

## A multi-institute european project for providing improved and simpler traceability to the kelvin

D. del Campo<sup>1,a</sup>, J. Bojkovski<sup>2</sup>, M. Dobre<sup>3</sup>, E. Filipe<sup>4</sup>, M. Kalemci<sup>5</sup>, A. Merlone<sup>6</sup>, J. Pearce<sup>7</sup>, A. Peruzzi<sup>8</sup>, F. Sparasci<sup>9</sup>, R. Strnad<sup>10</sup>, D. Taubert<sup>11</sup> and E. Turz6-Andr6s<sup>12</sup>

<sup>1</sup>Centro Espa1ol de Metrolog1a, Tres Cantos, Spain

<sup>2</sup>Univerza v Ljubljani, MIRS/UL/FE-LMK Ljubljana, Slovenia

<sup>3</sup>FPS Economy, DG Quality and Safety, Metrology Division, Brussels, Belgium.

<sup>4</sup>Instituto Portugu6s da Qualidade, Caparica, Portugal

<sup>5</sup>TUBITAK Ulusal Metroloji Enstitusu, Gebze, Turkey

<sup>6</sup>Istituto Nazionale di Ricerca Metrologica, Torino, Italy.

<sup>7</sup>National Physical Laboratory, Teddington, United Kingdom.

<sup>8</sup>VSL B.V., Delft, The Netherlands.

<sup>9</sup>LNE-Cnam, Paris, France.

<sup>10</sup>Cesky Metrologicky Institut, Brno, Czech Republic.

<sup>11</sup>Physikalisch-Technische Bundesanstalt (PTB), Berlin, Germany.

<sup>12</sup>Magyar Kereskedelmi Engedelyezesi Hivatal, Budapest, Hungary.

**Abstract.** In the framework of the European Metrology Research Program, NOTED is a multi-institute project focused on the development of new advanced techniques for providing improved traceability to the kelvin to support its wider and simpler dissemination to science, industry and, in general, every user of temperature measurements. The main objectives of the project are to develop simpler and cheaper primary thermometers, new robust high performance sensors, optimisation of calibration procedures and reduced uncertainties in temperature measurement. This paper gives an overview of the project and the different activities that are being developed.

### 1 Introduction

Temperature is one of the most frequently measured physical quantities in science and industry, with over two thirds of industrial processes monitored and controlled by temperature measurements. Abnormal variations in temperature can be an early indicator of an impending failure of a device or process. As a result temperature control and sensors find applications in many different industrial sectors such as automotive, consumer electronics, process industries, pharmaceuticals, food and beverage industries among others.

The optimisation of temperature sensors and methods to disseminate temperature have a direct impact in the quality assurance of the industrial processes, minimisation of energy consumption and improving, in general, the industrial competitiveness. In addition, after the foreseen new definition of the temperature unit, the kelvin, it will be necessary to establish practical links between the thermodynamic temperature  $T$  and the temperature defined according to the International

Temperature Scale of 1990 (ITS-90),  $T_{90}$ , that is currently used throughout the world to calibrate temperature measurement equipment. In the mid term future this scale will continue to be used, but it also has some limitations that need to be addressed.

EURAMET, being aware of these issues, is funding a multi-institute project in the frame of the European Metrology Program, which is jointly funded by the EMRP participating countries within EURAMET and the European Union. The project "Novel techniques for traceable temperature dissemination" (NOTED) will provide 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. NOTED is focused on the development of new advanced techniques for providing improved traceability to the kelvin to support its wider and simpler dissemination to the users, resulting in simpler and cheaper primary thermometers, new robust high performance sensors and reduced uncertainties in temperature measurement. The project has a website

<sup>a</sup> Corresponding author: ddelcampo@cem.minetur.es

(www.notedproject.com) where more details of the project and its different workpackages can be found.

## 2 Current methods for disseminating temperature

The internationally accepted methods to realize and disseminate temperature are currently the International Temperature Scale of 1990 (ITS-90) [1] and the Provisional Low Temperature Scale of 2000 [2]. These two scales are, at the moment, the only possibilities envisaged for the *Mise-en-pratique* for the definition of the kelvin [3] to perform practical measurements of temperature in accord with the International System of Units (SI) [4].

