High-accuracy radiation thermometry at the Physikalisch-Technische Bundesanstalt (PTB)

D. R. Taubert, K. Anhalt, B. Gutschwager, C. Monte, J. Hartmann, J. Hollandt

Physikalisch-Technische Bundesanstalt
**Introduction**

**Temperature**

- governs most production processes in industry
- crucial for optimized productivity and quality assurance

frequent measurement technique – radiation thermometry

- annual growth rate: more than 10%
- 600,000 radiation thermometers sold / year
- 15,000 thermography systems sold / year
- market volume: > 1 billion €
Introduction

Increased demand of calibrations in the field of radiation thermometry

**PTB** operates two calibration facilities:

- **Low-/ mid-temperature calibration facility**; temperature range: -60 °C to 962 °C
- **High temperature calibration facility**; temperature range: 900 °C to 3000 °C

Realization and dissemination of radiation temperatures
Low- / mid-temperature calibration facility

schematic view

<table>
<thead>
<tr>
<th>Substance</th>
<th>Temperature Range</th>
<th>Calibration Factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonia (NH₃)</td>
<td>-60 °C to 50 °C</td>
<td>ε = 0.9996</td>
</tr>
<tr>
<td>Water (H₂O)</td>
<td>50 °C to 270 °C</td>
<td>ε = 0.9996</td>
</tr>
<tr>
<td>Caesium (Cs)</td>
<td>270 °C to 650 °C</td>
<td>ε = 0.9998</td>
</tr>
<tr>
<td>Sodium (Na)</td>
<td>500 °C to 962 °C</td>
<td>ε = 0.9999</td>
</tr>
</tbody>
</table>

Thermal camera or radiation thermometer on translation stage:
- x: 3000 mm
- y: 600 mm
Low- / mid-temperature calibration facility

- cross-section of sodium / cesium heat-pipe blackbody

- heater temporal stability: 0.1 K
- heat-pipe temperature stability: 10 mK
Low- / mid-temperature calibration facility

- cross-section of H₂O / NH₃ heat-pipe blackbody

- housing
- cooling element
- aperture
- N₂ purging
- PRT
- heat-pipe
- heating elements

- heater temporal stability: 0.1 K
- heat-pipe temperature stability: 10 mK
## Low- / mid-temperature calibration facility

### PTB heat-pipe blackbodies

<table>
<thead>
<tr>
<th>Blackbody</th>
<th>Temperature range / °C</th>
<th>Cavity diameter / mm</th>
<th>Cavity Emissivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>NH₃-BB</td>
<td>-60 to 50</td>
<td>60</td>
<td>0.99990 ± 0.00006</td>
</tr>
<tr>
<td>H₂O-BB</td>
<td>50 to 270</td>
<td>60</td>
<td>0.99980 ± 0.00015</td>
</tr>
<tr>
<td>Cs-BB</td>
<td>270 to 650</td>
<td>41</td>
<td>0.99960 ± 0.00017</td>
</tr>
<tr>
<td>Na-BB</td>
<td>500 to 962</td>
<td>41</td>
<td>0.99960 ± 0.00017</td>
</tr>
</tbody>
</table>
Low- / mid-temperature calibration facility
Low- / mid-temperature calibration facility

辐射温度标准不确定度 (k=1)

<table>
<thead>
<tr>
<th>Radiation temperature / °C</th>
<th>Uncertainty of radiation temperature / °C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1.6 µm</td>
</tr>
<tr>
<td>-60</td>
<td>0.1</td>
</tr>
<tr>
<td>0</td>
<td>0.035</td>
</tr>
<tr>
<td>50</td>
<td>0.035</td>
</tr>
<tr>
<td>100</td>
<td>0.035</td>
</tr>
<tr>
<td>200</td>
<td>0.08</td>
</tr>
<tr>
<td>300</td>
<td>0.1</td>
</tr>
<tr>
<td>400</td>
<td>0.02</td>
</tr>
<tr>
<td>600</td>
<td>0.03</td>
</tr>
<tr>
<td>800</td>
<td>0.06</td>
</tr>
<tr>
<td>960</td>
<td>0.08</td>
</tr>
</tbody>
</table>
Realization and dissemination of radiation temperature scale above the Ag-FP (961.78 °C):

- Gold fixed-point (Au-FP) blackbody radiator: 1064.18 °C
- High temperature blackbody (HTBB): variable temperature, 900 °C to 3000 °C
- High quality transfer standard radiation thermometers (LP3)
- Tungsten strip lamps operated as radiation temperature standards
Gold fixed-point blackbody

- **heat-pipe based design**
- **cavity aperture diameter**: 3 mm
- **$\epsilon = 0.99999$**
- **large fixed-point material amount (~ 3 kg Au)**

excellent performance
Gold fixed-point blackbody

- Freezing plateau

![Graph showing detector signal (normalized) over time.]

