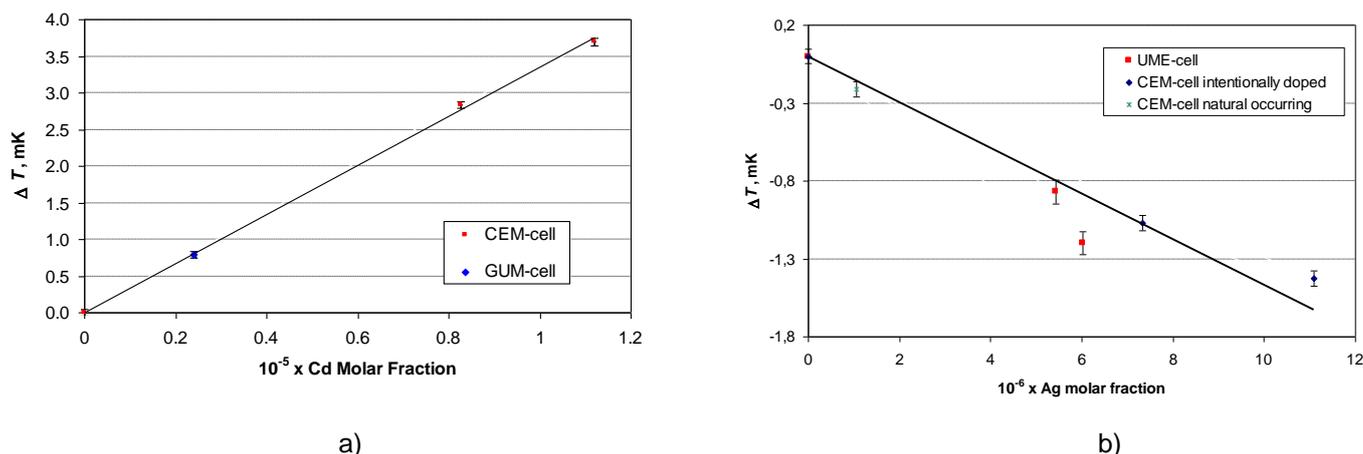


Figure 1. a) Results of the temperature variation of two Hg triple point cells doped with Cd. b) Comparison between CEM and TUBITAK (UME) results of the temperature variation of Hg triple point doped with Ag at different concentrations

With regards the triple point of water VSL, TUBITAK, SMD and GUM (in the frame of a RMG) worked together. For the first time a good consistency between a) the results of two independent ICPMS analysis and b) the impurity calculations based on the gravimetric data of the prepared mixtures and manufactured cells, was obtained. A re-sealable triple point of water cell was constructed allowing unprecedented flexibility in cell doping experiments: the re-sealable cell can be re-evacuated and measured in between repeated doping without the need of glass manufacturing. A photograph of the re-sealable cell is showed in figure 2. The cell has shown results over the expectation: stability over one month and differences of the order of 20 μK with respect the triple point of water temperature proving that this new concept of fixed point cell is

feasible, offering clear advantages with respect to permanently sealed cell, and a cost-saving approach, when doping experiments are needed to investigate the effects of both impurities and isotopes.

Continuing with the studies of the triple point of water, the impact of the oxygen isotope content on the temperature of the triple point of water has been investigated by VSL and GUM. The work aimed at finding new isotopic depression constants, which allow the reduction of uncertainty in the isotopic correction and the overall uncertainty in the realization of the triple point temperature.

The uncertainties due to thermal effects on the realisation of the ITS-90 defining fixed points correspond on average to 20 % of the total uncertainty but this can be >75 % in the case of aluminium. Consequently the improvement of our understanding of thermal effects is essential to achieve a reliable estimation of the uncertainty arising from them and where possible to minimise this uncertainty. The approach adopted to achieve this objective was twofold; Firstly models have been developed to describe the time evolution of the phase transformation (essentially the evolution of the solid-liquid interface) as a function of given theoretical assumptions and given parameters such as furnace thermal gradients, freezing initiation, type of furnace, furnace thermal gradients, ambient temperature and insulation. Secondly the validity of the adopted models will be experimentally verified by assessing the impact of the selected parameters on the corresponding observed melting curves. These works were undertaken by NPL, CEM, CMI, UL and UC.



Figure 2. The re-sealable triple point of water cell

One of the most relevant results obtained in this study is the development of a phase-field model that does not require explicit tracking of the phase boundary. It can therefore be used to study the morphology of the phase boundary as a function of time. The model handles all important physics associated with solidification of very pure metals, and has 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. The applicability of the Scheil impurity correction technique and a clear idea of its range of applicability are emerging. A set of data on typical Al fixed point cells has been accumulated, which is complete enough to compare the various impurity correction methodologies. 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 advances the state of the art by highlighting the strengths and weaknesses of all the available techniques. Ultimately the uncertainty associated with the correction will be better characterised at National Metrology Institutes and primary calibration laboratories. A side-benefit of the impurity correction work is the accumulation of a vast database of liquidus slopes (giving the effect of a given impurity on the freezing temperature of the fixed point material). This has been published already and is being taken up by users around the globe. Figure 3 represents a time slice illustrating the development of cellular growth in a full 3D simulation. The interface location and temperature are shown (the colour scale

covers the range temperature range ± 1 mK). The simulation geometry considers a segment of the lowest 10 cm of the fixed-point cell, to compensate for significantly increased computation.

A new experimental method based on the difference of the electrical conductivity of solid and liquid metals has been developed by MKEH to allow the study of the solid fraction and melt fraction along the phase transformation in the case of high temperature fixed points. This new measurement technique will help in increasing the theoretical knowledge of the phase transitions of the fixed points, ultimately to obtain an accurate theoretical model. As part of this microscopic modelling of solidification of highly pure metals a survey of distribution coefficients has been performed and recently been published. This provides, for the first time, a clear picture of the overall trends in distribution coefficient for impurities across the periodic table. Finally, the transient heat conduction model and the spatial distribution model of eddy currents have been developed. This study has been carried out thanks to the developed two-zone impedance measurement and a targeted furnace-controller, increasing the duration of the temperature plateau from a few hours to an unlimited amount of time.