Although not impossible, direct measurements of thermodynamic temperature are difficult, very expensive and time consuming; primary thermometers based on a well-understood physical system are required that are not practical ways to disseminate the kelvin. The Temperature Scales provide an internationally accepted recipe for realizing temperature in a practical way. The *MeP-K* also contains the differences between thermodynamic temperatures and the ITS-90,  $T-T_{90}$ , together with their uncertainties. In consequence, the user can easily convert measurements obtained in terms of  $T_{90}$  to  $T$  and vice versa.

The *MeP-K* was created in 2006 by the Consultative Committee for Thermometry (CCT) as a mechanism to update and to expand the thermometric methods in common use without imposing the high costs of changing the International Temperature Scale, in order to come closer to the user needs. It is foreseen that in the near future primary methods for the direct realization of the thermodynamic temperature at high temperatures will be included together with the future definition of the kelvin in terms of the Boltzmann constant. But it will be also possible to include even indirect approximations capable of exceptionally low uncertainties (similar that that of the ITS-90) or increased reliability. In consequence the *MeP-K* means a greater flexibility in traceable temperature measurements.

Anyway, it is also foreseen that the ITS-90 will continue to be in use in the future because it is a practical, robust and reliable tool. But in the past years with the increasing accuracy of the instrumentation some issues have arisen and brought to light by the CCT itself and some National Metrology Institutes [5-8].

Therefore more research is necessary to solve these issues and to achieve the promised expansion and flexibility of the *MeP-K* demanded by industry and technology to allow CCT the inclusion of new methods, assuring compatibility with the ones in use: different implementations of the *MeP-K* must provide compatible measures to preserve the uniqueness of the definition of the standard. In this line this project is being developed with two main objectives:

- a) The development of new interpolation instruments and techniques and, through implementing practical primary thermometers, the calibration of Standard Platinum Resistance Thermometers (SPRT) directly to the new kelvin definition.
- b) To solve current outstanding questions related to the ITS-90 temperature fixed points to clarify the discrepancies in their realization thus facilitating a reduction of their uncertainty.

In the next sections a brief summary of the different technical activities are presented.

## 3 REDUCING THE UNCERTAINTIES OF THE ITS-90 REALIZATION

The ITS-90 is made up three elements: the defining fixed points (changes of state of pure substances like the triple point of water), the interpolation instruments and the interpolation equations. In the temperature range covered by the project, from -218 °C up to 1000 °C, the interpolation instrument is the SPRT and its interpolation equation are defined by the ITS-90.

With regards the fixed points the main problems lie in the uncertainties linked to their realization due to the impurities content (it is not possible to realise a fixed point cell with a 100 % pure substance) and the undesirable thermal fluxes due to the influence of the surroundings. The SPRTs, on the other hand, have a critical limitation in practical applications: their sensitivity to mechanical shocks, that changes the physical state of their sensing element, and prevents them from meeting the required measurement uncertainty. A change of several millikelvin at the triple point of water is not unusual, that is several times greater than the more common calibration uncertainties, causing unacceptable propagated errors.

The project has defined several technical activities aiming at solving these issues.

### 3.1. Study of the influence of impurities and thermal fluxes on the realization of the fixed points

Phase diagrams for low impurities concentration in the fixed point materials do not currently exist, so it is not possible to perform any corrections due to impurities in the fixed point temperatures. For the first time this project will attempt to deepen the theoretical knowledge of the phase transitions of the defining fixed points using different approximations ultimately to obtain accurate theoretical and numerical models. These will be used practically by the end users to compute corrections for impurities and uncertainties. The aim is to precisely quantify the temperature effect of some common impurities in the defining metal fixed points (mercury, gallium and aluminium) and the triple point of water. This information will enable the correction of the fixed point temperature to that of a purer system with a

consequent reduction in the uncertainty due to impurities. The uncertainties due to impurities correspond, on average, to about 20 % [9] of the total uncertainty. In the case of the fixed points materials available with the highest purities (Hg and Ga), the total uncertainty for impurities could be reduced up to approximately 40 %; in the case of the fixed points available with lower purities the reduction could be up to about 20 %.