- Freezing plateau duration: ~ 90 minutes
- Temperature stability: ~ 10 mK

but: Limitation to one temperature: 1337.33 K
High Temperature Blackbody (HTBB 3200pg)

- **PTB primary standard**
  - Radiation temperature above the Ag-FP
  - Spectral radiance

- Directly Joule-heated cavity (DC, 700 A max.)

- Operating temperature range: 1000 K to 3200 K

- Temporal stability: better than 250 mK

- Large aperture diameter: 20 mm

\[ \varepsilon = 0.999 \pm 0.001 \]
Linear pyrometer LP3

Transfer radiation thermometer for:
- International scale comparisons
- Internal high-temperature scale dissemination

Main characteristics:
- $\lambda_{\text{eff}} = 650 \text{ nm (950 nm)}$
- Temperature range: 800 °C to 2900 °C
- FOV: 0.8 mm Ø at 690 mm
- Good stability and linearity
- Small SSE

$$I_{\text{photo}}(T) = C \cdot \exp\left(-\frac{c_2}{A \cdot T + B}\right)$$

Standard uncertainty:
- 0.3 °C at 800 °C
- to 1.0 °C at 2900 °C

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Radiation temperature calibration artefact

Comparison / Dissemination

Temperature $T_{90}$ (ITS-90)

Primary standard

GOLD-FIXED-POINT BLACKBODY (Au BB)

Detector $\Phi$

High temperature scale – realization and dissemination
Spectral radiance comparator facility

Step 1: Determination $T_{90}$ (HTBB)

Step 2: Determination $T_{(HTBB)}$ (alternative calibration)

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1. **Detector translation unit**: 800 mm
2. **Grating double monochromator**: 0.25 m, additive mode
3. **Chopper/filter wheel**: Imaging mirror $f = 600$ mm, F/#: 8..20
4. **Linear Pyrometer (LP3)**
5. **Ambient temperature blackbody**
6. **Tungsten strip lamp**
7. **Gold-fixed-point blackbody (Au-BB)**
8. **High temperature blackbody (HTBB, 960 °C to 3000 °C)**

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Spectral Radiance Comparator Facility
ITS-90 and high-temperature fixed points

Interpolation

Pt-resistance thermometer

Radiation thermometer

<table>
<thead>
<tr>
<th>Element</th>
<th>ITS-90</th>
<th>High-Temperature Fixed Points</th>
</tr>
</thead>
<tbody>
<tr>
<td>H2O</td>
<td>0.01 °C</td>
<td>660.323 °C</td>
</tr>
<tr>
<td>In</td>
<td></td>
<td>1084.13 °C</td>
</tr>
<tr>
<td>Zn</td>
<td></td>
<td>1492 °C</td>
</tr>
<tr>
<td>Ag</td>
<td></td>
<td>1953 °C</td>
</tr>
<tr>
<td>Cu</td>
<td></td>
<td>2290 °C</td>
</tr>
<tr>
<td>Fe-C</td>
<td>419 °C</td>
<td>1492 °C</td>
</tr>
<tr>
<td>Co-C</td>
<td>527 °C</td>
<td>1953 °C</td>
</tr>
<tr>
<td>Pd-C</td>
<td></td>
<td>2290 °C</td>
</tr>
<tr>
<td>Pt-C</td>
<td>961 °C</td>
<td>2761 °C</td>
</tr>
<tr>
<td>Ru-C</td>
<td>78 °C</td>
<td>2883 °C</td>
</tr>
<tr>
<td>Ir-C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Re-C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ZrC-C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>TiC-C</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Uncertainty ITS-90 (k=2) = 0.2%

Additional fixed points

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Eutectic fixed-point blackbodies

**Metal-Carbon-Fixed points in graphite crucibles**

- crucible material is part of the fixed point material
- no contamination through crucible material
- higher stability
- better reproducibility

<table>
<thead>
<tr>
<th>Metal-Carbon</th>
<th>Fixed Point Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-C</td>
<td>1153 °C</td>
</tr>
<tr>
<td>Co-C</td>
<td>1492 °C</td>
</tr>
<tr>
<td>Pd-C</td>
<td>1738 °C</td>
</tr>
<tr>
<td>Pt-C</td>
<td>1953 °C</td>
</tr>
<tr>
<td>Ru-C</td>
<td>2290 °C</td>
</tr>
<tr>
<td>Ir-C</td>
<td>2474 °C</td>
</tr>
<tr>
<td>Re-C</td>
<td>2761 °C</td>
</tr>
<tr>
<td>TiC-C</td>
<td>2883 °C</td>
</tr>
<tr>
<td>ZrC-C</td>
<td></td>
</tr>
</tbody>
</table>
Design / construction of eutectic fixed-point blackbodies

