Joint International Symposium on Temperature, Humidity, Moisture
and Thermal Measurements in Industry and Science

Book of Abstracts
Volume A

TEMPMEKO & ISHM 2010
31 May - 4 June 2010, Portorož, Slovenia

Edited by:
Jovan Bojkovski, Gregor Geršak, Vincencij Žužek,
Igor Pušnik, Domen Hudoklin, Gaber Begeš, Valentin Batagelj, Janko Drnovšek

Joint International Symposium on Temperature, Humidity, Moisture and Thermal Measurements in Industry and Science

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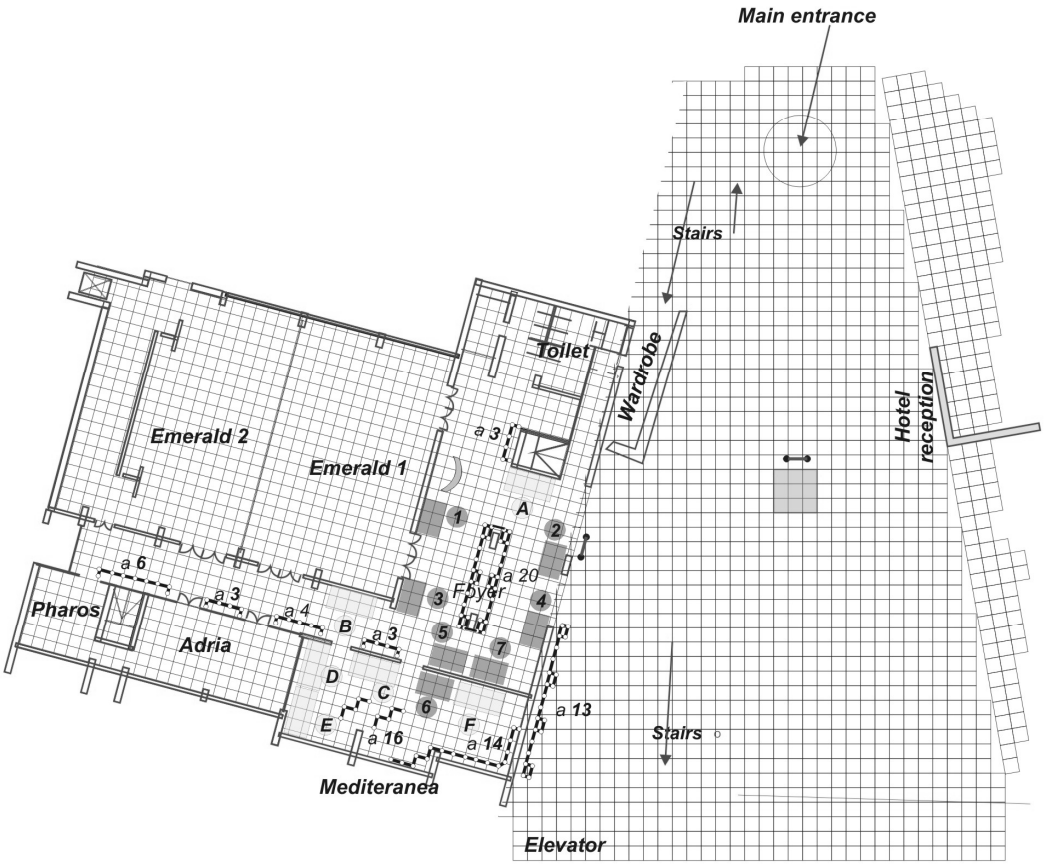
Floor plan

A Fluke - Hart Scientific
B Kambič Laboratory Equipment
C MBW Calibration, RH Systems
D ROTRONIC AG Schweitz
E Isothermal Technology
F Measurements International

1 Pond Engineering Laboratories
2 Guildline Instruments
3 Michell Instruments
4 Vaisala
5 Automatic System Laboratories
6 Mettler Toledo
7 INSCO

Poster panels
Front panels



Conference program

Sunday, May 30th

14:00	Registration *Foyer*		
19:00	Welcome Party	ISOTECH	Tigeroptics

Monday, May 31st

9:00	Opening *Emerald*		
9:30	Plenary session I *Emerald*		
10:00	Plenary session II *Emerald*		
10:30	Coffee break		
10:45	POSTER SESSION I *Foyer*		
11:30	Temperature Scales *Emerald 1 room*	Industrial Applications *Emerald 2 room*	Humidity and Moisture Standards I *Adria room*
13:10	Lunch	Vötsch Industrietechnik	Slovenian representative LT d.o.o.
14:15	POSTER SESSION I *Foyer*		
15:00	Fixed Points - M-C eutectics I *Emerald 1 room*	Calibration Procedures and Facilities I *Emerald 2 room*	Radiation Thermometry I *Adria room*
16:20	Coffee break		
16:50	Resistance Thermometry I *Emerald 1 room*	Fixed Points I *Emerald 2 room*	Interlaboratory Comparisons I *Adria room*
18:10	End of day		

Tuesday, June 1st

9:00	Plenary session III *Emerald*		
9:30	Plenary session IV *Emerald*		
10:00	Plenary session V *Emerald*		
10:30	Coffee break		
10:45	POSTER SESSION II *Foyer*		
11:30	Fixed points - M-C eutectics II *Emerald 1 room*	Fixed Points II *Emerald 2 room*	Water vapour pressure data *Adria room*
13:10	Lunch	Kambič Laboratorijska oprema	
14:15	POSTER SESSION II *Foyer*		
15:00	Resistance Thermometry II *Emerald 1 room*	Calibration Procedures and Facilities II *Emerald 2 room*	Interlaboratory Comparisons II *Adria room*
16:20	Coffee break		
16:50	Uncertainty Estimation *Emerald 1 room*	Fixed Points III *Emerald 2 room*	Radiation Thermometry II *Adria room*
18:10	End of day		

Wednesday, June 2nd

8:20	Thermodynamic Temperature Determinations *Emerald 1 room*	Thermophysical Properties *Emerald 2 room*	Moisture profile and transport *Adria room*
10:20	Coffee break		
10:50	Fixed Points IV *Emerald 1 room*	Calibration Procedures and Facilities III *Emerald 2 room*	Humidity and Moisture Standards II *Adria room*
12:30	End of day		
12:45	Cultural visit	MBW calibration	RH systems

Thursday, June 3rd

8:20	Thermoelectric Thermometry *Emerald 1 room*	Determination of the Boltzmann constant *Emerald 2 room*	Other applied measurements *Adria room*
10:20	Coffee break		
10:30	POSTER SESSION III *Foyer*		
11:10	Fixed points - M-C eutectics III *Emerald 1 room*	Mise en pratique for the definition of the kelvin *Emerald 2 room*	Hygrometers and Moisture Sensors *Adria room*
13:10	Lunch		
14:15	POSTER SESSION III *Foyer*		
15:00	Fixed Points - M-C eutectics IV *Emerald 1 room*	Resistance Thermometry III *Emerald 2 room*	Radiation Thermometry III *Adria room*
16:20	End of day		
19:00	Conference dinner	FLUKE	

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VOLUME A

Plenary session I

Monday
9:30 to 10:00
Emerald
Session Chairman: Jovan Bojkovski

RE-DETERMINATION OF THE BOLTZMANN CONSTANT WITH FIXED-LENGTH CYLINDER

J.T.Zhang[1], H. Lin[1], J. P. Sun[1], X. J. Feng[2], K. A. Gillis[3], M. R. Moldover[3]
[1] *National Institute of Metrology, Beijing 100013, China*
[2] *Tsinghua University,Beijing, China*
[3]*National Institute of Standards and Technology, Gaithersburg, USA*
E-mail (corresponding author): zhangjint@nim.ac.cni

We report progress in using fixed - length cylinders to re-determinate the Boltzmann constant. We investigated perturbations to the modes of gas-filled cylindrical cavities, methods to increase the signal – to – noise ratios, and a method to determine the cavity's length. The perturbations to the acoustic modes due to the thermoacoustic boundary layer, fill ducts and the compliance of the shell were calculated using first - order perturbation theory. The perturbations generated by motion of the shell and fill duct were estimated analytically and measured experimentally. The +measured perturbations from the fill duct were in good agreement with theory. Piezoelectric transducers (PZTs) were used to drive and detect resonances. For our cavities, the PZTs were observed to increase significantly the signal – to – noise ratios of the resonance line – shape measurements giving fractional deviations of 0.2×10^{-6} compared with measurements with traditional condenser microphones with fractional deviation of 1.5×10^{-6}. The PZTs were mounted to diaphragms that were machined into the endplates, so there are no perturbations from gaps or slots and no contamination from elastomers. The compliance of the diaphragm was modeled and measured. The heat dissipated in the source PZT was measured to be less than 1 microwatt at 1 kHz under typical measurement conditions. For reasonances with quality factors Q < 500, we fit the acoustic data with a resonance function that included a frequency-dependent half-width to remove the systematic shifts accounting for boundary-layer perturbations to the measured resonance frequencies. A two - color laser interferometer used to precisely deteminate the cylindrical cavity's length in the room temperature. The thermal expansion of the cavity was measured while the cylinder cooled down to the triple point of water. We completed a preliminary re-determination of Boltzmann constant kB using two cylinders. Our two – cylinder procedure determines kB from the differences between the optical lengths that are identical to the differences of the acoustic lengths of the two cylinders when they are closed with identical endplates. The additional benefit of this comparative procedure is the partial cancellation of the perturbations from the thermoacoustic boundary layer, the transducers, and fill ducts. Our result for kB is compared with the recently published values and the CODATA - recommended value. Our uncertainty budget indicates that the fixed path length cylindrical resonator can achieve a small uncertainty that is comparable to the uncertainties of shperical resonators.

Plenary session II

Monday
10:0 to 10:30
Emerald
Session Chairman: Janko Drnovšek

CHALLENGES OF FINANCING METROLOGY

Dušan Mramor et al.
University of Ljubljana, Faculty of Economics, Slovenia

Temperature Scales

Monday
11:30 to 13:10
Emerald 1
Session Chairman: Wolfgang Buck

COMPARISON OF COULOMB BLOCKADE THERMOMETERS WITH THE INTERNATIONAL TEMPERATURE SCALE PLTS-2000

M. Meschke[1], J. Engert[2], D. Heyer[2], J. Pekola[1]
[1] *Low Temperature Laboratory, Helsinki University of Technology, Espoo, Finland*
[2] *Physikalisch-technische Bundesanstalt, Berlin, Germany*
E-mail (corresponding author): meschke@boojum.hut.fi

The Coulomb blockade thermometer (CBT) is a primary thermometer based on arrays of tunnel junctions between normal metal electrodes. CBTs are usually operated in the regime of weak Coulomb blockade where the charging energy EC is small compared to temperature $E_C << k_BT$ (k_B - Boltzmann constant). Then the absolute temperature is derived by the measurement of the half width $V_{1/2}$ of the dip in the bias voltage dependent conductance curves according to

$$T = \frac{eV_{1/2}}{Nk_B} \frac{1}{5.439(1+0.4\Delta G/G_T)} \qquad (1)$$

where $\Delta G/G_T$ is the conductance dip, N is the number of tunnel junctions in series, and e is the electron charge. Due to the correction term proportional to ΔG/GT in the denominator equation (1) also holds when the condition of small charging energy starts to fail. Alternatively the conductance curves can be calculated numerically without any approximation. An advantage of the numerical calculation is that overheating effects due to small electron-phonon coupling can be included.

CBT has the advantage that many (100) junctions in series produce an enhanced signal, but the distribution in the junction resistances introduces errors in the absolute temperature readout. We expect the fabrication inhomogeneity of the junction resistances to be below 10% resulting in an uncertainty in temperature lower than 2%. The CBTs investigated were fabricated from aluminum. To keep the metal in normal state during the measurements a small magnetic field of 30 mT was applied to the CBT.

Here we report on a comparison of a CBT with a high accuracy realization of the PLTS-2000 in the range from 0.008 K to 0.65 K. An overall agreement of about of 1% was found for temperatures above 0.25 K. For lower temperatures increasing differences are caused by thermalization effects which are described by numerical calculations.

REALIZATION OF ITS-90 ABOVE SILVER FREEZING POINT AT NIM

Z. Yuan, X. Lu, J. Wang, C. Bai, W. Dong, X. Hao, T. Wang
National Institute of Metrology, Beijing, China
E-mail (corresponding author): yuanzd@nim.ac.cn

The high temperature primary standard system has been improved gradually at the National Institute of Metrology of China (NIM) since 2004. A new primary standard pyrometer has been developed, which is of smaller volume and size-of-source effect (SSE). The temperatures of interference filters, photoelectric detector and amplifier are controlled respectively in it. The optical shutter, wheel held the interference filters, and amplifier are programmable.

The new relative spectral responsivity calibration facility, is double grating monochrometer based, and non-linearity measurement facility have established, and they are employed for measuring the pyrometer spectral responsivity and its non-linearity. The results are shown.

The International Temperature Scale of 1990 (ITS-90) above silver freezing point has been realized and maintained by improved scheme. The silver freezing point blackbody is adopted as reference point, and a practical copper freezing point blackbody is used as the transfer reference point. The reference point-pyrometer assemble is used to maintain the scale, which avoids the influence of the instability and inhomogeneity of tungsten strip lamps and can correct the drift of pyrometer.

The primary standard calibration method is adopting to calibrate the standard pyrometers for simplifying the dissemination and improving calibration uncertainty.

Keywords: International Temperature Scale of 1990 (ITS-90), realization, pyrometer, freezing point, blackbody, uncertainty.

TOWARDS AN INTERNATIONAL HIGH-TEMPERATURE THERMODYNAMICTEMPERATURE SCALE ABOVE THE SILVER POINT ?

P. Bloembergen , Y. Yamada
National Metrology Institute of Japan, AIST, Tsukuba, Japan
E-mail (corresponding author): p.bloembergen@xs4all.nl

With the advent of high-temperature eutectics and peritectics with transition temperatures covering the range from 1427 K (Fe-C) to 3458 K(HfC-C) a new era in temperature measurement at high temperature, i.e. above the silver point, will be entered. Under the auspices of WG-5 of the CCT these systems are presently investigated for their suitability to serve as reference points for dissemination of T (and T_{90}) within the context of the '*Mise en pratique of the definition of the kelvin*' (*MeP*-K) at high temperature: *MeP*-K-HT. The interpolating thermometer has to be calibrated against a number n of reference points out of a maximum of n_{max}, as selected; interpolation is essentially done by means of Planck's law.
MeP-K-HT is fully exploiting the advantages of the new scheme by providing multiple ways of realizing temperature including any case $n \leq n_{max}$, allowing the user to realize the scale where it is needed for his application, and to build in redundancy, to safeguard robustness. On the other hand: a new scale to replace or to parallel T_{90} at high temperatures is not envisaged in the foreseeable future. To the opinion of the authors not only industry, but also internationally branched quality-oriented institutions (calibration organizations, quality-assurance systems, standardization bodies), and legal metrology would embrace an officially endorsed, international high-temperature thermodynamic temperature scale (IHT-TTS), underpinned by a
selected set of reference points, to replace or to parallel the high-temperature part of ITS-90.
In this paper it is evidenced that the scheme $n = 2$ provides an optimum in the ways of disseminating T. An optimum combining flexibility in coverage of temperature - in conformity with the application- with effectiveness in terms of accuracy, and with efficiency in terms of expenditure. Redundancy can be implemented to enhance robustness. As such it constitutes an ideal frame for an officially endorsed IHT-TTS. This would not exclude any other scheme in conformity with the *MeP*-K-HT being put into practice but that scheme would not be part of the IHT-TTS; it should be stressed though that results obtained in either scheme are of equal status. Effectively, delineating IHT-TTS would enhance the scope of *MeP*-K-HT in answering user's demands, but any-one would be free to measure T outside the constraints set by IHT-TTS within the free space allowed by the *MeP*-K-HT.
In practice constraints, chosen with care, would add to the reproducibility and comparability of results ensuing from global applied research and global industrial production processes. We would not need a special subscript attached to T within IHT-TTS (although T_I might be considered). When reporting results it could be stated that these are in conformity with IHT-TTS, supplemented by a list of thermodynamic temperatures and associated uncertainties proper to the set of reference points involved, marked and periodically updated by the CCT.

COMPARISON OF EUTECTIC-BASED AND ITS-90 RADIATION THERMOMETRY SCALES

A. Diril, Ö. P. Güven
TÜBĐTAK-UME, Kocaeli, Turkey
E-mail (corresponding author): ahmet.diril@ume.tubitak.gov.tr

Eutectic phase transitions are commonly considered for use as fixed-points in future 20XX temperature scales. Despite their potential as possible interpolation points in high temperature radiation thermometry scale (1000°C and above), the uncertainty in their plateau temperature values seems to be a major problem. Various ongoing research projects on the long-term stability and reproducibility of the eutectic fixed-points will likely improve the uncertainties enough to allow for use as reference temperature points.

In this article we compare the eutectic-based radiation thermometry scale with the well-practiced ITS-90 radiation thermometry scale in the range 1000°C…2500°C. The phase transition values of the eutectic points (Co-C: 1324°C; Re-C: 2474°C) and copper freezing point (1084.62°C) are determined by thermodynamic temperature measurement using filter detectors. In this process the temperature values indicated by the reference radiation thermometer, TSP-2, against a variable temperature blackbody set at 1084°C, 1324°C and 2474°C, are compared with filter detector indications. Later the copper freezing point and Co-C and Re-C plateau values are corrected accordingly to obtain the thermodynamic temperature values.

In the last part of the study, three new scales are established using Cu, Co-C and Re-C temperature values and ITS-90 scheme. A fourth scale is established using the interpolation of the above fixed-points and these four scales are compared in terms of difference and uncertainty.

SPECTRAL RADIATION DRIFT OF LEDS UNDER STEP-MODE OPERATION AND ITS EFFECT ON THE MEASUREMENT OF THE NONLINEARITY OF RADIATION THERMOMETERS

D. Wei[1,2], Y. Zundong[2], P. Bloembergen[2], L. Xiaofeng[2], D. Yuanyuan[1]
[1] *Key Laboratory for Thermal Science and Power Engineering of Ministry of Education, Depart of Thermal Engineering, Tsinghua University, Beijing, 100084, China*
[2] *National Institute of Metrology,Beijing, China*
E-mail (corresponding author): dongw@nim.ac.cn

The non-linearity of radiation thermometers is critically involved when realizing ITS-90 above the silver point. It has to be corrected for and its uncertainty should be adequately specified. In this paper we present results of non-linearity measurements based upon the superposition method and involving LEDs with high radiance output, peaked at a wavelength of 645 nm. To this end the two LEDs in question have been operated in the pulse mode with fixed phase shift. Their spectral radiances have been measured by the radiation thermometers to be tested, separately or superimposed by means of a beam splitter. The LEDs were air-cooled to enhance stability.

Still the drift in spectral-radiance observed after switching on the LEDs has to be taken into account and corrected for, since this drift, interfering during the superposition procedure, could corrupt the sum rule for the fluxes involved, which is the crux of the superposition method. Therefore experimental research has been carried out to characterize the drift of LEDs operated in the pulse mode with fixed phase shift. The result indicated that the drift could be roughly specified in terms of characteristic time intervals: nonlinear drift in the first several seconds followed by a quasi-linear drift in the subsequent time interval. The cross-over time from nonlinear to quasi-linear drift could be ascertained by experiment. In the course of the superposition process switching of the LEDs was done in such a way that only the quasi-linear part of the drift was involved which allowed for the correction of the drift in the NL measurements, as will be reported.

Non-linearity measurements have been performed for two pyrometers at radiance temperatures between the silver point and 3000 K: The primary standard pyrometer of NIM (PSP-NIM) and an LP-4 thermometer, supplied by IKE, with central wavelengths of 660 nm and 650 nm, respectively. For PSP-NIM the range in photocurrent was limited to 10^{-7} A, and radiation temperatures, as observed, had their upper limit at 2800 K. For the LP4 the range in photocurrent was again limited to 10^{-7} A, but still radiance temperatures up to 3000 K could be measured, because of a better overlap of the spectral output of the LED and the spectral responsivity function of the thermometer associated with the shift from 660 to 650 nm. At the same radiance, the drift effects observed for PSP-NIM were a bit higher than that for LP-4, because of the shift of its operational wavelength towards the long-wavelength flange of the LED emission spectrum, where it is more sensitive to temperature variations. Still in both cases the standard uncertainty of the nonlinearity measurement -in relative measure - was better than 10^{-4}.

Industrial Applications

Monday
11:30 to 13:10
Emerald 2
Session Chairman: Yong-Gyoo Kim

THERMAL IMAGE DEGRADATION THROUGH A HEATED SAPPHIRE WINDOW

S.-N. Park, Y. S. Yoo, G. W. Lee, C.-W. Park
KRISS, Daejeon, Korea
E-mail (corresponding author): snpark@kriss.re.kr

Optical windows are indispensable for monitoring industrial processes under vacuum or high pressure by using thermal imagers and radiation thermometers. When the windows are heated by conduction, convection or friction of aerodynamics, the window emits thermal radiation and increases the background signal of the thermal images under observations, which results in image quality degradation. Especially when the wavelength of absorption band edge of the window is sensitive to temperature, and the spectral band of the thermal imager overlaps with the band edge, the situation become more serious. Furthermore, temperature distribution in the heated window caused by non-uniform heating and cooling may introduce extra image quality degradation.

We measured standard four-bar images with various radiance temperature differences using a thermal imager with the sensing spectral band from 3 μm to 5 μm through a UV-grade sapphire window. The four-bar image is generated a blackbody masked with gold coated patterns and transferred to the imager through a collimator. The window is indirectly heated in a furnace and then suddenly extracted from the furnace to be placed in the optical path between the collimator and the thermal imager. The four-bar image degradation is measured as a function of the window temperature and the radiance temperature difference of the four-bar image. We propose an equation which can describe the contrast of the four-bar image by using emissivity of the sapphire window and by assuming the temperature uniformity of the heated window. We have confirmed that the theoretical model agrees well with the experimental observation.

TEMPERATURE MEASUREMENT OF SEMITRANSPARENT SILICON WAFERS BASED UPON ABSORPTION EDGE WAVELENGTH SHIFT

T. Iuchi, T. Seo
Toyo University, Kawagoe, Japan
E-mail (corresponding author): iuchi@toyonet.toyo.ac.jp

The temperature measurement of a silicon wafer is critical for the precise control of any thermally dependent semiconductor processes. The inability to obtain an accurate and reproducible measurement of a wafer temperature, however, remains a major problem obstructing the achievement of reliable and high-quality processing. In semiconductor processes, a non-contact temperature measurement of wafers is a crucial because it is a contaminant-free method. Radiation thermometry is a typical method that meets this requirement. The accuracy of this method is, however, limited by the uncertainties in the emissivity of the silicon wafer and thin film layers deposited on the wafer such as an oxide film. The radiative properties of silicon wafers are complex and changed during the processes. In order to overcome the problem described above, we have developed radiation thermometry that enables a simultaneous measurement of emissivity and temperature of a silicon wafer based upon a polarization technique [1]. This method is, however, limited to relatively high temperature range over 900 K where the wafer is opaque. We are now aiming at the development of an alternative non-contact measurement method that is available for relatively low temperature and semitransparent silicon wafers.

The temperature dependence of transmittance of a silicon wafer is well known. There are various absorption mechanisms over wide range of spectrum. As the wavelength increases, the photon energy decreases until it becomes smaller than the minimum energy gap in the silicon band structure. At this point there is a rapid drop in absorption, which is often referred to as absorption edge wavelength. This wavelength shifts to a longer wavelength with increasing temperature because the band gap energy reduces with increasing temperature.

We designed a measurement system to find a relationship between absorption edge wavelength and temperature. Experiments were carried out from the viewpoints of wavelength (900 ~ 1700 nm), polarization (p- and s-polarized) and direction (normal~80°) for specimens with different resistivity (0.01~2000 Ωcm) that corresponded with the impurity concentration doped into a silicon wafer. The characteristic curve between absorption edge wavelength and temperature of a silicon wafer is obtained and discussed. Once the absorption edge wavelength is measured, the temperature is uniquely determined from this relationship. In this paper, an experimental system and experimental results are described in detail and some remaining problems are discussed.

[1] T. Iuchi and A. Gogami, “Uncertainty in the temperature of silicon wafers measured by radiation thermometry based upon a polarization technique”, XIX IMEKO World Congress, Lisbon, September, 1487-1492 (2009).

ULTRASONIC HIGH TEMPERATURE SENSORS: PAST EXPERIMENTS AND PROSPECTIVES FOR FUTURE USE

M. Laurie[1], D. Magallon[1], J. Pierre[2], C. Marquié[2], S. Eymery[2], R. Morice[3]
[1] *European Commission - Joint Research Center – Institute for Energy, Petten, The Netherlands*
[2] *Institut de Radioprotection et de Sureté Nucléaire, Cadarache, France*
[3] *Laboratoire Commun de Métrologie LNE-CNAM, Paris, France*
E-mail (corresponding author): mathias.laurie@ec.europa.eu

Ultrasonic thermometry sensors (UTS) have been intensively studied in the past to measure temperatures in the range 1800 — 3100 °C. This sensor type using the temperature dependence of acoustic velocity in materials was developed for experiments in extreme environments. Its major advantages, which are (a) recording capability of a temperature profile deduced from the notches on the sensor rod and (b) measurement near the sensor material melting point can be of great interest when dealing with on-line monitoring of high temperature safety tests or long-term high temperature fuel irradiation experiments.

Ultrasonic techniques were successfully applied in several severe accident related experiments under challenging conditions. In the FARO experiments two UTS were used to determine the initial temperature of a UO_2 molten fuel prior to delivery of the melt to the test section for studies of fuel-coolant interaction and erosion of stainless steel structures. In PHEBUS FP experiments, two of these robust temperature sensors were embedded in the experimental bundle to measure up to 8 different zones during the whole degradation transient that led to the formation of a pool of molten UO_2. For these applications, mainly related to severe accident tests to improve safety of nuclear reactors, these sensors have been successfully applied with an accuracy that has been estimated to +/- 50 °C. For these applications and the targeted range of temperature, tungsten sensor rods had to be used. If some new developments are conducted with other materials, this sensor type may be used in other experimental areas where robustness and compactness are required. Long-term irradiation experiments of nuclear fuel to extremely high burn-ups could benefit from this previous experience. This ultrasonic technology could solve the known problem of massive failures of classical N or K type thermocouples during irradiation tests above 950 °C. These sensors may even be up-graded with adapted fixed point cells located in low neutron flux areas that would enable detection and correction of UTS drift.

After an overview of UTS technology, this paper summarizes experimental work performed to improve the reliability of these sensors. The various designs, advantages and drawbacks are outlined and future prospects for long term high temperature irradiation experiments are discussed.

FIXED-POINT THERMOCOUPLES IN POWER PLANTS–OPERATIONAL EXPERIENCES

H. Lehmann
electrotherm GmbH, Geraberg, Germany
E-mail (corresponding author): h.lehmann@electrotherm.de

Since 2005, fixed-point thermocouples have been in routine operation in hot steam pipelines of different power plants. Those sensors are equipped not only with a well chosen fixed-point substance but also with an internal heater and a second thermocouple for service. The implemented software controls a self-adjustment run at least every two week. This procedure involves 3 thermal cycles passing the well-known melting point of the fixed-point substance. The mean deviation of the recorded thermocouple voltage during melting from the reference table value results in an individual offset correction for the considered thermocouple measuring circuit. Hence, a highly reproducible temperature signal is available which serves as a reference value for other conventionally designed thermocouples which are also still in use.
The thermometers reported here have been operating at temperatures ranging from 535 °C to 545 °C. Their fixed-point substance is eutectic silver-aluminium, which melts at 567.7 °C. Before their installation the instruments were calibrated in a laboratory heat pipe furnace with a standard platinum resistance thermometer. By applying very slow heating rates (0.05 K/min), the beginning of the melting could be determined with an accuracy level of 0.3 K (K=2).
During the last four years of operation in hot steam pipelines, their periodical self-adjustment has been observed and analyzed for changes in melting plateau characteristics. The instruments have been exposed to about 200 self-adjustment cycles per year.
As long as no mechanical damage or leakage of fixed-point material occurred, the melting plateau shape did not change significantly. Nevertheless, from time to time one of the operating fixed-point thermometers was exchanged for inspection and recalibration under laboratory conditions. Meanwhile 4 of in total 13 AgAl-fixed-point thermometers have been investigated this way.
One of the most surprising results is that no significant drifts of the melting point were found after 2, 3 or meanwhile also after 3 ½ years. Melting plateaus recorded in the laboratory before and after long-term operation differ by 0.2 K maximum, which can be regarded as non significant. So, the described fixed-point measuring system has proved to be a highly reproducible thermometer for temperatures close to the particular melting point - even in rough industrial environments. In addition, the poster will give an introduction to the basic design and function of the measuring system. Also, the calibration process in the laboratory and the in situ self-adjustment procedure will be illustrated.

Humidity and Moisture Standards I

Monday
11:30 to 13:10
Adria
Session Chairman: Li Wang

NOVEL FLOW CONTROL/MEASUREMENT SYSTEM IN MAGNETIC SUSPENSION BALANCE/DIFFUSION-TUBE HUMIDITY GENERATOR

H. Abe
National Metrology Institute of Japan (NMIJ), AIST, Tsukuba, Japan
E-mail (H. Abe): abe.h@aist.go.jp

The demand for a trace moisture standard has recently increased rapidly in the field of gas analysis because the accurate measurement of residual moisture in gases is considered to be important in industry. To meet this demand, the National Metrology Institute of Japan (NMIJ) has established a primary standard of humidity in the trace moisture region using a diffusion tube method. This method requires the precise and accurate control and measurement of the flow rate of a gas passing through a humidity generator to produce a standard gas of trace moisture, and the uncertainty of the flow rate contributes directly to the uncertainty of the standard.

A thermal mass flow controller (MFC) has been used to control the flow rate at NMIJ. However, the capability of controlling the flow rate using the MFC is reduced at a small flow rate relative to the full scale of the MFC. Furthermore, the MFC may be affected by the fluctuation of ambient temperature. These two effects increase the uncertainty due to the instability of the flow rate.

In this study, a novel flow control/measurement system was developed to improve the control and measurement of the flow rate in the NMIJ magnetic suspension balance/diffusion-tube humidity generator. This system uses a mass flow meter (MFM) composed of multiple critical flow Venturi nozzles, also known as sonic nozzles. The flow rate was measured using the MFM and was adjusted to a set value by controlling the pressure of the gas upstream of the nozzles using a feedback loop with the values measured using the MFM. Using this system, the uncertainty due to the instability of the flow rate was reduced to less than 0.0023 % (expressed as relative standard uncertainty), approximately 65 times smaller than that previously reported, 0.15 %.

A SECOND-GENERATION NIST GRAVIMETRIC HYGROMETER

C. W. Meyer[1], J. T. Hodges[1], R. W. Hyland[1], G. E. Scace[1], J. Valencia-Rodriguez[2], and J. R. Whetstone[1]
[1] *National Institute of Standards and Technology, Gaithersburg, Maryland, USA*
[2] *Advanced Scientific Applications de México, Puebla, Pue, Mexico*
E-mail (corresponding author): christopher.meyer@nist.gov

A second-generation primary hygrometer has been completed at the National Institute of Standards and Technology (NIST). A gravimetric hygrometer measures humidity by separating the water from the carrier gas and afterwards measuring the water mass and carrier gas mass. These two measurements determine the mass ratio r (the ratio of the measured water mass to the measured dry-gas mass). The new design allows automated continuous gas collection at up to 3 L/min. This enables the hygrometer to collect larger amounts of gas and thereby measure humidity values lower than that measured by the previous NIST gravimetric hygrometer.

We present here a brief description of the design and operation of the gravimetric hygrometer. This description includes the recent addition of a cold trap to the desiccant-based water separation/collection system; when included, this trap enables the hygrometer to measure humidity values for gas streams with dew points above 20 °C. We also provide a detailed uncertainty analysis for the hygrometer, including equations relating the total expanded (k=2) relative uncertainty $U(r)/r$ to the uncertainty elements, a table displaying the standard values of the uncertainty elements, and plots of $U(r)/r$ as a function of r. Finally we present the results of recent comparisons of the mass ratio measured by the hygrometer with that generated by the NIST Hybrid Humidity Generator (HHG) over the range −12 °C to 71 °C.

When operated in an optimal thermal environment (minimal thermal loads in the laboratory), the total expanded relative uncertainty of the gravimetric hygrometer is approximately 0.1 % for atmospheric-pressure frost points higher than −35 °C (r = 250 µg/g). Below this frost point the total expanded relative uncertainty gradually increases to approximately 1% at −55 °C (r = 13 µg/g). In the comparisons with the HHG, the differences between the humidity generated by the HHG and that measured by the gravimetric hygrometer are less than the combined expanded uncertainties of the generator and hygrometer.

REALIZATION OF HIGHER HUMIDITY STANDARD BASED ON WATER VAPOR PRESSURE

H. Yi
NIM, Beijing, China
E-mail (corresponding author): yihong@nim.ac.cn

For the present the highest accuracy humidity standard is gravimetric hygrometer and about 10 mK uncertainty of frost/dew point temperature is achieved. In this paper higher humidity standard is proposed based on water vapor pressure. By the new approach, about 1 mK uncertainty of frost/dew point temperature can be achieved. For triple point of water less than 0.1 mK uncertainty of triple point temperature has been achieved. The purity of water and isotope compositions must be clarified on higher humidity standard. Gas-controlled heat pipe can be used to realize liquid-vapor transitions of pure water in the range of 1 °C to 100 °C . In order to determine a new water vapor pressure temperature relations, between –100 °C and 100 °C, with a lower uncertainty than the before, some theoretical work and experimental work must be done.

Keywords: humidity standard, water vapor pressure, triple point of water, gas-controlled heat pipe.

THE NIST STEAM GENERATOR

D. Vega-Maza[1], W. W. Miller[2], D. C. Ripple[2], G. E. Scace[2]
[1] *University of Valladolid, Valladolid, Spain*
[2] *NIST, Gaithersburg,United States of America*
E-mail (corresponding author): dean.ripple@nist.gov

We have constructed a new humidity generator that produces gas streams of known moisture content at temperatures from 85 °C to 200 °C, absolute pressures from 0.2 MPa to 1.6 MPa, and relative humidities from 10 % to 90 %. Unlike most steady-flow generators operating at lower temperatures, the NIST Steam Generator does not rely on saturation of a gas stream with water vapor at a known temperature. Instead, the generator first produces a moist gas stream by injecting fixed-rate streams of dry gas and liquid water into a vaporizer, where the water evaporates into the gas. Next, the gas stream passes into a re-entrant RF cavity. We have previously demonstrated the use of this cavity as a high-temperature hygrometer (the resonant frequency of the cavity depends on the dielectric constant, which in turn depends on the water concentration). Once calibrated, the RF cavity will serve as our reference hygrometer. From the RF cavity, the gas passes into a test chamber where we will mount hygrometer probes under test. From the test chamber, the gas stream passes through one of a set of capillaries. To prevent condensation in the pressure transducer line, this line is purged with nitrogen; the dry nitrogen combines with the moist gas stream upstream of the capillaries. The flow impedance of the capillary, combined with the volumetric gas flow set by water and gas injection rates, determines the operating pressure. Modulation of the purge or moist gas rates, which can be done manually or by an automated feedback loop, gives a simple method for controlling pressure. To maximize operational flexibility, each major component is individually enclosed in an insulated, temperature-controlled aluminum oven. A larger oven encloses these ovens and connecting tubing. Gas entering both the RF-cavity and the test chamber first passes through heat exchangers integral to the aluminum ovens. To minimize corrosion, all of the components exposed to moisture at elevated temperature, except the downstream pressure-control components, are constructed of high-nickel alloys. In the future, we wish to run hydrogen or oxygen through the generator to mimic fuel-cell conditions. The small total volume (< 1 L) and small flow rate (<0.5 L/min) reduce operational hazards from steam scalding or from gas explosion when operating with reactive or explosive gases.

AN INVESTIGATION OF THE CORRELATION BETWEEN CONTACT THERMOMETRY AND DEW-POINT TEMPERATURE REALIZATION

R. Benyon[1], N. Böse [2], H. Mitter[3], D. Mutter[4]
[1] *Instituto Nacional de Técnica Aeroespacial (INTA), Torrejón de Ardoz. Spain*
[2] *Physikalisch-Technische Bundesanstalt (PTB), Braunschweig, Germany*
[3] *E+E Elektronik Ges. m.b.H. (BEV/E+E), Engerwitzdorf, Austria*
[4] *MBW Calibration, Ltd. (MBW), Wettingen, Switzerland*
E-mail (corresponding author): benyonpr@inta.es

Precision optical dew-point hygrometers using chilled mirrors are commonly used in the comparison of standard humidity generators and in the practical dissemination of dew-point temperature. These instruments are also used by many National Metrology Institutes and Designated Institutes as a secondary standard with traceability to a primary realization in another country. In well defined and carefully executed measurement configurations, these transfer standards can be used with reproducibilities better than ± 0.02 °C in the range -50 °C to +95 °C.

The construction of a chilled mirror hygrometer revolves around the basic configuration of a gold or rhodium plated copper mirror mounted on a thermoelectric cooler with a miniature platinum resistance thermometer embedded below the mirror surface in contact with the gas.

Temperature metrologists are well aware of the difficulties of surface temperature measurement, even in carefully designed guarded hotplate designed specifically for the purpose. The dew-point hygrometer sensors are unavoidably in a considerable heat flux, in an assembly with reduced dimensions and close configuration, as required in these devices, with inherently dominating conduction effects affecting the measurement performance.

Whilst there have been many claims by instrument manufacturers as to the “fundamental” nature of the chilled mirror hygrometers, no precise experimental quantification of the uncertainty contribution due to mirror gradients has been published to date.

The correlation between the conventional calibration and characterization of miniature platinum resistance thermometers prior to their assembly in the mirrors of a series of state-of-the-art optical dew-point hygrometers and the subsequent calibration as dew-point temperature measurement devices, are presented and discussed.

POSTER SESSION I

Thermodynamic Temperature Determinations

Monday
10:45 to 11:30
and
14:15 to 15:00
Foyer

DETERMINATION OF THE THERMODYNAMIC TEMPERATURE OF GOLD FIXED-POINT CELLS

M.Battuello, F.Girard, M. Florio
INRIM, Turin, Italy
E-mail (corresponding author): m.battuello@inrim.it

Since the value T_{90}(Au) was fixed on the ITS-90, some determinations of the thermodynamic temperature of the gold point have been performed which, with other renormalized results of previous measurements by radiation thermometry, form the basis for the current best estimates of $(T\text{-}T_{90})_{Au}$ = 39.9 mK as elaborated by the CCT-WG4. Such a value, even if consistent with the behavior of $T\text{-}T_{90}$ differences at lower temperatures, is quite influenced by the low values of T(Au) as determined with few radiometric measurements.

At INRIM an independent determination of the thermodynamic temperature of gold was performed by means of a radiation thermometry approach. A fixed-points technique was used to realize approximated thermodynamic scales from the In point up to the Ag point. Two precision radiation thermometers were used, a Si-based one working at 950 nm and an InGaAs-based at 1.6 µm. The low uncertainty presently associated to the thermodynamic temperature of fixedpoints and the accuracy of INRIM realizations, allowed scales with uncertainty lower than 0.03 K in terms of thermodynamic temperature to be realized.

Two fixed-point cells filled with gold 99.999 % in purity were measured and their freezing temperature was determined by extrapolation with a combined standard uncertainty of 0.05 K. The paper describes the approach and the experimental set-up and presents the results in terms of $(T\text{-}T_{90})$ at the Au point.

THERMODYNAMIC TEMPERATURE MEASUREMETS TRACEABLE TO PHOTOMETRIC STANDARDS

M. J. Ballico
NMIA, Lindfield, Australia
E-mail (corresponding author): mark.ballico@measurement.gov.au

The replacement of ITS-90 temperature measurements by direct thermodynamic temperature measurements based on radiometric techniques in the temperature range above 1000 °C has been proposed by many national measurement laboratories. At the National Measurement Institute of Australia, NMIA, two routes to thermodynamic temperature measurement are underway: (a) the calibration of radiation thermometers using illumination from a laser illuminated sphere and (b) the use of precision apertures and photometers to measure the luminance of a blackbody source. The filter radiometers used in radiation thermometry are generally narrow band interference filters, although this is largely a historical artefact of the common practice of assigning an effective centre wavelength for use with thermal sources. Manufacturers of photometers have great experience in the development of systems with good long-term stability, and use systems based on coloured glass based filters rather than interference filters. In photometry, there is wide experience in the absolute calibration of photometers, and the NMIA scale has demonstrated uncertainties of below 0.14% (k=2). This paper reports on work at NMIA to develop a simple and robust traceability scheme for thermodynamic temperature, based on the use of photometers and a Thermogage furnace with a graphite tube element modified to improve its temperature uniformity and emissivity. A standard calibration of the illuminance responsivity of a photometer was performed by NMIA's photometry group, and the associated relative spectral responsivity measurements were used to determine the colour-temperature-correction between the calibration illuminant and the Thermogage blackbody. A simple luminance meter was constructed using the photometer and pairs of precision apertures 4 mm to 8 mm in diameter, separated by 500 mm, viewing a 6 mm to 14 mm diameter region of the blackbody cavity. Thermodynamic temperature determinations made using various combinations of apertures and photometers showed a range of less than 0.2 °C at 1700 °C, consistent with the calculated uncertainty of 0.29 °C (k=2). ITS-90 measurements made by NMIA's LP5 and HTSP primary radiation thermometers with an uncertainty of 0.16 °C (k=2), are consistent with the thermodynamic measurements. It is suggested that routine thermodynamic temperature determinations can now be made with an effort comparable to that required to realise the ITS-90 above 1000 °C.

ABSOLUTE RADIOMETRY OF METAL-CARBON EUTECTIC FIXEDPOINTSFOR A NEW TEMPERATURE SCALE

K. Anhalt[1], J. Hartmann[1], G. Machin[2], E. Woolliams[2], M. Dury[2], H. Yoon[3]
[1] *Physikalisch- Technische Bundesanstalt, Berlin, Germany*
[2] *National Physical Laboratory, Teddington, UK*
[3] *National Institute of Science and Technology, Gaithersburg, USA*
E-mail (corresponding author): klaus.anhalt@PTB.de

The high-temperature fixed-point (HTFP) research plan was launched under the auspices of the CCT WG 5 with the aim of establishing HTFPs as routine metrological tools [1].

In six work-packages the HTFP research plan develops procedures for the reliable construction of the fixed-point cells, investigates their long-term stability and assigns thermodynamic temperatures to a selected set of fixed-points.

Work-package four, piloted by PTB, is dedicated to improvements of the local realization of thermodynamic temperature measurement for the participating laboratories. This work-package commenced with PTB measuring the thermodynamic temperature for three HTFPs, namely, Co-C, Pt-C and Re-C. These cells were then circulated between the participants in the following sequence NPL, NIST, NMIA, LNE-INM/CNAM, VNIIOFI, KRISS. They will be re-measured at the pilot laboratory in the end of 2010.

The present paper is an interim report of the progress of WP4 providing a summary of the initial measurements made at PTB compared with those of the first two participants.

[1] Machin, G., Bloembergen, P., Hartmann, J., Sadli, M., Yamada Y., “A concerted international project to establish high temperature fixed-points for primary thermometry”, *Int. J. Thermophys.*, 28, 1976-1982, 2007

PREPARATION TO THE MEASUREMENT OF THERMODYNAMIC TEMPERATURE OF HIGH TEMPERATURE FIXED POINTS

B. Khlevnoy, M. Samoylov, S. Ogarev and D. Otryaskin
VNIIOFI, Moscow, Russia
E-mail (B.B. Khlevnoy): khlevnoy-m4r@vniiofi.ru

Within the scope of the CCT-WG5 project on investigation of high temperature fixed point (HTFP) VNIIOFI is going to measure thermodynamic temperature of three HTFP, namely Co-C, Pt-C and Re-C with approximate temperatures of 1597 K, 2011 K and 2748 K.

A radiance mode radiation thermometer of TSP type with the target size of about 1x1 mm will be used for measuring temperature of HTFP cells with a 3 mm blackbody cavity. The TSP is based on a silicon photodiode combined with a narrow band interference filter centered at 650 nm assembled in the temperature stabilized block together with an amplifier. Calibration of the radiation thermometer is traceable to a cryogenic radiometer. The procedure of the calibration is following: cryogenic radiometer – trap detector – irradiance mode filter radiometer – variable temperature blackbody – radiance mode radiation thermometer.

The filter radiometer is a silicon photodiode of S1227 type with a broadband glass filter and a 5 mm aperture. The range of spectral responsivity is about from 400 nm to 600 nm with a maximum at 510 nm. The temperature of the filter radiometer is stabilized using a liquid thermostat. The filter radiometer is calibrated in terms of irradiance spectral responsivity against the trap detector by means of precise 1 m double grating monochromator.

The calibrated filter radiometer is used then for measuring the temperature of a high temperature blackbody equipped with a known area aperture. Immediately after that the TSP thermometer is put in front of the blackbody. Therefore the radiation thermometer is calibrated against the filter radiometer using the blackbody as a comparator.

The paper will describe the facility to be used for the measurements and estimation of the uncertainty.

PHOTOELECTRON THERMOMETERY AS A NOVEL METHOD TO MEASURE THERMODYNAMIC TEMPERATURE

I. Kinoshita[1], J. Ishii[2]
[1] *Yokohama City University, Seto 22-2, Kanazawa-ku, Yokohama, Japan*
[2] *National Metrology Institute of Japan (NMIJ), AIST, Tsukuba, Japan*
E-mail (corresponding author): ikinoshi@yokohama-cu.ac.jp

In an electron gas in thermal equilibrium, using Fermi distribution it is possible to calculate the probability that an orbital at certain energy will be occupied. Since the distribution function depends only on temperature, we propose to use the technique to determine the temperature using Fermi distribution as a good method to measure the thermodynamic temperature. The principle of the method has been established and widely known in physics; however, there are not experimental reports which present practical proposals for the thermometry.

Ultraviolet photoelectron spectroscopy (UPS) measures the kinetic energy of electrons, which emit from a sample surface irradiated by ultraviolet lights. Measuring the electron occupancy as a function of electron energy by UPS and fitting the spectrum to the Fermi distribution function could determine the thermodynamic temperature uniquely. However, to achieve the temperature resolution of 1 K, the required energy resolution of UPS measurements is better than 1 meV, which is not possible even by the recent state-of-the-art UPS equipments.

We are now developing a new electron energy analyzer as “photoelectron thermometer” with higher energy resolution and higher sensitivity than those of existing systems. A new design of hemispherical electrodes to measure the electron energy has been introduced and the possibility of higher energy resolution was verified. Our numerical calculations show that the “photoelectron thermometer” will have the energy resolution around 1 meV.

The “photoelectron thermometry” is a novel techinique for thermodynamic temperature measurement. Since the process of photoemission in principle is not restreicted by temperatures, it can be applied for a wide range of temperatures. Moreover, since the measured photoelectron spectrum originates from a few atomic layers of surfaces, it will be an advanced non-contact technique of surface temperature sensing. We believe that our proposed “photoelectron thermometry”, independent of other physics value of materials, will be a new standard for calibration of practical thermometers.

