

# MEASUREMENT OF $T-T_{90}$ DOWN TO ZINC POINT TEMPERATURES WITH ABSOLUTE FILTER RADIOMETRY

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## ABSTRACT

In 1995 at the Physikalisch-Technische Bundesanstalt (PTB) measurements of  $T-T_{90}$  for the temperature range 660 °C to 962 °C have been performed using a large area blackbody (LABB) and absolute filter radiometry.

Although the inner cavity of the LABB is formed by a sodium heat pipe, it has been shown in [1] that the LABB can be used as an accurate source of blackbody radiation at temperatures as low as 400 °C. Applying interference filter radiometers with center wavelengths of 800 nm, 900 nm, and 1000 nm, the temperature range for thermodynamic temperature measurements of the LABB has been extended to temperatures as low as the zinc freezing point temperature of 419 °C. The achieved standard uncertainty was about 20 mK for thermodynamic temperatures of 419 °C. The measured thermodynamic temperatures have been compared with the temperature of the blackbody cavity measured according to the International Temperature Scale of 1990 (ITS-90) sensed by three high temperature standard platinum resistance thermometers (HTSPRT) located at the bottom of the radiating cavity. These HTSPRT were calibrated in terms of the ITS-90.

Comparing the thermodynamic and the ITS-90 temperatures it was found that the former is always higher than the latter, with decreasing difference towards lower temperatures.

## 1. INTRODUCTION

The International Temperature Scale of 1990 for temperatures above 457 °C is based on radiation thermometry using a temperature near 456 °C as a reference [2, 3]. The temperature of 456 °C was the highest thermodynamic temperature, which could be measured by gas thermometry and this thermodynamic temperature was determined by two groups, but the obtained values differed by 0.03 K [4, 5]. In order to generate a unique temperature scale, the average of the two values was used as the reference temperature for the radiation thermometry [2, 3, 6]. Recently, PTB has undertaken thermodynamic temperature measurements by absolute filter radiometry to investigate the difference of the thermodynamic temperature  $T$  and the ITS-90 temperature  $T_{90}$  in the range from 962 °C down to 660 °C [7]. The identified differences indicate that the higher of the two temperature values determined by gas thermometry should be the more accurate one. To further support this evidence PTB has extended the applicability of absolute filter radiometry using silicon photodiodes to temperatures as low as 419 °C. In a first step, the practicability of the used radiation source, the large area blackbody (LABB) at temperatures down to 400 °C has been successfully proven [1]. In a second step the spectral responsivity scale for silicon photodiodes has been extended to 1014 nm with a relative uncertainty below 0.01% for the visible and below 0.05% for the near-infrared wavelength range [8], which includes a critical consideration of the temperature dependence of the spectral responsivity of silicon for wavelengths above 950 nm [9]. Additionally, the influence of the stray light increases with increasing wavelength. Measuring temperatures below 500 °C with silicon photodiodes and narrow bandwidth interference filters requires the application of filters with center wavelengths above 900 nm. At these long wavelengths the influence of the stray light increases significantly, when calibrating the narrow bandwidth interference filter radiometers with respect to silicon trap detectors. To overcome this problem a double monochromator system, consisting of a grating monochromator and an additional prism pre-disperser has been developed. This preparatory work provides the

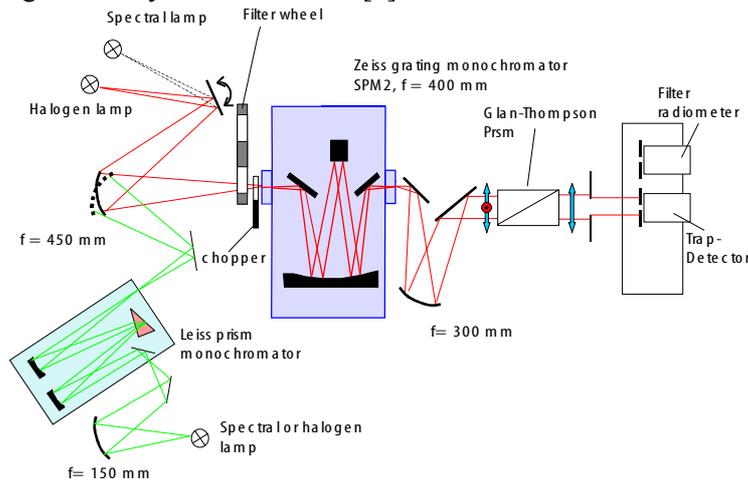
essentials for the accurate calibration of interference filter radiometers to perform measurements of thermodynamic temperatures by silicon filter radiometry at temperatures down to 400 °C.