Both fixed-point cells designs have:

- the same outer diameter
- the same length/diameter ratio of the cavity
- a calculated emissivity 0.9997
- several layers of C/C sheet insulation

Anhalt et al. (2008)
Large- and small- aperture fixed-point cells of Cu, Pt-C, and Re-C
Eutectic fixed-point cell relative comparison

2 identical furnaces
2 radiation thermometer LP3
15 fixed point cells
(NMIJ, BNM-INM, NPL)

- reproducibility single cell: < 50 mK
- difference $T_{\text{melt}}$ Co-C, Pt-C, Re-C: ~ 200 mK
- difference $T_{\text{melt}}$ Pd-C, Ru-C: ~ 400 mK
Thermodynamic temperature determination

Reproducibility
0.2 K – 0.5 K

Measurement uncertainty
k=2, 0.6 K – 1.2 K


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Thermodynamic temperature determination

- radiance comparison method
  1. HTBB temperature \( \sim T_{\text{melt}} \)
  2. Measurement of thermodynamic temperature of HTBB with FR

\[
\frac{L_{\lambda_s}(\lambda, T_{\text{melt}})}{L_{\lambda_s}(\lambda, T_{\text{HTBB}})} = \frac{\exp\left(\frac{c_2}{\lambda T_{\text{HTBB}}} - 1\right)}{\exp\left(\frac{c_2}{\lambda T_{\text{melt}}} - 1\right)}
\]
Thermodynamic temperature determination

- radiance comparison method
  1. HTBB temperature \( \sim T_{\text{melt}} \)
  2. Measurement of thermodynamic temperature of HTBB with FR
  3. Spectral radiance measurement of the HTBB with the LP3

\[
L_{s \lambda} (\lambda, T_{\text{melt}}) = \frac{\exp\left(\frac{c_2}{\lambda T_{\text{HTBB}}} - 1\right)}{\exp\left(\frac{c_2}{\lambda T_{\text{melt}}} - 1\right)}
\]
Thermodynamic temperature determination

**radiance comparison method**

1. HTBB temperature \( T_{melt} \)
2. Measurement of thermodynamic temperature of HTBB with FR
3. Spectral radiance measurement of the HTBB with the LP3
4. Spectral radiance measurement of the eutectic cell in the Nagano furnace with the LP3

\[
\frac{L_{\lambda s}(\lambda, T_{melt})}{L_{\lambda s}(\lambda, T_{HTBB})} = \frac{\exp\left(\frac{c_2}{\lambda T_{HTBB}} - 1\right)}{\exp\left(\frac{c_2}{\lambda T_{melt}}\right) - 1}
\]

Nagano S furnace

<table>
<thead>
<tr>
<th>Nagano S furnace</th>
</tr>
</thead>
<tbody>
<tr>
<td>( T_{melt} )</td>
</tr>
</tbody>
</table>

HTBB 3200pg

<table>
<thead>
<tr>
<th>HTBB 3200pg</th>
</tr>
</thead>
<tbody>
<tr>
<td>( T \sim T_{melt} )</td>
</tr>
</tbody>
</table>

LP3
Thermodynamic temperature determinations of Co-C, Pd-C, Pt-C and Ru-C eutectic fixed-point cells

Anhalt et al.
Thermodynamic temperature determinations of Co-C, Pd-C, Pt-C and Ru-C eutectic fixed-point cells
Metrologia, 43, 2006, pp. S78-S83
Absolute temperature comparison NIST – PTB using NPL cells

<table>
<thead>
<tr>
<th></th>
<th>PTB</th>
<th>U, k=2</th>
<th>NIST</th>
<th>U, k=2</th>
<th>PTB-NIST</th>
<th>U(PTB/NIST), k=2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co-C</td>
<td>1597.16</td>
<td>0.22</td>
<td>1597.43</td>
<td>0.17</td>
<td>-0.27</td>
<td>0.28</td>
</tr>
<tr>
<td>Pd-C</td>
<td>1765.02</td>
<td>0.27</td>
<td>1764.95</td>
<td>0.21</td>
<td>0.12</td>
<td>0.34</td>
</tr>
<tr>
<td>Pt-C</td>
<td>2011.67</td>
<td>0.32</td>
<td>2011.21</td>
<td>0.27</td>
<td>0.53</td>
<td>0.42</td>
</tr>
<tr>
<td>Ru-C</td>
<td>2227.12</td>
<td>0.41</td>
<td>2226.74</td>
<td>0.34</td>
<td>0.48</td>
<td>0.52</td>
</tr>
</tbody>
</table>
Summary