RADIOMETRIC TEMPERATURE COMPARISONS OF THREE NIST GOLD FREEZING-POINT BLACKBODIES

N. Sasajima[1], C. E. Gibson[2], V. Khromchenko[3], R. D. Saunders[2], H. W. Yoon[2]
[1] *NMIJ, AIST, Tsukuba, Japan*
[2] *NIST, Gaithersburg, MD, USA*
[3] *NIST/USU Joint Program in Optical Sensor Calibration, Space Dynamics Laboratory, Utah State University, North Logan, UT, USA 84341*
E-mail (corresponding author): howard.yoon@nist.gov

In the International Temperature Scale of 1990 (ITS-90), the temperatures above the freezing temperature of Ag can be derived from the fixed-point temperatures of Ag, Au, or Cu freezing temperature blackbodies and radiances from Planck's radiation law. Although in ideal situations, the freezing temperatures of the respective metals are presumed to be equivalent, the transition temperatures will be depend upon many factors such as the material purity, cavity design, furnace design and operational parameters. The long-term use might also be compromised by the possible contaminations of the pure metal during use. Thus, to determine whether there are intrinsic differences in the temperature scales at the different national metrology institutes (NMI) related to the ITS-90 realizations, the fixed-points should be directly compared.

Direct fixed-point blackbody comparisons are difficult due to the need to transport the furnaces and the power supplies between the institutes. Furthermore, the cost of the quantity of the pure metal required can be a barrier to constructing the number of fixed-points needed for comparisons, and due to the expense of acquiring the quantity of pure Au, many NMIs utilize fixed-points made from either Ag or Cu.

We compare three Au fixed-point blackbodies with three different furnaces utilizing Na-heatpipe liners. Two of the blackbodies are constructed with same physical dimensions and the other blackbody was slightly smaller. The blackbodies were operated in two furnaces with different physical dimensions but with a similar design. Measurements of radiance temperatures were performed with and without a graphite aperture to determine the change in the cavity emissivity. The emissivity was also modeled, and the results were compared with the experimental change in the radiance temperatures.

RECENT PROGRESS IN NOISE THERMOMETRY AT 693 K USING QUANTIZED VOLTAGE NOISE RATIO SPECTRA

W. L. Tew[1], S. P. Benz[2], P. D. Dresselhaus[2], H. Rogalla[3], D. R. White[4], and J. R. Labenski[5]

[1] NIST, Gaitherbsurg, MD USA
[2] NIST, Boulder CO USA
[3] University of Twente, Enschede, The Netherlands
[4] Measurement Standards Laboratory, Lower Hutt, New Zealand
[5] BAE Systems, Arlington, VA USA
E-mail (corresponding author): wtew@nist.gov

We report on technical advances and new results in noise thermometry at temperatures near the zinc freezing point using a Quantized Voltage Noise Source (QVNS). The temperatures are derived in a series of separate measurements comparing the synthesized noise power from the QVNS with that of Johnson noise from a known resistance at both 505 K and 693 K. Reference noise power spectra are digitally synthesized to match the thermal noise powers producing noise ratio spectra which are close to unity in the low-frequency limit. The relative ratio spectra contain information on the ratio of the two noise temperatures. Direct comparison of noise temperatures to ITS-90 is achieved in a comparison furnace with standard platinum resistance thermometers.

The data are analyzed as spectral ratios over the 4.8 kHz to 450 kHz range using tone spacings of 1.6 kHz and 3.2 kHz. A three-parameter model is used to account for differences in impedance-related time constants that are inherently temperature dependent. We report on the observed noise temperature ratios and related statistics together with estimated uncertainties. These relative noise thermometry results are combined with results from other thermodynamic determinations at temperatures near the tin freezing point to calculate a value of $T - T_{90}$ for temperatures near the zinc freezing point. These new results achieve a lower uncertainty than that of our earlier efforts and we compare the present value of $T - T_{90}$ to other published determinations from noise thermometry and other methods.

POSTER SESSION I

Temperature Scales

Monday
10:45 to 11:30
and
14:15 to 15:00
Foyer

INVESTIGATING THE INCONSISTENCY OF ITS-90 FOR SPRTS IN THE SUB-RANGE 0 °C TO 419.527 °C

J. P. Sun[1], J. T. Zhang[1], Z. R. Kang[2], Y. Duan[1]
[1] *National Institute of Metrology, Beijing 100013, China*
[2] *Institute of Metrology of Hebei Province, Shijiazhang City 050051, China*
E-mail (corresponding author)：sunjp@nim.ac.cn

The sub-range inconsistency is a significant factor to uncertainty in the standard platinum resistance thermometer (SPRT) sub-ranges of ITS-90. This paper investigated the inconsistency between the water-zinc and water-aluminum sub-ranges. The calibration data for 60 SPRTs from four manufacturers were analyzed, and the result conformed that the coefficient c in the interpolation of ITS-90 is available to determine the inconsistency in this temperature range again. The inconsistency, Δt, can be simply equal to 59.83c.

Key words: inconsistency; SPRTs; fixed points; interpolation equation.

ANALYSIS OF INCONSISTENCY BETWEEN THE ITS-90 - INTERPOLATIONS ABOVE 0.01°C

KangZhiru[1], Lanjinbo[2], Z. Jintao[3], Sunjianping[3], Jincheng[1]
[1] Metrological Institute of Hebei Province, Youyi Southern Street 175, Shijiazhuang City 050051, P.R.China
2 Work Safety Bureau of surpervision of Hebei province
3. National Insititute of Metrology

The inconsistencies in the ITS-90 interpolations above 0.01 ▫ were investigated. All of inconsistency functions in 15 overlap regions in the range are analyzed by means of factoring and extramum analysis. There is a very closed relation between the roots and extrema for any of 15 functions and them can be divided into two classes according to the properties of roots of them. Of the functions, 5 ones are only with the fixed real roots which occur at the fixed points shared by two interpolation equations and of simple structure and metremum representation. Others have one or two real roots or a pair of complex roots varying with the coefficients of interpolation equations involved bisides a fixed real root at the triple point of water. These variable real or complex roots effect the extrema of the functions and result in some of them complex. More than 20 SPRTs are used to determined the magnitude of inconsistency above 0.01 ▫ . The result shows that maximum of them are 7 mK .

COMPARISON OF TEMPERATURE SCALES BELOW SILVER POINT REALIZED BY RADIATION THERMOMETERS

T. Keawprasert, N. Rujirat, P. Wongnut, E. Puttiwong
National Institute of Metrology Thailand, Pathumthani, Thailand
E-mail (corresponding author): thada@nimt.or.th

At National Institute of Metrology Thailand (NIMT), a 0.65 mm radiation thermometer is calibrated by the extrapolation method according to the ITS-90 using a reference fixed point of silver or copper, in combinations with a spectral responsivity measurement and a non-linearity measurement to realize the temperature scale above the freezing point of silver (Ag, 961.78 °C) up to 2,500 °C. Below the freezing point of silver, a 0.9 mm and a 1.6 mm radiation thermometer are generally calibrated by the multi-point interpolation method using a series of fixed point blackbodies with out spectral responsivity characteristics to realize the temperature scale down to the freezing temperature of zinc (Zn, 419.527 °C) and indium (In, 156.5985 °C).

The most significant uncertainty component in this calibration is the long term drift of the fixed points due to the oxidation especially for zinc and other fixed point metals with lower transition temperatures. To validate these fixed point temperatures, a correlate temperature scale following to the extrapolating method is realized by the relative spectral responsivity of radiation thermometers and the Planck function. The difference between these temperature scales should be within their combined uncertainty.

This paper describes the methods used and the comparison results between the multi-fixed points scale and spectrally characteristic temperature scale with their combined uncertainty. An outlook to absolute spectral radiance calibrations of the radiation thermometers is also discussed for determinations of the thermodynamics temperature.

REALIZATION OF THE ^{3}He VAPOR PRESSURE SCALE AND DEVELOPMENT OF A LIQUID-He-FREE CALIBRATION APPARATUS

T. Shimazaki, K. Toyoda, O. Tamura
National Metrology Institute of Japan, AIST, Tsukuba, Japan
E-mail: t.shimazaki@aist.go.jp

The ^{3}He vapor pressure temperature scale was realized using an apparatus based on a continuously operating ^{3}He cryostat at National Metrology Institute of Japan (NMIJ), AIST. The cryostat has two operational modes; a ^{3}He circulation mode and a 1 K pot mode. The ^{3}He circulation mode can be used for ^{3}He vapor pressure measurements below 1.6 K, and the 1 K pot mode can be used for measurements above 1.3 K. Either mode can be selected for measurements from 1.3 K to 1.6 K. The realization of the ^{3}He vapor pressure temperature scale in this work covers fully its defined temperature range from 0.65 K to 3.2 K in the International Temperature Scale of 1990. The latest realization results are presented in this paper. In addition, a liquid-helium-free calibration apparatus was developed. It does not require liquid helium as a cryogen, which usually entails cumbersome handling and periodic refilling. The apparatus was designed for the calibration of capsule-type resistance thermometers from 0.65 K to 24.5561 K (the triple point of neon). The cooling system of the apparatus consists of a commercially available pulse tube refrigerator and a ^{3}He Joule-Thomson cooling circuit developed at NMIJ, AIST. The pulse tube refrigerator is used in a precooling stage and cools the apparatus to approximately 5 K. The ^{3}He Joule-Thomson cooling circuit is used to cool the apparatus from 5 K to below 0.65 K. Since the ^{3}He Joule-Thomson cooling circuit is a closed circuit, the apparatus can run continuously for at least several weeks with only simple maintenance operations required. The basic characteristics of the apparatus are described in this paper.

THE DISCONTINUITY IN THE FIRST DERIVATIVE OF THE ITS-90 AT THE TRIPLE-POINT OF WATER

R. L. Rusby
NPL, Teddington TW11 0LW, UK
E-mail (corresponding author): richard.rusby@npl.co.uk

This paper investigates the discontinuity in the derivative [dW/dT90]TPW of the ITS-90 at the triple-point of water, using data for over 40 calibrated SPRTs. It finds that the discontinuity is in most cases somewhere between 0 and -6 parts in 105, in relative terms, but that the higher numerical values are obtained for 'less ideal' SPRTs (those with lower temperature coefficients of resistance), and also for sub-ranges not extending beyond the indium point.

These results are investigated vis-à-vis the long-standing observation that the ITS-90 reference values Wr(Ga) and Wr(Hg) are not completely consistent with data for W(Ga) and W(Hg) for real SPRTs. It discusses what may be done in a future scale to ensure continuity in the first derivative, and it concludes with a comment about the acceptance criteria for SPRTs in the scale.

REALIZATION OF TEMPERATURE SCALE UNDER FIXED POINT OF SILVER BY MEANS OF UNFILTERED RADIOMETERS

P. Nemeček, R. Dubnička
SMU, Bratislava, Slovak Republic

Non-contact measurements in the range of lower temperatures under freezing point of silver become more and more interesting at all levels of non-contact thermometry measurements. Slovak Institute of Metrology is trying to evolve of method in this range by unfiltered radiometers using of trap radiometers with silicone photodiodes and plane detectors with InGaAs photodiodes for measuring thermodynamic temperature of models of blackbody radiators. This method should disseminate of realization actually used international temperature scale in accordance with ITS-90 document towards lower temperatures by means of non-contact thermometry. Furthermore this method would provide continuously measurements of unknown thermodynamic temperatures of blackbody radiators at primary standard level without needs of using interpolation or extrapolation methods between fixed points which these mathematical methods oftentimes leads to imprecision in non-contact realization of temperature scale. Fixed points of ITS-90 in that temperature range will serve for verifying of invented method. The base of evolved method is in using of Planck's radiation law with combination theory about radiometers and their physical properties. For realization of temperature scale in considered temperature range is appearing as one way of possible method using of unfiltered selective radiometers. In this group of radiometers can belong for example detectors in trap configuration with silicone photodiode or single InGaAs photodiode which are temperature stabilized. Advantages for this experiment with bare radiometers are various. One of them is higher level of radiation power at considered temperatures and therefore higher signal to noise ratio in comparison with filtered radiometers using for non-contact temperature measurements. Mathematical model presents following equation that is relation of signal of radiometer $S(T)$ and measured temperature T (K)

$$S(T) = G_{A_1 \to A_2} \cdot \prod_i Ki \cdot \int_\lambda H(\lambda, T) R(\lambda) d\lambda$$

where $G_{A_1 \to A_2}$ is geometrical factor that represents interchanging of radiation between surface A_1 of aperture in front of blackbody source and surface of aperture A_2 in front of detector, $H(\lambda,T)$ is spectral intensity of emittance of blackbody source from Planck's radiation law, $R(\lambda)$ is absolute spectral responsivity of used detector and $\prod_i K_i$ is product of correction factors due to various influences at practical realisation of proposed method. Some of correction factors depend on used radiometer for detecting radiation from blackbody source. For example if it use InGaAs radiometer owing to his better sensitivity in near infrared radiation, then it arise the problem with absorption

of water vapour in that range. Presented mathematical model above can already indicate technical realisation of proposed method. It is very simple idea. Two apertures that one is in front of blackbody source, second is in front of detector and distance between these apertures limited geometry of whole system. From equation presented above than it is possible determine the thermodynamic temperatures of blackbody source. Preliminary measurements in Slovak Institute of Metrology was performed in 2007 and indicated possibility of using unfiltered trap or single photodiode detectors as detectors used for realisation of thermodynamic temperature scale by radiometric means.

THE COMPARISON OF THE PERFORMANCE OF HTSPRT VERSUS THERMOCOUPLE AROUND FREEZING POINT OF SILVER

R. Strnad, M. Šindelář
CMI, Prague, Czech Republic

The International temperature scale ITS-90 defines the scale by means of standard platinum resistance thermometer (SPRT) or high temperature SPRT (HTSPRT) to the freezing point of silver. In this article will be presented the investigation of four HTSPRTs from two manufactures to maintain the scale. The special attention were paid for possibility to use for higher temperatures with implementing their long and short term stabilities.

The second part is focused to the comparison of these four HTSPRT with several thermocouple of type S, B, Pt-Pd and Pt-Au to the temperature of freezing point of copper. The different calibration equations will be applied to fulfill the experimental results. The special attention was paid to the possibility of interpolating and extrapolating of the results.

The results of these experiments will be used for the EURAMET project for the investigation of the possible interpolation instrument in the range of 660 °C to 1080 °C which is prepared with the cooperation of ČMI, GUM and SMU.

POSTER SESSION I

Fixed Points

Monday
10:45 to 11:30
and
14:15 to 15:00
Foyer

THE INFLUENCE OF TITANIUM ON THE ALUMINIUM FIXED-POINT TEMPERATURE

P. Petchpong[1], D. I. Head[2], J. Y. H. Au[3]
[1] *Department of Thermometry Metrology, National Institute of Metrology (Thailand), 3 / 4-5 Moo 3 Klong V, Klong Luang, Pathumthani 12120, Thailand*
[2] *Thermometry Consultant, 21 Chelsea Close, Hampton, Middlesex, TW12 1RS, UK*
[3] *Advanced Manufacturing and Enterprise Engineering Group, Brunel University, Uxbridge, Middlesex, UB8 3PH, UK*
E-mail (corresponding author): patchariya@nimt.or.th

This work describes the deliberate doping of high purity (99.9999%) aluminium with pure (99.8%) titanium impurity and the effect of this on the temperature of the aluminium liquid-solid phase transition (660.323 °C). The concentration of the Ti impurity was of the order of 0.9 and 1.8 ppm (parts per million, 1 in 10^6) in the aluminium. The aluminium sample was in the form of ~0.3 kg ingot that would normally be used to realise an ITS-90 fixed point. Measurements were made using equipment and a fixed-point ingot normally available in a metrological thermometry laboratory, rather than using a specially prepared experiment. Samples from the ingot were chemically analysed (by Glow Discharge Mass Spectrometry (GD-MS)) before and after the doping. Using the mass of dopants introduced, and/or the chemical analysis data, the measured temperature changes were compared with those calculated from a standard text on the thermal effects of impurities. The experimental undoped liquid-solid transition curves were also compared against theoretical curves (calculated from a theoretical model MTDATA developed by NPL, UK). The melting and freezing curves of aluminium indicate that the temperatures were increased by adding 0.9 ppm Ti while, unexpectedly the temperature remained unchanged after doping to 1.8 ppm of Ti. Therefore, it is calculated that the titanium impurity increases the aluminium temperature by +5 mK/ppm, (our first experimental result) or +2.5 mK/ppm (2nd result).However if we use the final temperature deflection and the GD-MS measured value of impurity (rather than that based on our measured masses) then one obtains 3.2 mK/ppm. It is interesting to note that from the phase diagram calculation [Hansen, 1958], the fixed-point temperature of aluminium was interpolated to be elevated by 3.31 mK/ppm of titanium impurity. It is possible that this closer value is by chance as the GD-MS claims a factor of 2 uncertainty (though it has been suggested that this is an overestimate).The results also suggest that perhaps not all the Ti is evenly distributed even though the sample was left molten for periods of 72 hours. Some of the freezing curves of the aluminium doped with Ti may also show evidence of a “concave up” shape at the start of the freezing curve, as previously calculated by MTDATA, though the effect is not as pronounced. This is significant as previous report experiments have not been able to see this.
Key words: Fixed-point, Aluminium temperature; ITS-90; Temperature calibration; Titanium metal impurity.

THE INFLUENCE OF LOW LEVEL ANTIMONY ON THE TIN FIXED-POINT TEMPERATURE

P. Petchpong[1], D. I. Head[2], J. Y. H. Au[3]
[1] *Department of Thermometry Metrology, National Institute of Metrology (Thailand), 3 / 4-5 Moo 3 Klong V, Klong Luang, Pathumthani 12120, Thailand*
[2] *Thermometry Consultant, 21 Chelsea Close, Hampton, Middlesex, TW12 1RS, UK*
[3] *Advanced Manufacturing and Enterprise Engineering Group, Brunel University, Uxbridge,Middlesex, UB8 3PH, UK*
E-mail (corresponding author): patchariya@nimt.or.th

This work focuses on the affects of low level concentrations of antimony impurity, doped in high purity (99.9999%) tin, particularly on the tin melting and freezing curves. Investigations are made in order to improve the measurement of the temperature shift caused by the dopants at low concentrations, in order to test the interpolation of previous data; all of which were obtained from relatively high impurity concentrations. Impurities are a major source of uncertainty in a fixed-point temperature (of the order of 1 mK). A better understanding of the impurity effect is required to improve top-level metrological thermometry. Most historical experiments with impurities have worked at a much higher level of impurities – say of the order of 100 ppm - and in experimental arrangements that are not used on a day-to-day basis in a metrology laboratory. The freezing point temperature of tin (231.928 °C) is one of a series of metallic fixed points on the International Temperature Scale of 1990 (ITS-90). The temperature offsets and shapes of melting and freezing plateaus of high purity tin were investigated as a function of low levels impurities (mass fraction ~1-25 parts per million (ppm)) of antimony metal. The melting and freezing curves, before and after doping, confirm the reproducibility of the temperature measurements in this tin fixed-point cell. The freezing temperatures of the tin after adding antimony are higher than for the undoped sample. It indicates that at low levels, antimony elevates the melting and freezing temperatures although the temperature change is less than expected.

Furthermore, the tin samples were analysed by glow discharge mass spectrometry (GDMS) before and after doping to compare the impurity level calculated by addition of known masses of impurity with that obtained by GD-MS chemical analysis of the impurity elements. There was evidence that the thermal history of the metal phase transitions can cause considerable segregation of some impurities, particularly those likely to increase the phase transition temperature through a peritectic ("positive" impurities), and that the effects of this segregation can be clearly seen on the shape of the melting curves of the tin doped with Sb.
Keywords: Fixed-Point Temperature; Antimony Impurity; Tin Melting/Freezing Curves; Thermometry; Metrology.

STUDY FOR THE REPRODUCIBILITY OF THE SUPERFLUID TRANSITION TEMPERATURE OF HELIUM IN THE PAST TEN YEARS

L. Peng[1], Y. Lihong[2], Z. Zhe[1]
[1] *National Institute of Metrology, Beijing 100013, China*
[2] *Technical Institute of Physics and Chemistry, Chinese Academy of Sciences, Beijing 100190, China*
E-mail (corresponding author): linp@nim.ac.cn

Based on the self-adjusting effect of the height of the normal helium column within the capillary, a method has been developed in China to realize the superfluid transition temperature of helium using a sealed cell with a capillary. A temperature plateau is obtained with a small heat flow along the capillary such that an interface of HeI/HeII is maintained within the capillary. Because there is a depression of the superfluid transition temperature of helium by a heat flux, a serial of heat flows are applied to the capillary and the temperature plateaus are recorded. An extrapolation is employed to determine the superfluid transition temperature of helium with zero heat flow. This paper reports the study for the reproducibility of the superfluid transition temperature of helium in the past ten years. The measurements have been made in four laboratories with a regular procedure since 2000, as six cells sealed in 2000 are used. A rhodium-iron resistance thermometer with series number of 229841 is used at forty-three measurements to realize the superfluid transition temperature of helium with the standard deviation of 0.035 mK. The measurements have been made for the new cells sealed in 2009 and the realization results are agreed well with that of the cells sealed in 2000.

IMPACT OF THE TIME SPENT IN THE LIQUID PHASE ON THE LIQUID-SOLID TRANSITION

E. Renaot, M. Elgourdou, C. Martin
Laboratoire commun de métrologie /LNE-CNAM
61, rue du Landy
93210 La Plaine Saint Denis, France
e-mail : eliane.renaot@cnam.fr

An earlier study completed in our laboratory showed that, in regular realization of the melting-freezing plateaus, there is no diffusion of the impurities within the ingot [1]. This is probably related to the fact that the speed of diffusion of the impurities is infinitely slow, even in the liquid phase. On the other hand, it is frequently noticed that the experimental conditions before the freezing plateau have an impact on its characteristics (value, slope, ...). Up to now, no systematic study was performed on the influence of this parameter. So, the objective of the task started recently in our laboratory is to investigate the influence of the time spent in the liquid phase on the phase transition.

An open cell was filled by using the method known as “the funnel method” with small cylinders of aluminum of nominal purity 99.9999 % (6N). It was observed that the time spent in the liquid phase after melting has a noticeable impact on the freezing range. The freezing range ΔT changes from 6 to 1.5 mK depending of the time t in liquid phase. The relation ΔT versus t is not linear.

Taking into account the Brownian nature of the movement, it is possible to conclude that the diffusion front advances according to a law proportional to the square root of time. The experimental results are in agreement with this formula as ΔT is proportional to that of t^2.

As a final result, it is demonstrated that in order to reach the equilibrium of the concentration of impurities, it is necessary to ensure that the metal remains in the liquid phase at least 24 hours before initiating the freeze.

At the end of the process the aluminium ingot was chemically analyzed. The analyses reveal large contaminations of the surface of the ingot (sodium, sulphur and phosphorus). In order to investigate the pollution brought by various furnaces (heat pipe, 3 zones) and the impact of diverse protective devices a new exploration is in progress. The results of this study, at the time of the congress, will be presented.

[1] *Aluminium cell contamination and impurities diffusion* EURAMET 732 Workshop, NPL, 3rd and 4th June 2008

THE EFFECT OF THE INITIATION PROCESS OF FREEZE ON THE MEASUREMENT RESULT OF AL FREEZING POINT TEMPERATURE

M.Y. Abasov, S.F. Gerasimov, A.G. Ivanova, A.I. Pokhodun, O.S. Shulgat
D.I. Mendeleev Institute for Metrology, Saint Petersburg, Russia
E-mail (corresponding author): S.F.Gerasimov@vniim.ru

In conformity with CCT documents, "…for the freezing curves of the metallic fixed points, the maximum observed temperature on the plateau should be taken as the best approximation of the liquidus temperature. The fixed points should be realized with the inner and outer liquid-solid interfaces and extend past the maximum by 10% to 20% of the fraction frozen, to clearly establish the value of the maximum and the resolution of its determination." Also, it is accepted that "…the inner interface is essentially static. It is the temperature of the inner liquid/solid interface that is measured by the thermometer."

Analysis of freezing curves obtained by using the standard method of fixed point realization shows, that parameters of initial segment of freezing curve, which mean temperature value is usually taken as liquidus temperature, depend on the way of inner interface initiation. Variations in initiation duration and intensity lead to alterations in initial segment of freezing curve and in SPRT measurement result. Moreover, duration ratio of plateau initial segment with minor temperature change and final segment with significant slope also depends on initiation method and furnace temperature.

The influence of freezing initiation conditions on the measurement result is individual for each fixed point because of the differences in thermophysical properties of metals and in conditions of heat transfer from liquid-solid interface to thermometer. Aluminium has maximum value of specific melting heat in comparison with other metals used in ITS-90 fixed points; at present study the influence of intensity and duration of inner liquid-solid interface initiation was investigated both experimentally and by calculation method.

A COPPER POINT FOR CONTACT THERMOMETRY

J. P. Tavener
Isothermal Technology Ltd (Isotech), Southport, United Kingdom
E-mail: info@isotech.co.uk

It would be nice if the temperature scale for contact thermometry ended at Copper (not Silver).
Two problems would have to be overcome before a truly successful copper point could be available for Contact Thermometrists.

1. A stable high temperature thermometer is required to measure the properties of a Copper cell (the melt curve of a copper cell 6N pure should be around 1 to 2 mK) and to define the freeze slope we need to measure to 1/10 mK.

2. As a cladding for the cell an alternative to quartz glass is required as the integrity of the quartz glass is suspect at this temperature (1084.62 °C).

This article describes attempts to create a stable and robust copper point cell and measure its performance to 1/10 mK.
The temperature scales for Contact Thermometry ends at the Silver Point (961.78 °C). However this was not the original plan. To have extended the scale to Copper Point meant having accurate and stable thermometry that would work up to 1100 °C.
These were not available during the 1980's. Evidence that the Copper Point is need by Contact Thermometrists is shown by the number of such cells in use.
The current Copper Cells have limitations in that they are normally clad, like the lower temperature cells, in quartz glass. At 1090 °C the quartz is soft and so the cell tends to have a limited life.
In Radiation Pyrometry the Copper Point Cell is housed in a metallic container and is purged during use with pure Argon, the cell is mounted horizontally.
A new approach is described here which combines the best of the radiation pyrometry solutions with the requirements of Contact Thermometrists to produce a robust and stable solution.
As part of the development the performance of a Platinum/Palladium Thermometer and a new design of High Temperature Standard Platinum Resistance Thermometer (HTSPRT) were investigated to improve measurement at the Copper Point.
The presentation describes the progress made to date and the uncertainties achieved, together with suggestions for further work.

WATER TRIPLE POINT ANALYSIS COMPARING CELLS MADE FROM QUARTZ AGAINST THOSE FROM BOROSILICATE GLASS

J. P. Tavener, N. Davies
Isothermal Technology Ltd (Isotech), Southport, United Kingdom
E-mail: info@isotech.co.uk

Three years ago, at Tempmeko we presented the results of over 100 Water Triple Point Cells comparing these cells to a reference cell and to V-SMOW. These cells were made between 2001 and 2005 when K7 report of 22 laboratories Water Cells were intercompared by BIPM, and BIPM adopted V-SMOW as the isotopic ideal for the ITS-90 Water Triple Point Cell.

This article updates this information by adding a further three years results to the above and attempts to compare results of cells made of quartz glass to those made from borosilicate glass.

During 2005 and following the K7 data we modified our still to produce water of V-SMOW composition.

Of the hundreds of cells produced during 2005, 2006, 2007, 2008 and 2009 many have been UKAS certified, by comparing them to a Reference Cell.

Others have been sampled and the samples sent for isotopic analysis.

More and more customers have preferred quartz glass containers for the pure water in preference to the traditional borosilicate glass containers, on basis that the quartz cell has a drift rate 1/10 of that of borosilicate cells.

The authors have analysed the results of the comparisons and isotopic analysis to give an idea of the spread of results, and also to attempt to show what improvements result from the use of quartz rather than borosilicate glass.

INVESTIGATION OF THE PARAMETERS OF SEALED TRIPLE-POINT CELLS FOR CRYOGENIC GASES

B. Fellmuth, L. Wolber
Physikalisch-Technische Bundesanstalt (PTB), Berlin, Germany
E-mail (corresponding author): bernd.fellmuth@ptb.de

Within the framework of an international star intercomparison of realisations of temperature fixed points, the parameters of a large number of sealed triple-point cells for the cryogenic gases hydrogen, neon, oxygen, and argon have been investigated at PTB. The work was directed to optimise the measurement of the melting curves as well as to establish complete and reliable uncertainty budgets. The investigated cells are quite different with respect to their design, the construction materials, and the amount of fixed-point substance. The parameters determined are internal thermal resistances, thermal time constants, heat capacity, heat of fusion, the sample portion showing pre-melting and the supercooling temperature. The influence of the freezing and measuring conditions on the parameters has been carefully checked. In the paper, an overview of typical parameter ranges is presented. Special emphasis is given to the question, whether the parameters are primarily influenced by the cell design or the properties of the fixed-point substances.

In view of the small heat capacities of the cells and the fixed-point samples, a surprising result is the fact that very large time constants of the thermal recovery after the heat pulses of the intermittent heating through the melting range may exist. Furthermore, the time constants may depend on the freezing conditions, which has been observed especially for neon [L. Wolber, B. Fellmuth, 2008, Proceedings of TEMPMEKO 2007, Int. J. Thermophys. 29, pp. 82-92]. In the paper, a simple model is developed, which explains very large recovery times of several hours if the thermal equalization requires melting and refreezing, respectively, of sample portions, which have different melting temperatures. According to this model, melting and freezing can be treated by a newly defined heat-capacity equivalent, which considers the heat of fusion of the fixed-point substance as well as the melting-temperature inhomogeneity. The equivalent yields together with the internal thermal resistances of the cell an estimate for the resulting time constant.

The thermal recovery follows usually not a simple exponential law because different thermal resistances and processes are involved in the thermal equalization. An analysis of the recovery requires, therefore, a description with a graded set of exponential functions containing different time constants. The way of separating these functions is described, too.

PRODUCTION OF A NEW TIN CELL AT INRIM

P. P. M. Steur, R. Dematteis
Istituto Nazionale di Ricerca Metrologica, Torino, Italy
E-mail (corresponding author): p.steur@inrim.it

During the years some of the INRiM metal fixed-point cells, dating back some twenty years or more, have shown a variable deterioration showing in the leaking of some of the metal through the walls of the crucible. The variation in the deterioration is assumed to be connected with the frequency with which the cells have been used. New cells are therefore being produced at INRiM as substitutes for the older cells. A description of the new cells and their preparation is given, along with a preliminary comparison between the new cell and the older cell(s).

SOME CURIOUS RESULTS WITH A GALLIUM FIXED POINT CELL

P. P. M. Steur, R. Dematteis
Istituto Nazionale di Ricerca Metrologica, Torino, Italy
E-mail (corresponding author): p.steur@inrim.it

During 2009 most of the Gallium fixed-point cells in use in different INRIM laboratories were compared with Italy's National Standard. The comparison has uncovered problems with one of the commercial devices, realizing a temperature about 7 mK too low which initially was even changing linearly with time. An additional series of measurements was undertaken to find out the reason for this behaviour, but not being allowed to open the cell, only a suspicion on the possible cause has remained. During these measurements a way was found that might give users an indication of such misbehaviour of their cell. The results underline the importance for those NMIs with only a single cell, for any fixed point, to undertake regular comparisons with another cell as a check on its behaviour.

THE α-β TRANSITION OF OXYGEN AS A SECONDARY FIXED POINT OF ITS-90

A. Szmyrka-Grzebyk[1], L. Lipiński[1], A. Kowal[1], P.P.M. Steur[2], F. Pavese[2]
[1] *INTiBS, Wrocław, Poland*
[2] *INRIM, Torino, Italy*
E-mail (corresponding author): A.Szmyrka@int.pan.wroc.pl

Below the triple point of oxygen two solid-solid phase transitions occur, recommended by BIPM as secondary fixed points of ITS-90: the *β–γ* transition at a temperature of about 43 K and the *α–β* transition at the lower temperature of about 23.8 K. The value of the latter phase transition temperature, T_{90} = 23.868 K as recorded in BIPM documents, is given with an associated uncertainty of 5 mK. At INTiBS investigations of this transition were carried out using a few different thermometric cells produced by IMGC (presently INRIM). A small latent heat of about 80 J/mole was found for the transition. The temperature of the phase transition was defined as the temperature of the heat-capacity maximum during the transition. Temperature was measured by means of RhFe resistance thermometer B178, calibrated by NPL. The transition temperature was observed to change by several millikelvin without any correlation with the experimental conditions (cooling speed, heating power, annealing at a chosen temperature). The numerical values found for the temperature of the *α–β* transition in oxygen in the tested cells were higher by about (20 – 30) mK than the BIPM recommended value. The results obtained at INTiBS were confirmed by measurements carried out at INRIM.

During the work on the phase transition hysteresis effects were observed to occur. The temperature difference between the *α–β* transition and the *β– α* in oxygen was about 150 mK.

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AUTOMATED REALIZATION OF THE TRIPLE POINT OF WATER WITH THE MUSH METHOD

X. K. Yan[1], Y. N. Duan[1], J. H. Yang[2], J. Li[3]
[1] *National Institute of Metrology (NIM), Beijing, China*
[2] *Beijing University of Chemical Technology, Beijing, China*
[3] *University of Science and Technology, Beijing, China*
E-mail (Xiaoke Yan): yanxk@nim.ac.cn

According to the different crystallization mechanisms of high purity water, there are two methods to freeze an ice mantle in a triple point of water (TPW) cell. One is the sheath method, which prepares the ice mantle from the inside outward using the coolants such as liquid nitrogen, solid carbon dioxide, liquid-nitrogen-cooled rods, immersion cooler, and other low temperature liquids. The other is the mush method, which creates the ice mantle from the outside inward. Compared with the sheath method, the mush method is more convenient for checking the stability of reference standard platinum resistance thermometers (SPRTs) in a secondary level temperature calibration laboratory.

In order to investigate the mechanism of phase transition of water using the mush method, many efforts have been devoted to developing an automated device based on the thermoelectric cooling and heat pipe technologies. In this paper, we describe the design principle, apparatus, and procedure for automated creating the ice mantles in the small TPW cells.

It has been found that the supercooled water in the cell can spontaneously transform into the uniform metastable fine crystals throughout the cell, when the supercooling reaches a certain temperature. Also, the temperatures of spontaneous phase transition differ for different small cells. However, for a certain cell, these crystallization temperatures vary gently. Moreover, the metastable crystals can change into the stable structure crystals, forming the ice mantle from outside inward. Furthermore, in terms of the thermodynamic solidification theory, some pertinent explanations are given to describe the whole phase transition procedure when using the mush method.

According to our obtained experimental results, after approximately 6 hours when transient forming the metastable ice crystals, the realized triple point temperatures of water are in good agreement within 0.1 mK. Furthermore, the small TPW cells (s/n 008 and s/n 001) in the abovementioned apparatus were directly compared against a conventional TPW cell (s/n NIM-1-211). The obtained results indicate that the equilibrium temperatures realized by the cells (s/n 008 and s/n 001) are –0.21 mK and –0.23 mK lower than that of the cell (s/n NIM-1-211), respectively.

DESIGN, CONSTRUCTION AND TEST OF MERCURYTHERMOMETRIC CELL IN CENAMEP AIP

R. Ortega-Cortés, A. Araolaza-Rodríguez
CENAMEP AIP, Panamá, Panamá
E-mail : rortega@cenamep.org.pa

The temperature measurements with high accuracy are possible due to the materialization of the International Scale of Temperature of 1990; also known as ITS-90. The ITS-90 is formed by a group of thermometric cell built with pure elements, measuring instruments and interpolation rules; that allow to extend it from 0,65 K (-272,5 ºC) to the highest temperature in the practice that can be measured in terms of Plank's radiation law.

The thermometry laboratory of the National Metrology Center of Panama AIP (CENAMEP AIP, for its acronyms in Spanish), who has the mission to keep and disseminate the national measurements standards of temperature quantity, it has seven (7) thermometric cells commercially acquired allowing to cover an interval between 0 ºC and 1084 ºC; however, there was no available primary references for temperatures under 0 ºC.

The cell built in CENAMEP AIP, allow to obtain the temperature primary reference defined by the ITS-90 of -38,8344 ºC. For the construction of the cell, were used 0,68 kg of high pure mercury, coded as reference material 743 and certified by NIST (USA). The container for the mercury was made considering the amount of mercury available and the required immersion length for a Standard Platinum Resistance Thermometer to obtain correct measurements values at these temperatures.

The value of reproducibility obtained in the realizations of the triple point of mercury, in the thermometric new cell by the laboratory personnel of CENAMEP AIP, is below one millesimal of Kelvin (1 mK). This value meets the current needs the accuracy of our laboratory and has been confirmed through technical inspections conducted by Phd. Edgar Méndez (CENAM, Mexico) and by assessing the quality of the results conducted by the expert in thermometry of fixed point Mrs. Patricia Giorgio (INTI, Argentina), whom we thank for their collaboration.

With this new reference CENAMEP AIP may calibrate, the Standard Platinum Resistance Thermometers, interpolators of ITS-90. These thermometers are used to calibrate the working standards used to verify sensors and controllers of low temperature in refrigerators, cold rooms and food & medicine chambers.

Keywords: Thermometric cells, ITS-90, National Standard, Traceability.

COMPARISON OF THE TRIPLE POINT OF NEON USING DIFFERENT SEALED CELLS AT NMIJ/AIST

T. Nakano, O. Tamura and H. Sakurai
National Metrology Institute of Japan (NMIJ), AIST, Tsukuba, Japan
E-mail (corresponding author): tnt@ni.aist.go.jp

The triple point of neon (Ne) is included among the defining fixed points of the International Temperature Scale of 1990 (ITS-90). At NMIJ/AIST, we realized the triple point of Ne using a NMIJ cell (S/N NMIJ Ne-5) (Int. J. Thermopysics 28 (2007) pp. 1893-1903) and a modular sealed-cell model (TEMPERATURE Vol. 7 part 1 (2003) pp.173-178) made by Istitute Nazionale di Ricerca Metrologica (INRIM) in Italy, and compared their results. The isotopic composition of the sources will be analyzed in a Joint Research Project. The results of the isotopic analysis will be also presented elsewhere in this conference [1].

The melting curves of the triple point of Ne using the both cells are obtained by the same procedure and the same calorimeter using a two stage Gifford-McMahon type cryogenic refrigerator as reported earlier.

The melting curves obtained by using the NMIJ cell show narrow widths (0.1 mK) in the wide region of the inverse of the fraction of melt ($1/F$) from $1/F$=1 up to $1/F$ =20, which is consistent with our former report. On the other hand, the width of melting curves obtained by using the INRIM cell is 0.2 mK from $1/F$=1 up to $1/F$ =20. The liquidus point T_{tp} estimated by the melting curves using the NMIJ cell is 0.07 mK hotter than that using INRIM cell. In the final manuscript, we will discuss the origin of the difference of T_{tp} estimated by using both cells.

[1] F. Pavese et al.,"Recent progress toward the determination of T-x(22Ne) for neon isotopes" in this conference.

ARGON TRIPLE POINT FOR LONG STEM SPRTS: THERMAL BEHAVIOUR

M. Dobre[1], I. Didialaoui[2], Y. Hermier[2]
[1] *SMD, 16 Bd. Du roi Albert II, 1000 Brussels, Belgium*
[2] *Laboratoire commun de métrologie, LNE-CNAM, 61, rue du Landy, 93210 La Plaine St Denis,France*
E-mail (corresponding author): Miruna.Dobre@economie.fgov.be

For more than thirty years, the French national laboratory, INM (now LNE-INM/CNAM) has been developing an argon triple point device for long stem thermometer calibrations. The original idea was to propose a very simple system, robust, and avoiding any electronic instrumentation, except the one associated to the thermometer under calibration. In such a system, the phase transition is observed by a so called “heat flux method”, by setting the temperature of the “surroundings” of the thermometric cell “slightly” higher than the temperature transition. In the particular case of the argon triple point realization, the temperature of the liquid nitrogen bath is controlled by adjusting the pressure within the cryogenic Dewar.

In the frame of the EURAMET project 732, it was proposed to re-visit this argon triple point device in order to ameliorate the uncertainty value, mainly due to the thermal aspects. In the actual system, due to the lost of liquid nitrogen with time, the thermal immersion of the thermometer is changing with time accordingly. Temperature in the liquid nitrogen bath is controlled by a mechanical pressure regulator, and particularly at the level of the thermometric cell, is dependant of the atmospheric pressure and hydrostatic pressures.

The improvement of the system is a goal shared by two laboratories, LNE for the design and realization of the device and SMD for the electronic regulation of the pressure. LNE developed a system allowing constant thermal conditions for the thermometer under calibration during the whole argon transition time. First, the dewar design permits the same thermal immersion of the thermometer during the experiment time. Second, the regulation principle has been revised: the pressure control is driven by the liquid nitrogen temperature at the level of argon cell.

In this paper, the authors presents the results obtained in the new system, compared them with the results obtained in the previous system and re-visit the uncertainty budget for long stem thermometer calibrations at the argon triple point.

FIXED-POINT-COMPARISON UNCERTAINTIES AT THE ZINC POINT WITH TWO CELL GEOMETRIES

A.-K. Gerlitzke[1], S. Rudtsch[1], M. Fahr[2]
[1] *PTB, Berlin, Germany*
[2] *NRC, Ottawa, Canada*
E-mail (corresponding author): anne-kathrin.gerlitzke@ptb.de

To realize the ITS-90 according to its definition, the melting and freezing temperatures of ideally pure metals are needed. Therefore, at most National Metrology Institutes, for each temperature instead of one single cell, a group of cells containing the same metal and realizing the same phase transition is considered as the national reference.

By direct fixed-point cell comparisons on a regular basis, these allow to account for the small differences between the individual fixed-point temperatures and to detect possible temperature drifts of the cells. At PTB, during recent years, these groups of national standard cells and socalled transfer cells for calibration work, have been complemented by newly-developed slim fixed-points. These cells typically contain (75-80) % less fixed-point material compared to standard cells.

Slim cells are used for homogeneity investigations of large batches of fixed-point material, for doping experiments to determine the influence of very small amounts of impurities on the fixedpoint temperature with very small uncertainties, and for the investigation of contamination or purification effects after the manufacture of a fixed-point cell.

Our investigations have shown that the main limitation of slim cells is the quality of the phase boundary. The small dimensions of the cell allow neither for the formation of a closed phase boundary nor the formation of two phase boundaries. However, this can be compensated using a quasi-adiabatic realization procedure, and uncertainties comparable to those of standard fixedpoint cells can be achieved.

In this paper we present the design of the cells, as well as typical measurement results and uncertainties for the direct comparison of fixed-point cells for both, standard size and the slim design.

TOWARDS CARBON DIOXIDE VAPOUR PRESSURE THERMOMETER

A. Kartal Dogan, G. Bonnier, A. Uytun, I. Kocas, Y.Durgut,
TUBITAK Ulusal Metroloji Enstitüsü (TUBITAK UME), Gebze-Kocaeli, Turkey
E-mail (corresponding author): aliye.kartal@ume.tubitak.gov.tr

Temperature Group Laboratory of National metrology Institute of Turkey (TUBITAK UME) has been realizing the scale between the range from argon triple point (83.8058 K) to copper freezing point (1357.77 K) and also constructing ITS-90 defined fixed points.

The aim of TUBITAK UME Temperature laboratory is to use the properties of carbon dioxide (CO_2) vapour pressure to realize a vapour pressure thermometer for covering the range from 216 K up to room temperature. This realization is intended to be considered as an approximation of the international temperature scale in this range of temperature.

The vapour pressure thermometer will be assessed by using the triple point of carbon dioxide and by measuring the pressure at the mercury point temperature.

At the present time three cells having different designs has been constructed and characterized. Besides a new design of new cell so-called double cell which contains mercury and carbon dioxide is designed and used at low temperatures.

The thermal effect and the effect of impurity on temperature will be investigated and presented in this paper.

INVESTIGATION OF GALLIUM MELTING POINT AS A CROSSCHECK FOR WATER TRIPLE POINT MEASUREMENTS

R. Silva, H. D. Vieira, R. N. Teixeira, S. G. Petkovic
INMETRO, Rio de Janeiro, Brazil
E-mail: rdsilva@inmetro.gov.br

The Thermometry Laboratory of INMETRO has recently started investigating the use of Gallium Melting Point as a crosscheck for Water Triple Point - WTP - measurements when it comes to checking SPRT (Standard Platinum Resistance Thermometer) drifts after other fixed point measurements. Some WTP measurements may present deviations that are due to the cell itself and not to the thermometer under calibration. This allows room for improvement regarding the use of the WTP cell, once there are many points to be considered such as isotopic composition of the water used (which must be known and corrected mainly for deuterium, ^{2}H, in order to approach Vienna Standard Mean Ocean Water VSMOW), age of the cell (due to dissolution/leaching of the components of the borosilicate glass envelope over time, mainly silicon and boron) and age of the mantle (recommended time to achieve best accuracy, as the impurities get incorporated in the solid phase and therefore change the realisation temperature). Many researchers in the field of Thermometry have published papers dealing with the issue of water, providing results after massive investigation of WTP cells. These results claim that the major problems regard impurities in the water, which can be determined by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with samples of the source water. Additionally, a study published a couple of years ago states that the leaching of impurities from the borosilicate glass envelope into the water represents a decrease in the WTP realisation of approximately 14 µK per year. Minimal influence is due to deviations in isotopic composition of the water. Due to the problems and difficulty of use/maintenance the water cells present, authors claim Gallium to have a special place in the next international temperature scale or even substitute for the WTP as a reference point for SPRT resistance measurements (Rs).

The investigation proposed consists of measuring both Gallium and WTP cells in order to establish their typical profiles and observe the influence of the problems mentioned above. This would comprise repeated measurements of Gallium MP from frozen to total molten without SPRT withdrawal (total curve) so as to determine the typical slope value (intrinsic to melting realizations) and its repeatability/reproducibility. After that, intermittent measurements will be performed so as to check if they reproduce the profile observed before in total curve measurements. As for water, measurements are being performed from 24 hours after mantle formation to at least 15 days of mantle growth to test stability of the mantle and determine the best period to be utilised (considering totally frozen mantles condenses almost all impurities). The same calibrated 25,5 Ω SPRT, the thermometric bridge and resistors will be used in all measurements. After this stage, tests will be done with thermometers under calibration in order to check results after exposure of the SPRTs to other temperatures (mainly the range covering up to Zn). The results achieved so far show good accordance with the literature, in which after the first ten days, the values measured at the WTP showed a high level of stability. Tests are still being performed to evaluate long-term mantle stability.

DEVELOPMENT OF THE TRIPLE POINT OF ARGON SYSTEM

R. Ding, M. Zhao, E. Nerdrum, D. Meier
Fluke Corp.- Hart Scientific Division, American Fork, UT, U.S.
E-mail: rong.ding@hatscientific.com

A triple point of argon system was developed to realize the argon triple-point temperature defined in the International Temperature Scale of 1990 (ITS-90) and to calibrate long-stem standard platinum resistance thermometers (SPRTs). The system structure, the techniques for realization of the triple-point temperature, and the testing results are presented in this paper.

In this system, there is a central argon cell that includes four re-entrant wells with immersion depth of 160 mm which can be used to calibrate four long-stem SPRTs simultaneously. A high-accuracy temperature controller is used to control the realization process of the argon triple-point temperature plateau. The argon triple-point plateau can be easily realized with this system. The influence of different experimental parameters on the triple point of argon plateau was investigated and is discussed. The testing results show that the duration of the plateau can be over 100 hours with the temperature change less than 0.05 mK. The temperature homogeneity of the four re-entrant wells was tested. The immersion profile of the system was measured, and the measurement results were compared with the ITS-90 hydrostatic values. The uncertainty analysis shows that the uncertainty of the argon system is 0.25 mK (k=2).

