

Research highlights

New thermodynamic temperature references based on high-temperature fixed point (HTFP) cells

Realisation of new cells

Several NMIs have constructed and characterised new cells to be used during this project:

- CEM: 4 Fe-C (1153 °C) and 3 Pd-C (1492 °C) cells
- PTB: 2 Fe-C
- VNIIOFI: 5 Ru-C (1953 °C) cells
- NIM: 2 Pd-C cells
- NMIJ 1 Pd-C cell
- NPL: 2 Ru-C and 2 WC-C (2749 °C) cells
- LNE-Cnam: 2 Fe-C and 5 WC-C cells

The filling of the cells is performed in vertical furnaces (**Figure 1**) starting with high purity metals and graphite powder slightly below the eutectic or peritectic composition.

The filling process and the design of the cells was left free for each laboratory to define except for the dimensions of the cells (25 mm in diameter, 40-45 mm length) and the use of the hybrid design which were mandatory to ensure the exchange of the cells between labs and the robustness of the cells, respectively (**Figure 2**).

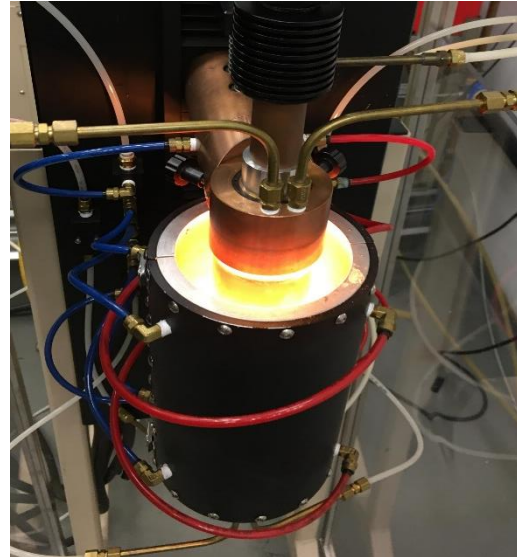


Figure 1: furnace for the filling of the cells (NPL)



Figure 2: set of 5 WC-C cells based on the hybrid design (sleeve and CC sheets) constructed at LNE-Cnam using the piston method

Thermal characterisation of the cells

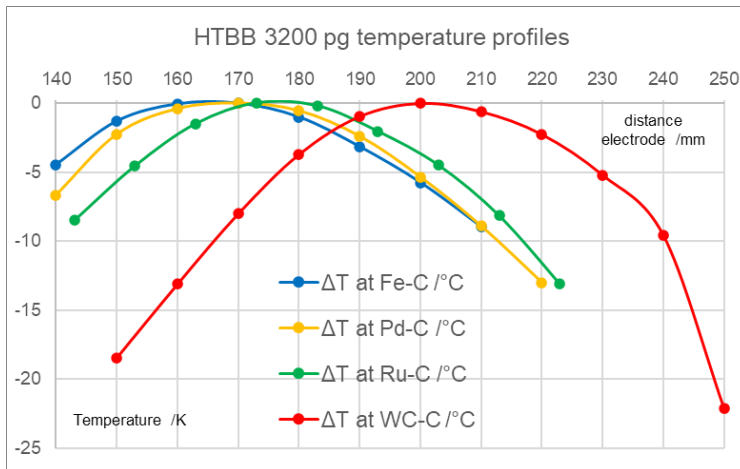


Figure 3: temperature distribution inside the high-temperature furnace HTBB 3200 PG determined at LNE-Cnam with the moving target technique

The characterisation of the cells included the assessment of the effects of thermal gradients on the temperature of the phase transition. This requires moving the cell inside the furnace so that it rests in different temperature distributions (**Figure 3**).

The first results show that the effect of temperature steps and temperature gradients are limited to a few tens of millikelvins in the case of cells with a high filling rate. In all cases, the lower the temperature gradient along the cell, the flatter the melting plateau.

Comparison and selection of the cells

The comparison of the cells is ongoing and should yield a batch of four cells at each HTFP: the best two of which will be used for the assignment of thermodynamic temperature and two others would be devoted to traceability tests towards industry or NMIs.