The uncertainties due to thermal effects correspond on average to 20 % of the total uncertainty but this can be >75 % in the case of aluminium. The activities of this project will enable a reliable estimation of the uncertainty due to thermal effects and, where possible, its minimisation. The approach adopted to achieve this objective is twofold:

- Firstly models will be developed 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 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.

### 3.2. New and improved calibration procedures

For capsule type SPRTs there are currently no internationally agreed procedures for their calibration in conventional fixed point cells [7], the work in this project will lead to best practice recommendations on their calibration. This is important due to the fact that these thermometers are often employed with thermodynamic thermometers such as acoustic resonators, to determine the difference between thermodynamic temperature  $T$  and ITS-90 temperature  $T_{90}$ .

For long stem SPRTs the lack of new data on non uniqueness (differences in interpolated values between fixed points between different SPRTs), oxidation, strain effects and contamination, particularly for recently developed commercially available models prevents optimal calibration. These aspects will be comprehensively investigated in this work. Finally an analytical model for furnace optimisation, which does not exist at present, will be developed and will facilitate the improvement of optimum calibration procedures for SPRTs.

### 3.3. New fixed points

Some work has been carried out in the past on possible new fixed point candidates which could be used as future ITS defining fixed points or as substitutes of the existing ones (e.g. the need to replace the mercury point is pressing). In this project two issues are specifically addressed:

- a) the step change improvement of the most promising new fixed points (in order to obtain measurement uncertainties able to improve the present temperature scale)

- b) the development of new fixed points to fill the large temperature gaps that exist between some established defining fixed points.

The current SPRT interpolation scheme in the ITS-90 is Lagrangian, i.e. the interpolation equation has the same number of fitting parameters as calibration points. If additional high quality calibration points could be introduced then least squares fitting could be applied, reducing the propagated uncertainty by a factor of  $\sqrt{\rho/N}$  (in the case of evenly spread points, [10] where  $\rho$  is the number of fitted parameters and  $N$  is the number of measurements). For instance, in the temperature range from the argon point to the water triple point the availability of additional fixed points (for eg Xe or C2F6) could reduce the total calibration uncertainty by 50 %.

In this project several new fixed points are being investigated. In the temperature range below the triple point of water (0,01°C), the triple point of carbon dioxide (CO<sub>2</sub>, 56,558 °C), carbon dioxide liquid-to-vapour phase transitions above 56 °C, and the triple point of sulphur hexafluoride (SF<sub>6</sub>, 49.595 °C) will be studied as possible replacements for the triple point of mercury (-38,83,442 °C) and the triple point of xenon (Xe, 111,745 °C) and hexafluoroethane (C<sub>2</sub>F<sub>6</sub>, 100,07 °C) to fill the gap between the triple point of argon (-189,3442 °C) and mercury, the largest in the ITS-90. In the temperature range above the triple point of water the objective is to look for secondary points in the ITS-90 subranges covered will be the water – indium (0.01 °C to 156,598 5 °C) and aluminium – silver (660 °C to 961,78 °C). These will be the Iodine (I<sub>2</sub>, 114,7 °C) and the Al-Cu eutectic (548,16 °C)

## 4 ALTERNATIVES WAYS FOR DISSEMINATING THE KELVIN

### 4.1. Covering the range between 500 °C and 1000 °C

The temperature range from 500 °C up to 1000 °C is especially problematic in the ITS-90 because the lack of reliability of the high temperature SPRT due to its instabilities and the contamination problems. The project aims at developing and validating new procedures and equipment that provide uncertainties at a similar level to that of the high temperature SPRT, but have none of its drawbacks. It is envisaged that these new approaches could be included as alternative methods in the future *MeP-K*.