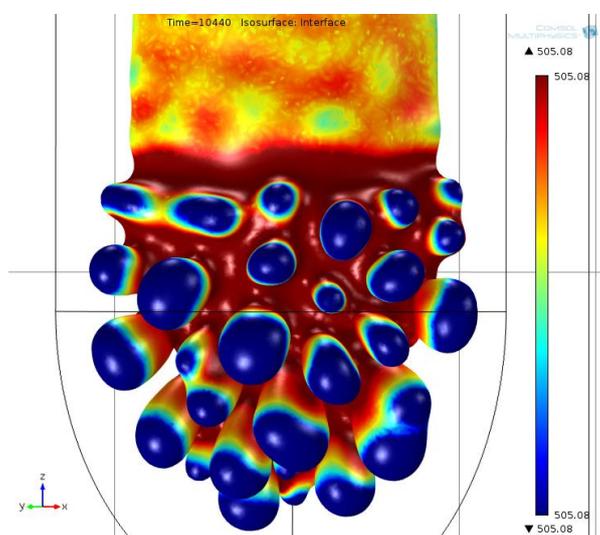


Figure 3. Example of a 3D simulation to study the thermal fluxes in the ITS-90 fixed points

Objectives 1b: Improvement of the calibration procedures of standard platinum resistance thermometers

To improve the calibration procedures of resistance thermometers several effects have been thoroughly studied, the influence of the calibration environment on the capsule type standard platinum resistance thermometers and the self heating effects, use of bushings, oxidation and annealing effects in the case of long stem standard platinum resistance thermometers.

The capsule type standard platinum thermometers are used as ITS-90 interpolation instruments at low temperatures for many years, and due to their compact form they are also often employed with thermodynamic thermometers such as acoustic resonators, to determine the difference between thermodynamic temperature T and ITS-90 temperature T_{90} . They have played a large part in the methods and equipment used to transfer the water triple point temperature during the determination of the Boltzmann constant. The calibration of these thermometers in conventional fixed point cells (used for long stem standard platinum resistance thermometers) is often performed in a very different environment to that when they are used in thermodynamic temperature sensors, so CEM, INRiM and INTiBS in the frame of a RMG have worked to characterise the influence of the environment: gas, oil, and vacuum on the capsule type thermometers performance. Different adapters to use these thermometers in the conventional cells have been constructed and used proving to be equivalent. The studies have demonstrated that the different media, even vacuum, do not increase dramatically the calibration uncertainty provided a good thermal contact is assured.

A dedicated instrument for integrated and non-intrusive direct measurement of bridge current (see figure4), already available, to measure the current of both AC and DC resistance bridges have been designed by UL. It will help to evaluate the uncertainty linked with the self-heating of thermometers when calibrating at the highest level of accuracy. This works are part of the optimization of procedures to calibrate standard platinum resistance thermometers. In this line, guidance for usage of metal bushings in order to estimate decrease of self-heating and increase of heat-loss, an experimental investigation of standard platinum resistance thermometers non-uniqueness in the range between 500 °C and 1000 °C or studies about annealing and oxidation effects for standard platinum resistance thermometers for old and new designs will be published soon in scientific peer review journals.

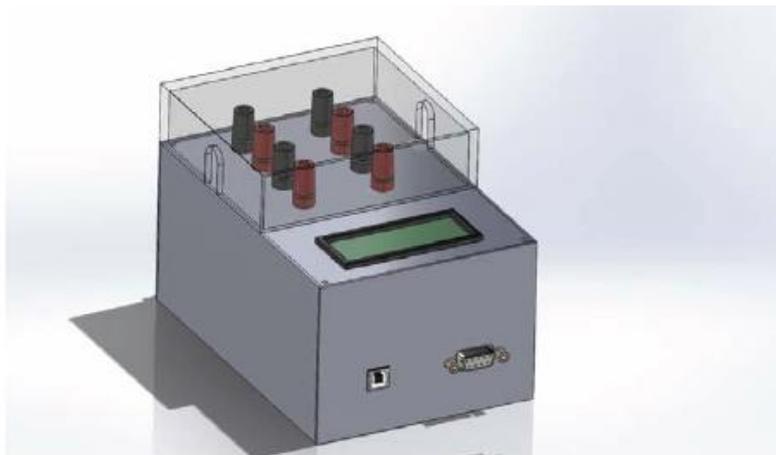


Figure 4. A dedicated instrument for integrated and non-intrusive direct measurement of bridge current

Linked to this development UL has thoroughly study the effect of bushings to improve the calibration procedures of platinum resistance thermometers. Different types of bushings (brass, copper and aluminum) and two main types of thermometers encapsulated in silica and inconel. It was concluded that despite the bushings can improve the thermal contact of the thermometer In the fixed point, Improving at the same time its repeatability, they have to be used carefully, for instance It was demonstrated that although copper bushings considerably reduces self-heating, they actually increases the temperature difference due to radiative heat loss. In all these studies UL has count on the collaboration of DMDM and MER in the frame of a RMG.

Objectives 1c: ITS-90 non-uniqueness studies

Continuing with the improvement of the calibration methods for standard platinum resistance thermometers, INRiM, CMI, IPQ and VSL have carried out the study of the ITS-90 non uniqueness. These thermometers, as interpolating instruments, need interpolating functions to relate temperature to resistance between the defining fixed points. However, the functional form of these equations does not take into account the many physical effects associated with real thermometers, and so the calibration equations are always subject to an interpolation error of the order of 0.5 mK. This so-called 'type 3 non-uniqueness' arises from the differences in interpolated values between fixed points between different thermometers. It affects all the ITS-90 subranges but it is particularly severe in the range between 660.323 °C – 961.78 °C. In this range there is very limited data on type 3 non-uniqueness because of the drift and oxidation of thermometers. The laboratories have performed measurements to evaluate this source of uncertainty by using some of the new fixed points mentioned before and gas controlled heat pipes (devices capable of generating a very uniform temperature zone inside their working volume). In addition a statistical study on historical data of a different standard platinum resistance thermometers from different manufacturers with calibration data between fixed points have been carried out. Figure 7 presents the results for the interpolation error of ten 25 Ω in the water-zinc ITS-90 subrange estimated by their historical calibrations using the value of the Ga fixed point.

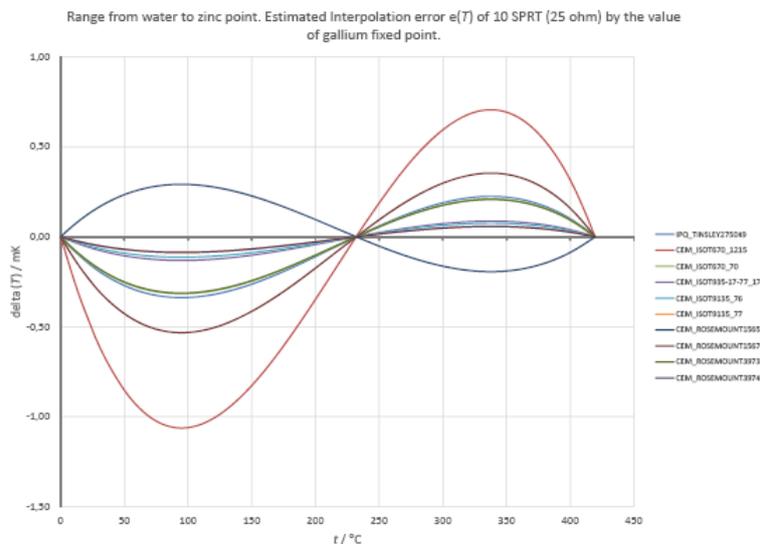


Figure 7. Estimated Interpolation error for a batch of standard platinum resistance thermometers.