Next steps

A technical on-line meeting was held by the participants on March the 30th 2021. The progress of the workpackage activities is satisfactory given the particularly complicated conditions due to the Covid-19 crisis which has an impact on the original plans in terms of organisation and timing. The main and rare technical difficulties encountered so far were discussed. The planning for the forthcoming activities was sketched and agreed:

- the ranking of the cells will be agreed in the next weeks when the last comparison results will be available for Ru-C, by end of April
- a batch of cells will be sent for further thermal characterisation to INRIM, CMI and UME to perform tests in three-zone furnaces on Fe-C and Pd-C cells, during May-June
- a protocol for the measurement of the thermodynamic temperatures of the HTFP cells will be issued by end of April
- a timetable for the sequential measurements of thermodynamic temperatures by the participants will be agreed in the last two weeks of April.

Demonstrating practical primary thermometry for temperatures below 25 K

One of the main goals of the current development of the primary magnetic field fluctuation thermometer (pMFFT) is to reduce the uncertainty of the temperature measurement. As a side effect, this will simplify its construction and shorten the preceding measurements required for calibration. To this end, a revised layout of the detection and calibration coils was developed, where both coil sets are now located on a single silicon chip instead of two. Nonetheless, the full functionality comprising the calibration of the SQUID channels and the in-situ measurement of the electrical conductivity is maintained without adding non-thermal noise to the evaluated signal thanks to the cross-correlation technique and a fully (anti-)symmetric coil configuration. The new layout has several advantages. Firstly, it reduces the complexity of the setup of the pMFFT. Secondly, the only relevant (or critical) parameter of the pMFFT assembly that must be determined is the distance between this single chip and the noise sensor. This reduces the main uncertainty component of the noise temperature, which was so far related to the geometrical parameters of the assembly. Thirdly, improvements are also expected for the calibration of the detection coils and for the in-situ measurement of the electrical conductivity of the noise sensor as the influence of surface related effects should be better predictable. Finally, the dimensions of the detection and

calibration coils themselves were changed in comparison to the temperature sensor and its surrounding to further reduce the uncertainty in the temperature determination.

Real-K sheds light on SPRT non-uniqueness

Standard platinum resistance thermometers (SPRTs) are the defined interpolating instrument for the realisation and dissemination of the International Temperature Scale of 1990 (ITS-90) up to 961.78 °C. One important source of uncertainty in SPRT measurements is the interpolation of the calibration equations, which are subject to ‘non-uniqueness’. This manifests itself as differences in the temperatures inferred using different thermometers or different equations. In Real-K work package 3 ‘Extending the life of the International Temperature Scale of 1990’, the subject of recent study has been the type of non-uniqueness arising from the difference between the interpolating equations for two different subranges for the same SPRT – the so-called Type 1 non-uniqueness or ‘subrange inconsistency’, which occurs when using the SPRT in a temperature range where two different interpolating equations overlap. Guidance for the end-user on how to incorporate this effect in their uncertainty budgets is very scarce.

Sub-range inconsistency has been investigated by several groups, although so far the studies published have concerned specific regions or specific laboratories (mainly concerning thermometers in the USA, China and Europe). The Real-K study has

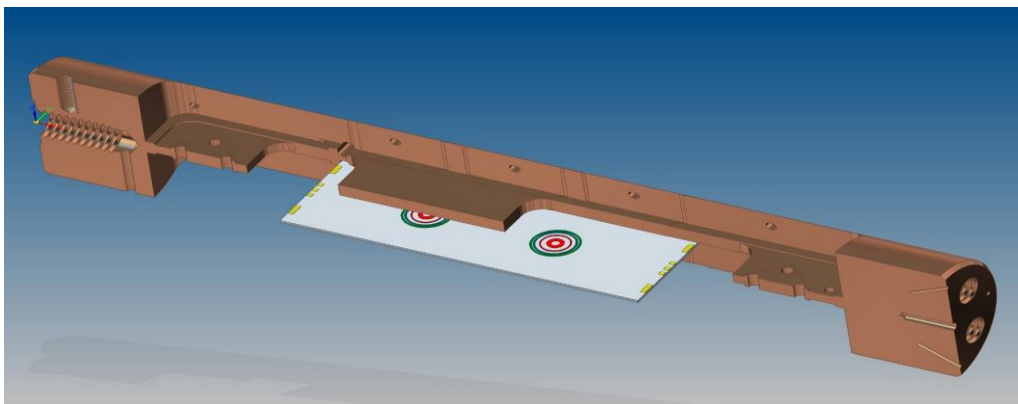


Figure 4: Assembly of the copper body of the pMFFT (sectional view) and the single silicon chip with detection coils (red) and calibration coils (blue, green). Each coil consists of two identical subsets of coils: one beneath the temperature sensor for thermal sensing and another one in the void.

been truly international, drawing on the results of 80 long-stem SPRTs from 12 different manufacturers for a total of 25 different models from regions all over the world. The analysis, performed by NRC, NPL, IPQ and UL, has yielded a comprehensive characterisation of the Type 1 non-uniqueness associated with contemporary SPRTs, and will feed into guidance to the community through a report to the CCT.

