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and Thermal Measurements in Industry and Science

Book of Abstracts
Volume B

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

Edited by:
Jovan Bojkovski, Gregor Geršak, Vincencij Žužek,
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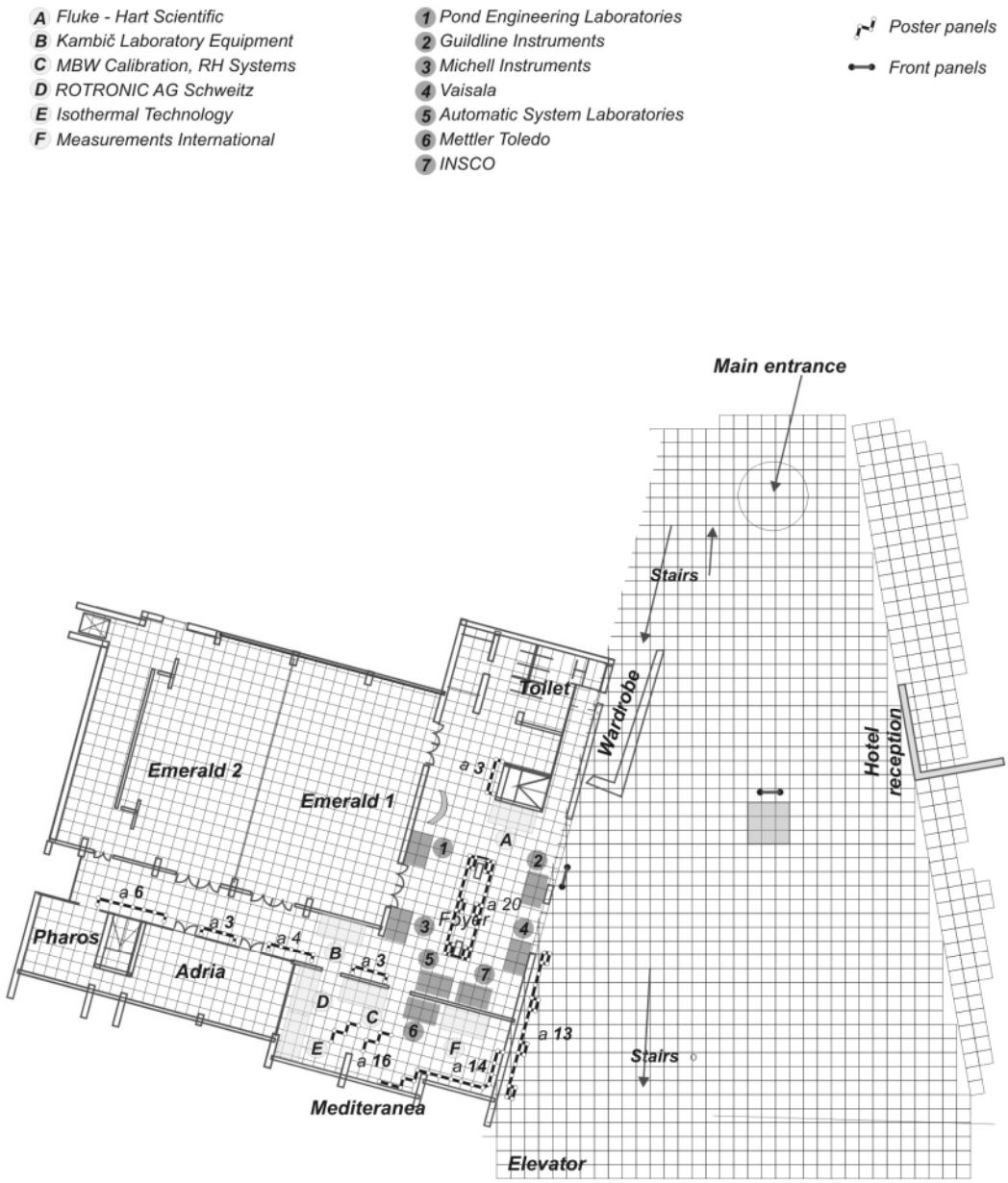
Floor plan


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
Main entrance
Stairs
Toilet
Wardrobe
Hotel reception
Emerald 2
Emerald 1
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Stairs
Mediteranea
Elevator

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 B

Resistance Thermometry II

Tuesday
15:00 to 16:20
Emerald 1
Session Chairman: Rod White

SPRT CALIBRATION INTERVAL DETERMINATION USING THE CALIBRATION HISTORY AND THE *R*(0.01 °C) VALUE

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The length of time that an ITS-90 calibrated standard platinum resistance thermometer (SPRT) may be used as a defining interpolation instrument for the measurement of temperature is limited by the stability of the SPRT. The use of the water-triple-point resistance [R(0.01 °C)] value for a calibrated SPRT can be used in several ways that can impact the calibration interval. The preferred method that reduces measurement uncertainty and increases the calibration interval is to use a new R(0.01 °C) value after temperature measurements [$R(T_{90})$] so that the $W(T_{90})$ properly reflects changes in the SPRT sensor element. In theory, the change in the R(0.01 °C) value and a change in the $R(T_{90})$ value should scale proportionally so that the overall change in $W(T_{90})$ is negligible (*e.g.* no change in the SPRT calibration status). However, in practice the changes in the R(0.01 °C) and $R(T_{90})$ values do not scale proportionally, thus the SPRT temperature measurement uncertainty (e.g. calibration status) will worsen over time. This paper explores the viability of using a change in the R(0.01 °C) value and the calibration history [fixedpoint $W(T_{90})$ values] of the SPRT as a predictive method to determine the calibration status of the SPRT with respect to required measurement uncertainty. Over 100 SPRTs, representing 5 manufacturers and 7 model types submitted to NIST for multiple calibrations since 1990, were investigated over the calibration range from the argon triple point (–189.3442 °C) to the zinc freezing point (419.527 °C).

The SPRT calibration history combined with a change in the R(0.01 °C) value for determining a calibration interval with respect to required temperature measurement uncertainty was quantified. Using Multivariate Analysis of Variance (MANOVA), it was determined that a predictive algorithm can not be unilaterally applied to SPRTs for predicting a change in the calibration status from a change in the R(0.01 °C) value to within an uncertainty that is commensurate with capabilities of an SPRT. However, it was found that it is possible to create an SPRT specific algorithm that can be used with an uncertainty (k=1) of 1 mK to calculate a change in the calibration status using a change in the R(0.01 °C) value. The use of such a predictive algorithm can be readily applied to determine the calibration interval based on changes in the R(0.01 °C) value for most measurement uncertainty requirements.

COMPARISON MEASUREMENT FOR THE DEVELOPMENT OF HIGH TEMPERATURE PLATINUM RESISTANCE SCALE

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Platinum Resistance Thermometers (PRTs) are used for interpolation up to the Ag freezing point temperature in the International Temperature Scale of 1990 (ITS-90). For higher temperatures, radiation thermometers are specified as the interpolating instrument of the scale. In practice, thermocouples are generally used for this temperature range, however, the measurement uncertainties would be slightly below 100 mK.

The authors have been developing PRTs for high temperatures, aiming to extend the applicability range of the PRTs and reduce the temperature measurement uncertainty for the temperatures between the silver freezing point (961.78 °C) and the copper freezing point (1084.62 °C). This development has two aspects; the development of high temperature PRTs, and the considerations upon the interpolation for the temperatures in between. Although it is known that characteristics of PRTs at high temperatures have been evaluated by H. J. Jung (PTB) before the revision of ITS-90, the data has been remaining unpublished.

In this paper, we report the construction of a comparison furnace to conduct the comaprison measurement between a PRT and a radiation thermometer, for the objective of making considerations upon the high temperature PRT scale. The uncertainties of the comparison measurement are first evaluated. Considerations upon the interpolation based upon the comparison data and the fixed point calibartion at Sn, Zn, Al, Ag and Cu of 5 PRTs will be done.

EVALUATION OF MODIFIED ADC BASED THERMOMETRY BRIDGE

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This paper presents the modification and testing of an ADC based thermometry bridge. The instrument in mind is an Anton Paar MKT50 resistance bridge (developed by TU-Graz) based on a precision analog to digital converter (ADC). During preliminary testing it was found that the MKT 50 performs better than its declared uncertainty and is in fact comparable to the thermometry bridges typically used in secondary thermometry laboratories. The modifications to the original bridge were undertaken by the development team of the MKT50 at the Graz University of Technology, Austria. Measurements and evaluation of the modified instruments were performed at the MIRS/UL-FE/LMK.
For the MKT 50 to be used in thermometry laboratories as a reference unit, measuring parameters of the instrument had to be changed. During the first modification the instrument range was reduced from 400 Ω to 133 Ω, this being a preferred range for standard platinum resistance thermometers (SPRTs). This also meant an increase in the measuring current from 0.5 to the more frequently used 1 mA. A software upgrade increased the resolution from 24 to 27 bit and enabled the use of an external standard resistor. After this stage of the modification the first tests on the instrument were performed. The second stage included removal of all the unnecessary components, which could generate noise. The instrument was prepared in such a way that it had only two input channels, one connected to the SPRT and other one to the standard resistor. Also the components of the ADC were upgraded to further reduce noise. A further software upgrade sped up measurements, making the PC software capable of taking several readings in a shorter time period.
All tests were performed in laboratory conditions, where precision AC and DC resistance bridges are typically being used. The non-linearity was assessed by use of an automated resistance bridge calibrator (RBC, model RBC100), while the noise value was gathered both from the standard deviation of RBC measurements as well as from comparisons done between two standard resistors with stable values. All tests were repeated several times to assure confidence in results.
With its lowered range of 133 Ω and increased resolution of 27 bit, the instruments non-linearity, being valued at around 2 μΩ, was comparable to primary resistance bridges such as the ASL F900 or MI6010T. However the noise of the instrument remained high at 4 μΩ.
Keywords: thermometry resistance bridge, non-linearity testing, precision analog-to-digital converter.

Calibration Procedures and Facilities II

Tuesday
15:00 to 16:20
Emerald 2
Session Chairman: Davor Zvizdić

DETERMINATION OF HYSTERESIS IN CALIBRATION OF PRTS

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The paper discusses the contribution of hysteresis to the total uncertainty of calibration of industrial platinum resistance thermometers (IPRTs). Hysteresis is one of the sources of uncertainty that has so far not been sufficiently researched and documented. The term hysteresis applies to any system that is path-dependent: the output depends on the history of the input. In our case, thermal hysteresis results in different resistance values at the same temperature point, whether the temperature was increasing or decreasing. The reason for such behavior is related to the construction of the thermometer (strain due to thermal expansion and contraction) and also to possible moisture inside the encapsulation.

In the process of evaluation of industrial platinum resistance thermometers calibration and measurement capabilities (CMCs) within Working Group 8: Calibration and measurement capabilities of the Consultative Committee for Thermometry (CCT WG8) it has been concluded that uncertainty due to hysteresis is not uniformly defined and being determined.

In order to estimate uncertainty contribution due to the hysteresis and compare different procedures, resistance measurements were carried out on a number of IPRTs of different qualities and tolerance classes. The temperature span was between –50 °C and 300 °C, which is the most frequent temperature range in practical use of IPRTs. The hysteresis was then determined in different ways (change of resistance at ice-point and at the midpoint temperature, according to ASTM International Standard E644 and according to the new version of IEC Standard 60751) and comparison of results was made.

HUMIDITY CONTROL IN A CALIBRATION SYSTEM FOR PTU DEVICES

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Devices measuring pressure, temperature and humidity simultaneously are known as PTU devices. Nowadays most of the transducers of PTU devices are calibrated separately, namely humidity and temperature sensors together and pressure transducers on their own. Simultaneous control and measurement of pressure, temperature and humidity is challenging. Especially the low and high ends of the humidity range are difficult, combined with the low and high ends of temperature and pressure ranges. A calibration system for PTU devices is developed at MIKES for studying humidity, temperature and pressure related characteristics of different kinds of devices. In this PTU Apparatus, all these three quantities can be controlled simultaneously.

The mixing flow principle is applied to control the humidity inside the measuring chamber of the apparatus. Air is supplied from a compressed air system to a dryer and on the other hand, to a saturator. Two mass flow controllers regulate the mixing of these two air streams. The mixture is supplied into the measuring chamber. The saturator is located in the same thermally and pressure controlled enclosure as the measuring chamber.

The dew-point temperature of air entering and exiting the chamber is monitored with a chilled mirror hygrometer. For the temperature control, heat transfer oil circulates in a system consisting of a thermostat and a coiled tube surrounding the measuring chamber. The temperature inside the chamber is measured with two Pt-100 thermometers. The pressure inside the measuring chamber and in the enclosure surrounding it is controlled using line pressure regulators in the pipelines before and after it. The pressure inside the chamber is measured with a digital barometer.

The nominal ranges of the PTU Apparatus are the following: relative humidity 10% … 95%, temperature –50 °C … +80 °C and absolute pressure 500 hPa … 1200 hPa. All combinations over the humidity, temperature and pressure ranges are possible. The estimated uncertainties (k = 2) of the humidity, temperature and pressure are 1 %RH to 3 %RH, 0,1 °C to 0,3 °C and 10 Pa respectively.

The PTU measurement chamber system was introduced in IMEKO 20th TC3, 3rd TC16 & 1st TC22 International Conference (27 November – 1 December 2007, Merida, Mexico). Since then the humidity control was designed and tested. Also, further tests on temperature and pressure control have been carried out. This paper reports the new design of the complete PTU Apparatus and results showing the performance of the system.

A RELIABLE AND INEXPENSIVE RELATIVE HUMIDITY CALIBRATION IN ORDINARY CLIMATIC CHAMBER

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The paper presents an inexpensive, but reliable method used in the Institute VINČA for the calibration of relative humidity hygrometers. The method uses an ordinary climatic chamber as the humidity generator, capacitive humidity probes as the humidity reference, and thermocouples as the temperature reference probes. The hygrometers under calibration (HUC) are calibrated by comparison with reference humidity and temperature probes inside the climatic chamber and a corresponding calibration uncertainty is assessed. Inside the chamber the air flow is affected only by the presence of the HUCs and reference probes and there are no additional air flow-control elements installed. The calibration method assumes the uniformity of the vapour pressure inside the working volume of the chamber.

The HUCs can use either electrical (capacitive or resistive) or mechanical types of sensors, external or internal humidity probes, and their readings can be either digital or analogue. In principle, the only limiting condition is that the overall dimensions of the HUC must be smaller than the working volume of the used climatic chamber.

In the first part of the paper, the description of the calibration setup, calibration procedure, and the analysis of the calibration uncertainty are given. In the second part, the description of a usermade LabVIEW application is presented. The application has been uniquely developed to meet several requirements: synchronizing acquired data from different reference instruments and those directly read and entered by the user, displaying data graphically for easy monitoring of the calibration process, saving data in a single text document for subsequent data reduction, and easily adapting the application to various combinations of reference instruments and HUCs. In the last part of the paper, several calibration examples are given and discussed.

REPRODUCIBILITY IN CALIBRATIONS OF THE THERMOHYGROMETERS

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Relative humidity, temperature and pressure are the three most important parameters in control of environmental conditions. Most of relative humidity measurements are carried out with capacitive or impedance sensors, which are based on the electrical properties of thin, water-sorbing films. Although the accuracy of these sensors is about ten times worse than achieved with standard chilled mirror hygrometers, their low price, wide range and convenience of measurement contribute to their usefulness for control of environmental conditions in most laboratories.

In capacitive sensors are used dielectrics in which take place absorption and desorption of water vapour particles. Depending on the *RH* range the sorption in dielectric structure may have respectively the surface (in low *RH*) or volume character (in high *RH*). When the sensor is polluted there may occur water solutions as well. Wide range of *RH* measurements is connected with large or small quantity of absorbed particles of water and is the important source of measurement errors which depends on history of measurements.

The uncertainty of measurement will increase if sensors will be calibrated in wider range. Reproducibility depends mainly on hysteresis, accumulation of impurities and ageing of sensors. The long-term stability of these sensors is not guaranteed and they require frequent calibration. The large number of calibration per year give us possibility of analysing all important factors which have influence on measurement.

In this paper is reported the determination of the important sources affecting the sensor response. There is also considered the clarity of calibration method, the reporting of calibration results and its interpretation which is very important to allow the user to get the most accurate and reliable results of measurements. And analysed how to fit the form and range of given information to the real necessity of the user of instrument.

Interlaboratory Comparisons II

Tuesday
15:00 to 16:20
Adria
Session Chairman: Michael de Podesta

COMPARISON OF BLACKBODIES FOR CALIBRATION OF INFRARED EAR THERMOMETERS

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The article will present the results of the EURAMET project no. 927 "Comparison of blackbodies for calibration of infrared ear thermometers (IRETs)". For the comparison MIRS/UL-FE LMK was chosen as the pilot laboratory. The objective of the comparison was to determine the agreement of blackbodies used for the calibration of IRETs among European national laboratories. To verify the accuracy of an IRET a suitable blackbody is needed. An essential requirement is related to accuracy. There are several standards in the world, which describe requirements for IRETs and a blackbody. In the EU this is the standard EN 12470-5, which is a harmonised standard and supports the Medical Device Directive (MDD). Other standards are ASTM standard, Designation E 1965 – 98 and the draft of Japan Industrial Standard JIS T 4207, 2005: Japanese Industrial Standard: “infrared ear thermometers.”. The International Organization for Standardization (ISO) is developing a new standard for clinical thermometers, which will include also IRETs. The basic requirement for accuracy in EN 12470-5 is that the maximum permissible error of IRET is ±0,2 °C in the range from 35,5 °C to 42,0 °C, while the uncertainty of a blackbody is required to be at the most ±0,07 °C. An example of a blackbody is presented in Annex C of the standard EN 12470-5. Such a blackbody related to this standard, designed for the calibration of ear thermometers and immersed in a stirred water bath, was provided for the comparison by the pilot laboratory. The difference to the standard was

horizontal positioning of the blackbody, while in the standard a vertical position is suggested.

The comparison was performed in one loop with 9 participating laboratories. The pilot laboratory provided the bath with the “EN-type” of a blackbody and the transfer IRET. The IRET and the bath were evaluated in the pilot laboratory before and after comparison measurements in participating laboratories.

Each participating laboratory calibrated at least the transfer IRET against the transfer blackbody and against a local blackbody in the range from 35,5 °C to 42 °C. The transfer IRET was capable of operation with and without a probe cover therefore two sets of measurements were performed at each temperature. Important was that participating laboratories could not measure the temperature of the transfer blackbody with their own thermometer. They could only measure the resistance of a 4-wire platinum resistance thermometer embedded in the transfer blackbody. After calibration of the transfer IRET and a local IRET, if it was available, a laboratory sent a report with results to the pilot laboratory. After all measurements were concluded the pilot laboratory performed an analysis, which was sent to all participants for checking the entries and comments.

The article will present the results of agreement of blackbodies for calibration of IRETs at participating NMIs. The agreement was evaluated based on measurements with the transfer IRET with and without probe cover. If a local IRET was available the agreement was evaluated in the same way but based on measurements with a local IRET with and without probe cover. The results, which will be presented, show, in general, relatively good agreement between the blackbodies for calibration of IRETs at majority of participating NMIs. A good agreement is necessary for further dissemination of the temperature scale to IRETs, which is one of essential requirements in the first and final drafts of the international standard for clinical thermometers (IEC ISO/FDIS 80601-2-56 Particular requirements for basic safety and essential performance of clinical thermometers for body temperature measurement).

A COMPARISON OF THE ITS90 BETWEEN, NPL, NIM AND CEM ABOVE THE SILVER POINT USING HIGH TEMPERATURE FIXED POINTS

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The ITS-90 above the silver point is realised, according to the definition, using Planck's law in ratio form, referenced to a blackbody cavity at the freezing point of one of the defining fixed-points. Due to the relative looseness of the definition there is some freedom in how to realise the scale: for instance the reference fixed point blackbody could be Ag, Au or Cu, and the selection of wavelength, optical system or detector of pyrometer is left to the users discretion.

Due to this variation it is essential that regular comparisons of internationally realised and disseminated versions of the ITS-90 are performed to ensure that the radiance temperature scales realised in different National Measurement Institutes (NMIs) are equivalent. Unfortunately, due the lack of suitable artefacts, comparisons are not really able to probe the equivalence of local realisations of the ITS-90 to the uncertainty level claimed. The development of high temperature fixed points (HTFPs) has resulted in artefacts that could possibly be used in a key comparison to demonstrate the equivalence of locally realised NMI scales at high temperatures.

This study reports the results on a prototype scale comparison undertaken between the UK, Chinese and Spanish NMIs. The comparison was performed by using HTFPs of Co-C, Pt-C and Re-C. Each of these cells was assigned an ITS-90 temperature by its respective institute. The HTFPs of NIM and CEM were then transported to NPL and their ITS-90 temperatures measured using a linear pyrometer. The temperature difference of the respective institute cells was also determined. A detailed uncertainty analysis was performed of the comparison results.

It was shown that the performance of the cells, in terms of temperature stability and robustness, was far superior to any other potential method of scale comparison at these temperatures and may well be the only practical way of conducting a key comparison that tests the claimed uncertainties of local realisations of such scales in a rigorous way.

DISSEMINATING THE ITS-90 TRACEABILITY IN INDUSTRY – AN INTERCOMPARISON OF TEMPERATURE BLOCK CALIBRATORS

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An international intercomparison has been carried out with a commercial dry block calibrator as transfer standard by Danish Technological Institute (DTI). The intercomparison involved 16 participating laboratories from five European countries. The intercomparison comprised five measurement points in the range from –20 °C to +150 °C. The purposes of the intercomparison were twofold: To compare the results of the participating laboratories during calibration of a dry block calibrator; to establish the dry block calibrators' reproducibility and suitability both as a transfer standard and as a working measurement standard for disseminating the ITS-90 traceability in industry.

The characterisation and performance of state-of-the-art multi zone dry block calibrators and the results of the intercomparison are presented.

Keywords: dry block calibrator, intercomparison.

EVALUATION OF CALIBRATION LABORATORIES PERFORMANCE

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One of the main goals of interlaboratory comparisons (ILCs) is to evaluate the laboratories performance for the routine calibrations they perform to the clients. In the frame of Accreditation of Laboratories, the National Accreditation Boards (NABs) in collaboration with the National Metrology Institutes (NMIs) organise the ILCs needed to comply with the requirements of the regional and international organizations, namely the European Accreditation (EA) and the International Laboratory Accreditation Cooperation (ILAC).

In order that an ILC is a reliable tool for a laboratory to validate its best measurement capability (BMC), it is needed that the NMI provides a travelling standard better – in terms of accuracy class or uncertainty, than the laboratories BMCs. Although this is the general situation, there are cases where the NABs ask the NMIs to evaluate the performance of the accredited laboratories when calibrating industrial measuring instruments (MIs). These MIs usually have a fixed resolution that is the major component of uncertainty (case of mercury-in-glass thermometers, sensors with digital display device, etc…). Even with the best instrumentation the NMI is not able to "decrease" this intrinsic component of uncertainty and consequently the reference value will have an uncertainty that will "penalise" the laboratory comparison uncertainty.

The aim of this paper is to discuss the existing approaches for the ILCs evaluation and propose a basis for the validation of the laboratories measurement capabilities. An example is drafted with the evaluation of the results of a mercury-in-glass thermometers ILC with 12 participant laboratories.

Uncertainty Estimation

Tuesday
16:50 to 18:10
Emerald 1
Session Chairman: Martti Heinonen

UNCERTAINTIES IN THE SPRT SUB-RANGES OF ITS-90: TOPICS FOR FURTHER RESEARCH

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Working Group Three of the CCT has completed the guide summarizing the uncertainties in the realization of the SPRT subranges of ITS-90 between the triple point of neon (24.5561 K) and the freezing point of silver (961.78 °C). The document provides users of ITS-90 with guidance for assessing the uncertainty in both SPRT calibrations and temperature measurements made with SPRTs. During the preparation of the guide, it became apparent that there were many aspects of standard platinum resistance thermometry where either data or models were lacking. This paper summarizes the most significant areas where further research is required.

In the calibration of SPRTs, the two largest sources of uncertainty are impurities, and the thermal management of the fixed points. For the metal fixed points particularly there is a shortage of data on liquidus slopes for the different impurities necessitating the use of the OME uncertainty-assessment method, which yields a larger uncertainty than the alternative SIE method.

In the use of SPRTs, the two largest sources of uncertainty are non-uniqueness (both Types 1 and 3) and oxidation. The causes of Type 3 non-uniqueness are not understood, especially at low temperatures, and there is a paucity of data for the high temperature subranges. At present our understanding of oxidation effects in 25 Ω SPRTs is based almost entirely on Berry's work. However Berry's work has never been replicated and independent validation of Berry's models and numerical results would be valuable, especially given the changes in SPRTs design that followed Berry's work.

A range of less significant effects are also discussed including vacancy effects in SPRTs, isotopic effects in fixed points, and improved statistical methods.

THE USE OF REFERENCE FUNCTIONS IN HUMIDITY CALCULATIONS

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A variety of reference functions for the saturation water vapour pressure and the water vapour pressure enhancement factor for moist air are used by the humidity community. Such functions are inherently inexact and potential error in their use is usually characterised by simple uncertainty functions or tables. While many variants of the vapour pressure reference functions have been published, they present a number of problems for those needing to correctly account for the uncertainty associated with their use.

Questions concerning which particular expression is to be used, must also consider the uncertainty function accompanying each variant, if it exists. There are significant differences between values obtained using different expressions and this might not be reflected in their uncertainty. Secondly, there is no one expression which is accepted as the "best" approximation by the humidity and moisture community, just as there is no commonly accepted "best" expression for the uncertainty associated with each approximation. Thirdly, the lack of "autocorrelation" information, that is, information concerning the correlation between two evaluations of the same expression, can lead to significant over- or under-estimation of uncertainty when the expression is used more than once in a calculation, as is the case for many humidity calculations.

In this paper, the above questions are addressed and recommendations are given for future analytical expressions, theoretical or experimental, of vapour pressure data. To illustrate, model expressions are given for the pure-phase water vapour pressure and the water vapour pressure enhancement factor for moist air, which are derived from published expressions but which enable assessment of autocorrelation and hence a correct analysis of the propagation of uncertainty.

MEASUREMENT UNCERTAINTY OF DEW-POINT TEMPERATURE IN A TWO-PRESSURE HUMIDITY GENERATOR

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This paper describes the measurement uncertainty evaluation of dew-point temperature when using a two-pressure humidity generator as a reference standard. The main difficulty related to this problem arises from the non-linear and iterative nature of the applied mathematical model that provides the measurand estimate. In this case, the analytical method although providing an exact solution, would require a substantial calculus effort and the conventional GUM method is not suitable to provide accurate solutions for this type of measurement uncertainty evaluation. Two alternative approaches are proposed: the forward measurement uncertainty propagation by the Monte Carlo method; and the inverse measurement uncertainty propagation by Bayesian Inference.

The broad application of this study, from the numerous sectors where the measurement of hygrometric conditions is a requirement to any other areas involving the solution of an iterative model, makes it particularly relevant. Thus, besides the information on the use of a two-pressure humidity generator as a dew-point reference standard, the research aimed to establish the influencing factors affecting the use of the Monte Carlo method and the inverse uncertainty propagation using Bayesian Inference in terms of their accuracy and adequacy.

Keywords: Dew-point temperature; Iterative model; Measurement uncertainty.

Fixed Points III

Tuesday
16:50 to 18:10
Emerald 2
Session Chairman: Anatoly Pokhodun

DOPING EXPERIMENTS IN MERCURY TRIPLE POINT CELLS

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The use of the SIE method to asses the uncertainties due to impurities in the ITS-90 fixed points requires the knowledge of the liquidus slopes of the alloys for each fixed point and the amount of impurities at low concentrations. In this sense, CEM has started doping experiments in triple point of mercury cells in order to obtain a more accurate estimation of the uncertainty contribution and to correct the influence of the dominating impurities on the mercury triple point.

The working cells are made in borosilicate glass. They are initially filled in vacuum distillation with highly purified mercury 99.999 999 + % (6 times distilled) in order to avoid air bubbles and mercury oxidation. A concentrated amalgam with the chosen impurity is prepared at room temperature. Each component of the amalgam is weighted in a high sensitivity analytical balance in order to know accurately the amount of amalgam sample to be added to the working cell.

The experiments consist of gradually doping a cell (initially filled with “pure” Hg) with impurities, recording the melting plateaus after fast and slow freezing and comparing it with a commercial closed cell (used as check standard) in order to estimate the differences in the triple point of mercury temperature caused by the doping. The measurements are performed in the two cells simultaneously using two SPRTs in an alcohol stirred bath with a DC bridge.

The measurements are carried out in two types of cells: open and sealed in vacuum. The results obtained for metallic impurities like Zn and Ag and atomic fraction between 10^{-7} and 10^{-5} will be showed.

IMPURITIES IN WATER TRIPLE POINT CELLS

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The clarification of the definition of the Kelvin (2005) prompted the large majorities of the National Metrology Institutes to found their national definition of the water triple point temperature on VSMOW (Vienna Standard Mean Ocean Water). As a result of this evolution, the uncertainty component linked to the isotopic composition of the cell water is currently reduced with a moderate effort to a few microkelvin (typically 1 to 3 µK).