Keywords: fixed point, ITS-90, SPRT calibration, temperature, triple point of argon.

BEHAVIOUR OF TRIPLE POINT OF WATER CELLS FOR DIFFERENT CONDITION OF ICE MANTLE

K. Kodaira
Japan Electric Meters Inspection Corporation(JEMIC), Tokyo, Japan
E-mail: k-kodaira@jemic.go.jp

The Triple Point of Water (TPW) is usually realized by preparing ice mantle in a sealed glass cell containing pure water that has substantially the isotopic composition of ocean water. The shape and amount of the ice mantle formed in the TPW cell are different in the condition every preparation. The preparation of the ice mantle is required suitable techniques to obtain the uniform ice mantle.

The water sealed in the TPW cell slightly contains impurities even if the pure water is used. Further it is considered that the elements of the glass of TPW cell dissolve in the water due to deterioration with time. As the results, the triple point temperature is changed owing to impurities. The degree of the temperature change depends on the impurities concentration. As most of impurities exist in liquid water, the impurities concentration changes with the amount of ice in the TPW cell. Consequently, there is a possibility that the realization temperature change by the difference of the amount of ice mantle even if the identical TPW cell is used.

In the present work, the effect of the difference of the amount of ice mantle on the realization temperature was investigated. Three TPW cells with different manufacturing time and a standard TPW cell were used in this experiment. First, an ice mantle about 5 mm thick is formed along the thermometer well for three TPW cells, and these TPW cells were compared with the standard TPW cell. Next, an ice mantle about 25 mm thick is formed along the thermometer well for three TPW cells. And, these were similarly compared with the standard TPW cell. In the case of the standard TPW cell, the ice mantle about 10 mm thick is formed along the thermometer well in each case. These measurements were alternately repeated. As the results, although the identical TPW cell was used, the realization temperature of TPW changed when the amount of ice mantle was different.

This paper is intended to report the experimental results.

STUDY ON THE REALIZATION OF ZINC POINT AND THE ZINC-POINT CELL COMPARISON

J. V. Widiatmo, M. Sakai, K. Satou, K. Yamazawa, J. Tamba, M. Arai
National Metrology Institute of Japan, (NMIJ/AIST), Tsukuba, Japan
E-mail (corresponding author): janu-widiatmo@aist.go.jp

Continuing our study on aluminium, tin and silver points, study on the realization of zinc point was conducted. Zinc-point cells were newly fabricated using 6N-nominal grade zinc samples, impurity elements of which were analyzed extensively based on the GDMS. The present paper reports the temperature measurements done using the newly fabricated cells during zinc freezing process, under which the zinc fixed-point is defined, and the analysis of the freezing curve obtained. Comparisons of zinc-point temperatures realized by the newly fabricated cells (cell-to-cell comparisons) were also conducted. Zinc-point depression due to impurity elements was calculated based on the sum of individual estimates and the impurity element analysis. One of the cells evaluated was drawn out from its crucible and analyzed by GDMS at four points, namely at around the center of the top, of the middle, of the bottom and around the outer part of the middle area. The purpose of this cell disassembly is to see whether or not there has been some difference before and after cell fabrication, as well as difference in impurity element distribution within the ingot.

From the aforementioned studies some findings were obtained. First finding is that the homogeneity of impurities in the zinc ingot was within 30%, except for Pb, which was more concentrated in the center part. Accordingly, temperature obtained when the zinc point is realized by the inner mantle would be different from that by the outer mantle. Second finding is that cell-to-cell temperature difference changes along with the progressing solidification process. As consequence, for an accurate cell-to-cell comparison, location in freezing plateau where the comparison is done should be determined. Third finding is that the slope analysis estimates accurately the cell-to-cell comparison, and is consistent with the impurity analysis. It is, therefore, applicable for evaluating the effect of impurity to the zinc point realization, especially after the cell fabrication.

CONFIRMING IMPURITY EFFECT IN SILVER POINT REALIZATION FROM CELL-TO-CELL COMPARISONS

J. V. Widiatmo, K. Harada, K. Yamazawa, J. Tamba, M. Arai
National Metrology Institute of Japan, (NMIJ/AIST), Tsukuba, Japan
E-mail (corresponding author): janu-widiatmo@aist.go.jp

As continuation to our earlier work on silver point realization, already reported in Tempmeko 2007, a new silver point cell has been fabricated using 6N-nominal grade material, having been analyzed by means of a glow discharge mass spectrometry (GDMS) by the manufacturer. This new cell is evaluated by a direct cell comparison with one of the existing cells, which was already reported in Tempmeko 2007. One of those existing cells is drawn out from its crucible and its ingot is analyzed by GDMS at four points, namely at around the center of the top, of the middle, of the bottom and around the outer part of middle areas for a purpose of confirming whether or not there has been difference in impurities before and after cell fabrication, as well as difference in impurity homogeneity within the ingot.

As results of the aforementioned measurements, it is found that the homogeneity of impurities in the zinc ingot was in average within 50%, and there was a tendency that impurities were concentrated around the middle part of the thermometer well. It is also found that cell-to-cell temperature difference changes along with the progressing solidification process. As consequence, for an accurate cell-to-cell comparison, location in freezing plateau where the comparison is done should be determined. Also obtained here is that the slope analysis was consistent with both the cell-to-cell comparison and the impurity analysis.

COMPARISON OF NEON SAMPLES OF DIFFERENT ISOTOPIC COMPOSITION AND DETERMINATION OF THE TRIPLE POINT TEMPERATURE OF PURE ^{20}Ne AND ^{22}Ne WITH $U \approx 50$ μK

F. Pavese[1], P. P.M. Steur[1], S. Valkiers[2], N. Bancone[1], D. Ferri[1], D. Giraudi[1]
[1] *INRIM, Torino, Italy*
[2] *IRMM, Geel, Belgium*
E-mail (corresponding author): f.pavese@inrim.it

The paper reports on new determinations of the triple point temperature, Ttp, of neon for seven samples of different 22Ne isotopic fractions, 22x, partly already involved in collaborative studies aimed at establishing the Ttp vs 22x relationship. Several problems remained unsolved in the presently published results. The new measurements at INRIM intend to supplement thermal data of lower uncertainty to help resolving some of these problems. The detailed results of the INRIM measurements are reported, characterised by a much improved uncertainty, concerning 35 meltings and including calorimetry, thermometry and sample-related effects, for the first time performed at INRIM on cryogenic cells sealed by PTB, NPL, NMIJ and INRIM. First, a detailed analysis is provided of the broader range of information collected and processed in order to assess with sufficient confidence expanded uncertainty levels of the order of 30 μK for a single cell and 50 μK for the comparison of pairs of cells. Then, the quality of the results is discussed, leading to an evaluation of the uncertainty budget for these measurements, which in most cases reached the aimed improved uncertainty level. This analysis includes the effect of chemical impurities and the hydrostatic head correction. Finally, the results are discussed in relation to their effect on the future assignment by the collaborative study of a sufficiently accurate and reliable Ttp vs 22x relationship. The paper discusses the improvements that have been obtained, regarding the influence of the thermal measurements uncertainty on the relationship uncertainty, and to the ability of discriminating between effects induced by the latter and those induced by the possible presence of isotopic fractionation in sample preparation, or by so far undetected incorrect values of 22x in some measured samples.

In addition, the paper reports on the first results on new determinations at INRIM of the triple point temperature of pure neon isotopes 20Ne and 22Ne, obtained on one sample for each isotope sealed in cryogenic cells and with an uncertainty much lower than that of the old literature data. Preliminary temperature values on ITS-90 are reported for both isotopes, together with the value of the difference between them, which is useful to the studies about methods to take into account the isotopic variability of natural neon in the realisation of the ITS-90. The results are compared with those obtained on samples of natural neon, and the problems involved in the correction for the residual content of isotopic impurities and of chemical impurities are discussed.

THE EFFECT OF MAGNETIC FIELD ON THE REALIZATION OF TRIPLE POINT OF WATER

G. Geršak and J. Bojkovski
MIRS/UL-FE/LMK, Ljubljana, Slovenia
E-mail (corresponding author): jovan.bojkovski@fe.uni-lj.si

In every day life we are subject to ac and dc extraneous magnetic fields (from electromagnetic sources like transformers, fans, heaters, Earth magnetic field, indoor lightning, medical devices like MRI scanners, etc). The effect of magnetic field on water molecules is well-known from numerous references in the field of chemistry, e.g. crystal growth and water research. When water freezes and crystals are formed, changes in water molecules crystallization can be observed when exposed to magnetic fields. The crystal shape, size and speed of crystallization correlate to the magnetic flux density of the applied magnetic field. Also, there are number of papers dealing with introduction of strains in the ice during the process of realization of triple point of water and formation of fine ice crystals during the preparation of the mantle and the relief of strains and the gradual conversion of fine ice crystals to larger ice crystals.

In this paper we would like to present effect of magnetic field on the realization of the triple point of water. First method includes magnetic treatment of water prior to triple point of water realization and the second one during the actual triple point realization. The magnetic treatment is performed by different direct and alternating magnetic fields (permanent magnets, air-cored Helmholtz coils and electromagnets with magnetic flux densities from 50 uT to 250 mT for dc and 50 Hz). After the realization, the triple point of water cell has been relaxed for at least a week in maintenance bath and then its value was measured. The differences in values will be presented.

ASSESSMENT OF METHODS FOR DETERMINING THE IMPURITY CONCENTRATION IN MERCURY CELLS

M. Kalemci[1], A.T. Ince[2], G. Bonnier[1]
[1] *TUBITAK UME, Kocaeli, Turkey*
[2] *Yeditepe University, Istanbul, Turkey*
E-mail (corresponding author): murat.kalemci@ume.tubitak.gov.tr

Uncertainty arising from the chemical impurities is the most contributing factor in the uncertainty budget of primary level temperature measurements. Impurities in any substance generally decrease the freezing (or triple) point temperature of substance and their influence is governed primarily by their behaviour at low concentrations in the host material. The depression in temperature due to impurities is theoretically expressed by Raoult's law which at the final analysis expresses the linearity between ΔT ($T_{observed}$ – T_{pure}) and inverse of fraction frozen (1/ F).

Recently, TUBITAK UME carried out a new project on the construction of new reference mercury fixed point cells. Within the scope of this study, three different sets of mercury cells were fabricated. The first set has been filled with the mercury of low purity, the second set with mercury having 99.99995% purity and the final set with 99.9999999% pure mercury. The mercury used for the latter two sets had been certified by the supplier. Borosilicate glass which has an outer diameter of 25 mm and a wall thickness of 2 mm was used for the construction of all cells. The same procedure was applied for cleaning of the borosilicate cell material and each cell has been prepared by pour and pump method.

Three methods were employed to assess the impurity concentration in the cells. First method is called as mole fraction sum of impurity components and the chemical assays form basis for this kind of assesment. The second method of evaluation is based on the 1/F versus ΔT curve and the slope values obtained from these curves is of importance. The final method is to directly compare the new cells with the national (or reference) standard mercury cell. For this final method of evaluation, several approaches to obtain triple point value of mercury were employed and the outcomes were compared.

The results obtained from three methods of evaluation showed consistency in terms of qualitative analysis.

THE INVESTIGATION OF THE IMMERSION PROFILES IN THE GALLIUM FIXED POINT AND IN TRIPLE POINT OF WATER CELLS

R. Strnad[1], M. Šindelář[1], O. Prokeš[2]
[1] *CMI, Prague, Czech Republic*
[2] *RWE Transgas Net, s.r.o., Czech Republic*

The standard validation procedure of the quality of the measurement in fixed points includes among others the checking of the immersion profile of the standard platinum resistance thermometer (SPRT). This profile should follow of the theoretical value assigned by the ITS-90. The real profile depends on the number of influences, e.g. furnace gradient, ambient temperature, insulation; type of the thermometer used, quality of the fixed point, etc.

In this article will be presented the experimental results of the investigation of the measurements of the immersion profile of the several type of triple point of water cells (TPW) and of Gallium cells. The measurements were done with several thermometers with showing of the dependency of the results on type of initialization of the cell, time of the plateau and external gradients. The measurements in the bottom of the cells were made for checking of the influence of the buoyancy effect in the TPW cells of different design.

POSTER SESSION I

Industrial Applications

Monday
10:45 to 11:30
and
14:15 to 15:00
Foyer
room: Emerald 2

THERMAL MANAGEMENT OF LIGHT SOURCES

J. Hartmann[1], S. Völker[2]
[1] *PTB, Berlin, Germany*
[2] *TU Berlin, Berlin, Germany*
E-mail (corresponding author): Juergen.Hartmann@ptb.de

The primary task of light sources is illumination, i.e. the emission of visible radiation – light. However, depending on the generation principle besides light, also heat will be dissipated to the surrounding.

Classical thermal light sources generate light by the electrical heating of a tungsten wire to temperatures of about 3000 K. Even at this temperature the majority of the emitted radiation is within the long wavelength range of the spectrum, i.e. not in the range of visible radiation.

Generation of light with discharge lamps is totally different and non-thermal, however, even in this case the electrodes are heated up to temperatures well above 2000 K. Thus also discharge lamps suffer from thermal problems.

In case of solid-state light sources, also non-thermal light sources, the electrical current causes a heating of the device, which is or should usually be below 150 °C for proper operation. Such relatively low temperatures of solid-state light sources prevent an efficient cooling by thermal radiation, requiring a convective or conductive cooling.

For all mentioned light sources the *Thermal Management*, i.e. the adjusting and maintaining of an optimum operation temperature is vital for the efficiency and life time of the light sources. The paper deals with the methods of generation and measurement of the thermal load and discusses ways to optimize the efficiency and life time of such light sources. Also some practical examples are given to emphasize the relevance of such Thermal Management for industry, pointing out the potential for future more energy-efficient light source concepts.

SILICON WAFER SURFACE TEMPERATURE MONITORING SYSTEM FOR PLASMA ETCHING PROCESS

Y. Yamada[1], J. Ishii[1], A. Nakaoka[2], Y. Mizojiri[2]
[1] *National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology(NMIJ, AIST), Tsukuba, Ibaraki, Japan*
[2] *Mizojiri Optical Co. Ltd., Tokyo, Japan*
E-mail: y.yamada@aist.go.jp

An *in situ* silicon wafer surface temperature monitoring system for plasma etching is reported for the first time. In plasma etching, heat is introduced from the plasma which makes measurement of the top surface necessary. However, the semiconductor wafer being etched is transparent in the infrared, and radiation thermometry is impractical. Therefore, temperature monitoring by thermoreflectance method was applied.

Thermoreflectance detects variation in temperature through change in optical reflectance. The technique has so far not been considered for *in situ* measurement for process control, due to the low sensitivity of the measurement principle. Accurate detection of small change in reflectance in the order of 10^{-5} is required, which is an extremely difficult task in the presence of background noise from the plasma, and vibration of the process chamber and the wafer being processed. Physical limitation in the installation position in the process chamber is another major obstacle: measurement needs to be preformed through a narrow gap between the wafer and the plasma electrode.

To overcome the above difficulties, thermoreflectance utilizing two orthogonal polarizations have been introduced. The system introduces a laser beam through the gap between the wafer and the electrode, and the reflected beam from the wafer surface is detected. The system detects the difference of the relative change in the reflectance for the two polarizations. Noise such as fluctuation in the incident beam intensity or change of loss in the optical path would affect both polarizations equally and would not affect the measurement. The large angle of incidence of the beam allows measurement to be performed from outside the viewing ports of existing plasma etching process chambers.

In this paper measurement result is described, which was made off-line to evaluate the performance of the developed thermoreflectance system. Silicon wafer samples are statically heated from room temperature to around 140 °C. The obtained signal corresponding to change in reflectance is compared with reference temperature and good correlation was confirmed. Results are presented for bare wafers as well as for wafers with metal film depositions.

A "DISAPPEARING OBJECT" RADIATION THERMOMETER FOR LOW-EMISSIVITY PROCESS CONTROL

P. Saunders, D. R. White
MSL, Lower Hutt, New Zealand
E-mail (*corresponding author*): *p.saunders@irl.cri.nz*

One of the long-standing problems in metals-processing industries is the measurement of the temperature of low-emissivity materials. Such measurements are typically accompanied by problems with reflections, highly variable emissivity, and moving targets. Examples include the monitoring of steel strip in galvanising processes and aluminium extrusion, both of which exhibit large relative changes in the target emissivity and have relatively tight temperature requirements.

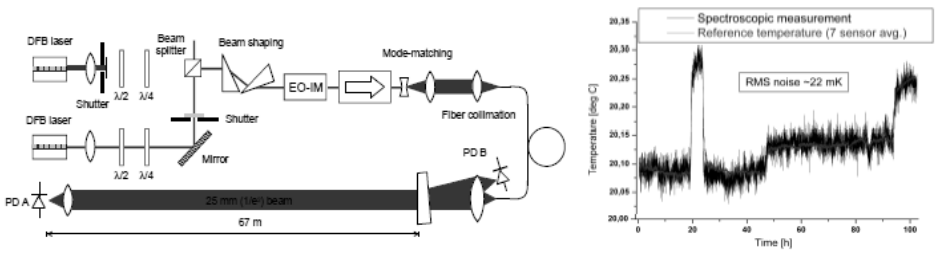
This paper presents a novel radiation thermometer that places the target inside a blackbody cavity at the desired process temperature. Uncertainties in the measurements of target temperature due to the background radiance temperature and the target emissivity, therefore, fall in proportion to the difference between the target and process temperatures. When the target and the process temperatures are the same, the uncertainty is at a minimum of perhaps less than 1 °C. Although the thermometer does not give an accurate measure of the temperature difference, it enables control of the process to a level near the minimum uncertainty. The paper presents the design principles based on the uncertainty analysis, and suggests practical realisations for steel strip mills and aluminium extrusion applications. The technique can also be used with imaging systems, which would show the target object disappearing against the blackbody background when the object temperature is at the required process temperature.

SPECTROSCOPIC MEASUREMENT OF AIR TEMPERATURE

T. Hieta[1,2], M. Merimaa[1]
[1] *Centre for Metrology and Accreditation (MIKES), Espoo, Finland*
[2] *Helsinki University of Technology (TKK), Espoo, Finland*
E-mail (corresponding author): tuomas.hieta@mikes.fi

Optical distance measurements have to be corrected for the refractive index of air. The refractive index is conventionally calculated from parameters of the ambient air using either Edlén or Ciddor equations. However, these equations require accurate knowledge of the air temperature. For example, to reach 10^{-7} uncertainty in distance, the air temperature has to be known at 100 mK level. This does not necessarily cause problems in stable laboratory environment. However, if measurements are done outdoors or in industrial environment, variations in temperature can be very rapid and local temperature gradients can cause significant error if not taken into account. Moreover, if the required distance is long, the temperature over the whole length can be impractical to measure by using conventional temperature measurement techniques. The developed method based on molecular spectroscopy allows good spatial and temporal overlap of the temperature measurement with the actual distance measurement.

Temperature measurement using spectroscopy is based on line intensity ratio measurement of two absorption lines, previously applied for measurements of high temperatures in flames [1]. Oxygen absorption band at 762 nm is convenient since the line strengths are practical for long distance measurements and suitable DFB-lasers are commercially available. Our measurement setup, based on of commercial laboratory equipment, is schematically shown in the figure below. Normalized lock-in detection is done to reduce interferences and noise. Example of 100 hour measurement including intentional temperature changes over a 67 m long path is given below. Scale of the spectroscopic measurement is obtained from comparison to conventional temperature sensors and an empirical pressure correction of–45 mK/mbar has been used.



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ARTIFICIAL FRUITS - POST HARVEST ONLINE MONITORING OF AGRICULTURAL FOOD BY MEASUREMENT OF HUMIDITY AND TEMPERATURE

T. Hübert, C. Lang
BAM - Federal Institute for Materials Research and Testing, Berlin, Germany
E-mail (corresponding author): thomas.huebert@bam.de

An on-line monitoring of environmental conditions and food transpiration losses is required in order to avoid quality downslides of fruits and vegetables during transportation and storage.

Therefore temperature and humidity was measured by appropriate small adapted sensors and the electronic data were transmitted by a transportable minaturised radio communication unit. We demonstrate that regular and also extreme values in low or high humidity (condensation) and temperature can be continuously measured and transmitted on demand up to 16 month.

The transpiration of fruits as an indicator of firmness results in gas releases and mass losses what is extensive to determine. We suggest controlling transpiration losses also by humidity measurements. For that purpose, a humidity sensor is surrounded by wet porous fibrous material what is in contact with the outer atmosphere. The sensor measures the equilibrium humidity inside of the wet material envelope. The transpiration of this artificial fruit reduces the water content of the porous material and thus also of the measured internal humidity. The decrease of humidity indication was calibrated against the mass loss of fruits under different influencing external parameters like temperature, humidity and air flow velocity. The system provides data from a sensor which is embedded in a fruit crate via radio transmission for three weeks.

INHOMOGENEITY ANALYSIS OF THERMOCOUPLES USED IN PRODUCTION PROCESSES IN THE STEEL INDUSTRY

M. Holmsten, S. Ljungblad, L. E. Josefson
SP, Borås, Sweden
E-mail (corresponding author): magnus.holmsten@sp.se

An investigation of the influence of inhomogeneity in different types of thermocouples is presented. In cooperation with a company within the steel industry, a number of thermocouples of types K, N and S have been selected. The thermocouples included in this investigation have been used in the industries' standard measuring applications and processes. For the different thermocouples, there is a documented user history available, including temperatures and time in use.

The investigation is still in progress and not quite concluded, but preliminary results indicate large influence after a time in use of two months, especially for the type K thermocouples. For some of these thermocouples, which have been in use at temperatures just above 1000 °C, temperature shifts of over 40 °C have been found in parts of the thermocouples. In some cases, the changes found are not linearly connected to the emf voltage of the thermocouple. That the thermocouples are this heavily influenced by inhomogeneities, leads to practical problems when the internal calibration resources within the industries are limited, lacking tools to identify the magnitude of measurement errors.

To investigate the inhomogeneities of the thermocouples, a method with a short moveable heating zone is used, as presented at Tempmeko in 2007. In the method, a hot-air fan is producing a short, well-defined heating zone, which is moved along the thermocouple. The measuring and reference junctions are placed in ice baths. The emf voltage from the thermocouple is measured continuously during the time the heating zone is moved. From the measurements, the corresponding temperature variation can be calculated. Since the method was presented in 2007, further developments of the measuring method and calculation algorithms have been made.

OPTIMAL PARAMETRIC SINTHESIS OF THIN-FILM THERMORESISTIVE SENSORS

A. G. Kozlov
Omsk State University, Omsk, Rassia
E-mail: kozlov@phys.omsu.omskreg.ru

Thin-film thermoresistive sensors are widely used as thermosensitive element in various types of thermal microsensors. The important parameter of the sensors is a measurement error which should be as small as possible. The basic component of the error is that due to a self-heating by a measurement current. It depends on many parameters (measurement current, constructive parameters, heat transfer conditions), therefore, in designing thin-film thermo-resistive sensors one should be used such values of the parameters which ensure the minimum of this error. The given problem is an optimization one. The aim of this paper is to present the method of the optimal parametric synthesis of thin-film thermoresistive sensors on support thermally isolated structures. Here the sensors are considered as the system with distributed parameters and the temperature distribution in it is determined with the following algorithm.

1. The 2D structure of a thermal microsensor is divided into rectangular regions depending on the composition of layers and heat-generating conditions. Each region is replaced by an equivalent region with the homogeneous parameters.
2. For each region, the heat exchange conditions are determined.
3. For each region, the steady-state heat deferential equation is solved by the eigenfunction method. The heat flux densities between the regions are presented as the sums of orthogonal functions with unknown weighting coefficients.
4. The unknown weighting coefficients are determined using the adjoint boundary conditions between regions.

The algorithm of the optimal parametric synthesis includes the following steps.

1. In the structure of thermal microsensor, the region occupied by the thin-film thermoresistive sensor is marked out and the dependence of the region area on the width of the thermoresistive layer is determined.
2. The analytical dependence of the average weighted overheating temperature in the separated region on the width of the thermoresistive layer is determined. Toward this end, the above-mentioned algorithm for determining the temperature distribution is used.
3. Minimization of the goal function in the form $Z = (\Delta T_j^{av} - \Delta T)^2$ (where ΔT_j^{av} is the average weighted overheating temperature in the region; ΔT is the absolute error of measuring a temperature by the thermoresistive sensor) is performed. The independent variable is the width of the thermoresistive layer. The obtained value of the width will be the value which ensures the adjusted absolute error of measuring a temperature.

The method of the optimal parametric synthesis of the thin-film thermoresistive sensor can be used in designing these sensors.

MULTI-CHANNEL OPTICAL FIBRE THERMOMETER FOR PEM FUEL CELL APPLICATIONS

L. Rosso, V. Fernicola
INRIM Istituto Nazionale di Ricerca Metrologica, Torino, Italy
E-mail (corresponding author): l.rosso@inrim.it

Miniature, durable and fast responding temperature sensors are needed for proton exchange membrane (PEM) fuel cells. When embedded in a single cell or in a cell stack, they can provide useful information both at the design stage for optimising their efficiency and during their operation for monitoring the working conditions thus preventing failures.

Optical fibre sensors offer the ability to perform in-situ multi-point temperature measurements because they are small, rugged and inexpensive. In addition, they can provide safe temperature measurements in an electrically-hostile environment. These features make such sensors potentially suitable for fuel cell applications.

A four-channel optical fibre temperature system, based on fluorescence thermometry has been developed. The system consists of an photonic unit for the excitation/detection of the fluorescence signals and a set of custom optical fibre probes based on a temperature-sensitive fluorescent material attached to the distal end of an optical fibre.

The temperature system was calibrated by comparison against a platinum resistance thermometer traceable to ITS-90 in the range from room temperature to about 150 °C. The system affords a temperature repeatability to within 0.1 °C with a response time lower than 0.1 s to a step temperature change.

The system design, the probe construction and its laboratory testing are presented in the work together with an assessment of the overall system performance. The application of such system in a fuel cell test rig for point temperature measurements in a cell stack is presented. The experimental results show the potential use of the system in real-time temperature mapping in operating fuel cells.

ALL FIBEER FABRY-PEROT TEMPERATURE SENSOR WITH LOW COST INTEROGATION SYSTEM

E. Cibula, M. Njegovec, D. Đonlagić
University in Maribor, Faculty of Electrical Engineering and Computer Science, Laboratory of Electro Optics and Sensor Systems, Maribor, Slovenia
E-mail (corresponding author): ddonlagic@uni-mb.si

This paper presents miniature optical temperature sensor based on all-fiber Fabry-Perot interferometer that can be interrogated by cost-effective optical signal processing system. The sensor is suitable for use in harsh and other critical environments, like for example volatile or explosive environments, in-vivo medical devices, corrosive environments, environments reach in electromagnetic interferences, etc.

The presented sensor consists of two low reflectance mirrors build within optical fiber that are produced by combination of wet etching and fiber fusion splicing processes. The mirrors thus form low fines all-fiber Fabry-Perot interferometer. The spectral characteristic of the sensor depends on the refractive index of the fiber core between both mirrors, which is temperature dependent. The selection of the distance between in-fiber mirrors can be used to set the desired sensor measurement span, which can exceed –270 to 800 °C range. The adjustment of the distance between the mirrors also affects the sensor sensitivity, resolution and achievable accuracy. The temperature interrogation is performed by wavelength sweeping of the optical source that is used to record sensors spectral characteristic, followed by digital post-signal processing to extract sensor temperature. For this purpose we successfully applied standard telecommunication DFB laser diode. Only few additional standard telecommunication components are needed to build entire measurements system, which makes the proposed system cost effective and attractive for variety of practical applications. As an example, we evaluated 1 mm long temperature sensor that was optimized for temperature range from 0 to 100 °C. The optical temperature measurement system showed temperature resolution better than 0.2 °C and repeatability better than 0.4 °C. Small dimensions (1 mm x 0.125 mm) also guarantee fast response time that is in the range of few hundreds of milliseconds for particular system.

POSTER SESSION I

Hygrometers and Moisture Sensors

Monday
10:45 to 11:30
and
14:15 to 15:00
Foyer

THE DEVICES M002 WHICH REPRESENT SIGNALING DEVICES OF THE COMPRESSED AIR HUMIDITY AT PRESSURE UP TO 39,2 MPA (400 KGS/SM^2)

V.A. Urakov, B. V. Kosenkov, Y. M. Ivanov
ZAO "Mera", Saint-Petersburg, Russia
E-mail:zaomera@mail.ru

The device M002 is intended for installation in systems of high pressure air (HPA) both a part of the automated compressor stations, and on the sites of pipelines. It provides the automatic continuous and periodic humidity content control in values of moisture dew point on free air with signal delivery on excess of admissible values of the dew point (d.p.) to system of automatic control of the compressor station (CSAC) or to the control panel of HPA system.

The device M002 is used for the control of compressed air humidity with following parameters:

- the range of absolute humidity of controllable compressed air: from –75 °C to –30 °C of dew point on free air,
- temperature: from +15°C to + 35 °C
- pressure: from 14,9 MPa to 39,2 MPa (from 150 to 400 kgf/cm^2), short-duration admissible, by the periods up to 30 min, but totally no more than 300 hours during the full assigned resource, increase up to 40,2 MPa (410 kgf/cm^2),
- oil content on free gas: no more than 2*10^{-7} kg/m^3 (2*10^{-4} mg/l),
- size of mechanical particles: no more than 5 μm.

The board M002-01-1 is intended for transformation of sensitive elements resistance of humidity and temperature detectors to a corresponding analogue signal on voltage of the direct current.
The microprocessor module is intended for transformation of analogue signals to digital ones, carrying out the automatic control of operating capacity of the device, change compensation of influence of controllable environment temperature on the detector of humidity and signal development on excess of the admissible limit at humidity measurements.
The input-output terminal is intended for input of the installed detector number in the instrument control program, instrument self-checking control and indication of self-checking and measurement results on LCD display.
The internal circuitry of the instrument is energized from the power supply MAA50-3C051515СУН.
The device M002 includes the following instruments (elements):

- M002-01 – the block of transformation and data display,
- M002-02 – the assembly of the detector M002-03 with the setting block of the detector in HPA system.

The detector M002-03 is a typical element of replacement, and it represents a set of the sensitive elements structurally closed in the rectangular metal

housing, realizing the function of transformation of the humidity characteristic into corresponding electric quantity with the account of automatic compensation of dependence of humidity characteristic transformation of factor from the controllable ambient temperature.
The metal housing of the detector has apertures for air flow and contacts of electric wires for connection of sensitive elements in the measuring circuitry of the instrument.
The sensitive element of the detector realizing the function of humidity characteristic transformation, represents 2 (measuring and compensatory) sorbed resistor arrays on the plates sized 15,0x17,0x0,5 mm on which thin-film interdigital structure (IDS) of the electrodes, consisting of a carbon film, is put under special technology using a photo mask with a step of 0,1 mm and with a nickel dusting on a chrome intermediate layer. Width of electrodes (IDS) ~ 0,35 mm. Terminal pads to IDS are made of aluminium film 0,001 mm in thickness. From above the hydro-sensitive multicomponent sorbent is put on a plate with electrodes. Compensatory array is intended for temperature compensation of dependence of measuring array transformation from the temperature of the controllable environment.

Operating principle of the device M002 is based on direct measurement of sensitive elements resistance in the humidity and temperature detectors of HPA against alternative and direct current, accordingly, and their further hardware-software transformation to values of absolute air humidity, expressed in dew point units.
Electric signals on voltage (V_h) and (V_t) through a connecting cable from the instrument arrive on the electric circuit of the measuring device M002-01, which is intended for hardware-software transformation of the data received from the detector M002-03, to values of absolute air humidity of the dew point expressed in temperature units and their digital indication on LCD display, and also signal deliveries on excess of admissible limits of controllable humidity in equipment of the compressor station automatic control.
The instrument operates in the automatic mode without operator's participation.
The signal on a normal condition of compressed air humidity automatically arrives to the equipment alarm module of the compressor station automatic control.

PERFORMANCE OF A NOVEL MICROSENSOR-BASED DEW POINT TRANSMITTER

T. J. Lehtola, J. A. Leppänen, T. J. Salo
Vaisala Oyj, Microsensor Technologies, P.O. Box 26, 00421 Helsinki, Finland
E-mail: terhi.lehtola@vaisala.com

A novel microsensor-based dew point transmitter has been developed for measuring frost points in the range of –80 … –10 °C. The transmitter utilizes a novel capacitive thin-film polymer sensor, novel mechanics and a new measurement algorithm. The transmitter has been on market since 2008. The most typical areas of use are air and plastic dryers, dry chambers, pure gases, high voltage circuit breakers and other industrial applications where it is necessary to control very low humidity. In plastics drying, a typical measurement environment is a challenging combination of low humidity and hot air. On the other hand, fast response time is appreciated in air drying applications.

The performance of the transmitter has been measured during the product development phase. Series of tests were done in pre-set laboratory conditions. Frost point was measured at controlled humidity levels and compared to a chilled mirror reference. Overall, the results lie within ±2 °C of the readings of the ten-fold more expensive reference, and show highly competitive performance in comparison to main competitors' devices. Due to the proprietary polymer structure and measurement algorithm, the response time for frost point change from –10 °C down to – 80°C is in the range of minutes. Moreover, good stability in dry conditions has been achieved.

DETERMINATION OF METROLOGICAL CHARACTERISTICS OF INTEGRATED DEW POINT HYGROMETER

J. Weremczuk, R. Iwaszko, D.Paczesny, R.S. Jachowicz.
Institute of Electronic Systems, Warsaw University of Technology, Warsaw, Poland
E-mail (corresponding author): jwer@ise.pw.edu.pl

Dew point hygrometers are known to be the most accurate technical systems for measurements of gas humidity. The hygrometer with novel dew point detector, based on small semiconductor microsystem, was build. The system was especially designed for medical applications in laryngology, where humidity of air during breathing must be measured with time constant less than 1.5 s and accuracy better than 0.5 K of dew point temperature. To decrease the time constant of the hygrometer operation the samll heater is embedded beneth the dew point detector surface. The whole microsystem structue is chilled down by Peltier module of relatively high time constant and after crossing the dew point temperature the heater injects a small amount of heat to rapidly stop the process of temperature decreasing which protects the dew detector from overcondensation of water.

To achieve high metrological characteristics of the hygrometer (accuracy of dew point temperature measurements and fast operation as well) the special care should be placed on dew point detector design. Our integrated detector structure dimmensions are 4 mm x 4 mm x 0.46 mm. The detector structure (microsystem) contains impedance condensation detector (surface interdigitated capacitor), thermoresistor and heater which are located beneeth the condensation area (500 nm in depth). The paper presents evaluation results and discussion of detector sensitivity measurements. The minimum amount of water molecules required to obtain dew detection was measured. Also temperature uniformity of condensation/detection area and thermoresistor stability of two different microsystem designs are evaluated.

EXTREMELY FAST 1 PPM WATER VAPOR MEASUREMENT BY A 1 MILLIMETER DAIMETER SPHERICAL SAW DEVICE

N. Takeda[1], M. Motozawa[2]
[1] *Ball Semiconductor Incorpolated, Frisco, TX, USA*
[2] *Ball Semiconductor Co. Ltd., Nagareyama, Chiba, Japan*
E-mail: ntakeda@ballsemi.com

A new propagation phenomenon of surface acoustic wave (SAW) on a spherical substrate was discovered by Yamanaka *et. al.* in 2000 [1]. Once SAW is generated on the spherical surface, a collimated SAW propagates around the equator hundreds of times. This unique propagation enables highly sensitive gas detection by measuring amplitude change and/or phase shift of SAW traveled after equivalently long distance [2].

In this paper, we report on the development and testing of a 1 mm diameter spherical SAW device designed for measurement of very low water vapor concentration. The so-called "Ball SAW sensor" was manufactured using a maskless exposure system and other semiconductor / MEMS process technologies for a spherical substrate. The spherical substrate is single crystal quartz and the SAW is excited and detected by an inter-digital transducer (IDT), based on Au/Cr with 5 um line width and spacing.

To test response speed of the Ball SAW sensor, water vapor was introduced into dry N2 through a thin metal tube to yield a concentration of 1 ppm by the diffusion theory. Then dry N_2 and 1 ppm H_2O / N_2 are alternatively introduced to the sensor at 4 minutes intervals. The rise time from 10% to 90% of 1 ppm response was found to be about 15 sec. Accordingly, the sensor has a potential to be a highly sensitive and extremely fast response hygrometer.

References

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[2] S. Ishikawa, N. Nakaso, N. Takeda, D.Y., Sim, T. Mihara, Y. Tsukahara and K. Yamanaka, Appl. Phys. Lett. vol. 83, pp. 4649-4651, (2003).

COMPARATIVE STUDY OF HUMIDITY ANALYSERS IN NITROGEN BELOW 1 µMOL/MOL

P. Hernandez, R. Benyon
Instituto Nacional de Técnica Aeroespacial (INTA), Torrejón de Ardoz, Spain
E-mail (corresponding author): benyonpr@inta.es

The lower limit of humidity traceability available in Spain is that attainable with the frost-point temperature realization of the low-range national humidity standard generator, –75 °C, equivalent to approximately 1 µmol/mol. The uncertainty of this standard is limited by the generation method that requires the use of the equations that relate the water saturation vapour pressure over ice and the corresponding enhancement factors to take into account the non-ideal behaviour of water vapour, only experimentally determined down to –50 °C.

One of the activities of the INTA national humidity standards Project is the improvement of the declared calibration and measurement capabilities (CMC), both in range and uncertainty. The two-pressure generator is to be replaced by a low-range single pressure standard humidity generator, designed in collaboration with, and constructed by NPL. One of the aims of the project is the determination of the level of consistency and robustness of the estimation of uncertainty of the realization of dew-point temperature, in relation to a standard based on a gravimetric system, comprising a magnetically-coupled suspension balance and an atmospheric pressure ionization mass spectrometer. This is to be used to calibrate permeation tubes that are subsequently employed as water vapour sources in trace humidity measurement and generation systems.

As a first step, a calibration system has been set-up in order to compare different analyzers and establish the design and implementation characteristics of the future standard generation system, as well as obtaining experience in the use of the essential components necessary for the primary realization. A trace humidity generator using permeation tubes has been implemented, and measurements have been performed in the range between 100 nmol/mol and 250 nmol/mol with flow rates between 1 l/min and 5 l/min.

An optical dew-point hygrometer, a quartz crystal microbalance, and a cavity ringdown spectrometer have been evaluated. The preliminary results of the comparison of these humidity analyzers based on three different measurement principles, are presented and discussed. The results include response times and repeatability. The design criteria and the characterization of the system, together with an estimation of measurement uncertainty in each case, are addressed.

IMPROVING THE UNCERTAINTY OF CALIBRATION USING A TRANSPORTABLE HUMIDITY AND TEMPERATURE GENERATOR

R. S. Farley[1], B. Ramsey[2], R. Gee[3]
[1] *Rotronic Instruments (UK) Ltd., Crawley, UK*
[2] *VCT Ltd, Egham, UK*
[3] *Rotronic Instruments (UK) Ltd., Crawley, UK*
E-mail (corresponding author): richardg@rotronic.co.uk

A transportable humidity and temperature generator has been developed as a solution for on-site engineers and industrial calibration laboratories as a cost effective method of transferring traceability and providing confidence to measurements made on a daily basis. The second generation of calibrator builds on the first series with improved specifications, additional features and optimised design.

Developments in mechanical design mean an improved thermal performance in terms of speed of response, control stability and temperature gradients giving rise to improved performance in terms of the humidity control. The humidity generation is maintained by a piezoelectric element from a digital PID control with response optimised across the temperature range of 0 °C to 60 °C.

A bespoke controller with touch screen user interface has been developed to provide the user with simplified setup and configuration tools that includes a programmer function so that multiple set point changes can be user defined. The embedded platform also provides additional features such as an integrated nine port USB hub, external DVI monitor connection, integrated data-acquisition and calibration adjustment software.

This paper will show that with the enhancements to the existing generator the calibration uncertainties achievable with the system have been improved.

POSTER SESSION I

Procedures and Facilities

Monday
10:45 to 11:30
and
14:15 to 15:00
Foyer

PRACTICAL STUDY OF PSYCHROMETER CALIBRATIONS

A. Kentved[1], M. Heinonen[2], D. Hudoklin[3]
[1] *Danish Electronics Light & Acoustics (DELTA), Hoersholm, Denmark*
[2] *Centre for Metrology and Accreditation (MIKES), Espoo, Finland*
[3] *MIRS/UL-FE/LMK, Ljubljana, Slovenia*
E-mail (A. Kentved): abk@delta.dk

Psychrometers remain the most widely used instruments for controlling the humidity in climatic test chambers. The typical uncertainty requirements for calibration of such psychrometers are around 1-2 %r.h., which is close to the best measurement uncertainties claimed by National Metrology Institutes (NMI). Psychrometer calibrations are known to be specifically challenging due to several factors, including: wick fitting, water evaporation and heat dissipation from a possible built-in fan. This, combined with the demand for low calibration uncertainties, makes inter-comparison of psychrometer calibrations particularly interesting.

In EURAMET Project no. 1033, a practical comparison of the psychrometer calibrations performed by the National Metrology Institutes in Denmark, Slovenia and Finland has been carried out. The aim of the comparison was to investigate the equivalence of the psychrometer calibrations performed by different laboratories at the highest level and gather practical experience to be used in future comparisons in this field. An aspirated electropsychrometer was used for the comparison and calibrations were carried out in the range from 15 %r.h. to 93 %r.h. in a temperature range of 15 °C to 70 °C.

This paper presents the results of the comparison, which show good agreement at high relative humidity. At low relative humidity, reported disagreement of the results is judged to be caused by psychrometer wick contamination combined with higher sensitivity coefficients.

Keywords: calibration; psychrometer; relative humidity measurement, wick contamination.

TRANSFER CHARACTERISTIC LINEARIZATION OF RELATIVE HUMIDITY SENSORS

Y.A. Barbar, M.N. Golikov, K.A. Tomsky
TKA Scientific Instruments, St. Petersburg, Russia
E-mail: ybarbar@yahoo.com

For production of portable thermohygrometers, the company TKA utilizes different humidity sensors: analog of type HIH-4000 (Honeywell) and digital of type SHTxx (Sensirion). In the course of production we studied the metrological and operational properties of the utilized sensors.
The linearity rate of the humidity sensor transfer characteristic affects the overall instrumental error of the instrument: than linear transfer characteristic of the less relevant instrumental error.
Thus, by having studied the sensors of type SHT71, SHT75 in the reference generator of humid gas, we determined that their transfer characteristic differs from the linear characteristic. The measurement error of relative humidity RH caused by this difference in characteristics has the delineated maximum and is non-linear and alternating. We have determined that such error can be approximated analytically as:

$$\delta RH_{TRUE} = a + b \cdot \left| RH_{TRUE} - RH_0 \right|^{\alpha} \qquad (1)$$

where a, b, RH_0 – constants; α can take values of 1 or 2; RH_{TRUE} – humidity value measured by sensor, taking into account its temperature.
Record correction $\Delta RH_1 = -\delta RH_{TRUE}$ improves linearity of the transfer characteristic and permits to reduce significantly the measurement error of relative humidity:

$$RH_1 = RH_{TRUE} + \Delta RH_1 \qquad (2)$$

where RH_1 – stable value of measured humidity.
There are two ways to determine the constants a, b, RH_0, α for humidity sensors:
1. Individual calibration of the humidity sensor in the reference humidity generator.
2. Using the averaged values. For this purpose, we investigated 100 pieces of SHT71 sensor type.
Results were then processed and averaged.
For case 1: the application of the proposed method of error reduction for sensors SHT71, SHT75 provides the measurement of relative humidity in the range of 2 … 99 %RH with error not more than ± 1%RH relatively to the used reference gage.
For case 2: the remaining instrumental error will be somewhat more than 1 %RH. Its value depends on the control sample studied humidity sensors and frequency of control measurements.

DETERMINATION OF HOMOGENEITY AND STABILITY OF HUMIDITY TEST CHAMBERS

A. T. İnce[1], G. Aslanbay[1], A. Davut[2]
[1] *Yeditepe University, Istanbul, Turkey*
[2] *Turkish Standards Institution, Kocaeli, Turkey*
E-mail: aince@yeditepe.edu.tr

Calibration of measuring instruments must be carried out regularly. Calibration is the set of operations that establishes the relationship between values of quantities indicated by a measuring instrument or measuring system represented by a reference material and the corresponding values realized by standards.

In this study, humidity and temperature is observed and the measuring system is prepared for the calibration of humidity and temperature measurements. In a test chamber, relative humidity measurements are performed between %20 and %80 at 15 °C and 40 °C, respectively. The homogeneity and stability of the test chamber is determined by the experimental setups and uncertainty budgets of humidity and temperature are prepared for calibrations.

The two-pressure humidity generator covered relative humidity range of %10RH to %95RH and temperature range of 0 °C to 70 °C. The results show that the value of uncertainty components of homogeneity are 1,85 %RH for humidity and 0,59 °C for temperature in the humidity generator. The results show that the value of uncertainty components for stability are 0,192 %RH for humidity between %20RH and %80RH, and 0,53 °C for temperature between 15 °C and 40 °C.

In a humidity calibration laboratory, some basic reference instruments are used for calibration. The humidity generator is the most commonly used instrument for humidity calibration laboratories. The stability and homogenity of the humidity generator have to be known in order to utilise the humidity generator as a reference instrument for calibrations.

RAPID RELATIVE HUMIDITY CALIBRATION

J.W. Lovell-Smith, R.S. Mason.
The Measurement Standards Laboratory of New Zealand Lower Hutt, New Zealand
E-mail (corresponding author): j.lovell-smith@irl.cri.nz

The calibration of hygrometers having impedance-based humidity sensors can require lengthy calibration times in order to adequately characterise the hygrometer response, which can exhibit marked hysteresis, temperature dependence, long response times and partly linearised complex behaviour. During a thorough calibration at the Measurement Standards Laboratory of New Zealand (MSL), the relative humidity is changed many times with set-points stepping up and down the humidity scale. The set-points (or a subset) may be repeated at different temperatures. Usually, only the near steady-state response from the reference and the device under calibration (DUC) are sampled and used in the analysis, although reference and DUC data is logged continuously.

In this paper we describe the development of a new method known as Total Data Calibration (TDC), which requires very rapid and stable, humidity step changes. In the method, all data logged during the calibration is used to characterise, and hence calibrate, the sensor response. This requires an adequate model of the sensor response that includes time constants, hysteresis and steady state response. Since the model can be fitted to all data collected, not just to that collected at the end of a lengthy soak, set-point transitions can be made well before the system has reached equilibrium and thus calibration time can be significantly decreased. Furthermore, typical time-constants and hysteresis behaviour will be derived for particular sensor types and these may be used as starting points to calibrations of that type of sensor, further reducing calibration times and the amount of data needing to be collected.

RELATIVE HUMIDITY SENSOR BEHAVIOUR AND CALIBRATION AT 100 %RH

J.W. Lovell-Smith, R.S. Mason.
The Measurement Standards Laboratory of New Zealand Lower Hutt, New Zealand
E-mail (corresponding author): j.lovell-smith@irl.cri.nz

Humidity sensors for general use are not commonly calibrated right up to 100 % relative humidity (100 %rh) because of the likelihood of condensation on the sensor and the surfaces of the calibration chamber, because condensation on the sensor is often thought to result in a temporary or semi-permanent shift in calibration, because for most applications condensing humidities are not encountered and because, in any case, most dynamic humidity generators are not designed to operate so high. In meteorological and green-house applications, however, condensing and near-condensing conditions are regularly encountered so that methods to characterise behaviour before, during and after the condensing condition, need to be available.