Section 2 describes the calibration procedure of the interference filter radiometer with respect to trap detectors, previously calibrated at the radiation thermometry cryogenic radiometer (RTCR) at several laser lines. The traceability to the ITS-90 is performed by standard platinum resistance thermometers and their accuracy and stability is discussed in section 3. The uncertainty for the  $T-T_{90}$  measurements is given in section 4. New preliminary results of  $T-T_{90}$  are presented in section 5.

## 2. CALIBRATION OF NARROW BANDWIDTH INTERFERENCE FILTER RADIOMETER

The calibration of the filter radiometers is performed with reference to silicon reflection trap detectors, which itself were calibrated against the cryogenic radiometer RTCR at several laser wavelengths. The spectral responsivity of the traps between the laser lines was interpolated using a physical model [8]. It has been reported in [10] that the spectral responsivity of narrow bandwidth interference filter radiometers based on silicon photodiodes can be calibrated with a relative uncertainty of a few  $10^{-4}$  in the spectral range between 400 nm and 800 nm. However, applying interference filter radiometers for temperature measurements below 500 °C requires a shift to infrared wavelengths. Today's infrared sensitive photodiodes like InGaAs or Ge diodes still do not have the high homogeneity and shunt resistance of silicon photodiodes. However, the application of silicon photodiodes is restricted to wavelengths below 1020 nm, but even silicon shows some disadvantages when it is applied at those wavelengths. One problem is the increasing temperature dependence of the spectral responsivity and the increasing non-linearity. Another problem arising in this wavelength range is linked to the increasing stray light generated in single grating monochromators and the overall temporal stability of the calibration. In the following the solution to these four problems is discussed shortly

To minimize the effects of the temperature dependence of the spectral responsivity of silicon, the temperature of the trap detector during its calibration with respect to the cryogenic radiometer was monitored. During the calibration of the filter radiometer with respect to the trap detectors and during the application of the filter radiometer at the LABB the temperature was carefully stabilized within  $\pm 50$  mK and recorded. Care has been taken to keep the trap and the filter radiometer during the calibrations and the application at the same temperatures. Small temperature differences still present were corrected using the theory outlined in Ref. [9].

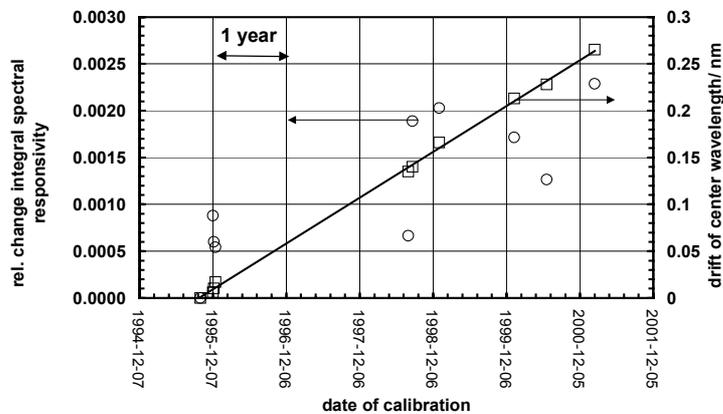


**Figure 1:** Schematic drawing of the spectral comparator facility. The set-up may be operated with and without the prism pre-disperser by simply rotating the spherical mirror.