There is general consensus that, when the isotopic composition has been taken in due account (either by applying an isotopic correction or by using quasi-VSMOW water) the dominant uncertainty becomes the presence of impurities in the cell water (typically 10 to 50 µK).

In spite of the relevant efforts dedicated to the origin and the effects of impurities on the water triple point temperature by the thermometry community, our understanding of the water contamination mechanisms and their impact on the realized triple point temperature is still limited.

In this paper we report on the results obtained on cells manufactured with ad-hoc addition of known amounts (1 to 10 ppm) of impurities (boron and silicon) to the cell high-purity water. The triple point temperatures realized by the manufactured doped cells were measured and correlated with the dope concentration as calculated from the known dope solution added to the high-purity water in the cell and with the global impurity content measured by ICPMS (Inductively-Coupled Plasma Mass Spectroscopy).

IMPROVED ESTIMATES OF THE ISOTOPIC CORRECTION CONSTANTS FOR THE TRIPLE POINT OF WATER

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In 2006, the CIPM clarified the definition of the kelvin by specifying the isotopic composition of the water to be used in the realisation of the triple point. At the same time, the Consultative Committee on Thermometry gave recommended values for the isotopic correction constants to be used for water departing from the specified composition. The values of the correction constants were based largely on the cryoscopic measurements of Kiyosawa [1], with supporting evidence from White *et al* [2] and the historical measurements of LaMer and Baker. Unfortunately, Kiyosawa did not provide estimates of the uncertainties in his measurements. The problem of estimating the uncertainty is complicated by possibility that Kiyosawa's water samples were contaminated by other isotopologues [3]. The consistency between the various measurements indicates that the uncertainties in the correction constants are of the order of 20 mK, 50 mK, and 5 mK respectively for deuterium, ^{18}O and ^{17}O respectively.

Van't Hoff's relation, which relates the freezing-point temperature depression to the fractionation constants for the isotopologues, provides an alternative means for estimating the correction constants. Values of the corrections constants inferred from the fractionation constant measurements of Lehmann and Siegenthaler [4] are A_D = 680 mK ± 7 mK, and A_{18O} = 602 mK ± 11 mK. These results are more consistent with the historical results of Lamer and Baker, White *et al*, and thermophysical data for the water isotopologues.

[1] Kiyosawa, K., Freezing point mixtures of $H_2{}^{16}O$ and $H_2{}^{18}O$, *J. Soln. Chem.* **20**, 583-588, 1991
[2] White D.R, Dransfield T.D., Strouse G.F., Tew W.L., Rusby R.L., Gray J., Effects Of Heavy Hydrogen and Oxygen on the Triple-Point Temperature of Water" in *Temperature: its Measurement and Control in Science and Industry*, Vol 7 , Ed. D. C. Ripple, AIP, New York, 221-226, 2003
[3] Tew W. L. and White D. R., "Comment on 'Freezing Point Mixtures of $H_2{}^{16}O$ with $H_2{}^{17}O$ and those of Aqueous CD_3CH_2OH and $CH_3{}^{13}CH_2OH$ Solutions'", *J. Soln. Chem.*, **34**, 1191- 1196, 2005
[4] Lehmann M, and Siegenthaler U., Equilibrium oxygen- and hydrogen-isotope fractionation between ice and water, *J. Glaciol.*, **37**, 125, 23-26, 1991

Radiation Thermometry II

Tuesday
16:50 to 18:10
Adria
Session Chairman: Renato Teixeira

DISTANCE EFFECT AND SIZE-OF-SOURCE EFFECT OF RADIATION THERMOMETERS

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The radiation thermometer with center wavelength of 660 nm has been established and improved several types since 1990 at NIM of China. Compared with older type radiation thermometer (named RT9031), RT9032-660 is of smaller volume, lighter weight and portable. The center wavelength of 900 nm of the standard radiation thermometer, named RT9032-900, has been setup for one year at NIM of China. The uncertainty of RT9032-660 and RT9032-900 is 0.3 k for copper point, respectively.

This paper describes the distance effect (DE) between the source and the thermometer and the size-of-source effect (SSE) of RT9032. The SSE and DE experiments employed a large integrating sphere source of 800 mm inner diameter and an exit port of 97 mm in diameter. It has six apertures from 5 mm to 97 mm in diameter and one quartz plate with black spot of 3 mm in diameter. The SSE is measured with different distances between the source and the thermometer. The radiation thermometers on a computer-controlled stage plate can be moved in an area ranging from 550 mm to 1000 mm relative to the exit of the source. The DE result would be affected by the stability and uniformity of integrating sphere source. We try to improve the stability and uniformity, such as changing the type and position of halogens, adding a monitor and so on. On the basis of the comparison between the DE results before and after the improvement of the source, we analyze the degree of influence of the stability and uniformity of source on DE. For verifying the method, the DE and SSE of the LP4 of IKE and Chino IR-IST 65H are also measured. The relationship between DE and SSE is discussed.

Keywords: radiation thermometer, size of source effect, distance effect.

CALIBRATION OF THE SPECTRAL RESPONSIVITY OF FILTER RADIOMETER FOR T MEASUREMENT AT NIM

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At the National Institute of Metrology of China (NIM), the silicon photodiode based narrow-band interference filter radiometers (FRs) are designed for the radiometric determination of thermodynamic temperature of new metal carbon fixed points. The calibration of the FRs is performed at a new spectral comparator with a trap detector which was calibrated at the cryogenic radiometer at several discrete laser lines. The new spectral comparator is constructed by two grating monochromators assemble with lower stray light and higher transmitted flux. Applying a correction for the transmittance of filter and a careful control of the temperature, the responsivity of a 633 nm centered FR is got over a dynamic range of nearly eight decades in the wavelength range 450 nm to 1200 nm. The relative uncertainty of the responsivity is also analyzed and less than 6×10^{-4} at the 1σ level.

A DYNAMIC METHOD TO MEASURE EMISSIVITY AT HIGH TEMPERATURES

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The laser-flash pulse-heating method is a widely used and well established technique for the measurement of thermal diffusivity. Major advantages are its high measurement speed and its applicability over wide ranges of temperature and thermal diffusivity. Since its introduction in the 1960s it has been proposed to extend this technique to the absolute measurement of heat capacity and emissivity.

The direct measurement of emissivity as well as heat capacity via the laser-flash method requires the knowledge of the absolute laser pulse energy and the resulting temperature rise, but these parameters are difficult to gain. It is however, highly desirable to overcome these difficulties and to establish this technique as the emissivity at high temperatures is not sufficiently known for important applications e.g. in lighting industry and fusion research.

In the presented work, a theoretical study is given on the possibility of emissivity measurement using the laser-flash method. Two mathematical approaches are discussed which solve the problem, that a measured temperature rise – necessary to calculate the emissivity – itself depends on the emissivity. With these approaches the emissivity can be calculated from the measured temperature rise, the energy of the incident laser pulse and the knowledge of the relevant material parameters (i.e. heat capacity and mass of the sample). The true temperature of the sample can be calculated subsequently with the determined emissivity. And the emissivity at other wavelengths can also be determined by measuring the emitted thermal radiation of the sample at these wavelengths. It is shown that both mathematical approaches have a negligible arithmetic error, making them applicable to be used in future work. Moreover, these theoretical studies are supplemented by first measurement results and some details of the recently assembled set-up.

CALIBRATION OF RADIATION THERMOMETERS AND FIXED POINTS IN THE MIDDLE-TEMPERATURE RANGE FROM 160 °C TO 420 °C

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The NMIJ had conventionally been disseminating radiation temperature scales traceable to the ITS-90 from –30 °C to 160 °C in the low-temperature range, and from 400 °C to 2500 °C in the high-temperature range. A new calibration service in the middle-temperature range has recently been established to bridge the gap, realizing a radiation temperature scale calibration service covering the entire range from –30 °C to 2500 °C.

The temperature scale is realized by interpolation on a 1.6-μm reference standard radiation thermometer calibrated at the three fixed-point blackbodies of indium (156.5985 °C), tin (231.928 °C), and zinc (419.527 °C). The blackbody cavities all have an opening of 6 mm and depth of 93 mm, resulting in an effective emissivity of 0.99989. The fixed-point blackbodies are placed in three-zone furnaces. The uncertainties of the fixed points are evaluated to be better than 0.1 °C (k=2). A radiation thermometer (working around 1.6 μm) is calibrated by comparison with the reference standard radiation thermometer employing a variable-temperature blackbody, whose cavity has opening of 65 mm and depth of 160 mm, and the effective emissivity is evaluated to be 0.995 (LANDCAL P550P). The large opening of the blackbody makes it possible to calibrate thermometers with large field of view. The uncertainty (k=2) in the calibration is estimated to be 0.16 °C to 0.20 °C, taking into account the difference in the field of view of the standard thermometer and the thermometer under calibration, as well as the instability of the blackbody temperature.

Fixed-point blackbodies are calibrated by radiance comparison with the standard fixed-point blackbody with the 1.6-μm radiation thermometer. The main uncertainty component in the radiance comparison is the size-of-source effect due to the difference in the source size between the standard blackbody and the blackbody under calibration. The estimated calibration uncertainty(k =2) is 0.12 °C for both indium and tin points.

In order to calibrate radiation thermometers working in the thermal-infrared wavelength range (8 μm to14 μm), we are constructing a variable-temperature blackbody cavity placed in an air bath, whose opening diameter is 40 mm, and depth is 400 mm. The temperature range from 100 °C to 500 °C can be covered by the air-bath blackbody. High emissivity and good temperature uniformity of the blackbody cavity are required to enable calibration of the thermal-infrared radiation thermometers against the standard thermometer with 1.6 μm wavelength. The design and the performance evaluation of this air-bath blackbody are presented.

Thermodynamic Temperature Determinations

Wednesday
8:20 to 10:20
Emerald 1
Session Chairman: Ken Hill

DIELECTRIC-CONSTANT GAS THERMOMETRY WITH NEON AND HELIUM FROM 25 K TO 36 K

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New dielectric-constant gas-thermometry (DCGT) measurements were performed at PTB in the temperature range from 25 K to 36 K with the recently established setup used for measurements between 2.4 K and 27 K. This extension to higher temperatures allows for the first time to use neon as measuring gas. Furthermore, the measurements with helium yielded the first thermodynamic-temperature data obtained at PTB in the range between 27 K and 37 K.

At more than eight temperatures, DCGT isotherms were measured with neon and helium. The evaluation was performed via single- and multi-isotherm fits and the uncertainty of the fit results was estimated via the mathematical methods already used for the DCGT measurements in the range below 27 K [Ch. Gaiser, B. Fellmuth, 2009, Metrologia 46, pp. 525-533], e.g. the application of Monte-Carlo simulations.

Concerning the temperature measurements with helium, a comparison between the realisation of the ITS-90 at PTB and the DCGT thermodynamic-temperature data is presented. In addition, a comparison between new, highly accurate ab initio calculations for the second and third density virial coefficient of helium with the new DCGT results of the isotherm fitting is given. The evaluation made for the third virial coefficient of helium is of special interest because very recently two new, completely different ab initio calculations have been performed for the range above 24 K. Theory and experiment coincide within the combined uncertainty estimates. Therefore, the DCGT data in this range is of certain impact for theory.

Also very recently, new highly accurate ab initio calculation results for the second and third virial coefficients of neon became available. Due to the fact that for neon almost no experimental data of acceptable uncertainty exist for the low-temperature range, the comparison of the calculation results with the DCGT data is highly important. The DCGT measurements provide a quality check of the interatomic potentials used for the ab initio calculations. Furthermore, the “calibration” of the measuring gas neon against helium, for which the polarizability has been theoretically calculated with sufficiently small uncertainty, yielded a preliminary value for the molar polarizability of neon.

COMPARISON OF A DETECTOR-BASED NIR RADIANCE-SCALE TO AN ITS-90 CALIBRATED RADIATION THERMOMETER

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Although thermodynamic radiation thermometry in the wavelength range of Si detectors is well established, the rapidly decreasing spectral radiances with decreasing temperatures restrict the temperature range of measurements that Si-based radiation thermometers can perform. In order to extend the temperature range to lower temperatures, radiometric calibrations must be performed with near-infrared detectors. Such extension to lower temperatures would have several objectives. The comparisons of radiometric scale against the calibrations obtained using the temperature scale would validate the infrared responsivity scale for use in satellite sensors for climate change applications. If the uncertainties of the thermodynamic radiation thermometry were sufficiently low, then these measurements could be used to determine systematic differences between fixed-point temperatures of the International Temperature Scale of 1990 (ITS-90) and the thermodynamic temperatures.

We describe the use of an integrating-sphere-input InGaAs radiometer for detector-based radiance responsivity calibrations. This radiometer has radiometric and electronic characteristics between 950 nm and 1650 nm similar to those of silicon trap detectors between 400 nm and 960 nm. This near-IR radiometer is calibrated against the primary-standard cryogenic radiometer for radiant power responsivity. Knowing the area of its input aperture, it can be used as a power-to-irradiance converter with spatially uniform and Lambertian input characteristics. With non-uniformity of spatial responsivity of < 0.05% and with < 0.03% deviation of the angular responsivity from the cosine function in a 5 o field-of-view, it is possible to use it as a reference irradiance meter in the Spectral Irradiance and Radiance Responsivity Calibrations using Uniform Sources (SIRCUS) facility. Since a spatially-uniform exit-port radiance is needed to calibrate the radiance responsivity of the radiation thermometer, Spectralon-coated integrating spheres of different sizes were compared for their spatial uniformity. The off-axis angular dependence of the sphere-source irradiances was also checked. We describe the calibration and the uncertainty budget of an InGaAs-based radiation thermometer centered at 1550 nm which is calibrated for radiance responsivity. The thermodynamic temperatures and their uncertainties obtained with the ITS-90 calibration and the near-IR detector-based calibrations are compared and evaluated.

PRESENT ESTIMATES OF THE DIFFERENCES BETWEEN THERMODYNAMIC TEMPERATURES AND THE ITS-90

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In 2005, the Consultative Committee for Thermometry (CCT) recommended the creation of a Mise en pratique for the definition of the kelvin and envisioned that future versions of the Mise en pratique would recommend values of the differences between thermodynamic temperatures and ITS-90 temperatures ($T - T_{90}$). At the CCT's request, Working Group 4 (WG4) critically reviewed all available measurements of $T - T_{90}$ including constant-volume gas thermometry, acoustic gas thermometry, spectral radiation thermometry, total radiation thermometry, noise thermometry, and dielectric-constant gas thermometry.WG4 also reviewed the uncertainties and documented the conversion of older data to the ITS-90.

From the review, we provide consensus estimates of $T - T_{90}$ for selected measurements from 4.2 K to 1358 K as well as a proposal for $T - T_{90}$ spanning the range 0.65 K to 4.2 K. In the temperature range from 25 K to 255 K, the review found unexplained inconsistencies between the uncertainties claimed for specific data sets and WG4's consensus estimates of $T - T_{90}$. This was addressed by expanding the uncertainties of the consensus estimates. WG4 needs more information before it can provide a low-uncertainty estimate of $T - T_{90}$ in this temperature range. Also, more measurements are needed between 550 K and 693 K and at the copper point (1358 K). WG4 strongly encourages researchers to undertake additional high-accuracy measurements of $T - T_{90}$ in these temperature ranges.

In order to disseminate the differences $T - T_{90}$ via the Mise en pratique, we provide two analytic functions for $T - T_{90}$, one for use below T_{TPW} and one for use above T_{TPW}. Below T_{TPW}, we provide a polynomial function of the variable $x \propto \log_{10}(T_{90}/273.16\ \mathrm{K})$, similar to the SPRT reference function in the ITS-90 below T_{TPW}. Above T_{TPW}, we provide a polynomial function of the dimensionless variable $x \propto (273.16\ \mathrm{K}/T_{90})$. The discontinuity of the derivative dT_{90}/dT at TTPW is discussed and the consistency with the results of recent acoustic gas thermometry and with the resistance ratios of platinum resistance thermometers is investigated.

MONOCHROMATOR BASED ABSOLUTE CALIBRATION OF RADIATION THERMOMETERS

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Filter radiometers with interference filters are used for a long time at PTB for measuring thermodynamic temperatures in irradiance mode. Recently these filter radiometers have been applied to measure indirectly the thermodynamic temperature of small aperture hightemperature fixed-points with uncertainties significantly lower than those achievable according to ITS-90 methods. Direct measurements of the thermodynamic temperatures of these fixedpoint with small apertures require imaging systems. For these imaging systems the absolute responsivity must be calibrated. In literature integrating spheres have been used as a uniform extended source together with tuneable lasers for the absolute radiance calibration. To cover a wide wavelength range, the laser-based method requires a series of tuneable lasers to measure the absolute responsivity directly or a single laser line near the centre wavelength of the radiation thermometer in combination with the relative result from the monochromator based method.

However, the small bandwidths of most laser systems can introduce large uncertainties due to interference effects at the optical elements of the radiation thermometers. This effect is absent in the broad band monochromator-based method. In this paper the monochromator based technique combined with an integrating sphere is used for an absolute calibration of a focussing system.

This paper will describe the set-up and preliminary results of measurements obtained both, at the monochromator based spectral radiance comparator, and the tunable laser in photometry (TULIP) facility of PTB.

CONSTANT-VOLUME GAS THERMOMETRY WITH DIFFERENT HELIUM GAS DENSITIES AT NMIJ/AIST

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Constant-volume gas thermometer measurements are performed for thermodynamic temperature measurement in the range from 3 K to 24 K using ^{3}He of three different densities as the working gas. The experimental apparatus for the present work is the same as that used in the measurement reported previously (Int. J. Thermophys. **29**, 31 (2008)). The temperature of the triple point of neon (about 24.5561 K) realized by using a sealed cell of NMIJ/AIST is used as a reference temperature for the gas thermometer. Densities of 127 mol m^{-3} and 277 mol m^{-3} are used for the working gas in addition to a density of 168 mol m^{-3} used in the previous measurement. The pressure of the working gas is measured for each gas density at more than 25 different temperatures in the range from 3 K to 24 K. The dependence of the gas pressure on the density is observed to be consistent with the virial coefficient of 3He adopted for the interpolating constant-volume gas thermometer in the International Temperature Scale of 1990 (ITS-90). The present results are compared with those obtained previously from the measurement using only a gas density of 168 mol m-3 and with the ITS-90 temperatures obtained from the interpolating gas thermometer.

ACOUSTIC THERMOMETRY: NEW RESULTS FROM 7 K TO 273 K AT LNE-INM/CNAM

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We used a quasi-spherical cavity as an acoustic and microwave resonator to measure the thermodynamic temperature T in the range from 7 K to 273.16 K, and we compared it with T90, the temperature of the International Temperature Scale of 1990 (ITS-90).

The quasi sphere used is one of the resonators developed for the redetermination of the Boltzmann constant. The cavity was intensively studied in helium at 273.16 K, its metrological characteristics are now very well known and it is possible to use it as a primary thermometer.

Two different methods were used to measure the temperature.

With the first method, microwave and acoustic resonance frequencies are measured both at the unknown temperature and at 273.16 K, and the unknown temperature is determined from the ratio of the two measurements.

With the second method, the measurements are carried out only at the unknown temperature and the temperature value is determined using the Boltzmann constant value.

The two methods were compared. The results obtained and the associated budgets of the uncertainties are presented in this paper.

In the range from 7 K to 24.5 K we obtained new values of T-T_{90}, in agreement with the recent results obtained from dielectric-constant gas thermometry, and we achieved uncertainties that are comparable or smaller than those achievable using the interpolating constant volume gas thermometer currently defined in the ITS-90.

Thermophysical Properties

Wednesday
8:20 to 10:20
Emerald 2
Session Chairman: Francesco Righini

DESIGN AND CAPABILITIES OF A CUSTOM-MADE THERMOSTAT FOR A HIGH-ACCURACY ADIABATIC CALORIMETER

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A high-accuracy adiabatic calorimeter (AC) is being developed at the Italian Istituto Nazionale di Ricerca Metrologica (INRiM) to investigate thermophysical properties of small amounts of solids and fluids, including bio-diesels and biological materials. The designed apparatus is intended to perform absolute heat capacity measurements in the temperature range between 240 K and 420 K. Such kind of investigations require accurate temperature measurements and controls. Therefore a dedicated thermostat has therefore been studied and assembled at INRiM in order to obtain temperature stability and uniformity within 1 mK all over the application range of temperature. The accuracy of the temperature measurements is obtained by means of a capsule standard platinum resistance thermometer calibrated at the national temperature fixed points.

This paper describes the AC calorimeter project and the dedicated thermostat design. The temperature automatic control principle is reported, together with the preliminary results obtained in terms of thermal conditions and measurements.

NORMAL SPECTRAL EMISSIVITY OF THE INDUSTRIALLY USED ALLOY HS2-9-1-8 AT 684.5 nm

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The Subsecond Thermophysics Workgroup at TUG uses a fast pulse-heating system, which allows the determination of normal spectral emissivity under pulse heating conditions. Although mainly intended for the investigation of thermophysical properties such as electrical resistivity, specific heat capacity and density of solid and liquid metals and alloys as a function of temperature, the apparatus can also be used for measuring normal spectral emissivity at melting and in the liquid phase. For this purpose, a laser polarimeter, proposed and later in the 1980's developed by R. M. A. Azzam for the determination of optical constants without any moving parts, was adapted for this µs-pulse heating experiment.

By recording the change in polarization of a laser beam reflected off the surface of the wire-shaped sample material during a pulse heating experiment, and evaluating data using the Stokes formalism, measurements of temperature-dependent normal spectral emissivity at the used laser wavelength are possible. As knowledge of emissivity and its behaviour throughout the liquid phase does improve the temperature determination, industry and especially the industrial cooperation partner Böhler Edelstahl GmbH & Co KG is interested in emissivity data for numerical simulations of plastic deformation and remelting processes as well as for process optimisation.

There are three different liquid state behaviours of normal spectral emissivity at 684.5 nm that occurred in numerous experiments with various sample materials, namely increasing, decreasing and constant emissivity with increasing temperature. It's obvious that the behaviour of normal spectral emissivity in conjunction with the radiometric temperature measurement is needed to achieve reliable thermophysical properties of liquid metals, and that's the main target of the present investigations.

Within this paper normal spectral emissivity data at 684.5 nm for HS2-9-1-8 steel at melting and in the liquid state is presented.

Research supported by Böhler Edelstahl GmbH & Co KG and the „Forschungsförderungsgesellschaft mbH, Sensengasse 1, 1090 Wien, Austria“, project 812972.

VIRTUAL EXPERIMENTS BY PULSE HEATING TECHNIQUES: CYLINDRICAL SPECIMEN MODELS

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A research project to evaluate virtual experiments by pulse heating techniques was recently started at IPCF-CNR. The work follows similar activities by other research groups, which have developed mathematical models for pulse heating techniques [1] or modelled specific geometries used in subsecond pulse heating experiments [2]. In our case, the experiment is simulated calculating the time dependent behaviour of the relevant quantities by means of a finite-element technique allowing the modelling of different physical processes.

The first stage of the project involves the development of bi-dimensional models for specimens heated by current pulses of subsecond duration. Some materials have been considered, after a careful literature search and evaluation of their thermophysical properties (heat capacity, electrical resistivity, thermal conductivity and hemispherical total emissivity) over the entire temperature range of operation. Different wire and rod shapes have been evaluated, to find an appropriate geometry so that the specimen may be used both as a heater and as a temperature sensor during the virtual experiment.

Preliminary results of optimal geometries for tungsten and niobium over the entire temperature range up to their melting points will be presented. The optimisation of the specimen requires a careful evaluation of its virtual temperature profile both in the longitudinal and in the radial directions, considering realistic values of the experimental apparatus.

The final aim of the project is the establishment of a reliable predictive numerical tool for subsecond current pulse heating methods, as well as the conception of new measurement techniques. The virtual experiment verifies the feasibility of the proposed measurement technique, in relation to the capabilities of existing instrumentation, and also the accuracy of measurement necessary to reach adequate results, without the necessity of an immediate realization of the experimental apparatus.

[1] J. Spišiak, F. Righini, G.C. Bussolino; "Mathematical models for pulse-heating experiments" *Intern. J. Thermophysics* **22**, 1241-1251 (2001).
[2] E. Kaschnitz, P. Supancic, “Three-dimensional finite-element analysis of high-speed (millisecond) measurements”, *Intern. J. Thermophysics* **26**, 957-967 (2005).

MEASUREMENT OF THERMAL CONDUCTIVITY OF CONSTRUCTION MATERIALS

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Guarded hot plate apparatus (GHPA) is a primary instrument to measure thermal conductivity of insulation solid materials. GHPA operates in a steady state of heat flow in one direction. This measurement technique is applied in a wide variety of samples and temperature ranges. The best results are obtained for homogeneous and flat isotropic samples.

We use at CENAM a double side GHPA as a national standard for thermal conductivity of insulation materials.

In Mexico there is the need to measure thermal conductivity of building materials because there is no data for many of them, or the information found in literature does not correspond to their usage conditions.

When measuring building materials, different problems arise: non-uniform thickness, non-homogeneous samples, rough and irregular materials, thus an experimental setup is required to measure the thermal conductivity of those materials.

We present the thermal conductivity of the following materials: solid adobe, perforated adobe, concrete slab, refined mortar slab, and two types of insulation materials, polystyrene and glass fiber, with their respective uncertainties.

THERMOPHYSICAL PROPERTIES OF MATERIALS: NEW CHALLENGES FOR REDUCING THE UNCERTAINTIES

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Within the international framework of the CCT -WG9 and taking into account the great challenge of the sustainable development, LNE as the French National Metrology Institute (NMI), has been developing in a recent past a unique metrological platform structured around complementary, versatile and very accurate facilities for measuring the main physical and chemical properties of materials. The objective is to answer to the new societal issues and industrial needs. Research, development of reference measurement methods, dissemination of measurements or SI -traceable calibrations, certification of reference materials and knowledge transfer to Industry, and Society constitute the usual and common cornerstones of the laboratory activities.

Beyond the basic activities necessary to strengthen the different metrological scales emerging in force relatively to the thermophysical properties of materials, this article aims to describe for the main thermophysical properties of materials, namely the radiative properties, the thermal diffusivity, the thermal conductivity, the specific heat, the calorific value ... a set of challenging projects that drive the laboratory at the heart of a European metrological network still under construction. These scientific developments are illustrated through some issues relatively to energy or environment. For instance, with regards to:

Buildings thermal efficiency: for improving the measurement of thermal resistances of materials used to reduce the heat transfer coefficients of building envelopes, new metrological developments have been suggested as a whole project of investigations. Thickness of traditional insulating products are increasing, 30 cm is becoming common. Low emissivity glass panes are now commonly used for insulating glass units and new super insulating products are appearing on the market. Existing reference instrumentation for thermal conductivity calibrations was optimized for insulating products with a thickness fro m 2.5 to 15 cm (uncertainties about 1 to 2%). For 30 cm thick insulating products the relative uncertainty with existing instrumentation is about 3% or more. The objective should be to reduce that relative uncertainty to about 1 to 2% for thick products.

In the same way, some other metrological investigations are presented and discussed as well.

Engines efficiency: for enhancing efficiency of turbines, generators and diesel engines: in term of metrology, the main objective for the laboratory is to

characterize the high temperature thermal properties of advanced materials, especially refractory alloys and ceramic coatings used in cogeneration systems, conventional fossil fuel power plants and vehicle engines, as well as to improve the traceability of the corresponding measurements.

Biofuels metrology: The physical properties (density, thermal conductivity, thermal expansion, specific heat, viscosity…) of biofuels mainly depend on the feedstock sources used for their production. These properties can also v ary depending on the water content in biofuel and on the biofuel blend ratio (biofuels being used alone or blended with fossil fuels). The growing use of biofuels leads to an increasing interest in modeling different parameters for instance during the development phase of engines or gas turbines. These simulations require the knowledge of the biofuels thermo chemical and thermophysical properties. Therefore it is necessary to determine accurately these properties in function of temperature …

Thus this article is intended to underline the metrological challenges, including the current developments in progress, endorsed by LNE in that field of thermophysical properties today and for the near future. Methods and associated uncertainties will be highlighted.

THE THERMAL CONDUCTIVITY OF HUMID AIR

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Humid air is one of the most important gaseous mixtures in science and engineering, due to the rising concerns about efficient air conditioning and economical solutions for reliable and more environmentally friendly heat transfer fluids. However, in spite of the use of steam and humid air in industry since the 19th century and all the efforts of IAPWS, there is almost no publish data in the literature for the thermal conductivity of humid air, since the pioneering work of Grüß and Schmick [1], in 1928, working for Siemens.

We report in this paper the measurement of the thermal conductivity of humid air as a function of pressure, temperature and molar fraction of water, for pressures up to 5 MPa and temperatures up to 400K, for different water contents (up to 5% w/w), completing a previous report [2]. Measurements were performed using a transient hot-wire apparatus previously reported [3], capable of obtaining data with un expanded uncertainty of 1% for gases. However, and because the air/water mixture in the vapor phase becomes corrosive above 373 K and pressures greater than 5 MPa, the apparatus, namely the pressure vessel and the cells had to be modified, by coating all the stainless steel parts with a TiN PVD thin film coating, about 4 μm thick.

Experimental details regarding the preparation of the samples, the precautions that were taken to avoid condensation in the tubes connecting to the measuring cell , and the method developed for obtaining reliable values of the water content for the gas mixtures, are discussed. Attempts to correlate our data using the kinetic theory for dense gases will be discussed.