Objective 1: Key research outputs and conclusions

This objective targeted towards the optimisation of the ITS-90 by increasing the knowledge on the defining fixed points and the improvement of the standard platinum resistance thermometers calibration procedures. Thanks to the experimental and theoretical works developed, the project has made available a large database where the influence of a given impurity on the ITS-90 defining fixed points is listed, allowing all kind of users (NMIs, manufacturers, etc) to perform the adequate correction to their fixed points. This result together with the new isotopic depression constants obtained, which allow the reduction of uncertainty in the isotopic correction and the overall uncertainty in the realization of the triple point temperature mean a significant step forward on the reduction of the uncertainties on the realization of the ITS-90 fixed points. The improvement of the standard platinum resistance thermometers calibration procedures achieved thanks to the studies on the effect of the use of bushings, oxidation and selfheating effects and the new data on the ITS-90 non-uniqueness have also contributed to the mentioned overall reduction of the ITS-90 uncertainties. In conclusion the attainment of this objective can be considered fully satisfactory.

3.2 Objective 2: New Methods to provide traceability to the kelvin

Objective 2a: New fixed points developed

New fixed point cells have been developed by CNAM, CMI, LNE and INTiBS in the frame of a RMG, namely Xe, CO₂, I₂ and Al-Cu eutectic, these new cells will enable studies to be performed on the possibility of improving the ITS-90 equation. The uncertainties on the realization of these fixed points range from 1 mK for the Xe and CO₂ up to 10 mK for the Al-Cu eutectic. The Xe triple point cell has showed a deviation of 1 mK with respect to the most accurate determination of the Xe triple point temperature performed up to now. Figure 5 presents the results of different realizations of a Xe triple point transition. The graph plots the variation in temperature versus the percentage of fraction of sample melted, F , where a good reproducibility of the plateaus can be observed.

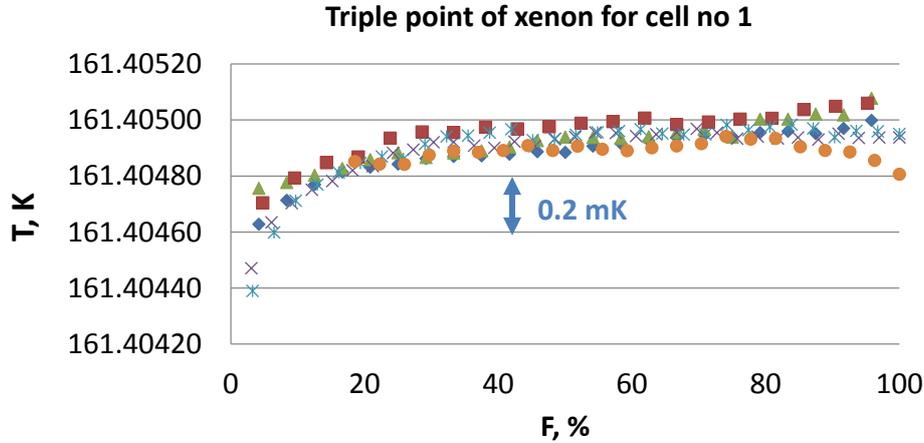


Figure 5. Different Xe triple point realizations.

Figure 6 shows from its part different realizations of the Al-Cu eutectic transition on one of the fixed point cells developed. It is important to mention that these cells have been also used in the ITS-90 non-uniqueness studies that are mentioned below.

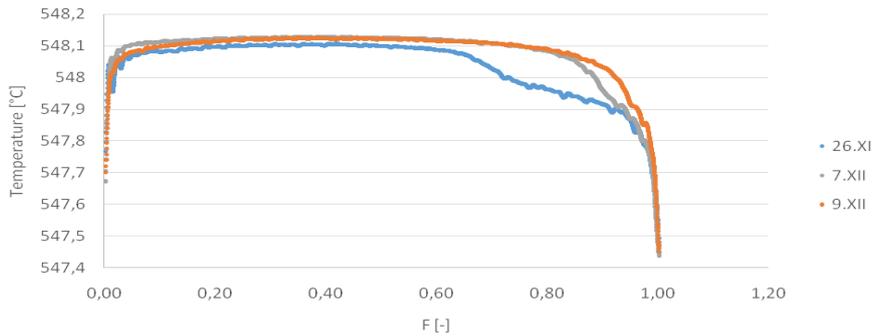


Figure 6. Different Al-Cu eutectic transition realizations.

Objective 2b: Approximations to the kelvin in the range from 500 °C to 1000 °C:

This temperature range is especially problematic in the ITS-90 because the lack of reliability of the high temperature standard platinum resistance thermometer. Above the zinc freezing point (419.527 °C) and up to 1000 °C their best attainable uncertainties are of the order of several millikelvin. However, as described earlier, they prone to significant drift and robustness problems. Therefore the availability of new equipment have direct impact in reducing the uncertainties in the temperature dissemination. The partners have tackled the problem developing new facilities and instruments for radiation thermometry and by using vapour pressure temperature scales.

CNAM has designed and constructed a new near infrared radiation thermometer with a tuneable operating wavelength, specifically engineered to facilitate laser calibration of its absolute spectral radiance responsivity. The calibration of this instrument is much simpler compared to filter radiometer based techniques as the wavelength is spectrally tuneable. This new type of radiation thermometer has been characterised and it proved to be more stable than a conventional, optical grating based spectroradiometer as it does not use any moving mechanical parts. The main features of this radiation thermometer are the following:

- wavelength range: 1.2 µm to 1.8 µm
- filter width (FWHM): 10 nm
- transportable (90 cm x 30 cm x 20 cm, < 20 kg)

- target size: 2.3 mm (diameter)
- working distance 740 mm

Due to the use of a tuneable filter device, no optical interference pattern (as is the case for fixed wavelength interference filters) is observed when spectral characterization with a narrow band laser system is used. Therefore for the calibration of the spectral responsivity and the characterization of the RT in terms of achievable signal/noise ratio, out-of-band blocking and reproducibility of wavelength and signal amplitude a tuneable external cavity diode laser (ECDL) can be applied. The characterization of its optical performance lead to the following results:

- signal-to-noise ratio: $5 \cdot 10^{-4}$
- out-of-band blocking level: several 10^{-5}
- center wavelength stability of the filter: 5 pm / 60 hours
- amplitude stability (max. of filter transmission): $2 \cdot 10^{-3}$ / 60 hours
- signal stability (spectrally integrated): $1.3 \cdot 10^{-3}$ / 60 hours

The relative spectral responsivity of the radiation thermometer was calibrated by means of an extended cavity, wavelength tuneable, feedback stabilized laser in conjunction with an integrating sphere (Lambertian source), applying two independent methods: fixed wavelength / wavelength tuning of the laser and tuning of the wavelength / fixed wavelength laser. The relative difference between the two methods was found to be below $5 \cdot 10^{-3}$. Figure 8 shows a schematic picture of this brand new radiation thermometer.