Above the zinc freezing point (419.527 °C) and up to 1000 °C the best attainable uncertainties [9] are of the order of several millikelvin using high temperature

SPRTs as interpolation instruments. Due to the drawbacks of high temperature SPRTs, particularly stability, the availability of new equipment will have a direct impact in reducing the uncertainties in the temperature dissemination.

Also planned is the development and characterisation of a new near infrared radiation thermometer and the development of a set up and the methodologies for the complete radiometric characterisation of selected InGaAs-detector based radiation thermometers, both intended for application in this temperature range and operating in the NIR wavelength range.

Another novel technique for the dissemination of the kelvin in this range is the use of coupled gas controlled heat pipes to thermodynamically relate two different temperature ranges, with the possibility of redefining one temperature range in terms of another. The relationship between the two ranges would be thermodynamically related by the two substances used for the vapour-liquid transitions in the heat pipes (Clausius-Clapeyron equations).

The third possible alternative is the Au/Pt thermocouple. The project aims to improve the performance of Au/Pt thermocouples, to challenge and supplant the high temperature SPRT, and to support the inclusion of Au/Pt thermocouples as interpolating instruments in the developing *MeP-K* that will increase the reliability of scale dissemination and the simplicity of its use.

#### 4.2. Novel practical primary thermometers

The project also included the development of practical primary thermometers (able to provide traceability directly to the thermodynamic temperature not to the ITS-90), which will be able to perform a calibration of SPRT directly to the new kelvin definition. It is planned to design and build an acoustic device which could be used practically whilst retaining many of the advantages of acoustic thermometry i.e. sensitivity and a simple physical principle of operation.

In addition a new thermometer based on the sapphire whispering-gallery-mode (WGM) resonator is being constructed. This new thermometer could overcome the mechanical instabilities of SPRTs in industrial applications. The temperature dependence of the refractive index of synthetic sapphire, together with the ease of measuring the frequencies of high-Q resonant modes, allows the use of a sapphire WGM as a thermometer rather than a frequency standard. The thermometer will initially cover the temperature range from -80 °C to 180 °C, but investigations will also be carried out to at least determine the additional requirements to extend the temperature range from -196 °C to 300 °C. The target uncertainty for the thermometer is 0,01 °C with sub-millikelvin resolution.

## 5. CONCLUSIONS

This paper presents a summary of the main technical activities developed in the project NOTED “Novel techniques for traceable temperature dissemination”. The project aims at resolving some of the most important problems associated with the thermometric methods in use and to provide a simpler dissemination of the kelvin to the end user. Detailed information can be found in the project web page [www.notedproject.com](http://www.notedproject.com).

## Acknowledgements

This research is included in an EMRP Joint Research Project jointly funded by the EMRP participating countries within EURAMET and the European Union.

## References

1. Preston-Thomas H., *Metrologia*, 1990, 27(1), 3-10
2. Procès-Verbaux du Comité International des Poids et Mesures, 89th meeting, 2000, pp. 63-64 (French version) and pp. 129-130 (in English).
3. [http://www.bipm.org/utis/en/pdf/MeP\\_K.pdf](http://www.bipm.org/utis/en/pdf/MeP_K.pdf)
4. [http://www.bipm.org/en/si/si\\_brochure/](http://www.bipm.org/en/si/si_brochure/)
5. CCT Workshop “Toward the ITS-XX”, Chicago, 25 October 2002, CCT Document CCT/03-01
6. D. R. White et al. *Int. J. Thermophys.* (2010) 31:1749-1761.
7. G. Machin et al. *Metrologia* 40 (2011) 68-69.
8. Bojkovski, J. et al. *International Journal of Thermophysics* vol. 30 issue 1 February 2009. p. 1 - 8
9. [http://www.bipm.org/cc/CCT/Allowed/24/D19\\_rev\\_WG3\\_Doc\\_rev\\_10July2009.pdf](http://www.bipm.org/cc/CCT/Allowed/24/D19_rev_WG3_Doc_rev_10July2009.pdf).
10. <http://www.bipm.org/cc/CCT/Allowed/21/CCT01-08.pdf>.