[1] Grüß, H, Schmick, H., Wissensch. Veröffentl. Aus Siemens Konzern, 203-224 (1928).
[2] S. G. S. Beirão, A. P. Ribeiro, M. J. V. Lourenço, F. J. V. Santos and C. A. Nieto de Castro, “Thermal Conductivity of Humid Air: A First Approach”, Thermal Conductivity 29, Eds. John Koenig and Heng Ban, DEStech Publs., Inc., (2008).
[3] Beirão, S. G. S., Ramires, M. L. V., Dix M., Nieto de Castro, C. A., Int. J. Thermophys., 27, 1018 (2006).

Moisture profile and transport

Wednesday
8:20 to 10:20
Adria
Session Chairman: Vito Fernicola

DETERMINATION OF MOISTURE DIFFUSIVITY AS A FUNCTION OF BOTH MOISTURE AND TEMPERATURE

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The moisture diffusivity is commonly determined from transient moisture profiles as measured in a capillary absorption experiment. In this experiment a specimen is at the bottom side brought in contact with a free water layer and water is taken up by capillary suction. Moisture contents are measured at different positions and time intervals during the experiment producing moisture profiles versus time. An inverse analysis of moisture profiles is then used for the calculation of moisture diffusivity as a function of moisture content. Temperature dependence of moisture diffusivity is mostly neglected, mainly for practical reasons, because it involves a laborious measuring procedure. However, it is not quite clear when (and if anything) this simplification can be used in computational models without a significant departure from the physical reality. In this paper, the moisture diffusivity is analyzed as a function of both moisture and temperature and the possibilities to neglect its temperature dependence are discussed.

The experiment is arranged in the form of 1-D water suction into dry samples of autoclaved aerated concrete which are placed in a climatic chamber. After a specific temperature is chosen, the sample is thermally homogenized and then subjected to water penetration from one of its face sides (the lateral sides are provided by a water- and vapor-proof coating). The movement of water front is continuously monitored, and the experiment is stopped before the water suction has reached the remote end of the measured sample. The moisture diffusivity is then calculated from the moisture profiles determined by the gravimetric method using an inverse analysis involving Boltzmann transformation.

Experimental results show that the dependence of moisture diffusivity of autoclaved aerated concrete on temperature can only be neglected if the temperature differences in a studied building structure, typically a building envelope, are limited to approximately 10 °C. Otherwise, the accuracy of hygrothermal calculations could be seriously impaired.

APPLICATION OF TIME-DOMAIN REFLECTOMETRY FOR MEASUREMENT OF MOISTURE PROFILES IN A DRYING EXPERIMENT

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The time-domain reflectometry (TDR) method can be generally classified as a dielectric method, based on an analysis of the behavior of dielectrics in a time-varying electric field, and consists in the measurement of permittivity of moist porous media. The determination of moisture content using the permittivity measurements is based on the fact that the static relative permittivity of pure water is equal to approximately 80 at 20 °C, while for most dry building materials it ranges from 2 to 6. In this paper, the TDR technique is used for measuring moisture profiles in calcium silicate during a drying experiment. The motivation for this experiment is to provide information on the possible differences in moisture transport in porous materials during wetting and drying phases which would cause hysteresis of transport parameters, thus affect hygric calculations.

In the drying experiment the calcium silicate samples are saturated at first by water, and their lateral sides and one of the face sides are water- and vapor-proof insulated to ensure 1-D water transport. Then, the drying process is started in an environment with a relative humidity close to 0%. Moisture profiles are measured in specified time intervals using the TDR method. The experiment is stopped when the moisture content along the whole length of the sample is lower than the maximum hygroscopic moisture content.

Experimental results show that moisture transport in calcium silicate is significantly slower in the drying phase than in the wetting phase. From the point of view of the theory of transport processes in porous materials, there are several effects which are responsible for these differences, namely the water adsorption on pore walls and subsequent surface transport of water during the wetting phase which is limited or absent during the drying phase, possible osmotic transport during the wetting phase, and the “bottleneck” effect during the drying phase which slows down the removal of water from bigger pores with narrow openings. The separation and quantification of these particular effects is, however, not feasible using a single type of experiment presented in this paper, and further experimental analysis is necessary.

RAPID MOISTURE MEASUREMENT WITH MICROWAVE RESONANCE TECHNOLOGY IN INFANT FORMULAS

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Besides breast milk, infant formula is the only other milk product that the medical community considers nutritionally acceptable for infants under the age of one year.

Water content is a very important value for the quality especially for the physical, microbial and shelf-life properties of a product. Accurate and rapid measuring of water or moisture content is the basis for producing dried powdered food like infant formulas.

The objective of this work was to investigate if the water content of infant formulas can be determined by microwave resonance technology.

Microwave resonance technology is a non-destructive, non-chemical and very fast method to determine moisture in various kinds of food.

For measuring moisture, a low-energy microwave field is generated which shows a sensor-specific resonance. Solid products and water molecules that are brought into the microwave field influence the resonance frequency and the resonance bandwidth. Because of their dipole property, the free water molecules permanently try to align in the alternating field but cannot follow it. Thus, they absorb energy. Frequency shift and attenuation of the resonance are measured and converted into a moisture value that is independent of varying product density. As all secondary methods, this techniques needs to be calibrated with a reference method.

It could be shown that water content in infant formulas can be determined by using the microwave resonance technique. Calibrations were based on different methods like oven drying, volumetric Karl Fischer titration and an automated Karl Fischer titration with a heating oven.

CAPABILITIES OF AUTOMATED KARL-FISCHER-TITRATION OF SELECTED DAIRY PRODUCTS

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In dairy products, the content of water or moisture is usually measured by drying techniques. Classical methods such as oven drying, vacuum drying, freeze drying, infrared or microwave drying do not distinguish between water and other volatile substances.
The result of these methods is not water content but the mass loss which the product undergoes under the conditions applied. Such measurements can last up to 6 hours.
In contrast, accurate and especially rapid determination of water content is an important aim in food analysis.
The most important primary method to determine water content is the Karl Fischer titration. It is based on a chemical reaction selective for water.
Conventional Karl Fischer titrators are computer-controlled but require an operator for the manual loading of samples, starting the measurement process and observing the results.
In order to simplify this procedure two different automated methods of the Karl Fischer titration were tested. In both methods, sealed vials containing the sample are loaded on a carousel and are automatically placed in the titrator.
Method 1 is a combination of a drying method and the volumetric Karl Fischer titration. The vial with the sample is automatically placed into a small heating oven. Subsequently, a stream of dry air or nitrogen is led into the vial. The released water, possibly together with other volatiles, is transported into the titration vessel by the carrier gas stream. Contained water is then determined specifically by volumetric titration. Operation temperatures can be set from 50 °C up to 250 °C.
Method 2 uses different solvents to bring a sample into solution. The products are weighed into sealed vials and placed into a sample rack. After starting the measurement the samples are prepared by automated injection of either one or two solvents and stirring the solution. Samples are then injected into a titration vessel.
Both methods were tested on different dairy products such as butter and cheese and the results compared with those obtained by conventional drying methods. According to operational experience, both methods provide an opportunity to measure the water content very simply and effectively without trained operators.

EXPERIMENTAL INVESTIGATION OF THERMAL PROPERTIES OF INSULATING MATERIALS AS THE MOISTURE CONTENT VARIES

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Moisture condensation causes negative effects on the behaviour of building envelopes in terms of deterioration of materials, increasing of the thermal conductivity of the insulating material, weakening of structures, growing of mould and mildew. The presence of moisture in building walls is due to different reasons such as the use of wet materials during construction, capillary rise from ground, infiltration of rainwater or melted snow, leakage of piped water, continuous high humidity conditions and condensation of water vapour in building envelopes.

Although the relevant international standards [1] seem to allow moderate condensation to be neglected, or define the thermal conductivity as the value obtained for a uniform moisture distribution [2] in absence of convective transport phenomena, thermal conductivity measurements made on wet porous materials require a thermal gradient to be applied across the sample. This gradient may cause vapour and liquid phase transport until a dynamic equilibrium is reached if the samples are sealed by means of impermeable covers. The measured (effective) thermal conductivity value, therefore will depend on the moisture profile established in the sample in stationary conditions and on the latent heat contribution due to phase change. In order to determine the effective thermal conductivity is, therefore, necessary to investigate heat and mass transfer phenomena which take place in the sample. In the present paper, the authors measure the effective thermal conductivity of some important insulating materials for building envelopes as the water content varies. Finally, the contribution of mass transfer to the effective thermal conductivity will be determined by means of the solution of inverse problems.

[1] ISO 13788: 2001 – Hygrothermal performance of building components and building elements. Internal surface temperature to avoid critical surface humidity and interstitial condensation. Calculation methods. ISO – International Organization for Standardization.
[2] ISO 10051:1996, Thermal insulation -- Moisture effects on heat transfer -- Determination of thermal transmissivity of a moist material. ISO – International Organization for Standardization.

NON-DESTRUCTIVE MOISTURE CONTENT MEASUREMENT OF BIOABSORBABLE POLYMERS USED IN MEDICAL IMPLANTS

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This paper describes measurements of moisture content of samples of a bioabsorbable polymeric material used for medical implants. The polymer is used to produce screws that are used in surgical procedures to attach tendons or ligaments to bone tissue. During the healing process, the polymer is intended to degrade and be absorbed into the body. This work is a step towards characterising the degradation process, by identifying a non-destructive moisture measurement that can be correlated with component degradation.

Polymer samples in the form of narrow cylinders were subjected to accelerated degradation for time intervals ranging from 1 hour to 24 hours in containers of water heated to 70 °C. Their moisture contents were then measured non-destructively, followed by further analysis using destructive methods.

Non-destructive measurements of the samples were made by a microwave resonance technique. A sample placed in a microwave resonant cavity induces a shift in resonant frequency within the cavity as a function of the moisture content of the sample. A second measurement was made by evolved vapour analysis. The water in each sample was evolved through heating to 180 °C (which was enough to melt the polymer). The water uptake of a dry air flow passed over the sample was measured using a P2O5 electrolytic cell. This technique enabled a measurement of the mass of water in each sample to be related to the shift in microwave resonant frequency. As a second measure, weighing before and after the evolved vapour analysis also gave values of moisture content (gravimetric loss on drying) of the same samples.

The gravimetric results for moisture content obtained after heating the samples to 180 °C were always higher than those for evolved vapour analysis water content and this difference is believed to be due to the evaporation of other volatiles from the polymer in addition to water. Overall, the study demonstrated that changes in water content corresponding to progressive degradation of the polymer samples can be measured non-destructively by a microwave resonance technique, with appropriate sensitivity.

Fixed Points IV

Wednesday
10:50 to 12:30
Emerald 1
Session Chairman: Stanislav Ďuriš

THERMAL ASSESSMENT AND REALIZATION OF THE INDIUM MELTING POINT BY USING AN ADIABATIC CALORIMETRY TECHNIQUE

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Within the framework of the Euramet project 732, LCM/LNE-CNAM has recently proposed a new device to assess the melting point of Indium by using the adiabatic calorimetry approach. A first apparatus based on a "Cell-Within-Cell" configuration was developed and experimentally exploited [1]. First results showed extraneous heat flows due to the geometrical characteristics of the cell, disturbing significantly the isothermal conditions within the calorimeter. Such thermal effects were also clearly highlighted by a numerical model developed in parallel [2]. Regarding the excellent agreement between the model and experiments, an optimization step has been carried out to develop most adapted cell's geometry [3]. A new cell was subsequently constructed and arranged into the calorimeter.

The purpose of this paper is to introduce the thermal behaviour of the apparatus, highly enhanced, while presenting some series of measurements; on the one hand, the melting point of Indium under adiabatic conditions is studied, and on the other hand the continuous heat flow method under isothermal conditions is assessed. The results obtained are compared and analyzed according to the impurities concentrations into the ingot (SIE method).

[1] R. Morice & al, «*Realization of the Indium Fixed Point by an Adiabatic Technique*», Proc. Of Tempmeko 2007, International Journal of Thermophysics **29** (5) pp 1785-1795, Springer Netherlands (2008)
[2] V. Le Sant, R. Morice & G. Failleau, «*Modeling of Transient Heat Transfer in Temperature Fixed Points: Indium Cell Design*», Proc. Of Tempmeko 2007, International Journal of Thermophysics **29** (5) pp 1772-1784, Springer Netherlands (2008)
[3] G. Failleau, V. Le Sant, R. Morice & P. Ridoux, «*Thermal Assessment Of Fixed-Point Cell Design By Numerical Modelling*», Proc. of Tempbeijing 2008, Acta Metrologica Sinica, **29** (4A) (2008)

PROCEDURE FOR THE IMPURITY-RELATED CORRECTION AT THE INDIUM FIXED POINT

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The determination of the influence of impurities on fixed-point temperatures of the ITS-90 requires the completion of several tasks. In this paper we present the progress made at Physikalisch-Technische Bundesanstalt (PTB) and BAM Federal Institute for Materials Research and Testing and discuss remaining questions.

We describe the projected characterization procedure at PTB that is based on the established SIE method, using a new indium fixed-point cell as an example. This procedure includes a traceable chemical analysis of the material in the fixed-point cell with sufficiently low uncertainties, the individual experimental determination of the influence of the quantified impurities on the fixedpoint temperature and the establishment of direct links to the phase transition temperatures of the national standard and of an assumed material of ideal purity.

A characteristic difference to the common practice is the chemical analysis of the material in the fixed-point cell after the determination of its freezing temperature. This allows the detection and consideration of contamination and purification effects due to the filling process or due to the contact with the carbon crucible and other parts of the fixed-point cell. A chemical analysis of an indium fixed-point was carried out by BAM with so-far unachieved relative measurement uncertainties below 50%. These provide experimental evidence for the precipitation of some impurities which is apparently inconsistent with the corresponding binary phase diagrams, but in accordance with a recent publication. Consequences for the use of the SIE method shall be described briefly at the end.

INVESTIGATION OF NEWLY DEVELOPED ITS-90 SILVER AND COPPER POINT BLACKBODIES

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For the realisation of the ITS-90, LNE-INM uses a series of Cu and Ag fixed point blackbody cavities and a monochromator-based radiance comparator or linear pyrometers. As it is usual in the field of contact thermometry, the national reference will now be represented a batch of fixed point cells. Decision was made to construct multi-purpose Cu and Ag cells which can be implemented in various types of furnaces depending on their size. The cells are to be compared, on a semi-annual or annual basis, to detect any unexpected drift of one of the cells in order to discard it.

In this study, the constructed cells, of different types and dimensions (3 sizes for the Cu point and 2 sizes for the Ag point) have been extensively implemented and characterised in different furnaces of different temperature uniformity capabilities. The temperature differences between cells, although small, are analysed and the effects of the temperature drop as well as the emissivity correction are estimated. The uncertainty budget for the realisation of these fixed points as well as the effects on the extrapolation to the highest temperatures are investigated.

In this paper, we will give a description of the cells and the furnaces used and present the results of the characterisation of the cells and their comparison.

MODELLING OF TRANSIENT HEAT TRANSFER IN ZINC FIXED POINT CELL

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Current practice in fixed-point calibration recommends usage of furnaces that provide best achievable uniform temperature distribution at the outer segment of the immersed portion of the fixed-point cell. The effect of any inhomogeneous temperature distribution is somewhat unrevealed, and its coverage in literature is insufficient.

In order to to establish some estimation of the influence of the inhomogeneous furnace temperature distribution to the calibrating process, a numerical study was conducted. The study was conducted using FLUENT software package employing finite volume method on non-structured grids. The problem was considered as axially symmetric. The domain of the calculation consisted of the whole cell volume and its geometry was modeled such to distinguish each of the individual elements of the fixed-point cell assembly.

The material properties of the assembly elementss were treated as temperature dependent, while their surface properties were kept constant. The mathematical model employs transient conductive heat transfer along with solidif ication model for Zinc so as radiation heat transfer between surfaces in all argon - filled cavities. The effect of natural convection was assumed to be of minor importance and was therefore neglected thus saving significant amount of computational effort. The calculations covered the whole duration of Zinc solidification process.

Several variants of boundary conditions at the fixed – point cell's outer surface were applied. The variants distinguish: the homogeneous temperature distribution at the outer skin of the immersed part of the fixed point cell, the plus and minus 1 K/m of gradient added to base temperature distribution in axial direction and the presence or absence of cold-roding.

The base outer temperature was kept 1 K below the solidus temperature giving approximately 6 hours of calibration process. The study shows that such linear gradient of +/- 1 K/ m influences the process solely in its duration, prolonging it or shortening it for approximately 20 minutes.

Calibration Procedures and Facilities III

Wednesday
10:50 to 12:30
Emerald 2
Session Chairman: Kazuaki Yamazawa

ANNEALING STATE DEPENDENCE OF THE CALIBRATION OF TYPE-R AND S THERMOCOUPLES

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Type R (Pt-13%Rh vs. Pt) and type S (Pt-10%Rh vs. Pt) thermocouples are widely used as a reference and working standard for temperature measurements both in calibration laboratories and in industry for temperatures up to 1600 °C. Many laboratories claim that the best achievable uncertainty is 0.1 °C up to 1000 °C and 0.3 °C up to 1550 °C, and international comparisons confirm that this is achievable practically. However due to (i) preferential Rh oxidation of the Pt-Rh alloy thermoelement and (ii) defect quenching effects, these thermocouples suffer from reversible hysteresis in their calibration. As a result calibration laboratories usually perform some heat treatment of the wire prior to calibration to attain a specific 'annealing state', at which the calibration is performed. Internationally there are two commonly used annealing states for these thermocouples: the '450 °C annealed state', and the '1100 °C quenched state'. High-level comparisons between laboratories in the calibration of type R or type S thermocouples will rigorously specify the annealing state in the protocol, so any systematic differences due to the choice of annealing state will be masked. This paper compares the calibration of several thermocouples using the two common annealing states, finding that the difference can be as large as 0.2 °C at 961 °C, larger than the best calibration uncertainties reported. The paper examines the advantages and disadvantages to the user of calibrations performed in each state, and the implications for the uncertainty analysis for calibration and use of type R and type S thermocouples.

Keywords: Annelaing state, Hysteresis, Inhomogeneity, Pt-Rh vs. Pt Thermocouple.

MELTING TEMPERATURE OF HIGH TEMPERATURE FIXED POINTS FOR THERMOCOUPLE CALIBRATIONS

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Thermocouples can be calibrated at pure metal ingot-based fixed points at temperatures up to the freezing point of copper (1084.62 °C). For Pt/Pd thermocouples, the deviation from the accepted reference function very often takes an approximately linear form up to the copper fixed point. The calibration of Pt/Pd thermocouples may therefore be more amenable to extrapolation than that of Pt/Pt-Rh thermocouples.

Here, the melting temperatures of a Co-C and a Pd-C eutectic fixed point are determined by extrapolating the deviation functions of several Pt/Pd thermocouples, after the fashion of Edler et al. (F. Edler et al., *Int. J. Thermophys.* (2007) **28**, 1983-1992). The results are compared with the melting temperatures measured using non-contact thermometry. The expanded uncertainty (k = 2) of the melting temperatures determined by extrapolation of the Pt/Pd thermocouple calibrations is ± 0.32 °C for the Co-C fixed point, and ± 0.49 °C for the Pd-C fixed point. For both fixed points, these uncertainties are comparable to those of non-contact thermometry measurements. While a number of assumptions are made in performing the extrapolation of the calibrations, the method does appear to offer a useful complement to non-contact thermometry measurements.

A DEVIATION FUNCTION USED SECOND RELIZATION OF THE ITS-90 IN THE SUBRANGE –83.8033 K TO 273.16 K

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This paper gives an correlation relation between the resistence ratios of triple point of argon and mecury. By this relation, the resistance ratio of the triple of argon can be exptrapolotes from that of mercury and a deviation function which defined in the range –83.8058 K to 273.16 K can be only determined from the calibration values at the triple points of water and mercury. It is a closed approximation to the ITS-90 deviation function in the subrange. Using it, we can be save the calibration at the triple point of argon. Twenty five SPRTs are used to check the function. The errors are less than 5mK. It is sufficient for secondary mesurement.

NEW PROTOTYPE APPARATUS FOR CALIBRATION OF SURFACE TEMPERATURE SENSORS

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An error that appears as a result of temperature measurement by contact sensors at solid contact surface depend on sensor construction and on the thermal properties of the object whose temperature we measure. In certain measurements the required uncertainty is very low, even smaller than 1 °C. In such cases it is very important that there is possibility of calibration with a system having proven traceability and small uncertainty.

By combining and careful choice of construction solutions, materials and measurement-regulation system of temperature, by combining different techniques of identification of temperature field it is possible to get homogenous temperature profile at the reference plate and achieve appropriate calibration points at the moment when the best thermal situation in whole system is gained, which can be achieved using an optimal measurement period.

In this paper new construction solutions will be presented. Moreover, a theoretical evaluation of the convective flow near the reference surface will be effectuated and will be estimated its influence on the measurement error. These will be implemented in prototype apparatus. In such a way we will have the possibility to properly estimate the most important contributions to the measurement uncertainty within this calibration method.

The paper will also present some basic characteristics and differences with some other systems for calibration of surface temperature sensors.

A CALIBRATION SYSTEM FOR HEAT FLUX METERS

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Improvements on the energy efficiency of buildings are called upon by European directives, such as the 2002/91/CE, which set maximum values for energy needs and for the thermal transmittance of building envelope components. In-situ measurements of envelope components are needed: i) to estimate the thermal transmittance of existing buildings in order to make the energy certification; ii) to validate the designed energy performance of new buildings. When the design data about existing building are not available, it can be necessary to perform invasive tests to detect stratigraphy and materials. Alternatively, the thermal transmittance can be measured in-situ, according to ISO 9869, by means of a heat flux meter (HFM). As thermal conductance is a steady-state thermal property, to perform in-situ accurate estimations, in not stationary conditions and at different thermal regimes, long measurement intervals and proper post processing procedures are required.

A HFM is basically made of a thin plate of known thermal conductivity and a thermopile to measure the temperature difference across the plate itself. In the last few years, several studies were carried out to better understand its performances in order to improve the calibration process. Many HFMs are calibrated at high heat fluxes (i.e., greater than 100 W/m^2) and, just relying on their linearity, are used to measure lower heat fluxes. On the other hand, only few NMIs are able to provide HFM traceability to lower heat fluxes commonly found in building applications.

In the paper, after a brief analysis of the HFM calibration approaches, the authors present a numerical study and an experimental analysis aimed at the realization of a HFM calibration system operating at moderate heat fluxes (i.e., lower than 100 W/m^2). Numerical predictions about the metrological performance of such a calibration system have been made by means of a commercial Finite Element Code (COMSOL®). On the basis of such numerical studies, a HFM calibration system has been constructed within a collaboration between INRIM and the University of Cassino. Laboratory experiments have then been carried out in order to: i) validate the numerical study; ii) characterize the proposed HFM calibration system in a temperature range from about 0 °C to 40 °C and with a heat flux range from 20 W/m^2 to about 200 W/m^2.

Humidity and Moisture Standards II

Wednesday
10:50 to 12:30
Adria
Session Chairman: Christopher Meyer

PRESSURE DROP CONSIDERATIONS IN THE CHARACTERIZATION OF DEWPOINT TRANSFER STANDARDS AT HIGH TEMPERATURES

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The development of precision optical dew-point hygrometers has experienced a very significant improvement in the last decade, with instruments now able to achieve levels of reproducibility better than ± 20 mK over a wide temperature range. These instruments are commonly used as transfer standards for the comparison of Standard Humidity Generators (SHGs). The current levels of CMC claims by the National Metrology Institutes (NMIs) and their Designated Institutes (DIs) require strict measurement protocols to reduce the impact of the dominating influence quantities on the comparison results. These include the minimization of mirror gradients through fixing the head to mirror differential, the loading effects, by fixing the gas flow and pre-conditioning the gas temperature, the reading of the Platinum Resistance Thermometer embedded in the mirror, using an external resistance bridge, amongst others.

Until very recently, the EURAMET and CCT Key and Supplementary comparisons have not covered the range above 20 °C (e.g. CCT/K6, EURAMET-T.K6 and APMP-K6, in the range –50 °C to +20 °C). However, the onset of EURAMET-T.K8 and CCT/K8, covering the range 30 °C to 95 °C, have required further considerations in defining the reproducible measurement conditions adequate for the different sampling configurations required by the various types of SHGs used in the national realizations of dew-point temperature.

The paper addresses the methodology developed for the measurement protocols of the highrange comparisons and the method validation in the range 30 °C to 95 °C, consistent with the specifications of the available state-of-the-art transfer standards.

Measurement results obtained in the laboratories of the three holders of national humidity standards are presented and discussed.

DEVELOPMENT OF A DEWPOINT GENERATOR FOR GASES OTHER THAN AIR AND NITROGEN AND PRESSURES UP TO 7 MPA

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The primary humidity standards currently available at the national metrology institutes throughout the world operate only with air or nitrogen as carrier gas and at quasi-atmospheric pressures. Industrial on-field humidity measurements are performed with commercial humidity sensors and analyzers on a variety of gases such as natural gas, SF_6 and Cl and at pressures up to 7 MPa and even higher. Although manufactures of humidity sensors and analyzers claim to be able to cope for the gas dependency and pressure dependency with different methods, no indisputable evidence of that exists, given the lack of a primary humidity standard operating with carrier gases other than air and nitrogen and at pressures far from atmospheric level.

VSL is developing a two-temperature dewpoint generator able to operate also with gases other than air and nitrogen and at pressures up to 7 MPa. In this paper we will report on the status of the development of this new dewpoint generator, with particular emphasis on:

- the design of the saturator,
- the saturation efficiency tests.

NEW PRIMARY DEW-POINT GENERATORS AT HMI/FSB-LPM IN THE RANGE FROM –70 °C TO +60 °C

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In order to extend the dew-point range and to improve the uncertainties of the humidity scale realization at HMI/FSB-LPM, the new primary low-range and high-range dew-point generators were developed and implemented in cooperation with MIKES in year 2009 through EUROMET project no. 912. The Low-range generator is designed for primary realization of the dew-point temperature scale from –70 °C to +5 °C while the High-range generator covers the range from 1 °C to 60 °C. The system is designed as the single pressure – single pass dew-point generator. MIKES designed and constructed both saturators to be implemented in dew-point calibration systems at LPM. The LPM took care of purchasing and adapting liquid baths, of implementing the temperature and pressure measurement equipment appropriate for use in the systems, gas preparation and flow control systems as well as the computer-based automated data acquisition. The principle and the design of the generator are described in detail and schematically depicted. The tests were performed at MIKES to investigate how close both saturators are to an ideal saturator. Results of the tests show that both saturators are efficient enough for a primary realization of the dew-point temperature scale from –70 °C to +60 °C in the specified flow rate ranges. The estimated standard uncertainties due to the non-ideal saturation efficiency are between 0.02 °C and 0.05 °C.

DESIGN AND MODELLING OF A LOW FROST-POINT HUMIDITY GENERATOR

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In recent years the demand for traceability to trace moisture has increased particularly in the pure gases industry where traceable measurements of the water mole fractions at sub-ppm level are needed. At INRIM, dew/frost-point temperature standards has been established in the range between –75 °C and 85 °C which, at lowest temperature corresponds to a water mole fraction in the moist gas close of approximately 1.2 µmol/mol.

To overcome the present range limits, a new low frost-point humidity generator intended for use as a primary standard in the range –98 °C to –10 °C was designed. The lowest temperature corresponds to a mole fraction of 20 nmol/mol. Among the available operating principle, a single-pressure, single pass, generator design was chosen, because of the lowest predicted uncertainty in water mole fraction. The most critical part of a single pass humidity generator is, of course, the gas saturation assembly which is made of a heat exchanger for temperature conditioning of the inlet gas and an isothermal saturator to achieve its saturation at the outlet.

The saturator is intended to work at flow rates in the range from 0.5 L/min to 4 L/min and at controlled pressures up to 250 kPa; the pressure being measured at a static access port near the saturator outlet. The system must fully saturate the carrier gas with a residual unsaturation lower than $1 \cdot 10^{-3}$. The temperature uncertainty goal at atmospheric pressure was set to 0.2 °C at the lowest temperature.

The saturator design was carefully validated by developing a non commercial modelling tool based on the Artificial Compressibility Characteristic Based Split (AC-CBS) procedure and on finite element spatial discretization. The AC-CBS code, recently stabilized by the authors, allows the numerical simulation of problems characterised by the presence of very large source terms. The explicit procedure adopted also allows an easy and efficient parallelization for the solution of complex 3D problems.

A NEW ATTEMPT ON A COULOMETRIC TRACE HUMIDITY GENERATOR

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The need for traceable primary standards in the low frost point region is characterized by meteorological measurements, semiconductor manufacturer, pharmaceutical industry, suppliers of pure gases and some other branches in industry and science. Some attempts were made in the past for the reliable generation of a water content by volume in a carrier gas of lower than 10^{-8} l/l.