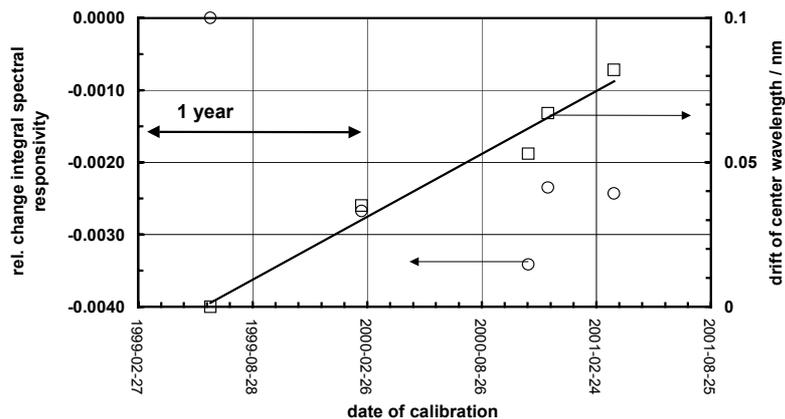
The trap detector was calibrated with respect to the cryogenic radiometer at a radiant power of about 0.5 mW, while the radiant power during the calibration of the filter radiometer at the spectral comparator is as low as 50 nW [10]. The radiant power received by the filter radiometer in front of the

LABB is as low as 10 pW. The major difference in the measured radiant powers is detected by the trap detector. Thus, the non-linearity of the spectral responsivity of a silicon trap detector has been measured at the center wavelength of the interference filter radiometer, showing that at wavelengths above 900 nm silicon photodiodes show a supra-linearity up to several  $10^{-4}$ , which have to be taken into account.

The stray light generated inside a grating monochromator is proportional to the wavelength and to the density of lines of the grating. Due to the narrow bandwidth of the interference filters gratings with more than 1000 lines per mm have to be used, resulting in a level of stray light of about  $10^{-5}$ . Application of a double grating monochromator is not feasible, as the transmitted radiant power would decrease dramatically. Therefore we decided to complement the existing single grating monochromator [10] with a prism pre-disperser (see Fig. 1), which provides sufficient stray light rejection and still enough radiant power for calibration of the filter radiometer. Applying the prism-pre-disperser the spectral comparator set-up now provides a stray light rejection of the order of  $10^6$ , sufficient for the calibration of narrow bandwidth near-infrared filter radiometers.



**Figure 2:** Time evolution of the relative change in spectral responsivity (circles) and the change in center wavelength of the 800 nm filter radiometer (squares) with respect to the calibration in October 1995. The solid line is a linear fit to the data, serving as guide to the eye.



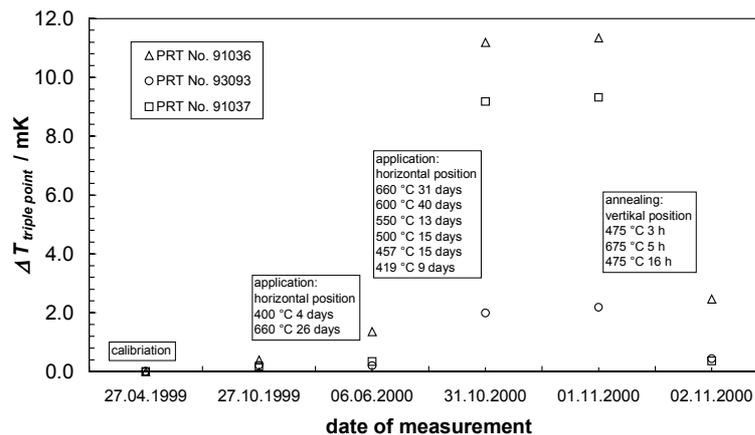
**Figure 3:** Time evolution of the relative change in spectral responsivity (circles) and the change in center wavelength of the 900 nm filter radiometer (squares) with respect to the calibration in June 1999. The solid line is a linear fit to the data, serving as guide to the eye.

