Aluminium fixed-point cells for thermodynamic temperature assignment

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Aluminium fixed-point cells for thermodynamic temperature assignment

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Abstract. In the frame of the EMPIR-funded joint research project 15SIB02 “Implementing the new kelvin – 2” (InK2) the determination of T-T⁹₀ over the temperature range between 430 K and 1358 K with a target uncertainty around the 10 mK level is under preparation in several NMIs, among which LNE-Cnam and CEM. The two laboratories have joined their efforts for the thermal characterisation of a set of three aluminium fixed-point pyrometric cells. The cells were constructed using the hybrid design based crucibles and the piston filling method both developed by LNE-Cnam over the past decade. To study the effect of the temperature gradients on the freezing temperatures of the cells, two three-zone furnaces were used for the implementations of the cells with different temperature distributions. Finally, a first determination of the thermodynamic temperature of the freezing plateau was determined by extrapolation from the thermodynamic temperature of the copper point of LNE-Cnam. The results of the characterisations and the temperature determination are presented.

1. Introduction

The mise-en-pratique of the new definition of the kelvin, due in 2019, will require the knowledge of T-T⁹₀ over a large range of temperatures. The European metrology research programmes (EMRP and EMPIR) have included joint research projects (JRP) striving to achieve this goal over the last 5 years [1]. The current JRP InK2 within which this work has been performed focuses on the temperature range between 430 K and 1358 K.

To achieve the lowest uncertainty on T-T⁹₀ over this range two different methods are intended to be implemented: high-temperature acoustic gas thermometry and medium-temperature primary radiometry with an overlapping temperature range aiming to increase the reliability in the determined values. Within the radiometric measurements scheme, it is planned to base the work on ITS-90 fixed-point cells (namely Zn, Al, Ag and Cu) for which low-uncertainty thermodynamic temperature determinations are sought.

This work is an important step in this direction for the aluminium point. Robust and reliable cells have been constructed using the up-to-date designs and their characteristics regarding thermal effects were determined as a collaboration between the two participating laboratories (CEM and LNE-Cnam) and a first thermodynamic temperature assignment to the Al cells was performed by LNE-Cnam using a monochromator-based InGaAs radiance comparator.

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2. Filling of the Al cells

One of the objectives of this work was the construction of a set of cells for the comparison planned in the project (3 Zn cells, 3 Aluminium cells and 3 Ag cells). This set of cells was constructed using the “piston” method developed by LNE-Cnam and described in figure 1 [2]. This method has the great advantages of being fast and non-polluting and it allows the preparation of a representative sample for impurity analyses. The graphite parts are all made of 5-ppm level of impurity graphite and the design of the cells reproduces the well-established hybrid design including C/C sheets surrounding the sacrificial graphite sleeve intended to improve the robustness of the cells. This design is particularly efficient for the metal-carbon eutectic/peritectic high-temperature fixed points (HTFPs).

The dimensions of the cells are well adapted to the medium temperature range and the possibilities offered by the available three-zone furnaces (larger and more uniform than the high-temperature furnaces used for HTFPs). The cells are therefore larger than the usual HTFP cells. The length of the cells is 70 mm and the diameter is 32 mm. The volume of metal forming the ingot is estimated to be about 14 cm³. The mass of the aluminium ingot, formed from a 6N Al from Material Research SA (bought about 30 years ago) is approximately 34 g.

![LNE-Cnam “piston” method for the filling of fixed-point cells](image)

3. Characterisation of the temperature gradient effect

The expected output of this characterisation work was the quantification of the effect of temperature gradients on the freezing temperature of the cells. A prerequisite for this task is then naturally to have the possibility of tuning the temperature gradients of the furnace. The furnaces used for the implementation of the Al cells were two 3-zone furnaces newly commissioned from Cerhec company (France) corresponding, in particular, to strict specifications for the achievable temperature uniformity along the cells. Figure 2 shows the three different cases of temperature distributions achievable in both furnaces by changing the temperature set-ups of the three heaters. Temperature differences of up-to ± 2 K between the ends of the cell can easily be obtained and this temperature gradients can be maintained for sufficiently long periods thanks to a very stable temperature control. In any given conditions, the temperature controllers are indeed capable of changing the temperature setpoints to maintain the gradient while initiating the freeze or the melt.
Figure 2. Temperature profiles of furnace A and furnace B. The blue rectangles represent the cell positions inside the furnaces (S.P = temperature set-point).

When the freezing plateau is realised in different furnace temperature profiles, the duration of the plateau was observed to change: it is shorter when the bottom of the cell is at lower temperature than the rest of the cell, as can be seen on fig 3 in the red line case. In this situation, the solidification of the aluminum at the bottom of the cell is much faster than in the rest of the cell and it reaches the temperature of the furnace earlier than the rest of the cell.

Figure 3. Comparison of the Aluminium plateaus under different furnace profile temperatures – the measurement consists of a comparison between one cell in furnace A (with changing conditions) and one cell in furnace B (in the most uniform conditions). The pyrometer observes the cells alternatively (cell in furnace B for a shorter time).

In this case, the reading of the thermometer during the freezing plateau is higher than in the other two cases probably due to the emissivity, since the thermometer is focused on the entrance of the cell, which is a higher temperature than the rest of the cell. The combination of these two effects, fast
solidification of the metal at the bottom and high temperature at the entrance gives as a result the poor quality of the plateau.

When the bottom of the cell is hotter than the entrance (blue line in fig. 3), the duration of the plateau is a bit longer and the end of the plateau is sharper than when the cell is subjected to the best furnace temperature profile (green line in figure 4). On the contrary, the reading of the thermometer in the plateau with the best temperature furnace profile is a bit higher than the reading of the thermometer in the plateau with the profile hotter at the bottom of the cell.

In all cases however, the thermal effects have been estimated to induce an error on the freezing temperatures of less than 3 mK.

4. Thermodynamic temperature of the Al freezing point
The assignment of the thermodynamic temperature value to the aluminum freezing point was performed by extrapolation from the temperature of the copper fixed point, whose thermodynamic temperature was determined at LNE-Cnam [3-5]. The extrapolation was performed using the monochromator-based radiance comparator of LNE-Cnam adapted to the IR range using an InGaAs detector.

This extrapolation was done by applying Plank’s law for radiation to derive the unknown Al fixed point temperature from the ratio of the radiance of both fixed points. The comparison between the two FP blackbodies was done in the infrared, between 1.2 µm and 1.6 µm.

The difference between the mean thermodynamic temperature of the phase transition and the ITS-90 temperature (933.473 K) is therefore of only 2 mK ± 83 mK. This uncertainty is considered too large for the moment and should be improved by a direct measurement using an InGaAs reference (trap) detector traceable to the cryogenic radiometer around 1.6 µm.

5. Conclusion
This work has allowed the validation of the constructed Al fixed point cells as suitable means to ensure the determination of $T - T_{90}$ and to act as transfer standards for the comparison of the realisation of thermodynamic temperature scales in the European region and beyond.

The feasibility of the extrapolation of the thermodynamic temperature of the copper point to lower temperatures has been demonstrated with an uncertainty level of about 40 mK (k=1) at the aluminium point, paving the way to possible improvements of the knowledge of $T - T_{90}$ over the whole temperature range from 430 K to 1358 K.

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6. References
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