These methods for the preparation of trace humidity reference gases include for example two-pressure generators (with saturation over ice), mixing flow generators, generators on the basis of diffusion or permeation and hybrid combinations of the mentioned principles. One of the most promising methods for the trace humidity area is the coulometric trace humidity generation which is directly traceable to the SI. The principle of a coulumetric traces humidity generator (CTHG) bases on Faraday's law. Defined amounts of hydrogen and oxygen are produced by water electrolysis as a function of the electrolysis current. In a subsequent step, the hydrogen and the oxygen are quantitative re-combined by means of a catalyst. There is a clear relation between the amount of water in the carrier gas flow and the electrolysis current *I*, no empirically determined factors are necessary. With the knowledge of the volume flow of the carrier gas nitrogen, the mixing ratio *r* can be determined. PTB has developed a generator of this type. This contribution will show how it works and give you an imagination about its capabilities in the ultra trace humidity region of water contents by volume of lower than $5 \cdot 10^{-9}$ l/l according to frost points about –105 °C. Measurements with classical chilled mirror hygrometers will be presented as well as spectroscopic systems including TDLAS and cavity ring down spectroscopy (CRDS).

Thermoelectric Thermometry

Thursday
8:20 to 10:20
Emerald 1
Session Chairman: Hans Liedberg

MATERIAL PROBLEMS IN USING HIGH TEMPERATURE THERMOCOUPLES

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The measurement of temperatures in the high temperature range between about 1600 °C and 2200 °C by using thermocouples is complicated by serious material problems with increasing temperature. Besides the difficulty to discover suitable and thermoelectric stable thermoelements (for instance high melting metals) also appropriate insulation materials are needed to protect the thermoelements from environmental influences and to provide electrical insulation. Typically refractory metals like tungsten (W) and rhenium (Re) and in particular some of their alloys are used for high temperature thermocouples. Molybdenum, niobium and tantalum play only an ancillary role. Measurement uncertainties achievable by using thermocouples of W/Re alloys are in the order of some Kelvin in the lower part of the temperature range mentioned above and increase to about several ten Kelvin by using them at temperatures higher than 2000 °C. Although such high measurement uncertainties are disappointing, up to now no alternative thermoelement materials are available.

Additionally, limitations of ceramic insulation materials become essential for thermocouple applications at temperatures above about 1600 °C to 1800 °C. Generally, the electrical resistivity of the ceramics decreases with increasing temperature, chemical reactions of the ceramics with the thermoelements may occur and some insulators melt or undergo changes in their crystal structure. For instance, a special problem arises by using alumina (Al_2O_3) which is considered as the most widely-used ceramic for standard thermocouple insulations. Reduction reactions of the Al_2O_3 start in contact with graphite at temperatures of about 1600 °C, i.e. for instance during a calibration of a thermocouple inside the graphite crucible of a metal-carbon eutectic fixed point.

The results of some investigations of the thermoelectric stability of alternative materials usable as thermoelements at higher temperatures as well as the results of annealing experiments of different ceramic protective layers deposited at alumina tubes (Al_2O_3) to prevent reduction processes of the alumina in the temperature range between 1750 °C and 1850 °C will be presented.

REPRODUCIBILITY OF THERMOELECTRIC CHARACTERISTICS FOR THE HIGH-TEMPERATURE THERMOCOUPLES 'W-Re 5/20'

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Features of development in Russia and application of thermocouples with elements made of tungsten-rhenium alloys with the rhenium contents 5 and 20% are briefly characterized in the paper. According to the national verification scheme (GOST 8.558-93) standard thermoelectric thermometers based on W-Re thermocouples had been developed to transfer temperature unit in the range 900…2500 °C to industrial thermoelectric thermometers. Expanded uncertainty of the calibration (k=2) was in the limit from 5 to 25 °C. To certificate W-Re wires the Ural's Institute of Metrology (Ekaterinburg, Russia) had developed and introduced into metrological practice standard samples of thermoelectric materials (wires) of W-Re5 and W-Re20 alloys. They were calibrated with expanded uncertainty about 11 °C at 2500 °C by a "wire bridge" method.

Now control of reprodusibility of the thermoelectric characteristics of the 'W-Re 5/20' thermocouples (type A) is induced by the fact a new edition of the standard IEC 60584-1, 2 preparing, and also by the fact of appearance of a new manufacturer of W-Re wires in Russia. The calibration program and comparison of calibrations results were performed by Russian metrologists (in ROSTEST-MOSKVA; VNIIM, OTC, SIA"LUCH"), and experts of foreign research laboratories (PTB, Berlin, Germany; NIST, Gaithersburg, USA; NIMJ, Tsukuba, Japan; NRC, Ottawa, Canada). All participants tested samples of thermocouples made of the same paired wire coils of Φ0,5 and Φ 0,35 mm.

Results received by the Russian researchers are generalized in the paper. At the first stage calibration of W-Re 5/20 thermocouples was realized in the range of 0…1800 °C. Wire-by-wire comparison with standard samples of W-Re wires and comparison of W-Re thermocouples with standard type B thermocouple or standard pyrometer have been performed. In the all range calibration data were, mainly, within 0,5% limit of deviations from the reference table (GOSTR 8.585-2001). The second stage of researches assumes realization of W-Re thermocouple calibration near the upper limit of measuring temperatures.
The first results received within the present research concerned to reproducibility of reference table for W-Re 5/20 (type A) thermocouple were submitted at the meetings of working group 5, TC65 IEC, held on May, 2008 in Tokyo and on May, 2009 in St-Petersburg.

INRIM-NMC COMPARISON OF PT/PD CALIBRATION ABOVE THE AG POINT

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A calibration of thermocouples above the temperature of the silver point (962 °C) directly traceable to the ITS-90 requires a radiation thermometer as reference. To fully exploit the potential of Pt/Pd thermocouples up to 1500 °C, specially designed blackbody cavities and multizone furnaces able to assure good axial temperature distributions have to be used as transfer temperature sources. For this purpose, INRIM and NMC developed facilities for the comparison of thermocouple and radiation thermometer based on three-zone furnaces and self-made silicon carbide blackbody cavities.

With the aim of validating the respective measuring systems, four Pt/Pd thermocouples were used. Two of them were commercially available thermocouples and the other two were fabricated at INRIM. They were investigated and calibrated at both institutes, at temperatures from 1000 °C to 1300 °C for the commercial ones and from 1000 °C to 1500 °C for the INRIM ones. Besides, INRIM's thermocouples were calibrated at the fixed points of tin, zinc, aluminum and silver while the commercial ones were calibrated at silver point at both institutes. As such, this exercise also compared the fixed point realizations for contact thermometry at the two institutes, within the capability of the Pt/Pd thermocouples. The inhomogeneity of the Pt/Pd thermocouples were tested before and after calibration against the radiation thermometer at both INRIM and NMC. At INRIM, a radiation thermometer carrying a multi-fixed point calibration was used to define T_{90} of the transfer source cavity by an extrapolation approach. At NMC, a local radiation thermometer calibrated according to ITS-90 definition was used instead. Consequently, the exercise was a useful comparison of different approaches to disseminate T_{90} above silver point.

CONCERNING CALIBRATION OF THE GOLD VERSUS PLATINUM THERMOCOUPLE

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The gold versus platinum (Au/Pt) thermocouple was first suggested as a reference thermometer in the late 1980s. A decade later several designs had been developed and two reference functions described; one by Gotoh et al. (1991) and one by Burns et al. (1992). Although the thermocouples were assembled in multiple batches, it is believed that each set of thermocouples, and consequently each reference function is based on single lots of material. The difference in EMF as a function of temperature between the two reference functions is significant relative to the stated uncertainties of the reference functions. The reference function developed by Burns et al. has been adopted as the de-facto standard for Au/Pt thermocouples.

The traditional approach for calibrating noble metal thermocouples is to determine EMF deviations from a reference function at the fixed point temperatures and fit a quadratic calibration function to the difference values. The quadratic calibration function is then used in conjunction with the reference function to describe the EMF-temperature relationship of the thermocouple. This approach is recommended in the literature and has been shown to be effective with first-generation Au/Pt thermocouples. However, structure is present in the resulting residuals, suggesting a quadratic function may be insufficient. Additionally, thermocouples produced from currently available lots of material do not appear to fit the reference function as well as the thermocouples produced from the original material. In some cases, the residuals resulting from the quadratic solution cause the chi-squared statistic to exceed expectations.

The differences between the two reference functions and the inconsistent results observed when using a quadratic calibration function suggest that Au/Pt thermocouples have more variability than previously believed. The variability negatively impacts the calibration uncertainties and challenges suitability of the Au/Pt thermocouple as a reference thermometer. This paper describes Au/Pt thermocouple calibrations over the course of many years and among several lots of material. The differences among the instruments and examples of excessive fitting residuals are shown. Finally, an alternative solution that may be generalized among lots of material is proposed.

A FURNACE FOR EVALUATING THE INHOMOGENEITY OF THERMOCOUPLES

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The emf of an inhomogeneous thermocouple depends on its longitudinal temperature profile, since it is generated in the thermocouple wires under a temperature gradient region due to the Seebeck efffect. Therefore, it is necessary to know the effect of temperature distribution on emf for each individual thermocouple.

If there is an inhomogeneity in a thermocouple wire, the emf varies with the change of the immersion depth in a furnace, although the temperature of the measurement junction and that of the cold junction are held at respective constant values. To measure such small emf change caused by inhomogeneity of wires, the temperature along the furnace should be maintained constant precisely during the measurement. To evaluate the inhomogeneity of the whole length of the thermocouple, a long immersion depth with sophisticated temperatureuniformity is required. Furthermore, the furnace should have a steep temperature gradient region to detect the inhomogeneous region of the wire.

In this study, aiming to measure the inhomogeneity of type R and Pt/Pd thermocouples, we designed and constructed a vertical furnace and evaluated its performance in the range from 200 °C to 400 °C. One of the features of this new furnace is that the immersion depth of the furnace is approximately 1000 mm, so as to measure long thermocouples. The furnace consists of six-zoned heaters with isolated temperature control systems to realize high temperature uniformity. A cooling block, which was chilled by flowing water, was attached at the top of the furnace and it made the temperature gradient steeper. The temperature stability at the full immersion position of the furnace was within +/- 5 mK for at least 10 hours in the range from 200 °C to 400 °C. The temperature uniformity within +/- 10 mK for 900 mm at 200 °C and that within +/- 20 mK at 400 °C were obtained. In conjunction with an automated actuator for the scanning, the inhomogeneity can be measured. As preliminary measurements, the inhomogeneity of several thermocouples, which had been exposed at high temperature, was evaluated using this furnace.

Determination of the Boltzmann constant

Thursday
8:20 to 10:20
Emerald 2
Session Chairman: Joachim Fischer

CAPABILITIES FOR DIELECTRIC-CONSTANT GAS THERMOMETRY IN A SPECIAL LARGE-VOLUME LIQUID-BATH THERMOSTAT

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The new, large-volume bath thermostat for the determination of the Boltzmann constant via dielectric-constant gas thermometry (DCGT) is now operating at PTB. Considering the special challenges of the DCGT experiment, the device was manufactured, assembled and studied within the framework of a scientific cooperation between INRiM and PTB. The main feature of the thermostat is to provide an instability and inhomogeneity of the bath temperature below 1 mK in a large central working volume of 500 mm diameter and 750 mm height covering the temperature range from the triple point of mercury to the melting point of gallium. In this working volume, the DCGT measuring chamber will be hosted. The measuring chamber has been designed to contain quite different special 10 pF capacitors for the DCGT measurements, including very large ring cross capacitors, where the temperature of the capacitors has to be controlled on a level of 0.1 mK. The meet of these extreme demands a huge device having a total bath volume of about 800 l is required. In the paper, the thermal features of the thermostat and the measuring chamber including the temperature control are described in detail.

A first series of tests has been performed to investigate and optimize the several parameters, which are important for the temperature control of the large amount of liquid under undisturbed conditions. These tests have been, therefore, carried out without the DCGT measuring chamber inside the bath. In the central working volume of the thermostat, the investigation showed, in accordance with the demands, an instability and inhomogeneity of the temperature well within the limits ±0.5 mK over many days. During the DCGT experiment, the temperature field in the working volume determines the thermal environment of the measuring chamber. Therefore, in a second series of tests, the temperature gradients at the boundaries of the central working volume have been measured with a mesh of 12 resistance thermometers at appropriate positions. The change of the temperature field due to the measuring chamber has been analysed in detail. But the most important result is the fact that the necessary quality of the thermal environment could be achieved.

Within the measuring chamber, the capacitors are housed in pressure vessels allow the use of measuring gas pressures up to 7 MPa. The pressure vessels are thermally anchored to a central copper plate and surrounded by an isothermal shield. The thermal conditions in this complex system have been investigated applying both resistive sensor elements and differential thermopiles. Since changes of the gas pressure cause temperature jumps within the vessel, controlling of the temperature of the capacitor requires also to know the time constants of thermal equalization after temperature jumps. The results of dedicated investigations comparing experimental data and results of finite-elements calculations are described in the paper, too.

DETERMINATION OF THE BOLTZMANN CONSTANT BY MEANS OF PRECISION LASER SPECTROSCOPY IN THE NEAR-IR

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The spectral line shape of an atomic or molecular resonance, when recorded very precisely in a linear regime of interaction, for any absorbing medium in the gas phase at thermodynamic equilibrium, provides a variety of detailed physical information on the gas itself from the macroscopic point of view, as well as on the individual particles. In fact, from the accurate analysis of an absorption profile, it is possible to retrieve, through the Beer-Lambert's law, the strength of a given line, from which the transition dipole moment between two quantum states can be inferred. Similarly, if the linestrength is known, the molecular density can be accurately determined.

Usually eliminated in any experiment of time and frequency metrology, the Doppler broadening effect can be regarded as a gift of nature for the purpose of measuring the thermodynamic temperature of a gaseous sample. In a proof-of-principle experiment, performed on CO_2 molecules in the wavelength window around 2 mm, Doppler broadening thermometry has been implemented and successfully used for the spectroscopic determination of the Boltzmann constant, with a relative accuracy of 1.6 10^{-4}.

Here, we report on the results of a second-generation experiment, carried out on the water vapour spectrum at 1.38 mm. A pair of phase-locked extended-cavity diode lasers were employed in order to ensure extreme levels of accuracy in controlling and measuring any variation of the laser frequency around a given absolute reference. The master laser was frequency stabilized against a saturated absorption of a $H_2{}^{17}O$ vibration-rotation line, observed inside a high finesse optical cavity. Intensity-stabilized laser absorption spectroscopy was performed for the extremely accurate observation of the line shape of a given $H_2{}^{18}O$ line.

Laser-gas interaction takes place inside an isothermal cell, referenced to the triple point of water. This latter system is based on three chambers, one inside the other, the inner one being the 20 cm long cell containing the ^{18}O-enriched water vapour sample. It is capable of ensuring a temperature stability within 0.1 mK for an unlimited time and a temperature uniformity better than 0.4 mK.

ACOUSTIC DETERMINATION OF THE BOLTZMANN CONSTANT WITH QUASI-SPHERICAL RESONATORS

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We report current progress in the INRiM experiment for the determination of the Boltzmann constant by means of acoustic thermometry, which is based on the simultaneous measurement, along an isotherm at 273.16 K, of the acoustic and microwave resonance frequencies of a cavity filled with a monoatomic gas. The results of a microwave determination of the resonator volume are compared for three different cavities, which differ significantly in shape, dimensions and comprising material. As to acoustic measurements, the modeling of several perturbing effects, which include the presence of holes and ducts, the coupling of gas and shell motion and geometrical imperfections, was improved and checked against the experimental results obtained in He and Ar with a steel resonator having an inner radius of 8 cm. The procedures, methods and results obtained in the calibration of several capsule-type SPRTs used in the experiment are illustrated and discussed along with the performance of our temperature measurement and control in terms of stability and uniformity across the apparatus. The methods used to enhance and maintain the high purity of monoatomic gases, which is needed during the experiment, are described. Preliminary results obtained in the determination of the molar mass of He and Ar samples are presented.

ASSESSMENT OF UNCERTAINTY IN THE DETERMINATION OF THE BOLTZMANN CONSTANT BY AN ACOUSTIC TECHNIQUE

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NPL is currently carrying out acoustic resonator experiments with the aim of determining the Boltzmann constant with an uncertainty below 1 part in 10^6. In this paper we present our latest results and critically assess the likely uncertainty of measurement.

We pay special attention to three areas. Firstly we discuss the design of the resonator and the manufacturing techniques used to ensure that accurate measurements of the resonator's form and dimensions are possible. Secondly we discuss the comparability of resonator dimensions determined by three techniques: dimensional measurements with a coordinate measuring machine; pyknometry using water; and a microwave technique. Finally we discuss uncertainties in the determination of the impurity and isotopic composition of measurement gas used.

THE EUROPEAN EXPERIMENTS ON THE DOPPLER BROADENING METHOD FOR THE BOLTZMANN CONSTANT DETERMINATION

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A EURAMET Joint Research Project (T1.J1.4) on the determination of the Boltzmann constant was started in 2007 in preparation for the proposed redefinition of the kelvin. The project involves three different measurement methods based on acoustic gas thermometry, dielectric constant thermometry and Doppler broadening thermometry. Each method is associated with a specific work package. This paper presents the recent advances in the work package dealing with Doppler- broadening thermometry, which involves research groups from LNE, LPL, UniNA2, PoliMI, INRiM and DFM.

Due to the thermal motion of the molecules constituting a gas in thermal equilibrium, the spectrum of laser absorption by a resonant molecular line is Doppler broadened. The method presented here determines the Boltzmann constant from accurate measurement of the Doppler width of a molecular absorption line of a given gaseous sample, probed by means of precision laser absorption spectroscopy. Following the Maxwell Boltzmann velocity distribution, the measured Doppler width is proportional to the quantity $\sqrt{k_B T}$, where k_B is the Boltzmann constant and T is the thermodynamic temperature of the gas. In order to eliminate any deviation between thermodynamic temperature and ITS90, the Boltzmann constant is determined at the temperature of triple point of water (273,16 K) on which the current definition of the kelvin is based.

This paper reviews the advances of three European experiments based on Doppler broadening thermometry, which has the advantage of being insensitive to the isotopic mixtures of the gas involved. The thermostats developed to maintain the gas in thermal equilibrium within 0.3 mK over a few hours are presented and discussed. A unified approach for the evaluation of the uncertainty of the measured Boltzmann constant is also presented.

The progress status of the experiments and the near future perspectives are reported. A comparison of the three different experiments and systems is presented too, in terms of capabilities, advantages and disadvantages.

ACOUSTIC DETERMIATION OF THE BOLTZMANN CONSTANT: PROGRESS WITH A DIAMOD TURNED QUASI SPHERICAL RESONATOR IN HELIUM

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There is a strong interest in the international community in metrology for new accurate determinations of the Boltzmann constant k, which could lead to a new definition of the unit of the thermodynamic temperature, the kelvin. Indeed k is related to the quantum of energy $k \cdot T$, where T is the thermodynamic temperature.

The value of the Boltzmann constant k can be derived from the measurement of the propagation velocity of the sound in a noble gas.

In the method described here, the experiment is performed in a closed cavity. To get an accurate determination of k, most parameters of the experiment (gas purity, static pressure, temperature of the device, exact shape of the cavity …) have to be carefully controlled. Since correction terms have to be applied to the acoustic signals, the relevance of the theoretical models from which they are derived is also a key issue.

The new determination carried out at the LNE-INM/CNAM is based on the same principles of the acoustic experiment performed by Moldover et al. at NIST in 1988, which led to the most accurate determination of the Boltzmann constant up to now.

However, several fundamental modifications and improvements have been achieved in this new experiment to measure and control the parameters affecting the value of k.

The results shown here are carried out with a diamond turned copper resonator filled with helium gas. The measurements are performed with two sets of acoustic transducers of different size, and the respective budgets of the uncertainties are presented.

Other applied measurements

Thursday
8:20 to 10:20
Adria
Session Chairman: Robert Benyon

HEAT AND MASS TRANSFER MEASUREMENTS FOR TRAY FERMENTED BIOLOGICAL AGENTS OF FUNGUS

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The cultivation of microorganisms on solid substrates in the absence or near absence of free water in the medium is called solid-state fermentation (SSF). Metabolic heat arising from respiration causes a temperature increase in the culture medium. Lack of free water and low conductivity of solid particles can lead to heat gradients in SSF. Therefore, the quantitative description of the influence of temperature and moisture on microbial growth is essential for modeling and optimization of solid-state fermentation. Meanwhile, high performance of SSF industrial and pilot-scale processes can only be attained by the application of effective control strategies over the environmental conditions within the bioreactor (temperature, water activity, nutrient concentration, illumination, pH value, and oxygen availability). Also, direct monitoring of fermentor conditions in large scale solid-state fermentation (SSF) bioreactors is difficult due to lack of reliable and affordable instrumentation. In this study, in order to accurately monitoring and control the growth conditions of biological agents of fungus such as Trichoderma and Beauveria bassiana inside a large fermentor chamber and to direct measurements of the two most important environmental variables of temperature and water content, and other fermentation parameters such as biomass content, air speed, illumination, pH values, substrate temperatures, etc, relevant meters are adopted, and the IR thermal imager is used to visualize the surface temperatures of solid substrates to investigate the fermentation uniformity of products. The tray system is chosen as the fermentation system because it is widely applied in SSF for production of enzymes or koji, for example. The width of the tray is assumed to remain infinite, allowing a one-dimensional mass and heat transfer approach in vertical direction. Substrate, water and air are assumed to be homogeneously divided in the tray. Heat and mass transfer resistance in the air above the tray are assumed to be absent. As a consequence, growth models of tray fermented biological agents of fungus can be derived from the interpolated data of relevance measurements. The models predict temperature, biomass-glucosamine, oxygen, water, and dry-matter concentration in the tray. Model calibration and simulation details are provided, while some results showing model performance are presented and discussed. Finally, the main conclusions and possible extensions and applications of this study are addressed.

Keywords: solid-state fermentation (SSF), tray fermentor, fungus, kinetic model, IR thermograph.

TEMPERATURE, HUMIDITY AND PRESSURE MEASUREMENTS TRACEABILITY FOR A METEOROLOGICAL WEATHER STATION

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In the context of the traceability for meteorological measurements, since 2007 a project is being developed at the Italian Institute of Metrology (INRiM). A weather measurements station is installed and used to measure and save temperature, air humidity and pressure values. Several are the scopes of this project. At first it aims at promoting the traceability of the weather measurements, which at present is not provided by any Italian Meteorological Service. It will be an example of application of metrological procedures to such kind of measurements. The station is in fact calibrated following a dedicated method, using sensors and instrument having a well known traceability to the national standards. As direct consequence, the weather station measurement results will then have an evaluated uncertainty, that also keeps into account the variance and co-variance matrixes due to the mutual influence of the parameters to the different sensors. Another goal is that to make recorded data archives available and to provide an online service, opened to public access, that makes instant data available together with daily, weekly and monthly evolutions.

The measurement conditions for the weather stations have been improved in terms of positioning and data collection. After an experimental period devoted to the evaluation of the data collection procedures, the dedicated software development and the calibration procedures definition, the system is now operative; a remote transmitter and a solar panel have been added in order to improve the siting uncertainty conditions.

The project finally aims at the proposal of a series of such calibrated stations, controlled in cooperation between the meteorological services and the national metrology institutes in Europe. Only if measurements values are associated to their uncertainties the evolution of weather parameter values can be compared year by year thus becoming a useful contribution for climate studies.

A CALORIMETRIC SCAN TECHNIQUE FOR SPECIFIC HEAT CAPACITY MEASUREMENTS ON SAMPLES IN THE 200 G RANGE

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A calorimetric technique is presented applying a temperature scan to a material inside a calorimeter of own design. The temperature rise can be varied depending on the size and type of material by adjusting the power to electrical heating elements in the calorimeter. The form and size of the samples is flexible and in the range of 10 to 500 g, determining the specific heat cp in the range of 5 to 60 °C (at the moment) in a representative way. This is in contrast to other methods, which request very small sample sizes of few mg's and isotropic behaviour. The calorimeter works usually with pure water, which well known specific heat capacity as function of temperature, is used as reference. The calorimeter is built of a light insulating material to keep energy losses low. The cp of any material can be measured as long as there is no chemical reaction with the working medium. Otherwise protective coating is necessary. Evaluation of cp is performed at different temperatures by comparing the electrical energy dissipated into the calorimeter with the absorbed thermal energy, during a chosen time interval. For this, the temperature rise in the sample and in the working medium and their respective masses must be known. The unavoidable heat losses are determined by a suitably chosen blind scan. Comparisons have been performed with other techniques like DSC and TPS (Transient Plane Source). The estimated measurement uncertainty is in the range ±3 to 5%.

PRACTICAL ACOUSTIC THERMOMETERY WITH ACOUSTIC WAVEGUIDES

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Acoustic Thermometry is capable of phenomenal accuracy, but it is a difficult technique to apply in many practical situations. Here we describe a modification of the technique which permits robust temperature measurements to be made, potentially with milli-kelvin resolution, over a temperature range extending from cryogenic temperatures to over 1000 °C.

The technique uses measurements of the time-of-flight of acoustic pulses in tubes usually filled with an inert gas such as argon. The tubes – typically made of stainless steel with an outer diameter of 6 mm – act as acoustic waveguides and can be several metres long and bent into complex shapes. The time-of-flight reflects the average temperature along the entire length of the tube. Local temperature information can be inferred in several ways. Typically a second shorter tube is used and the difference in time-of-flight reflects the temperature in the region at the end of the first tube. If the measurement length is sufficiently long – typically 1 metre of tube – then a measurement resolution of less than 1 mK is achievable.

The technique is well suited to measurements in harsh environments in which conventional sensors degrade. We show results from early tests which highlight the strengths and weaknesses of the technique.

HUMIDITY, PRESSURE AND TEMPERATURE MEASUREMENTS IN AN INTERDIGITATED-FLOW PEM HYDROGEN FUEL CELL

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Hydrogen fuel cells have great potential as portable sources of power that is pollution-free at the point of use, and can be low-carbon if the fuel (hydrogen) is generated by renewable methods. However, the reliability and durability of fuel cells need to be improved significantly if this technology is to be widely adopted. Testing under controlled conditions is a necessary step towards viability and uptake of fuel cells. Detailed measurement data are also needed as inputs to models of fuel cell operation. Yet, in spite of widespread interest, suitable measurement techniques are still only in the process of being developed.

There is strong research focus on low temperature PEM fuel cells (PEM stands for both “proton exchange membrane”, and “polymer electrolyte membrane”). Measurements of reactant gas humidity, temperature, flow, pressure and composition are central to the study of PEM cells, along with electrical measurements. Water is a product of the hydrogen fuel cell reaction. The presence of water within the fuel cell membrane electrode assembly usefully increases its proton conductivity. However, if conditions are too wet then transport of the fuel gases to the electrode surface is impeded. For these reasons, it is highly important to control and measure humidity in PEM fuel cell gases. Temperature measurements are also essential: not only does temperature influence cell operation, but localised overheating is an indicator of uneven current generation and a potential cause of membrane failure. All these effects can be studied if localised values of temperature and humidity can be correlated with localised current density within the cell. In support of this, localised pressure and flow measurements are also valuable.

In this work, in-situ measurements of humidity, temperature and pressure are demonstrated for a PEM fuel cell of interdigitated gas flow channel layout. Sensors are embedded at the flow field edges, sampling air and hydrogen in the flow channels near the edges of the active area. The measurements provide, for the first time, real-time localised temperature, humidity and pressure data for this configuration of fuel cell.

Fixed points - M-C eutectics III

Thursday
11:10 to 13:10
Emerald 1
Session Chairman: Jörg Hollandt

A DETERMINATION STUDY OF THE CAVITY EMISSIVITY OF THE EUTECTIC FIXED POINTS CO-C, PT-C AND RE-C.

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The eutectics Co-C, Pt-C and Re-C, with eutectic temperatures of 1597 K, 2011 K, and 2747K, 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. Temperatures are measured by means of radiation thermometry of cavity radiators imbedded in the associated eutectic. This paper deals with the determination of the respective spectral effective cavity emissivities, which are influenced by the reflective properties of the graphite constituting the cavity on the one hand, and by the temperature distribution within the cavity and over the radiation-shield structure in front of the cavity, on the other.

We have begun a comprehensive effort to determine the cavity emissivities. The overall program, as summarized in the steps 1 to 7 below, involves diverse measurements on representative graphite samples and calculations of the cavity-emissivity dependencies. The steps are: (1) Spectral hemispherical reflectance of graphite samples under neutral gas purge at temperatures above ambient (up to a maximum of about 1200 K). This would provide the nearnormal directional emittance. (2) Full room-temperature bi-directional reflectance distribution function (BRDF) at 405 nm and 650 nm. These measurements can be validated with hemispherical reflectance at the same wavelengths obtained in step 1. At the same time, the BRDF results can validate the restricted BRDF measurement set in step 3. (3) Temperature dependent BRDF samples at a few fixed geometries up to 3000 K. Assuming that the relative BRDF angular distribution does not change with temperature, these measurements will allow scaling the reflectance (from step 2), and hence the near-normal emissivity, up to 3000 K. (4) Angle-dependent relative spectral radiance at 405 nm and 650 nm under vacuum at temperatures up to 3000 K in the same setup as step 3 to obtain the angle dependent emittance. (5) Furnacetemperature profiles of the radiation-shield structure in front of the cavity measured at the eutectic temperatures of the eutectics in study. (6) Calculations of the temperature distributions within the cavity and along its outer environment, using a software package utilizing finite volume analysis, with input from step 5. (7) Monte Carlo ray-trace modeling of the effective spectral cavity emissivity with input of the results of all steps 1 to 6. We report here on the current status of the study, including cavity temperature distributions and Monte Carlo modeling results (steps 5, 6 and 7). The modeling assumes current estimates of the graphite emissivity and BRDF, which will be updated as data become available from steps 1 to 4.