In this paper we consider several ways to reliably produce 100 %rh including wrapping the sensor housing in wet cloth, use of a hybrid two-flow and two-temperature generator, suspending the sensor above a plane water surface and thoroughly wetting a ptfe or mesh filter enclosing the sensor. The results show that many sensors do actually respond quickly and reliably to condensing conditions and that slow recovery in still conditions is likely to be due to slow evaporation of condensation on the sensor and on the adjacent surfaces rather than to a slow response of the sensor itself. Finally we consider how 100 %rh set-points can be incorporated into a relative humidity calibration sequence and the consequences for the calibration corrections and uncertainty.

POSTER SESSION I

Determination of the Boltzmann constant

Monday
10:45 to 11:30
and
14:15 to 15:00
Foyer

MEASURING SHELL RESONANCES OF SPHERICAL ACOUSTIC RESONATORS

D. Truong[1], F. Sparasci[1], E. Foltête[2], L. Pitre[1]
[1] *LNE-INM, Saint-Denis, France*
[2] *FEMTO-ST, Besançon, France*
E-mail (corresponding author): daniel.truong@cnam.fr

We report here a work in progress to measure the impact of shell motion on acoustic resonances. LNE-CNAM is using an acoustic method to determine the Boltzmann constant k_B. This method consists mainly in measuring acoustic resonances within helium gas contained in spherical resonators. As shell walls of real resonators are not perfectly rigid, some perturbations occur in the frequency range of acoustic resonances. They are detected by the excess of half-width of the acoustic modes and the perturbed modes are rejected for the determination of k_B. A coupling between radial acoustic modes and radial shell motion, the "breathing" mode, is predicted by the theory.

But excess of half-width is not solely observed in the vicinity of the "breathing" frequency -and with a target of a 1 ppm uncertainty associated to k_B- an excess of half width of 1 ppm for non-rejected modes has now to be taken into account.

We have investigated the natural modes of an air-filled, copper-walled, half-litre quasi-spherical resonator. Using a hammer blow method together with modal analysis we have assessed the shell modes in the frequency range 1 Hz to 20 kHz. We observe multiple resonances for each mode, including for the "breathing" mode whose series ranges in frequency from 18.2 kHz to 20 kHz.

THE LENGTH DETERMINATION OF A FIXED PATH CYLINDRICAL RESONATOR WITH THE DUAL WAVELENGTH LASER INTERFERENCE METHOD

J. P. Sun[1], J. T. Zhang[1], X. Y. Zhang[2], H. Lin[1]
[1] *National Institute of Metrology, Beijing 100013, China*
[2] *Hebei University, Hebei 071002, China*
Email (corresponding author)：sunjp@nim.ac.cn

The length is one of the key parameters for cylindrical acoustic resonator used for the determination of the sound speed. Compared with variant path cylindrical resonators, fixed path cylinders inherit the advantages of the concise structure, the selection of the optimal size, the simplicity of installation, etc. One of the disadvantages lies in the absolute length determination. A research project has been conducted in the National Institute of Metrology, China, for the re-determination of the Boltzmann Constant with dual fixed path cylindrical acoustic resonators. This paper describes the procedure for the length determination. This method consists of two parts, the primitive measurement and the fine determination. In the primitive measurement, the length of cylinder is gauged to an uncertainty of ±3 μm. The excess fraction method was applied to determinate the accurate length of the resonator by the dual wavelength laser interferometer. The interferometer with the resolution of 1nm was composed of a 633 nm He-Ne laser and a 657 nm semiconductor laser. The difference of the wavelength was selected based on the balance obtaining meanwhile a sufficient resolution and enough uncertainty allowance for the primitive measurement. The selected wavelength difference allows the primitive measurement having an allowance of ±8 μm, which permits a minor length variation caused by the stress of cavity. Because conventional semiconductor lasers are not good for their frequency stability, a Michelson wavelength meter has been set up for the calibration of the wavelength of the semiconductor laser. The practical fine measurement is implemented in a controlled room temperature. The resonator operates at the temperature around the triple point of water (TPW). The length variation of the resonator has to be measured from the room temperature to the TPW. As a result, the laser interferometer can operate also as a precise dilatometer. The lengths of the dual cylindrical cavities are determined with this dual wavelength laser interferometer. The result and the measurement uncertainty are given in the paper.

Keywords: cylindrical resonator, excess fraction method, dual wavelength laser interferometer, wavelength meter

PERTURBATION MEASUREMENT OF WAVEGUIDE FOR THE ACOUSTIC THERMOMETRY

H. Lin, J. T. Zhang, X. J. Feng
National Institute of Metrology, Beijing 100013, China
E-mail (corresponding author): linhong@nim.ac.cn

Acoustic thermometers embed small acoustic transducers in the wall bounding a gas-filled cavity resonator for measurements of resonance frequency. Acoustic thermometry is recognized as the most accurate primary procedure for determination of the general gas constant and the thermodynamic temperatures below 500 K. This method is also applied widely in the field of sound speed measurements of fluids for very high accuracy. However, the application of this method is limited by high temperature because of some technical difficulties. At moderately high temperature, transducers outgas impurities to contaminate pure gases bounded in resonator cavity and insulators loss their electric insulations so to degrade the signal-to-noise ratio. One essential solution to those technical problems is by coupling sound between a resonator and the remote transducers at moderate temperature by a waveguide duct. For this technical treatment, resonance frequency perturbations generated by the waveguide duct must be addressed in order to maintain an equal accuracy. Accordingly, perturbations caused by circular ducts were investigated analytically and experimentally for longitudinal resonance modes in this paper. The resonance frequency shifts and the half-widths increases were obtained as functions of the duct's radius, length, and locations of the transducers along the duct length. For one case of the waveguides of 1.4 mm in diameter and 2 m in lenght, the resonance frequency shift $\Delta f/f$ and half-width increase $\Delta g/f$ were estimated to be -0.23×10^{-6} and -40×10^{-6} for the third longitudinal mode at different pressure and temperature, respectively. The perturbations caused by the duct were also measured at (240~380) K and (0.05~0.5) MPa for different longitude modes and radius modes for the fixed length cylinder. The data matched very well with the theoretical estimations.

ASYMMETRY AND BACKGROUND CONTRIBUTIONS IN FREQUENCY RESPONSE OF ACOUSTIC MODES WITH LOW QUALITY FACTORS

X. J. Feng[1], K. A. Gillis[2], J. T. Zhang[3], H. Lin[3], Y. Y. Duan[1]
[1] *Tsinghua University,Beijing, China*
[2] *National Institute of Standards and Technology, Gaithersburg, USA*
[3] *National Institute of Metrology, Beijing 100013, China*
E-mail (corresponding author): yyduan@mail.tsinghua.edu.cn

Fixed path cylindrical method is one of the primary acoustic resonance methods for the determination of the Boltzmann constant. Comparing to the spherical resonators, the quality factors (Q's) of cylindrical resonator are much lower (mostly less than 1000). Low quality factors were caused by thermal and viscosity boundary effects with cylinders which lead to a significant challenge for the precise measurement of acoustic resonant frequencies. The asymmetry of resonance frequency shapes caused by low Q's results in a non-negligible drift of the resonance frequency. This report described the analytical and the experimental investigations on this drift. The results conclude that this drift can be approximated by $-1/8Q^2$. The frequency drift correction by this derived relation leads the difference less than 2×10^{-7} from the frequency curve fitting results. Low Q modes may require non-linear background terms to describe the tails of other modes. The measured in-phase voltage and quadrature voltage by detectors of piezoelectric transducers were fitted to analyze the resonance frequency. The signal-to-noise of different terms of the correlation was calculated and variance ratio tests (F test) were also compared to make sure the signal of the detector reliable. After the correction and expressions optimization, the asymmetry and non-linear background contributions in frequency response caused by low Q's can be controlled less than 1×10^{-6} which is precise enough in the resonance frequency measurements with fixed path cylindrical method.

SHELL PERTURBATIONS OF AN ACOUSTIC THERMOMETER DETERMINED FROM SPEED OF SOUND IN GAS MIXTURES

R. M. Gavioso[1], D. Madonna Ripa[1], G. Benedetto[1], R. Cuccaro[1],
C. Guianvarc'h[1], L. Pitre[2], D. Truong[2], P. A. Giuliano Albo[1]
[1] INRiM, Torino, Italy
[2] LNE-INM/CNAM, France
E-mail (corresponding author): r.gavioso@inrim.it

With the goal of achieving a better understanding of the excess halfwidths of a quasi-spherical resonator used for the determination of the Boltzmann constant at INRIM, we measured the variation of its acoustic and microwave resonance frequencies induced by changing the composition of a binary He-Ar mixture which filled the cavity. The measurements were taken at a constant density of 300 mol/m^3, corresponding to a temperature of 273.16 K, and a pressure of 690 kPa. As a consequence of the progressive dilution with Ar of a sample of initially pure He, the first seven radial acoustic modes of the resonator spanned a decreasing frequency range between 77 kHz for mode (0,8) to 4.4 kHz for mode (0,2). Due to the linear pressure dependence of the coupling of the acoustic modes within the cavity with the elastic modes in the metal shell comprising the resonator, the identification of shell effects with respect to perturbations induced by other geometrical imperfections (like ducts and slits) is made possible by the comparison of the acoustic spectra recorded at high pressure with low pressure data. In addition to the expected breathing mode of the shell, the results evidenced the presence of several other shell resonances at lower frequencies, confirming that the elastic response of the assembled resonator significantly differs from that of a simple spherical shell.

PROGRESS TOWARDS AN ACOUSTIC DETERMINATION OF THE BOLTZMANN CONSTANT AT CEM-UVA

J. J. Segovia[1], D. Vega-Maza[1], M. C. Martín[1], C. García[2], D. del Campo[2]
[1] *Universidad de Valladolid, Paseo del Cauce s/n, 47011 Valladolid*
[2] *Centro Español de Metrología, Alfar 2, 28760 Tres Cantos*
E-mail (corresponding author): DDELCAMPO@cem.mityc.es

The Spanish Metrology Centre (Centro Español de Metrología-CEM) in consortium with the Research Group TERMOCAL of the University of Valladolid (UVa) are involved in a European project which main target is the determination of the Boltzmann constant k_B. It will be re-determined by using the simple, exact connection between the speed of sound in noble gases (extrapolated to zero pressure) and the thermodynamic temperature T , the molar mass of the gas M, and the universal gas constant R. The speed of sound will be determined in a spherical cavity of known volume V by measuring the acoustic resonance frequencies.

CEM-UVa have set up an acoustic gas thermometer (AGT), which consists in a stainless steel spherical resonator, 4 cm nominal radio, in an adiabatic enclosure. The temperature stability and uniformity obtained are about 0.1 mK. This system allows performing measurements of acoustic and microwave resonance frequencies. Microwave measurements characterize the resonator volume. Both hemispheres, joined by a flange in the equatorial band, can be misaligned up to 50 µm, splitting degenerated microwave modes in suitable triplets. Acoustic radial resonances in Helium and Argon compute zero-density limit speed of sound at T_{TPW}. Kinetic theory of gases and hydrodynamics reveals k_B from this speed of sound value.

In this paper, the first results obtained in the determination of the Boltzmann constant using argon, will be presented together with its associated uncertainty calculated using Monte Carlo approach.

POSTER SESSION I

Fixed points - M-C eutectics

Monday
10:45 to 11:30
and
14:15 to 15:00
Foyer

NUMERICAL PREDICTION OF EUTECTIC TEMPERATURE USING PHASE-FIELD MODEL

M. Ode[1], N. Sasajima[2], Y. Yamada[2], H. Onodera[1]
[1] *National Institute for Materials Science (NIMS), Tsukuba, Japan*
[2] *National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology (NMIJ, AIST), Tsukuba, Japan*
E-mail: ODE.Machiko@nims.go.jp

Metal (carbide)-carbon eutectic systems are widely investigated to serve as reference fixed points in thermometry and radiometry. The experimental observations reveal that shape of melting plateaux varies depending on the metal species, solidification condition, annealing time, impurities, and so on. Compared to experimental investigations, however, the number of theoretical studies is limited. For the eutectic solidification, Jackson-Hunt theory is widely accepted to predict the solid/liquid (s/l) interface temperature under constant temperature gradient. There are few studies on the melting behavior even for single-phase systems.

In the present study the multi-phase-field model has been applied to predict the s/l interface temperature (eutectic temperature) during both solidification and melting. The equation for heat conduction is solved simultaneously adopting double grid method to save computing time. First the s/l interface temperature during the eutectic solidification is investigated for the ideal system that has symmetrical eutectic phase diagram. The effects of furnace temperature, the heat transfer constant and the solid/solid (s/s) interphase width are studied. With the increase of the furnace undercooling and the heat transfer constant, the s/l interface temperature decreases. The effect of the s/s interphase width becomes large with the increase of the other two parameters. The effect of the metal species and the interface temperature during melting will be reported in the presentation.

INVESTIGATIONS ON TWO CO-C FIXED-POINT CELLS PREPARED AT INRIM AND LNE-INM

M. Battuello[1], M. Florio[1], M. Sadli[2], F. Bourson[2]
[1] *INRIM, Turin, Italy*
[2] *Institut National de Métrologie (LNE-INM/Cnam), Saint Denis, France*
E-mail (corresponding author): m.battuello@inrim.it

INRIM and LNE-INM agreed to undertake a collaboration aimed to verify, through the use of metal-carbon eutectic fixed-point cells, methods and facilities used for defining the transition temperature of eutectic fixed-points and manufacturing procedures of cells. To the purpose and as a first step of the cooperation, a Co-C cell manufactured at LNE-INM was measured at INRIM and compared with a local cell. The two cells were of different designs: the INRIM cell of 10 cm^3 inner volume and the LNE-INM one of 3.9 cm^3. The external dimensions of the two cells were noticeably different, namely 40 mm length and 24 mm in diameter for the LNE-INM cell 3Co4 and 110 mm in length and 42 mm in diameter for the INRIM cell. Consequently, the investigation of the effect of temperature distributions in the heating furnace was undertaken by implementing the cells inside a single-zone and a three-zone furnaces.

The transition temperature of the cell was determined at the two Institutes making use of different techniques: at INRIM radiation scales at 900 nm, 950 nm and 1.6 µm were realized from In to Cu and then used to define T_{90}(Co-C) by extrapolation. At LNE-INM, a radiance comparator based on a grating monochromator was used for the extrapolation from the Cu fixed point.

This paper presents a comparative description of the cells and the manufacturing methods and the results in terms of equivalence between the two cells and melting temperature determined at INRIM and LNE-INM.

THE DEPENDENCE OF THE MICROSTRUCTURE OF EUTECTIC FE-C ON FREEZING RATE AND IMPURITY CONTENT. COMPARISON WITH 2D THEORY.

P. Bloembergen[1], B. Illerhaus[2], Z. Wei[1], N. Sasajima[3], W. Wang[3], Y. Yamada[3]

[1] *National Institute of Metrology, Beijing 100013, China*

[2] *BAM, Bundesanstalt fuer Materialforschung und -pruefung, 12489 Berlin, Germany*

[3] *National Metrology Institute of Japan, AIST, Tsukuba, Japan*

E-mail (corresponding author): p.bloembergen@xs4all.nl

In this paper we present a study of the variation of the eutectic microstructure -in terms of the average spacing λ between the particles making up the graphite phase- with the variation in the velocity v of the solid-liquid (S/L) interface during freezing, induced by varying the heatextraction rate Q_f. Results are compared with the prediction of the available analytical 2D theory, developed for the case of unidirectional freezing at imposed growth velocity v of the solid phase during solidification and for the system in its pure state: $\lambda_{2D} \propto v^{-1/2}$. Four materials of eutectic Fe-C with differing types and levels of impurities are selected for a better understanding of the effect of impurities upon the dependency $\lambda = \lambda(v^{-1/2})$. Both 2D and 3D structural data are taken into account to check for the effect of dimensionality. The following factors (1) to (4) influencing the relation in question will be discussed:
(1) The effects of special kinds of impurities, denominated modifiers. These can be roughly categorized in (a) those promoting branching of the graphite structure and thus refining the structure and (b) those with the opposite effect i.e. inhibiting branching and by this coarsening the structure. (2) The effect of imposing the heat extraction rate, rather than imposing the interface velocity as in unidirectional solidification, to which the 2D theory applies (3) The effect of the anisotropy of the microstructure and the effect of the 2D counting method on the result obtained for λ_{2D} . (4) Last but not least: The influence of dimensionality, the estimate of which is enabled by the determination of the 3D structure for one of the samples for two different growth rates by X-ray computer tomography in an experimental setting involving BESSY. The 3D structure has been quantified in terms of V_g/A_g the volume-to-surface ratio of the graphite phase, providing an upper limit to λ_{3D}, the average spacing in 3D. Applying the respective corrections (2), (3), (4) reduces the relative differences between the dependencies $\lambda \propto v^{-1/2}$, as observed for the samples in question, and pull them downwards towards and partly beyond the 2D prediction and towards the 3D prediction, which is situated well below its 2D counterpart. The residual dispersion between the dependencies $\lambda \propto v^{-1/2}$ is ascribed to the effect of impurities (1).
From our study it appears that the existing analytical 2D theory for unidirectional freezing at imposed growth velocity is of qualitative value only to the practice followed in realizing hightemperature fixed points i.e. freezing by imposed heat extraction rate. Numerical 3D approaches like the one incorporated in the phase-field model, supported by 3D structural analysis, should be initiated.

COMPARISON OF RE-C FIXED POINT CELLS AND THEIR T-90 TEMPERATURES BETWEEN NMIJ AND VNIIOFI

B.B. Khlevnoy[1], I.A. Grigoryeva[1], M.L. Samoylov[1], Y. Yamada[2]
[1] *VNIIOFI, Moscow, Russia*
[2] *NMIJ, AIST, Tsukuba, Japan*
E-mail (B.B. Khlevnoy): khlevnoy-m4r@vniiofi.ru

The previous comparison of Re-C fix point cells built by NMIJ and VNIIOFI took place in 2003 and discover a relatively high difference of melting temperatures of about 1 °C [1]. The reason of such disagreement was associated mostly with impurities of the VNIIOFI made cell.

Since that VNIIOFI has significantly improved his Re-C point, first of all by using purer rhenium powder and improving preparation and filling procedure in a way to prevent contaminations during a process of cell building. Some changes in the design of crucibles have been also done by both NMIJ and VNIIOFI.

New comparison was carried out in 2009 at VNIIOFI. Both NMIJ and VNIIOFI cells had similar dimensions and were built using 5N purity rhenium of the same supplier. The cells were heated in the same furnace and measured on consecutive days using the TSP-2 radiation thermometer. To eliminate probable instability of the thermometer a third Re-C cell was used as reference. It was placed in another furnace and measured each day just after a cell to be compared.

As a result of the comparison it was found that the difference of the melting temperatures was less than 0.1 °C and that the NMIJ cell had higher temperature.

High-temperature fixed points, due to their high reproducibility, are perfect artifacts for radiation temperature scale comparisons. Therefore, cell comparison described above gave a good opportunity to compare NMIJ and VNIIOFI T-90 scales, at the temperature of Re-C point, which is about 2475 °C. The scales agreed within the uncertainties.

[1] N. Sasajima, M. K. Sakharov, B. B. Khlevnoy, Y. Yamada, M. L. Samoylov, S. A. Ogarev, P. Bloembergen and V. I. Sapritsky. 'A comparison of Re-C and TiC-C eutectic fixed-point cells among VNIIOFI, NMIJ and BIPM'. *Proceedings of TEMPMEKO 2004, edited by Bermanec L.G., Stasic T. and Veliki T.*, Cavtat-Dubrovnik, 2004, pp.1107-1115.

PALLADIUM-CARBON EUTECTIC FIXED POINT FOR THERMOCOUPLE CALIBRATION

M. Gotoh, A. D. W. Todd, D. J. Woods, and K. D. Hill
Institute for National Measurement Standards, National Research Council of Canada, Ottawa, Canada, K1A 0R6
E-mail (Masahiko Gotoh): masahiko.gotoh@nrc-cnrc.gc.ca

Metal-Carbon eutectic fixed points are poised to reduce the uncertainties associated with the calibration of contact thermometers at temperatures above the freezing point of silver. At 1492 °C, the melting plateau of the Pd-C eutectic point is well-located near the upper temperature limit of the Pt/Pd thermocouple, and also suited to calibrations of thermocouple types R, S, B and W-Re.

A Pd-C eutectic fixed point suitable for thermocouple calibration was produced in a single zone, vertical tube furnace having molybdenum disilicide heating elements. The axial temperature distribution of the furnace was found to vary 0.3 K over an 80 mm distance at the location of the fixed point crucible at a temperature of approximately 1310 °C. Palladium powder (99.995% purity) was mixed with 2.7 wt. % carbon powder (99.9999 % purity) in a high purity graphite (less than 20 ppm ash content) crucible. The crucible is a simple two-piece, single inside wall design with a 33 mm outer diameter. The full height of the eutectic material space is 110 mm and the calculated volume is approximately 14 cm3. The Pd-C eutectic crucible was filled in multiple steps, beginning with a powder charge (Pd and C powder) totaling 70 g and ending with a crucible filled with approximately 120 g of eutectic material. The ingot height contained in the final cell is approximately 80 mm.

The melting plateau was measured using a platinum/palladium thermocouple that was calibrated at the freezing points of zinc, aluminium, silver and gold. The repeatability of the melting point has been investigated for one palladium-carbon cell.

A W-CELL FIXED POINT FOR *IN SITU* CALIBRATION OF RADIATION THERMOMETERS IN AN INDUCTION FURNACE

N. Sasajima, Y. Yamada, J. Ishii
National Metrology Institute of Japan, AIST (NMIJ, AIST), Tsukuba, Japan
E-mail (corresponding author): n.sasajima@aist.go.jp

UO_2 and mixed oxides of plutonium and uranium (MOX) are used as fuels in nuclear power plants. To obtain reliable thermodynamic data such as melting temperature of fuels is very mportant from the point of thermal design for nuclear power plants and safety evaluation during a severe nuclear accident. Although the melting temperature of fuels has been investigated ever since the development of nuclear reactors, reliable data with small uncertainty are limited due to difficulty in precise measurement at high temperature. Measurement of melting and freezing plateaus of oxide samples encapsulated in tungsten (W) crucibles is the most reliable method to obtain melting temperature of UO_2 (approximately 2865 °C [1]) because it can avoid composition change of $UO_{2.00}$. To accurately measure the melting temperature, high-temperature fixed points filled in W crucibles are desired to provide means of calibrating radiation thermometers *in situ* in a furnace located in a radiation protection area. In this paper we present the first results of the investigation to realize fixed points in W cells with temperature exceeding 2500 °C.

An induction furnace, which can be operated up to 3000 °C with W reflectors, was constructed at NMIJ. A W-γW_2C eutectic alloy (approximately 2710 °C) was selected as the fixed point material to be realized in a W crucible. The W crucible has a dimension of 14 mm outer diameter and 24 mm length. The blackbody cavity has an aperture diameter of 3 mm. Tungsten powder was mixed with graphite powder at a hypo W-γW_2C eutectic (i.e. W rich) composition. The mixture was filled in W crucible, which in turn was placed in the induction furnace and raised to a temperature above the eutectic temperature in Ar atmosphere. The process was repeated until the crucible was filled.

The melting and freezing plateaus were observed by means of a radiation thermometer. Although the duration of the melting plateaus was short with large melting range due to poor temperature distribution and imperfect filling, W-γW_2C eutectic point cell showed repeatable melting temperature, demonstrating their possibility to become *in situ* calibration means for radiation thermometer in induction furnaces.

[1] M. Baichi, et al., “Thermodynamics of the O-U system: 3 - Critical assessment of phase diagram data in the U-UO2+x composition range”, J. Nucl. Mater. 349 (2006) 57-82.

RESULTS OF LONG-TERM STABILITY TESTS PERFORMED FOR THE EUTECTICS CO-C AND PT-C

W. Tie-jun, B. Cheng-yu, D. Wei, Y. Zun-dong, P. Bloembergen
National Institute of Metrology (NIM), Beijing 100013, China
E-mail (corresponding author): wangtj@nim.ac.cn

Under the auspices of WG-5 of the CCT the eutectics Co-C, Pt-C and Re-C, with eutectic temperatures of 1597 K, 2011 K, 2747K, respectively, are presently investigated, within the context of a high-temperature fixed point (HTFP) research project [1], for their suitability to serve as reference points for dissemination of T and T_{90}. HTFP cells to serve as reference standards should show a long-term stability and a high robustness (immunity to breakage). So just these parameters have been tested in the first workpackage of the HTFP research project, referred to. However, it still makes sense to supplement this work by dedicated long-term stability studies performed either for cells of special design or by extending the test period or varying the test method for existing cells.

In recent years NIM has been working steadily on constructing HTFPs cells of various designs including the evaluation of their stability. This work has been continued in two directions.

(a) A Pt-C cell of new design has been constructed in hybrid manner including sleeve and foil lining, the cavity being protected against breakage by buoyancy forces, by means of a fragmented circular support of pure graphite embedded in the ingot. Its stability was tested and compared with that shown by cells of current design.

(b) Long-term stability tests, earlier performed for a Co-C cell with C/C sheet incorporated and a Pt-C cell equipped with graphite sleeve, involving 80 and 50 melting and freezing cycles, respectively, have been pursued, to check for anomalous drift or degrading robustness, possibly appearing when extending the test period.

Procedures relied on and results obtained under (a) and (b) will be presented in the paper. Possible mechanisms underlying instability, drift, saturation of drift, and failure by fracture, inferred from the analysis of the results of our tests, will be discussed.

[1] Machin, G., Bloembergen, P., Hartmann, J., Sadli, M., Yamada Y., “A concerted international project to establish high temperature fixed-points for primary thermometry”, *Int. J. Thermophys.*, 28, 1976-1982, 2007

A JOINT WC-C PERITECTIC FIXED POINT PROJECT BETWEEN THE NIM AND THE NMIJ

C. Bai[1], N. Sasajima[2], Y. Yamada[2], Z. Yuan[1], C. Ara[2], T. Wang[1]
[1] *National Institute of Metrology (NIM), Beijing, China*
[2] *National Metrology Institute of Japan, AIST (NMIJ, AIST), Tsukuba, Japan*
E-mail (corresponding author): baichy@nim.ac.cn

WC-C peritectic fixed point (2749 ºC) has transition temperature higher than any of the metal-carbon (M-C) eutectic high-temperature fixed points (HTFPs), currently being developed in many metrology institutes. We have shown in a recent investigation that WC-C has repeatability of 11 mK for a single cell, melting range of 117 mK, long term stability within 34 mK, and reproducibility among different cells of 50 mK [1]. This performance is comparable to the best of the M-C eutectic HTFPs. In the two-fixed-point interpolation scheme, which utilizes one metal fixed point (e.g. Cu point) and one HTFP, an HTFP of good enough performance with the highest transition temperature gives the smallest uncertainty over the whole interpolation range [2]. From this point, a joint research project is being carried out between the NIM and the NMIJ to establish the WC-C peritectic point as a national primary standard. This project comprises of 1) establishment of filling method of WC-C peritectic cells, 2) improvement of high-temperature furnace, 3) studies of impurity effect and furnace effect for the transition temperature, 4) comparison of T_{90} with WC-C fixed point between the NIM and the NMIJ, 5) realization of two-point interpolation scale by use of WC-C and copper point. As parts of the joint research project, extension of the Nagano furnace operating temperature to reach the WC-C peritectic point, construction of WC-C cells, and a comparison of T_{90} with WC-C peritectic fixed point between the NIM and the NMIJ were carried out.

Three WC-C peritectic blackbodies were constructed by the authors at the NMIJ. The three WC-C peritectic blackbodies have the same design, but were constructed from different sources of tungsten powder. First, T_{90} measurement with the WC-C blackbodies was carried out at the NMIJ using an LP5 radiation thermometer and a Nagano furnace. The Nagano furnace was improved to be operational up to 2800 ºC. During the measurement, a Re-C eutectic fixed-point blackbody was measured to monitor drift of the LP5 radiation thermometer if any. Three WC-C peritectic blackbodies were then transported to the NIM, and T_{90} measurements were carried out in a Thermo Gauge furnace and an LP4 radiation thermometer. The comparison result of T_{90} with WC-C fixed point between the NIM and the NMIJ with the estimated uncertainty is presented in this paper.

[1] N. Sasajima and Y. Yamada, Performance evaluation of WC-C peritectic high-temperature fixed point, Proceeding of ICCAS-SICE 2009, pp. 3307-3310.
[2] Y. Yamada, Uncertainty of radiation thermometers calibrated by interpolation between fixed points, Proceeding of ICCAS-SICE 2009, pp. 2812-2815.

DEVELOPMENT AND INVESTIGATION OF WC-C PERITECTIC HIGH TEMPERATURE FIXED POINT CELLS

B.B. Khlevnoy and I.A. Grigoryeva
VNIIOFI, Moscow, Russia
E-mail (B.B. Khlevnoy): khlevnoy-m4r@vniiofi.ru

Recently suggested new high temperature fixed point WC-C [1] is especially attractive for people involved both in radiation thermometry and radiometry. Its melting temperature, which is about 3033 K, is very close to colour temperature of tungsten halogen lamps, which are usually used for spectral irradiance.

Although the TiC-C fixed point has similar melting temperature, it has some disadvantages in compare with WC-C that was clearly shown in [2]. One of the disadvantages is the absence on the market titanium powder purer than 4N [2,3], while tungsten can be found as pure as 5N or even 6N. Another one is some difficulty of filling a TiC-C cell. Finally, the WC-C shows better plateau shape and repeatability.

However, up to now only one laboratory [2] has published the results of investigation of the WCC fixed point. To make certain of its high reproducibility we have to compare WC-C cells built in several different laboratories.

VNIIOFI started working with WC-C shortly after the first publication [1] and finally built cells out of 5N purity tungsten powder using two different methods of filling. Both methods cells showed almost identical melting plateaus with the melting range of about 100 mK and repeatability of 20 mK. The paper will present the experience in developing the WC-C fixed point cells and the results of their investigation.

[1] Y. Yamada, Y. Wang, N. Sasajima, Metal carbide-carbon peritectic systems as high-temperature fixed points in thermometry, *Metrologia* 43, L23 (2006)
[2] N. Sasajima and Y. Yamada, Investigation of TiC–C Eutectic and WC–C Peritectic High-Temperature Fixed Points, *Int J Thermophys* (2008) 29: 944–957, DOI 10.1007/s10765-007-0334-4
[3] M. Sakharov, B. Khlevnoy, V. Sapritsky, M. Samoylov, S. Ogarev, Development and investigation of high- temperature Fixed Point based on TiC-C Eutectic, in *Proceedings of TEMPMEKO 2004, 9th International Symposium on Temperature and Thermal Measurements in Industry and Science*, ed. by D. Zvizdić, L.G. Bermanec, T. Veliki, T. Stašić (FSB/LPM, Zagreb, Croatia, 2004), pp. 319–324

CONSTRUCTION AND EVALUATION OF A SET OF Co-C AND Re-C EUTECTIC CELLS

M. Sadli, F. Bourson, B. Rougié, S. Briaudeau
Laboratoire Commun de Métrologie LNE-CNAM, La plaine Saint-Denis, France
E-mail: mohamed.sadli@cnam.fr

LNE-INM is involved in the research about metal-carbon eutectic for almost a decade and has aimed at developping robust, repeatable and stable cells. After having devoted large efforts to the design of the cells and the achievement of a simple, fast and clean filling technique, our latest version of cells showed satisfactory behaviour in terms of robustness and reproducibility.

Two metal-carbon eutectic points were under study recently: the Co-C (1324 °C) and the Re-C (2474 °C) eutectic points. The design of the cells is based on the "hybrid" structure first proposed by our institute [1]; namely, using a sleeve and two C/C sheets to separate the eutectic ingot from the outer wall of the crucible and consequently thermally (almost) insulating the ingot from the furnace while avoiding direct contact of the ingot with the C/C sheets, thus increasing the robustness of the cell. The crucibles were filled inside the Vega HTBB 3200pg high-temperature furnace using an original method, with a reduced number of filling steps, which allows finalising the cells with repeatable high filling rates. Impurity analyses performed on the Co-C and the Re-C samples obtained during the filling showed that the method does not introduce noticeable pollution of the eutectic ingot.

In this paper, we will describe the filling method applied up to the Re-C point and the results of the characterisation of a set of cells as well as the results of the comparison of the cells performed in two different furnaces.

[1] F. Bourson, S. Briaudeau, B. Rougié, M.Sadli "Developments around the Co-C eutectic point at LNE-INM/Cnam" Tempbeijing, 20th-23rd October 2008

COMPARISON OF PYROMETRIC Co-C AND Re-C EUTECTIC POINT CELLS BETWEEN LNE-INM AND VNIIM

M. Sadli[1], M. Matveyev[2], F. Bourson[1], V. Fuksov[2], Yu. A. Sild[2], A. I. Pokhodun[2]
[1] *Laboratoire Commun de Métrologie LNE-CNAM, La plaine Saint-Denis, France*
[2] *VNIIM, St-Petersbourg, Federation of Russia*
E-mail (corresponding author): mohamed.sadli@cnam.fr

VNIIM and LNE-INM have collaborated for several years in the field of metal-carbon eutectic points. The first action was the construction of a Pt-C cell at VNIIM using the LNE-INM technique and cell design. The cells constructed in each of the labs have been studied and compared [1].

The two laboratories have followed their common work on studying and comparing Co-C and Re-C cells. Different designs and filling techniques were applied. The comparison of the cells was performed at LNE-INM using the high-temperature blackbody furnace HTBB-3200pg which was thermally optimised prior to the implementation of the cells.

The results of the comparison showed that the Co-C cells were comparable to the level of 0.01 K while the Re-C cells were quite different in terms of melting temperatures and this difference reached about 0.7 K.

In this paper we will describe the cells used and the methodology of the comparison. We will focus on the temperature differences that were obtained at the highest temperature to derive an explanation for this temperature difference.

[1] M. S. Matveyev, M. Sadli, Yu. A. Sild, A. I. Pokhodun, F. Bourson «Experience of Construction and Study of Pt–C Eutectic in VNIIM and Cooperation with LNE-INM» Int J Thermophys (2009) 30:47–58.

Fixed Points - M-C eutectics I

Monday
15:00 to 16:20
Emerald 1
Session Chairman: Andrea Peruzzi

DESIGN, CONSTRUCTION AND EVALUATION OF NI-C AND CO-C EUTECTIC FIXED POINTS

J. Bojkovski, M. Hiti, V. Batagelj and J. Drnovšek
MIRS/UL-FE/LMK, Ljubljana, Slovenia
E-mail (corresponding author): jovan.bojkovski@fe.uni-lj.si

A new design of eutectic fixed point for contact and non-contact measurements has been developed at MIRS/UL-FE/LMK. The cell is designed in such way that it can be used for only thermocouple calibration in vertical or horizontal furnace, for calibration of pyrometers in horizontal furnace and for simultaneous calibration of contact and non-contact thermometers in horizontal furnace. Special care has been taken in order to achieve repeatability, reproducibility and robustness. This includes also usage of carbon sleeve in combination with carbon sheets. With such design, significant improvement in robustness and stability of the cell has been achieved.

The inner part of the cell comprises a thermometer well with an inner diameter of 4 mm and/or a black body cavity with inner diameter of 6 mm inserted into a graphite tube with 1 mm wall thickness. The outer part of the cell comprises a graphite tube with 24 mm outer diameter and two end caps.

The inner part that can be filled with fixed-point material has a volume of about 9 cm^3. A graphite foil is wrapped around the inner part of the cell before it is inserted into the outer cell tube. The assembly is closed by screwing an end cap to each end of the outer tube. Graphite felt was put between the inner part of the cell and the end caps. Since the inner and outer parts of the cell are not firmly attached an expansion of the material in the inner part of the cell during heating can be compensated by the graphite felt. The multilayer design of the cell ensures that in the case of cracking of the cell the material does not run out.

All the measurements are performed within specially designed seven-zone furnaces within which different gradients can be realized. The melting temperatures of NI-C and CO-C measured with thermocouple type R, calibrated at zinc, aluminium, silver, copper and palladium fixed points, and non-contact thermometer, calibrated by comparison against different black bodies, agree within their uncertainties.

REALIZATION OF FE-C EUTECTIC POINT USING AN ALUMINA CRUCIBLE

Y.-G. Kim, K. S. Gam and I. Yang
KRISS, Daejeon, Korea
E-mail (corresponding author): dragon@kriss.re.kr

As new high temperature fixed-points above the freezing point of Cu, metal-carbon eutectic points have been widely investigated in many National Metrology Institutes. Usually, graphite is used for a crucible material because it is one of the constituents of eutectic alloys, leading to chemical inertness. Also, it is easy to machine and available to obtain as a high-purity form. However, it is inevitable that the eutectic composition be changed to hyper-eutectic due to the excessive carbon from the crucible. This composition change may lead to instability of melting behavior after long-term exposure to high temperature. In this work, alumina crucible instead of graphite material is adopted to minimize the change in the chemical composition of metal-carbon eutectics.

Fe-C eutectic alloy, which is known to have the lowest melting temperature among the metal-carbon systems that have been published so far, is investigated at various melting offset temperatures. Double-wall alumina crucible and alumina thermometer-well were prepared for the purpose of thermocouple calibration. The length of the cell was 10 cm and Fe-C mixture of about 54.5 g was filled in pure Ar atmosphere. A type S thermocouple was used to monitor the melting and the freezing behavior. The melting plateaus were obtained at three different melting offset temperatures (+4 °C, +9 °C and +19 °C). Freezing tests were performed at the fixed offset temperature of –11 °C during the experiments. Total of 26 melting and freezing tests were done to examine the reproducibility. During the tests, furnace temperature was intermittently lowered to room temperature.

The melting emf was determined from the inflection point of the melting plateau, and the freezing emf was from the maximum emf in the freezing curve. The melting emf was very reproducible regardless of melting offset temperature. The standard deviation of 26 melting emf was calculated to 0.2 μV (equivalent to 0.02 °C). The average freezing emf was lower by 14.5 μV than the average melting emf, and standard deviation of freezing emf was five times larger than that of melting. After the experiments, it was found that alumina crucible was not broken. Therefore alumina crucible can be used as a container of the Fe-C eutectic point, leading to very high reproducibility. At higher temperature range, we expect that alumina is safe as a container material for metal-carbon eutectic cells.

COBALT-CARBON EUTECTIC FIXED POINT FOR CONTACT THERMOMETRY

M. Gotoh, A. D. W. Todd, D. J. Woods, and K. D. Hill
Institute for National Measurement Standards, National Research Council of Canada, Ottawa, Canada, K1A 0R6
E-mail (Andrew D.W. Todd): Andrew.todd@nrc-cnrc.gc.ca

The Co-C eutectic point has been realized at the National Research Council of Canada. The furnace used was a single zone, vertical furnace with molybdenum disilicide heating elements. The axial temperature distribution of the furnace was found to vary less than 0.3 K over a distance of 80 mm in the vicinity of the fixed point crucible at the eutectic temperature. The purity of the materials used was 99.995% and 99.9999% for the Co slugs and graphite powder, respectively. The crucibles were made of high purity graphite containing less than 20 ppm ash content.

Three Co-C cells were produced; the first two cells contain approximately 90 g of the eutectic material and their performance validated the fabrication process. The Co-C eutectic cells were produced using a single filling, and x-ray imaging confirmed that the eutectic material occupies approximately 90% of the cell's interior volume. The third cell containing approximately 45 g of eutectic material was produced to probe the optimum size of the eutectic ingot within the cell and to minimize the effect of the furnace gradient on the melting plateau. The crucible is a simple two-piece design, and the central portion consists of a single inside wall with a thermowell in the centre. The outer diameter of the crucible is 33 mm. The interior volume of the crucible was calculated to be approximately 14 cm^3 and this volume occupies approximately 110 mm in height. Many melt-freeze cycles were performed without damage to the crucibles, thereby demonstrating the robustness of the crucible design.

The melting plateaus were measured using a platinum/palladium thermocouple calibrated at the freezing points of zinc, aluminium, silver and gold. The inflection point of the melting plateaus was found to be reproducible, with a standard deviation of 20 mK. The effect of the quench rate (from the melt) on the inflection point of the melt plateau was investigated by choosing furnace offsets that varied from 7 K to 26 K below the freezing temperature. Over this range of quench rates the effect was negligible.

Calibration Procedures and Facilities I

Monday
15:00 to 16:20
Emerald 2
Session Chairman: Andrea Merlone

ESTIMATING UNCERTAINTIES IN INTERPOLATIONS BETWEEN CALIBRATION DATA FOR THERMOCOUPLES

J. V. Pearce[1], P. Harris[1], J. Greenwood[1]
[1] *National Physical Laboratory, Teddington, United Kingdom*
E-mail (corresponding author): jonathan.pearce@npl.co.uk

Two methods for evaluating thermocouple calibration uncertainty over the temperature range of the calibration are presented, when the thermocouple is calibrated at only a few temperatures. The evaluation of the uncertainty at fixed point temperatures is well established, but it is often not clear how the uncertainty arising from interpolation between fixed points can be determined.

We present a conventional method, based on that described in the "Guide to the expression of uncertainty in measurement" (GUM), and a numerically based Monte Carlo method, for quantifying the calibration uncertainty arising from the use of an interpolating polynomial defined by calibration data. The two methods are compared and found to be in excellent agreement, but the Monte Carlo method is, in general, more flexible, for example, when measurements are described by non-normal distributions.

CONSTRUCTION OF SODIUM HEAT PIPE FURNACES AND THE ISOTHERMAL CHARACTERISTICS OF THE FURNACES

X. K. Yan[1,2], Y. N. Duan[1], C. F. Ma[2], Z. F. Lv[3]
[1] *National Institute of Metrology (NIM), Beijing, China*
[2] *Beijing University of Technology, Beijing, China*
[3] *Beijing University of Chemical Technology, Beijing, China*
E-mail (Xiaoke Yan): yanxk@nim.ac.cn

The high temperature sodium heat pipes are widely used in the thermometry field due to their good isothermal characteristics over the temperature range from 500 °C to 1200 °C. At present, a number of national metrology institutes (NMIs) have used sodium heat pipe furnaces for the realization of ITS-90 freezing points of aluminum and silver. In order to perform a similar study, in recent years, the National Institute of Metrology (NIM) has set up a new system of fabricating sodium heat pipes and the corresponding furnaces have been constructed.

In this paper, we describe the newly developed apparatus for the fabrication of sodium heat pipes, sodium heat pipe furnaces and their isothermal characteristics.

The system for the fabrication of sodium heat pipes is capable of leak detecting, cleaning, vacuum degassing, fluid charging, sealing and wick wetting. During the construction of heat pipes, filling high purity sodium into the inside of heat pipes and keeping simultaneously the sodium from oxidizing and contaminating are crucial to the performance of heat pipes. In order to solve this key technology, a special charging system was designed.

The isothermal characteristics of the sodium heat pipe furnaces were studied. The experimental results show that the vertical largest temperature differences over the 150 mm distance of the thermometer well bottom of the aluminum fixed cell in the sodium heat pipe furnaces (s/n SHPF-1 and s/n SHPF-2) did not exceed 15 mK and 11 mK respectively, when the temperatures of the furnaces were set to approximately 3 °C below the fixed-point temperature of aluminum. Furthermore, some factors influencing the temperature uniformity are discussed.

A NEW DIPHENYL GAS CONTROLLED HEAT PIPE

O. Kerkhof[1], J. L. W. A. van Geel[1], A. Merlone[2]
[1] *VSL BV*
[2] *Istituto Nazionale di Ricerca Metrologica, Str. delle Cacce 91, 10135 Torino, Italy*
Email (corresponding author): okerkhof@vsl.nl

Under a scientific cooperation contract between the Italian Istituto Nazionale di Ricerca Metrologica (INRiM) and the Dutch Metrology Institute VSL, a new gas controlled heat pipe (GCHP) has been made. Since several years the diphenyl has been used with good results for sealed heat pipes for fixed point furnaces; for the first time this synthetic compound has been used as a working fluid in a six wells GCHP using helium as controlling gas. The diphenyl heatpipe approximately has the same operating range as the mercury heatpipes and may provide for a safe alternative if the slightly added uncertainty is accepted. The GCHP has been coupled to an existing Na GCHP heatpipe in the VSL laboratory to create a temperature amplifier. This temperature amplifier will be used as calibration facility for standard platinum resistance thermometers and thermocouples. It will perform calibration by comparison of such instruments in the field between 500 °C and 950 °C, corresponding to a pressure ranging between 0,01 and 2,0 bar. This pressure range corresponds to temperatures between 320 °C and 760 °C in the biphenyl heatpipe.

The device has been manufactured and tested at INRiM in the heat pipes laboratory of the thermodynamic division, then transported to the VSL laboratories for the filling procedure and startup. This paper describes the GCHP, all the auxiliary devices, the startup procedure and the first results obtained in terms of temperature stability and uniformity.

Radiation Thermometry I

Monday
15:00 to 16:20
Adria
Session Chairman: Mark Ballico

EXPERIMENTAL AND NUMERICAL INVESTIGATION OF THE TEMPERATURE FIELD OF FIXED POINT'S CAVITY

V.M. Fuksov, A.I. Pokhodun
D.I. Mendeleev Institute for Metrology (VNIIM), St. Petersburg, Russia
e-mail: V.M.Fuksov@vniim.ru

A temperature field non-uniformity of a blackbody cavity is one of components of uncertainty of fixed points realization. At present study the design and opportunities of the temperature furnace used in VNIIM is described. Dependence of uniformity of a temperature field on various factors is shown by the example of results of numerical calculations of a temperature field of VNIIM's copper and gold cells, realized in software packages Elcut and Ansys. The basic accent is made on calculation of radiation heat exchange between crucible elements and an environment and furnace cavity, as a dominating component heat transfer. Results obtained using analytically and numerically calculated angular factors of radiation of heat exchange.

The data obtained during measurements of temperature field of a cavity fixed points during phase transitions of copper and gold by spectrocomparator with high sensitivity, are also shown here. Both theoretical calculation and experiment were realized at various distributions of temperature along an external surface of crucible. Good concurrence of results between steadystate calculation of a temperature field and the measured data with the best entry conditions is observed, that confirms adequacy of thermal model and an opportunity of its use further for more detailed analysis of problems of phase transition. Average value of non-uniformity of a temperature field on a cavity for points of phase transition of copper and gold was 40 mK, and on thickness of a graphite wall –20 mK. In this paper the reasons of occurrence of the big gradients inside a cavity fixed points during the phase transition, received during some experimental researches, are also discussed.

Results of this investigation have helped to reduce uncertainty of type A realization of fixed points, and also to estimate uncertainty of type B, connected with heating leaks through aperture of blackbody.

TEMPERATURE AND THICKNESS DEPENDENCE OF IR OPTICAL PROPERTIES OF SAPPHIRE AT MODERATE TEMPERATURE

G.W. Lee[1,3], S. Jeon[1,2], S. Park[1], Y. S. Yoo[1], C. Park[1]
[1] Korea Research Institute of Standards and Science, Daejon, Repulbic of Korea
[2] University of Science & Technology, Daejon, Repulbic of Korea
E-mail: gwlee@kriss.re.kr

Infrared (IR) windows are used to separate radiation thermometers and thermal imagers from a hot object. When the windows are heated directly or indirectly by hot objects, the IR window itself emits radiation, which gives additional signal to the detectors. Moreover, the radiation from the object and heated IR window varies with the thickness of IR windows in the semitransparent region. Therefore, the optical properties of the heated IR windows, such as temperature and thickness dependence of transmittance and emittance, should be scrutinized before determining the temperature and image of the object. So far, the temperature dependence of transmittance has been studied at high temperature on sapphire. Here, we report temperature and thickness dependence of IR optical properties of UV grade sapphire at moderate temperature, which has been rarely reported.