Figure 5 shows one example of the distribution of the SRI, in this case for the overlap between the argon triple point to the water triple point subrange (83.8058 K to 0.01 °C) and the mercury triple point to the gallium melting point (-38.8344 °C to 29.7646 °C). This can be used to produce a refined estimate of the associated uncertainty.

NRC, NPL, IPQ and UL have submitted a paper to Metrologia on the findings; a briefing note for the CCT is forthcoming.

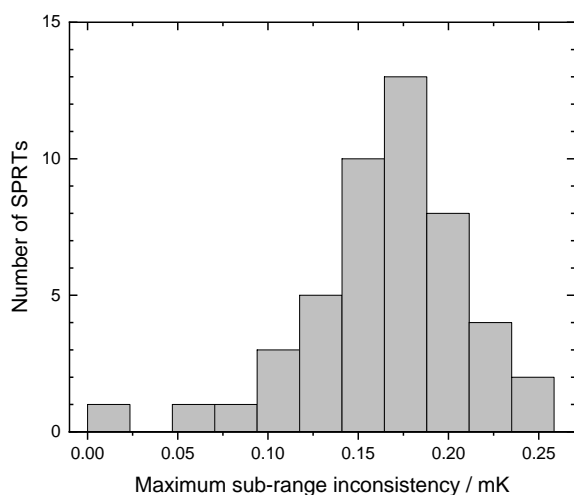


Figure 5: example of the distribution of the SRI

Facilitating full range primary thermometry

The aim of this objective is to reduce the uncertainty of several primary thermometry methods, which are included in the Mise en Pratique for the definition of the kelvin, namely acoustic (AGT), dielectric constant (DCGT) and refractive index (RIGT) gas thermometry, and to extend the application range of these methods. These achievements will promote the development

of simplified procedures for the direct dissemination of the thermodynamic temperature.

Theoretical *ab initio* calculation of thermometric gas properties

The improvement and simplification of primary thermometry methods requires the accurate knowledge of several thermophysical properties which enter in the corrections applied to account for the non-ideality of thermometric monatomic gases. For the sake of accurate acoustic thermometry (AGT) these properties include density- and acoustic virial coefficients as well as thermal conductivity. For thermometers based on the experimental determination of electromagnetic properties of gases, like dielectric constant (DCGT) and refractive index gas thermometry (RIGT), accurate estimates of properties like electrical polarizability, magnetic susceptibility and dielectric virials are also needed. The substantial theoretical and computational effort implied by these requirements is being shared among the Real-K and the **QuantumPascal EMPIR projects**, with a focus of Real-K on the improved determination of thermodynamic and transport properties of He, Ne and Ar. Remarkable progress has been recently achieved and published by the European Centre for Theoretical Studies in Nuclear Physics (ECT*) of the Bruno Kessler Foundation (FBK) in cooperation with the National Institute of Standards and Technology (NIST), reporting the improved calculation of the 4th density virial coefficient $D(T)$ of helium isotopes (^4He and ^3He), by path-integral Monte Carlo (PIMC) method and state-of-the-art two-body and three-body potentials between 2.6 K and 2000 K (see **Figure 6**). In the course of 2020, the same collaboration also published corrections to their previous estimates of the 3rd density virial coefficient $C(T)$ of helium.

Together with the remarkably accurate calculations of the second virial coefficient $B(T)$ and second acoustic virial coefficient $\beta_0(T)$ of He previously reported by the Quantum Chemistry Laboratory of the University of Warsaw (UW), and their anticipated improved three-body potential of He, the progress so far achieved reinforces the choice of He as the reference substance for most accurate thermometric work. Future work of the Real-K

project will focus on the improved calculation of the properties of heavier atomic gas, like Ne and Ar, which are less sensitive to contamination issues and may significantly increase the practical, reliable application of the same primary thermometry methods.