T_{90} MEASUREMENT OF THE HIGH TEMPERATURE FIXED POINTS AT NIM

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The high temperature fixed-point (HTFP) blackbodies, Co-C, Pt-C, and Re-C point, have been gradually established at the National Institute of Metrology of China (NIM) since 2005, and their characteristics are studied.

Recently, the International Temperature Scale of 1990 (ITS-90) above silver freezing point has been realized at NIM by improved high temperature primary standard pyrometer, which is of smaller size-of-source effect, programmable, and partial temperature controls. The pyrometer is characterized by new facility for calibrating spectral responsivity, and its non-linearity of spectral responsivity is measured extending to around 2500 °C. The temperatures of the ITS-90, T_{90}, of these HTFPs have been measured by the primary standard pyrometer with 660 nm interference filter and validated by other standard pyrometers with 660 nm and 900 nm interference filters.

This paper describes the characteristics of the primary standard pyrometer, the HTFP blackbody cells and the CHINO high temperature furnace which is employed to realize the HTFPs, and the experimental procedures of realization and measurement. The measurement results and corrections are presented and the associated uncertainties are evaluated.

The calibrations of standard pyrometers by silver or copper point and these HTFPs are being studied for improving the uncertainty of calibration, especially at high temperature.

Keywords: temperature, measurement, fixed-point, blackbody, pyrometer, uncertainty.

INTERNATIONAL STUDY OF THE LONG-TERM STABILITY OF METAL-CARBON EUTECTIC CELLS

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For high temperature fixed points to be accepted as temperature references it is important that their long-term stability be objectively demonstrated. This evaluation is part of the CCT-WG5 high temperature research project [1] devoted to the comprehensive evaluation of three hightemperature fixed points Co-C (1324 °C), Pt-C (1738 °C) and Re-C (2474 °C). The assessment of the long-term stability as well as the robustness of the cells is examined in the first workpackage.

Four cells for each of the eutectic points have been constructed by NMIJ (4 Co-C, 2 Pt-C and 4 Re-C) and NPL (2 Pt-C) and the stability tests have been performed by NMIJ (Co-C), NIM (Pt-C) and VNIIOFI (Re-C). These tests consisted of the aging of one cell, among the set of four, for a period of 50 hours around the melting temperature. For each of the three eutectic points, before and after aging, the tested cell was compared to one of the three cells so that the drift, due to the aging if any, can be evaluated. All three cells which underwent the aging process showed no breakage and demonstrated extremely repeatable melting plateaus.

In this paper, after a short description of the protocol and the cells (described more completely elsewhere [2]), the results of the aging tests obtained for the three fixed points are presented and 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

[2] Sadli M., Yamada Y., Wang T., Yoon H.W., Bloembergen P., Machin G. “Stability and robustness tests on Co-C, Pt-C and Re-C cells: the first results” Tempbeijing, 20th-23rd October 2008.

NEW FILLING TECHNIQUE AND PLATEAU OBSERVATIONS OF CR_3C_2-C PERITECTIC FIXED POINT

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Since the emergence of high-temperature fixed points (HTFPs) based on metal-carbon (M-C) and metal carbide-carbon (MC-C) eutectics, many national metrology institutes have been developing high quality eutectic cells to be used for the calibration of radiation thermometer and high-temperature thermocouples. In addition to M(C)-C eutectics, MC-C peritectic fixed points have also showed promising performance to serve as high-temperature reference points in thermometry. Among the MC-C peritectic fixed points Cr_3C_2-C (1826 °C) can take the place of Pt-C (1738 °C) or Ru-C (1953 °C) eutectic fixed points; the chromium has an advantage over precious metals in that it is much cheaper. The construction of Cr_3C_2-C peritectic cell, however, is difficult due to the good wetting property and the high viscosity of molten chromium, resulting in large voids inside of the ingot. In this paper a new filling technique for Cr_3C_2-C peritectic cells and the results of plateau observations and microstructure analysis are presented.

In a previous study [1], some of the authors had devised a new filling method to use rolled up graphite cloth material ("C/C sheet" TCC-019, manufactured by Toyo Tanso Co. Ltd) as a wick to absorb molten metal to overcome the formation of voids. However, large gaps were observed in the ingots. During the filling process, short lengths of rolled up C/C sheet layers were added in stages on top of the previously formed ingot section. The new C/C sheet layers suck up the metal from the previous filling stage, resulting in large gaps. We report here a method that overcame this problem by using a single roll of C/C sheet packed to fill the full length of the crucible. A tube was formed of graphite paper that extended out of the crucible and was used as a hopper to hold enough chromium and carbon mixture ideally to fill the crucible in a single stage. After the filling, plateau observations were carried out by use of Nagano three-zone furnace. X-ray transmission photos were taken for some cells to check the filling. The results of the filling, plateau observations, and microstructure analysis are presented in the paper.

[1] Y. Yamada, Y. Wang, W. Zheng, and N. Sasajima, "A Study of the Metal Carbide-Carbon Peritectic Phase Transition for the Cr-C System", Int. J. Thermophys. 28 (2007) 2028-2040.

Mise en pratique for the definition of the kelvin

Thursday
11:10 to 13:10
Emerald 2
Session Chairman: Graham Machin

THE ROLES OF THE MISE EN PRATIQUE FOR THE DEFINITION OF THE KELVIN

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Motivated by the need for a formal document to give definitive guidance for the practical realization of the kelvin in accordance with the International System of Units (SI), the Consultative Committee for Thermometry (CCT) created the *mise en pratique* for the definition of the kelvin (MeP-K) in 2006. Subsequently, the CCT considered constructing a new International Temperature Scale (ITS) to replace the present ITS-90 and its low-temperature counterpart, the Provisional Low Temperature Scale (PLTS-2000). However, the CCT decided not to do this because a new temperature scale imposes large burdens on industry, which has a large installed base of hardware and process algorithms specific to the ITS-90. Instead, the adoption of the MeP-K opens a new and flexible path for updating and expanding the thermometric methods in common use without changing the basis of the ITS-90. Additionally, the CCT foresaw that adoption of the proposed new definition of the kelvin, based on a fixed value for the Boltzmann constant, would require an MeP-K. Future versions of the MeP-K, updating the 2006 version, will cover the following:

1. An Introduction will outline the definition of the kelvin and its extension to primary realizations and the ITS.
2. The MeP-K will describe direct, primary methods for measuring thermodynamic temperature T. In particular, absolute radiometric methods above the silver point have lower uncertainties than ITS-90 methods.
3. The ITS-90 and PLTS-2000 provide formal approximations to T. The MeP-K currently includes the text of the scales and a Technical Annex of essential additional information. Future versions will include recommended differences between thermodynamic temperature and temperature on the ITS-90, $T - T_{90}$, along with the associated uncertainty. Documentation of known ITS-90 biases supports thermodynamically accurate measurements without mandating replacement of the ITS-90 in industry.
4. In a new category of SI realizations, the MeP-K will describe realization of the kelvin by indirect methods, giving the temperatures for selected fixed points (not defined on the ITS-90) and outlining interpolation or extrapolation methods. Realizations using high-temperature eutectic fixed points and radiometric interpolation are one example of an indirect method promising greater flexibility in traceable temperature measurements.

PRIMARY RADIOMETRY FOR THE MISE-EN-PRATIQUE FOR THE DEFINITION OF THE KELVIN: THE HYBRID METHOD

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A task group of CCT-WG5 (radiation thermometry) was established in May 2008 to write text for the mise-en-pratique for the definition of the kelvin (MeP-K) for high temperatures. This task group reviewed and gave summaries for the existing techniques for filter radiometry as a means of determining absolute radiance, and hence thermodynamic temperature of a blackbody source. Three approaches were described – the radiance method, which calibrates the radiation thermometer for radiance responsivity, the irradiance method, which calibrates a filter radiometer for irradiance responsivity and then measures the source through two apertures, and the hybrid method that introduces a lens to the irradiance method.

In the 'hybrid method' the radiation thermometer consists of a filter radiometer, a double aperture system and a lens. The lens allows the instrument to view a small area blackbody source. The system is calibrated 'in parts' – that is, the filter radiometer is calibrated for irradiance responsivity and the transmittance of the lens and the geometric factor are determined separately.

The main drawbacks of this single lens instrument is its high size-of-source effect (~0.2 %), and that this effect has to be determined in an 'absolute' sense – relative to a theoretical infinite source. However, although the correction is large, with careful evaluation the associated uncertainty can be made sufficiently small to measure the temperature of fixed-point cell transitions with low uncertainties.

This paper reviews the hybrid method and gives a comprehensive discussion of the associated uncertainty components.

UNCERTAINTIES IN THE REALISATION OF THERMODYNAMIC TEMPERATURE ABOVE THE SILVER POINT

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In May 2008, a task group of the CCT-WG5 (radiation thermometry) was established to examine the possibilities of realising and disseminating thermodynamic temperature above the silver point by means of both primary radiometry and radiation thermometry with the aid of high temperature fixed points with known thermodynamic temperatures. These realisation schemes are denoted n = 0, 1, 2, 3, 3+, where n refers to the number of fixed points used in the realisation. The WG5 task group has prepared text describing these schemes for *the mise-en-pratique for the definition of the kelvin* (*MeP*-K) for the temperture range above the silver point.

A major advantage of this approach is that it provides a great deal of flexibility and can accommodate the varying resources and needs of a wide range of laboratories and/or users. This paper outlines the uncertainty analysis for each of the $n \geq 0$ schemes, which provides a useful tool for assessing the suitability of any particular scheme and for selection of the appropriate fixed points. In all cases, the analysis is based on a modified Sakuma–Hattori model of the underlying measurement. This allows simple analytical expressions to be derived for all sensitivity coefficients, identification of the dominant uncertainty components, and each method to be easily compared. Current estimates for each uncertainty component are given, and these are propagated to give a total uncertainty for each scheme. It is expected that over time, total the uncertainties will be reduced, particularly as the quality and reliability of the high-temperature fixed points improve.

PRIMARY RADIOMETRY FOR THE MISE-EN-PRATIQUE: THE LASER-BASED RADIANCE METHOD APPLIED TO A PYROMETER

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The CCT working group 5 (radiation thermometry) is on the way of proposing a new *mise en pratique* of the kelvin at high temperature based on thermodynamic temperature mediated, or not, by high-temperature fixed points.

LNE–INM/Cnam is involved in the research on high temperature metal-carbon eutectic (or pertitectic) fixed points and on the thermodynamic temperature measurement of these fixed points. The method used for thermodynamic temperature measurements is a «radiance method» based on the determination of the absolute spectral responsivity of a low-SSE, high-stability and linearity radiation thermometer. The absolute spectral responsivity of this pyrometer is measured with a specific Ti-Sa tunable-laser based set-up with an integrating sphere whose absolute radiance is traceable to a cryogenic radiometer *via* a silicon trap detector.

As a validation of this method, a complementary experiment using LNE-INM's monochromator based radiance comparator instead of the radiation thermometer is presented. The radiance comparator is based on a Czerny Turner monochromator which does not require a laser wavelength scanning over a broad range (typically 40 nm) for the spectral responsivity to be determined while this is necessary for an interference filter with the known risks of interreflexions.

This paper presents the experimental method. It describes the principle of the specific pyrometer filter used to facilitate its calibration, the laser based set-up and the sphere radiance lockin technique involving an innovative two stages feedback loop which includes an acousto optic modulator. This method uses a unique combination of the absolute radiance method and a specific interference filter used to overcome stray interferences appearing in its spectral sensitivity when using a tunable laser. The uncertainty budget is discussed.

ABSOLUTE RADIOMETRY FOR THE MeP-K: THE IRRADIANCE MEASUREMENT METHOD

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The "Mise en pratique for the definition of the kelvin" (MeP-K) as a guide for the realization of the kelvin is presently revised to incorporate the temperature scales in current use as the best formal approximations to thermodynamic temperature and, additionally, direct methods of thermodynamic temperature measurement. Within the MeP-K the CCT WG 5 "Radiation Thermometry" develops a document to facilitate the implementation of thermodynamic temperature measurements at temperatures above the silver fixed-point (961,78 °C). Three thermodynamic temperature measurement methods, all relying on absolute spectral-band radiometry, are proposed. To make these techniques more accessible for the broader thermometry community each of this method is described in detail in a dedicated paper. These descriptions shall not be understood as a restriction to other possible radiometric methods of thermodynamic temperature measurement.

One of the three presently applied methods of radiometric temperature measurement above the silver point is the absolute measurement of irradiance in a well defined spectral-band. This is a straight forward method, which relies on filter radiometers with known spectral irradianceresponsivity and a well defined observation geometry, not needing any imaging technique. It has been developed for thermodynamic temperature measurements of precision blackbodies as primary standards for radiometry and thermometry and for the investigation of the uncertainty of the approximation of ITS-90 to thermodynamic temperature over a period of nearly 20 years at PTB. The paper describes the design features of the applied filter radiometers, their calibration chain and their long term stability. Additionally, the details and requirements of the experimental procedure for obtaining thermodynamic temperatures with this method are presented. Finally, outstanding results obtained with this method, which is also applicable for temperatures significantly below the silver fix-point, are reviewed.

THERMODYNAMIC RADIATION THERMOMETRY USING RADIOMETERS CALIBRATED FOR RADIANCE RESPONSIVITY

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A primary objective in the redefinition of the *Système International d'Unités* (SI) is to have, as the basis of the SI units, constants which are invariant in space and time. In the temperature community, the movement to adopt a fixed value of the Boltzmann constant as the basis of the unit of temperature is well underway. However, with the redefinition of the kelvin, guidance from the Consultative Committee on Thermometry (CCT) on the practical realization or the *mise en practique* for the kelvin (*MeP-K*) is needed. The new *MeP-K* will include descriptions and references to all primary thermometry techniques including, among others, constant-volume gas thermometry, acoustic thermometry, noise thermometry, dielectric-constant gas thermometry, and thermodynamic radiation thermometry.

Using radiometry, thermodynamic temperatures can be determined by a variety of experimental techniques. Radiometers without imaging optics can be calibrated for spectral power or spectral irradiance responsivity, and radiometers with imaging optics can be calibrated for radiance responsivity. These separate approaches can have different uncertainty components with different uncertainty values. At NIST, thermodynamic radiation thermometry is performed using radiation thermometers calibrated for radiance responsivity using laser-irradiated integrating sphere sources (ISS). The radiance of the ISS is determined using Si-trap detectors whose spectral power responsivity is traceable to the electrical-substitution cryogenic radiometer. The radiometric basis of the NIST approach will be discussed. The uncertainty budget for the measurements as well as the characterizations to determine the component uncertainty values will be discussed.

Hygrometers and Moisture Sensors

Thursday
11:10 to 13:10
Adria
Session Chairman: Norbert Böse

A NEW THIN-FILM TRACE MOISTURE SENSOR USING NANOPOROUS SOL-GEL LAYERS

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A new thin-film trace moisture sensor consisting of a ceramic substrate and three thin-film layers, namely base electrode, sensing layer and cover electrode, is presented. The sensor is combining the advantages of well known capacitive polymer humidity sensors and common metal-oxide trace moisture sensors. This is achieved by replacing the polymer of a conventional capacitive humidity sensor by a nanoporous sol-gel layer. The used sol-gel technology provides a high flexibility in thin-film designing and a rapid, flexible and economic manufacturing process. With respect to the aimed fields of application, the new trace moisture sensor has been optimized by consequent variations of the thin-film parameters. Furthermore, the device was tested in relevant industrial applications. The measurement results are summarized and the specific advantages of the device are discussed with regard to common trace moisture sensors. For the device a typical uncertainty of measured frostpoint better than ±1,5 K has been determined experimentally. It is shown that the sensor resists to condensation often occurring in industrial applications and therefore provides excellent long-term stability over years. Additionally a short response time is achieved. Regarding the outstanding characteristics the new sensor is qualified for different industrial applications like drying processes, clean gas production and air service units for pressurized air. The applied sol-gel technology enables a variety of sensor designs to fulfill different customer needs in the near future.

EXPANDED RANGE MOISTURE ANALYSIS USING CONTINUOUS-WAVE CAVITY RING-DOWN SPECTROSCOPY

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Continuous-Wave Cavity Ring-Down Spectroscopy (CW-CRDS) is a laser-based state-of-the-art detection technique that does not need calibration. Based on first principles, it directly derives the absolute optical loss due to molecular absorption inside the cavity from a simple time-based measurement, independent of laser intensity noise and environmental conditions. CW-CRDS has already become a field-proven trace gas measurement technique widely used in both standards labs and industry. With particular emphasis on expanded range moisture analysis in different matrix gases, this paper demonstrates the strong capability and versatility of CW-CRDS beyond applications requiring the highest sensitivity. For example, in addition to H2O performance data on sub-ppb to low ppb levels, detailed H2O step intrusion or pulse intrusion data of up to several hundred PPM in various carrier gases will be presented. This self-verifying measurement method can cover any measurement range, from parts-per-trillion up to percent levels, with excellent linearity and repeatability. Thus, it provides an ideal analytical solution that is fast, sensitive, precise, and yet extremely robust and easy to operate.

MICROWAVE DETERMINATION OF WATER MOLE FRACTION IN HUMID GAS MIXTURES

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A small volume (65 cm^3) gold plated quasi-spherical resonator has been used to measure the water vapor mole fraction x_w of standardized N_2/H_2O and CO_2-free air/H_2O mixtures prepared with a INRiM standard humidity generator and a commercial two pressure generator. For nominal frost points between 242 K and 270 K, the relative difference Δx_w between the water mole fractions in the resonator and in the samples prepared by the standard generator was less than 5%. The microwave technique exploits the high precision achievable in the determination of the cavity microwave resonance frequencies. It is particularly sensitive to the presence of small concentrations of water vapor in reason of the high polarizability of this substance. The determination of the concentration of the mixture components requires the knowledge of their density and dielectric virial coefficients as a function of temperature; for common substances these quantities are accurately known from experiment; in one case, that of He, the same quantities are amenable to extremely accurate calculation from theory, making it a reference substance for the calibration of the resonator dimensions as a function of pressure, and for detection of systematic errors. Thanks to its simple and rugged design the microwave hygrometer is suitable for accurate measurements at temperatures up to 500 K and pressures up to 4 MPa, far above the current upper working range of standard humidity generators.

EVALUATION OF THE LONG-TERM STABILITY OF DEW-POINT HYGROMETERS AND RELATIVE HUMIDITY SENSORS

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The continuous quest for improved specifications of optical dew-point hygrometers and polymer relative humidity sensors has raised customer expectations on the performance of these devices. Users with a limited prior experience in the measurement of humidity, in the absence of a long calibration history, often rely solely on manufacturers specifications in assigning the components of uncertainty due to the long-term stability of the instruments, as would be reasonable for example in the case of measurement of electrical quantities. This leads in many cases to rather optimistic estimations of uncertainty. This is particularly problematic in he assessment of new calibration laboratories seeking accreditation according to ISO/IEC 17025, where laboratories have significant difficulties in justifying the contributions due to long-term stability in the actual conditions of use, in many cases leading inevitably to surprises in the first surveillance visit.

During 15 years of calibration activity, within the accredited scope of the Laboratory, first under UKAS accreditation and later under ENAC Accreditation (see http://www.enac.es for the current schedule), the INTA Temperature and Humidity Laboratory has accumulated a vast wealth of data on its own and customer instruments in diverse levels of demanding applications, from which the long-term performance can be evaluated.

One of the largest customers of the Laboratory are the Measurement and Testing laboratories of other departments of the Institute. The present work presents a study of the drift of a representative sample of hygrometers and analyses their performance over the lifespan of the instruments through regular calibration using the INTA humidity calibration facilities.

The results of the investigations provide typical realistic figures for the long-term stability for theabove instruments in laboratory and selected industrial applications that can serve as a referencewhen establishing initial contributions in uncertainty budgets.

A TRULY DISTRIBUTED OPTICAL FIBRE HUMIDITY SENSOR

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Optical fibre sensors have many advantages over conventional sensors such as freedom from electromagnetic interference, intrinsic safety and their inherent distributed nature. There are many applications where monitoring humidity at several points is desirable. This paper presents a novel truly distributed optical fibre distributed humidity sensing system based on Fresnel reflection using Optical Time Domain Reflectometry (OTDR).

The cladding of an optical fibre is replaced by a hygroscopic material of a refractive index higher than that of the core over the full humidity operating range. The moisture content of the cladding varies with humidity in the surrounding environment resulting in a change in the refractive index of the cladding. As light experiences a change in refractive index in its path Fresnel reflection occurs. Using an OTDR the value of the Fresnel reflection is measured along the optical fibre. The level of Fresnel reflection represents the variation in refractive index hence the humidity. A self referencing technique has been implemented within the cladding to provide accurate self calibration for the sensor without the need for any additional software or hardware.

The basic operation of the sensors is detailed in the paper, a fully working sensor is demonstrated and the experimental results are presented. A numerical model for the sensor has also been developed.

POSTER SESSION III

Moisture profile and transport

Thursday
10:30 to 11:10
and
14:15 to 15:00
Foyer

A METHOD FOR DETERMINATION OF WATER CONTENT IN LIQUIDS BY A LONGITUDINAL SLOT WAVEGUIDE

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We propose a simple method for the determination of water content in liquids (e.g. in alcohol) that is based on the on-line measurement of a single parameter – attenuation of a wave traveling through a rectangular waveguide with a longitudinal slot. The use of electromagnetic waves of the millimeter-wave band allows one to make the device compact and to realize non-destructive monitoring in real time without chemical reagents.

A waveguide with a longitudinal slot is well known as a leaky-wave antenna (see, for example, [1]). The operation of the device is based on the dependence of the complex propagation constant of the operating mode in the waveguide on the complex dielectric permittivity of the external medium, i.e., the medium on the outer side of the slot (outside the waveguide). In the narrow-slot approximation $l \ll a$, where l is the slot width and a is the width of the narrow wall of the waveguide, the attenuation per unit length of a wave propagating in such a waveguide as a function of the parameters of the medium outside the waveguide can be calculated for alcohol solutions in water (for alcohol content from 0 to 50% vol.) using the formula given in [2]. These calculations show that such a waveguide allows for the measurement of the concentration of water in alcohol (and per contra) in the range of frequencies from 20 to 40 GHz with accuracy of about 1% per unit length of slot. Experiment is carried out on a measurement cell designed on a rectangular waveguide section at frequencies from 29 to 37 GHz with a solution of ethyl alcohol in water as the external medium. The measurement accuracy is 0.3% of alcohol for the 3-cm long cell.

This method can be used for the on-line monitoring of concentration of alcohol in water as well as for monitoring other water solutions.

[1] C. H. Walter, *Traveling Wave Antennas,* New York: Dover, 1965.

[2] E. I. Nefedov and A. N. Sivov, *Electrodynamics of Periodic Structures,* Moscow: Nauka, 1977.

RETRIEVAL OF ATMOSPHERIC MOISTURE PROFILES USING FY-3 MICROWAVE HUMIDITY SOUNDER

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Back-propagation neural networks (NN) are applied to retrieve humidity profiles from FENGYUN-3 (FY-3) microwave humidity sounder (MHS-A). FY-3 microwave sounder consists of a five channel microwave radiometer. Its channels are centered on 150 GHz, 150 GHz and 183.31±1 GHz, 183.31±3 GHz, 183.31±7 GHz, known as channels 1 to 5, respectively. The first two of these channels fall in an atmospheric window (affected mainly by the water vapor continuum), the three high frequency channels are centered on a water vapor absorption line. The instrument's antenna scans in cross-track type, sampling 98 earth-views within of nadir, each separated by (16 km on surface at nadir and 41 km * 27 km for the outer pixel), referred to pixels 1 to 98. It also views an internal calibration target and space, which are used as radiometric calibration points. The period of one full scan is 2.667 s. Combined with radiosonde and other microwave sensors, these channels will allow regional sounding and global sounding of atmospheric humidity. This paper mainly using regional sounding datasets to retrieve regional humidity profiles. In the certain radisonde datasets, the paper extracts clear-air and over-ocean datasets randomly. It derives atmospheric absorption spectrum from accurate MPM93 model (liebe'93 model) and simulates brightness temperature using radiometric transfer equation. The retrievals yield good results in the humidity profiles from the surface to 400 hpa approximately. When compared to a linear regression approach, the neural network retrieval yields significantly better results for the retrieval atmospheric levels.

Keywords: FY-3; MHS; back-propagation neural network; humidity profiles; MPM93.

INTERFACIAL PROPERTIES OF LENNARD-JONES FLUIDS ON THE FUNDAMENTAL OF VAN DER WAALS GRADIENT THEORY

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Interfacial tension and the relevant properties is a basic thermophysical properties which plays an important role in many industrial process. The liquid-vapor surface tension influences and controls many processes in chemical or reservoir engineering applications, especially the heat transfer through heat-exchanger surfaces with bubbles or fluid drops. In recent years, the van der Waals gradient theory and its modification has been widely used to predict the interfacial properties. The only inputs to the gradient theory are the Helmholtz free energy density of the homogeneous fluid and the influence parameter of the inhomogeneous fluid. The Helmholtz free energy density of homogeneous system f_0 (ρ ,T) at a fixed temperature in the whole range between the densities of liquid and vapor existing in equilibrium was assumed to be an analytic function of the local density ρ which can be defined by many thermodynamic models, for example, cubic EOSs. The influence parameter then is an expression for the coefficient of proportionality that precedes the square gradient in terms of density or temperature. In this work, the interfacial properties of Lennard-Jones (LJ) fluids were studied using the van der Waals gradient theory. The Orstein-Zernike (OZ) equation using the Choudhury-Ghosh (CG) closure model was solved to determine the molecular distribution function over large density and temperature ranges. The vapor-liquid equilibrium of the LJ fluids was studied using the pressure equation and the compressibility equation. The gradient theory model was then used to calculate the surface tension of the LJ fluids from the distribution function results.

THE EFFECT OF PRESSURE ON MOISTURE DIFFUSION IN POLYMER MATRIX COMPOSITES

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Temperature and humidity play a significant role in the mechanical behavior and long-term durability of polymer matrix composites. While the effect of temperature on diffusion has been studied extensively, the effect of pressure has received less attention. Recent results have shown that diffusion in polymer composites can be accelerated by increasing the ambient pressure. This study seeks to more fully interrogate the effect of pressure on diffusion. Test chambers were built to maintain a constant relative humidity of 80% at 60 °C at three different pressures (1 atm., and 5 atm., and 10 atm.). The absorbed moisture content increased with increasing chamber pressure indicating pressure assisted diffusion. In comparison to the 1 atm., environment, the through thickness diffusivity increased by 36% at 5 atm., and 69% at 10 atm., whereas the time to saturation decreased by 27% at 5 atm., and 41% at 10 atm.

MOISTURE CONDITIONING SYSTEM FOR GRAINS

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The moisture conditioning systems are very important in the calibration process of grain moisture meters, because they provide grain samples with different moisture content levels. These systems also are used for conditioning of other solid materials such as wood, construction materials, foods, etcetera, that can be used as reference materials.

In general, the moisture conditioning methods can be classified as indirect and direct. Indirect methods relate the equilibrium relative humidity with the moisture content of the material (sorption isotherms) by mean of empirical or semi empirical relationship. In other words, the sample to be conditioned is placed in a relative humidity chamber, e.g. salts solution chamber and the moisture content is related to the value of relative humidity; in the direct methods the moisture content is obtained by the mass of water added in the sample.

A direct method for grain moisture conditioning was implemented. In this system a flow of air pass first through a water reservoir and the resulting saturated air pass through the sample. The flow is kept until all water is evaporated and absorbed by the sample. The moisture content value of the conditioned grain is obtained by weighting the sample.

This system includes a relative humidity hygrometer which measures continuously the relative humidity inside of the test chamber. When the relative humidity is stable the process is concluded. With the equilibrium relative humidity value, the temperature and a sorption model is possible to calculate the moisture content value in the grain.

In this work the description, operation and results of the conditioning system are presented.

A PRACTICAL APPROACH TO THE TRACEABILITY OF MOISTURE CONTENT IN WOOD

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The measurement of the moisture content in wood has important industrial issues related to the assessment of the quality of raw and processed materials. Wood exchanges moisture with air, where the amount and direction of the exchange depend on the relative humidity and temperature of the air and the current amount of water in the wood. This relationship has an important influence on wood performance, on its physical and mechanical properties (density, thermal expansion, tensile strength, etc.) and on its evaluation for engineering applications and trading purposes.
A widely used electrical wood moisture content (MC) measurement technique is the DC resistance/conductance method. An electrical conductivity moisture meters is essentially a MΩ-meters whose pin electrodes are driven into the specimen at given depths. The method is invasive because of the electrode design. The meter is generally supplied with an empirical calibration chart by the manufacturer to allow for the conversion from the primary measured quantity (conductance) to the moisture content for different wood species. Of course, traceability exists for the primary quantity, but the lack of a reliable traceability chain for MC can be a significant source of uncertainty in the measurement of wood properties.
At INRIM, a practical approach has been developed within an industrial collaborative project to cope with the above traceability issues. The method is based on the measurement of the sorption isotherms at ambient temperature (23 °C) of several wood specimens. The sorption isotherm function characterizes the storage of moisture within a material.