The temperature and thickness dependence of transmittance is measured by FT-IR spectrometer (Nicolet 6700) and a heating furnace. IR windows are placed at the center of the furnace with a slit and directly heated from room temperature to 500 °C. The heating zone of the furnace is covered by water-cooled stainless steel with a slit so that IR light in FT-IR spectrometer transmits through the IR windows. With careful correction, temperature and thickness dependence of transmittance is obtained. From the thickness dependence of transmittance, extinction and reflection coefficients are calculated to get emissivity and reflection of sapphire. Interestingly, the reflection shows thickness dependence.

We also directly measure the emissivity of sapphire at moderate temperature (300 °C) using heating subplate method which is used for opaque materials. For the measurement, gold coated subplate is used to reduce the radiation of the subplate. With the transmittance of sapphire and reflectance of gold coated subplate, the emissivity is measured, and compared with the calculated emissivity obtained from extinction and reflection coefficients. The two emissivities are overall agreed with each other in the semitransparent region.

LOW TEMPERATURE BLACKBODIES FOR TEMPERATURE RANGE FROM –60°C UP TO 90 C

S. P.Morozova[1], B. E. Lisyanskiy[1], A. A. Stakharny[1], M. L. Samoilov[1], S. A. Ogarev[1], Y. S. Yoo[2], C.-W. Park[2] and S.-N. Park[2]
[1] *VNIIOFI, Moscow, Russia*
[2] *KRISS, Daejeon, Korea*
E-mail (S.A.Ogarev): ogarev-m4@vniiofi.ru

Two cavity-type models, VTBB and BB100K1, of low temperature blackbodies (BB) are developed at VNIIOFI for operation as IR radiation sources of the Middle Background Calibration Facility in the temperature range from –60 °C to 90 °C, which is being constructed by KRISS for calibration of multi-spectral cameras for space applications.

VTBB model with 30 mm output aperture is utilized as a standard radiation source featuring high emissivity. VTBB covers the whole temperature range under vacuum environment (up to $1\cdot10^{-4}$ mbar), and the temperature range from 20 °C to 90 °C under open air conditions. For operation under vacuum conditions VTBB has hermetic housing and flange for mounting to vacuum chamber. BB100K1 has a wide aperture of 100 mm diameter, which shows stable operation in the temperature range from –60 °C to 90 °C inside vacuum chamber, and in the temperature range from –40 °C to 90 °C in dry-air or inert gas environment. BB100K1 can also operate in the temperature range from –20 °C to 90 °C under open air conditions with the usage of an extra hood with an aperture. The aperture is convenient for carrying out routine calibration procedures of laboratory equipment. The radiating cavity temperature of VTBB and BB100K1 is stabilized at the level of ±0.01 °C by means of external precise closed-loop liquid thermostat HUBER of UNISTAT 705 model. Temperature distribution along the radiating cavity and across the BB bottom is monitored by 5 precision PRT thermometers and digital multimeter equipped with a scanner card.

The paper describes VTBB and BB100K1 design, their metrological characteristics and budget of measurement uncertainties. Effective emissivity of radiating cavities of both blackbodies, covered with LORD Aeroglaze Z306 black paint, was calculated with the usage of STEEP3 Monte-Carlo simulation software, taking the measured temperature gradients into account. The numerical calculations yield emissivity of at least 0.9997 for VTBB cavity, and 0.997 for BB100K1 cavity. Radiance temperature distributions of BB are measured by using a spot radiation thermometer (HEITRONICS) and a thermal camera (ThermoCAM SC640, FLIR). Temperature gradient along the VTBB cavity does not exceed ±40 mK under vacuum operation conditions at the temperature range between –60 °C and 90 °C. Temperature gradiant across 100 mm aperture of BB100K1 does not exceed ±50 mK in the temperature range between –40 °C and 90 °C under dry-air environment.

CORRECTING RADIATION THERMOMETRY MEASUREMENTS FOR THE SIZE-OF-SOURCE EFFECT

P. Saunders
MSL, Lower Hutt, New Zealand
E-mail: p.saunders@irl.cri.nz

The size-of-source effect (SSE) is a major contributor to error and uncertainty in both the calibration and use of a radiation thermometer, and applying corrections for the SSE is necessary to achieve high accuracy. There are several recognised methods for measuring the SSE, including the newly developed scanning method, and each one measures a slightly different quantity. Moreover, the measured SSE values also depend on various parameters, such as diameter of the black spot or maximum aperture diameter. When applying corrections, it is necessary, in principle, to recognise which SSE quantity has been measured, as the correction formulae for each differ. The corrections should also be independent of the measurement parameters.

In this paper the different formulae for correcting thermometer signals to a common target size are derived for each SSE quantity. It is shown that the differences are only second order, so that for sufficiently small SSE, a single simple approximation can be made. Thus, the correction methods are the same for each SSE quantity, irrespective of whether the SSE values are close to 1, as in the direct method, or close to 0, as in the indirect method. It is also shown that measurements of the radiance distribution surrounding the target do not require corrections for the SSE, even at the highest level of accuracy.

Resistance Thermometry I

Monday
16:50 to 18:10
Emerald 1
Session Chairman: Wes Tew

TYPICAL R(T90)-CHARACTERISTICS OF PLATINUM THIN FILM RESISTANCE THERMOMETERS IN THE TEMPERATURE RANGE -50 … 660 °C

D. Boguhn
Endress+Hauser, Nesselwang, Germany

The temperature measurement by using the temperature-dependent changes of metals is an established method for many years. If wire wound (WW) platinum resistance elements in capsules of glass or ceramic were primarily used in industrial applications, thin film (TF) measurement resistances have been on the advance for a number of years. Instead of a wrapped wire some about 1 µm thick conductors which form the Pt measurement resistance of such a TF sensor are produced by sputtering and structuring photo lithographically of a highly purified platinum layer on a ceramic substrate. In addition covering and passivation layers protect these thin platinum layers from pollutants even at high temperatures. The advantages of TF sensors lie in the small dimensions, mechanical ruggedness and a considerably more reasonable price clearly due to automated mass production.

However, until now, Pt thin film elements could only partly replace the WW sensors. On the one hand, many TF sensors in the past have not proved to be adequately long term stable at higher temperatures. Especially if they are used under harsh industrial conditions often any contaminations of the highly pure platinum layer caused also by changes of the physical and chemical properties (aging effects) of the covering and protecting layers as well as an unsteadiness of the contact resistances etc. can be observed which cause temporary or even permanent changes in the total $R(t_{90})$-characteristic of a thin film resistor. New materials and optimized production technologies have brought about clear improvements within the last few years.

On the other hand the temperature/resistance characteristics of both sensor types differ noticeably at very low, but especially at higher temperatures >250 °C. Compared to the standard characteristic (defined e.g. in IEC 60751) originally devised using wire wound Pt resistances TF sensors show a clear deviation towards lower resistance values. The narrow tolerances of class A, e.g., can only be adhered to in a restricted temperature range. This effect is typical of conventional TF sensors and is due in their constructive construction, particularly in the different thermal expansion behaviour of the different layers. In order to investigate more in detail this behaviour and to determine more exactly the typical $R(t_{90})$ characteristics of platinum TF temperature resistors extensive calibration measurements with different sensor types provided by various suppliers were carried out. A handful of wellequipped calibration laboratories of German manufacturers of industrial thermometers, the PTB temperature lab as well as some well-known manufacturers of TF platinum sensors have taken part in this study.

Within the scope of a paper for the TEMPMEKO 2010 it is intended to introduce the special properties of the current platinum TF sensors which are not so widely known yet. Basing on the results of the mentioned extensive comparison measurements with a multitude of current platinum thin-film sensors the typical and systematic deviation of their $R(t_{90})$ characteristics in comparison to the classical wire-wound resistors will be demonstrated as well as some facts and reasons causing this effect.

Besides, some actual innovations on platinum TF sensors and their improvements, especially with regard to their characteristic and long-term stability, will be presented by means of some corresponding measurement results as well. Based on the analysis of those effects and their influencing factors on the sensor characteristics some manufacturers of platinum TF sensors have recently intensified their work on a further improvement of their TF sensors. By means of some optimizations in the sensor design, the usage of new materials and structures which are better adjusted each to another with regard to their thermal expansion behaviour, new technologies in the electrical contacting and wiring of the resistor,… in the meantime a remarkable long-term stability of TF sensors even at temperatures up to 1000 °C at oxidizing atmosphere has achieved. Recently even a completely new generation of Pt thin film sensors has been introduced that are able to almost perfectly follow the standard characteristic curve of IEC60751 over the complete temperature range of –200 … 600 °C with a quite high long term stability. Especially for industrial applications this presents some new possibilities in the practical use of Pt TF sensors and is one step more toward the aimed reduction of the often still high uncertainties in temperature measurement there.

MEASUREMENT OF AC AND DC INSULATION LEAKAGE IN PLATINUM RESISTANCE THERMOMETERS UP TO 960 °C

C. García, D. del Campo, F. Raso
Centro Español de Metrología, Tres Cantos, Spain
E-mail (corresponding author): mcgarciaiz@cem.mityc.es

It is well known that insulation breakdown at high temperatures can be one of the major contributions to uncertainties in measured temperatures above 600 °C. From one side, this insulation leakage causes a shunt across the sensor resistor leading to systematic errors in the measured temperatures, which are, in principle, characteristics of each thermometer. On the other side at high temperatures, degradation of the insulation materials used in the furnaces provokes the decrease of the furnace insulation impedance between the thermometer and the furnace what also causes a systematic temperature measurement error.

Two high temperature standard platinum resistance thermometers have been used to measured this effects in different heat pipes and three zone furnaces. One of them was open circuited by cutting the sensing element end allowing the measurement of the resistance between the two pairs of current-potential leads.

Two different instruments were used to measure the ac and dc insulation between leads and thermometer-to-furnace. To measure dc isolation a teraohmeter was used. In order to better reproduce the effect of insulation on the readings of thermometer resistance, the method measures the variation of measurements of a high resistance standard shunted by it.

To determine ac shunt admittance, a LCR meter is used to perform the measurements at different frequencies. It allows to measure not only leakage resistance but shunt capacitance too.

In this paper the different results are shown and the effect on temperature readings estimated.

IMPROVED HIGH-TEMPERATURE STANDARD PLATINUM RESISTANCE THERMOMETER

M. Zhao, D. Chen, M. Newman, R. Ding
Fluke Corporation-Hart Scientific Division, American Fork, U.S.A.
E-mail:mzhao@fluke.com

High-temperature standard platinum resistance thermometers (HTSPRTs) are used to interpolate temperature in the range from 661.323°C to 961.78°C on the International Temperature Scale of 1990 (ITS-90). In the past 20 years, the reliability and performance of HTSPRTs has been troubled by instability and short circuits in the sensor coil. During the EUROMET regional key comparison, EUROMET.T-K4 found that the selected HTSPRTs were less stable than desired, and one of the BIPM HTSPRTs failed due to a short circuit in the sensor. Similar problems were reported in other laboratories such as NIMJ. There has been discussion about the possibility of replacing the HTSPRT with the Au/Pt thermocouple as the ITS interpolating instrument above the freezing point of aluminum. To continue using the HTSPRT as the ITS interpolating instrument, it is critical that its performance be improved.

In response to requests from NMIs, the HTSPRT was redesigned three years ago. The objective was to prevent short circuits, improve stability, and raise the upper temperature limit to the freezing point of copper (1084.62°C). The most important change was an improvement in the structure of the sensor support. The strip support was replaced by a new specially designed cross support. The structure and design of the new HTSPRT are briefly discussed in this paper.

Since the new HTSPRT was developed three years ago, no sensor coil short circuits have been reported. Thorough testing has shown that the HTSPRT can operate well at temperatures as high as 1085°C. Results from tests of a group of thermometers are presented in this paper. The test included long-term drifts of the thermometers at the triple point of water and freezing point of silver during a period of a few hundred hours operation at 1085 °C, short-term stability of R(tpw) and W(Ag) in a period of five days, and thermal cycles between room temperature and 1085 °C. The test results show that the thermometer performance is improved and the new HTSPRT can operate up to the freezing point of copper (1084.62°C).

THE IMMERSION CHARACTERISTICS OF INDUSTRIAL PRTS

D. R. White and C. L. Jongenelen
MSL, Lower Hutt, New Zealand
E-mail (*corresponding author*): *r.white@irl.cri.nz*

Immersion effects are one of the most significant sources of error in the use of industrial platinum resistance thermometers (PRT) for laboratory applications. The error arises from the flow of heat along the body of the thermometer in response to the temperature difference between ambient and the system under test. The error in the temperature measurement is conventionally modelled by an exponential function of immersion depth for which the $1/e$ characteristic is assumed to be similar to the diameter of the thermometer. This leads to rules of thumb for usage of the thermometer, such as "for 1% accuracy, immerse the sensor to a minimum of 5 diameters", or "for 0.01% accuracy, immerse the sensor to a minimum of 10 diameters". Common practice is to demonstrate the absence of a significant error by changing immersion a few centimetres and noting that the reading does not change.

This paper investigates the validity of the simple exponential model and shows that in the oil baths typically used for calibration, the effective diameter of the thermometer can be very much larger than the physical diameter of the probe. The increased diameter appears to arise from the non-turbulent layer of oil, the boundary layer, attached to the thermometer. The paper reports measurements of the immersion characteristics of industrial PRT probes of different diameter, in different fluids, and at different temperatures.

Fixed Points I

Monday
16:50 to 18:10
Emerald 2
Session Chairman: Richard Rusby

IMPROVED INITIATION TECHNIQUE FOR THE METAL FIXED POINTS

D. R. White and R. S. Mason
MSL, Lower Hutt, New Zealand
E-mail (*corresponding author*): *r.white@irl.cri.nz*

Traditionally, the metal fixed points of indium, tin, zinc, aluminum and silver are realized with two solid-liquid interfaces surrounding the thermometer. One interface is formed on the inner surface of the outside wall of the crucible and is initiated by lowering the furnace temperature or by withdrawing the cell from the furnace. The inner interface is formed around the thermometer well and is initiated by the insertion of cool rods or thermometers into the well. For some time we have been concerned that cooling of most of the liquid, which is a consequence of the conventional method for initiating the outside interface, encourages the formation of dendrites that may provide 'thermal short circuits' between the inner and outer interfaces. Similarly, insufficient cooling of the inner interface, due to the high thermal conductivity of the liquid metal and the proximity of the outer interface, may not allow the formation of a complete inner interface without the formation of excessive solid.

Working on the principle that one complete solid-liquid interface is better than two incomplete interfaces we have investigated initiating techniques for establishing a single highquality interface around the thermometer well. We have found such realizations offer a faster recovery from recalescence, higher realized temperatures, and longer duration plateaus than achieved with the conventional procedures. Thermal-coupling testing, both by the furnaceoscillation method and immersion profiling, shows that the thermometer-furnace coupling is undetectable with the new procedures.

FURTHER FINDINGS ON IMPURITY PRECIPITATION IN METAL FIXED POINTS

M. Fahr[1], S. Rudtsch[2]
[1] *NRC, Ottawa, Canada*
[2] *PTB, Berlin, Germany*
E-mail (corresponding author): martin.fahr@nrc-cnrc.gv.ca

Impurities are the source of the largest contribution to the uncertainty budgets in realizing the metal-fixed-point reference temperatures of the ITS-90. Currently, the smallest uncertainties are achievable by accounting for the influences of the impurities independently, as given by the product of the amount of dissolved impurity and the specific influence of that impurity on the phase-transition temperature. The former has to be determined through chemical analysis, which can deliver very detailed information about the concentrations of each element in the metal and even its spatial distribution. However, at these low concentrations, chemical analysis is unable to determine whether or not the impurities are dissolved, because the known methods cannot distinguish between a (solid) solution and a (solid) suspension. The latter, the sensitivity coefficient for each impurity, has to be determined by doping experiments until sufficient public data is available, an activity that several NMIs are currently engaged in.

A few of these experiments have indicated that some impurities do not dissolve in the fixed-point metals. Based on this observation, we recently published a paper presenting thermodynamic calculations that predict the formation and precipitation of impurity oxides in metal fixed points. Here we provide further, more distinct proof, based on doping experiments with other material combinations, illustrate the precipitation process on a microscopic level, and present new calculations considering other chemical reactions that may lead to precipitation of even more elements.

NUMERICAL MODELLING OF HEAT FLUX IN FIXED-POINT CELLS DUE TO THE HIDROSTATIC-HEAD EFFECT

V. Batagelj, J. Bojkovski and J. Drnovšek
MIRS/UL-FE/LMK, Ljubljana, Slovenia
E-mail (corresponding author): valentin.batagelj@fe.uni-lj.si

Perturbing heat exchange and the resulting temperature gradients are one of the major sources of uncertainty in the calibration of standard platinum resistance thermometers in fixed-point cells. The commonly accepted tests for the estimation of this uncertainty source are the measurement of the thermometer immersion profile, the use of different bushings, changing the temperature of the thermal enclosure and numerical modeling.

The perturbing heat exchange is closely related to the depth of immersion of thermometer inside the fixed-point cell. It is a common belief that if immersion depth is sufficient, the effects of the perturbing heat exchanges are negligible. In other words, if the immersion depth was infinite, the associated uncertainty contribution would be zero. This hypothesis would be completely valid only, if the temperature field inside the thermometer well would be homogeneous and there would consequently be no heat flux. However, the temperature gradient is inherently present in every fixed-point cell due to the hydrostatic-head effect and inevitably there is a resulting heat flux. Since thermal resistances within the cell's thermometer well are not axially symmetric, the temperature field is always distorted.

In order to get a better insight into this phenomenon, a numerical model based on finite difference method was developed. The model takes into account only heat conduction and not also heat convection and radiation, as these would only further add to the complexity of the problem. The cell is modeled as infinitely long, so there is no influence of the thermal enclosure. The temperatures at the point of phase transition ideally follow the hydrostatic-head curve. The model allows the simulation of the measurement of the thermometer immersion profile and of the use of different bushings.

The results of the modeling showed that there is an inherent difference between the temperature measured by the thermometer sensor and the temperature at the point of the phase transition, even if the immersion depth was infinite. Nevertheless, the thermometer would still almost perfectly follow the immersion-profile curve. The only exception is near the bottom of the thermometer well, where a small deviation from the immersion profile was observed. This is in agreement with previously presented results, where this behavior was noticed, but never satisfactory explained.

A SOLIDIFICATION APPROACH TO CORRECTING FOR THE EFFECT OF IMPURITIES IN FIXED POINTS

Z. Malik[1], J. D. Hunt[2], H. Davies[3], P. D. Lee[1], D. Lowe[3], P. Petchpong[3], P. N. Quested[3]
[1] *Imperial College, London, UK*
[2] *Oxford University, Oxford, UK*
[3] *National Physical Laboratory, Teddington, UK*
E-mail (corresponding author): zohaib.malik@imperial.ac.uk

The uncertainty in the realisation of a fixed-point cell is usually dominated by the difference from the true freeze temperature due to impurities in the material. If the amounts of impurities are known and thermodynamic data is available the Sum of Individual Estimate (SIE) method can be used to apply a correction. If only the amount of impurities is known an uncertainty can be estimated by the Overall Maximum Estimate (OME) method in which it is assumed that all the solutes are insoluble in the solid. Both these methods rely on an accurate assay of composition. The measurement uncertainty of this assay is usually significant leading to uncertainties comparable to the correction. Since higher purity materials are unlikely to become readily available there is a drive to develop a method to correct the temperature that is independent of the precise chemical analysis.

The departure of the solidification temperature from the ideal value for dilute solutions is given by the sum of the product of the liquidus slopes m_i and the compositions C_i of the binary system of each impurity in the fixed-point material. During freezing the liquid composition changes according to the relative solubility of the impurity in the solid and liquid phases (the distribution coefficient) and so the solidification temperature changes, affecting the shape of the freeze curve. Given complete mixing in the liquid the shape of the freeze curve can be determined analytically – the Scheil equation. Under suitable freeze conditions this can predict the shape of a freeze curve and can be used to determine the liquidus temperature. Alternatively if there is only one main impurity the composition and fixed point temperature may be predicted.

Two methods have been developed for fitting experimental curves. In the first a program has been written to give the best fit for the four terms in the Scheil equation. In the other a graphical method is used that assumes all the distibution coefficients are small (i.e. the impurities are rejected from the solid and build up in the liquid).

The methods were first performed on simulated freeze curves derived using a thermodynamic database (MTData) for a range of mixtures with varying levels of impurities that have been found in tin. Even when there is more than one solute it has been found that fitting a Scheil curve for one solute gives a reasonable estimate of the fixed point temperature. Tests on actual fixed

points with both single and combined impurities were investigated. Finally some cases where the technique does not work so well, possibly due to non-uniformity in the freeze, have also been considered.

The preliminary results are promising demonstrating that in a number of cases the parameters can be determined by fitting to the freezing curve without any prior knowledge of impurity level. This could allow correction of the freezing temperature based solely on freeze curve shape with potentially reduced uncertainty.

Interlaboratory Comparisons I

Monday
16:50 to 18:10
Adria
Session Chairman: Jintao Zhang

INVESTIGATION OF THE EQUIVALENCE OF NATIONAL DEW-POINT TEMPERATURE REALIZATIONS IN THE RANGE -50 °C TO +20 °C

M. Heinonen[1], M. Anagnostou[2], S. Bell[3], M. Stevens[3], R. Benyon[4], R. A. Bergerud[5], J. Bojkovski[6], R. Bosma[7], J. Nielsen[7], N. Böse[8], P. Cromwell[9], A. K. Dogan[10], S. Aytekin[10], A. Uytun[10], V. Fernicola[11], K. Flakiewicz[12], B. Blanquart[13], D. Hudoklin[6], P. Jacobson[14], A. Kentved[15], I. Lóio[16], G. Mamontov[17], A. Masarykova[18], H. Mitter[19], R. Mnguni[20], J. Otych[21], A. Steiner[22], N. S. Zsófia[23], D. Zvizdic[24]

[1] Centre for Metrology and Accreditation (MIKES), Espoo, Finland
[2] Hellenic Institute of Metrology (EIM), Thessaloniki, Greece
[3] National Physical Laboratory (NPL), Teddington, UK
[4] Instituto Nacional de Técnica Aeroespacial (INTA), Madrid, Spain
[5] Norwegian Metrology Service (JV), Oslo, Norway
[6] MIRS/UL-FE/LMK, Ljubljana, Slovenia
[7] VSL, Netherlands, Delft, Netherlands
[8] Physikalisch-Technische Bundesanstalt (PTB),Braunschweig, Germany
[9] National Metrology Laboratory (NML), Dublin, Ireland
[10] Ulusal Metroloji Enstitüsü (UME), Turkey
[11] Istituto Nazionale di Ricerca Metrologica (INRiM), Turin, Italy
[12] Central Office of Measures (GUM), Warszawa, Poland
[13] Centre Technique des Industries Aérauliques et Thermiques (CETIAT), Lyon, France
[14] Swedish National Testing and Research Institute (SP), Borås, Sweden
[15] Danish Electronics Light & Acoustics (DELTA), Hoersholm, Denmark
[16] Instituto Português da Qualidade (IPQ), Lissabon, Portugal
[17] D. I. Mendeleyev Institute for Metrology (VNIIM), St. Petersburg,Russia
[18] Slovak Institute of Metrology (SMU),Bratislava, Slovakia
[19] BEV / E+E Elektronik (BEV), Engerwitzdorf, Austria
[20] National Metrology Institute of South Africa (NMISA),Pretoria, South Africa
[21] Czech Metrology Institute (CMI), Brno, Czech Republic
[22] Swiss Federal Office of Metrology (METAS), Bern, Switzerland
[23] Hungarian Trade Licensing Office (MKEH), Budapest, Hungary
[24] University of Zagreb, FSB-LPM, Zagreb, Croatia
E-mail (corresponding author):martti.heinonen@mikes.fi

To support worldwide recognition, national metrology institutes (NMI) regularly investigate the equivalence of the national realizations of SI units by arranging key comparisons. The national standards of a region are compared to each other in regional key comparisons arranged by the relevant regional metrology organization (RMO), e.g. EURAMET in Europe. Regional key comparison results from different regions are linked to each other through CIPM key comparisons.

In the field of humidity quantities, the first CIPM key comparison, CCT-K6, is at its end. The corresponding European regional key comparison, EUROMET.T-K6,

was completed in early 2008, about four years after the starting initial measurements in the project. In total, 24 NMIs from different countries took part in the comparison. This number includes 22 EUROMET countries, and Russia and South Africa. Both CCT-K6 and EURAMET.T-K6 cover the dew-point temperature range from –50 °C to +20 °C.

The comparison was carried out in three parallel loops with two specially manufactured precision chilled mirror hygrometers as transfer standards in each loop. Each NMI calibrated two instruments out of six that were used as transfer standards. In addition, each pair of loops was interlinked through two NMIs that calibrated the transfer standards of the two loops. Before starting the comparison, the pilot laboratories of the loops run initial tests for the instruments of their loop. They also carried out an additional set of measurements after all other participants of their loops for long-term stability monitoring. This comparison scheme reduced the sensitivity of the final results to the quality of results at individual NMIs and split the workload of piloting and establishing links among 9 NMIs.

There are two PRTs embedded in the mirror of each transfer standard. One of them has direct access for electrical resistance measurement which was used as the primary output. For each nominal measurement point, all participants reported the resistance and corresponding NMI's reference dew-point temperature value with assigned uncertainties. Also, other supporting information was reported to enable a comprehensive analysis of results.

This paper describes the applied comparison and analysis method and summarizes the results. It is shown that the standard uncertainty due to the long-term instability was smaller than 0.008 °C in all loops. The standard uncertainties due to links between the loops were found smaller than 0.025 °C at –50 °C and 0.010 °C elsewhere. Conclusions on the equivalence of the dew-point temperature standards are drawn on the basis of calculated bilateral degrees of equivalence and deviations from the EURAMET comparison reference values (ERV).

Taking into account 16 different primary dew-point realizations and 8 secondary realizations, the results demonstrate the equivalence of large number of laboratories at an uncertainty level that is better than achieved in other multilateral comparisons so far in the humidity field.

EURAMET.T-K7 KEY COMPARISON OF WATER TRIPLE POINT CELLS

A. Peruzzi[1], R. Bosma[1], O. Kerkhof[1], P. Rosenkranz[2], M. D. Del Campo Maldonado[3], M. Smid[4], J. Nielsen[5], M. Anagnostou[6], D. Zvizdic[7], E. Grudnewicz[8], M. Nedea[9], D. M. Neagu[9], P. Steur[10], E. Filipe[11], I. Lobo[11], I. Antonsen[12], E. Renaot[13], M. Heinonen[14], T. Weckstrom[14], J. Bojkovski[15], E. Turzo-Andras[16], S. Nemeth[16], M. White[17], E. Tegeler[18], M. Dobre[19], S. Duris[20], A. Kartal Dogan[21], A. Uytun[21], V. Augevicius[22], A. Pauzha[22], A. Pokhodun[23], S. Simic[24]

[1] *VSL, Delft, Netherlands*
[2] *BEV, Wien, Austria*
[3] *CEM, Madrid, Spain*
[4] *CMI, Brno, Czech Republic*
[5] *DTI, Aarhus, Denmark*
[6] *EIM, Thessaloniki, Greece*
[7] *FSB-LPM, Zagreb, Croatia*
[8] *GUM, Warsaw, Poland*
[9] *INM, Bucharest, Romania*
[10] *INRiM, Torino, Italy*
[11] *IPQ, Caparica, Portugal*
[12] *JV, Kjeller, Norway*
[13] *LNE-INM/CNAM, Paris, France*
[14] *MIKES, Espoo, Finland*
[15] *MIRS/UL-FE/LMK, Ljubljana, Slovenia*
[16] *MKEH, Budapest, Hungary*
[17] *NSAI NML, Dublin, Ireland*
[18] *PTB, Berlin, Germany*
[19] *SMD, Bruxelles, Belgium*
[20] *SMU, Bratislava, Slovakia*
[21] *UME, Kocaeli, Turkey*
[22] *VMT/PFI, Vilnius, Lithuania*
[23] *VNIIM, S. Petersburg, Russia*
[24] *ZMDM, Beograd, Serbia*
E-mail (corresponding author): a.peruzzi@vsl.nl

EURAMET.T-K7 key comparison of water triple point cells was carried out in 2006-2007 as EURAMET regional extension of the corresponding CIPM key comparison CCT-K7 (2002-2004). A total of 24 National Metrology Institutes (NMIs) took part in the EURAMET.T-K7 comparison, 9 of which had participated in CCT-K7 as well. The results of EURAMET.T-K7 comparison were linked to the results of CCT-K7 comparison and published in the BIPM KCDB (Key Comparison DataBase) in January 2009.

Two facts complicate the interpretation of the cross equivalence of CCT-K7 and EURAMET.TK7:

1. In CCT-K7 only three participants applied a definition of the triple point temperature based on ocean water (VSMOW, Vienna Standard Mean

Ocean Water). All the other participants, even those having sufficient information, did not apply corrections for the deviation of the isotopic composition of the cell water from VSMOW.

2. In the time window between the end of CCT-K7 and the beginning of EURAMET.T-K7, the clarification of the definition of the kelvin (2005) prompted many NMIs to redefine their national reference for the water triple point temperature, which resulted in a modified equivalence in EURAMET.T-K7 with respect to CCT-K7 for those NMIs that changed their national reference in between the two comparisons.

In this paper we will:

I. Summarize the results of EURAMET.T-K7 comparison
II. Calculate the best estimate of the SI unit from the results of EURAMET.T-K7 and compare it to the best estimate of the SI unit
III. Evaluate the impact of chemical impurities on the water triple point temperature.

ROUND ROBIN TEST OF HEAT FLUX SENSORS

E. Turzo-Andras[1], H. Blokland[2], U. Hammerschmidt[3], S. Rudtsch[3,5], C. Stacey[4], C. Krös[6], T. Magyarlaki[1], S. Nemeth[1]
[1] *MKEH, Budapest, Hungary*
[2] *TNO, Delft, Netherlands*
[3] *PTB, Braunschweig and Berlin, Germany*
[4] *NPL, Teddington, United Kingdom*
[5] *BTU, Cottbus, Germany*
[6] *SABS, Pretoria, South Africa*
E-mail (corresponding author): thurzo-a@mkeh.hu

The first intercomparison on density of heat flow rate measurements has been organized by MKEH (Hungarian Trade Licensing Office, Metrology Division) within the framework of EUROMET (project No. 426). The objective of this round robin test was to improve the accuracy in the realisation of a density of heat flow rate scale up to 100 $W \cdot m^{-2}$.

Two types of heat flux plate sensors differing in their size and here denoted as “NL” and “HU” were circulated among five (NL) and two (HU) partner laboratories, respectively. Each one of the six partners calibrated the sensors using its individual heat source, a guarded hot plate or a heat flow meter apparatus. Measurements were performed at nominal temperatures of 20 °C and 30 °C.

The report compares all the results of the round robin test and gives the mutual differences among the partners. Representative results are presented in the figures 1 to 3. Figure 1 shows four calibration curves of sensor NL at 20°C, each for three nominal densities of heat flow rate of 10, 50 and 100 $W \cdot m^{-2}$. In Figures 2 and 3, the individual results of the partners on both sensors, NL and HU, are depicted for the same temperature of 30 °C and density of 50 $W \cdot m^{-2}$.

The mutual deviations in the partner’s results are well within their measurement uncertainties.

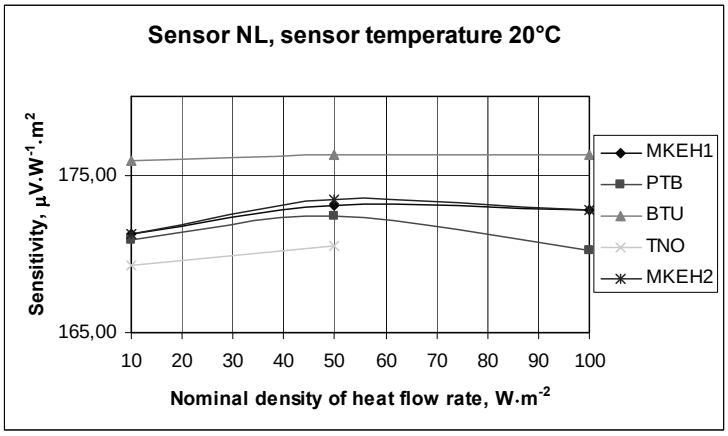

Fig. 1

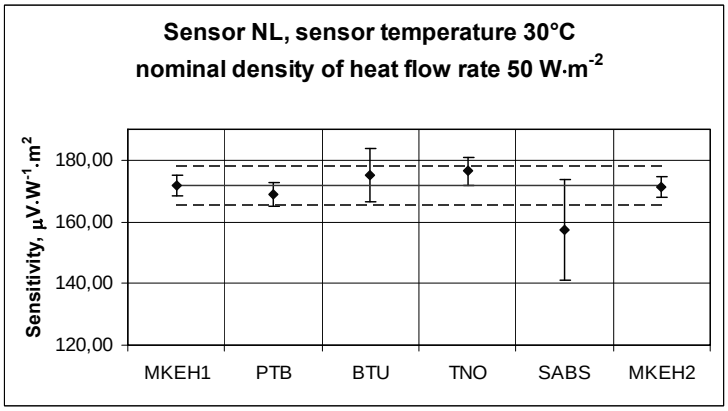

Fig. 2

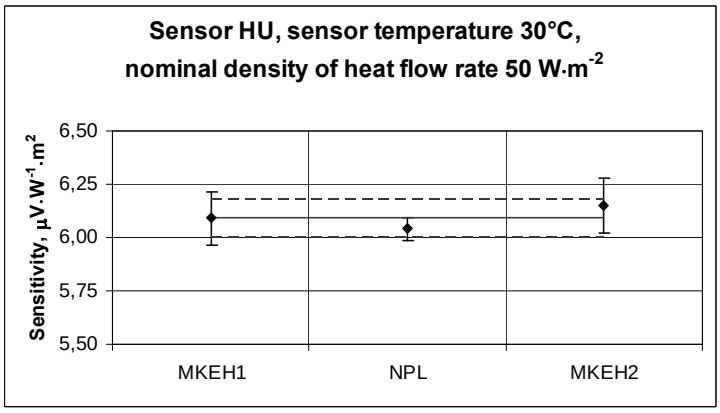

Fig. 3

PILOT STUDY: COMPARISON OF RADIANCE TEMPERATURE IN THE RANGE OF -40 °C TO 300 °C BETWEEN NIST, NPL, PTB AND NRC

S. Mekhontsev[1], V. Khromchenko[1], L. Hanssen[1], H. McEvoy[2],
R. Underwood[2], J. Hollandt[3], B. Gutschwager[3], K. Hill[4], D. Woods[4]
[1] *NIST, Gaithersburg, USA*
[2] *NPL, Teddington, United Kingdom*
[3] *PTB, Berlin, Germany*
[4] *NRC, Ottawa, Canada*
E-mail (corresponding author): snm@nist.gov

A number of national metrology institutes realize and maintain radiance temperature scales at near-ambient temperatures in the spectral bands from 3 to 5 µm and 8 to 14 µm with claimed total uncertainty at the level of 25 - 50 mK. In recent years, certain applications, such as climate monitoring, have generated additional interest in establishing and confirming such levels of uncertainties at the NMI level.
The main objective of the reported pilot study was to establish uniformity and equivalence of local scales of radiance temperature at temperatures from –40 °C to 300 °C. Additional goals included: (1) study of the long term stability of the transfer standard sources, (2) evaluation of uncertainties for SSE (size-of-source effect) characterization of pyrometers, (3) compare results obtained using the delivered transfer pyrometer as a comparator vs. those obtained using regular comparators (scale transfer techniques) of the participants, and (4) compare IR spectral radiance scales in the spectral range of 3 µm to 14 µm.
All participants performed radiance temperature calibration of two variable transfer standard sources (TSS) and one transfer standard pyrometer (TSP), with the laboratory-pilot performing measurements twice, once at the beginning and again at the end of the comparisons. Selected TSS were portable thermal radiation sources with high reproducibility of contact temperature measurements and alignment. In the temperature range of their operation (15 °C to 300 °C) they were serving as primary transfer standards because of potentially significant long term drift of the pyrometer. Each TSS was equipped with a 40 mm diameter room temperature aperture and is intended to be compared with a primary standard blackbody of each participant with an identical aperture size to reduce possible errors due to SSE. The TSP was intended to serve, at most temperatures, primarily as a comparator between the participant reference BB sources and transfer standard sources. For sub-ambient temperatures, where TSS could not be used without purge or window because of moisture condensation, the TSP served as the transfer standard.
Results of the comparisons have shown a good agreement, in most cases not exceeding combined uncertainty of the respective participants. Along with analysis of the obtained results, report contains discussion of further cooperation needs and possibilities in the area of radiation thermometry and spectroradiometry at the near-ambient temperatures.

Plenary session III

Tuesday
9:00 to 9:30
Emerald
Session Chairman: Jeremy Lovell-Smith

TEOS-10: A NEW INTERNATIONAL STANDARD FOR SEAWATER, ICE, FLUID WATER, AND HUMID AIR

R. Feistel
IOW, Warnemuende, Germany
E-mail (corresponding author): rainer.feistel@io-warnemuende.de

Very accurate empirical thermodynamic potential functions are available for fluid water, ice, seawater and humid air covering wide ranges of temperature and pressure conditions, including those of the terrestrial hydrosphere and atmosphere. They permit the consistent computation of all equilibrium properties as, for example, required for coupled atmosphere-ocean models or the analysis of observational or experimental data (http://www.ocean-sci.net/6/91/2010/os-6-91-2010.html). These potential functions are formulated as international standards endorsed (humid air in preparation for 2010) by the International Association for the Properties of Water and Steam (IAPWS, http://www.iapws.org).

A new seawater standard referred to as the International Thermodynamic Equation of Seawater 2010 (TEOS-10) was adopted in June 2009 by UNESCO/IOC, as recommended by the SCOR/IAPSO Working Group 127 (WG127) on Thermodynamics and Equation of State of Seawater (http://www.teos-10.org). In force since January 2010, TEOS-10 has replaced the previous 1980 International Equation of State of Seawater, EOS-80, by a combination of IAPWS documents. To support the adoption process, WG127 developed a comprehensive source code library for the thermodynamic properties of liquid water, water vapour, ice, seawater and humid air, referred to as the Sea-Ice-Air (SIA) library.

The modular architecture of TEOS-10 and its supporting SIA library is guided by the axiomatic principles of consistency, independence and completeness of the primary standard. Virtually unlimited sets of property equations for the geophysical substances and their mutual phase equilibria can be derived from the primary standard by rigorous thermodynamic relations and their numerical implementation, without additional empirical constants or correlation equations. Among the derived quantities are, e.g., Gibbs functions for composite systems such as sea ice, or enthalpy potentials for the convenient description of adiabatic oceanic or atmospheric processes. Linked via a well-defined interface, the algorithms for the derived properties are unaffected by future partial update, substitution or extension of the primary standard. Similarly, the set of derived quantities is easily and arbitrarily extendible to provide additional required properties without modification of the primary standard. Alternatively, the modular semi-order structure in combination with the mutual independence of the primary standard modules permits the separation of smaller, self-contained sub-libraries from TEOS-10 that are restricted to special tasks. For speed-critical applications, tailored equations or look-up tables can be compiled with high accuracy for arbitrary parameter combinations.

Plenary session IV

Tuesday
9:30 to 10:00
Emerald
Session Chairman: Richard Davis

PRACTICAL IMPLEMENTATION OF THE MISE-EN-PRATIQUE FOR THE DEFINITION OF THE KELVIN ABOVE THE SILVER POINT

G. Machin[1], P. Bloembergen[2], K. Anhalt[3], J. Hartmann[3], M. Sadli[4], P. Saunders[5], E. Woolliams[1], Y. Yamada[6], H. Yoon[7]

[1]*National Physical Laboratory (NPL), Teddington, Middlesex, UK*

[2]*National Institute of Metrology (NIM), Beijing 100013, China*

[3]*Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587, Berlin, Germany*

[4] *Institut National de Métrologie (LNE-INM/Cnam), 61 rue de Landy, Paris 93210, France*

[5] *Measurement Standards Laboratory of New Zealand (MSL), Industrial Research Ltd, P. O. Box 31-310, Lower Hutt, New Zealand*

[6] *National Metrology Institute of Japan (NMIJ), AIST, AIST Tsukuba Central 3, 1-1-1 Umezono, Tsukuba, Ibaraki, 305-8563, Japan*

[7] *National Institute of Standards and Technology (NIST), Gaithersburg, MD, USA*

E-mail: graham.machin@npl.co.uk

In April 2006 the Consultative Committee of Thermometry (CCT) adopteded the *miseen-pratique for the definition of the kelvin (MeP-K)* with the purpose of providing information necessary for a practical realisation of temperature according to the *Système International d'Unités* (SI). The MeP-K included direct realisation of thermodynamic temperature, independent of the defined scale, ITS-90, where the primary thermometry had advanced sufficiently to confer benefit. This was thought apply at very low and very high temperatures.

A task group of CCT-WG5 (radiation thermometry) was established in May 2008 to examine the different methods of direct mesaurements of thermodynamic temperatures above the silver point, and, in particular, to write text for the MeP-K for this temperature range. Although the ITS-90 definition is experimentally simple to implement other methods could convey significant advantages, either in terms of lower uncertainties or improved robustness/security of realisation. The methods are; a) absolute radiometry linked to appropriate radiometric units, b) use of high temperature fixed points (HTFPs) as defining fixed points or as radiometric reference standards for a thermodynamic realisation/dissemination of temperature.

The WG5 task group has prepared text for the MeP-K that describes the possible methods of high temperature realisation: absolute radiometry (n=0, T), ITS-90 (n=1, T or T_{90}), or by high temperature fixed points (n=2, 3 and 3+, T or T_{90}), n referring to the number of fixed points that are used in the realisation of temperature. This paper gives a summary of the WG5 task group text for the MeP-K, with an outline of the proposed approaches, including a discussion of the advantages and disadvantages of the above possible methods. This paper serves as an introduction to linked papers by the task group members on absolute radiometry methods, comparative uncertainty studies and interpolation schemes.

Plenary session V

Tuesday
10:00 to 10:30
Emerald
Session Chairman: Mickey Haynes

IN SITU SILICON WAFER SURFACE TEMPERATURE MEASUREMENTS UTILIZING POLARIZED LIGHT

Y. Yamada, J. Ishii
National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology (NMIJ, AIST), Tsukuba, Ibaraki, Japan
E-mail: y.yamada@aist.go.jp

As the miniaturization of transistors in integrated circuits progresses, new refined processes for silicon device fabrication are introduced, which require improved temperature measurement and control. Such temperature measurement set new challenges which cannot be overcome by simple extension of conventional temperature measurement techniques. For instance, reduction in process time does not allow thermal equilibrium of the silicon wafer under process with the surrounding to take place, or even within the wafer itself, so that direct temperature measurement of the device side surface of the wafer is inevitable. However, such measurements conventionally could not be performed effectively due to difficulties of unknown and varying emissivities and intense background radiation. Poor accessibility of the wafer top surface in the process chamber is also a problem. In this paper, we present results of two recent research developments made at the NMIJ in collaboration with industrial partners to accomplish such semiconductor process in situ temperature measurements: one on the millisecond annealing process by flash lamp (Flash Lamp +Annealing, FLA), and the other on the plasma etching process. In both processes, the wafer top surface faces either the heating lamp source unit or the plasma source electrode, and the wafer surface can only be viewed from an extremely inclined angle from the side ports. Such an arrangement on the other hand makes utilization of polarized light effective. Automatic emissivity compensating radiation thermometry based on the classical polaradiometry[1]) was applied to measurement in the FLA. Measurements have been made both off-line and on-line to evaluate the performance of the automatic emissivity compensated radiation thermometry[2]). For temperature monitoring in the plasma etching process, a novel technique was devised combining thermoreflectance method and ellipsometry[3]). The low sensitivity of the change in reflectance with change in temperature has been an obstacle in applying thermoreflectance technique in industrial environments. The proposed method accomplishes robust monitoring of variation in temperature by high-sensitivity detection of the variation in the difference of the relative changes in reflectances at two orthogonal polarizations by a rotating analyzer.

[1] C. Tingwaldt: Z. Metallkunde 51 (1960) pp.116-119
[2] Y. Yamada, T. Aoyama, H. Chino, K. Hiraka, J. Ishii, S. Kadoya, S. Kato, H. Kiyama, H. Kondo, T. Kuroiwa, K. Matsuo, T. Owada, T. Shimizu, T. Yokomori: Extended Abstracts of SSDM 2009 (JSAP, 2009) 58
[3] Y. Yamada, J. Ishii, A. Nakaoka and Y. Mizojiri, in these abstracts

Fixed points - M-C eutectics II

Tuesday
11:30 to 13:10
Emerald 1
Session Chairman: Jürgen Hartmann

THE USE OF DIFFERENT FURNACES TO STUDY REPEATABILITY AND REPRODUCIBILITY OF THREE PD-C CELLS

M. Battuello, M. Florio, F. Girard
INRIM, Turin, Italy
E-mail (corresponding author): m.battuello@inrim.it

Three different Pd-C eutectic fixed-point cells were prepared and investigated at INRIM. Several tens of phase transition runs were done and recorded with both a Si-based radiation thermometer at 950 nm and a precision InGaAs-based thermometer at 1.6 µm. Two of the cells were of the same design with an inner volume of 10 cm^3. The third one was smaller with an inner useful volume of 3.6 cm^3. All the three cells were filled with palladium powder 99.995% in purity and graphite powder 6N pure. Repeatability and stability of the inflection point were investigated over a period of one year. The noticeably different external dimensions of the two cells, namely 110 mm and 40 mm in length, respectively, allowed the influence of longitudinal temperature distribution to be investigated. To the purpose, two different furnaces, a single-zone with SiC heaters and a three-zone with MoSi2 heaters were used. Different operative conditions, namely temperature steps, melting rate, longitudinal temperature distributions and position of cells within the furnace, were tested to investigate the reproducibility of the cells. Effects on the duration and shape of the plateaux were also studied. This paper gives details of the measurement set-up and analyses the melting plateaux obtained with the different conditions.

SPACE EXPERIMENTS IN RUSSIA AIMED AT DEVELOPING ONBOARD LOW-TEMPERATURE FIXED-POINT BLACKBODY

V. Sapritsky[1], A. Burdakin[1], B. Khlevnoy[1], S. Ogarev[1], A. Panfilov[1], A. Puzanov[1], V. Rakov[1], M. Samoylov[1], V. Krutikov[2], M. Kudashkina[3], V. Levtov[4]

[1] *All-Russian Research Institute for Optical and Physical Measurement (VNIIOFI), Moscow, Russia*

[2] *Federal Agency on Technical Regulating and Metrology, Moscow, Russia*

[3] *Rocket and Space Corporation «Energia», Russia*

[4] *Central Scientific Research Institute for Machine Building (TsNIIMASH) , Korolev, Moscow region, Russia*

Small-size fixed points based on melting phase transition of Gallium eutectics were designed as a first step in a chain of works aimed at developing super-stable onboard fixed-point standard radiation sources for in-flight calibration of satellite optical radiometric instruments within thermal IR spectral range [1, 2].