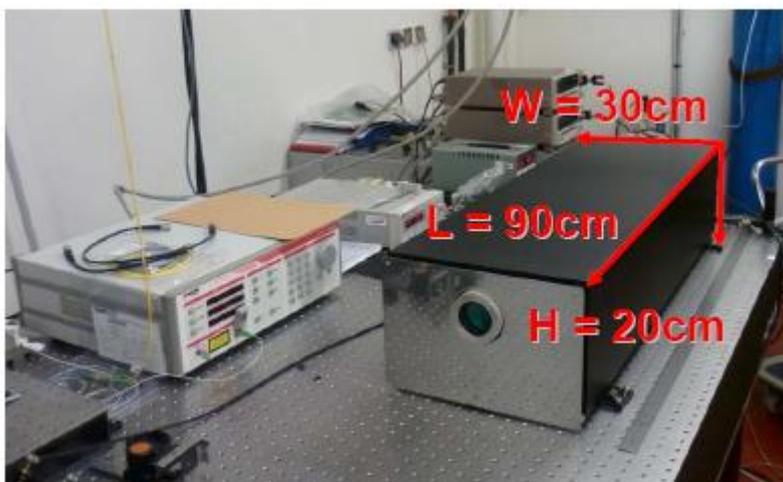


Figure 8. The tuneable near infrared radiation thermometer.

To assess the range from 500 °C to 1000 °C with absolute radiation thermometry requires radiation thermometers operating in the near infrared wavelength range. These types of radiation thermometers are routinely calibrated according to the ITS-90 via standard platinum resistance thermometers/blackbody based facilities. At present, nobody performs a detector based characterisation of these devices in the near infrared wavelength range, in terms of their absolute radiance responsivity i.e. thermodynamic temperature. This activity, carried out by PTB, has established beyond state-of-the art techniques and facilities enabling absolute radiometric characterisation of near infrared radiation thermometers to be applied for the approximation and dissemination of the kelvin below 1000 °C. The radiometric characterisation procedure included the determination of the key parameter for thermodynamic temperature determination, the absolute radiance responsivity, the radiation thermometer short term stability and the investigation of the linearity within the dynamic range of the radiation thermometers. Figure 9 presents a picture of the developed facility for the spectral radiance responsivity characterization.

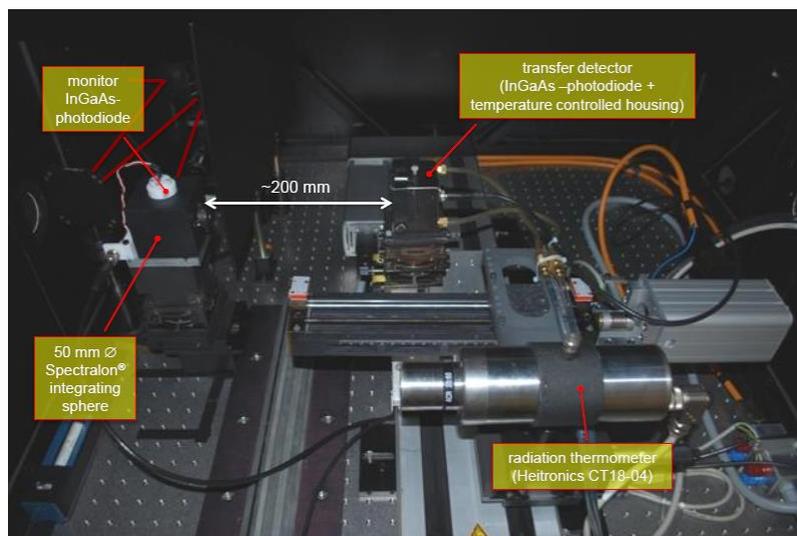


Figure 9. Developed facility for the spectral radiance responsivity characterization.

For the first time in radiation thermometry, the absolute radiometric calibration of an imaging type, single detector radiation thermometer (RT) operating in the Near Infrared (NIR) wavelength range was successfully completed and validated. The achieved relative standard uncertainty of the calibrated spectral radiance responsivity is 0.1 %, being comparable to the achievable uncertainties with non-imaging filter radiometers at the highest metrological level in this wavelength range. As NIR-RT are widely used in industry in the temperature range below 1000 °C, the developed instrumentation and calibration procedure allows, alternatively to the ITS-90, a direct thermodynamic temperature determination in this temperature range. Moreover, the performance of the newly developed continuously tuneable infrared pyrometer were evaluated and showed satisfactory capabilities though the signal-to-noise that is considered too large and requires the replacement of the photodiodes.

Recent studies on the uncertainties of temperature measurements using high temperature standard platinum resistance thermometers have shown that, at present, the overall uncertainty attainable is close to 10 mK over the entire range between the Al (660.323 °C) and Ag (961.78 °C) fixed points. Vapour pressure temperature scales could be used to fill this temperature range by continuous thermodynamic phase transition curves, ideally with a significantly reduced uncertainty. There is a good agreement between the measurements of the immersion characteristics in a mercury Gas-Controlled Heat Pipe and the Clausius-Clapeyron profile for a column of Hg vapour. This raises the possibility of using coupled Gas-Controlled Heat Pipes to thermodynamically relate two different temperature ranges, with the possibility of redefining one temperature range in terms of another: this is the “Temperature Amplifier”. The ITS-90 could therefore be defined in a new way: any accurate reproducible temperature range may be used to generate another temperature interval, ranging between 660 °C and 962 °C, with the relationship between the two ranges being thermodynamically related by the two substances used for the vapour-liquid transitions in the Gas-Controlled Heat Pipe. With this objective in mind INRiM has designed and constructed a new “Temperature Amplifier” system based on two coupled gas-controlled heat pipes of new generation, manufactured for the purpose under INRiM design, taking advantage of a 20 years experience achieved on this technology. Figure 10 presents two graphs where the performance of the developed Temperature Amplifier can be observed in terms of its temperature stability with time and the differences among the thermometric wells.