The used filter radiometers have been calibrated and re-calibrated over a longer period of time and they were found to be remarkable stable. Figures 2 and 3 show the time evolution of the relative change in spectral responsivity and the change in the center wavelength for the filter radiometers at

800 nm and 900 nm. It can be seen that the drift in center wavelength is typically in the order of a few pm per month, as has been found previously for an interference filter at 974 nm [3], while the change in spectral responsivity critically depends on the filter radiometer and varies in the range from  $10^{-5}$  to  $10^{-4}$  per month. To reach an relative uncertainty in spectral responsivity in the order of  $10^{-4}$  it is sufficient to re-calibrate the filter radiometers every month.

### 3. TRACEABILITY OF THE LABB TEMPERATURE TO ITS-90

The traceability of the LABB temperature to the ITS-90 is performed by three high temperature standard platinum resistor thermometers (HTSPRT), calibrated at tin, zinc, aluminium, and silver freezing point temperatures. These HTSPRTs have been applied for temperature measurements of the LABB over several years and have been recalibrated several times. The trend of the difference of the temperatures of the triple-point of water measured after application with respect to the calibration at the beginning of the measurement campaign is shown in Fig. 4 for the three HTSPRTs. One remained very stable, two HTSPRTs showed a remarkable change in the temperature at the triple point of water after being applied several days at temperatures below 500 °C. The reason for this probably is the horizontal application of the HTSPRTs at the LABB. This prevents a circulation of the argon gas inside the HTSPRT, eventually resulting in an accumulation of impurities at the sensor. Subsequent annealing of the HTSPRTs recovers the previous values. In order to prevent this effects, the time the HTSPRTs were exposed to temperatures below 500 °C in a horizontal position was kept to an absolute minimum.



**Figure 4:** Change of the temperature of the triple point of water measured with the HTSPRT's with respect to the calibration at 27.04.1999. The insets shows the history the HTSPRT's had undergone between the several measurements of the triple point of water. Between the 1.11.2000 and 2.11.2000 they had been annealed.

### 4. UNCERTAINTY BUDGET

The uncertainty in terms of spectral irradiance for the measurement of  $T-T_{90}$  using the filter radiometers is presented in Table 1. The overall uncertainty depends on four major contributions. The geometry contribution includes the uncertainty in the radiating area of the LABB, the diameter of which is measured with an uncertainty of 0.4  $\mu\text{m}$  and its thermal expansion, resulting from an uncertainty in aperture temperature measurement of about 0.1 K. Also considered is a diffraction component and an uncertainty of 50  $\mu\text{m}$  in the distance measurement. The uncertainty in measurement of the ITS-90 temperature considers the realisation of the ITS-90 (6 mK at 660 °C) and the temperature stability of the LABB (2 mK). Uncertainty contributions to the thermodynamic temperature measurement arise from the uncertainty in the photo current measurement, the calibration of the I/U converter, the calibration of the voltmeter and the numerical integration of the photo current

integral. For the uncertainty of the filter radiometer the calibration at the spectral comparator and a diffraction contribution have to be considered. The final relative uncertainty in the spectral irradiance measured with the filter radiometer is in the order of some  $10^{-4}$ , which gives a temperature equivalent of few tens of mK.