The shape of the eutectic melting plateau, which is just the reference mark for determining sequential metrological characteristics of the fixed point, obviously depends on its “thermal history” [1]. One can improve the eutectic fixed point’s characteristics by varying parameters of the preceding (preparatory) melt-freeze cycles. The structure of the liquid eutectic just a bit over the freezing temperature is especially important to obtain optimal crystal structure of the alloy that ensures maximally isothermal and prolonged working melting plateau [3, 4].

All the stated above justifies a need for investigation of the eutectic fixed-point devices under zero-gravity conditions including on-orbit calibration procedures. Studies of Pb-Sn eutectic system on board the ISS revealed strong impact of zero-gravity conditions on the resultant alloy structure [5]. Now preparation for space experiment with the fixed points based on Gallium eutectics is progressing at VNIIOFI.

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NEW METHOD OF FILLING OF HIGH TEMPERATURE FIXED POINT CELLS BASED ON METAL-CARBON EUTECTICS/PERITECTICS

B.B. Khlevnoy, I.A. Grigoryeva and N.A. Ibragimov
VNIIOFI, Moscow, Russia
E-mail (B.B. Khlevnoy): khlevnoy-m4r@vniiofi.ru

High temperature fixed point (HTFP) cells based on metal-carbon (metal carbide – carbon) eutectic and peritectic alloys (M(C)-C) is developed at VNIIOFI for several years side by side with NMIJ, NPL, LNE-INM/Cnam and some other institutes. Experience shows that quality of a cell depends on its filling method as well as on a crucible design.

The method of HTFP cells filling that was initially used at VNIIOFI was the following: the crucible was filled with a mixture of metal and carbon powders, then melted in vertical position, then filled and melted again and so on until the crucible was full. The same method with some variations was used at all other institutes. This method has some disadvantages: 1) metal reacts not only with carbon powder of the mixture but also with graphite walls of the crucible; as a result locally the walls become thinner 2) foundry cavities tend to appear inside the eutectic ingot; 3) porous crust is formed on the top of the alloy; 4) numerous iterations of filling and melting are necessary to fill the cell completely. In [1] it was shown that the number of iterations could be reduced by means of using a C/C-cloth extension. However, an additional problem appears with such an extension: 5) the extension can also be eaten by pure metal, so holes are often appear in the cloth, and sometimes eutectic alloy sticks on the cloth surface and partly blocks the channel.

A new method of filling was suggested and tested to avoid the mentioned disadvantages. In this method the powder mixture is introduced not directly into the crucible, but into an additional container located just above the crucible. The eutectic forms and melts inside the container and the already molten eutectic drops through a small hole in the bottom of the container and fills the crucible. The crucible design was changed a bit as well: a graphite sleeve with one layer of cloth in between the sleeve and the wall were used in instead of the cloth extension.

The method was successfully tested for Re-C and WC-C. The most disadvantages were overcome. However, an overfilling was a problem for the new method. To solve this problem the method was combined with the filling finalizing technique developed at LNE-INM/Cnam [2].

The paper will present the details and results of the developed method in comparing with the previous one.

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ON THE IMPURITY PARAMETERS ASSOCIATED WITH IMPURITIES DETECTED IN THE EUTECTICS CO-C, PT-C AND RE-C

P. Bloembergen, D. Wei, W. Tie-jun
National Institute of Metrology, Beijing 100013, China
E-mail (corresponding author): p.bloembergen@xs4all.nl

Under the auspices of WG-5 of the CCT the eutectics Co-C, Pt-C and Re-C, with eutectic temperatures of 1597 K, 2011 K, 2747 K, respectively, are presently investigated for their suitability to serve as reference points for dissemination of T (and T_{90}) within the context of the '*Mise en pratique of the definition of the kelvin*' (*MeP*-K) at high temperature: *MeP*-K-HT.
In the *MeP*-K-HT the reference temperatures of reference points are defined for the transition temperature of the reference material in its pure state. From direct comparisons of the eutectic transition temperatures of samples of a given eutectic originating from different sources it appeared that impurities, even at trace levels, can significantly affect the transition temperature. For the correction of the effect of impurities the knowledge of the liquidus slopes m_i associated with the impurities X_i involved is required; in practice a full uncertainty budget presupposes at least the feasibility of estimating the maximum $k$max to the distribution of the distribution coefficients k_i.
Impurity parameters m_i and k_i have been calculated for a range of metallic impurities X_i for Co-C, Pt-C and Re-C by means of the software package Thermo-Calc within the ternary phase spaces Co-C-X_i, Pt-C-X_i and Re-C-X_i. The choice of the impurities is based upon a selection out of the results of impurity analyses performed for a representative set of samples for each of the systems in study. The analyses performed were GDMS or ICP-mass. As reported not all of the selected impurities were represented in the databases SSOL4 and SSUB4 associated with Thermo-Calc. Especially for the eutectic Re-C these databases need to be supplied with new data derived from dedicated experiments.
For the three systems in study it will be checked whether the impurity parameters fulfill the van't Hoff equation -valid in the ideal-solution limit- up to the impurity levels as measured in practice, or higher. Plots of the parameters against the atomic number Z_i of the impurities will be presented. These might enable indirect derivation of the parameters m_i and k_i by interpolation in accordance with the van't Hoff equation for those impurities for which no direct information is available. In each case the correction T_{TR}–$T_{liq}(0)$ will be presented for representative sets of impurities, where T_{TR} and $T_{liq}(0)$ refer to the transition temperature of the pure system and to the liquidus temperature in the limit of zero growth rate of the solid phase during solidification, respectively. Uncertainty estimates based upon the sum of individual estimates (SIE) and the overall maximum estimate (OME) are included. Finally, doping studies will be suggested for those impurities which are preponderant, but for which as yet the required information is lacking in the databases.

Fixed Points II

Tuesday
11:30 to 13:10
Emerald 2
Session Chairman: Yves Hermier

IMPROVED PROTOCOL FOR THE REALISATION OF THE TRIPLE POINTS OF CRYOGENIC GASES AS TEMPERATURE FIXED POINTS

L. Wolber, B. Fellmuth
Physikalisch-Technische Bundesanstalt (PTB), Berlin, Germany
E-mail (corresponding author): lutz.wolber@ptb.de

Because of the poor thermal conductivity of the liquefied gases, precise calibrations at their triple points require to take into account the temperature difference $\Delta T = P_u R_{cs}$ caused by the heat load P_u flowing across some effective thermal resistance Rcs from the cell wall to the solid-liquid interface acting as a heat sink.

As an improvement of the previous protocol [B. Fellmuth, D. Berger, L. Wolber, 1999, Proceedings of TEMPMEKO '99, pp. 233-8], the thermal model is now generalized to include the coexistence of different melting temperatures and various thermal resistances. Further uncertainty components are considered for

- correction of the self-heating by the usual √2-variation of the measuring current,
- observed melting-curve shape,
- extrapolation to the liquidus point.

It will be described in detail how P_u can be obtained from the drift of the cell outside the melting range. The optimum choice of P_u with respect to the uncertainty estimates is discussed. Precise determinations of heat capacity and heat of fusion also require P_u to be included in the energy budget.

As a basis for the optimum realisation of the triple points, an explanation is given

- for the extremely long time constants of the thermal recovery after heat pulses of the intermittent heating,
- for the equilibrium condition,
- for the convergence of the measured melting curves towards the liquidus point, and
- why the observed thermal resistance Rcs often remains small and constant over a wide range of the fraction of sample melted.

Finally a detailed uncertainty budget is established for the melting temperature at the liquidus point that is used as the best approximation of the triple-point temperature for a given fixed-point sample. The budget is based on deducing the final result as weighted mean of many data obtained from several melting experiments performed under different conditions as well as applying at least three resistance thermometers and both ac and dc resistance-measurement techniques.

FURTHER PROGRESS TOWARD THE DETERMINATION OF T_{tp}-x(^{22}Ne)

F. Pavese[1], P. P. M. Steur[1], S. Valkiers[2], T. Nakano[3], H. Sakurai[3], O. Tamura[3], A. Peruzzi[4], J. van Geel[4], L. Lipinski[5], A. Szmyrka-Grzebyk[5], W. L. Tew[6], K. D. Hill[7], A. D. Steele[7], Y. Hermier[8], F. Sparasci[8]

[1] *Istituto Nazionale di Ricerca Metrologica (INRIM), Torino, Italy*
[2] *Institute for Reference Materials and Measurements (JRC-IRMM), Geel, Belgium*
[3] *National Metrology Institute of Japan (NMIJ), AIST, Ibaraki, Japan*
[4] *VSL Dutch Metrology Institute (VSL), Delft, The Netherlands*
[5] *Instytut Niskich Temperatur i Badan Strukturalnych (INTiBS), Wroclaw, Poland*
[6] *National Institute of Standards and Technology, (NIST), Gaithersburg, USA*
[7] *National Research Council of Canada (NRC), Ottawa, Canada*
[8] *Institut National de Métrologie (LNE-INM), Paris, France*
E-mail (corresponding author): f.pavese@inrim.it

Following the finalisation of the work performed in order to establish the *T-x* relationship for hydrogen isotopes (*Metrologia* **42** (2005) 171–193), already adopted into the ITS-90 definition by CIPM in 2005, and the start of exploration of the similar problem for neon isotopes (*Analytical Chemistry* **77** (2005) 5076–5080), further progress has been achieved with respect to the 2007 still preliminary results (*Int. J. Thermophysics* **29** (2008) 57–66). The paper will summarise the to-date advancement in understanding that was obtained from further work on three basic aspects of the problem:

(a) new isotopic assays, to gain more confidence on the actual accuracy of the measured values of the isotopic composition x, namely for ^{22}Ne, whose large concentration (close to 10%) places special problems;

(b) new thermal measurements, to determine with decreased uncertainty the differences of the values of the liquidus point T_{tp} between samples of natural neon, in order to check for: (i) the feasibility of obtaining a value of the slope $dT_{tp}/dx(^{22}\mathrm{Ne})$ with an accuracy sufficient for the purpose and, (ii) the possible occurrence of isotopic fractionation during the process of sealing the samples in the cells;

(c) new thermal measurements on the pure isotopes ^{20}Ne and ^{22}Ne, to obtain their T_{tp} with an accuracy much higher than the one associated with the previous old literature data, to check the alternate possibility to define the T_{tp}-x(^{22}Ne) relationship for neon isotopes with the help of pure isotopes.

The recently collected information is becoming sufficient to state that it will eventually be possible to fulfill within 2010 the goal of recommending to CCT solutions for the correction, with an accuracy fitting the purpose, of neon isotopic effect on T_{tp}, to be included in a future revision of the Technical Annexe to the 'mises en pratique" of the kelvin.

THE TRIPLE POINT TEMPERATURES OF ^{20}Ne AND ^{22}Ne

K. D. Hill and M. Fahr
National Research Council of Canada, Ottawa, Canada, K1A 0R6
E-mail (corresponding author): ken.hill@nrc-cnrc.gc.ca

Interest in the triple points of neon isotopes has recently arisen as a result of the multi-institute project to understand how variations in the isotopic composition of natural neon influence the triple point temperature of a particular sample. Given the challenges in determining both the relative isotopic concentrations, particularly of ^{22}Ne relative to ^{20}Ne, and the temperature differences among cells filled from different gas sources with sufficiently low uncertainty to determine the sensitivity coefficient with adequate confidence, modeling of the isotopic influence becomes an attractive alternative approach to correct the triple points of 'natural' neon samples to a common composition. This modeling would start with, among other things, knowledge of the triple point temperatures of ^{20}Ne and ^{22}Ne. In addition, the triple points of these pure neon isotopes have utility in their own right as secondary reference temperatures on the ITS-90, and one or both of these could replace 'natural' neon in a revised International Temperature Scale or approximation of the ITS.

Aside from recent measurements at INRiM, the neon isotope triple point temperatures (on the IPTS-68) reported by Furukawa, Kemp, and Sakurai in various publications from 1972 to 1986 predate the ITS-90, and so there is utility in contemporary measurements (on the ITS-90) with gas samples of recent production. At NRC, we find the triple point of ^{20}Ne to be 24.5424 K (13.68 mK below the triple point of 'natural' neon) and that of ^{22}Ne to be 24.6887 K (132.63 mK above the triple point of 'natural' neon) with our current sample of 'natural' neon defining a temperature of 24.5561 K.

DEVELOPMENT OF NEW GALLIUM CELLS AT THE SMU IN THE FRAME OF THE PROJECT EURAMET 732

J. Ranostaj[1], S. Ďuriš[1], M. Kaskötö[1], R. Knorová[1], R. Palenčár[2]
[1] *SMU, Bratislava, Slovak Republic*
[2] *STU, Bratislava, Slovak Republic*
E-mail (Juraj Ranostaj): ranostaj@smu.gov.sk

The article informs about the progress in the work on development of new gallium fixed point cells at the SMU in the frame of the project EURAMET 732. It provides information about the designs of new cells, used materials, cleaning and filling the cells with high purity gallium. Article presents also the results of performed experiments.

SMU built and studied three gallium fixed point cells of different designs. Cell Ga01 consists of PTFE crucible placed into the silica glass outer envelope, cell Ga02 consists of PTFE crucible placed into the metallic outer envelope and the cell Ga03 is all PTFE design. All cells are filled with gallium of purity 99.99999%. In consideration of practical realization of gallium point at the SMU, all cells are designed for using in the stirred liquid bath. To enable setting and measuring of the inner pressure inside the cell, cells Ga01 and Ga02 are equipped with flange for connection to the vacuum and gas handling system. Performed experiments were oriented on the study of several factors affecting the temperature of gallium phase transition, e.i. effect of previous freezing, temperature of thermal enclosure (liquid bath), presence of the inner solid-liquid interface. Article presents also the results of the comparison of new cells with national reference gallium cell.

Water vapour pressure data

Tuesday
11:30 to 13:10
Adria
Session Chairman: Stephanie Bell

SPECTROSCOPIC MEASUREMENT OF THE VAPOR PRESSURE OF ICE

K. Bielska[1], D. K. Havey[2], G. E. Scace[2], D. Lisak[1], J. T. Hodges[2]
[1] *Instytut Fizyki, Uniwersytet Mikolaja Kopernika, ul. Grudziadzka 5/7, 87-100 Torun, Poland*
[2] *National Institute of Standards and Technology, 100 Bureau Drive, Gaithersburg, Maryland 20899, USA*
E-mail (corresponding author): gregory.scace@nist.gov

We present a laser absorption technique to measure the vapor pressure of ice. This method is referenced to the triple point state of water and uses frequency-stabilized cavity ring-down spectroscopy to probe four rotation-vibration transitions of $H_2{}^{16}O$ at wave numbers near 7180 cm. Laser measurements are made at the output of a temperature-regulated standard humidity generator which contains ice. The dynamic range of the technique is extended by measuring the relative intensities of three weak/strong transition pairs at fixed ice temperature and humidity concentration. Our preliminary results agree with Wexler's ice vapor pressure correlation [A. Wexler, J.Res. NBS 81A, 5 (1977)] over the temperature range 0 °C to 70 °C to within 0.5%.

ON THE WAY TO THE DETERMINATION OF THE VAPOUR PRESSURE CURVE OF PURE WATER

S. Mokdad[1], E. Georgin[1], I. Mokbel[2], J. Jose[2], Y. Hermier[3], M. Himbert[3]
[1] LNE-CETIAT, Villeurbanne, France
[2] LSA UMR 5180 équipe THERMALI, Lyon, France
[3] Laboratoire commun de métrologie LNE-CNAM, Saint-Denis, France

The determination of the physical properties of pure water, especially of the vapor pressure curve of water, is one of the major issues identified by the Consultative Committee for Thermometry (CCT) of the International Committee for Weights and Measures (CIPM) to improve the accuracy of the national references in humidity. Actually most references consist in a humid air generator in which the dew/frost temperature is monitored by the temperature at which a thermodynamic equilibrium can be achieved between the partial pressure $e_w'(p,T)$ of water vapor, contained in moist air, and a sheet of water, either liquid or solid. The partial pressure is linked to the vapour saturation pressure of water via a so called “enhancement factor”. Progress in the knowledge of the saturation curve will directly improve the accuracy of the calibrations in humidity. One of the mostly used reference formula has been established in 1990 by Sonntag, using the original data of Goff and Gratch (1948) and some data from the equation set up by Wexler (1975). It covers the range from –100 °C to +100 °C and is in agreement with the International Temperature Scale (ITS-90). No other experimental data covering this whole range has been obtained since 1975.

The present work presents measurements of the static pressure and the temperature of pure water in a closed, thermometrically controlled vessel. The temperature range lies from –40 °C to +100 °C, and the pressure range from 12,8 Pa to 101419 Pa.

Temperature and pressure sensors were calibrated against the references in use at the LNE-CETIAT laboratories. An automatic data acquisition program was required and was realized with LabVIEW© software. The influence of several parameters has been studied, such as the presence of dissolved gases, the presence of impurities, the effect of thermal transpiration … Water has been analysed by capillary electrophoresis. The results have been compared with previous experimental and theoretical vapor pressure equations.

A first uncertainly budget has been set up. The final uncertainty takes into account components coming from the pressure measurement, from the temperature measurement and from different environmental error sources such as transpiration and hydrostatic effects.

INVESTIGATION OF THE SATURATED WATER VAPOR PARTIAL PRESSURE IN PRESENCE OF OTHER GASES

K. Flakiewicz, R. Jarosz
Central Office of Measures/GUM, Poland
E-mail (corresponding author):humidity.kf@gum.gov.pl

The saturated water vapor partial pressure in pure phase over plane surface of water or ice may be calculated with the number of computer equations worked out for this application. The experimental basis for these is not so large and rather old. In the environment and in industrial applications water vapor exists the most in presence of air and other gases. The saturated water vapor partial pressure in such mixtures in comparison to the pure phase depends on the total pressure, temperature and kind of gases. Because of different possible values of those quantities their influence on the saturation pressure in pure phase is characterized with enhancement factor $f(P,T,x)$:

$$f(P,T,x) = \frac{p_{smix}(P,T,x)}{p_{spure}(T)} \tag{1}$$

Where: p_{smix} - saturated water vapor partial pressure in mixture with other gases over plane surface of water or ice,
p_{spure} saturated water vapor partial pressure in pure phase over plane surface of water or ice,
P – total pressure,
T – temperature,
x - characteristics of mixture.

In the paper is described the method, original draft of the project of the experimental setup and results of measurements p_{smix} in the temperature range (-50 ÷ +50) °C for the total pressure range 0,1 to 2 MPa for compressed air and Nitrogen. Then the enhancement factor is calculated. There are compared the experimental results and values calculated with the accepted datum. The uncertainty estimation for the experimental results obtained in this work is presented. The differences of chosen results obtained with the calculation, these and former measurement are analysed.

INVESTIGATION OF THE SATURATED WATER VAPOR PARTIAL PRESSURE IN PURE PHASE

K. Flakiewicz
Central Office of Measures/GUM, Poland
E-mail (corresponding author):humidity.kf@gum.gov.pl

The number of computer worked out equations for saturated water vapor partial pressure is very large, but support of the experimental results for these equations seems to be not very strong. The most of measurements was carried out a many years ago with instruments which were available in that time and uncertainty analysis was also not adequate to contemporary requirements. That was the reason for drawing up the method, instruments and devices for measurement of this quantity in the wide range temperature. The subject of these measurements is water vapor partial pressure in pure phase over plane surface of water/ice.

In the paper is described the method, original draft of the project of the experimental setup and results of measurements in the temperature range from –50 ºC to +50 ºC. The measurements are going on and extension to the measuring range from –85 ºC to +100 ºC may be soon completed. There are compared the experimental results and values calculated with the commonly accepted equations. The uncertainty estimation for the experimental results obtained in this work is presented. The differences of chosen results obtained with the calculation, this and former experiment are analysed. As the uncertainty targets were assumed the following criteria:
- below 0,01% for water in the range from 0 ºC to 100 °C,
- below 0,6% for overcooled water in the range from –50 ºC to 0 °C,
- below 1,0% for ice in the range down to –100 °C.

AN INVESTIGATION OF THE ICE-WATER VAPOUR EQUILIBRIUM ALONG THE SUBLIMATION LINE

V. Fernicola, L.Rosso, L. Iacomini, M. Giovannini
INRIM Istituto Nazionale di Ricerca Metrologica, Torino, Italy
E-mail (corresponding author): v.fernicola@inrim.it

The thermodynamic properties of water at low temperature play an important role in atmospheric processes where its accurate knowledge impacts on the models of our climate system. Vapour pressure data are also needed for chemical physics and metrology applications. Humidity standards benefit from any improvement in the measurement of the ice-water vapour equilibrium curve. The uncertainty associated with the correlation between the saturation vapour pressure and the temperature affects the calculation of the vapour mole ratio of humid gas mixtures. At present, the stated relative uncertainty in the temperature range from 220 K to 273 K is lower than 0.25%.

Saturation vapour pressure, in the pure phase, in equilibrium over ice has been investigated by several scientists with the first measurements dating back to forties by Goff and Gratch. The latest, most accurate, measurements were probably carried out by Marti and Mauersberger. Most of the known experimental determinations of saturation water vapour were carried out with direct static measurements.

At INRIM, a set up was assembled in order to make a preliminary investigation of the water vapour-ice equilibrium along the sublimation line. The measurements covered the temperature range from 0 °C to –50 °C, corresponding to saturation vapour pressures from 611 Pa to ca. 4 Pa. A static method was used to perform the measurements in a small gold-coated cell kept in a liquid bath at a constant temperature to better than 2 mK. The sample cell was connected to a manifold were two capacitive diaphragm pressure gauges were used for pressure measurements.

The paper presents the experimental apparatus and gives details about the sample preparation in order to preserve its purity. Preliminary results compare favourably with published literature data and enabled the validation of the measurement method. The applied corrections, the uncertainty sources and the way to improve them is discussed in the work.

POSTER SESSION II

Humidity and Moisture Standards

Tuesday
10:45 to 11:30
and
14:15 to 15:00
Foyer

STUDY ON ICE CRYSTAL FORMATION IN A FROST POINT GENERATOR

H. Kajastie and M. Heinonen
Centre for Metrology and Accreditation (MIKES), Espoo, Finland
E-mail (corresponding author): heikki.kajastie@mikes.fi

MIKES performs calibrations for dew-point meters in the range from –80°C to +84 °C. For the purpose, MIKES has developed several primary dew/frost-point generators. Recently, increasing emphasis has been given to very low dew point temperatures. Technical solutions are searched for the range down to –100 °C. Demands for the ultra low dew-point temperatures is coming from industry.

In low temperature saturators, small ice crystals in the sample gas may cause significant errors in dew-point measurements. When melting and evaporating in the sample tube between the saturator and a hygrometer, the water vapour pressure changes causing an error in dew-point reference value. Ice crystals in gas stream may form as a result of supersaturation when gas is entering the saturator or they can come off from condensed phase on the walls of the saturator.

Because of small size and light mass, airborne small crystals can pass through even very complicated flow path in the saturator. In the work reported in this paper, we study the ice particle formation using two methods: At first ice formation on inner wall surface of circular tubes is visually observed using an endoscope. The tip of the endoscope can be moved along the tube to observe the ice layer at different locations. Tubes with different surface quality will be studied. In the second method, ice crystals are collected applying centrifugal force. Gas is forced to circular flow and ice crystals are collected in a container. Initial studies are carried out at about –40 °C. The methods will then be applied to lower temperatures.

THE RUSSIAN NATIONAL STANDARD OF GASES HUMIDITY AND TRACEABILITY SYSTEM OF HUMIDITY MEASUREMENTS

A. F. Vinge, N. I. Dubovikov, O. A. Podmurnaya, I. A. Sokov,
N. P. Skryabikov
VNIIFTRI East-Siberian Branch, Irkutsk, Russian Federation
E-mail: dep15@niiftri.irk.ru

The Russian national gas humidity standard of gases has been modernized in order to increase the number of reproducible quantities of humidity (relative humidity, dew/frost-point temperature, mole fraction) and to extend the humidity and operating temperature range. The basis of the standard comprises two humidity generators with working range from 5 °C to 90 °C and from –60 °C to 15 °C respectively. The common working range (from 5 °C to 15 °C) allows comparison of the generators.

The generators use the two-pressure method to generate humid gas defined in terms of relative humidity (from 5% to 98%) and the phase equilibrium method to generate humid gas defined in terms of the vapour mole fraction (from 0,6 ppm to $700 \cdot 10^3$ ppm) and dew/frost-point temperature (from –79 °C to 90 °C). The expanded uncertainties in relative humidity is no more 0.2%, in vapour mole fraction 1.2%, in dew/frost-point temperature 0.12 °C.

The ordinary hygrometers are traceable to the national primary standard in accordance with the state hierarchical chain for measuring means of gas humidity. The state hierarchical chain consists of three branches: first branch for measuring means of relative humidity, second branch for measuring means of mole fraction and third branch for measuring means of dew/frostpoint temperature. Each branch can be represented as the scheme: primary standard - secondary standard - working standard - ordinary hygrometer. Method of direct measurements, method of direct comparison, method of comparison with a comparator or method of indirect measurements can be used for calibration and verification of working standards and ordinary hygrometers and for their traceability to the primary standard.

HUMID AIR GENERATOR IN LOW-RANGE: QUALIFICATION

E. Georgin[1], B. Blanquart[2]
[1] LNE-CETIAT, Villeurbanne, France
[2] Blanquart, Nancy, France
E-mail (corresponding author): eric.georgin@cetiat.fr

The LNE-CETIAT has developped a humid air generator dedicated to the low range of dew/frost-point, that is to say until –80°C. This generator has been involved, recently, in comparisons which covered low range (regional comparaison 715), medium range (regional key comparison 621-K6) and high range (regional comparaison 717).

In previous works, a presentation has been done concerning the comparison between this new generator and the generator in use at LNE-CETIAT to calibrate instruments from industry and research laboratories.

This paper presents a synthesis of the technical improvements which have been brought to this generator during the last years.

These improvements deal with different aspects of the humid air generator. Concerning temperature equilibrium and measurement, a new thermostatic bath has been selected for its better speed to reach the equilibrium, with an increased temperature stability. The efficiency of the saturator due to the heat exchanger has been also qualified. Concerning the flow motion, a piston pump substitutes a vane pump. Concerning the data acquisition, all the intruments are now monitored; including pressure fluctuations in the bench and environnement parameters such as pressure, temperature and humidity.

We tried to quantify the effects of these improvements on the bench, and to express them in terms of uncertainties, when it is possible.

IMPROVEMENT OF THE NMISA TWO-PRESSURE HUMIDITY GENERATOR

M. R. Mnguni, D. Jonker, H. G. Liedberg
NMISA, Pretoria, South Africa
E-mail (corresponding author): rmnguni@nmisa.org

The humidity laboratory of NMISA is in the process of developing a two-pressure humidity generator to operate over the range –25 °Cfp to 60 °Cdp, to replace unsaturated salt solutions as the reference standard for the calibration of relative humidity meters. Traceability for relative humidity calibrations will then be obtained from local temperature and pressure measurement standards, and greater confidence will be obtained in relative humidity calibrations at temperatures far from ambient.

During initial testing of this generator, it was found that both temperature equilibration and saturation of the air appeared to be less than ideal. For this reason, a pre-saturator (with independent temperature control) has now been added to the system. This paper describes the changes made to the generator, and the more comprehensive evaluation of saturation efficiency that these changes made possible.

HUMID AIR GENERATOR AT LNE-CETIAT: MODELLING ACTIVITY

E. Georgin[1], J. Ouazzani[2]
[1] *LNE-CETIAT, Villeurbanne, France*
[2] *ARCOFLUID, Talence, France*
E-mail (corresponding author): eric.georgin@cetiat.fr

The humid air generator developed at LNE-CETIAT has been designed in 2001 and underwent different upgrade in order to reduce uncertainties in: temperature measurement, homogeneity and stability temperature improvement, airflow motion. In spite of its great return of experience and so as to go deeper in its global understanding concerning this apparatus, the LNE-CETIAT started a numerical work. The numerical simulations have been done with the general finite volume code PHOENICS with customization in order to tackle the evaporation/condensation process. Depending on the aim of the simulation, steady cases and unsteady cases have been solved in 2D and 3D polar coordinates.

Basically, the first step of this numerical work has been to solve the coupled conservation equations for mass, momentum and energy applied to a cylindrical geometry. In addition as the humid air may be considered like a binary mixture between dry air and water steam, a specie equation is also solved in order to describe the advection-diffusion of water steam. At last, as the main purpose deals with humidity, fundamental scalar equations of humidity are also computed. The system studied is a water pool inside a cylinder, the main part of heat and mass exchange appear at the interface level between air and water. Thus an original work has been done about the boundary conditions at the interface to account for the evaporation and/or condensation occurring at this interface in terms of mass and energy sinks and/or sources.

The first part of this work has been to validate the case dealing with pure diffusion model. Then, transition, due to thermal steps, is studied and the equilibrium state, reached by the system, is compared to humid air table.

The second part of this work has been to add a circular inlet and outlet airflow. Thus a parametric study has been done about, temperature difference, airflow, concentration and geometry.

HOW CONTINUOUS-WAVE CAVITY RING-DOWN SPECTROSCOPY AND DERIVATIVE METHODS ARE TRANSFORMING METROLOGY LABORATORY PRACTICE

L. Bergson
Tiger Optics, Warrington, Pennsylvania, USA
lbergson@tigeroptics.com

Today, from Thailand to Finland to South Africa, national metrology labs are increasingly relying upon Continuous Wave Cavity Ring-Down Spectroscopy (CW-CRDS) and its offshoots to achieve fast, precise, sensitive, and highly efficient measurements. The widespread acceptance and growing use of these techniques will be discussed by one who has witnessed this transformation first-hand. Going back to 2001, when the Dutch NMi purchased the first commercial CW CRDS analyzer through to the present, this advanced spectroscopic method has been adopted both for research and as a transfer-standard for gas calibration. Starting with ultrahigh-purity, low parts-per-billion measurement, the technology is increasingly sought for higher parts-per-million applications, taking maximum advantage of its exceptional dynamic range. In so doing, it has begun to replace traditional means, such as chilled mirror, electrochemical, and oscillating crystal analyzers. Its ease-of-use and low cost-of-ownership favor such expanded use.

LOW COST DEW-POINT TEMPERATURE STANDARD FOR THE FIXED POINT 0 °C

R. Jarosz, K. Flakiewicz
Central Office of Measures (GUM), Warszawa, Poland
E-mail (corresponding author): rjarosz@op.pl

Calibration of the standard dew point hygrometers with chilled mirror is realized mainly on the measuring stand S01 in Humidity Laboratory of GUM. This measuring stand contains: dew point generators DPG1 and DPG2, thermostats, resistant bridge, multimeters, PRT sensors, standard hygrometers. Range of calibration is about from –80 °C to +95 °C what is covered whole measurement market requirements of hygrometers. The best measuring capability on this stand is about 0,03 °C. Standard measuring setup is very expensive and calibration for chosen frost or dew point temperature is rather long time process. Time period between calibration for standard hygrometers is about 13 months. To assure quality of calibration results is needed suitable standard. Low cost dew-point temperature standard for the fixed point 0 °C is used to check hygrometers between calibration and helps to check the current status of instrument. The best for easy and reliable periodical checks is dew point temperature standard for the nominal value 0 °C, which was designed and worked out by original conception in Humidity Laboratory. The source of reference value 0 °C is used mixture of water and ice. The air stream is circulated in close system. Where excess of water stay in saturator (heat –exchanger). Saturator is constructed as coil pipe made of stainless steel and immersed in mixture water and ice. By means of fast check saturator standard we can control the drifts, stability and easy detect the most of possible damages of hygrometers. This standard enables check hygrometers between calibrations quickly and systematically. It helps to fulfil requirements p. 5.9 norm PN-EN 17025 – assurance of quality results.

The designed and worked out prototype of fast check standard to periodical check does not require any other standard. This improves functionality of this device especially in situation when there is no possibility of using computer record of standard dew point temperature.

As the correct reference value 0 °C is used the temperature of freezing/melting point of water in atmospheric conditions. The check with this standard is the considerably simpler process, shorter and less expensive.

NEW SATURATOR FOR LOW RANGE DEW/FROST-POINT GENERATOR DPG-1 – PRIMARY HUMIDITY STANDARD IN GUM

K. Flakiewicz
Central Office of Measures/GUM, Poland
E-mail (corresponding author):humidity.kf@gum.gov.pl

The saturators of different construction are the basic components of different humidity generators. Dew-point generators DPG-1 and DPG-2 are the primary humidity standards in Humidity Laboratory in GUM. The chilled mirror (or SAW) dew-point hygrometers used as the secondary humidity standards are calibrated against these generators. These generators were used in European regional key comparison EUROMET.T-K6 (P-621) or supplementary EUROMET P-511.

The DPG-1 measuring range is below the ambient temperature, down to –80 °C (in practice even below –85 °C). It operates in closed circuit in recirculation mode. The reference saturator of DPG-1 is immersed in this dew-point range in ethanol bath. It consists of two elements and has the connection (with gasket) immersed in bath. It was observed that after few days of continuous work generator the mirror look in hygrometer being calibrated against the generator become less correct than at the beginning of measurement. It causes the increasing of the uncertainty. Finally the drift of the hygrometer become significant and is necessary to clean an dry the saturator and pipe system. The explanation of this effect is disturbing of the ice layer building on the mirror surface by diffusion of ethanol vapour to the measuring circuit across the connection immersed in ethanol bath. The changes are not important for the accuracy level of the industrial measurements but are significant for the calibration of the dew-point hygrometers used as the secondary standards. That was the reason for develop a new saturator without any junction in the bath. In the former saturator the sealed connection has been situated under the ethanol level.

The new saturator has been made due to own project and applied in the generator successfully. The stability of the hygrometer indications especially in the low humidity range (approx. < –0 °C) is better. In the paper is presented schematic draft of the generator DPG-1, the saturator and examples of comparisons of the results for former and new saturator.

CONSITENCY OF THE NATIONAL REALIZATION OF DEWPOINT TEMPERATURE USING STANDARD HUMIDITY GENERATORS

R. Benyon, T. Vicente
Instituto Nacional de Técnica Aeroespacial (INTA), Torrejón de Ardoz. Spain
E-mail (corresponding author): benyonpr@inta.es

The Spanish high range national humidity standard in the range –10 ºC to +95 ºC is maintained using two standard humidity generators of completely different saturator designs, measurement configurations and flow-rate capabilities. The generators used in this work are based on the "two-pressure" principle, that is, they rely on the saturation of a constant flow of air with respect to a plane surface of liquid water at elevated pressure and subsequent isothermal expansion. In the calculation of the reference dew-point temperature generated, knowledge of the vapour pressure formulation and enhancement factors is required. The second generator can also be operated in single pressure mode, eliminating the uncertainty introduced by these equations.

The first generator is an optimized commercially available generator used in the range –10 °C to +75 ºC. It has a maximum saturator pressure of 2.0 MPa and the saturator temperature range used is 4 ºC to 78 ºC. A chamber of internal dimensions (305 x 305 x 305) mm is located above the saturator. The gas flow rate used is in the range 30 to 100 l/min. Saturator (low and high) and chamber pressure measurement are made with precision quartz absolute pressure gauges with full-scale values of 0.31 MPa, 2.0 MPa and 0.15 MPa, respectively. Measurement of saturator temperature is performed with two metal-sheathed 25 Ω standard platinum resistance thermometers meeting the requirements of ITS-90 together with a precision AC resistance bridge, 25 Ω and 100 Ω standard resistors.

The second generator, was constructed using generator components supplied by BEV/E+E as part of a EURAMET project, and is operated in the range –10 ºC to 95 ºC with pressures up to 1.0 MPa and flow-rate of up to 2 l/min. Measurements of temperature and pressure are as in the first generator with the addition of two pressure transducers in a thermally controlled enclosure, connected in parallel with the other digital pressure gauges.

Although, in principle, the internal consistency of two-pressure generators can be checked by generating the same nominal values using different temperature and pressure combinations, one of the most robust tests of saturator efficiency is to compare different saturator designs using the same nominal temperature and pressure combinations (thus minimizing the influence of the SVP formulations and enhancement factors). In the work reported, three precision optical dew-point hygrometers have been used to investigate the level of agreement between the two generators in the overlapping range from –10 ºC to + 75 ºC in order to quantify the consistency of the two realizations that conform the Spanish National Humidity Standard. The measurement procedures adopted to minimize the effect of the influence quantities due to the transfer standards are described. The results are presented and discussed in the context of the declared CMCs in this range.

REVISION OF THE HIGH TEMPERATURE DEWPOINT GENERATOR IN USE AT VSL

R. Bosma and A. Peruzzi
VSL, Delft, the Netherlands
E-mail (corresponding author): rbosma@vsl.nl

At VSL a humidity generator was built in the early 1990s. This generator was of the recirculating two-temperature single-pressure type. The generator had a mayor revision in the early 2000s, when some critical components were changed. In 2007 the pre-saturator for the high temperature range of the generator was changed from piezo-electric type into a porous chalk oil filter. The high efficiency of this new pre-saturator made it possible to change the high temperature generator from re-circulation to single pass mode. As consequence of this modification, the flow through the saturator decreased from 75 $l \cdot min^{-1}$ to nominal 0.5 $l \cdot min^{-1}$ and the saturator was re-designed in order to increase turbulence at the lower flow rate. Experiments showed that the range of the new setup could be extended from 70 °C to 95 °C with a uncertainty of 0.08 °C (k=2) at the maximum temperature.

In this paper we will report on the revision of the high temperature dewpoint generator, with particular emphasis on:

- the design of the pre-saturator and saturator
- the saturation efficiency tests
- the uncertainty budget.

TOWARDS A STANDARD FOR MULTI-GAS AND MULTI-PRESSURE HUMIDITY CALIBRATIONS

M. Stevens, S. A. Bell, P. Carroll, S. Mortimer
National Physical Laboratory, Teddington, TW11 0LW, United Kingdom
E-mail (corresponding author): mark.stevens@npl.co.uk

The provision of primary dew-point standards for humidified air and nitrogen at atmospheric pressure is well established at many national institutes, and consistency has been demonstrated by international comparisons. Measurement traceability to these standards provides confidence in a vast number of air humidity measurements. However, hygrometers are used industrially in various non-air gases and at a wide range of pressures. Both the performance of hygrometers and the properties of humid gases are known to vary with gas species and pressure. There is a lack of both gas non-ideality data (water vapour enhancement factor) and instrument performance data that would allow humidity calibrations in air at 1 atmosphere to be applied more widely. Calibrations in air at 1 atmosphere only weakly underpin measurements in other gas conditions. To address this, a humidity calibration capability of wider scope is being developed at the UK National Physical Laboratory (NPL).

This paper describes the first steps at NPL towards facilities for the calibration of hygrometers in a range of gases, and at above-atmospheric pressures. This has been approached in stages. Initially, facilities have been developed separately for humidity generation at pressure and for humidity generation in non-air gases. For use at elevated pressure, a commercial two-pressure generator (Thunder Scientific Model 3900) has been modified to provide gas of defined dew- point (frost-point) at a range of pressures, operating initially in air. For providing humidified non-air gases, an initial capability has been developed using flow mixing of small amounts of saturated air with selected other gases (experimentally: methane, argon and helium, at approximately atmospheric pressure). Facility details and performance are presented for humidity generation in air at pressures up to 10 atmospheres, and in non-air gases at atmospheric pressure. Preliminary results are reported for hygrometers of different types compared under these conditions. Proposals are made for further development towards a combined multi-gas multi-pressure humidity calibration facility.

POSTER SESSION II

Thermophysical Properties

Tuesday
10:45 to 11:30
and
14:15 to 15:00
Foyer

THERMO-MECHANICAL ANALYSIS OF RUBBER COMPOUNDS FILLED BY CARBON NANOTUBES

P. Koštial[1], I. Ružiak[1], Z. Jančíková[1], R. Hrehuš[2], J. Kršková[2], R. Puchký[1]
[1] *VŠB-Technical university of Ostrava, Czech Republic*
[2] *Alexander Dubček University of Trenčín, Púchov, Slovak Republic*
E-mail (corresponding author): pavel.kostial@vsb.cz

Thermal and mechanical properties of solid state play an important role in the process of material optimization, mainly in the case of composites, concrete, polymers, steels, glass and ceramics, which are in the practice applied usually in non equilibrium states.

The presented work is devoted to the investigation of the single wall carbon nanotubes (*SWCNT*) influence on the thermal and mechanical properties of rubber blends.

The use of reinforcing fillers gives the material unique properties, a combination of high elasticity with high strength. The addition of the filler to the polymer can increase both modules *G* and *E* according to a hydrodynamic effect.

Materials under investigation were rubber blends filled by carbon black *(CB)* or mixture of *CB* with single wall carbon nanotubes *(0.6 wt. % of SWCNT)*. For all measurements two kinds of samples were prepared. One as standard, filled by *CB* only and second one filled by *SWCNT-CB* mixture.

The thermal parameters of rubber samples were measured on the basis of exponential model of cooling body. From the measured cooling curve of the sample after its reversible deformation (in the Hooke's law region) specific heat capacity c_p *[J/kgK],* thermal diffusivity a $[m^2/s]$ and thermal conductivity k *[W/m.K]* were measured. The Young's modulus *E* was also measured. The rising of all thermal and mechanical parameters under investigation *(c_p, a, k* and static Young's modulus *E)* was observed for rubber blends filled by mixture of *CB* and *SWCNT*.

The filler polymer interactions are determined by the structure of the filler. Blending of fillers with rubber leads also to a nonlinear phenomenon characterized, for instance, by Payne's effect caused by a filler-filler interaction.

To obtain an information about it, experimental results are completed by measurements of the complex Young's modulus E^* and loss factor $tg\delta$ as a functions of the temperature and frequency. Rising of both physical parameters under investigation caused by presence of *SWCNT* in the blend together with Payne's effect was observed.

REDUCTION OF SYSTEMATIC UNCERTAINTY OF A MEASURE OF THERMAL CONDUCTIVITY CAUSED BY THERMAL EXPANSION

N. A. Sokolov, A. N. Sokolov
D. I. Mendeleyev Institute for Metrology, St. Petersburg, Russia
E-mail (N. A. Sokolov): N.A.Sokolov@vniim.ru

In D. I. Mendeleyev Institute of Metrology, an essentially new way of reproduction of unit of thermal conductivity in any point of the set range was offered, the way is based on the fact that a certain system of solids in the certain conditions by means of operating influence gets any set thermal conductivity [1]. In other words, it has been possible to create a multiple-valued measure (MVM) of thermal conductivity [2] for the first time.

According to the offered way, MVM is made on the basis of Plexiglas with geometrical dimensions of 300 x 300 x 30 mm for reproduction of a unit within a range from 0.01 to 0.2 W/(mK) at temperature from 250 to 350 K.

Plexiglas is characterized by rather high coefficient of thermal expansion. Thermal conductivity measurement under ISO 8302 requires creation of a considerable gradient of the temperature reaching 40 K. Unaccounted influence coefficient of thermal expansion at measurement of thermal conductivity leads to occurrence of systematic uncertainty reaching 10 - 15%.

The uncertainty caused by the coefficient of thermal expansion also has dominant meaning in the state primary standard of thermal conductivity, but it is considerably reduced due to the special design. MVM consists of several thin flat plates. Their quantity is estimated by a method of computer modeling so that at the given mechanical loading deformation (bend) of MVM would be rather small.

MVM was included into the structure of a new state primary standard of D. I. Mendeleyev Institute for Metrology. This state primary standard participated in international comparison on thermal conductivity measurements of insulating materials. The analysis of first results shows that the obtained values are within a range of approximately 1.5% [3].

[1] N.A. Sokolov. Measurement Techniques, Vol. 49, No. 4, 2006, pp. 386-390
[2] N.A. Sokolov and A.N. Sokolov. Measurement Techniques, Vol. 52, No. 7, 2009, pp. 751-754
[3] B. Hay, L. Cortes, B. Doucey, J.-R. Filtz, U. Hammerschmidt, N. Sokolov, C. Stacey, R. Zarr, J. Zhang. 30-th International thermal conductivity conference. DEStech Publications, 2009, p. 79.

THERMOPHYSICAL PROPERTIES OF URANIUM BASED NIOBIUM AND ZIRCONIUM FROM 23 °C TO 200 °C

D. M. Camarano, F. L. Migliorini, E. H. C. Silva ,P. A. Grossi, W. B. Ferraz, J. B. Paula
CNEN/CDTN - LMPT, Belo Horizonte, Brazil
E-mail: dmc@cdtn.br

The thermal diffusivity of alloy uranium were measured by the laser flash method between room temperature and 200 °C, the thermal conductivity were determined by the measured thermal diffusivity and density, and the reported specific heat capacity. These properties were measured by using laser flash method. This method is conceptually simple, and presents advantages with respect to the various methods used in the past. In this method, the front face of a small wall-shaped sample receives a pulse of radiant energy coming from either a laser. The apparatus is specially designed to operate under conditions imposed by the requirement to measure the thermal diffusivity of nuclear fuels. Certain alloys of uranium containing the transition metals are of considerable interest in the development of nuclear fuels with low enrichment uranium for high-neutron-flux research reactors or low power reactor. In recent years several UZrNb alloys has been studied for many researchers and it has been known that additions of niobium and zirconium improve the properties of the uranium base alloys. Our work began with uranium of technical purity with about 500 ppm of metallic impurities, zirconium of purity 99,8% and niobium of purity of 99,9%. The purpose of this paper is to summarize thermophysical property measurements made on two alloys of uranium that have been studied by Centro de Desenvolvimento da Tecnologia Nuclear (CDTN) in a program of development of fuel for low power reactor. One alloy was of the nominal composition U4Zn6Nb while the other composition was nominally U3Zr9Nb. The results obtained by the original flash laser method and by the mathematical model developed by the laboratory were compared to the literature data. The GUM (Guide to the Expression of Uncertainty in Measurement) uncertainty framework and the adaptive Monte Carlo method were used to obtain the associated standard uncertainty with an estimate of the output quantities.
Keywords: Flash Method, Thermophysical Properties, Nuclear Fuels.

DESIGN AND CONSTRUCTION OF A SYSTEM TO MEASURE THERMAL CONDUCTIVITY OF SOLID CONDUCTOR MATERIALS

L. Lira-Cortés, González Rodríguez O. J.,Garcia Duarte S., Méndez-Lango E.
Laboratorio de Propiedades Termofísicas División de Termometría, Área Eléctrica, Centro Nacional de Metrología, Km 4,5 Carretera a Los Cués, Municipio El Marqués, Querétaro
E-mail (corresponding author): llira@cenam.mx,

It is important to know the values of thermophysics properties of materials for all the processes where there is energy exchange, particularly heat exchange.

For the design, operation, maintenance, simulation and optimization of mechanical systems and equipment, it is important to know the thermal conductivity of materials.

At CENAM we have developed a measurement system of thermal conductivity of metals with a secondary method; the method of cut bars. The measurement system operates on a steady state heat flow and uses a set of metal bars made from high purity copper as reference material.

The design criteria for this system was obtained from a parametric study about heat transfer for the bar conduction. In this study the parameters are the bar length and its diameter, and isolation thickness.

The system was constructed and used to measure the thermal conductivity of some materials from room temperature up to 300 °C range.

We present the design criteria, the main features of the system and the obtained values.