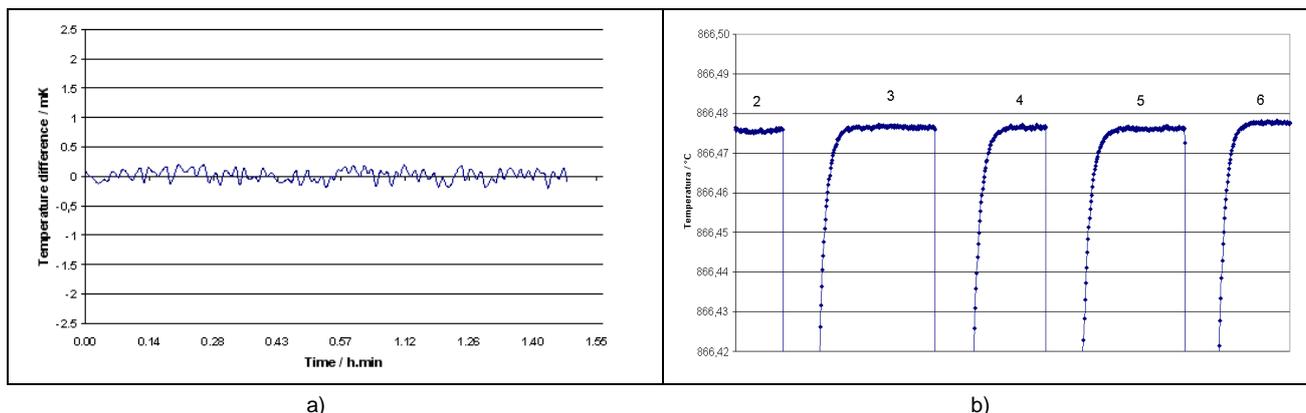


Figure 10. Performance of the “Temperature Amplifier” a) temperature stability b) differences in temperature among the different thermometric wells.

This new Temperature Amplifier represents a significant improvement in the present technology between the Au and Ag fixed points. It is intended as a commercially available device for accurate temperature measurements and calibration. The special dedicated pressure controller adopts a fundamental thermodynamic principle to direct control pressure (therefore temperature) through a temperature value and set point. This last is a further progress beyond the state of the art not limited to temperature metrology but extended to pressure control.

Objective 2c: Alternatives to standard platinum resistance thermometers

As it was mentioned several times above, high temperature standard platinum resistance thermometers are defined as standard interpolating instrument of the ITS-90 in the temperature range between 660.323 °C and 961.78 °C. Their susceptibility to contamination, lack of stability, and poor repeatability are well known problems so it prevent its use in industry. Modern techniques for the production of wires of pure noble metals provide the possibility of using Au/Pt thermocouples as potential replacements for the resistance thermometers. Au/Pt thermocouples have a high sensitivity (Seebeck coefficient) which is about a factor of two larger than that for Pt/Rh alloyed thermocouples, and they have better thermoelectric homogeneity and stability. Furthermore, they are much more robust, are more easily manageable thermometers, and are much cheaper than the resistance thermometers. The CCT recommended further research on this type of thermocouple as a potential candidate for an interpolating thermometer between the aluminium and silver fixed points and has highlighted the need for comparisons between Au/Pt thermocouples and high temperature standard platinum resistance thermometers. To this end several partners joined their efforts: PTB, CEM, CMI, NPL, TUBITAK together with IMBiH, GUM, SMU, DMDM and BRLM in the frame of RMGs.

The reference functions published in the international standard IEC 62460, that specifies the equations and reference tables relating the temperature to EMF (electro-motive force) for Au/Pt and Pt/Pd thermocouples were based on a single investigation of only one group of Au/Pt thermocouples. The problem nowadays is that the thermoelectric behaviour of wire from different batches/providers exhibits enormous variation, and it is necessary to develop and validate a new reference function which better reflects the properties of commercially available Au and Pt wire, to improve their repeatability and to reduce the measurement uncertainty. This requires the investigation of a number of different thermocouples, with proven stability and homogeneity, constructed with wires from different sources and with different assembly techniques than has been made possible thanks to the combination of skills and capabilities of the partners and the mobility grant researchers.

These works have led to some unexpected conclusions. Measurements performed with different sets of Au/Pt thermocouples constructed with the most pure elements available nowadays has reinforced the notion that the present reference function of Au/Pt thermocouples is not applicable to currently available thermocouple wires (see figure 11). Furthermore, a comparison study on the performance of the Au/Pt thermocouples versus high temperature standard platinum resistance thermometers has shown on a systematic basis the relative merits of the two types of sensors. It is clear that in general the high temperature resistance thermometer is more repeatable, provided it is not moved. If it is moved, the Au/Pt thermocouple is more reproducible.

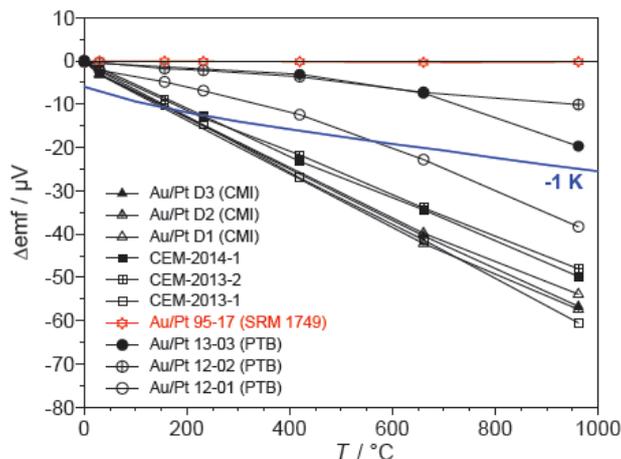


Figure 11. Difference of Au/Pt thermocouples with respect the reference function.

Thermometers based on the sapphire whispering-gallery-mode resonator are also good candidates to overcome the mechanical instabilities of platinum resistance thermometers in industrial applications. The intrinsic temperature dependence of the refractive index (or its near equivalent, the permittivity) of synthetic sapphire, coupled with the ease of measuring the frequencies of high-Q resonant modes, allows the use of a sapphire whispering-gallery-mode as a thermometer rather than a frequency standard. Compared to platinum, synthetic sapphire is thought to be mechanically stable up to 1800 °C and less subject to changes in physical state (e.g., oxidation, growth of crystal defects). Due to the inherent stability issues associated with platinum thermometers, a sapphire whispering-gallery-mode thermometer represents a potential replacement in industrial applications where measurement uncertainty below 10 mK is required.

INRiM has collaborated with POLITO in the field of whispering gallery thermometry. A new prototype of this kind of thermometer has been constructed (see figure 12) showing a temperature resolution better than 1 mK and a temperature stability at 0 °C better than 3 mK. This is a promising result showing the way for considerable improvements in electromagnetic thermometry methods.

