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Thermodynamic measurements of Zn and Al freezing temperatures using an InGaAs-based, near-infrared radiation thermometer 3 (NIRT3)

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Abstract. Thermodynamic temperature measurements of Al and Zn points using a near-infrared radiation thermometer 3 (NIRT3) are described. The NIRT3 was constructed for long-term stable operation, low size-of-source and characterized for linearity. The spectral responsivity was measured using tuneable near-infrared lasers and window-less InGaAs diodes. The spectral responsivity of the NIRT3 was scaled using a Au freezing-temperature blackbody and an absolute visible radiation thermometer. We then measured both the Al and Zn freezing temperatures and these measurements are compared against T-T⁹⁰ values estimated by the Consultative Committee for Thermometry.

1. Introduction

The upcoming redefinition of the kelvin has led to intense activities at national measurement institutes around the world to develop capabilities for directly measuring thermodynamic temperatures [1]. Much of this work centre around better thermodynamic temperature measurements of existing and new freezing- and melting-point cells to improve the uncertainties in current and future temperature scales [2]. At temperatures near the triple point of water, acoustic thermometry can be used with the lowest uncertainties, however, these uncertainties increase with increasing temperatures due to material and sensor limitations. At high temperatures, detector-based thermodynamic temperature measurements by numerous NMI’s have been shown to result in low uncertainties in international temperature assignments of metal-carbon eutectic [3] melt inflections. At intermediate temperatures between 400 K and 1400 K, thermodynamic, spectral-radiation thermometry must be performed in the near-infrared wavelength region due to the low radiiances of the Planck radiation in the visible wavelength region. Recently, thermodynamic temperatures in the near-infrared wavelength region were measured using radiation thermometers calibrated with monochromator-based systems utilizing supercontinuum sources [4, 5]. These radiation thermometers could be directly calibrated using near-infrared detectors or be calibrated using a chain of measurements relying on initial measurements with silicon detectors which operate in the visible wavelength range.

In this paper, we describe the characterization of the NPL Near-Infrared Radiation Thermometer 3 (NIRT3) [6] and it absolute calibration using the Absolute Pyrometric Transfer Radiometer 650 (APXR650) and a gold-freezing temperature blackbody. We use this single-point radiometric calibration along with the trans-impedance amplifier gain calibrations and knowledge of the size-of-source effect and relative radiance responsivity of the NIRT3 to determine thermodynamic temperatures of NPL Al and Zn points. Our thermodynamic temperature measurements are compared with those compiled by the Consultative Committee for Thermometry Working Group 4 [7].

2. NIRT3 calibrations and characterizations

The near-infrared radiation thermometer 3 (NIRT3) was designed and constructed for low uncertainty determinations of thermodynamic temperatures. In the following sections the calibration and characterization of the NIRT3 are described followed by the measurements of the Al and Zn freezing point temperatures.
2.1. Thermodynamic-temperature traceability

The thermodynamic temperature traceability is shown in Figure 1. Initially both the 650 nm channel of the Absolute Pyrometric Transfer Radiometer (APXR650) and the NIRT3 were calibrated in the NIST facility for the calibration of detectors and radiometers for spectral irradiance responsivity and spectral radian ce responsivity (SIRCUS) [8]. The initial determinations of thermodynamic temperatures of ITS-90 fixed points using the NIRT3 calibrated in this way showed systematic 0.38 % lower signals than those measured at the fixed points. The discrepancies were possibly due to the higher uncertainties in the power-to-irradiance responsivity transfers using the single-element InGaAs as compared to the lower achievable uncertainties using Si-trap detectors.

![Traceability flow diagram of the NPL Al and Zn point measurement using the NIRT3. The thermodynamic temperature determination of the Au freezing temperature using the APXR650 which is centred at 650 nm is used to assign the absolute calibration of the NIRT3.](image)

2.2. NIRT3 preamplifier calibration and SSE measurements

The NIRT3 was designed to view a 2.0 mm diameter spot at a distance of 75 cm from the front of the objective lens. The optical layout was slightly modified from a previously described radiation thermometer for these requirements [9]. The size-of-source effect (SSE) was measured using a 10 mm diameter central obscuration and the outer diameter of the diffuse radiance source was varied from 15 cm to 50 cm in 5 mm steps. The SSE was found to be < 5x10⁻⁵ with source diameters within the above range of measurements. The transimpedance amplifier was calibrated separately using measurements traceable to the latest realization of voltage and resistance, and the gains are shown in Table 1.

| NIRT3 Gain Range | Gain (V/A)  | Measurements          |
|------------------|------------|-----------------------|
| 10               | 1.00352E+10|                       |
| 9                | 9.9916E+08 | NPL Zn                |
| 8                | 1.00026E+08| SIRCUS Calibrations, NPL Al |
| 7                | 1.00017E+07|                      |
| 6                | 9.99969E+05| NIST Au               |