DEVELOPMENT OF A PRIMARY MEASUREMENT SYSTEM FOR MEASURING THERMAL CONDUCTIVITY OF FLUIDS

L. Lira-Cortés, Garcia Duarte S., Méndez-Lango E.
Laboratorio de Propiedades Termofísicas, División de Termometría, Área Eléctrica, Centro Nacional de Metrología, Km 4,5 Carretera a Los Cués, Municipio El Marqués, Querétaro
E-mail (corresponding author): llira@cenam.mx

Transient hot wire technique is suitable for measurement of heat conductivity of simple and complex fluids. This technique is used to measure thermal conductivity of gases, organic liquids, electrical non-conducting and conductor liquids. Best measurement capabilities ranges from ±0.3% to ±0.5%. It is used a infinite line heat source with zero heat capacity and infinite heat conductivity. The heat source is immersed in a dense and isotropic fluid whose thermal properties are independent from temperature changes being at thermal equilibrium with the source as initial condition and heat transfer from source to fluid is only conductive.

There is an ongoing project in the Centro Nacional de Metrología (CENAM) of Mexico to design a measuring system to implement this technique for both simple and complex fluids.

To measure heat conductivity of fluids with this technique it is required to measure the temperature change of the wire as function of time.

To avoid convection heat transfer the test last one second or less, in that time the measuring system should be able to collect enough data to evaluate the thermal conductivity. Thus, cell dimensions and measuring system is designed by using design criteria developed for this purpose.

We present the cell design and measuring system developed to implement this technique.

DYNAMIC MEASUREMENTS OF THE THERMAL CONDUCTIVITY OF INSULATORS

M. Bezjak[1] and D. Zvizdić[2]
[1] *INSTITUTE IGH, Zagreb,Croatia*
[2] *HMI/FSB-LPM, Zagreb,Croatia*
E-mail (corresponding author): mladen.bezjak@igh.hr

Measurements of thermal conductivity of insulators that are commonly used in civil engineering are as a rule being performed using the Pönsgen's guarded hot plate method under the steady state conditions. Achieving those steady state conditions is a time consuming and relatively expensive procedure. Therefore the application of a method that is less time consuming and less costly to common building insulating materials is of interest. The method should also have comparable accuracy and repeatability to presently used methods. One of such methods is a hot wire method (predominantly used for liquids, non-Newtonian fluids, plastic, semi-plastic and similar materials) it is a dynamic method that uses a very thin pure platinum wire that functions as a thermal source in combination with a temperature sensor that detects temperature transients.

This work describes the application of the hot wire method to most commonly used building thermal isolating materials. The dynamic hot wire measurements of thermal conductivity were performed on many building material samples. For the sake of comparison, the thermal conductivity of the samples made from the same materials was also tested using a stationary Pönsgen's guarded hot plate method. This work describes the comparison and evaluation of the measurement results obtained from both methods as well as the estimation of the pertinent measurement uncertainties. The results are presented in graphical and numerical form in tables and diagrams for each type of thermal insulator.

NEW MODELS FOR THE PULSE TRANSIENT TECHNIQUE

V. Boháč[1], P. Dieška[2] and Ľ. Kubičár[1]
[1] *Institute of Physics SAS, Dúbravská cesta 9, 845 11 Bratislava, Slovakia*
[2] *Department of Physics, Faculty of Electroengineering and Informatics, STU, Ilkovičova 3, Bratislava, Slovakia*
Email: bohac@savba.sk

Transient as well as stationary methods for the measurement of thermophysical parameters of the materials usually require large specimen size to get reasonable degree of measurement accuracy. The most of ideal models are assuming infinitively large specimen size that causes a problem in real experiment when the size of the specimen is limited due to any reasons. Use of ideal models in connection with finite specimen geometry causes decrease of the data accuracy. The reason is that additional effects like heat losses from the sample surface as well as heat capacity of the heat source influence the measured transient response. As a consequence the measured transient response is distorted in space and time. Then, the calculated values of thermophysical parameters are out of range of recommended values.

In this paper new models were introduced. The first one includes the effects of heat capacity of the heat source that is important at the measurement of materials having low thickness or low volume density. The second one accounts limited geometry of the specimen, the effects of the heat losses from the sample free surface as well as the thermal contact of the sample with the stabilized heat sink exchangers. These models match the real experimental arrangement. The second model estimates four parameters, e.g. thermal diffusivity, thermal conductivity specific heat and heat transfer coefficient from the sample surface from single measurement. The models were tested on several building construction materials as well as Polymethyl metacrylate (PMMA) of different geometry.

THE MOISTURE MONITORING AS A NEGATIVE FACTOR OF DETERIORATION OF MASSIVE ROCKS

V. Boháč[1], Ľ. Kubičár[1], M. Brček[2], J. Vlčko[2], V. Greif[2]
[1] *Institute of Physics, Slovak Academy of Sciences, Dúbravská cesta 9, 845 11 Bratislava*
[2] *Department of Engineering Geology, Comenius University in Bratislava, Faculty of Natural Sciences, Mlynská dolina G, 842 15 Bratislava, Slovakia*
Email: bohac@savba.sk

Moisture in porous materials may cause different thermal behaviors and may trigger different thermo-mechanical processes frequently leading to the deterioration of these materials. The natural rock deterioration present an important problem in engineering geology, especially when massive rocks form the foundation ground of historic buildings, thus contributing to their instability. The deterioration of the rocks (weathering, rock breakdown) is caused by exogenous factors with the main influence of the temperature and moisture regimes. Therefore a monitoring of temperature and moisture regimes and their impact on the natural rock deterioration to a great extent depends on the lithological, mineralogical, structural and thermophysical properties of the particular rock type.

The authors describe utilization of the transient Hot-ball method for measuring the temperature and thermal conductivity of materials that was used to the moisture monitoring at the Perún`s rock at the Spiš Castle (UNESCO site). For the long-term monitoring we used the hot ball sensor placed in cylindrical probes (samples) prepared from the cores drilled from the natural rock body. Thermal conductivity of probe body made of the porous stone is measured in dependency on moisture content under laboratory conditions in between dry and water saturated state. The extrapolation of this calibration allows direct measurement of moisture content.

The estimation of the water content in various depths inside the rock mass helps to assess its effect on the temperature distribution beneath the surface, which might control the thermal expansion of the rock cliff upon the Spiš Castle stands and its potential instability.

The results obtained during long-term monitoring showed relation between temperature distribution and the thermal conductivity within the probe and the precipitation registered by the meteorological station.

POSTER SESSION II

Medical and Biological measurements

Tuesday
10:45 to 11:30
and
14:15 to 15:00
Foyer

APPLICATIONS OF TEMPERATURE, HUMIDITY, MOISTURE AND OTHER THERMAL MEASUREMENTS IN THE MEDICAL AND BIOLOGICAL FIELDS - THE UGANDAN CONTEXT

S. R. Rwashana
Uganda National Bureau of Standards, Kampala, Uganda
E-mail: simon.rwashana@unbs.go.ug

Metrology, the Science of Measurement is often overlooked and yet it is one of the most critical elements of primary health care and a core component of Biological experimentation and tests. This is because all measurements, if inaccurate, can lead to improper research and diagnosis of diseases, prescription, storage and administration of treatment (drugs).

This paper introduces the challenges facing Metrology in health care and the Biological fields and defines the importance of improving and in some cases introducing this strategy by involving all the stake holders.

A Case study is taken from Uganda, East-Africa where out of all the health centers in the 80 districts of Uganda there is no evidence of calibration / verification of Medical and Laboratory equipment.

Solar system and battery enabled measurement is proposed in the countryside where there is completely no electrical power.

NEW EQUATION FOR CALCULATION OF WET-BULB TEMPERATURE, INSTRUMENTAL IMPLEMENTATION

Y. A. Barbar, M. N. Golikov, S. L. Solovyov
TKA SCIENTIFIC INSTRUMENTS, St. Petersburg, Russia
E-mail: ybarbar@yahoo.com

To estimate the most important parameters of microclimate, is necessary to obtain information about the temperature of wet bulb t_{nw}. Where t_{nw} – natural temperature of wet thermometer, located in conditions of natural convection.
It is desirable refuse from the direct measurement of temperature t_{nw} by means of wet thermometer, because of the difficulties in correct operation of wet channel occur during the wide-scale measurements, which can cause the significant errors at determination of t_{nw}.
Statistics such practical measurements show that errors of temperature t_{nw} measurement are some degrees by scale Celsius, but it is unacceptable.
The analytic expression which couples inter se the values of relative humidity RH, temperatures t_a, t_{nw}, pressure of water vapors P, where the parameter t_{nw} is one of the arguments of P function, is taken from work of Y. Nishi [1].
We found the calculation of temperature tnw values by the Nishi expression leads to significant errors which reaches unity of degrees, as opposed to the values of t_{nw} indicated in the psychrometric tables, which is inadmissible.
In order to improve the reliability of mathematical calculation the authors carried out the mathematical analysis of psychrometric tables, as a result the expression which connects in a parametric manner the value of t_{nw} in function of the measured values of RH and t_a, was obtained:

$$t_{nw} = (A \cdot RH + B) \cdot t_a + C \cdot RH - D \cdot (0{,}01 \cdot RH - 0{,}5)^2 - E, \qquad (1)$$

where A, B, C, D, E – constants.
The requirements for instrumental precision of measurements are established: with respect to humidity – not worse than ±3% RH, with respect to temperature – not worse than ±0,3 °C, at which the determination error of t_{nw} by (1) doesn't fall outside the limits of ±0,60 °C in the ranges of measured temperatures of 20±50 °C and humidity of 5 ... 100% RH.
For example the digital Sensirion SHT71, SHT75 sensors of humidity and temperature correspondto such requirements in case of introduction of correction functions by the humidity channel, suggested by the authors, which provides the humidity measurement error at a level of ±1% RH.
The resulting equation (1) is used in thermohygrometer series "TKA" for calculating the values of tw and values WBGT -index, according to ISO7243: "Hot environments. Estimation of the heat stress on working man, based on the WBGT-index (Wet bulb globe temperature)."

[1] "Y. Nishi. Field assessment of thermal characteristics man and environment by using a programmable pocket calculator. ASRAE Trans. Vol. 83 (1), 1977, p. 103-111"

COMPARISON MEASUREMENTS OF INFRARED EAR THERMOMETERS AGAINST THREE TYPES OF BLACKBODIES

S.-F. Tsai, H.-T. Lee
Center for Measurement Standards, Hsinchu, Taiwan
E-mail (corresponding author): Shu-Fei_Tsai@itri.org.tw

Body temperature regularly monitored by utilizing clinical electrical thermometers, infrared ear thermometers, or forehead thermometers is demanded in schools and assembly places, especially being faced with the worsening global influenza infection nowadays. Among those thermometers, infrared ear thermometers capable of rapid response are designed to measure the ear canal temperature because the ear canal is located in close proximity to major brain arteries and veins, and its temperature is relatively close to that of the tympanic membrane, which is a recognized measure of core temperature.

Three types of cavity-shaped blackbodies vertically immersed in a temperature controlled stirred water bath were developed in CMS to calibrate and verify several commercial IR ear thermometers produced by six manufacturers. One of them was designed according to the mandatory information required in ASTM E-1965, and the other two were referred to the informative examples in EN 12470-5, and JIS T 4207 standards respectively. The temperature of blackbody cavity shall be represented by the water temperature near the bottom of cavity that is measured using an immersed platinum resistance thermometer (PRT) for which the calibration is traceable to our national standard and with an uncertainty no greater than 0.03 °C (k=2).

The water bath was evaluated through PRT to be stable and uniform within 7 mK, in which three blackbodies were characterized via the analysis of variance (ANOVA) method using the IR ear thermometer. Similarly, IR ear thermometers were compared and analyzed by the maximum permissible error (MPE) against blackbodies' temperature and the readout of PRT at three blackbody temperatures of 35.5 °C, 37 °C and 41 °C. The analysis results of ANOVA manifested that there is no significant temperature difference among three different structured blackbodies, and approximate 65% of tested IR ear thermometers conform to the MPE requirements regulated in ASTM E-1965, EN 12470-5, or JIS T 4207 standards.

POSTER SESSION II

Other applied measurements

Tuesday
10:45 to 11:30
and
14:15 to 15:00
Foyer

TEMPERATURE MEASURING TECHNIQUES: CLIMATE AND WEATHER APPLICATIONS

V. L. Mwazi
Zambia Bureau of Standards, Lusaka, Zambia
E-mail: vlmwazi@yahoo.com

The growing interest of global warming in the recent past has led to an intensive development of fast response measurement techniques. The most commonly discussed measure of global warming is the trend in globally averaged temperatures near the earth's surface in which meteorological observations show a warming of approximately 0.6-0.8 °C since the late 1800's. The instrumental temperature record indicates the fluctuations of the temperature of the atmosphere and the oceans as measured by temperature sensors .The sea surface temperature is monitored from satellites and ships at sea. Land and atmosphere temperatures are similarly reported and recorded.

The natural habitat of animals and plants together with several existing environmental factors are continuously investigated, observed and analyzed on in terms of weather reports, climate change, etc. It is noted that even small measurement errors can have large consequences, so temperature probes used in these observations must be of the highest quality. The widespread availability and application of thin film deposition techniques for metals and ceramics coupled with advances in micro technology have seemingly expanded the range of devices available for temperature measurements.

The paper reviews the various types of temperature sensors available. Vital to the use of temperature measurement techniques is accurate calibration. The various techniques in common use for calibration are also described.

Keywords: global warming, natural habitat, weather, climate, thin film, temperature measurements, calibration.

DEVELOP AN IMAGE PROCESSING AIDED AUTOMATIC APPARATUS FOR IMPROVING THE TEMPERATURE MEASUREMENTS

S.-F. Tsai, W.-C. Chang, Z.-Y. Chung
Center for measurement standards/ITRI, Hsinchu, Taiwan
E-mail (corresponding author): Shu-Fei_Tsai@itri.org.tw

Formerly, in CMS, the temperature reading of liquid-in-glass thermometer (LIGT) was decided by human eyes through an auxiliary CCD which was equipped with enlarging lens and then transferred the magnified image of LIG to a monitor to be observed. On the other hand, clinical electrical thermometers (CETs) are used extensively nowadays to check the body temperature to avoid the transmission of influenza, for example H1N1 flu. An apparatus composed of mechanical mechanism and optical module was developed to automatically measure the LIGTs and CETs in CMS recently. It was designed to raise the resolution of LIGTs to one more digit during the calibration in thermometry metrology field and automatically recognize the digital readout of CETs during the verification in legal metrology field.

The mechanical mechanism functions to hold the thermometers within its two annular fixtures which are able to make a rotation and up-down movement to finely adjust both of the immersion depth and plane angle by driving two θ-z motor stages while doing the calibration or verification. Two annular fixtures; which are inner and outer ones with different diameters; allow accommodating more thermometers, and outer one is lifted upwards when measurements are taken on the thermometers held within inner fixture.

The Optical module comprised of charge couple device (CCD) and optical lens was set up for image processing. For LIGTs, the images of liquid column in the capillary tube as well as the scale on the stem are taken by CCD, and then one additional digit is accurately attainable by the pixel ratio of protruding column above the scale to the two adjacent scales via the LabVIEW software. An image processing approach was utilized to automatically record the readings of clinical electrical thermometers successfully in CMS even though that kind of thermometers normally is not provided with communication interface. The image of readout on the LCD display is captured similarly as stated above, however advanced optimized processing upon CCD and image binarization, and even a light source to differentiate the digit numbers from backlit are necessitated in case the display window of CET is with a poor contrast.

EVALUATION ON RAIN-DEFENCE PERFORMANCE OF TEMPERATURE SENSORS

J. Zhao, Y. Wang, P. Wang, J.-H. Wu
Beijing Changcheng Institute of Metrology and Measurement, Beijing, China
E-mail (Jian Zhao): zhaojianbj73@sina.com

Rain-defence performance of temperature sensors is concerned in many important fields, while there has no suitable method to evaluate this performance before. To solve the problem, an experiment is designed in this paper. The experiment device used is normal temperature calibration wind tunnel. To simulate the practical environment as rainfall of 8mm per hour, a water tank is set above the open experiment section of the wind tunnel, at the bottom of which many minor holes are drilled so that rain can run down. A new-typed temperature sensor and an old-typed temperature sensor are calibrated in the wind tunnel. The calibration method is as same with recovery characteristic and dynamic characteristic calibration except that the environment here is a mixture of spray and air. As the authentic total temperature is not able to be obtained, aquivalent recovery factor is defined, which uses the total temperature outside the calibrated sensor to participate in the calculation instead of the authentic local total temperature. And through dynamic characteristic calibration, time constant of the sensor is obtained too. During the calibration experiment, gas Mach-number, water temperature and attack angle of the temperature sensors are changed. The result shows that aquivalent recovery factor of the new-typed temperature sensor is higher than the old-typed one when the water temperature is 15 ▫ and 4 ▫,which proved that rain-defence performance of the new-typed temperature sensor is better than the old-typed one, and some other comparisons can also prove this point. Associate the design structure of the temperature sensor with the experiment result, the evaluation method is proved reasonable and feasible.

STUDY OF TFTC DYNAMIC CHARACTER CALIBRATION TECHNIQUE

Caijing[1], Z. Xue-cong[2], Y. Yong-jun[1,2], Zhaojian[1]
[1] *Beijing Changcheng Institute of Metrology and Measurement, Beijing, China*
[2] *Beijing University of Aeronautics and Astronautics, Beijing, China*
E-mail (corresponding author): yangyj007@sina.com

TFTC (Thin film thermocouple) is an advanced thermal sensor which is used to measure transient temperature. The time constant is so short that the normal calibration instrument and method already cannot meet the request of high response velocity. Up to today, there is no uniform instrument and method to calibrate the TFTC. Normally TFTC is assumed as a first-order system, and the input is an ideal step-input temperature signal producing by pulse laser. According to the response curve, the time of 63.2 percent of temperature step is time constant. While the method has 3 problems as below:

(1) Laser can produce much quickly transient temperature increase on the material surface. Using Positive step-input, the mutual operation between laser bean and material is very complicated transient process. It is difficult to produce regular temperature wave on the material surface. Using negative step-input, the influence of substrate conduction is much larger than using positive step-input, which would have great effect on temperature decrease velocity.

(2) The bandwidth of pulse laser is very short relative to the constant time, so the indicative value of TFTC begins to drop before the peak value is achieved. For this reason the time constant cannot be calculated by TFTC's output.

(3) The storage energy component is more than one because of the influence of substrate conduction. TFTC departs far from the one rank system. Time constant cannot be used to evaluate the dynamic character.

An adjust temperature ascend can be obtained by using a heat source produced by a large power laser which Q can be modulated. The voltage output of TFTC is recorded by high velocity data collection system. Because of the input signal is not a strict temperature rank, it is necessary to record the transient surface temperature by high-respond none-contact temperature measurement instrument as input signal. The transfer function can be derived by system identification method.

TFTC is calibrated by the method in which the dynamic character measurement and system identification are combined. In the method the assumption of ideal temperature step, first-order system and the limit of pulse laser bandwidth are departed . The calibration becomes much more flexible and practical.

AUTOMATION OF A NVLAP ACCREDITED PRT CALIBRATION PROCESS

M. J. Coleman, T. J. Wiandt
Fluke Corporation, Hart Scientific Division, American Fork, UT, USA
E-mail: mike.coleman@fluke.com

Calibration of PRTs is an important part of the calibration program in Fluke's Hart Scientific Calibration Lab. PRTs are calibrated by comparison in a liquid nitrogen comparator and a series of stirred-liquid baths over the range –197 °C to 500 °C. The reference temperatures are determined by a metal sheath SPRT and a commercially available precision digital thermometer.

Since PRTs are not strain-free devices and vary greatly in design and wire purity, many factors must be accounted for in their calibration. Among these factors are curve fit, repeatability and insulation resistance. Other issues that affect the quality of the calibration are heat source uniformities, reference equipment reliability, pre-calibration heat treatment, and statistical process control.

In order to improve calibration process efficiency and control of the aforementioned factors, a comprehensive automation approach has been introduced into Hart Scientific's PRT calibration process. One of the main goals of the automation project was to not simply avoid affecting the process' NVLAP accreditation, but to improve upon its compliance.

The purpose of this paper is to present how the software and other process controls have been designed and implemented, and the manner in which they address these important calibration issues. For completeness, the overall calibration process will be presented as well as the improvements that were made. In addition, methods for ensuring proper curve fit using the software and a process for reporting failed calibration data to the customer will be described.

MEASUREMENT UNCERTAINTIES INFLUENCE ON THE THERMAL ENVIRONMENT ASSESSMENT

M. Dell'Isola[1], A. Frattolillo[1], B. I. Palella[2], G. Riccio[2]
[1] *Dipartimento di Meccanica, Strutture, Ambiente e Territorio (DiMSAT) Università degli Studi di Cassino, Cassino (FR), Italia*
[2] *Dipartimento di Energetica Termofluidodinamica Applicata e Condizionamenti Ambientali (DETEC), Università degli Studi di Napoli Federico II, Napoli , Italia*
E-mail (corresponding author): frattolillo@unicas.it

ISO 7726 Standard describes in a very careful way both the measurement techniques and the related metrological performances of instruments required for the thermal environment assessment. On the contrary hand, the analysis in situ of assessment indices rarely takes into account the evaluation of the single measurement uncertainties and their inevitable effect on the thermal environment assessment. The reasons of these lacks are several: the high costs for the calibration of devices, the complex metrological traceability, the variety of measurement parameters, the late application of both ISO GUM on the accuracy assessment and the related Standards for the management and certification of measurement hardware of laboratories.

Concerning thermal comfort assessment, another critical facet is related to the limit values for the percentage of dissatisfied for each comfort category required by ISO 7730 Standard. As a matter of fact, such values (especially for the category labelled as "A") can be so strongly prescriptive to make almost impossible their practical use, especially if the high uncertainty of some quantities involved for the assessment is taken into account. As far the mean radiant temperature and the radiant asymmetry is concerned, ISO 7730:2005 suggests three direct (black-globe, two-sphere radiometer and constant air temperature sensor) and two indirect (view factors and plane radiant temperature) measurement methods. Although this Standard sets up their different and not easily comparable ranges of accuracy, there is no clear reference about the effect of such values (differentiated in "required" and "desirable") on the environment classification ("A", "B" or "C").

This work deals with the effect of measurement accuracy of both physical (the air temperature, the mean radiant temperature, the air velocity and the relative humidity) and subjective parameters on the assessment of moderate thermal environment. A compared assessment of comfort indices in most significant environments carried out by means of different measurement techniques and devices is also reported. This procedure can highlight both the compatibility of measurement and the suitability of devices required for the verification of comfort classes settled by ISO 7730 Standard.

INTEGRAL QA SYSTEM OF ENIRONMENTAL MONITORING AT EARS

D. Groselj, J. Knez
Environmental Agency of the Republic of Slovenia
E-mail (corresponding author): drago.groselj@gov.si

Environmental Agency of the Republic of Slovenia (EARS) operates a network of automatic stations for meteorology (temperature, humidity, air pressure ...), hydrology, ambient air-quality, water quality and ionizing radiation. At present, there are about 125 automatic stations in our network, sending 170 different parameters in real time to the central database at EARS, which are quite different regarding measuring parameters and purpose. Strategic issue of EARS is quality of measured data. Very important goal is to operate the common integral quality assurance system for different network type, for different quantities such as temperature, humidity,..., which will base on ISO/IEC 17025 standard. New legislation that covers environmental monitoring, especially modern European directives in the field of air and water quality, requires that quality systems has to be established to assure technical competence, quality and international comparability of data. Meteorology, on the other hand, is bounded mainly to WMO (World Meteorological Organization) guides and recommendations, ICAO (International Civil Aviation Organization) and other relevant documents. To achieve this goal, we have elaborated QA Guide for EARS measuring network, which defines requirements regarding quality of data and prescribe QA/QC procedures for the network and supporting services.

EARS Calibration laboratory is an important subject in an integral QA system in terms of periodical recalibrations of field measuring instruments. EARS Calibration laboratory has long history of performing accredited calibrations of measuring instruments in the field of temperature, relative humidity, air pressure and air-quality parameters: carbon monoxide, sulphur dioxide, ozone and nitrogen oxides. EARS Calibration laboratory is also Regional Instrument Centre (RIC) within WMO framework.

Important infrastructure of the QA system is the Information System of Measuring Network (ISMN). Available through an intranet portal, it enables data visualization and control as well as operations with meta data (data about instruments). Applications based on a relational database manage planning, equipment management and correlate data to any operational event, such as maintenance and calibration.

The goal was to make the measurement process transparent, so the user knows the type of the measuring equipment and method used in the 'on-site' measuring process, the site characteristics etc., and very important - the measurement traceability and uncertainty of the measured data. For example, measurements of air temperature from the past 40 years are problematic, since we have identified some problems with traceability and especially measurement uncertainty. This can result in inaccurate interpretation of global warming problem. With new system in place, our measurements are more reliable, trustworthy and can be used for long term analysis.

POSTER SESSION II

Resistance Thermometry

Tuesday
10:45 to 11:30
and
14:15 to 15:00
Foyer

CONSTRUCTION AND CHARACTERIZATION OF NTC THERMISTORS AT LOW TEMPERATURE

Y. Lan[1], L. Yu[2], A. Chang[3], G. Chen[4], S. Yang[1]
[1] *Beijing Institute of Aerospace Testing Technology, Beijing 100074, China*
[2] *Cryogenic Laboratory, Technical Institute of Physics and Chemistry, Chinese, Academy of Sciences, Beijing 100080, China*
[3] *The Xingjiang Technical Institute of Physics and Chemistry of the Chinese Academy of Science, Urumuqi, China*
[4] *Institute of Refrigeration and Cryogenics, Zhejiang University, Hangzhou, China*
E-mail address (corresponding author): hangtiandiwen@163.com

Nano-powder of negative temperature coefficient (NTC) ceramic with spinel structure of Mn-Ni-Cu-Co-La-O composition was prepared by the Pechini method. A type of NTC thermistor sensor (3.0 mm diameter × 1.5 mm high) was designed by the in-situ lead wire attachment method (ISAM) and made by using the synthesized powder. NTC thermistors were packaged in the glass sealed package. The phase composition and microstructure of the materials were characterized by means of X-ray diffraction and scanning electron microscope (SEM). The effect of aging and magnetic field on thermistors and reproducibility were investigated. Six independent NTC thermistors were calibrated by using cryostat, standard platinum-sheathed platinum resistance thermometer and a FLUKE 1590 super thermometer meter over the range from 18 K to 120 K. The data was interpolated to obtain calibration tables with 2 K intervals from 18 K to 30 K and 5 K intervals from 30 K to 120 K, respectively. The tables were averaged to fit a mean curve with the equation: $1/T = A_0 + A_1\ln(R) + A_2\ln(R)^2 + A_3\ln(R)^3 + A_4\ln(R)^4$. The values of $B_{20\ \mathrm{K}/100\ \mathrm{K}}$ thermistor constant and activation energy of NTC thermistors were counted severally. The results show that the values of aging coefficient and reproducibility of NTC thermistors did not exceed 5% and ± 3 mK, respectively. The biggest errors induced by magnetic field in the magnetic field which is less than 14 T at the temperature of 20 K and 77 K were –33 mK and 41 mK, respectively. A curve-fit equation was obtained: $1/T = 5.5\times10^{-3} + 4.8\times10^{-4}\ln(R) + 6.7\times10^{-4}\ln(R)^2 - 6.3\times10^{-5}\ln(R)^3 + 3\times10^{-6}\ln(R)^4$. All sensors agreed with this fit within an error 1.5 K in the temperature range from 18 K to 120 K. The values of $B_{20\ \mathrm{K}/100\ \mathrm{K}}$ constant and activation energy of the NTC thermistors range from 204 K to 207 K and from 0.0174 eV to 0.0178 eV, respectively.

Keywords: cryogenic; electrical properties; low temperature; spinel; thermistors.

INVESTIGATION OF 50 Ω RHODIUM-IRON SENSORS WITH CERAMIC SHELL

L. Yu[1,2], C. Yao[3], Q. Zhang[1], Z. Huang[1]
[1] *The Key Laboratory of Cryogenics, TIPC ,CAS , Beijing, China*
[2] *Graduate School of Chinese Academy of Sciences, Beijing, China*
[3] *Shanghai Institute of Process Automation Instrumentation, Shanghai, China*
E-mail: lhyu@mail.ipc.ac.cn

Since 1990s, the copper-cell and ceramic-shell resistance thermometers with 0.03 mm rhodium-iron alloy wire have been developed by us. They have been widely used in cryogenic applications in China. Because the original 0.03 mm diameter wire ran out in 2009, we start to obtain this diameter by drawing the 0.05 mm diameter wire to 0.03 mm.

Preliminarily, we produced a few thermometers without annealing the wires and found that they cannot be used as temperature sensors because of the stresses generated in the drawing process. The following sensors are then made by capsulating the wires inside ceramic shell and annealing therein.

Stability of 30 thermometers were investigated after they have been exposed to 20 thermal cycles between 4.2 K and room temperature. The thermometers remained stable at 273.15 K within 10 mK. A reference standard curve was obtained by 58 calibration points from 1.2 K to 300 K for ten thermometers. According to the curve, we can simply fit the R-T relationship for the other sensors in the same batch within 0.1 K accuracy with just a few temperature points. Finally, the self-heating effect and sensor interchangeability in the same batch were discussed. The results indicated that the performance of the thermometers with drawn wires are the same as that of the thermometers with the original 0.03 mm diameter wires.

DEVELOPMENT OF PRECISION Rh-0.5%atFe THERMOMETERS OF CHINESE PRODUCTION: FINAL TESTS

L. Lipinski[1], A. Szmyrka-Grzebyk[1], P. Lin[2], X. W. Li[3], H. Manuszkiewicz[1], D. Jancewicz[1], A. Grykalowska[1], P. P. M. Steur[4], F. Pavese[4]

[1] *Instytut Niskich Temperatur i Badan Strukturalnych (INTiBS), Wroclaw, Poland*

[2] *National Institute of Metrology (NIM), Beijing, China*

[3] *Kunming Dafang Science & Technology Ltd, Kunming, China*

[4] *Istituto Nazionale di Ricerca Metrologica (INRIM), Torino, Italy*

E-mail (corresponding author): f.pavese@inrim.it

Following the practical impossibility to obtain new precision Rh-0.5at%Fe thermometers in the last years, re-starting of the commercial production of such thermometers in Yunnan (China) was explored by INRIM in co-operation with NIM and with the help of INTiBS for prototype characterisation. After the production in 2006 of ten prototypes of the new production, the published preliminary test data showed satisfactory results as to the reproducibility on thermal cycling, but the *R-T* characteristics with lower sensitivity was unsatisfactory, a problem attributed to the alloy composition. Further studies, concerning the alloy composition, the thermal pre-treatment of the alloy wire used in new prototypes and some further measurements of the basic characteristics, namely sensitivity and *R-T* characteristics, were performed and published in 2007-08. In 2008 a new batch of 8 prototypes was produced, showing *R-T* characteristics basically identical to that of the prototypes of the 2007 publication. This paper reports the results of the measurements on the full characteristics of these prototypes in the range 2.5–25 K, which confirm the similarity to the typical characteristics of previous commercial RhFe thermometers, and of the effect of thermal cycling, showing for 6 thermometers out of 8 a stability better than 1 mK (the measurement uncertainty) at 4.2 K, with one further thermometer slightly more unstable than 1 mK and the last even more unstable.

These results indicate that all production problems are basically resolved, so that these thermometers can soon become commercially available.

AUTOMATIC LINEARITY CALIBRATION IN A RESISTANCE THERMOMETRY BRIDGE

R. Walker
Fluke Corporation-Hart Scientific Division, American Fork, U.S.A.
E-mail: rick.walker@fluke.com

Present methods of testing and evaluating uncertainties associated with a resistance thermometry bridge, including linearity, involve expensive equipment and time-consuming procedures. As a consequence, bridge calibrations are performed infrequently. This situation increases risk that due to unrecognized defects in the equipment, errors in the measurement system may be greater than expected. In an effort to make calibration of a thermometry bridge more convenient, the author has proposed a method of testing bridge linearity using resistance voltage dividers. The technique is simple enough to be integrated into the resistance thermometry bridge's design, allowing quick, automatic linearity calibration that can be performed routinely without external equipment.

The paper explains the proposed technique and its implementation in a resistance thermometry bridge. To evaluate the effectiveness of the technique in recognizing possible errors and defects, a series of tests were performed. First, a properly functioning resistance thermometry bridge was tested. The proposed technique was used to evaluate the linearity of the bridge and these results were compared against a calibration of the same bridge using a Resistance Bridge Calibrator. Then tests were performed on several resistance thermometry bridges that had a variety of in-tentionally introduced defects. The results presented in this paper show that the proposed linear-ity calibration technique could be a valuable tool in maintaining low, traceable uncertainties in a robust temperature measurement or calibration process.

THERMAL HYSTERESIS IN THIN-FILM TYPE PLATINUM RESISTANCE THERMOMETERS

K. S. Gam, I. Yang and Y.-G. Kim
KRISS, Daejeon, Korea
E-mail (corresponding author): ksgam@kriss.re.kr

The use of platinum resistance thermometer (PRT) for industrial applications has increased markedly in the last twenty years. The main reason for this has been the need for the application in new fields and improved temperature measurement accuracy. The industrial PRTs have three types, such as ceramic-type, glass-type and thin-film type. The ceramic-type and glass-type PRTs made of high-purity platinum coil. But thin-film type PRTs are manufactured generally using a laser trimming method and the deposition of platinum thin-film upon the alumina substrate. Considering the strong adhesion between platinum thin-film and alumina substrate, the thin-film PRTs have inevitably strain over the operating temperature range. This causes anomalies and instabilities in the resistance versus temperature characteristics (R-T). The most prominent and observable effect of thermally induced strain is the thermal hysteresis in the R-T characteristics. Thermal hysteresis is one of the main uncertainty factors in the calibration of industrial platinum resistance thermometers in laboratories. Thermal hysteresis for ceramic-type and thin-film type PRTs has been investigated extensively by D. J. Curtis in 1982 [1]. Since his study the manufacture technology of thin-film PRTs improved much and there was very little data for the thermal hysteresis of thin-film PRTs.

We used 3-zone furnace (Isotech 511Medusa-3, England) and a Super Thermometer (Hart Scientific 1575, U.S.A.) to construct a measurement system for the thermal hysteresis of thinfilm PRTs. In this study the furnace temperature increased from room temperature to 500 °C with 100 °C step and decreased reversely. Temperature-resistance data measured for two hours when the furnace temperature at the every step was stable within 10 mK. The furnace temperature, and the resistance measurement of the reference SPRT and thin-film PRT were controlled and measured automatically by a personal computer using a LabVIEW program. The thermal hysteresis for 30 pieces of the industrial PRT that were fabricated by three Korean companies was measured. The thin-film sensor elements of industrial PRTs were supplied by IST, Switzerland and Omega, U.S.A. The thermal hysteresis was distributed from 16 mK to 156 mK for 30 sensors, and the lowest hysteresis was 16~31 mK in the all temperature range tested. We compared the thermal hysteresis values against the thermal hysteresis shown in reference values.

[1] D.J. Curtis, "Thermal hysteresis and stress effects in platinum resistance thermometers", TMCSI, Vol.5, p. 803(1982)

CALIBRATION OF CAPSULE-TYPE SPRTS AT THE TRIPLE POINT OF WATER FOR THE BOLTZMANN CONSTANT DETERMINATION

F. Sparasci, L. Pitre, D. Truong
LNE-INM/CNAM, Paris, France
E-mail (corresponding author): fernando.sparasci@cnam.fr

The determination of the Boltzmann constant by an acoustic method is performed at the temperature of the triple point of water (TPW). In this experiment, a copper acoustic resonator is installed in a cryostat at the temperature of the TPW, and the temperature is measured with a set of capsule-type standard platinum resistance thermometers (CSPRTs) calibrated at the triple point of water. The aim is to obtain a temperature measurement with an associated uncertainty of the order of 0.1 mK, corresponding to a relative uncertainty contribution of $0.4 \cdot 10^{-6}$ on the determination of the Boltzmann constant.

Contrary to long-stem standard platinum thermometers, designed to easily fit in the thermometric well of TPW glass cells, capsule-type thermometers need to be adapted with specific thermometer fittings, with the risk of degrading the thermal contact between the thermometer and the phase transition, and hence the final calibration uncertainty.

Moreover, when used on the acoustic resonator, the thermometers are subjected to pressures varying between 0.05 MPa and 0.7 MPa. This can induce resistance shifts and, more in general, a loss of stability of the thermometer.

A batch of CSPRTs was calibrated at the triple point of water under several calibration conditions. Tests were performed with different resistance bridges and reference resistors and by connecting the thermometers to the bridge by either short cables or the long cryostat wiring, in order to evaluate systematic effects coming from the experimental setup. Two different fittings were tested, with three different contact fluids, and the self-heating effect was analyzed, to determine the goodness of the thermal contact between the thermometer and the phase transition. Finally, measurements were performed changing the external pressure, to observe the stability of the thermometers.

The results obtained on the 0.5 dm^3 acoustic resonator named “BCU3” show that an accuracy of 0.1 mK in the measurement of the resonator temperature can be achieved, but frequent checks and recalibrations of the CSPRTs are necessary.

THE HYSTERESIS CHARACTERISTICS OF SOME INDUSTRIAL PRTS

D. R. White, C. L. Jongenelen, and P. Saunders
MSL, Lower Hutt, New Zealand
E-mail (*corresponding author*): *r.white@irl.cri.nz*

Hysteresis in industrial platinum resistance thermometers (PRT) is caused by the tension and compression induced in the wire due to the differential thermal expansion of the platinum wire and the substrate. Although the hysteresis is a key performance parameter for high-accuracy applications, very little is known about the effect, except that the more robust the PRT, the greater the amount of wire in contact with the substrate, and the greater the hysteresis. Industrial PRTs used in laboratory measurements at the level of 10 mK or better tend to employ sensors exhibiting very low hysteresis, so that the effect is not usually a significant contributor to total uncertainty. For this reason most calibration laboratories do not measure the hysteresis, except perhaps as a single measurement at a single temperature. Additionally, the extra cost involved in measuring the temperature characteristic on both rising and falling temperatures is a discouraging factor. However, as laboratory measurements are pushed to higher accuracies, the need to measure and understand hysteresis effects becomes more pressing.

This paper reports the measurement of hysteresis in a wide range of industrial PRTs including film, glass-encapsulated, wire-wound ceramic, and low-hysteresis partially-supported wire sensors. Hysteresis is assessed as a function of temperature and temperature range and possible guidelines for the design of calibration procedures are discussed.

DRIFT STUDY OF THERMISTORS USED BETWEEN 90 °C AND 150 °C

G. F. Strouse, C. W. Meyer, W. W. Miller
NIST, Gaithersburg, MD, USA
E-mail: gstrouse@nist.gov

A 27 week long thermal aging (drift) study of thermistors was initiated at NIST to quantify and characterize the thermal aging of thermistors subjected to temperatures between 90 °C and 150 °C. Two hundred and one thermistors from two manufacturers were subjected to five different thermal aging temperatures during the course of measurements. All of the thermistors studied are of the bare element bead-in-glass style. One of the manufacturer's thermistors was separated into three groups of heat pretreatment performed prior to measurements: no heat treatment ($M1_{none}$), 4 weeks at 130 °C ($M1_{130}$), and 4 weeks at 150 °C ($M1_{150}$). The second manufacturer's thermistors received no heat pretreatment ($M2_{none}$). The study used five thermal soak temperatures (23 °C, 90 °C, 100 °C, 120 °C, and 150 °C). In order to quantify drift, all thermistor measurements were performed at 85 °C in a comparison water bath. Each thermistor group was subjected to only their thermal soak temperature, ambient conditions (23 °C) and the 85 °C measurement temperature. The combined expanded uncertainty of the thermistor aging study measurement process [$U(k=2)$] is 2.8 mK.

After a baseline measurement set was established, each thermistor group was placed in a furnace operating at the pertinent thermal soak temperature for one week before another 85 °C measurement was performed. Measurement sets were acquired once per week for each batch of thermistors over the first nine week period and then every three weeks. We found that there are significant differences in the thermal aging (drift) results between the thermistor groups. For example, after 9 weeks at the 120 °C thermal soak temperature the average thermistor thermal aging (drift) was equivalent to 1%, 0.3%, 0.3% and 6.2% for $M1_{none}$, $M1_{130\,°C}$, $M1_{150\,°C}$, and $M2_{none}$, respectively. This paper gives the results of the thermal aging (drift) of the thermistors with respect to each of the thermistor groups and thermal soak temperatures.

INFLUENCE OF PRT THERMOPHYSICAL PROPERTIES ON THE MEASUREMENT RESULT OF AL FREEZING POINT TEMPERATURE

A.G. Inanova, A.I. Phokhodun, O.S.Shulgat
D.I.Mendeleev Institute for Metrology, Saint Petersburg, Russia
E-mail (corresponding author): A.G.Ivanova@vniim.ru

Differences between measurement results of main fixed points temperatures of national laboratories and unrepeatability of resistance ratio $W_{f.p}$ could be caused by nonregulated demands to some characteristics of the using thermometer, and consequently by absence of the corresponding corrections. The main characteristics that should be regulated are:
1. The thickness of silica glass sheath from which conductive heat leakage depends on.
2. The matted area size influencing on radiation leakage.
3. Thermal resistance between thermometer sensitive element and liquid-solid interface.

In order to maintain the correlation between the investigated characteristics and measuremen result of Al freezing fixed point temperature and, as a consequence, the uncertainty of calibration of SPRT at Al fixed point, some experiments were carried out in VNIIM.

The influence of the matted area size, the thickness of silica glass sheath and the thermal resistance between sensitive element and liquid-solid interface, were investigated both experimentally and by calculation method. Preliminary tests indicated that existence of 15 cm matted area on a thermometer sheath can increase thermometer reading in 0,06 °C. The difference in measured values of temperature gradient in thermometer well at 6 cm height caused by the differences in thermometers sheath thickness achieves 0,5 – 1,5 mK.

EVALUATION OF SMALL SIZED PLATINUM RESISTANCE THERMOMETERS WITH ITS-90 CHARACTERISTICS

K. Yamazawa, K. Anso, J. V. Widiatmo, J. Tamba and M. Arai
National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology (NMIJ, AIST), Tsukuba, Japan
E-mail K. Yamazawa: kazuaki-yamazawa@aist.go.jp

Many platinum resistance thermometers (PRTs) are applied for high precision temperature measurements in industry. Most of the applications use the PRTs with Pt100 characteristics. However, recently, some applications, such as measurements of the temperature distribution within equipments, require a more precise temperature scale at 0.01 °C level.

In this paper we report the evaluation of remarkably small sized PRTs that have resistance-totemperature characteristics very close to that of standard PRTs of the ITS-90. Two types of the sensing element were tested, one is 1.2 mm in diameter and 10 mm long, the other is 0.8 mm and 8 mm. The resistance of the sensor is 100 ohms at the triple point of water temperature. The resistance ratio at the Ga melting point temperature of the sensing elements all exceeds 1.11807.

To verify the closeness of the resistance-to-temperature characteristics, we employed comparison measurements from 0 °C up to 157 °C. A water bath for temperatures up to 30 °C (comparison uncertainty 0.43 mK), and a pressure controlled heat pipe furnace for higher temperatures were used for the comparison measurement. Characteristics of 19 thermometers with these small sized sensing elements were evaluated. The deviation from the temperature measured using a standard PRT used as a reference thermometer in the comparison was remarkably small, when we apply the same interpolating function for the ITS-90 sub-range to these small thermometers. Results including the stability of the PRTs and the uncertainty evaluation of the comparison measurement, and the comparison results showing the small deviation from the ITS-90 resistance-to-temperature characteristics will be reported. Development of such PRT might be a good solution for applications such as temperature measurement of small objects or temperature distribution measurements that need ITS-90 temperature scale.

SELF-HEATING ANOMALIES IN STANDARD CAPSULE RESISTANCE THERMOMETERS DUE TO AIR CONTAMINATION

W. L. Tew[1], C. W. Meyer[1], M. L. Huber[2], and E. W. Lemmon[2]
[1] *NIST, Gaitherbsurg, MD USA*
[2] *NIST, Boulder, CO USA*
E-mail (corresponding author): wtew@nist.gov

Standard capsule-type resistance thermometers are necessary to disseminate the ITS-90 over its cryogenic ranges at the lowest achievable uncertainties. These include standard platinum varieties for the range 13.8 K to 273 K, and rhodium-iron and platinum-cobalt varieties for the range 0.65 K to 273 K. All standard capsule-type resistance thermometers designed for cryogenic service are filled with a helium heat-exchange gas. For temperatures above the depressed λ-point transition (e.g. ~1.3 K) of condensed He films, the gas-mediated self-heating coefficient should be a well-behaved decreasing function of temperature. The typical values for the self-heating coefficient range from ~0.01 mK/μW near 273 K to ~1 mK/μW near 1.3 K. For some thermometers, however, a self-heating anomaly can be readily observed in the region 40 K $\leq T \leq$ 70 K. We show that this anomaly can be modeled as the self-heating thermal resistance of a helium-air mixture. When a sufficient range of self-heating data is available, the relative proportions of helium and air can be extracted from the data. We also show that over the 15-year period that data has been taken on our thermometers, this air contamination is a stable function of time, indicating that the contamination is most-likely an original manufacturing defect rather than something due to a leak. Also, the contamination does not appear to affect the stability of the thermometers' calibrations. The experimental aspects of the self-heating measurements are described as well as certain aspects of the thermal transport modeling.

POSTER SESSION II

Calibration Procedures and Facilities

Tuesday
10:45 to 11:30
and
14:15 to 15:00
Foyer

CALIBRATION OF STANDARD THERMOCOUPLES BY COMPARISON METHOD IN FLUIDIZED BATHS

V. Karzhavin[1], S. Nenashev[2]
[1] *Tesey Ltd.,Obninsk, Russia*
[2] *Rostest, Moscow, Russia*
E-mail (corresponding author): ms@tesey.com

Modern calibration laboratories need reliable and high accuracy temperature measurement tools. In recent years demands to accuracy of temperature measurements in many industrial processes also became more strict. That's why aim of our investigations was to increase accuracy of calibration works made in laboratory of Tesey company, to give our customers standard thermocouples with low uncertainty of calibration. Russian official metrology rules for this moment allow to do calibration of standard thermocouples by comparison method in tube furnaces, but uncertainty of calibration results should be from 0.5 °C to 0.9 °C in temperature range from zinc freezing point to copper freezing point.

We have studied possibility of making calibration of standard thermocouples type S by comparison method in temperature range from 200 °C to 1100 °C with uncertainty not greater than 0.6 °C in full range. Calibration of thermocouples were made in two fluidized baths with different constructions. We have used standard thermocouples type S and standard platinum resistance thermometers as reference thermometers. To increase reliability of obtained data calibration were made at 5 temperature levels: tin, zinc, aluminum, argentum and copper freezing points. In article data about repeatability of calibration results for more than 10 thermocouples made from one batch of thermo electrodes is presented. It shown that repeatability is about 0.2 °C. The data about reproducibility of calibration result by comparison method and freezing points calibration results for 4 freezing point is presented. Reproducibility was checked for 5 thermocouples. Freezing point calibrations were made in laboratory of Rostest. It's shown that such reproducibility is about 0.3 °C. Obtained results allows to assert that it's possible to make calibration of thermocouples type S by comparison method with extended uncertainty from 0.4 °C to 0.6 °C in temperature range from 200 °C to 1100 °C. Basing on this result we plan to officially certify method of comparison calibration of thermocouples in fluidized baths with mentioned uncertainties.