**Table 1:** Uncertainty budget in terms of irradiance for the temperature difference  $T-T_{90}$  given in  $10^{-4}$ .

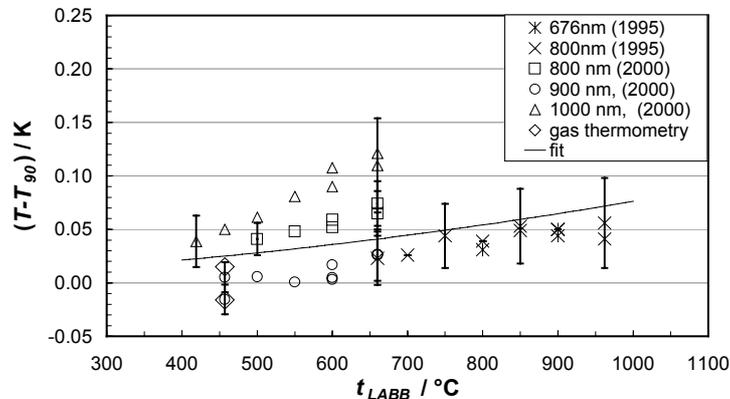
Uncertainty contribution	filter radiometer					
	800 nm		900 nm		1000 nm	
	500 °C	660 °C	450 °C	660 °C	419 °C	660 °C
geometry	1.5	1.5	1.5	1.5	1.5	1.5
measurement ITS 90	1.3	1.3	1.3	1.2	1.3	1.1
measurement of T	1.5	1.4	1.5	1.4	1.5	1.4
filter radiometer	3.6	3.6	3.6	3.6	6.9	6.9
total uncertainty	4.4	4.4	4.4	4.3	7.3	7.3
temperature equivalent (mK)	15	21	14	24	24	44

## 5. PRELIMINARY RESULTS OF $T-T_{90}$

The interference filter radiometers with central wavelength at 800 nm, 900 nm and 1000 nm have been applied to measure the thermodynamic temperature between 500 °C and 660 °C. Below 500 °C only the 900 nm and the 1000 nm filter radiometer could be applied. The results of  $T-T_{90}$  are presented in Fig. 5 together with the results based on absolute calibrations of the filter radiometers published in [7]. It can be seen that the general trend of the former data is confirmed by the present data, although the scatter in the results at low temperatures is increased. This is due to the fact that the measured photo currents were as low as 10 pA, which were at the lower end of the measurement range of the equipment. However, all results agree within their combined uncertainties. The solid line additionally shown in Fig. 5 is a fit to the data according to

$$\Delta T = \left( \frac{T}{T_{ref}} \right)^2 \Delta T_{ref}, \quad (1)$$

with  $T_{ref} = 729$  K and  $\Delta T_{ref} = 25$  mK (solid line). This line describes the propagation of the difference in temperature, if the reference temperature of 729 K was about 25 mK too low. The results of Fig. 5 strongly support the idea that the higher of the two temperatures determined by gas-thermometry, which are also included in Fig. 5, should have been used for the establishment of ITS-90.



**Figure 5:** Measured differences of  $T-T_{90}$  in the range 419 °C up to 962 °C. The solid line is a fit to Eq. 1 with  $\Delta T_{ref} = 25$  mK

The results presented in Fig. 5 are preliminary only as the non-linearity of the filter radiometers, which significantly affects the measurements for the 900 nm and 1000 nm filter radiometers has to be checked again.

## 6. CONCLUSIONS

An improved spectral comparator set-up has been presented, which allows the calibration of narrow bandwidth interference filter radiometers with relative uncertainties in the order of some  $10^{-4}$ . This set-up has been used to calibrate near infrared small bandwidth interference filter radiometers based on silicon photodiodes at 800 nm, 900 nm, and 1000 nm for determination of thermodynamic temperatures of the large area blackbody radiation source below 500 °C by absolute filter radiometry. The intention of these measurements was to investigate the difference between thermodynamic and ITS-90 temperature. The improved accuracy in the filter radiometer calibrations results in an uncertainty of the temperature difference  $T-T_{90}$  in the order of 20 mK, with values as low as 14 mK at 450 °C. The obtained temperature differences  $T-T_{90}$  exhibit an increasing difference with increasing temperature, confirming the general trend observed in [7]. The obtained temperature difference can be explained by a propagating temperature error at the temperature of 456 °C, which has been used as the reference temperature in the ITS-90 for extrapolation of the scale to higher temperatures by radiation thermometry. The thermodynamic temperatures measurements are preliminary in that sense that the non-linearity corrections for the filter radiometers must be confirmed.

## ACKNOWLEDGEMENTS

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