2.3 NIRT3 spectral responsivity measurements

The NIRT3 radiance responsivity was determined using tuneable near-infrared lasers which were guided into a 30 cm diameter integrating sphere whose inner walls are coated with sintered polytetrafluoroethylene (PTFE). A small, 8 mm diameter precision aperture was placed at the output of the integrating sphere. The spectral irradiances were determined using a calibrated, 5 mm diameter, window-less InGaAs diode. The presence of a window on the diode resulted in interference fringing issues. The InGaAs diode was calibrated in spectral power responsivity
mode using the NIST primary cryogenic radiometer using tuneable lasers. A precision aperture was also placed on the InGaAs diode for assignment of spectral irradiance responsivities. The measured spectral radiance responsivities of the NIRT3 are shown in Fig. 2.

Fig. 2. The absolute spectral radiance responsivities of the NIRT3 measured in the NIST SIRCUS facility using a window-less 5 mm InGaAs diode.

3. NIRT3 measurements of NPL Al and Zn freezing temperatures
The measurements of the NPL Al and Zn blackbodies were performed in Sept. 2017. A 3-zone furnace along with the cells were shipped to NIST from NPL for these measurements. The NIRT3 was calibrated by measuring the NIST small, Au cell with its temperature measured using the APXR650 to be 1337.36 K which is 30 mK higher than the ITS-90 value. This thermodynamic temperature of the gold point is consistent with the most recent recommended value by CCT.

The APXR650 was calibrated on the NIST SIRCUS facility. The radiance responsivity of the NIRT3 was corrected by a factor of 1.003768 to account for the difference between the SIRCUS calibrations and the transfer calibrations from the APXR650.

The NIRT3 was aligned visually at the opening of the NPL Al and Zn point blackbodies. The measurement of the NPL Al and Zn points were performed at preamplifier gains of $10^7$ and $10^8$, and the respective gain corrections were applied from Table 1. The photocurrents at the freezing plateau were determined from voltages converted to photocurrents. This conversion is needed to compare voltages measured at different gains and also to compare the measured currents to the those calculated using the SIRCUS calibrations. The measured photocurrents as compared to those calculated at the ITS-90 temperatures are shown in Table 2. The differences in temperatures or $T-T_{90}$ were found by using the differences in signals converted to temperatures from the derivative of the Wien approximation. A global fit of the Sakuma-Planck function resulted in residual temperatures of up to 8 mK which were close to the measured differences.

Table 2. Results of the NIRT3 thermodynamic temperature determinations of the NPL Al and Zn freezing temperatures.

|          | ITS-90 Temperature, K | Calculated using NIRT3, A | Measured by NIRT3, A | Ratio Measured/Calculated | NIRT3 ($T-T_{90}$), K |
|----------|-----------------------|---------------------------|----------------------|---------------------------|-----------------------|
| NPL Zn   | 692.68                | 2.11980E-09               | 2.12022E-09          | 1.0001996                 | 0.010                 |
| NPL Al   | 933.47                | 6.69962E-08               | 6.70083E-08          | 1.000181                  | 0.017                 |

4. Discussion
The measured thermodynamic temperatures are compared to the best estimates of $T-T_{90}$ calculated by the CCT WG4 in Figure 3. We estimate that our expanded uncertainties are at 0.1 %
\( (k=2) \) in radiance responsivity at the Au point which corresponds to 81 mK using the APXR650. Such uncertainties are consistent with those reported by other NMIs in a recent comparison. We utilize the calculated emissivity of the Au freezing cell, 0.99964, to calibrate the absolute spectral responsivity of the NIRT3 to 0.06 % \( (k=2) \). This uncertainty in radiance responsivity corresponds to estimated uncertainties of 31 mK and 56 mK at Zn and Al freezing temperatures respectively. A more complete uncertainty budget will be described in a later publication.

![Fig. 3. Differences in \( T-T_{90} \) temperatures as compiled by the CCT WG4 and as measured by the NIRT3. The Au freezing temperature was measured by the APXR650 and used to scale the NIRT3 measurements.](image)

5. Conclusions
The thermodynamic temperatures of Al and Zn freezing temperature were measured using the NPL NIRT3 using a calibration path which utilizes the lower uncertainties in absolute spectral radiance responsivities achievable in the visible wavelength region. The radiance responsivity of the NIRT3 was calibrated using tuneable lasers and a window-less InGaAs diode. The NIRT3 was designed for low SSE, and also the preamplifier was calibrated at each gain. Our measured temperatures are compared with the estimates in the CCT WG4 document.

We demonstrate that thermodynamic temperature measurements in the infrared are possible using a radiation thermometer calibrated in the visible wavelength range where lower uncertainties can be achieved.

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