CALIBRATION OF THERMOCOUPLES AND BROAD BAND RADIATION THERMOMETERS BY COMPARISON TO RADIATION THERMOMETRY

M. J. Martin, M. Zarco, D. Del Campo
Centro Español de Metrología (CEM), Tres Cantos, Spain
E-mail (corresponding author): mjmartinh@cem.mityc.es

A new calibration by comparison system for thermocouples and broad band radiation thermometers up to 1600 °C has been designed, characterized and setting up. This system is based in a $MoSi_2$ three zone furnace with a graphite blackbody comparator inside. Two interchangeable alumina tubes with different parts inside are used for thermocouples and radiation thermometers calibrations respectively. The reference temperature of the calibration is given by a standard radiation thermometer which disseminates the ITS-90 up to 2200 °C referred to the Cu fixed point.

Several noble metal thermocouples and infrared radiation thermometers with central wavelength near 1 μm have been calibrated and the uncertainty budgets have been obtained. The method has been validated using thermocouples calibrated at fixed points (Ag, Cu and Pd) and broad band radiation thermometers calibrated in Na heat pipes (up to 950 °C).

The calibration uncertainty obtained for thermocouples (considering an inhomogeneity of 7 μV) and broad band radiation thermometers (considering a typical size of source effect) is from 0,6 °C at 900 °C to 1,1 °C at 1600 °C.

The characterization of the system, the calibration procedure and the uncertainty calculus will be provided with detail.

MEASURING SYSTEMS FOR THERMOMETERS CALIBRATION IN LOW TEMPERATURE RANGE

A. Szmyrka-Grzebyk, L. Lipiński, H. Manuszkiewicz, A. Kowal, A. Grykałowska, D. Jancewicz
INTiBS, Wrocław, Poland
E-mail (corresponding author): A.Szmyrka@int.pan.wroc.pl

At INTiBS in Poland the national temperature standards for low temperature range between 13,8033 K and 273,17 K has been established and is maintained. The standard consists of four sealed cells for realization of gaseous fixed points – the triple point of hydrogen, neon, oxygen and argon produced by IMGC (presently INRIM). Metrologic parameters of these cells were tested during the MULTICELLS project realization between 2000 and 2003. These triple points are realized in an adiabatic condition. A parasitic heat to the measuring chamber of a cryostat is less than 10 μW. The triple points of mercury and water are realized in ISOTECH apparatuses. CSPRT - Leeds&Northrup – has participated in the CCT-K2.4 key comparisons. Resistance of thermometers is measured with the MI 6015 T resistance bridge.

The Laboratory of Temperature Standards at INTiBS calibrates CSPRTs at all low temperature fixed points and make calibration platinum resistance thermometers by comparison with calibrated standards between 77 K and 273 K. For this calibration a home-made cryostat or liquid bathes produced by Hart/Fluke, depends on a needed temperature range, are used. Digital thermometers could be calibrated too. For the thermometer parameters Superthermometer or Chub produced by Hart/Fluke are used.

A comparison method is applied for calibration of RhFe thermometers in the temperature range from 2,5 K up to 25 K. A control RhFe thermometer used for temperature value determination participated recently in the CCT-K1.2 comparison carried out by PTB. RhFe thermometers can be calibrated at the triple point of hydrogen and neon as well.

The Laboratory calibrates also resistance and digital thermometers above 273 K – up to about 400 K.
At the Laboratory a quality system has been maintained according to ISO 17025. The Laboratory was accredited by the Polish Centre for Accreditation in 2009.
The work is partially financed by the Ministry of Sciences and High Education in Poland under the project no KB/58/13484/IT1-B/U/08.

A CHANGEABLE APERTURE BLACKBODY FOR LOW TEMPERATURE REFERENCE SOURCE TO 245 K

H.-Y. Ko
CMS/Industrial Technology Research Institute, Hsinchu, Taiwan
E-mail Shelley_Ko@itri.org.tw

The CMS is developed a facility for infrared radiation thermometers in the low temperature range from 245 K to 363 K cover two liquid bath. The blackbodies with changeable apertures of 28 mm to 70 mm of diameter are constructed and characterized. Two blackbody cavities were separately immersed horizontally in two stirring liquid baths; dry gas is filling of the cavity opening aperture via a circular inlet at the top of the front end of the blackbody cavity to reduce dew on the surface. The cavity is made of oxygen-free copper with thickness less than 3 mm and coated with Nextel Velvet 811-21 no thicker than 100 mm as its black coating. The temperature of blackbody cavity is monitored using Platinum Resistance Thermometers (PRTs) which were calibrated according to the International Temperature Scale of 1990 (ITS-90). The effective emissivity of the cavity is maintained over 0.99 units for the range between 263 K and 363 K. For converging factor (k) equals to 2, the uncertainties of the whole standard blackbody system, without device-under-test (DUT), at 245 K and 363 K are 0.36 K and 0.11 K, respectively, which were also traceable to ITS-90.

CALIBRATION OF RADIATION THERMOMETRY FIXED POINTS USING AU/PT THERMOCOUPLES

F. Jahan, M. J. Ballico
NMIA, Lindfield, Australia
E-mail (corresponding author): ferdouse.jahan@measurement.gov.au

At NMIA, radiation thermometers are calibrated by comparison with a number of reference radiation thermometers which are themselves calibrated using fixed point cells on the ITS-90 temperature scale (In, Sn, Zn, Al, Ag and Au). The suitability of NMIA fixed point cells used for the SPRTs is evaluated by the comparison of ensembles of cells at each fixed point, and participation in international BIPM Key-Comparisons K3 and K4. However, the NMIA fixed point cells used for radiation thermometry are typically much smaller (only 110 mm in length) and the thermowell length immersed in the metal much shorter (85 mm) than those used for SPRTs. Further, the cell assemblies need to accommodate the F/10 viewing cone of the radiation thermometers, so significant temperature gradients exist near the top of the crucible. As a consequence, the conduction errors obtained using SPRTs are too large to be of practical use. We have developed a convenient methodology based on the use of a Au-Pt thermocouple, together with a protective tube assembly to reduce conduction errors. This allows the convenient measurement of the phase transition temperature traceable, at the 30 mK level, to the fixed point cells used at NMIA to realize and maintain the ITS-90 scale. As the measurements are made in-situ, the temperature environment and hence interface geometry and formation during melting and freezing is similar to that occurring in use with radiation thermometers. Results are presented for ITS-90 fixed points up to Ag, establishing formal traceability of radiation thermometry fixed point cells NMIA's primary ITS-90 cells.
In alphabetical order:

Keywords: Au-Pt Thermocouples; conduction error; fixed point; melt-freeze.

A CRYOSTAT FOR AUTOMATED milli-Kelvin LEVEL THERMOMETER CALIBRATIONS from -202 to 250 °C

M. J. Ballico
NMIA, Lindfield, Australia
E-mail (corresponding author): mark.ballico@measurement.gov.au

The National Measurement Institute of Australia (NMIA) has developed a vacuum cryostat capable of calibrating precision electronic thermometers with a transfer error less than 2 mK over the range -202 °C to 250 °C. The calibration of precision temperature measurement probes such as platinum resistance thermometers (PRTs) is usually performed in circulated fluid baths to achieve mK-level calibration uncertainties, and requires the use of several baths to cover the commonly-used range –80 °C to 250 °C. Below –80 °C, dry-well systems cooled by liquid nitrogen are available down to –196 °C, but achieve poorer uniformity and stability. The increased use of cryogenic preservation in the bio-medical area has seen an increase in demand for precision calibration of electronic thermometer systems, in particular down to a few degrees below the boiling point of nitrogen (–196 °C). This has prompted NMIA to develop a new design of dry-well calibrator, based around a 380 mm long, 50 mm diameter, oxygen-free copper block insulated by gold-plated radiation and guard shields. Temperatures down to –202 °C are achieved by controlling the flow of liquid nitrogen through a restricting orifice into an evacuated heat-exchanger. Computer control of the nitrogen flow and of several immersion heaters achieve a temperature stability of a few mK at all temperatures over the operational range, requiring typically 60 minutes to equilibrate at each new setpoint. Radiative transfer limits operation to 250 °C where the uniformity is 0.5 mK/cm (and becoming negligible at lower temperatures). A significant design innovation is the thermometer entry region, which has a purge system to keep the wells free of condensed moisture or atmospheric gases without the need for a seal. As the block is only 50 mm from the face of the cryostat, thermometers as short as 250 mm can be calibrated. The system is now in regular use at NMIA providing fully automated calibrations of precison temperature measurement systems without the need to use multiple temperature enclosures.

USE OF GRAPHITE ELEMENTS IN PYROGRAPHITE HEATER OF BB3200/BB3500 TYPE FURNACES FOR HTFP APPLICATIONS

B.B. Khlevnoy, I.A. Grigoryeva and N.A. Ibragimov
VNIIOFI, Moscow, Russia
E-mail (B.B. Khlevnoy): khlevnoy-m4r@vniiofi.ru

BB3200/3500 furnace is one of three furnace types that are used for high temperature fixed points (HTFP) applications [1]. One of the advantages of the BB-type furnace is its extremely high working temperature up to 3300 °C due to use of pyrolytic graphite (PG) as material for a heater. However, high cost of PG limits a number of users and applications of the furnace.

Another advantage of the BB-type furnace is a ring design of the heater, which makes it very flexible. The structure of the heater can be changed easily. For instance, some PG rings can be partly (or even totally) replaced by peaces made of usual graphite. Such replacements could significantly reduce the cost of some applications of the PG furnace or even improve its characteristics. The present paper describes examples of using usual graphite in the PG heater.

The first example is replacing the central (therefore, hottest and faster degrade) half of the PG heater rings of the BB3200pg by a graphite thin-wall tube for cells filling. The tube with inner diameter of 38 mm (which is equal inner diameter of a normal BB3200pg heater) and wall thickness of 2 mm made of high resistance graphite (nominally 15 μΩm at room temperature) enables to reach the Re-C temperature at current of 550 A and the W-C temperature at the current less than 700 A, which is within the limits of a regular power supply. A few Re-C cells were filled using such a heater without any evidence degrades of the tube.

Another example is related to uniformity of the furnace. The PG heater rings is usually ordered in such a way that the lowest resistance rings are placed in the center and the highest resistance ones are placed at the ends of the heater to achieve the lowest gradient [2]. However, the difference between the highest and lowest resistance is not enough. In this case normal graphite rings can be placed in between PG rings one by one in the central part.

The paper will describe more details of these and other examples.

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[2] B. Khlevnoy, M. Sakharov, S. Ogarev, V. Sapritsky, Y. Yamada, K. Anhalt, Investigation of Furnace Uniformity and its Effect on High-Temperature Fixed-Point Performance, International Journal of Thermophysics (2008) v.29, N.1, p.271-284 // TEMPMEKO 2007 Proceedings, Issue 2 // DOI 10.1007/s10765-007-0347-z

DEVELOPMENT OF ONBOARD GALLIUM FIXED-POINT BLACKBODY FOR PROSPECTIVE IN-FLIGHT CALIBRATIONS

A. Puzanov, L. Samoylov, A. Burdakin, B. Khlevnoy
VNIIOFI, Moscow, Russia
E-mail (Andrey Puzanov): a_pu@list.ru

The development and the use of space-based blackbodies that incorporate the phase transition phenomenon is of great interest for metrology as a mean for calibration of onboard radiometric instruments. This work deals with a projected low-temperature Gallium blackbody intended for testing its performance under zero-gravity conditions.

The aperture of the projected blackbody is 10 mm, emissivity – about 0.9997, and the mass of working substance – about 500 g. The blackbody is operated in fully automated mode by microprocessor-based control. Heating/cooling of the blackbody and, thereafter, melting/freezing of Gallium is realized by means of thermoelectric elements with total electric power about 100 watts.

The Gallium melting/freezing plateaus are indicated with the help of 100 ohm PRTs energized by current of 1.0 mA magnitude. The PRTs' voltage is gained by precision amplifiers and after that comes to measuring circuit with temperature resolution of the order of 0.001 mK.

The prospective space experiment aimed at facilitating the development of onboard standard fixed-point blackbodies will be a significant step in a chain of projected works for the purpose of establishing space-based radiometric instruments calibration system.

METHOD FOR EFFECTIVE CALIBRATION OF TEMPERATURE LOGGERS WITH AUTOMATED DATA SAMPLING AND EVALUATION

S. Ljungblad, L. E. Josefson, M. Holmsten
SP, Borås, Sweden
E-mail (corresponding author): sara.ljungblad@sp.se

A highly automated calibration method for temperature loggers is presented. By using an automated procedure, a time and cost efficient calibration of temperature loggers is made possible.

The method is directed at loggers that lack the function/property of direct reading from a display. This type of logger has to be connected to a computer for the setting up of the measurement and again for collection of the measurement results. Calibration normally takes place in liquid baths, and for loggers which have external sensors, only the sensor is normally placed in the bath. Loggers with internal sensors are protected from the liquid by placing them in an exterior plastic or metallic cover and there after the entire logger is placed in the bath. A digital temperature meter measures the reference temperature of the bath and transmits it to a computer by way of Bluetooth. The developed calibration programme controls the logger software and thus the communication protocol of the logger software does not need to be known.

The previous method, with manual handling of start and termination of every measuring sequence, evaluation of the resulting data and its corresponding uncertainty components, can be replaced by this automated method. Both the logger and reference measurement data are automatically read once the logger has been connected to a computer after the calibration, and the calibration programme started. The data is then evaluated automatically and by statistical analysis of confidence coefficient and standard deviation, the temperature plateaus that the calibration includes are identified. If a number of control parameters comply with requirements, correction, resolution and short term stability are calculated for each calibration temperature. The calculated values are saved in a data base, along with information about what instruments were used during the calibration, after which calibration certificates and measurement uncertainty calculations can be generated. Up to ten loggers can be connected at the same time and are then evaluated in sequence.

The paper will show that the system can correctly analyze different types of measurement sequences and what measurement uncertainties are associated with this.

ESTABLISHMENT OF NIMT ZINC FIXED POINT CELL

C. Yaokulbodee[1], U. Norranim[1], J. V. Widiatmo[2], K. Yamazawa[2], J. Tamba[2]
[1] *National Institute of Metrology (Thailand), Pathumthani, Thailand*
[2] *National Metrology Institute of Japan, Tsukuba, Japan*
E-mail: charuayrat@nimt.or.th, janu-widiatmo@aist.go.jp

One of the research programs for Thermometry Metrology Department at National Institute of Metrology (Thailand), NIMT, is establishment of its own fixed point cells. Among the fixed point cells adopted for the realization of the International Temperature Scale of 1990 (ITS-90), NIMT has chosen the zinc fixed point to start the program. The fabrication and the initial evaluation of the zinc fixed point cell were conducted at the National Metrology Institute of Japan, NMIJ. The cell fabrication was following the design and procedures developed by the NMIJ.

In the cell fabrication, a 6N-nominal purity zinc metal cylinder ingot was used. The metal ingot was collected in a graphite crucible under an argon gas atmosphere. The new fixed point cell was compared with the old fixed point cells already owned by NIMT, namely, one open type cell and one sealed type cell by direct cell comparisons. Since the ingot was equipped with a detail impurity element analysis, it is possible to calculate the effect coming from the existence of the impurities based on, for example, the sum of individual estimates (SIE) method. This effect can then be used to correct for the influence impurities on the realization of the temperature fixed point. Using such cells, it is expected that the dissemination of temperature standard based on the ITS-90 will be possible with a reduced uncertainty.

TRACEABILITY AND QUALITY CONTROL IN A REFERENCE RADIOMETRIC CALIBRATION PROGRAM

F. E. Liebmann[1], M. J. Coleman[2], T. J. Wiandt[3]
Fluke – Hart Scientific, American Fork, Utah, U.S.A.
E-mail: frank.liebmann@fluke.com

In the world of radiometric thermometry, quite a bit of progress has been made in the past few years at the national laboratory level. Fluke – Hart Scientific has been able to leverage this progress to set up its own radiation thermometry program to measure traceable temperatures from–15°C to 500°C. This program is based on a set of traceable variable temperature blackbody cavities covering this entire temperature range. It uses this traceability in its radiometric calibration of flat-plate infrared (IR) calibrators. These flat-plates are used for the calibration of hand-held IR thermometers. The radiometric calibration of the flat-plate calibrators uses a commercially available radiometric transfer standard with a pyroelectric detector. The radiometric transfer is between the variable temperature cavities and the flat-plates. Both the transfer standard calibration and the flat-plate radiometric calibration have received National Voluntary Laboratory Accreditation Program (NVLAP) accreditation.

This paper gives a description of the traceability for the Fluke – Hart Scientific radiometric calibration program. It discusses the standards used for both the transfer standard calibration and the flat-plate radiometric calibration. The paper gives a schematic description of both of these processes to include detail of the cavity geometry and measures taken to maximize cavity uniformity. Discussion is made on the transfer standard uncertainty budget and steps that have been taken to make the uncertainty budget conform to the International Bureau of Weights and Measures (BPIM) Working Group 5 standard. The paper also briefly discusses the flat-plate radiometric calibration uncertainties. It then gives brief detail of transfer standard drift management and other steps to ensure the quality of these calibrations.

CAN SALT BATHS BE USED IN TEMPERATURE RANGES BETWEEN 180 °C TO 550 °C WITH UNCERTAINTIES OF LESS THAN 30 m°C

F. M. P. Alper, A. T. İnce and M. Aiordachioaiei
Department of Physics, Yeditepe University, Istanbul, Turkey
E-mail: mpatan@yeditepe.edu.tr

One of the most important characteristics differentiating temperature calibrations from other calibration fields is the requirement for overlapping measurement systems in order to perform an efficient calibration. The increase in the temperature range of a calibration process, increases the number of bath requirements during the process. For example, to calibrate a thermometer with a measurement range above 0 °C to 95 °C, only requires a water bath however, in the thermometer measurement range –60 °C to 550 °C, generally alcohol, water, oil, and salt baths or block calibrators must be employed. If there is a need for measurements with less uncertainity, liquid baths are preferred. In temperature calibrations, salt baths with wider calibration temperature ranges than liquid baths are more common. Salt baths provide a working range of 400 °C (180 °C to 550 °C). They also have a more stable and homogeneous temperature distribution than dry block calibrators or furnaces. These properties should ensure the advantage of lower measurement uncertainity.

In this study, investigations of the uncertainity at different salt bath temperatures in the range 180 °C to 550 °C are carried out. Results show that using the salt baths in this wide range of temperature, provides opportinitues to calibrate referance or/and working thermometers with an uncertainty below 30 m°C. The main components of uncertainty for utilising a salt bath over a wide temperature range will also be discussed.

TEMPERATURE PROFILE CHARACTERIZATION OF BATHS IN THREE DIMENSIONS

M. K. Torun and A. T. İnce
Department of Physics, Yeditepe University, Istanbul, Turkey
E-mail: quantumsicramasi@hotmail.com

Traceable temperature measurements require that temperature sensors should be regularly calibrated with respect to the internationally agreed temperature standards . To do this, two methods are used i.e comparison calibrations and fixed point calibrations. The latter is the most common method used by secondary temperature calibration laboratories. Liquid baths are the main calibration equipment that directly affect the measurement uncertainty in temperature calibrations. Therefore, the characteristics of baths need to be well investigated and understood.

The temperature profile and stability of liquid baths are one of the most important contributions to the calibration uncertainty of platinum resistance thermometers, digital thermometers, liquid-in glass thermometers, thermocouples and etc.

In general, commercial baths only present a two-dimensional bath profile or sometime just a stability of the baths are given. However, thermometers, thermocouples and etc. are immersed into the body of the bath. Thus the depth or z axis is also very important.

In this work a measurement system has been designed using three-stepper motors and a data acquisition system (DAS). The DAS is developed using object oriented algorithms called a three dimensional (3-D) scanning system. The 3-D scanning system is homemade and used in conjunction with a Hart Scientific reference platinum thermometer, the temperature profiles and stability of several types of baths: water, oil and salt were carried out in the temperature range 30 °C to 450 °C. This will then contribute towards a more accurate uncertainty budget.

A NEW BASIC METHOD TO CALIBRATE IR THERMOMETER CALIBRATORS

D. Cardenas-Garcia, S. J. Luyo-Alvarado, H. Rodriguez-Arteaga, E. Mendez-Lango
CENAM, Querétaro, México
E-mail (corresponding author): dcardena@cenam.mx

This paper presents a basic method for calibrating infrared radiation sources known as flat disc calibrators (FDCs) based on radiation energy conservation, the FDC emissivity, and the correction of the radiance temperature value due to different wavelength ranges for.

An FDC has a black surface that serves as a target for IR temperature measurement. FDC´s are very popular because they are easy to operate, transport, maintain and there is an increasing need to calibrate inexpensive IR thermometers that work in the 8 µm to 14 µm wavelength range. A number of those calibrators are considered to have a nominal emissivity of 0.95 in the specified wavelength range because most of IR thermometers that are calibrated with them are usually set for that emissivity value and work in the mentioned wavelength range.

We use a blackbody to calibrate the radiation thermometer that is going to be used as reference. Then, we calculate a set of temperatures corresponding to a gray body with 0.95 emissivity, and calibrate the radiation thermometer at 0.95. It may happen that the radiation thermometers used for calibration of FDCs as reference instruments work in a slightly different wavelength range as compared to nominal “operating range” of the FDC. Thus, when a calibration is carried out we deal with two problems: one is related to the FDC nominal emissivity value and the other to a discrepancy of working bands between the reference IR thermometer and the FDC nominal operating range.

An FTIR is used to measure the spectral radiance emitted by the FDC, so we can compare it with that of a black body cavity at the same temperature. With the experimental data, we calculated the normal spectral emissivity in the wavelength range of 8 µm to 14 µm from ambient temperature to 500 °C. The results indicate significant deviations of about 1% to 3% from the nominal value of emissivity of 0.95 leading to temperature errors of about 7 °C to 20 °C, thus corrections should be done for the radiation temperature measurements with these instruments.

To calibrate the FDC using an IR thermometer, we use the FDC calculated emissivity, and then we make the correction to the temperature reading due to the different wavelength range.

PRACTICAL HIGH-TEMPERATURE FURNACE FOR THE APPLICATION IN THERMOMETRY AND RADIOMETRY

K. Anhalt[1], L. Bünger[1], E. Schreiber[2] and J. Hartmann[1]
[1] *Physikalisch-Technische Bundesanstalt, Berlin, Germany*
[2] *KE Technology GmbH, Stuttgart, Germany*
E-mail (corresponding author): klaus.anhalt@PTB.de

High-temperature furnaces have become important tools in modern measurement technology, they are used as blackbody radiators for the realization of spectral radiance and to operate temperature fixed-points for the realization of the temperature scale in practical thermometry over a wide temperature range.

In addition to radiation thermometers, which measure the average temperature of a small area, thermal imagers are used at high temperatures for a precise measurement of a two dimensional temperature distribution. The calibration of a thermal imager requires a wide aperture radiator.

This paper describes the development and characterisation of a black-body radiator for temperatures up to 3000 °C. The high-temperature furnace is equipped with an up to 50 mm wide aperture and a special cavity-design, which allows to optimize the temperature homogeneity along the furnace axis.

The optimisation of the furnace cavity and operational characterisation of the furnace has been supported by finite element simulations.

SIMULATION OF A HIGH TEMPERATURE FURNACE FOR OPTIMISATION OF THE TEMPERATURE CONTROL

E. Schreiber
KE Technology GmbH, Stuttgart, Germany
E-mail (corresponding author): schreiber@ke-gmbh.de

The optimization of the temperature control of a furnace is very important for its application in radiation thermometry. Novel simulation software simplifies this optimization by means of fast simulation of the closed loop response.

The optimization of the temperature control of a furnace can be a time-consuming task due to the slow response of the furnace. In radiation thermometry, large thermal masses such as fixedpoints are placed in the furnace tube, and time constants are frequently larger than one minute even at highest temperatures. Using a time-dependent simulation of the furnace, the development of the temperature control of a high temperature furnace can be speeded up without the risk of furnace or fixed-point damage by improper control. However, commercially available simulation software is designed for best adaptability to a wide range of scientific problems. Due to this design rule, the time-dependent furnace simulation with multi-purpose software is too slow for the development of the temperature control. A drastically higher simulation speed can be obtained if a specialized simulation program is developed in particular for the time-dependent simulation of a thermal system with radiative and conductive heat transfer.

The novel finite difference simulation software uses a number of measures to improve simulation speed. The restriction to rotational symmetry yields the most important acceleration. The calculation of the radiative heat transfer, which dominates at high temperature, is separated in two major steps, firstly the calculation of the geometry factors (configuration numbers) between the finite elements of the furnace, and secondly the calculation of the heat exchange between these elements in dependency of the individual temperatures during simulation. The geometry factors are pre-calculated before starting the simulation. Heat exchange terms are not calculated if the geometry factor is too small for a significant contribution to the heat transfer. Diffuse reflection can be optionally considered. The emissivity, the thermal and electrical conductivity, and the heat capacitance are modeled as function of the temperature. The resistive furnace heating is simulated in dependency of the variable heater current, so that the closed loop behavior of the furnace temperature control can be analyzed. Assuming a furnace modeling with about 500 cylindrical finite elements, the simulation speed is at least 10 times faster than real time even on an older PC.

CALIBRATION BY COMPARISON OF PLATINUM RESISTANCE THERMOMETERS USING SLOW RESISTANCE BRIDGES

V. Batagelj, J. Bojkovski
MIRS/UL-FE/LMK, Ljubljana, Slovenia
E-mail (corresponding author): valentin.batagelj@fe.uni-lj.si

Platinum resistance thermometers (PRTs) are calibrated at the highest level in fixed points, as specified in the International temperature scale ITS90. However, in order to reduce cost and time, platinum resistance thermometers can also be calibrated by comparison. In the temperature range from –50 °C to 300 °C it is possible to achieve uncertainties of down to 5 mK, which is 3 to 5 times larger compared to calibration in fixed points.

PRT is calibrated by comparison by comparing its reading with the reading of a reference thermometer, placed at the same temperature inside the temperature-controlled calibration medium. The reference thermometer is commonly also a resistance thermometer and both thermometers are measured with the same resistance bridge. The resistance bridge uses a switching matrix (scanner) to switch between both thermometers and alternately take resistance readings. This procedure is applicable only to fast resistance bridges, which are capable of taking a stable resistance reading within 10 to 20 seconds from the connection of the thermometer.

High accuracy resistance bridges are often too slow to meet the above criteria, either because it takes too long to produce a stable reading or because they require an initialization procedure after the switching of the thermometer. This means that a simple alternating procedure is not applicable, but such resistance bridges can still be used in calibration by comparison, if proper procedures are developed, implemented and validated. Such procedures are inherently more complex and therefore require the development of custom-made calibration software, which controls the bridge and also performs the necessary data post-processing.

In the paper, several procedures of calibration by comparison of PRTs with slow resistance bridges will be presented and validated with analytical and experimental results. The procedures include simple approaches such as taking a set of measurements with the reference PRT, followed by a set of measurements with the PRT under calibration or more complex approaches, which may include also the direct measurement of the ratio between the two PRTs. Each procedure presents a different compromise between the accuracy, reliability, complexity and measurement time.

THE DEVELOPMENT OF A DEVICE FOR ON-SITE CALIBRATION OF THERMOMETERS

M. J. De Groot
MartinDeGroot Consultancy, Vught, The Netherlands
E-mail (corresponding author): mdg@martindegrootconsultancy.nl

Prototype devices have been designed and constructed for the calibration of thermometers on site. The device is an alternative to block calibrators. The uncertainties that can be obtained using this device compare favourably with those of block calibrators. The system has reduced stem conduction losses through better adaptation to heat flows in practice. Presently, the range of the prototype applications is limited to between –90 °C and +150 °C.

The paper shows details on the construction and discusses evaluation tests, calibration results and gives example uncertainty budgets.

CALIBRATION OF PLATINUM RESISTANCE THERMOMETERS (PRTs): CONTROL AND AUTOMATION OF THE CALIBRATION SYSTEM

D. Nyamba
Inspection de la Qualité et de la Métrologie (IQM),
Ouagadougou, Burkina Faso
E-mail : dnyamba@yahoo.fr

In our Sub region the reference platinum resistance thermometers (PRTs) are calibrated at the Regional Calibration Laboratory of Thermometry. The thermometry laboratory of Burkina Faso as been designate by the project PTB/UEMOA as the regional calibration laboratory of West Africa.

The standards platinum resistances are calibrated by comparison in bath on the International Temperature Scale of 1990. The automated calibration system done with software LabVIEW will also be described in this paper.

The measurement of temperature plays a key role in checking the quality of products. For many applications thermometers (PRTs, thermocouple, Liquid in glass) are commonly used.

That why in our sub region the economic union as establishes in Burkina Faso a Regional Calibration Laboratory for the calibration of PRTs. This calibration is done by the method of comparison.

The calibration system for the beginning covers the range from 0 °C to 80 °C.

The measurement system witch is used to measure the reference temperature in the baths includes: a standards platinum resistances (SPRT), a resistance-ratio bridge ASLF700A, a 100 ohms Tinsley ac/dc resistor and an 8½ digit multimeter for the measurement of resistance.

A calibration laboratory of temperature as been established in West Africa. He needs to be performed for internationally recognized measurement of thermometers instruments in West Africa.

EUTECTIC Ga-In AS CALIBRATION REFERENCES FOR CALIBRATION LABORATORY AMBIENT THERMOMETERS

E. Mendez-Lango
CENAM, Queretaro, Mexico
E-mail: emendez@cenam.mx

Ga-In eutectic phase transition temperature has been used for calibration of low accuracy thermometers at about 15.7 °C.

We decided to reproduce this point with two purposes: to provide a reference to calibration laboratories for calibration of their ambient thermometers and to develop an experimental technique to handle eutectic experiments.

Samples of Ga and In of 6N purity were used. Eutectic concentration of In in Ga is 20.5% weight. When preparing this experiment we decided add In to Ga sample "step by step" with steps of about 2%.

When realizing the experiments a well defined phase transition was observed at 15.7 °C even though the concentration was far from the eutectic one and another well defined phase transition was detected at 24 °C.

Realizations of both points require very simple and common laboratory apparatus: a flask with ice, a water bath at above 32 °C and a Styrofoam pouch. To prepare the "regular" eutectic point (15.7 °C), the cell is placed in ice until it reaches a near 0 °C value measured with a PRT. Then it is placed in the Styrofoam pouch and let it warm at room temperature. Phase transition is reached few minutes later and the melting range is about 0.05 °C. The melting time depended from the concentration but it lasted at least half of an hour.

To prepare the "high" temperature point, the cell was placed in a water bath at about 40 °C for several minutes until it reached the bath temperature. Afterwards the cell was placed in the Styrofoam pouch and let it cool down until it reached the "freezing" point at about 24 °C.

Reproducibility of both transitions was studied while a realization technique was developed. Estimated uncertainty for these two phase transitions is less than 0.1 °C.

By assessing temperature values to these phase transitions a scale of two fixed points may be realized in one cell.

FREEZING POINT TEMPERATURE OF H_2O-NaCl SOLUTIONS FOR CALIBRATION OF CRYOSCOPES

E. Mendez-Lango[1], L. Lira-Cortes[1], R. Ibarra-Quiñones[2]
[1] *CENAM, Queretaro, Mexico*
[2] *Universidad de la Sierra, Moctezuma, México*
E-mail: emendez@cenam.mx

Cryoscopes are instruments to measure the freezing point of a given substance and they are used for purity determination. In milk industry cryoscopes are used to identify if some water has been added to milk: by adding water to milk its freezing point rises.

Milk has about 87% of water and a wide melting range, thus "the freezing point" is defined as the observed "equilibrium" temperature after recalesence where equilibrium means that temperature value does not change more than 0.5 mK during 20 seconds.

Calibration of cryoscopes is done by using reference solutions that its freezing value is stated to be in ±1 mK range and the expected repeatability of the freezing point, realized in the cryoscope, is about ±2 mK.

For milk test cryoscopes calibration two types of solution are used as reference standards, solute may be sucrose or NaCl. We dealt with NaCl solutions.

Concentration of NaCl in water is stated in the standards to be used to certain temperature values but it was no found published data of temperature determination for these solutions.

We prepare four solutions with nominal freezing pints of –0.300 °C, –0.406 °C, –0.512 °C and –0.598 °C values used for preparation of reference solutions. The melting curves of these solutions were measured in an adiabatic calorimeter. The obtained melting curve for each case was used to assess the freezing point: extrapolated temperature value when solid phase tends to zero (liquidus point).

Experiment description and results are shown.

PRACTICAL CALIBRATION OF PLATINUM RESISTANCE THERMOMETERS IN THE RANGE –190 °C TO 0 °C

J. De Lucas, R. Benyon
Instituto Nacional de Técnica Aeroespacial (INTA), Torrejón de Ardoz. Spain
E-mail (corresponding author): delucasvj@inta.es

In the temperature range between the triple point of Argon and the freezing point of Aluminium, INTA maintains the traceability of the Institute and the Ministry of Defence, using fixed point cells, together with Standard Platinum Resistance Thermometers (SPRT) as interpolation devices, with traceability to the realization of the International Temperature Scale (ITS-90) of Centro Español de Metrología (CEM)

In order to offer the lowest possible uncertainty, SPRTs are used as standards to calibrate other thermometers, (including Platinum Resistance Thermometers that do not necessarily meet the ITS-90 requirements for use as interpolation devices), by comparison in temperature controlled liquid baths. Using conventional fluids, in the range from –100 °C to 550 °C, expanded measurement uncertainties (for a confidence level of approximately 95%), of between ± 10 mK and ± 30 mK, can be routinely achieved.

The increasing demand for calibrations with lower uncertainties and wider ranges, as a result of the technical requirements of the Defence and Aerospace sectors, and the better availability of commercial instrumentation, have triggered INTA to contract the development and manufacture of precision comparators that jointly cover the range from –190 °C to 660 °C.

The results obtained in the characterization of the low temperature comparator and its performance in relation to the calibration of metal and borosilicate sheathed SPRTs calibrated at the ITS-90 fixed points, are presented and discussed.

The principal influence quantities are addressed and the estimation of measurement uncertainty supporting the measurement capability recently accredited by ENAC, are presented and discussed. (The current scope of accreditation is available on http://www.enac.es).

THE CALIBRATION OF DATALOGGERS FOR THE CHARACTERIZATION OF STERILIZATION AUTOCLAVES

R. Benyon, J. De Lucas, H. Fuentes
Instituto Nacional de Técnica Aeroespacial (INTA), Torrejón de Ardoz. Spain
E-mail (corresponding author): benyonr@inta.es

The availability of high-quality dataloggers for the measurement of temperature and pressure have seen the proliferation of the use of these devices in the characterization and validation of sterilization autoclaves. It is common practice to calibrate these devices by comparison in liquid baths, at atmospheric pressure, using Standard Platinum Resistance Thermometers (SPRT) as the reference standards.

In order to assess the validity of the conditions of calibration and the technical specifications for the pressure coefficient of the temperature indication of the dataloggers used by the INTA Temperature and Humidity Laboratory in site characterizations of autoclaves, the complete characterization of the loggers in the range from 0 °C to 125 °C and atmospheric to 0.3 MPa absolute pressure.

The experimental set-up and the results obtained, are presented and discussed in the context of the manufacturer's specifications. Recommendations on the calibration of these devices and the estimation of the contributions to the expanded uncertainty of measurement for the characterization of autoclaves are presented and discussed.

A LOW TEMPERATURE COMPARATOR FOR CALIBRATION OF INDUSTRIAL THERMOMETERS IN THE RANGE –215 °C TO –60 °C

C.K. de Bruin-Barendregt, R. van Breugel, R. Bosma and A. Peruzzi
VSL, Delft, the Netherlands
E-mail (corresponding author): rbosma@vsl.nl

A low temperature comparator was developed for the purpose of calibrating industrial thermometers in the range –215 °C to –60 °C. This comparator will replace an existing system which uses a controlled liquid nitrogen flow. The valve used to control the nitrogen flow needed frequent service. To simplify the operation of the comparator and to improve the temperature stability, a new system was developed based on conventional cryostat principles, where the copper comparator block is located inside a vacuum chamber immersed in liquid nitrogen. Two temperature control loops were implemented: one loop to control the temperature of a shield in between the block and the vacuum chamber, another loop to control the temperature of the block itself. The operating range of the comparator can be extended below the boiling point of nitrogen by pumping on the liquid nitrogen bath.

In this paper we will report on the status of the development of this comparator, with particular emphasis on:

- the design of the comparator,
- the temperature control system,
- the evaluation of the stability and uniformity.

REALISATION OF THE INTERNATIONAL TEMPERATURE SCALE AT ČMI

R. Strnad, M. Šindelář
CMI, Prague, Czech Republic

This article presents the realization of the international temperature scale ITS-90 from triple point of mercury to freezing point of silver. In the first part will be presented the current status of the laboratory with showing the uncertainty budget for all of the cells, including the uncertainty budgets of the calibration schemas for calibrating of the standard platinum resistance thermometers (SPRTs) and high temperature standard platinum resistance thermometers (HTSPRTs).

In the second part of this article will be presented the current validation procedure for maintaining of the temperature standards including all of the parts of measurement chain. The investigation of the sub range inconsistency of the monitoring SPRT will be also presented.

The third part will be focused to the development of the laboratory setup for creating the primary and secondary fixed points at CMI. It includes the laminar box with possibility of implementing of the pure argon atmosphere and chemical equipment for washing and preparing of the fixed point materials. It will be also showed the setup for maintaining of the open cells at CMI.

The next part of this article will be focused on the status of the development of the laboratory setup for the calibration of the infrared thermometers for the past two years. The laboratory of the CMI focused on the low temperature range (below 1000 °C), where the most of the demands for the calibration from the industry occurs. The international temperature scale to freezing point of silver is maintained by means of the fixed points. To have the possibility to compare new developed laboratory for infrared thermometers all of the setup was creating in the way to have the possibility to compare the calibration results of SPRT (or thermocouple) on the standard fixed points calibration scheme with the calibration in fixed point black bodies. The heat pipe furnaces of Sodium and Water was used for variable temperature black bodies including the commercial black body fixed points of In, Sn, Al, and Cu was implemented. For the industrial infrared thermometers is used the variable temperature blackbodies with different geometries. As a transfer standard is used infrared thermometers Heitronic with wavelengths of 3.9 µm and 8-12 µm. It will be also present the uncertainty budget of the calibration scheme used.

THE LABORATORY SETUP FOR CALIBRATING OF THE SURFACE TEMPERATURE SENSORS

R. Strnad, M. Šindelář
CMI, Prague, Czech Republic

The calibration of the surface temperature sensors depends on number of influencing parameters including the calibrated sensor, design of the surface, ambient environment and methods of the calibration. The results also often depend on the operator experiences. In this article will be presented the laboratory setup for surface temperature calibration at ČMI. The design is based on the block of compact material with 4pc of industrial platinum resistance thermometers (IPRT) to measure the temperature gradient along the block. These measurements were used for extrapolation of the surface temperature.

In the experimental part will be showed the comparison of this block with the commercial system used at ČMI. It will be also shown the influence of different block materials and different designs to the results. The investigation on the external environment was made. The results were compared with numerical analysis and the uncertainty budged will be presented.

POSTER SESSION II

Uncertainty Estimation

Tuesday
10:45 to 11:30
and
14:15 to 15:00
Foyer

UNCERTAINTY BUDGET FOR THE NIST HYBRID HUMIDITY GENERATOR

C.W. Meyer, W.W. Miller, D.C. Ripple and G.E. Scace
National Institute of Standards and Technology, Gaithersburg, MD, USA
E-mail (corresponding author): christopher.meyer@nist.gov

A new humidity generator has been constructed at the National Institute of Standards and Technology (NIST) and is now fully operational. The NIST Hybrid Humidity Generator (HHG) is the NIST primary humidity generation standard for frost/dew points from –70 °C to +85 °C using calibration gas flows up to 150 standard liters per minute. The HHG combines the twopressure and divided-flow humidity generation techniques (hence the name "hybrid"). The centerpiece of the HHG is a heat-exchanger/saturator that is immersed in a temperaturecontrolled bath stable to within 1 mK. A pre-saturation process minimizes sensible and latent heat loading on the final saturator. For dew/frost point temperatures above –15 °C, the two-pressure principle is employed. For frost points at or below –15 °C, the divided-flow method is used. For this method, the water-vapor/air mixture is produced by mixing metered streams of moist air produced by the two-pressure principle with purified, dry air; here, the HHG saturates the wet air stream at a temperature close to the water triple point, reducing the uncertainty of the water vapor pressure.

We provide here a detailed uncertainty budget for the HHG. The total expanded (k=2) uncertainty is estimated for HHG generation of the following quantities: dew/frost point, mole fraction, mass ratio and relative humidity. The total uncertainty is estimated separately for the cases of two-pressure mode and divided-flow mode. First we present equations relating the total uncertainty of each quantity to the uncertainty elements for the HHG. We then provide a table listing all the uncertainty elements and their estimated uncertainty values, describing the basis for these values. Finally we plot the uncertainties of each quantity as a function of the quantity.

When the HHG is operated in two-pressure mode, the dominant experimental uncertainty is usually from the pressure measurement of the final saturator; the uncertainty of the saturator temperature is dominant when it is above ~70 °C due to significant temperature non-uniformities in the saturator. When the HHG is operated in divided-flow mode, the dominant experimental uncertainty is from the flow measurements of the gas streams. In both modes the uncertainty of the water enhancement factor is significant and is often the dominant uncertainty element. For dew/frost point temperatures, the uncertainty budget yields a total expanded uncertainty (k = 2) of less than 0.025 °C for dew/frost point temperatures above –60 °C. For mole fraction, the budget yields a total expanded relative uncertainty of less than 0.2% for mole fractions above 2×10^{-5}. When the uncertainty budget for the HHG described above is used, comparisons of moisture generated by the HHG with that produced by other NIST humidity generators show agreement to within the combined expanded uncertainty. Comparisons of moisture generated by the HHG with that measured by the NIST gravimetric hygrometer show similar agreement.

DEW/FROST-POINT UNCERTAINTY ANALYSIS OF THE SWSY-S HUMIDITY GENERATOR

J. Gong, Y. Zhang, Y. Z. Han
Changcheng Institute of Metrology & Measurement, AVIC, Beijing, China
E-mail (corresponding author): gongjuan@ tsinghua.org.cn

A new frost-point generator SWSY-S was constructed and evaluated in order to extend the range of the humidity standard to –80 °C. This paper details the design of the Dew/Frost-point humidity generator. In the design, special attention has been given to a system of pre-saturator and saturator. SWSY-S humidity generator design favor the single-pressure principle without any recirculation. This paper presents the uncertainty of dew-point and frost-point uncertainty analysis for SWSY-S generator manufactured by Changcheng Institute of Metrology & Measurement, AVIC. The improvements in design and high performance instruments have enabled a smaller uncertainty in generated dew-point and frost-point. This uncertainty is now 0.05 °C at the 10 °C dew-point, and rising to 0.09 °C at the –80°C frost-point.

UNCERTAINTY PROPAGATION FOR PLATINUM RESISTANCE THERMOMETERS CALIBRATED ACCORDING TO ITS-90

P. Rosenkranz
BEV, Vienna, Austria
E-mail: peter.rosenkranz@bev.gv.at

A new supplement to the GUM outlines uncertainty calculation using matrix algebra for models with more than one output quantity. This technique is applied to the problem of uncertainty propagation for platinum resistance thermometers (PRTs). PRTs are calibrated at specified sets of defining fixed points dependent on the desired temperature range. With these calibration results, the resistance value of the thermometer in use can be converted into temperature through specific reference and deviation functions. This presentation discusses the problem of uncertainty propagation of the fixed point calibrations plus the resistance value of the PRT in use as input quantities, the coefficients of the deviation function as intermediate results and the temperature as output quantity.

A general solution in matrix formulation for any temperature range of the ITS-90 defined by PRTs will be highlighted. The presented method allows for an easy consideration of the input quantities correlations, which differ with the circumstances of the accomplishment of the fixed point calibrations and the resistance measurement of the thermometer in use. An example calculation for a specific temperature range based on a simplified model for the input quantities correlations will show this benefit.

UNCERTAINTY OF THE KRISS LOW FROST POINT HUMIDITY GENERATOR

B. I. Choi, J. C. Kim and S. B. Woo
Division of Physical Metrology, Korea Research Institute of Standards and Science, Daejeon, Korea
E-mail: cbi@kriss.re.kr

The KRISS Low Frost-point Humidity Generator (LFPG), developed for the temperature range (–95 ~ –40) °C in order to extend calibration capabilities at 2006, was evaluated it uncertainties. Each uncertainty component was categorized, and estimated by experiments and calculations. The uncertainty of LFPG depends on the generated frost point, gas flow rate, and change of moisture concentration in transportation. The standard uncertainty of LFPG is less than 32 mK at frost point (–70 ~ –40) °C region. However in the lower frost point region the uncertainty increases to 135 mK at –90 °C, which is mainly due to the water adsorption (or desorption) in the tubing from saturator to hygrometer.

Keywords: dew point, humidity standard, low frost point generator, uncertainty.

ALGEBRAIC APPROACH FOR CALCULATING OF TOTAL UNCERTAINTIES IN SPRT MEASUREMENT

R. Palenčár[1], S. Ďuriš[2], J. Ranostaj[2]
[1] *STU, Bratislava, Slovak Republic*
[2] *SMU, Bratislava, Slovak Republic*

Document CCT 08-19/rev *Uncertainties in the Realization of the SPRT Subrange of ITS-90* prepared by CCT-WG3 mentions four basic methods for calculating of total uncertainties in SPRT measurement: numerical analysis, direct algebraic application of the ISO Guide, mixed numerical and algebraic approaches and application of the ISO guide via interpolation theory.

Presented article describes the algebraic approach for calculating of total uncertainties in SPRT measurement in the ITS-90 sub-range from 0 °C to the freezing point of aluminium (660.323 °C). Presented approach is based on direct application of ISO Guide and for specific cases use the orthogonal interpolating equation as presented in the CCT 08-19/rev.

Authors presume that presented algebraic approach is more universal and enables to solve also cases that are not mentioned at the CCT 08-19/rev. Moreover, this approach enables to calculate total uncertainty when covariances between resistances of the SPRT at the fixed points are considered. For application of presented approach is necessary to know resistances of the SPTR at the fixed points together with their uncertainties and also the covariances between them.

UNCERTAINTY EVALUATION OF THE NEW SET-UP FOR MEASUREMENT OF WATER VAPOUR PERMEATION RATE BY A DEW-POINT SENSOR

D. Hudoklin[1], J. Šetina[2] and J. Drnovšek[1]
[1] *MIRS/UL-FE/LMK, Ljubljana, Slovenia*
[2] *Institute of metals and technology, Ljubljana, Slovenia*
E-mail: domen.hudoklin@fe.uni-lj.si

Water vapour permeation rate (WVPR) through materials is a very important measurable parameter in many industrial applications such as development of new fabrics and construction materials, in semiconductor industry, packaging, vacuum techniques etc. The demand for this kind of measurement grows considerably and thus many different methods for measuring the WVPR are developed and standardized within numerous national and international standards. However, comparison of existing methods shows low level of agreement. It is therefore the objective of this paper to carry out the necessary uncertainty evaluation for WVPR measurement, which would provide a basis for development of corresponding metrological infrastructure.

Paper presents a specially developed measurement set-up, which employs precision dew-point sensor for WVPR measurement on a specimens of different shapes. The paper also presents a physical model, which tries to account for both commonly referred method types of WVPR measurement by standards and scientific publications; the dynamic and quasi-static. Uncertainty evaluation is carried out according to ISO/IEC Guide to the expression of uncertainty in measurement (GUM). Results show the relative expanded (k=2) uncertainty of 2.9% for WVPR of 6.71 $mg \cdot h^{-1}$ (permeance 30.4 $mg.m^{-2} \cdot day^{-1} \cdot hPa^{-1}$).

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