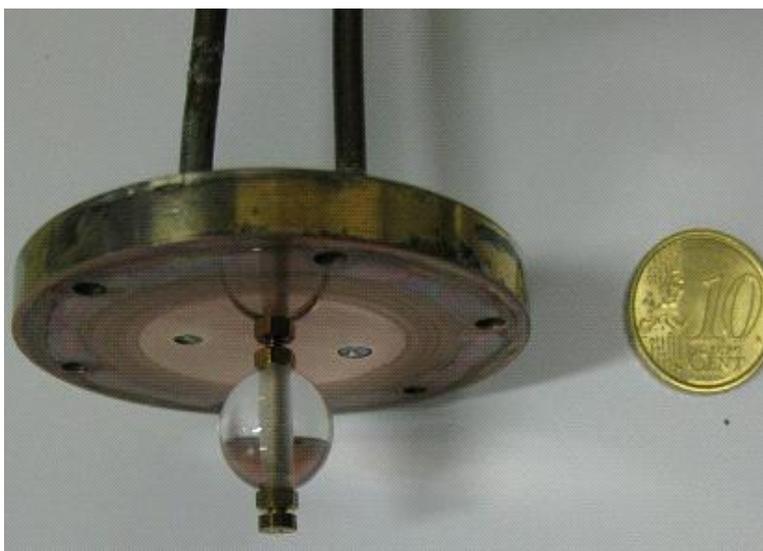


Figure 12. Detail of the whispering gallery mode thermometer constructed

To complement this thermometer, a low-cost (prototype cost around 5000 €) portable (weight less than 3kg, volume around 10 dm³) vector S2-meter covering the 13 GHz – 19 GHz frequency range has been designed realized and tested. The realized electronic system has been validated by POLITO against a commercial state-of-the-art VNA in measuring the resonances of a sapphire whispering gallery thermometer, assessing measurement accuracy and stability at the ice melting point. The final set-up composed by the resonator and

the developed instrument (see figre 13) has been validated at INRiM as thermometer against standard platinum resistance thermometers over a 100 K temperature range.

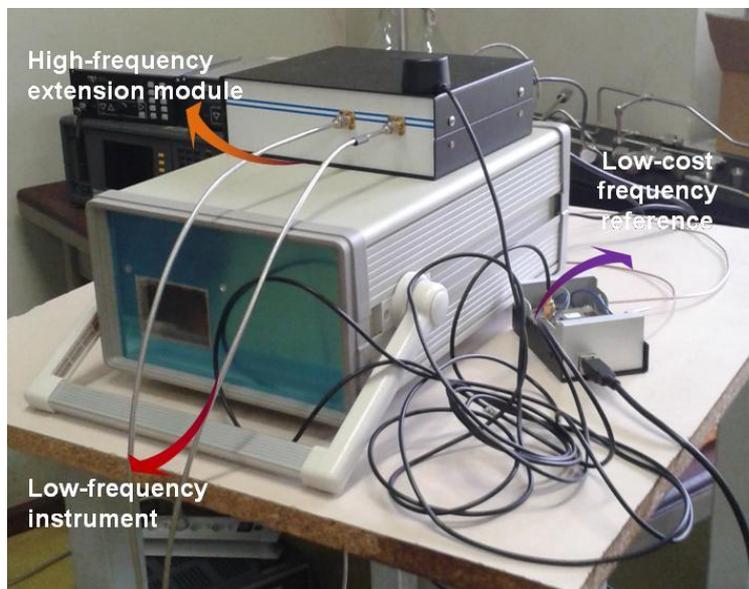


Figure 13. The low-cost, portable vector S2-meter constructed

Two different concepts of acoustic thermometer prototypes have been design and constructed with the main objective of enabling direct calibration of long stem standard platinum resistance thermometers to the thermodynamic temperature for the first time.



Figure 14. Acoustic resonator for the direct calibration of standard platinum resistance thermometers.

INRiM has design and develop a practical standard acoustic standard for thermodynamic temperature. Ideally, such instrument maintains the accuracy already demonstrated by primary acoustic thermometers (of the order of 5 ppm) though it is drastically simplified in its design, easier and quicker to operate and reduced in cost. The instrument is based on a sealed cavity filled with pure and calculable helium, operating over an extended temperature range (from the mercury fixed point up to the indium point, i.e. from $-38.834\ 4\ ^\circ\text{C}$ to $156.598\ 5\ ^\circ\text{C}$) and is suitable for direct thermodynamic calibration of different types of thermometers. The prototype was made in copper as a triaxialellipsoid with an internal volume of $260\ \text{cm}^3$ and a thick wall to minimize shell coupling. The cavity is designed to be vacuum- and pressure tight. The excitation of acoustic and microwave resonances is made by waveguides. It has embedded thermometer wells for capsule type and long stem standard platinum resistance thermometers. The temperature range of tests is from 230 K to

430 K with an accuracy: ± 5 ppm. Figure 14 shows a photograph of the resonator with the thermometer well around the central cavity.



Figure 15. Acoustic "dry block" calibrator.

From its part NPL has designed and built an acoustic device which can be used practically retaining many of the advantages of acoustic thermometry i.e. sensitivity and a simple physical principle of operation. A prototype of such a device, specifically designed for nuclear environments was developed the EMRP JRP ENG08 Metrofission, however that device covers a different range and is not suitable for calibrating standard platinum resistance thermometers. A demonstrator acoustic "dry block calibrator" prototype for calibrating/comparing conventional contact thermometers with an acoustic technique in the range 0 °C to 100 °C has been constructed and characterize. Figure 15 shows the picture of the device.

Objective 2: Key research outputs and conclusions

This second main objective of the project has been satisfactorily achieved. New fixed points with competitive values for their repeatability and reproducibility have been constructed. New instruments such as the tuneable NIR radiation thermometer, the new temperature amplifiers generation, and the whispering gallery thermometer together with the low-cost, portable vector S2-meter are now a reality thanks to the project. Furthermore, within this projects the first attempts to develop artefacts and methods to allow the calibration of long stem standard platinum resistance thermometers in terms of the thermodynamic temperature have been carried out. At the moment the reproducibility and the repeatability are not comparable to that of the ITS-90 but this has been really the first step towards the absolute calibration of this kind of thermometers.

4 Actual and potential impact

The outputs of this JRP have optimised the realisation of the ITS-90 and provided new methods and alternative instruments and procedures to provide traceability to the kelvin, NOTED has had a direct high impact in the thermometry community formed by all the NMIs and the laboratories (usually accredited) which represent the route for dissemination to European organisations, industries, etc. by the calibration of different high level types of thermometers. It has also had direct impact in the scientific community and industry that need to perform high accuracy temperature measurements.