Wood specimens were placed in small ventilated chambers - whose relative humidity (RH) was controlled by means of saturated salt solutions - until equilibrium with ambient RH was established. The equilibrium relative humidity (ERH) in the chambers was measured by means of a calibrated RH probe traceable to INRIM humidity standards. The equilibrium moisture content (EMC) of small wood samples cut from each specimen was determined by thermo gravimetric analysis. The EMC was then correlated with the equilibrium relative humidity (ERH) for each specimen to get the sorption isotherm.

The paper discusses such investigations and gives examples of conductivity-based moisture meters calibration. A comparison of the results with the manufacturer calibration charts is also made and a suitable calibration procedure developed.

MODELING OF WATER VAPOR DIFFUSION IN ELASTOMERS WITH IMPACT IN HUMIDITY AND VACUUM MEASUREMENTS

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Elastomer gaskets are frequently used in humidity measurement systems and high vacuum technology for demountable flange seals and vacuum valves. It is well known that atmospheric gases and particularly water vapor from humid air permeate through elastomer gaskets and thus influence the amount of water vapor being measured in a closed humidity measuring system, especially in case of very dry samples. The permeation also limits the lowest attainable pressure level in vacuum systems. Most common gasket material is fluoroelastomer known under trade name Viton. A limited data on water permeability of Viton can be found in vacuum literature. The results are also not consistent and important parameters such as relative humidity and temperature are often missing. Therefore we have performed a set of experiments to better clarify water permeation of Viton material.

Sample gasket has been degassed in high vacuum for sufficiently long period to remove more than 99% of dissolved water vapor. After that it was exposed to the ambient atmosphere with controlled temperature and relative humidity. The water vapor uptake curves were measured gravimetrically with precise balance. The physical process of water absorption can be described by the diffusion equation, which has been modeled numerically with finite difference method (FDM). Goal of the modeling was to determine diffusion constant and solubility of water vapor in the material from the best fit with experimental data.

Standardized methods for water vapor transmission rate (WVTR) like ISO15106, ISO21129 and ISO12572, are based on the measurement of steady state transport of diffusing water vapor through a sheet of sample material. Diffusion constant and thickness of the material are two parameters which determine the time delay to achieve a steady condition after the change of water vapor pressure at high pressure or low pressure side of the sample plate. To reduce measurement errors in WVTR experiments it is necessary to start measurements only after the steady state is reached.

Results of the work allow determination of the lowest attainable vacuum levels that can be measured using such elastomers and consequently the minimal moisture level that can be measured within specified uncertainty.

A FACILITY FOR MEASURING MOISTURE CONTENT OF MATERIALS

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The UK National Physical Laboratory (NPL) has established a new facility for measurements of moisture content of materials. This facility comprises a suite of instruments using a range of physical measurement principles based on loss of mass on drying, evolved vapour analysis, and microwave resonance.

Loss of mass on drying is considered a reference measurement of moisture content, although it does not distinguish water from other volatile contents. NPL carries out loss-on-drying measurements by oven drying together with weighing on a precision laboratory balance. A bench-top thermogravimetric analyser is also available, which integrates radiant heating with continuous weighing of the sample.

Evolved vapour analysis is related to classic loss-on-drying techniques. If a water-specific sensor is used to measure the total evolved vapour, then the measurement specifically identifies the water content of a sample. By selecting a suitable temperature profile for heating, it is possible to distinguish free water, capillary water, and chemically bound water in a sample under test. Tests using this facility on industrial powders sent to NPL have helped make drying processes more efficient, providing the user with optimum drying temperatures and durations to reduce water contents to specified levels.

Microwave resonance frequency analysis allows rapid, non-destructive analysis of bulk moisture content, provided it can be calibrated against a suitable reference method. It is particularly suited to moderate sized samples of granular materials (up to approximately 50 cm^3 of sample in the NPL laboratory). NPL is experimenting with using a microwave analyser in versatile ways, such as studying changes in water content of samples of polymer material during degradation.

An intercomparison of these moisture measurement methods has been carried out, as well as measurements of certified reference materials. Estimated uncertainties for the measurement methods are presented.

Future work planned for the facility is also described:

- To further develop this facility to support traceable calibration of instrumentation,
- To identify needs for reference materials within industry in order to develop simple, multi purpose, widely applicable certified reference materials,
- To study the relationship between water activity and moisture content using the microwave resonance technique in combination with water activity instrumentation.

POSTER SESSION III

Other applied measurements

Thursday
10:30 to 11:10
and
14:15 to 15:00
Foyer

A NEW DEVICE FOR SPECTRAL EMISSIVITY DETERMINATION USING A FT-IR SPECTROMETER

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To investigate the radiative properties of new ceramic coatings and heat-resistant materials, a new device for determination of infrared spectral emissivity has been developed in the past two years.The device employs two blackbody sources and two sample furnaces and covers low temperature 100 °C to 1000 °C and high temperature 1000 °C to 2400 °C, respectively. This device covers the spectral range of 1 μm to 20 μm. The degree of vacuum is less than 1 Pa by using thermocouple vacuum gauge. The operation principle is the comparison sample spectrum with variable temperature reference sources spectrum at the same temperature by means of FT-IR spectrometer. The temperature of blackbody source and sample surface at high temperature is measured using a radiation thermometer and a integral thermometer.

For the direct measurement of material spectral emissivity, one must compare the spectral radiance of a sample to that of a blackbody at the same temperature. This requires several system and measurement components : (1) Blackbody sources at the variable temperature, and a means of determining temperature; (2) Sample for holding, heating and controlling temperature; (3) A means of accurate temperature measurement; (4) A spectrometer such as FT-IR which employs the TGS sensor; (5) An interface optics for viewing blackbody and sample and switching between them; (6) A chamber that consists of sample furnace, blackbody and interface optics can be evacuated or filled with protect gases; (7) A cooling system for thermostatic background and aperture; (8) Labview software for heating control, operating display and data processing.

After construction of the the spectral emissivity measure systems, we have begun to make system performance tests including preliminary sample emissivity measurements. Several tests were performed to check characteristics of the spectrometer. We have performed the stability experiment by heating a period of time and obtained the spectral response function of FTIR by calibration functions and blackbody spectrum. The spectral peak response is near 16 μm.We calculated values of blackbody emissivity from modeling studies. The effective emissivity was obtained from the ratio of the calculated data and the measured data. We have estimated that the nonlinearity of response. Observed non-linearity error at the blackbody radiance of 2000 °C was smaller than 1% around 5 μm bands. We have performed the experiments to compare the difference between the sample source optics and blackbody reference optics. The results show that the difference is less than 1%. In order to study the radiative properties of new ceramic coatings, we performed emissivity determination experiments for several TiO_2 coatings at 800 °C. The results show that the emissivity of several TiO_2 coatings is higher than 0.85 at the 3 μm wavelength. The measurement results for the TiO_2 coatings has been analyzed for the details.

TRANSPORT AND OPTICS MEASUREMENTS OF CYCLO OLEFIN COPOLYMER SUBSTRATES DEPOSITED SILICON DIOXIDE FILMS

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Organic light-emitting devices (OLED) are extremely sensitive to moisture and oxygen. Recently, OLEDs have been fabricated on plastic substrates to form flexible organic light-emitting diodes (FOLEDs). Now, a new amorphous engineering thermoplastic, nominated cyclic olefin copolymer (COC) is used for plastic substrate applications, because of its higher transparence, lower birefringence, lower dispersion and lower water absorption. However, COC plastic substrates can't sustain plasma-based processing temperatures at 350 °C. In this study, silicon dioxide films are deposited on the COC substrate to act as a thermal barrier layer. To evidence transport characteristics of the composite SiO_2/COC structure, experiments of the moisture permeation rate testing and the thermal resistance experiments for transport measurements are conducted to explore the moisture diffusion barrier and thermal barrier characteristics of COC substrate deposited a layer of SiO_2 thin film on it. Silicon dioxide layers of thickness, 0.25 μm, 0.5 μm, and 1 μm, respectively, are fabricated by the PECVD process. Surface morphologies are explored by the three-dimensional atomic force microscopy to show its surface roughness. Sessile drop test for contact angle measurement are conducted for surface tension investigation of the SiO_2/COC structure. For the permeation rate measurement, the Ca-test method is adopted. For the thermal resistance measurements, two methods of the thermocouple in vacuum environment and the IR thermal imager are adopted and measured results are compared. Different surface temperatures, 323.15 K, 373.15 K, 408.15 K, and 473.15 K, respectively, are applied upon the silicon dioxide film and temperature differences for varied thickness of silicon dioxide film are measured. Experimental results are presented to investigate the behaviors of moisture diffusion barrier and thermal barrier characteristics of the COC/SiO_2 structure. Furthermore, the optical characteristics of transmissivity and refractive index of the SiO_2/COC structure is investigated by a spectrophotometer. From the optical experiments, thickness of silicon dioxide films is significant to optical properties of transmissivity, refraction, and reflection, respectively, and the experimental results show that effects of SiO_2 film thickness are contrary to the transport problems.

Keywords: substrate, cyclo olefin copolymer (COC), permeation, thermal resistance, optics.

THERMOMETERS IN LOW MAGNETIC FIELDS

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Most thermometers for laboratory and industrial use are subject to ac and dc extraneous magnetic fields (from electromagnetic field sources like fans, heaters, Earth magnetic field, medical devices like MRI scanners, etc). Thermometers themselves are at least partly composed of ferromagnetic materials, thus making their readings susceptible to surrounding magnetic fields. The effect has been researched in the cryogenic temperature range below 80 K. In this paper we are discussing the correlation of temperature indication for various types of temperature sensors due to extraneous dc magnetic flux densities in temperature range up to 200 °C.

Magnetic fields were generated by a water-cooled electromagnet capable of generating dc magnetic flux density of 2 T with relative homogeneity of $5*10^{-4}$ over an area of 1 cm^2 in the pole face centre, regulated by means of a precision power supply. Different temperature sensors (thermistors, thermocouples and platinum resistance thermometers) were maintained at a constant temperature and their temperature readings acquired. Changes in temperature indication at different magnetic flux densities were observed. Constant temperature was achieved by means of a liquid nitrogen container, ice-point bath, Peltier element cooled metal sheets and silicon oil closed-loop regulation system for the temperature range from cryogenic temperatures up to 200 °C.

The results indicate observable changes in temperature readings due to dc magnetic field for platinum resistance thermometers and thermistors, which corresponds to [1]. Differences are noticeable also for types of thermocouples, which are composed of ferromagnetic materials (Fe, Cr, Ni).

[1] Working Group 2 of the Comité Consultatif de Thermométrie. TECHNIQUES FOR APPROXIMATING THE INTERNATIONAL TEMPERATURE SCALE OF 1990, pp. 160-168, 1997, reprinting of the 1990 first edition.

Keywords: magnetic flux density, magnetic effect, magnet, thermistors, thermocouples, resistance thermometers.

CONVECTIVE EFFECTS OF A LARGE CAPACITY AUTOMATIC MASS COMPARATOR

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High accuracy application of a large capacity (64 kg) fully automated mass comparator with the four-place weight handler is strongly affected by convection effects. The comparator is used for calibration of weights by multiplication in the range from 2 kg to 50 kg and for oneto-one comparisons starting from 1 kg. Mass range from 1 kg up to 5 kg is mostly disturbed.

For quantification of convection effects comparisons of all possible independent mass differences with simultaneous temperature measurements under the individual draft shields covering each of the handler's positions were carried out. For the temperature measurement four reference sensors and 12 additional miniature temperature sensors with the two highprecision readout devices were used. Due to small relaxation time, small contact gradients and absence of internal heating thermocouple sensors were preferred. Calibration uncertainty of sensors is 10 mK.

Systematic deviations of mass differences obtained by using comparator and associated temperature gradients under the draft shields were determined. Maximum error for the difference of two 1 kg cylindrical loads with exactly the same shape was 0,3 mg, although temperature differences revealed were smaller than 0,1 K.

CALIBRATION OF SENSORS FROM –150 °C TO 60 °C, FOR USE IN THE MEASUREMENT OF AIR AND SURFACE TEMPERATURE OF MARS

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Within the misión of the MSL Mars Scientific Laboratory (MSL), the Astrobiology Centre (CAB), a joint INTA-CSIC facility, has developed the Rover Environmental Monitoring Station (REMS) for the measurement of meteorological parameters of Mars, in which are integrated amongst others, several Platinum Resistance Thermometers for the measurement o fair and thermopile temperatures operating in the (8 to 14) µm, (14.5 to 15.5) µm and (16 to 20) µm bands, that are intended for the remote measurement of the surface temperature of the planet.

The need for calibration of these particular sensors at temperatures down to –150 ºC, required the development of specific measurement techniques, as well as the optimization and validation of a precision low-temperature comparator. Unlike the ideal behaviour of Standard Platinum Resistance Thermometers (SPRT) calibrated at the ITS-90 fixed points and have defined interpolating functions, the non-standardized thermometers require a different approach and calibration at many more points over the range, in order to determine their response and sensitivity coefficients.

This work describes, in the context of the application of the Project of the REMS meteorological station, the optimization and characterization of the calibration system, with the detailed uniformity and stability Studies using Quartz-sheathed SPRTs (using the Triple Points of Argon and Mercury) and its optimization for the calibration of sensors of different characteristics.

The calibration results for thermocouples and resistance thermometers with nominal resistance of 1 kOhm at 0 ºC, are presented and compared with the overlapping ranges with the conventional calibration baths.

The procedure and calibration results of the sensors, both individually and integrated in the thermopiles, in the range from –150 °C to 60 °C and the preliminary results of the comparison with the radiometric measurements performed in the Astrobiology Centre.

MODELLING OF TEMPERATURE CONDITIONS BETWEEN TEMPERATURE ARTEFACT AND BLACK TEST CORNER

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The objective of the paper is elaboration of elements related to metrological analysis in the field of testing, with the respect to conformity assessment. Temperature measurement in testing is one of the most defined field of measurement, but still it is very important what is being measured and how the measurement is performed. Standards usually only describe what should be measured, in some cases also how accurate the measurement should be but not what kind of measuring instrument should be used. Therefore it sometimes happens that measurements are performed with improper equipment or in an improper way. It is important that measurement results and their associated uncertainties are correctly evaluated and on that basis right conclusions of conformity or nonconformity with specifications are made. Therefore the knowledge and awareness regarding all facts related to the used measuring equipment in testing is essential.

The case study in this paper will be temperature conditions modelling between a temperature artefact and a black test corner measuring instrument. The paper will focus on the measurement of heating of household appliances according to the standard (SIST) EN 60335-1, clause 11 using black test corner. The black test corner is an instrument which consists of two wooden walls and a floor, with build-in thermocouples fixed on the back side of cooper disks flashed with the surface. It is used to measure how household appliances temperature is influencing surrounding in real environment, e.g. in kitchen, leaving room, etc. Temperature heating that is measured with the black test corner is defined as temperature difference between temperature of the instrument, increased by the normal usage of an appliance and a room temperature.

For the same level of appliances conformance testing, laboratories shall use the same testing procedures and comparable measuring instruments. During the proficiency testing, in scope European project INCOLAB, all participating laboratories used black test corners made in accordance with standard. However, their results were significantly different. Therefore this paper will deal with analysis of influencing parameters when measuring heatings using the black test corner and modelling of temperature conditions between a temperature artefact and a black test corner. The temperature artefact is a specially developed heating plate which is very stable and can be set to different temperatures. Using commercial FEM COMSOL modelling software we will model changes of different parameters (thickness of the wooden plates, distance between the temperature artefact and the black test corner, etc), which influence the measurement result in the black test corner. As a result of analysis and modelling, important parameters for an optimal measuring instrument and thus an optimal experiment will be suggested.

INFLUENCE OF RESISTANT METHOD ON MOTOR WINDINGS TEMPERATURE RISE MEASUREMENT

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The objective of the paper is presentation of influencing parameters when measuring motor windings temperature rise in scope of safety testing of electrical appliances, with the respect to conformity assessment. Temperature measurement in testing is one of the most defined field of measurement, but it is very important how the measurement is performed. Standards only describe that the resistant method shall be used for determination of temperature rise (heating) of windings. It is not explicitly defined how to approach when using cooling characteristics of windings for determination of heating. Since the extrapolation curve is used, the procedure is also very important in order to obtain a result as accurate as possible. Therefore it sometimes happens that measurements and evaluation of results are performed with improper equipment or in an improper way. It is important that measurement results and their associated uncertainties are correctly evaluated and on that basis right conclusions of conformity or nonconformity with specifications are made. Therefore the knowledge and awareness regarding all facts related to the used procedure and measuring equipment in testing is essential.

Resistant method influence on motor windings temperature rise measurement will be a case study in this paper. The paper will focus on the measurement of heating of electrical motors used in electrical appliances according to the standard (SIST) EN 60335-1, clause 11. In the paper will be analysed what are the influencing parameters when measuring heating of electromotor windings. As a case study concrete measurements will be performed and analysed. Measurement uncertainties of the results will be presented in respect to target measurement uncertainty.

Upon this basis a general approach on evaluation of cooling characteristic and consecutively determination of windings heating will be proposed. It is intended to find out what is optimal time after performer has to start measuring the cooling characteristics, since the motors are typically mounted deep into appliances, specially for collector motor windings it takes some time for performer, to be able to connect measuring instrument to collector's lamellas.

DEVELOPMENT OF A TEMPERATURE AND HUMIDITY WIRELESS NODE FOR WIRELESS SENSOR NETWORK CALIBRATION

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Wireless sensor networks (WSNs) are constantly expanding their application field, from simple two-state measurements (e.g., on/off, proximity detection, etc.) to distributed many-parameter measurements. Commercial WSNs offer a wide range of functions and performance with the best sensors achieving accuracy comparable with desktop instrumentation.

The advantage of using such sensors for continuous in-situ monitoring is offset by the need of partially dismantling the network at the time of the sensor calibration. As a result, new reference standards suitable for in-situ calibration of such network sensors are needed in order to reduce the calibration cost, the inherent inefficiency and the logistic problems of a laboratory calibration, still exploiting the capabilities of a WSN.

The paper describes the development of a so-called wireless sensor reference node (WSRN) for temperature and relative humidity (RH) measurements based on a high-accuracy capacitive sensing module developed at INRIM. The basic module was developed as a general purpose wireless platform to be used for accurate measurements of several quantities by means of a capacitive-based sensing mechanism (e.g., acceleration, pressure, position, flow). After a suitable front-end re-design, a temperature and humidity sensing node resulted.

The WSRN performance was investigated in the temperature range from –10 °C to 50 °C and in RH range from 10% to about 85% for its potential use as a transfer standard for in-situ calibrations. It was firstly characterised in the test chamber of a humidity generator and, subsequently, it was tested in the field against a chilled mirror dew-point hygrometer and a portable RH hygrometer probe. Finally, its operation was assessed during in-situ calibration of commercial WSN.

The results of this work show that the present gap between expensive laboratory calibrations and simple in-situ operational testing can be filled by providing the traceability to WSN and, at the same time, by insuring the full network operation during a calibration process. Furthermore, the introduction of such WSRN devices prompt to the development of specific calibration methods for WSNs.

POSTER SESSION III

Thermoelectric Thermometry

Thursday
10:30 to 11:10
and
14:15 to 15:00
Foyer

COMPARATIVE STUDY OF PT/PD AND PT-RH/PT THERMOCOUPLES

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The Pt/Pd thermocouple has demonstrated superior thermoelectric drift and homogeneity performance over conventional Pt-Rh/Pt thermocouples. Here we present a systematic comparison of the drift and homogeneity performance of Pt/Pd and Type R thermocouples by ageing the thermocouples at 1350 °C for a total of 500 hours and measuring the performance at regular intervals during this time.

The thermocouples studied were 1 Pt/Pd thermocouple, 1 Type R thermocouple, and 1 'special' Type R thermocouple which was given the same preparatory annealing treatment as the Pt/Pd of each thermocouple was measured at the freezing point of silver (961.78 °C) and the melting point of Co-C eutectic (1324.29 °C).

The long term drift of the Pt/Pd thermocouple was around 50 mK after the first 100 hours ageing at 1350 °C, followed by a further 25 mK over the subsequent 400 hours ageing. This drift performance was an order of magnitude lower than the two Type R thermocouples. The Type R thermocouple which was given the 'special' preparatory treatment was slightly more stable than the conventional Type R.

The thermoelectric homogeneity of the thermocouples was also measured at regular intervals by a dedicated movable localised heat source, which demonstrated the evolution of the thermoelectric homogeneity profile with aging.

STABILITY OF PLATINUM/PALLADIUM THERMOCOUPLES UP TO COPPER FREEZING POINT

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The dissemination of traceability to the International Temperature Scale of 1990 (ITS-90) through comparison calibration of thermocouples, is playing an important role in metrology institutes, secondary laboratories and industrial measurements.

The previous studies of the Pt/Pd thermocouples at NIS-Egypt, shows promise of achieving significant improvements in the accuracy of temperature measurements up to 960 °C.

In this paper we describe stability and changes in the thermo EMF of Pt/Pd thermocouples in the temperature range from 420 °C up to 1100 °C following a series of heat treatment at 1100 °C. The magnitudes of the changes in EMF, the short and long term stability of the thermocouple were estimated, experimental results are presented.

The Pt/Pd thermocouples had been assembled at NIS-Egypt using a reference grade wires supplied by Johnson Matthey, England.

All the measurement possesses Pt/Pd thermocouples, carried out using NIS facilities as Zn, Ag and Cu freezing points to achieve highest calibration accuracy.

For the calibration of the thermocouples we have employed a reference polynomial function given in the literature for Pt/Pd thermocouples based on the ITS-90.

A detailed analysis of the different components of uncertainty of thermocouple calibration is also described in this paper.

After good annealing of the thermocouple, the EMF drift became very small typically 0.03 °C at 1100 °C.

A THERMOCOUPLE HOMOGENEITY SCANNER BASED ON AN OPEN PRESSURE-CONTROLLED WATER HEATPIPE

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The thermoelectric inhomogeneity of thermocouples is a major source of uncertainty in the use and calibration of thermocouples. It is therefore important that the inhomogeneity is assessed as a normal part of calibration. To date such assessments have been carried out using, for example, furnaces, oil baths, and highly localised heating. These methods variously have disadvantages that may include inaccurate quantification of the cumulative temperature gradient, nonuniformity of the nominal isothermal zone, limited capacity for long thermocouples, or limited spatial resolution of variations in Seebeck coefficient. Here we demonstrate a low-cost, highperformance homogeneity scanner based on a pressure-controlled water heatpipe that is open to the atmosphere. With this arrangement, very much like a steam-point apparatus, the atmosphere provides the controlled pressure and the buffer gas for the heatpipe, and the isothermal zone is close to 100 °C. The thermocouple is inserted into the steam within the heatpipe through a plastic membrane so that the gradient is imposed over a spatial region determined primarily by heat flow within the thermocouple body, and can be as small as a few millimetres. The paper explains the construction of the scanner, and presents the results of example scans demonstrating the uniformity and resolution of the scanner.

THERMOELECTRIC SCANNING OF PT/PD THERMOCOUPLES USING A FIXED-POINT FURNACE

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Platinum verses Palladium (Pt/Pd) thermocouples are widely using as standard thermometers in calibration services or in research fields to measure high temperatures more than before accurately. Stability and homogeneity of Pt/Pd thermocouples are superior to that of Type R or S thermocouples, however inhomogeneity of Pt/Pd thermocouples should be measured to estimate measurement uncertainty. Furnaces with excellent temperature stability and uniformity are necessary for Pt/Pd thermocouples to detect small changes of electro-motive-force (EMF). A heat-pipe furnace equipped with pressure-control system is the most suitable device for thermoelectric scanning but it is not affordable price for many calibration laboratories.

The aim of this report is to verify whether inhonogeneity measurements are feasible using an existing furnace in our laboratory. We made use of a commercially available sodium heat-pipe furnace manufactured by ISOTECH. This furnace is originally designed to realize freezing point of Al or Ag, and widely used in calibration laboratories in the world. The heat-pipe has 500 mm depth and 50 mm in diameter and maximum operating temperature is 1000 °C. To improve stability, a Pt/Pd thermocouple was attached to the sodium heat-pipe and connected to a temperature controller of resolution of 0.01 °C. Vertical temperature distribution in the furnace was measured by a newly fabricated Pt/Pd thermocouple which can be moved vertically by an electric slider. We obtained stability of ±10 mK and temperature uniformity of ±50 mK in the range from 450 mm to 200 mm at 600 °C.

We will report on the results that the performance of our furnace and thermoelectric scanning data obtained from Pt/Pd thermocouples at several temperatures.

ANALYSIS OF THE CONTACT THERMAL RESISTANCE IN CASE OF SEALED CONTACT SURFACE THERMOMETERS

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The aim of this paper is to determine the specific contact thermal resistance from measurements effectuated at different reference materials, different temperatures, with sealed contact surface temperature sensors.
In case of contact surface thermometers, the measurement error can be described with the following equation, where t_w is the temperature of the reference target determined by extrapolation method, t_s is the temperature measured by the sensor.

$$\delta t_s = t_s - t_w$$

The contact thermal resistance R_K is determined from the measurement error and the inlet heat flux at the frontal surface of the sensor head q_{Si} .

$$R_k = \frac{\delta t_s}{q_{si}}$$

Taking into consideration the measurement results, the contact thermal resistance R_K depends not only on the thermal conductivity of the reference material and on the rughness height, but also on the construction of the sensor head. The contact thermal resistance R_K can be taken as the sum of the contact thermal resistance of the reference surface R_{KW} and that of the surface sensor R_{KS}.

$$R_K = R_{KW} + R_{KS}$$
$$R_{KW} = R_{KW}(t_w, \lambda, R_z, \varepsilon_W, \sigma_c, F_c)$$

R_{KW} depends on the temperature of the reference target t_w, thermal conductivity λ, roughness height R_Z, total normal emittance ε_W, compressive yield point σ_C and compressive force F_C. On the other part, determination of R_{KS}, which depends on the sensor construction, is very difficult.
Due to the fact that the specific contact thermal resistance of the reference surface R_{KW} depends mostly on the roughness height R_Z, which can't be satisfactory determined by roughness measurements, this paper will present a better method for its determination. The analysis will be performed from measurements of temperatures using one given type of contact surface sensor (Ahlborn FTA 1202), employing three different types of reference materials (stainless steel, aluminium, brass), to determine the effect of roughness height R_Z of the reference surfaces and the contact thermal resistance R_{KS} of the surface sensor.

LIFE EXPECTANCY STUDY OF SMALL DIAMETER TYPE E, K, AND N MINERAL INSULATED THERMOCOUPLES ABOVE 1000 °C IN AIR

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Very little if any current data is available for the life expectancy of very small diameter thermocouples operating at high temperatures, greater than 1000 °C. Over the past 10 years significant changes in the supply stream of the materials used to manufacture base metal thermocouples have occurred. In many industrial applications small diameter thermocouples are the only solution for high temperature measurements. This study has been undertaken to assess the performance of small diameter magnesium oxide insulated metal sheathed thermocouple sensors at or above 1000 °C. Three different American Society of Testing and Materials (ASTM) standard letter designation thermocouple types have been included in this study, Types, E, K, and N. Inconel 600 and 316 Stainless, for three different sizes, 0.5 mm, 1.0 mm, and 1.5 mm, have been tested for different thermocouple types. Each group of sensors was placed in an equalization block in air for a maximum of 500 hours or until failure. Based on the data collected thermocouples as small as 0.5 mm outside diameter are capable of reading accurately for 75 to 100 hours in air at 1100 °C. Larger diameter thermocouples show very little drift up to 500 hours at 1100 °C. The data for each test was collected at 10 second intervals for the entire duration of the test. Data for the sensors drift and subsequent failure is presented.

A MECHANISM FOR THE OXIDATION-RELATED INFLUENCE ON THE THERMOELECTRIC BEHAVIOUR OF PALLADIUM

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Oxidation of thermocouple elements can degrade the accuracy of thermocouple-based temperature measurements. As a particular example of such effects, oxidation of the Pd element of a platinum/palladium thermocouple is known to increase the thermoelectric emf by an amount equivalent to a temperature change of order 100-200 mK (Hill 2002 *Metrologia* **39** 51–8).

We propose a possible physical mechanism to explain how oxidation affects the thermoelectric output of a Pt/Pd thermocouple. The analysis hinges on the hypothesis that the oxide-induced strain within the Pd thermoelement leads to a change in the Seebeck coefficient, and therefore changes in the thermoelectric emf.

A theoretical model relating deformation of the palladium lattice to the change in the Seebeck coefficient is presented. The level of agreement between the calculation and the experimental observations suggests that oxide-induced strain in the Pd thermoelement is a likely explanation for the change in thermoelectric output of a Pt/Pd thermocouple within the temperature range where oxidation is active.

STABILITY OF TUNGSTEN-RHENIUM THERMOCOUPLES IN THE RANGE FROM 0 °C TO 1500 °C

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Tungsten-rhenium (W-Re) thermocouples are widely used in industry for measurements at high temperatures since it is available to measure above 2000 °C. However, it is well known that emf of W-Re thermocouple changes during heating at high temperatures. This is serious problem for precise temperature measurements. Especially the change in emf during calibration is serious, because it will increase the uncertainty of calibration of the thermocouple.