- **PTB instrumentation and experimental techniques for the realization and dissemination of the temperature scale with optical methods**

- **Low-/mid-temperature calibration facility:** -60 °C to 962 °C
  
  **High temperature calibration facility:** 962 °C to 3000 °C

- **Standard uncertainty of the disseminated radiation temperature:**
  
  - 40 mK at -60 °C
  - 10 mK at the Au-FP (1064.18 °C)
  - 1000 mK at 3000 °C

- **Achievable standard uncertainties for radiation thermometer calibrations:**
  
  - 60 mK at -60 °C
  - 30 mK at 400 °C
  - 1000 mK at 3000 °C

- **PTB meets all industrial requirements in the range from -60 °C to 3000 °C**
Emissivity determination at the PTB in the temperature range from 0 °C to 600 °C

C. Monte, M. Becker, B. Gutschwager, E. Kosubek and J. Hollandt

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Outline

Motivation for an accurate emissivity determination

Experimental setup for emissivity measurement in air

Uncertainty

New experimental setup for emissivity measurement under vacuum

Summary
Motivation

\[ \Delta T \approx \frac{\lambda \cdot T^2}{C_2} \cdot \frac{\Delta \varepsilon}{\varepsilon} \]

Temperature Error resulting from 5% Emissivity Error

3000 K @ 1 μm: \( \Delta T = 30 \) K

300 K @ 10 μm: \( \Delta T = 3 \) K

Wavelength of Detection
- 1 μm
- 4 μm
- 10 μm
Motivation

Variability of INCONEL 600 samples

- Preparatory work for the spectral emissivity pilot study of CCT-WG9
- Identical sample preparation, two different sample manufacturers

Only individual emissivity determination allows an accurate temperature measurement
Spectral emissivity measurement in air - setup

Measurement principle:
- Ratio of the spectral radiance of the sample / spectral radiance of the reference blackbody
- Radiance of the detector / surrounding
- Reflectivity of the sample
### Spectral emissivity measurement ranges (air)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature range:</td>
<td>80 °C to 430 °C</td>
</tr>
<tr>
<td>Wavelength range:</td>
<td>4 µm to 40 µm (2500 cm(^{-1}) to 250 cm(^{-1}))</td>
</tr>
<tr>
<td>Direction of observation:</td>
<td>0° to 70°</td>
</tr>
<tr>
<td>Size of measured area:</td>
<td>circular, 10 mm diameter</td>
</tr>
<tr>
<td>Acceptance angle of the optics:</td>
<td>+/- 3°, NA 0.05</td>
</tr>
<tr>
<td>Homogeneity (camera):</td>
<td>circular measurement area, 20 mm diameter</td>
</tr>
</tbody>
</table>

The **directional total emissivity** and the **hemispherical emissivity** are calculated from the **directional spectral emissivity**.
Spectral emissivity measurement in air - example

High-emissivity coating at 250 °C
Application: reference coating for radiation thermometry up to 800 °C
Uncertainty

Emissivity of a SiC-sample at 150 °C

wavenumber / cm⁻¹

emissivity

+/- standard uncertainty

standard uncertainty

wavelength / μm
Emissivity measurement under vacuum - setup

- Radiation sources chamber
- FT-spectrometer
- Detector chamber
- Cooled beamline
- Vacuum-IR-transfer-radiation thermometer (VIRST)
Emissivity measurement under vacuum - setup

**reference blackbodies:** VLTBB and VMTBB

**bolometer**

**FT-spectrometer**

**sample holder**

**off-axis ellipsoid**

- No atmospheric interference
- No convection → accurate determination of the sample surface temperature

**Temperature range:** 0 °C to 600 °C

**Wavelength range:** 1 µm to 1600 µm (10000 cm\(^{-1}\) to 6 cm\(^{-1}\))
Emissivity measurement under vacuum - setup

LN₂- / water cooled enclosure

heating plate

e > 0.98

sample

rotation stage

vacuum sample holder 0 °C to 600 °C
Emissivity measurement under vacuum

Measurement principle:
measurement of the sample against two blackbodies at two different temperatures

Advantage of the method:
the background radiation, the warm components of the FT-spectrometer and the spectral responsivity of the detection system are cancelled
Summary

- Determination of the directional spectral emissivity in air in the temperature range from 80 °C to 430 °C (4 µm to 40 µm)
- Determination of the directional spectral emissivity under vacuum in the temperature range from 0 °C to 600 °C (1µm to 1600 µm)
- Determination of the total and hemispherical emissivity
- Standard uncertainty for the emissivity 1% for samples with an emissivity > 0.3 and a sample temperature starting from 150 °C
Muster