The work on impurity corrections (objective 1.a) and on the triple point of water has been discussed within different CCT working groups and the findings have already appeared in the revised BIPM document "Supplementary Information for the ITS-90". This document is essential in the current practice of high level fixed point thermometry. The formal text of the ITS-90 gives just a concise definition of temperatures. To accompany the formal text, supplementary information concerning the practical realization of the scale is published by the BIPM in the mentioned monograph which provides details in a comprehensive form. In particular, the chapter related to fixed points that were updated in 2015 contains some of the project outputs. All National Metrology Institutes all around the world use this document as the basic information to develop their calibration procedures. Even during the development of the project while the results on impurities corrections were being obtained some manufactures and laboratories started to use the tabulated distribution coefficients obtained for informing/validating the corrections for the influence of impurities in the ITS-90 fixed points they developed.

The evaluation of thermal effects (objective 1.a), and phase transition modelling in the defining fixed points significantly reduce their associated uncertainties directly benefiting primary calibration laboratories. The works developed in the project have demonstrated that computational fluid dynamics is a valuable tool to develop models that describe the time evolution of the phase transformation (essentially the evolution of the solid-liquid interface) as a function of given theoretical assumptions and given parameters (furnace thermal gradients, freezing initiation, ambient temperature and insulation). These models allow the design of optimal furnace conditions, cells and initiation techniques in a cheaper and faster way allowing to reduce the experimental procedures to the minimum required to realize the fixed points.

The ITS-90 non-uniqueness problem (objective 1b) arises from using different interpolating thermometers of the same kind, calibrated at the same fixed points and using the same interpolating equation, and manifests itself as differences, at a given thermodynamic temperature, in temperature values indicated by these thermometers. The failure of thermometers to indicate the same temperature (within experimental uncertainty) is due to the characteristics of different standard platinum thermometers and it is considered a problem of the scale itself, since it cannot take into account any variation in the behaviour of the thermometers. So this is a source of uncertainty to be taken into account when calibrating thermometers in the ITS-90 temperature ranges and sub-ranges. Its evaluation is based on literature i.e. published studies made on different thermometers (from different manufacturers, different designs, etc). But as it was pointed out by the CCT there were still a shortage of good quality data to assess the magnitude of the effect. The project consortium has contributed to produce new data, published in peer review journals and presented in congresses, which allow a more reliable assessment of this source of uncertainty.

To reach the objective of optimising the realisation of the ITS-90 the consortium has also worked on the improvement of the calibration procedures for standard platinum resistance thermometers (objective 1.c) to better account for the influence of the environment on capsule type thermometers and oxidation and self-heating effects, annealing, use of redundant fixed points on uncertainty propagation and bushings for long-stem SPRTs. The publication of the self-heating studies and the guidance for usage of metal bushings to estimate decrease of self-heating and increase of heat-loss is being followed by the thermometry community and the methodology implemented in the routinary technical procedures. In addition a dedicated instrument for integrated and non-intrusive direct measurement of bridge current has been developed and it is available for purchase.

In summary the project consortium has significantly contributed to reduce the three most important uncertainties on the dissemination of the ITS-90 and to improve the standard platinum resistance thermometer calibration procedures. The direct beneficiaries are the National Metrology Institutes which substantially can improve their capabilities and reduce their uncertainties what benefits their external customers in turn, helping them to remain competitive.

The new fixed points developed (objective 2.a) provide additional fixed points in temperature ranges where large temperature gaps currently exist between established fixed points, specifically between the triple points of argon and mercury and in the water-indium and aluminium - silver subranges, thus reducing uncertainties. The procedures for the construction of such fixed points and their performance have been published in peer review journals and in international congress so they are publicly available and can be implemented by manufacturers and other National Metrology Institutes. It is important to highlight the research carried out to look for potential alternatives to the mercury triple point (namely CO₂ and S₆F₆). The use of heavy metals, like mercury, entails an important risk for environment protection. European regulations are becoming progressively stricter due to the damage that mercury can cause in the environment. Despite the fact that the

quantity of mercury used in a fixed point cell is not very high (1 kg to 3 kg) there is always some risk of accidental spillage because of this the availability of an alternative benign fixed point will have clear environmental benefits. The results obtained in the project represent a further development on the final replacement of the mercury as one of the fixed point substances.

Infrared radiation thermometry (objective 2.b) has also undergone a significant step forward thanks to the works of the project consortium. For the first time a detector based characterisation of the radiation thermometers operating in the near infrared range (this corresponds to a temperature range from 500 °C to 1000 °C) has been developed. New facilities and new calibration services are now available establishing beyond state-of-the art techniques and facilities. This enables absolute radiometric characterisation of radiation thermometers to be applied for the approximation and dissemination of the thermodynamic temperature from 500 °C 1000 °C.

Furthermore a new radiation thermometer (objective 2.b) with a tuneable operating wavelength is now a reality, the first prototype has been constructed within this project and its performance published in international congresses. It has been specifically engineered to facilitate laser calibration of its absolute spectral radiance responsivity. It is remarkable the interest demonstrated by the glass industry in this new instrument.

The practical acoustic thermometers and the sapphire whispering gallery model resonator thermometer (objective 1.c) developed and evaluated within the project are the first step to enable the practical direct calibration to the thermodynamic temperature, which is not currently possible, at considerably lower cost. A key feature of this is the elimination of uncertainty arising from interpolation of the calibration between fixed points, which currently represents a large contribution to the uncertainty. Both instruments are of interest to users of sensors in the low-to-medium temperature range and will help to address difficulties experienced in the space industry, where accurate measurement of temperatures is required from -200 °C to about +200 °C, particularly for earth observation satellites and large space simulators, and where the currently used capsule and industrial platinum thermometers exhibit large uncertainties due to the large temperature range of interpolation, particularly -189 °C (argon triple point) to -39 °C (mercury triple point). Some European companies have demonstrated interest in the practical acoustic thermometry technology.

The thorough study performed on the Au/Pt thermocouples (objective 1.c) has been reflected in a peer reviewed paper and in a report that has been sent to the CCT and that will be taken into consideration in its 2017 meeting. It is foreseen that these results will be included and referred in the future update of the Guide on Secondary Thermometry published by the BIPM. This document provides guidance for thermometry at lower levels of accuracy. This guide should enable temperature measurement with greater convenience and lower cost than the more formal and primary realizations described in the *Mise-en-Pratique* for the kelvin. This guide, together with the previously mentioned "Supplementary Information for the ITS-90", are the cornerstones in which the current practice of thermometry all around the world is supported. . In addition discussions with European and non European sensor manufacturers have taken place regarding the behaviour of the Au/Pt thermocouples versus high temperature standard platinum resistance thermometers.