In this work the change in emf of type C (tungsten-5% rhenium versus tungsten-26% rhenium) thermocouples was evaluated. Thermocouple wires were 0.5 mm diameter and mounted in twin-bore beryllium oxide tubes. Tantalum tubes whose outside diameter and length were 6 mm and 700 mm, respectively, were used as thermocouple sheaths.

Two furnaces were used for W-Re thermocouple calibration, which was done by comparison with a calibrated noble metal thermocouple as reference. One furnace was used for the calibration in the range from 0 °C to 1000 °C. It consisted of iron-chrome-aluminum alloy heaters and a copper block with three wells in a quartz tube filled with argon gas to obtain uniform temperature distribution. Meanwhile, the other furnace was used for the calibration in the range from 1000 °C to 1500 °C. It consisted of C/C (carbon fiber reinforced carbon) composite heaters, graphite tubes and an alumina tube. After calibration by comparison, the W-Re thermocouples were exposed at approximate 1500 °C and their emf changes were evaluated.

A CRITICAL LOOK AT TYPE T THERMOCOUPLES IN LOW TEMPERATURE MEASUREMENT APPLICATIONS

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In the United States, Type T thermocouples are commonly used in industrial measurement applications due to their accuracy relative to other thermocouple types, low cost, and the ready availability of measurement equipment. Type T thermocouples are very effective when used in differential measurements, as there is no cold junction compensation necessary for the connections to the measurement equipment. Type T's published accuracy specifications result in its frequent use in low temperature applications. An examination of over 250 samples from a number of manufacturers has been completed for this investigation. Samples were compared to a Standard Platinum Resistance Thermometer (SPRT) at the LN2 boiling point along with four other standardized measurement points using a characterized ice point reference, low-thermal EMF scanner and an 8.5 digit multimeter, and the data compiled and analyzed. The test points were approximately –196 °C, –75 °C, 0 °C, +100 °C, and +200 °C. These data show an anomaly in the conformance to the reference functions used in the United States where the reference functions meet at zero. Additionally, in the temperature region between –100 °C to -200 °C, a positive offset of up to 5.4 °C exists between the reference function equations published in the United States in ASTM E230-06 for the nitrogen point and the measured response of the actual wire. This paper also examines the historical and technological reasons for this anomaly in the US reference function. The study concludes that Type T thermocouples typically do not conform to the ASTM E230-06 published reference function describing their performance when used to measure temperature in the range of -100 °C to -200 °C.

INFLUENCE OF DIFFERENT CALIBRATION PROCEDURES TO THE UNCERTAINTY AND MEASUREMENT RESULTS OF THERMOCOUPLES

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Calibration of thermocouples is performed by comparing the emf of a thermocouple being calibrated with the emf in the reference table of a standard thermocouple of the same type at a certain temperature. Afterwards a calibration curve is fitted to the differences from calibration of a standard thermocouple using a least square method. The comparison has to be performed over the whole temperature range of interest and at sufficient number of points for a calibration curve to be fitted properly.

The standard reference table represents the behavior of a particular type of thermocouple. Calibration of an individual thermocouple type is thus reduced to determining the difference between its behavior and that of the standard embodied in the reference table.

Usually, as a cold junction temperature reference point, an ice-point is used. Theoretically if hot and cold junction of the thermocouple are at the same temperature, for example ice-point, the generated voltage should be 0 V. However due to different reasons, in practice this is not always case.

Doing a survey among number of NMIs, we have found out that in post processing of data some laboratories force in their calibration reports 0 °C = 0 V, while other give actual measured value, which is different from 0. In this paper we are going to present what kind of influence such different procedures can have to the final result when using those thermocouples for measurements and calibration.

COMPARISON OF CO-C AND PD-C EUTECTIC POINT CELLS FOR THERMOCOUPLE CALIBRATION BETWEEN LNE AND NMIJ

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The melting temperature of cobalt-carbon eutectic (Co-C, 1324 °C) and palladium-carbon eutectic (Pd-C, 1492 °C) points are ideally distributed between the freezing point of copper (1084.62 °C) and the upper limit of use of the Pt/Pd thermocouple (1500 °C), which is considered to be the best contact sensor for accurate measurements in this temperature range.

In this work, an intercomparison of the melting temperatures of three Co-C eutectic fixed-point cells and two Pd-C cells were performed using LNE high-temperature furnace. Two Co-C cells and one Pd-C cells were designed and constructed in LNE, while one Co-C cell and one Pd-C cell were designed and constructed in NMIJ. Before performing the intercomparison, the repeatability of their melting plateaux and thermal parameters influencing the phase transition such as the effect of the surroundings temperature on the point-of-inflexion, the effect of conductive heat flow through the thermocouple stem while in melting conditions were evaluated using Pt/Pd thermocouples.

To measure the Co-C and Pd-C eutectic points in intercomparison, four Pt/Pd thermocouples were used. In spite of slightly different cell designs and different materials sources, the melting temperatures of the investigated Co-C and Pd-C eutectic fixed-point cells agreed within 10 mK and 100 mK, respectively.

POSTER SESSION III

Radiation Thermometry

Thursday
10:30 to 11:10
and
14:15 to 15:00
Foyer

DETERMINATION OF THE EFFECTIVE WAVELENGTH OF THE LINEAR PYROMETER (LP4) USING BLACKBODY FIXED POINTS

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This work will describe an experimental investigation to determine the effective wavelength and its temperature dependence for radiation thermometer "LP4" of thermal metrology laboratory at the National Institute of Standards (NIS-Egypt). In fact this work is decided to be conducted in place of having separate experiment for measuring the effective wavelength which is costly and time consuming.

The experimental work will include measuring the signal of the thermometer at Ag, Cu and Co-C blackbody fixed-points. So that the output signal can be used for two purposes, the first one is calibration of the radiation thermometer by using multi-fixed points techniques (using Sakuma equation). The second is calculation of the mean effective wavelengths and consequently calculation of the effective wavelengths.

The work will include the equipment, the construction and realization of the blackbody fixed points with their maintenance furnaces, results and discussion.

Calculation for the effective wavelength will be done at different temperatures. The associated calculated uncertainties in determination of the effective wavelength will be also reported.

For the purpose of accreditation and quality assurance of calibration results, this method for determination of the effective wavelength can be used to validate both the method " the separate experiment for determination of the effective wavelength" and to validate the calibration results at multi-fixed points using Sakuma equation by using the calculated effective wavelength in measuring the temperature.

VACUUM VARIABLE TEMPERATURE BLACKBODY (VMTBB)

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This paper describes the vacuum variable medium temperature blackbody (VMTBB) constructed to serve as a highly stable reference source with an aperture diameter of 20 mm in the temperature range from 150 °C up to 430 °C under medium-vacuum conditions (10^{-3} Pa) and a medium-background environment (liquid-nitrogen-cooled shroud). The VMTBB was realized for the calibration facility at the PTB in the field of reduced background radiation thermometry under vacuum. This facility is intended for performing radiometric and radiation thermometric measurements under vacuum conditions and spectral emissivity measurements in the range from 0 °C to 430 °C without atmospheric interferences. It is difficult to realize a precision black body with high emissvity for temperatures above 400 °C. Cavities of such blackbodies are made as a rule of copper and coated by a paint with high emissivity. But any paint put on copper, does not maintain several cycles of heating to temperature up to 450 °C. As a result of investigations in the PTB a special procedure of coating the surface of the cavity by paint with high emissivity has been developed. The cavity surface is coated by chemical nickel plating before covering it by a paint with high emissivity. The general concept and the design of the VMTBB are given. For realization of a good temperature uniformity along the complete radiating cavity a three modular design of the heat exchanger and a two stages temperature control of a cavity, based on two precision PID controllers, has been used. The temperature of the cavity is supervised by 15 precision Pt resistance thermometers. 6 of them are used for the VMTBB cavity and heat exchanger temperature control and the others for the cavity temperature measurement and correction. A description of the temperature control and measurement system of the VMTBB is presented. Optical ray tracing with a Monte Carlo method (STEEP 3) indicated that the effective emissivity of this blackbody cavity is not worse than 0.9997. Tests of the VMTBB were carried out on the PTB facility and the radiation of the VMTBB is measured by comparison with the vacuum variable low temperature blackbody (VLTBB) in the temperature range from 150 °C to 170 °C with the Vacuum Infrared Standard Radiation Thermometer (VIRST). The temperature uniformity of the blackbody from the bottom to the front of the cavity is better than ±50 mK in the whole temperature range. The stability of temperature of the blackbody is within 50 mK in the whole temperature range. A preliminary uncertainty budget of the blackbody is given.

ABSOLUTE RADIOMETER

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The standard facility for the heat-flux sensor calibration is developed in VNIIOFI. The structure of the standard facility includes a high-temperature blackbody as a source of radiation in a wide range of levels of a heat-flux and a high-precision absolute radiometer with electrical substitution as a radiation detector, which is devoted to the description of this article. The design and operational principle of the absolute radiometer are presented. An irradiance range of the absolute radiometer is from 20 W/m^2 to 20 kW/m^2. The construction specifics and operation principle of the radiometer permit to get a highly equivalent substitution of electric power for optical power. The absolute radiometer is completely automated. The results of the effective absorptions of the absolute radiometer cavity are given. A preliminary uncertainty budget of the irradiance measurement is presented.

Keywords: heat-flux; absolute radiometer with electrical substitution; high-temperature blackbody; irradiance measurement; irradiance range.

PROCEDURE FOR AUTOMATED EVALUATION OF A SURFACE CALIBRATOR WITH A RADIATION THERMOMETER

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The article will present the procedure for evaluation of temperature stability and homogeneity of a surface calibrator for non-contact thermometer, so called "surface blackbodies". For calibration purpose it is important to determine the temperature stability and homogeneity of a blackbody. The automated positioning system was very helpful tool for evaluation of so called "surface blackbodies", which are used for calibration of radiation thermometers and thermal imagers. This automated positioning system is a cartesian coordinate system with two added rotations at the top of it. All together it has 5 degree of freedom (DOF). Complex measurement procedure can be performed due to its mechanical characteristic and very good positioning accuracy and repeatability. For each joint a motorized movement control by means of a stepper motor it is used. The core of a control unit is a microcontroller with a built-in serial peripheral interface for communication with a PC. The user interface is implemented as a virtual instrument in LabVIEW programming environment, which allows manipulation of a particular joint as well as preprogramming of any complex positioning algorithm and sophisticated measurement procedures.

Evaluation of surface blackbodies was performed with a radiation thermometer. At each set temperature the radiation thermometer scanned a circular surface of a blackbody. The first measurement was made in the center a blackbody. The following sets of measurements were made in circles around the center. Each circle has a radius larger than a previous circle for the diameter of the nominal target size of the used radiation thermometer.

Two evaluated surface calibrators are designed for calibration of radiation thermometers and thermal imagers with spectral band between 8 µm to 14 µm. The first one has a temperature range from –15 °C to 120 °C, the second one has a temperature range from 35 °C to 500 °C. Both calibrators are of the same size, with a large target (surface blackbody) of circular shape. The diameter of target is 152,4 mm with the nominal emissivity of 0.95. The calibrators also have a variable emissivity adjustment to vary their apparent emissivity from 0.90 to 1.00. Evaluation results at different temperature and emissivity set points of surface blackbodies will be presented.

ANALYSIS OF THERMAL IMAGERS

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Thermal imagers with microbolometer focal plane array (FPA) detector which does not require cooling are relatively modern instruments. They are very useful in technical diagnostic (fault detection, performance analysis), safety imaging (military, police) but increasingly used also for absolute measurement of temperature (energy efficiency, research, human and animal temperature screening). Their use seems rather straightforward but in reality there are many parameters which must be encountered. Evaluation of thermal imagers with FPA detector was performed to identify parameters which affect their performance. Evaluation methods and measurement of parameters such as accuracy, temperature resolution, drift between internal calibration, non-uniformity, size-of-source-effect and distance effect is discussed. Based on the results it is indicated which parameters should be evaluated in the calibration procedure as well as uncertainty budget. Suitability of a blackbody for calibration was also addressed, especially in terms of temperature homogeneity and dimensions related to the field-of-view of a thermal imager at the minimum focal distance.

Keywords:* Blackbody, Calibration, Thermal imager, Thermogram, Uncertainty

RADIOMETRIC CALIBRATION OF THE RESPONSIVITY OF NPL'S LP3 PYROMETER

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The National Physical Laboratory (NPL, UK) has recently applied radiometric methods to calibrate a Linear Pyrometer 3 (LP3) as part of the high temperature fixed-point research plan [1]. The LP3 instrument is a portable, silicon-based, low size-of-source effect pyrometer traditionally calibrated against a fixed-point according to the ITS-90. It is envisaged that a direct radiometric calibration of the instrument will yield reduced uncertainties compared to ITS-90 approaches.

In this poster we describe a radiometric calibration according to the 'radiance approach' – that is for radiance responsivity. Traceability is to the cryogenic radiometer via a monochromatic source of known radiance. This complements and provides a comparison for work performed with filter radiometers in the 'hybrid approach', as described in [2].

During the calibration a series of interference fringes were observed at the peak of the instrument's responsivity spectrum, complicating the calibration. This poster presents the calibration methods and discusses the techniques to mitigate the effects of the interference fringes on the measured spectral responsivity. Results from fixed-point measurements based on both ITS-90 and radiometric calibrations will be presented.

[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

[2] E.R. Woolliams, M.R. Dury, T A Burnitt, P.E.R. Alexander, R. Winkler, W.S. Hartree, G. Machin. "Primary Radiometry for the *Mise-en-Pratique* for the definition of the kelvin: The Hybrid Method." Submitted to this conference.

EFFECTIVE RADIANCE TEMPERATURE AND ITS MEASURING EQUIVALENT WAVELENGTH

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The theories of classical radiance temperature (RT) and effective wavelength for pyrometry neglect the influence of ambient radiation as the early applications were for high temperature measurement. Nowadays the applications of radiation thermometry have been extended through to the temperature below room temperature and needed more accurately. In widespread use, the influence of ambient radiation on RT measurement is probable not to be negligible, and is considered and corrected as a kind of error factor.

Considering the effect of the ambient radiation, this paper presents the concept of the effective radiance temperature (ERT) which is suitable for a measurement at an arbitrary temperature, and furthermore, develops the theories of the integral effective radiance temperature (IERT) and the EW based on the band radiation thermometer (BRT), in which the deviations in actual thermometric theory from the strict definition of classical RT are avoided. The measurement result, *i.e.* IERT, of a BRT depends on not only the object temperature, emissivity and the ambient radiation characteristic, but also the BRT responsivity. Therefore it is not the simplex feature of measured object and ambient. The EW links the subjective measurement result, IERT, of a BRT to the objective radiation characteristic, ERT, of measured object.

The existence and the non-uniqueness of the EW for BRT measurement are discussed and the relation of geometric meaning and physical meaning of the EW is illuminated.

The ERT feature has marked differences with the one of the classical radiance temperature. The expression of the ERT needs to point out the measurement conditions, the spectrum condition and also ambient radiation condition. At an isothermal ambient, the value of the ERT is between the measured object temperature and ambient temperature, and the ERT is less than, equal to and more than the object temperature respectively if the object temperature is more than, equal to and less than the ambient temperature.

The ERT theory can cover the classical RT theory. The ERT is identical with the RT when neglecting the ambient radiation. The new ERT's definition is more consistent with the apparent temperature essence and benefit to study its characteristic and measurement uncertainty.

Keywords: Effective radiance temperature, Radiation thermometer, Equivalent wavelength, Effective wavelength.

RESEARCH ON ACCURACY OF 4-BAND PYROMETER USING MONTE-CARLO METHOD

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The principle of multi-band pyrometer is to measure radiant energy of each band radiating from high temperature object, and solve the corresponding mathematic model to get the measured temperature. It has the advantage of reducing or even eliminating the effect of object emissivity and surroundings condition, and has better prospect in the field of high temperature measurement. But it is difficult to evaluate the measurement uncertainty for its non-linear model. The calibration method, calculation model, effective wavelength, emissivity model, ambient temperature and measurement distance all affect the measurement uncertainty. This paper evaluates the measurement uncertainty of a 4-band pyrometer using Monte Carlo Method (MCM) to investigate the influence and optimize the pyrometer.

When blackbody furnace is used to calibrate the pyrometer at reference temperature, the furnace performance would have influence on the result. The effective wavelength of each band, which is calibrated using blackbody furnace also, is another factor influenceing the result obviously. Apart from blackbody performance, it relates to the interval of the temperature, spectral responsivity of detector and transmittance of optical system. The emissivity model, ambient temperature and measurement distance, which are relevant to property of measured object and operation condition, also contribute to the uncertainty.

Generated a series of random data according to the probability distribution function of each factor from theoretical and experiment analysis above, the Monte Carlo Method is used to get the uncertainty distribution of the pyrometer. The result shows that measurement uncertainty mainly arises from effective wavelength and reference temperature, and the pyrometer can measure the object high surface temperature in an acceptable uncertainty under the condition of emissivity increasing or decreasing along with wavelength slowly, measuring distance from 500 mm to 1000 mm, and ambient temperature below 400 °C. Optimized according to the result, the 4-band pyrometer accuracy can be better than 0.4% when measured temperature rang from 800 °C to 1600 °C.

CCD-CAMERA FOR MEASURING TEMPERATURE AND SPECTRAL RADIANCE

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The application of thermal imagers to measure the two dimensional distribution of temperature in the mid to the far infrared spectral range is an established technique. However, this wavelength range is not well suited for temperatures exceeding a few hundred degrees Celsius. For higher temperatures the application of imaging systems operating in the visible or even in the near UV spectral range is the method of choice. Although the application of imaging systems for the measurement of two dimensional luminance distributions has reached already an increased level of accuracy, up to now the application of such systems to measure temperatures and absolute spectral radiances is not widely and traceably performed. In this paper we describe the radiometric investigation of the camera system *TechnoTeam LMK 98-4* for traceable temperature and spectral radiance measurements at temperatures above 1000 °C.

To apply such a luminance camera for absolute measurement of temperature and spectral radiance it requires specially adapted filters and objectives. However, in contrast to point-like measurement systems, the application of interference filters causes problems when using a CCD sensor as a detector for measuring two dimensional distributions of temperature and radiance. Therefore, prior to the application of the camera, the usability of this camera system for the measurement of the radiance of high-temperature blackbodies and the applicability for an absolute calibration has been tested.

EFFECTS OF ENVIRONMENTAL CONDITIONS ON PERFORMANCE OF THERMAL IMAGERS

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In thermography of buildings, the thermal imagers are mostly used under ambient conditions, which differ substantially from the laboratory conditions during the calibration of the imagers. Thus the precision of the measurements can be significantly influenced by uncertainties related to the extrapolation of calibration to the lower temperatures. Nevertheless, the effects of ambient conditions on the output of thermal imagers are rarely taken into account by users. Thus there is a need to develop calibration setups for performing the calibrations at the ambient temperatures similar to the actual working conditions.

A method is described for determining the effects of the ambient conditions on thermal imagers in the ambient temperature range from –10 °C to 23 °C. A measurement setup consisting of a climatic chamber and a flat plate blackbody calibrator is developed to simulate the measurement conditions corresponding to the real outdoor conditions in thermography of buildings. The lower temperature limit has been selected based on typical field measurement conditions in northern countries, while the upper limit is a typical laboratory temperature at the calibration.

The targeted uncertainty level for the calibration, 1 K (k=1), has been derived from the practical needs of the research community working on simulation of the energy efficiency of buildings. The measurement setup along with the characterization measurements for uncertainty estimation are described in detail. The limiting uncertainty components are related to the emissivity of the blackbody and to the measurement geometry.

STUDY ON REDUCING THE SIZE-OF-SOURCE EFFECT OF PYROMETERS

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The size-of-source effect (SSE) corrections of radiation thermometers are required when realizing ITS-90 between the different blackbodies or lamps. In this paper a test pyrometer was established to study the source of SSE. The characteristic and cleanliness of objective lens, the design scheme of aperture stop and inner baffles were found the most dominant influence factor for the SSE. Many efforts were attempted and the SSE of test pyrometer is reduced to 2×10^{-4} when a 50 mm diameter radiance source with a 4 mm diameter central obscuration is measured. The experiment conclusions are applied in the primary standard pyrometer and the SSE is reduced to less than 1×10^{-4}.

Keywords: Aperture stop; Objective lens; Pyrometer; Radiation Thermometer; Size-of-Source Effect.

ON THE USE OF THE PRESENT MESUREMENT MODEL IN THE CALIBRATION OF RADIATION THERMOMETERS

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The model used nowadays to interpret the result of a measurement performed by a radiation thermometer is presented with some detail. It is explained how the expected temperature indication is obtained by comparing the signal that the thermometer should generate with that it actually interprets. It is shown how a radiation thermometer may be calibrated by means of another radiation or by a contact thermometer. Corrections for the calibration of 8 to 14 μm radiation thermometers configured with different instrumental emissivity values are calculated. They include the common case of factory configured thermometer with instrumental emissivity of 0.95. The use of the model is emphasized with other examples. It is employed to explain how a modern automatic radiant surface simulates a greybody of a desired emissivity and temperature to calibrate that type of thermometers and how its indication should be corrected to calibrate an infrared thermometer operating at other spectral band. Similarly the influence of an error in the measurement of detector's temperature is estimated.

LIGHT-EMITTING-DIODE-BASED, INTENSITY- AND WAVELENGTH-STABILIZED RADIANCE SOURCE

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Although laser-based radiance and irradiance responsivity calibration facilities are capable of achieving < 0.05 % (k=2) uncertainties in calibrations of radiation thermometers or filter radiometers traceable to the cryogenic electrical substitution radiometer, the long-term stability of the calibrated radiometers have been difficult to maintain with similar, low uncertainties. The main problem in the long-term stability of the responsivity scale is the often unpredictable change in the spectral responsivity of the filters, which is the primary reason for the change in the instrument throughput. The temporal drift can arise from a change in the amplitude of the spectral responsivity rather than a wavelength shift of the filter. One possible approach to monitor and correct these changes would be to have the filter radiometer measure a known spectral radiance source with uncertainties of < 0.10 % (k=2) prior to its operation.

We describe the design and construction of a light-emitting diode (LED) based integrating sphere source (LISS) which can act as source of stable radiance. The LISS is comprised of a temperature-stabilized high-power LED which is spectrally selected with a narrow-band interference filter prior to the introduction of the radiation into the pressed-PTFE integrating sphere. A Si diode without any spectral filter is attached the LISS for monitoring the quasi-narrow-band radiation. The sphere is used in conjunction with a detector-based radiation thermometer with an internal interference filter which is identical to the external filter on the LISS. For this particular study we have used filters which have a central wavelength of 650 nm with about 10 nm bandpass. We also study the suitability of using blue, green, red and white LEDs which are to be used in conjunction with the appropriate interference filters in a similar setup. Such sources could be used as a check standard to correct the temporal drift in the responsivities of the radiation thermometers or radiometers.

RADIATION THERMOMETRY BASED ON PHOTONIC CRYSTAL FIBRES

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Photonic crystal fibres (PCF) are being used to sense and measure various quantities, e.g., temperature, strain and gas chemical composition. This paper describes a radiation thermometry method based on solid core silica PCF fibre, in which thermal radiation propagates along a fibre from a hot target to an infrared detection system. In solid core PCF fibres, a radial step in refractive index for light guiding is achieved by surrounding the core with cladding containing a periodic array of parallel holes running along the length of the fibre. The advantage of PCF fibres is that they are more resilient to high temperatures than standard silica fibres. They can be used up to at least 600 °C, above which the devitrification (crystallization of the silica) occurs.

In our implementation, the target is an Inconel blackbody and the detection system is an InGaAs 256 array spectrometer with a wavelength range of 907 to 1681 nm. The temperature is determined from the spectrometer signal at particular wavelengths using Planck's relationship. A calibration is first carried out with the black body at a known temperature given by a thermocouple inserted into the rear of the black body. Two tests were performed:

(1) Long term high temperature soak-tests to measure drift and noise in thermal radiation levels, in which spectra are sequentially recorded over a long period of time with the blackbody cavity at a constant temperature.

(2) Temperature dependence tests, whereby thermal radiation spectra are recorded with the blackbody cavity at several temperatures.

The soak tests were carried out for 20 hours with the blackbody at temperatures 595 °C and 934 °C. At 934 °C, the transmission of the PCF fibre slowly decreased at a rate of 0.078% per hour, which compared with a rate of 0.5% per hour for conventional silica fibre. The decrease in transmission of PCF fibre is thought to be due to devitrification of the silica. At the lower temperature of 595 °C, the transmission of the PCF fibre was constant over time.

The temperature dependence tests have been carried out with the blackbody target at a series of temperatures in the range 400 °C to 800 °C. The test confirmed that the thermal radiance is following Planck's law. Single and two-colour analysis of the thermal radiation levels were made It was found that two-colour analysis did not reduce effects due to the decrease in transmission with time, and provides no advantage.

The thermometer could be employed in situations where there is electrical noise, limited optical access, or a need for low-drift and low maintenance, for example in solid oxide fuel cells.

CHARACTERIZATION OF THE 300 K AND 700 K CALIBRATION SOURCES FOR SPACE APPLICATION WITH THE BEPICOLOMBO MISSION TO MERCURY

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The Mercury Thermal Infrared Spectrometer (MERTIS) onboard the European-Japanese space mission BepiColombo to Mercury will be launched in 2014. MERTIS scientific objective is to identify rock-forming minerals and measure surface temperatures by infrared spectroscopy (7 µm to 14 µm) and spectrally unresolved infrared radiometry (7 µm to 40 µm). To achieve this goal MERTIS utilises two onboard infrared calibration sources, the MERTIS blackbody with 700 K (MBB7) and the MERTIS blackbody with 300 K (MBB3), together with deep space observations. All three sources can be observed one after the other using a rotating mirror system.

Both blackbody radiators have to fulfil the severe mass, volume and power restrictions of MERTIS. The radiating area of the MBB3 is based on a structured surface with a high-emissivity space qualified coating. The relatively high emissivity of the coating was further enhanced by a pyramidal surface structure to values over 0.99 in the wavelength range from 5 µm to 10 µm and over 0.95 in the wavelength range from 10 µm to 33 µm.

The MBB7 is based on a small commercially available surface emitter in a standard housing. The windowless emitter is an electrical heated resistor, which consists of a platinum structure with a blackened surface on a ceramic body. The radiation of the emitter is expanded and collimated through use of a parabolic mirror.

The design requirements and the radiometric and thermometric characterization of these two blackbodies are described in this paper.

CALIBRATION OF THERMAL IMAGERS BY EVALUVATION OF THE ENTIRE FIELD-OF-VIEW

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Calibration laboratories in industry and national metrology institutes commonly apply equipment and procedures developed for optical radiation thermometers also to calibrations of thermal imagers. Calibration of thermal imagers consists of positioning the thermal imager under calibration in front of a blackbody and comparing measured temperature with reference temperature. Reference thermometer is either a radiation thermometer or a contact thermometer. Such a calibration does not cover complete field-of-view (FOV) of the thermal image. This approach provides accurate calibration results only for a certain percentage of the detector pixels, which are in view of a blackbody. For the remaining pixels it is assumed that they have the same temperature characteristic. This inference may not be always true.

To obtain more comprehensive calibration data, special instruments and methods have been developed, but not introduced in calibration laboratories due to their complexity and high cost. To overcome the problems, two new calibration procedures will be presented on the basis of the methods currently in use in the calibration laboratories. A minimum set of additional features will be identified and implemented to obtain results necessary and relevant for the end-user applications.

First procedure consists of dividing the imager FOV in partial FOVs according to the certain blackbody aperture. Calibration is performed by moving thermal imager with positioning system in such a way that each of partial FOVs is progressively positioned in front of a blackbody aperture. This procedure needs accurate and repeatable positioning system. The idea of the second procedure is to position at the same time all of detector pixels (entire FOV) in front of a blackbody aperture. For this purpose a large aperture variable temperature blackbody will be constructed. The blackbody aperture will be at least 250 mm in diameter for the temperature range from –40 °C to +90 °C. Experimental work and analysis of calibration procedures of thermal imagers will be presented.

REALIZATION AND VALIDATION OF RADIANCE TEMPERATURE SCALE IN THERMAL INFRARED AT TEMPERATURES UP TO 1000 °C

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A recently developed AIRI capability for realization and transfer of infrared spectral radiance and radiance temperature scales has been expanded to cover temperatures up to 1000 °C. The paper summarizes the scale realization approach, including evaluation of spectral emissivity for newly built heat pipe blackbodies, their comparison with In, Sn, Zn, Al, Ag and Au fixed point blackbodies, and the uncertainty budget analysis.

The radiance temperature scale realization has been partially validated via international pilot study reported separately at this conference. The pilot study was limited to the temperatures up to 300 °C and was performed using a comparator with a spectral band from 8 to 14 microns.

In order to better understand and validate the spectrally dependent components of scale realization and transfer, an internal comparison in spectrally resolved radiance temperature with the NIST spectral emittance measurement facility has been performed. The NIST spectral emittance facility has complementary capabilities, including a completely different design of spectral comparator and another set of heat pipe blackbodies. The transfer standard source selected for this effort has a strongly selective spectral features, posing a challenge for a low resolution comparator of the AIRI facility as opposed by high spectral resolution of the Fourier Transform Spectrometer of the emittance facility.