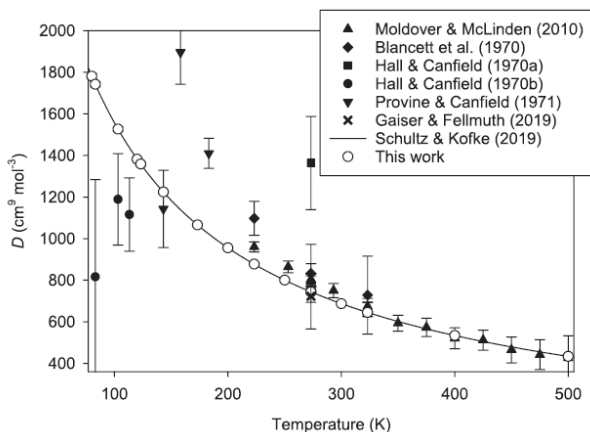


Figure 6: Comparison of the calculated values of the fourth density virial coefficient $D(T)$ of ^4He with experimental results and a previous first-principles calculation.

Measurement of thermodynamic properties of selected atomic systems

Among thermodynamic properties, speed of sound and density have a special importance, as they allow to determine directly the temperature-dependent deviations from ideality and can be determined experimentally with low uncertainties. These experiments can validate the theoretical results, where they currently suffer larger uncertainties, and drive the selection of appropriate alternative computational tools.

Speed-of-sound measurements in Ne between 200 K and 420 K at pressures up to 100 MPa have been previously reported by the Helmut Schmidt Universität (HSU), and of NPL speed of sound measurements in argon in the range 120 K to 330 K are currently being analysed. From these sets of data, estimates of the acoustic virial coefficients of Ne and Ar in these temperature range will soon become available which might later confirm the accuracy of future improved ab initio calculations of the same properties.

Coupled dielectric constant gas thermometry (DCGT) and Burnett expansion experiments with He and Ar have progressed at the Physikalisch-Technische Bundesanstalt (PTB). For He measurements at 273.16 K and 296 K were completed allowing to determine the 2nd and 3rd density virial coefficients $B(T)$ and $C(T)$. The relative standard uncertainty of the 2nd virial $u_r(B)$ is lower than 1% and the results found consistent with the most recent theoretical estimates. For Ar, previous measurements at 253 K, 273.16 K, 296 K and 303 K with relative standard uncertainties $u_r(B) = 0.5\%$ and $u_r(C)$ between 1.5% and 3% were obtained.

An acoustic gas thermometer for use in the cryogenic range down to 10 K has been completed at the Istituto Nazionale di Ricerca Metrologica (INRiM). Measurements of speed of sound in He and Ne will start in April 2021 and continue until the end of the project with the aim of providing accurate estimates of the second acoustic virial coefficient $\beta_a(T)$ of these gases. The comparison with the theoretical estimates of $\beta_a(T)$ in this temperature range is expected to be particularly interesting, especially for Ne. A RIGT version of the same apparatus recently provided estimates of the 2nd density virial of Ne between 54 K and 161 K which compare favourably with the most accurate theoretical estimates obtained by HSU and measurements of PTB which are in course of publication.

Implementing improved primary thermometry

A major objective of the Real-K project regards the simplification of two primary thermometry methods, namely DCGT and RIGT. It has been reported by PTB that a *commercial* version of DCGT, i.e. an apparatus based on commercially available instrumentation, has performed with absolute uncertainties of a few mK when operated with Ar at temperatures up to 303 K. At INRiM, a RIGT primary thermometer was operated between 13.5 K and 161 K with an absolute uncertainty of 1.8 mK at 161 K. From these measurements accurate estimates of the

differences ($T - T_{90}$) using He and Ne have been published (see **Figure 7**); development of a RIGT thermometer for use with Ar in the ambient temperature range is underway.