It is also important to mention the early uptake of the dedicated electronics (objective 1.c) for the temperature control of fixed point furnaces and for the very accurate temperature control required in innovative primary thermometry devices. European manufacturers contacted with the consortium and an exchange of information about the design and performance of this new electronics took place.

In addition to this direct impact there will be a substantial and moderate impact at environmental, financial and social levels. For instance, the ITS-90 temperature range between the mercury triple point and the freezing point of indium (38.834 4 °C to 156.598 5 °C) is crucial in the climatic data measurement. The very high accuracy required and the global nature of the measurements makes it essential that traceability of atmospheric and ocean temperature measurements performed worldwide is at a level suitable for meaningful measurements and data pooling. The improvement of the realisation of the fixed points in this range and the availability of new ones allowing the reduction of the interpolation uncertainties will allow increased reliability in the assessment of the climatic change.

The step change improvements achieved will substantially improve the capabilities and reduce uncertainties of European NMIs, many of which have substantial turnovers, as mentioned before. This will enable enhanced competitiveness of these NMIs with competitors outside Europe. This will benefit their external customers in turn, helping them to remain competitive. -European manufacturers bidding for high value tenders to supply metrology equipment that need to have lower calibration uncertainties from European

NMIs to be competitive in the global market will be also direct beneficiaries of the new capabilities and reduced uncertainties. Most industrial processes are monitored and controlled by measuring temperature and abnormal variations in temperature can be an early indicator of an impending failure of a device or process. These facts have resulted in temperature control and sensors (within the temperature range covered by this project) finding applications in many different industrial sectors such as automotive, consumer electronics, process industries, pharmaceuticals, food and beverage among others. The optimisation of temperature monitoring by the reduction of the uncertainties or the development of better equipment/sensors for temperature sensing facilitates the minimisation of energy use. This not only benefits the environment but also the company's finances by reducing production costs, minimising product rejection and improving product quality all of which will improve industrial competitiveness.

The temperature range covered by the project impacts some sectors that have a direct relationship with society like pharmaceuticals, and food processing and preservation industries. Any improvement in the temperature control and monitoring will improve the public safety, for instance with regards of the food transport (cool chain) or the use of medicines that need to be stored in a temperature controlled environment (e.g. vaccines).

The consortium has also made a big effort on dissemination the project outputs in order to increase the impact at maximum. The most remarkable results have been published in metrology related peer review journals like Metrologia, Measurement or International Journal of Metrology and Quality Engineering. Other scientific journals not directly related to metrology have been also targeted as IEEE Transactions on Instrumentation and measurements or Journal of Heat and Mass Transfer. At the moment of writing this report the consortium has 20 publications and at least 4 more have been submitted. A Ph D and a B Sc thesis were developed also in the frame of the project.

Continuing with the publications, some papers have been also published in journals addressed to a wider range of technical readers. Journals like e-medida, that is widely read in Latin America or Design products & applications magazine and Comsol news have been chosen in addition to newsletters like the one published by the IMEKO TC12 committee or Espaço, a Portuguese publication also widely read in Latin America.

Two workshops were organized during the life of the project. The first one (June 2012) took place in Tres Cantos (Spain) and it was addressed to stakeholders. The aim of this workshop was to give an overview of the project and to encourage the stakeholders to join the project as collaborators. As a consequence several "Exchange of Letters" took place with other NMIs and manufacturers and contracts for Research Mobility Grants were planned. Close to the end of the project (may 2015) a final workshop was also organized in Brussels. It was focused on presenting an overview of the most significant results obtained. It was also aimed to be an advanced training based on the project outputs. There were 50 attendees from 28 countries (see figure 16). All the presentations can be freely downloaded from the project web page that is still working.



Figure 16. NOTED Final workshop

Special care was taken to maintain up to date the EURAMET Technical committee for Thermometry because of the European National Metrology Institutes are the main stakeholders of this project. A brief report was made in all their annual meetings, and during the one of 2015 in Lisbon there were a special half day workshop. The CCT and their working groups have been also keep up to date as it was mentioned before.

Training has been also a key impact point of the project. A 1-day training course was organized in Brussels in May 2016 (see figure 17). It was addressed to new recruits of (contact) primary thermometry laboratories in Europe. Topics included practical and theoretical aspects, like uncertainties related to the realization of the fixed points or thermocouple thermometry. The course was based in some of the main and more practical improvements achieved in NOTED, the classes were given by some of the more recognized and respected scientist in the field. The course attendance was limited to 15 participants but due to the large number of requests it was extended to 22.



Figure 16. NOTED training course

One of the most successful stories of NOTED is the big number of Research Mobility Grants (RMG) and Early Stage Research Mobility Grants (ESRMG) undergone within the life of the project. A Researcher Mobility Grant provides financial support for a researcher at any stage of their career, to undertake research activities relevant to the JRP objectives aiming at increasing the capability of the European metrology researcher community. From their part, the Early Stage Research Mobility Grants provides financial support for a researcher with less than 4 years experience aiming to ensure sustainability of cooperation between the National Metrology Institutes and Designated Institutes. In total CEM, CMI, CNAM, NPL, TUBITAK, UL, VSL and PTB have hosted 9 RMGs and 2 ESRMGs from 7 organisations (BRML, DMDM, IMBiH, iNTIBS, MER, MG and SMU) from 5 countries (Bosnia Herzegovina, Montenegro, Poland, Romania, Serbia and Slovakia) outside the project. This is really a big success that makes NOTED the project with the largest number of mobility grants, among the ones funded within the EMRP.

5 Website address and contact details

A public website has been open, where the main public deliverables have been made available for the end users and keep them informed about project meetings and events: www.notedproject.com

The contact person for general questions about the project, is Dr. Dolores del Campo, CEM (ddelcampo@cem.minetur.es).

The contact person for topics related to the reduction of uncertainties on the realisations of the defining fixed points is Dr. Dolores del Campo, CEM (ddelcampo@cem.minetur.es).

The contact person for topics related to the standard platinum resistance thermometers calibration procedures and temperature scale non-uniqueness is Jonathan Pearce, NPL (jonathan.pearce@npl.co.uk).

The contact person for new temperature fixed points for improved dissemination of ITS 90 and future International Temperature Scales is Fernando Sparasci, CNAM, (fernando.sparasci@cnam.fr).

The contact person for approximation of the kelvin in the range from 500 °C to 1000 °C is Dieter Taubert, PTB (Dieter.Taubert@ptb.de).

The contact person for exploring new methods to establish traceability to the kelvin is Graziano Coppa , INRiM (g.coppa@inrim.it).

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