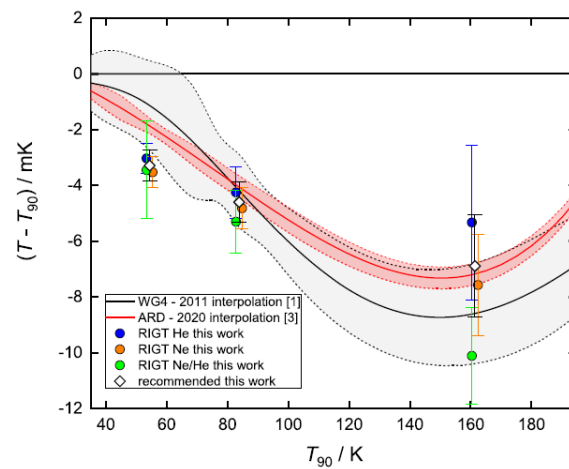


Figure 7: Comparison of the $(T - T_{90})$ determinations obtained using RIGT with two interpolating equations, respectively based on primary thermometry results obtained until 2011 or after that date.

Dissemination of project results

Scientific articles

1. GAISER, Christof; FELLMUTH, Bernd; HAFT, Norbert. Thermodynamic-temperature data from 30 K to 200 K. *Metrologia*, 2020.
2. GAO, Bo, et al. Measurement of thermodynamic temperature between 5 K and 24.5 K with single-pressure refractive-index gas thermometry. *Metrologia*, 2020.
3. WANG, Yanfei, et al. A method for spectral irradiance measurement based on a large area WC-C fixed point blackbody. *Optics Express*, 2020, 28.19: 28430-28440.
4. PAN, Changzhao, et al. Active suppression of temperature oscillation from a pulse-tube cryocooler in a cryogen-free cryostat: Part 1. Simulation modeling from thermal response characteristics. *arXiv preprint arXiv:2002.03177*, 2020.
5. PAN, Changzhao, et al. Active suppression of temperature oscillation from a pulse-tube cryocooler in a cryogen-free cryostat: Part 2. Experimental realization. *arXiv preprint arXiv:2002.03178*, 2020.
6. IMBRAGUGLIO, Dario; STEUR, Petrus Paulus Maria; SPARASCI, Fernando. Comparison of ITS-90 realizations from 13 K to 273 K between LNE-CNAM and INRIM. *Measurement*, 2020, 166: 108225.
7. HAHTELA, O. M., et al. Coulomb Blockade Thermometry on a Wide Temperature Range. In: **2020 Conference on Precision Electromagnetic Measurements (CPEM)**. IEEE, 2020. p. 1-2.
8. A. Peruzzi, et al. Survey of Subrange Inconsistency of Long-Stem Standard Platinum Resistance Thermometers, *Metrologia*. (currently under revision)
9. D. Madonna Ripa, et al. Refractive index gas thermometry between 13.8 K and 161.4 K, *Metrologia*. (currently under revision)
10. Y. Xie, et al. A fast Akima curve fitting method approximation of large-area HTPF melting plateau for spectral irradiance application, *Applied Optics*. (currently under revision)
11. P. Changzhao, et al. Acoustic measurement of T_{Ne} used as a reference for thermometry below 25 K, *Metrologia*. (currently under revision)

Presentations and other disseminations

1. **Poster of Real-K project**
2. Coulomb Blockade Thermometry on a Wide Temperature Range, Aug 2020, IEEE Precision Electromagnetic Measurements (CPEM 2020), United States.
3. Current thermometry research directions, invited seminar, January 2020, Aberdeen University, United Kingdom.
4. The kelvin redefinition and its implications, invited keynote, February 2020, European Society of Precision Engineering (EUSPEN), Germany.
5. Redefinition of the SI, November 2020, invited seminar, Glasgow Caledonian, United Kingdom.
6. Participation of VNIIOFI in EURAMET projects including the Real-K, September 2020, COOMET webinar, Russian Federation.
7. COOMET Tasks in the Light of the redefinition of the International System of Units (SI), October 2019, COOMET Seminar, Russian Federation.

Forthcoming events

- Invited keynote speech at Sensor and Measurement Science International 2021, May 2021, Germany
- Talk at International Congress on Metrology (CIM 2021) on Real-K project, Sep 2021, France

Consortium and contact information

The consortium consisting of national metrology, research institutes and universities brings together a critical mass of recognised world leaders in the field.



Project coordinator: Graham Machin (graham.machin@npl.co.uk).

Project website: <https://real-k.aalto.fi>.

Newsletter: Every nine months an e-Newsletter will be available via the project **website**.

Inquiries and more info, Contact Shahin Tabandeh (shahin.tabandeh@vtt.fi).