A newly realized and validated capability will become a part of offered calibration services, including characterization of infrared sources in spectral radiance over 3 – 14 μm spectral range, radiance temperature and its spatial uniformity, as well as pyrometers and imagers calibration.

POSTER SESSION III

Instrumentation

Thursday
10:30 to 11:10
and
14:15 to 15:00
Foyer

INRIM HEAT PIPES FOR USE WITH FIXED POINTS

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New heat pipes (HPs) have been developed at the Italian Istituto Nazionale di Ricerca Metrologica (INRiM) and the first HPs, with different working fluids, for use with fixed points have been installed and tested. A sealed stainless steel HP has been filled with biphenyl and is now regularly used for the realization of the Indium fixed point. After a previous prototype presented in 2007, a second HP, also in stainless steel, has been made and filled with mercury as working fluid. It is used with satisfactory results for the realization of the tin fixed point. This second one is equipped with a manual valve at the top and can also be partially controlled in pressure.

A description of the devices and some of the obtained results will be presented, along with a short summary of the progress made in the development of a new temperature control line with in-house made electronics for use with the same fixed-point furnaces. Future plans for this activity are discussed, as new Inconel HPs for high temperatures fixed points are now being constructed and will be subject of further investigations.

THE USE OF THERMOWELL BUSHES AT THE TRIPLE POINT OF WATER FOR IMPROVING REPEATABILITY

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Water triple point cells are essential for a realisation of the international temperature scale of 1990 (ITS-90). There is some evidence that the ultimate performance of water triple point cells may be restricted by the variation in the position of the platinum resistance thermometer at the bottom of the re-entrant well, and in addition that the variation in position is not completely compensated by correction to zero measurement sensing current.

This comparative study focuses on the use of quartz bushes of two different lengths and examined whether an improvement in repeatability of the resistance readings was possible. The experiment was conducted over a five-week period using a standard platinum resistance thermometer, one water cell and two different lengths of quartz bushes. The resistance measurements were performed using an F900 resistance bridge.

A description of the experiment and results are given. Significant improvements in the repeatability of the measurements were observed (factor >2) when quartz bushes were used.

THERMAL CHARACTERISITICS OF A SEALED GLASS-WATER HEAT PIPE OVER THE TEMPERATURE RANGE FROM 0 °C TO 60 °C

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Heat pipes are effective heat transfer devices by means of phase transitions of the working fluids. In the thermometry field, due to their good isothermal characteristics, diverse heat pipes operating in different temperature ranges have been applied to provide uniform and stable temperature environment. Therefore, the heat pipes play an important role in promoting the level of realizing the fixed points in the International Temperature Scale of 1990 (ITS-90) and of calibrating the standard platinum resistance thermometers (SPRTs), thermocouples and optical pyrometers. In view of these reasons, the National Institute of Metrology (NIM) is conducting investigations on sodium heat pipes, gas-controlled sodium heat pipes and water heat pipes.

By referring to literatures, water heat pipes operate over the temperature range from 30 °C to 200 °C. To the authors' knowledge, there is no report on the water heat pipes operating at the temperature lower than 30 °C. Moreover, the heat transfer mechanism inside the heat pipe is rather complicated due to the process of evaporation and condensation. Glass-water heat pipes are quite suitable for the visualization of the water phase transition inside the heat pipe. Consequently, borosilicate glass-water heat pipes with four thermometer wells were developed according to the procedure for the fabrication of the triple point of water (TPW) cells, which can make sure the good quality of the glass-water heat pipes.

An investigation on thermal characteristics was conducted when the heat pipe at 0 °C, 20 °C, 30 °C, 40 °C, 50 °C and 60 °C. The obtained results show that the water heat pipe can obviously improve temperature stability of the liquid bath, since it can keep the temperature constant by absorption or liberation of the latent heat to attenuate temperature variations of the surroundings. Meanwhile, the long-term temperature stabilities at the temperatures of 0 °C, 20 °C and 30 °C within at least13 hours are better than 0.2 mK. Furthermore, the ones are within 1 mK when the water heat pipe at approximately 40 °C, 50 °C, and 60 °C. Finally, the corresponding measures to further improve temperature stability and uniformity and the heat transfer mechanisms are discussed.

CONSTRUCTION AND CHARACTERIZATION OF A LARGE APERTURE BLACKBODY FOR INFRARED RADIOMETER CALIBRATION

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A large aperture blackbody with a diameter of 1 m has been constructed for calibrating infrared radiometers with wide field of view in temperature range between 10 °C and 90 °C. The blackbody is cylindro-conical cavity (length: 1 m) with conical bottom of 120 apex angle. The cavity is integrated into a water reservoir. A high pressure pump introduces water flow along the cavity to improve temperature stability and uniformity. A numerical analysis was carried out to find the design parameter such as water flow rate, water volume and electrical power. The inside of the cavity was purged by dried air through heat exchanger immersed in the water reservoir to reduce convection heat loss to ambient air. A reference radiation thermometer (RRT) and a platinum resistance thermometer (PRT) measured temperature stability of the blackbody, which was within 0.1°C in the temperature range between 10°C and 90°C. A thermal camera measured radiance temperature distribution on the blackbody aperture, of which uniformity was within 0.2°C in the range between 20°C and 70°C. Considering intrinsic emissivity of the coating material and temperature distribution along cavity, the Monte-Carlo simulation code (STEEP3) calculated effective emissivity of the blackbody to be 0.996 from 1 μm to 15 μm. The PRT was calibrated in terms of radiance temperature in reference to a reference radiation thermometer calibrated by the multiple fixed- point method. Uncertainty of the radiance temperature scale was evaluated by considering the transfer uncertainty, the uniformity and stability. The expanded uncertainty varies from 0.3 to 0.5 °C (k = 2) in the temperature range between 10°C and 90 °C.

REALIZATION OF A ^{3}HE – ^{4}HE VAPOUR PRESSURE THERMOMETER FOR TEMPERATURES BETWEEN 0.65 K AND 5 K AT LNE-INM/CNAM

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In the temperature range between 0.65 K and 5 K, the International Temperature Scale of 1990 (ITS-90) is based on ^{3}He and ^{4}He vapour-pressure thermometers. Between 0.65 K and 1 K, the ITS-90 overlaps with the Provisional Low Temperature Scale of 2000 (PLTS-2000), defined in term of the melting pressure of ^{3}He. Some differences, up to more than 1 mK, exist between the two scales in the overlapping interval.

The LNE-CNAM has recently started the construction of a ^{3}He – ^{4}He vapour-pressure thermometer to realize the ITS-90 in its lowest sub-range.

The device is provided with two separate vapour-pressure chambers, one for the ^{3}He and the other one for the ^{4}He, built in a single copper block, and is installed in the experimental space of a dilution refrigerator.

Two pressure tubes, dimensioned to minimize the thermomolecular pressure and aerostatic head corrections, are used to connect the thermometers to the pressure sensors located outside the cryostat. The tubes are designed to prevent the formation of cold spots, and are equipped with a series of thermometers and heaters to control their temperature, in order to evaluate the effect of thermal gradients on the pressure measurements.

The pressure sensors and the gas handling system are placed in a temperature-controlled enclosure, to minimize the perturbations.

The vapour-pressure thermometers are designed to accommodate on the same copper block several transfer standards, an acoustic thermometer and the ^{3}He melting pressure thermometer. This latter will allow the direct comparison between the ITS-90 and the PLTS-2000 in the overlapping temperature range.

The realization of the system has been recently accomplished and this report illustrates the characteristics of such experimental device.

Keywords: Vapour pressure thermometry; Cryogeny; ITS-90; PLTS-2000; Acoustic thermometry; Thermodynamic Temperature.

CRYOSTAT FOR FIXED-POINT CALIBRATIONS OF CAPSULE-TYPE SPRTS

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We built a cryostat for fixed-point calibrations of capsule-type SPRTs (standard platinum resistance thermometers). Using this system, we realized cryogenic fixed-points defined in International Temperature Scale of 1990 (ITS-90). The cryogenic cells were argon, oxygen, neon and two equilibrium-hydrogen (e-H_2) cells made by iNRiM, Italy. The uncertainty of the realization of each fixed points were estimated to be 0.53 mK to 0.43 mK (k = 2). The realizations of the two e-H_2 coincided within 0.1 mK. Therefore, we are able to calibrate capsuletype SPRTs down to 24.5561 K within the uncertainty of 1 mK (k = 2) by this system.

We used a closed-cycle helium gas refrigerator for the cryostat. The base temperature of this liquid-free cryostat, when a sealed cell and three capsule-type SPRTs were attached for calibrations, was ~ 17 K. For the realization of the e-H_2, we used liquid helium for additional cooling. Adiabatic melting of the triple point was realized by controlling the inner-most radiation shield to a temperature very close to that of the triple point, and applying pulse-heat by a heater directly wound to the cell. The adiabatic melting took ~ 6 h for each cell if appropriate amount of accumulated pulse-heat was applied to the cell. The triple points of each cryogenic fixed-point were deduced from the equilibrium temperature between the heat pulses. For the oxygen cell, temperatures of the two solid-solid transition (α–β and β–γ transitions) were also measured, and the results were consistent to the literature values within the uncertainty.

AN ADIABATIC CALORIMETER FOR THE REALIZATION OF THE ITS-90 IN THE CRYOGENIC RANGE AT LNE-INM/CNAM

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The LNE-CNAM, in cooperation with the IPN, has recently realized a new adiabatic calorimeter based on a cryogen-free refrigerator, to realize the International Temperature Scale of 1990 (ITS-90) in the temperature range between 6 K and 84 K.

The new calorimeter, cooled by a cryogen-free Gifford-McMahon refrigerator, is equipped with three thermal shields and two separate vacuum chambers, to minimize the effect of parasitic heat fluxes. The inner adiabatic chamber can accommodate either a multicompartment cell - containing the triple points of hydrogen, neon, oxygen and argon, to realize the ITS-90 between 14 K and 84 K - or a thermometer comparison block, for the calibration of rhodium-iron (RhFe) thermometers between 6 K and 24 K. Several control thermometers and heaters, mounted on the thermal shields and controlled by a computer, are used to regulate the temperature of the whole experimental environment. The use of a cryogen-free system and the fully computer-controlled measurement chain allow long lasting experiments and a good thermal control, bringing to a substantial reduction of the measurement uncertainties.

The new adiabatic calorimeter was successfully tested at the LNE- CNAM, and several measurement runs were performed using the last generation of multicompartment fixed-point cells.

In the range between 14 K and 84 K, while the overall uncertainties in the realization of the oxygen and argon triple points are almost the same as those obtained in the past, a strong improvement in the realization of the triple points of hydrogen and neon was achieved. The overall uncertainty was reduced from 2.08 mK to 0.37 mK at the hydrogen triple point, and from 1.40 mK to 0.30 mK at the triple point of neon.

In the temperature range between 6 K and 24 K, rhodium-iron resistance thermometers were calibrated by comparison with calibrated thermometers, and the overall uncertainty obtained was of the order of 0.80 mK, dominated by the uncertainty of the reference thermometer.

NOISE THERMOMETRY AT LOW TEMPERATURES: MFFT MEASUREMENTS BETWEEN 0.001 K AND 1 K

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Recently, we have developed the Magnetic-Field-Fluctuation Thermometer (MFFT) for practical thermometry in the low temperature range [1]. Its operational principle is based on the Nyquist theorem ensuring a linear characteristic over a wide range of temperatures. This makes the MFFT a thermometer capable to replace a variety of secondary thermometers which are usually required to cover the whole temperature range of the International Temperature Scale PLTS-2000 from 0.001 K to 1 K.

Here we describe a compact and easy to use MFFT system comprising a metallic temperature sensor with a SQUID magnetometer, a data acquisition unit and a software package. For measurements at very low temperatures, the MFFT setup is specially designed to have a high signal level and, at the same time, a sufficiently high bandwidth for fast measurements. For the first time, we report on MFFT measurements down to temperatures of about 1 mK. The results are given in comparison to a high accuracy realization of the PLTS-2000.

[1] J. Engert, J. Beyer, D. Drung, A. Kirste, M. Peters, Int. J. Thermophys. (2007) **28**:1800–1811

ADVANCEMENT IN THERMAL CONTROL FOR THE REFERENCE TEMPERATURE FACILITY USING ENHANCED HEAT TRANSFER

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The paper describes a control system development for the reference surface temperature facility. Facility is based on a heat pipe principle for the hot plate design developed and tested earlier by the authors. In order to provide the accurate calibration procedure the complete study of heat pipe thermal performance is necessary in temperature range from 20 to 200 °C.

Pressure and temperature control system is important for extensive tests of heat pipe providing the stability and reproducibility of small temperature gradients. Development of multi-parametric control system lead to the structural modification of heat transfer device with evaporationcondensation circle. Paper describes concept, structural and mass distribution peculiarities of modified heat pipe joined with auxiliary heater, with pressure measurement gauge and line. System requires the thermodynamic & thermal state detailed analysis.

Multi-parametric direct measuring system allows monitoring a vapor-liquid stability at saturation vapor set temperature. Improved mechanism of control is used for study a correction values, for uncertainty minimization in processes of surface temperature sensor calibration and for improving of reference facility self-calibration. Such an approach provides a self-compensation to environmental parameters fluctuations and is important for study of processes sensitive to temperature gradient for various applications, including microbiology.

EVALUATION OF FLAT SURFACE TEMPERATURE PROBES

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The objective of the paper is elaboration of elements related to metrological analysis in the field of surface temperature measurement. Surface temperature measurements are applicable in many fields. As an example safety testing of electrical appliances and pharmaceutical production line will be a case study for surface temperature measurement. In both cases correctness of the result of the surface temperature has an influence on final product safety and quality and thus conformity with specifications. This paper will deal with the differences of the flat surface temperature probes measuring surface temperature. Different measurement approaches for measuring surface temperature are possible. For the purpose of safety testing of electrical appliances surface temperature measurements are very important for safety of user. General requirements are presented in European standards, which support requirements in European directives e.g. European Low Voltage Directive 2006/95/EC and pharmaceutical requirements, which are introduced in official states legislation.

Paper will introduce comparison of temperature measurement between attached thermocouple on the measured surface and measurement with flat surface temperature probes. As a heat generator a temperature artifact will be used. Probes and thermocouples will be applied to the surface in horizontal and vertical position, using also different force for application of probes. Reference temperature will be measured by J-type fine-wire (0.2 mm) thermocouple. Two probes will be homemade according to requirements in European standard EN 60335-2-9/A12, but one with fine-wire (0.2 mm) thermocouple and one with 0.5 mm of thermocouple wire diameter. Additional commercially available probes will be compared.

It is expected that there will be differences between probes due to thermal conditions caused by application of the probe. Therefore it sometimes happens that measurements are performed with improper equipment or in an improper way. It is important that measurement results and their associated target uncertainties are correctly evaluated and on that basis right conclusions of conformity or nonconformity with specifications are made. Therefore the knowledge and awareness regarding all facts related to the used measuring equipment is essential.

LOW-COST RATIOMETRIC FRONT-END FOR INDUSTRIAL PRT APPLICATIONS

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Cost, size, speed and measurement range limitations make the resistance bridge not always suitable for temperature measurements with PRTs in industrial applications. However, high-accuracy resistance thermometer systems are often needed in many industrial applications, where measurement performance comparable to resistance bridges are often needed at a lower cost and size.

A compact, portable, ratiometric front-end exploiting a 24-bit analog-to-digital converter (ADC) with ΣΔ modulator is described. It was designed to measure the resistance ratio between a 100-ohm IPRT and a reference resistor with repeatability to within few ppm. Its small size makes it ideal to be integrated within the stem-handle assembly of a thermometer probe, enabling an early transmission of measurement data in a digital form.

The ADC system design, development and testing are discussed in the paper. Performance tests included repeatability, linearity, sensitivity to ambient temperature and electrical noise. They were investigated in a resistance ratio range from about 0.01 to unity. In addition, a comparison between the system performance and an AC resistance bridge was carried out and the results discussed in the paper.

An accurate thermometer for industrial applications, made of a Pt-100 sensing element, a ratiometric front-end and a digital readout module, resulted from the above developments. The compactness of the devices enabled an implementation of the 'smart sensor' concept in the measurement chain, where the front-end electronics was placed inside the IPRT handle together an integrated memory to hold device identification, calibration coefficients and the associated uncertainty. All data are transmitted to the readout module and are available to the user at a 2 Hz update rate for any further analysis.

POSTER SESSION III

Interlaboratory Comparisons

Thursday
10:30 to 11:10
and
14:15 to 15:00
Foyer

BILATERAL COMPARISON OF RELATIVE HUMIDITY STANDARDS BETWEEN NMISA AND MIKES

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This paper describes a bilateral comparison of the relative humidity measurement standards of NMISA (South Africa) and MIKES (Finland). The purpose of this comparison was to test the relative humidity calibration capabilities of NMISA, particularly at temperatures other than ambient (19 °C to 25 °C). This paper will present the results of the comparison, as well as further measurements performed to determine the causes of the differences between the participants.

Five thermohygrometers with capacitive sensors were used as comparison artefacts. The comparison range was 10 %rh to 95 %rh at temperatures from 5 °C to 55 °C. NMISA calibrated the artefacts against reference unsaturated salt solutions, while MIKES calibrated the artefacts using two chilled mirror hygrometers as reference standards.

Results obtained by the laboratories agreed fairly well for one thermohygrometer, which had a wire mesh filter, but measurement points performed with the other four, which had less porous filters, showed significant differences. Measurements at NMISA were performed at constant humidity and rising temperature, in static conditions (no air flow), without removing the filters from the sensors, while MIKES performed measurements at constant temperature and rising humidity, in flowing air, with filters removed. Further measurements are being performed at NMISA to evaluate the comparison differences. With these measurements the effects of filters, air flow and differences in measurement order are investigated.

BILATERAL COMPARISON BETWEEN CEM AND LACOMET IN THE RANGE 83,805 8 K TO 993,473 K, LINKING TO CCT COMPARISONS

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A bilateral comparison between Centro Español de Metrología (CEM) and Laboratorio Costarricense de Metrología (LACOMET) in the range 83.8058 K to 993.473 K has been developed during 2009 and it is aimed to provide linkage to the CCT key comparisons K3 and K4 to LACOMET. This comparison gives support to the Calibration Measurement Capabilities requested by LACOMET. The participation of CEM in the EURAMET regional comparisons EUROMET-T.K3 and EUROMET-T.K4 is the basis of the link.

Two 25 Ω standard platinum resistance thermometers (SPRTs) were used as traveling standards. One of them was used only in the aluminum freezing point while the other one covered the remaining fixed points. An exception was made at the temperature of 83.8058 K, CEM performed the measurements in an Argon Triple Point Apparatus but LACOMET calibrated the SPRT at a temperature close to the argon point using a Liquid Nitrogen Boiling Point Apparatus.

Both SPRTs were provided by LACOMET and they were measured before and after CEM measurements. The SPRTs showed no significant drifts during the comparison. The results for both laboratories agreed within their expanded uncertainties and are summarized. Proposals for key comparison reference values and for the linkage to the results of CCT-K3 and CCT-K4 are presented.

INTERCOMPARISON OF TEMPERATURE BLOCK CALIBRATORS

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During recent years temperature block calibrators became increasingly popular for thermometer calibrations, in industrial calibration laboratories but also for specific tasks at NMIs. With the publication of the EURAMET/cg-13/v.01 the users of temperature block calibrators have access to an agreed procedure to quantify different sources of uncertainty and to validate calibrations by means of block calibrators. At the other hand manufacturers of block calibrators became the advantage of transparent and uniform assessment criteria of their products and a better basis for further developments. As a result considerable improvements of the actual generation of temperature block generators were reported within the temperature community and as a consequence lower measurement uncertainties were requested within the framework of accreditations within the German Calibration Services (DKD).

Therefore, in 2008/2009 an intercomparison of temperature block calibrators was carried out between 17 DKD accredited laboratories and PTB as pilot. Within the framework of this intercomparison different types of block calibrators were investigated in the temperature range between -24 °C and +600 °C. The results demonstrate the achievable potential of the current generation of temperature block calibrators and the capabilities of accredited laboratories in Germany.

BILATERAL COMPARISON OF THE DEW POINT TEMPERATURE BETWEEN TUBITAK UME AND NIST

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This work describes the results of a bilateral comparison which has been conducted between the TUBITAK UME (Turkey) and NIS (Egypt) in the field of dew and frost point over the range from –40 °C up to 50 °C. This comparison is registered as EUROMET Project No. 1137.

Both institutes used two-pressure humidity generator in this comparison. Dew point meter (manufacturer MBW, model DP30) with its accessories belonging to NIS was transported to TUBITAK UME for bilateral comparison. After the measurements had been performed at TUBITAK UME by using dew point meters (manufacturer MBW, model 373 and 373H), the same measurements were carried out at NIS premises.

TUBITAK UME participated in EUROMET T.K6 key comparison in the range –30 °C to 20 °C. So, the results of this bilateral comparison can be used in terms of quality assurance and validation of calibration and measurement results.

The expected output of this bilateral comparison will provide valuable information concerning the equivalence of the dew-point temperature scales realized by the two laboratories. The detailed description for the equipment used and the analyses of the comparison results will be presented in the paper. The results of comparison will be evaluated using the technique of normalized error ratio E_n which will show that the measurements at both laboratories with their claimed uncertainties are compatible.

RADIOMETIC COMPARISON BETWEEN A NATIONAL LABORATORY AND AN INDUSTRIAL LABORATORY

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One of the disciplines that Fluke – Hart Scientific has is radiometric calibration. Part of this program involves use of a radiation thermometer with a pyroelectic detector. It is used as a radiometric transfer standard between a set of liquid bath variable temperature blackbodies and a flat-plate infrared (IR) calibrator. The flat-plate calibrator is designed for use in calibration of handheld IR thermometers.

The traceability of the variable temperature blackbodies comes by contact thermometry through the National Institute of Standards and Technology (NIST). A verification of these blackbodies is a comparison between a calibration done by the Radiance Temperature Laboratory at NIST and the blackbodies at Fluke – Hart Scientific. This comparison uses a transfer radiation thermometer (TRT) as a check standard. It would be more desirable to use radiometric traceability as an indication of the blackbodies' radiometric temperature. However, contact thermometry provides much better uncertainties. These uncertainties are needed for the radiometric transfer from the blackbodies to the flat-plate calibrators. Thus the NIST radiometric calibration of the TRT is used for verification of normal equivalence.

This paper discusses Fluke – Hart Scientific's blackbody traceability. It covers the Fluke – Hart Scientific and the NIST radiometric calibration procedures. It discusses the radiometric uncertainty budgets at both Fluke – Hart Scientific and at NIST. It then discusses the results of this comparison and analyzes the results. The comparison is in the temperature range of –15 °C to 500 °C. It showed a normal equivalence of less than 1.00 at all points. The paper concludes with a set of future actions to ensure quality in Fluke – Hart Scientific's radiometric calibration program.

INTER-LABORATORY COMPARISON OF INFRARED EMITTANCE SCALES

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NIST, PTB, LNE, INRIM and NMIJ, have joined in an inter-laboratory comparison of their infrared spectral emittance scales. This action is part of a series of pilot inter-laboratory comparisons (including thermal conductivity and thermal diffusivity) sponsored by the CCT – WG 9 - Thermophysical Properties. The objective of this collaborative work is to strengthen the major operative National Measurement Institutes (NMI) infrared spectral emittance scales and consequently the consistency of radiative properties measurements carried out worldwide. The comparison has been performed over a spectral range of 1 µm to 12 µm, and a temperature range from 23 °C to 800 °C. Samples included in the comparison are potential standards: oxidized inconel, boron nitride, and silicon carbide. The measurement instrumentation and techniques used for emittance scales are unique for each NMI, including the temperature ranges covered as well as the sample sizes required. For example, all three common types of spectral instruments are represented: dispersive grating monochromator, Fourier transform and filter-based spectrometers. As a result of the difference in temperature ranges, not all NMI scales are compared at all temperatures. However, the overlap of temperature ranges enables at least an indirect comparison between all NMIs. Due to the difference in sample size requirements for each NMI, the emittance inter-laboratory comparison was conducted in a "star" format, in which the pilot laboratory (NIST) distributed separate sample sets to each participant. NIST measured each sample at 23 °C (only) prior to and subsequent to the participant's measurements. The NIST 23 °C results are used to correct for sample to sample variation, as well as to monitor any change due to the measurement processes. Emittance results are compared both directly as a function of wavelength, as well as band-averaged for comparison with the filter instrument results. Final complete results of the comparison will be presented and discussed.

KEY COMPARISON OF WATER TRIPLE POINT CELLS EURAMET.T-K7.1

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Article presents the organization and preliminary results the EURAMET key comparison of water triple point cells EURAMET.T-K7.1 (Project 1080). The decision to organize this comparison was taken during the EUROMET Thermometry Technical Committee that was held in Delft in 31 March – 2 April 2008. Comparison was organized as additional comparison to previously realised regional key comparison of water triple point cells EUROMET.T-K7 (Project 899). The pilot was SMU, participating laboratories were GUM and CMI. The reason of organizing the comparison was a non-satisfactory results obtained for CMI and GUM during realization of EUROMET.T-K7 due to instability of the same model of TPW cell used as a transfer cell by both of the mentioned laboratories. Link to the CCT-K7 was established by means of SMU cells used as transfer cell and national reference during CCT-K7. Technical protocol was in most of its parts similar to that already used for CCT-K7. The main objectives of the comparison were to quantify the difference between the cells, providing the link to the CCT-K7 to GUM and CMI and comparison of national realization of water triple point in the light of adopting new methodologies (mostly concerning isotopic effect).

INTERCOMPARISON OF MERCURY AND GALLIUM FIXED-POINT CELLS USING STANDARD PLATINUM THERMOMETER

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The objective of project EURAMET 1127 (Bilateral comparison of triple point of mercury and melting point of gallium) in the field of thermometry is to compare realization of triple point of mercury (–38,8344 °C) and melting point of gallium (29,7646 °C) between the Slovenian national laboratory MIRS/UL-FE/LMK and Croatian national laboratory HMI/LPM-FSB using long stem 25 ohm standard platinum resistance thermometer (SPRT).

MIRS/UL-FE/LMK participated to number of intercomparisons on the level of EURAMET. HMI/LPM-FSB laboratory recently acquired new fixed point cell which had to be evaluated in the process of intercomparison.

Quartz sheathed SPRT has been selected and calibrated at HMI/LPM-FSB in triple point of mercury, melting point of gallium and water triple point. A second set of measurement was made at MIRS/UL-FE/LMK. After its return the SPRT was again recalibrated at HMI/LPM-FSB. In the comparison the W value of the SPRT has been used.

Results of the intercomparison confirmed that new cell of the HMI/LPM-FSB has the value which is within uncertainty limits of both laboratories that participated in the exercise.

INTERCOMPARISON OF ALUMINIUM FIXED-POINT CELLS USING STANDARD PLATINUM THERMOMETER

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The objective of project EURAMET 1114 (Bilateral comparison of freezing point of aluminium) in the field of thermometry is to compare realization of freezing point of aluminium (660,323 °C) between the Netherlands national laboratory VSL and Slovenian national laboratory MIRS/UL-FE/LMK using long stem 25 ohm standard platinum resistance thermometer (SPRT).

Both of the laboratories participated to number of intercomparisons on the level of EURAMET and also on BIPM CCT level (VSL). MIRS/UL-FE/LMK laboratory recently acquired new fixed point cell which had to be evaluated in the process of intercomparison.

Quartz sheathed SPRT has been selected and calibrated at MIRS/UL-FE/LMK in aluminium freezing point and water triple point. A second set of measurement was made at VSL (NL). After its return the SPRT was again recalibrated at MIRS/UL-FE/LMK. In the comparison the W value of the SPRT has been used.

Results of the intercomparison confirmed that new cell of the MIRS/UL-FE/LMK has the value which is within uncertainty limits of both laboratories that participated in the exercise. Further more, results were compared with results that both laboratories achieved in the EURAMET K4 (project 820) and found to be in agreement.

INTERCOMPARISON OF THE DEW-POINT TEMPERATURE REALIZATIONS AT LPM AND MIKES IN THE RANGE –70 °C TO +20 °C

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The first European humidity key comparison EURAMET-T.K6 was completed in 2008. This comparison was carried out among 24 countries and it covered the dew-point temperature range from –50 °C to +20 °C. Both LPM and MIKES participated in the comparison but a new low dew-point generator was introduced at LPM as a result of progress in the EUROMET P912 project. To extend the range of available comparison evidence down to –70 °C and to study the validity of improved uncertainties of LPM, bilateral comparison was carried out between LPM and MIKES in year 2009. The applied comparison procedure was similar to that applied in EURAMET-T.K6. However, only one transfer standard was used instead of two units and the measurement point –70 °C was added in the measurement scheme.

Applied comparison and analysis methods are described in this paper. Final results are reported in terms of bilateral equivalence. The results obtained with the new generator at LPM are linked to the EURAMET.T-K6 comparison reference values in the range from –50 °C to +20 °C through the MIKES results.

COMPARISON OF FROST-POINT TEMPERATURE SCALES BETWEEN –80 °C AND –10 °C

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In order to investigate the realisation of low frost-point temperature scales at INRIM, LNECETIAT and MIKES a comparison of humidity standard generators was carried out. The frost point temperature range was between –80 °C to –10 °C using, as the travelling standard, a precision chilled mirror dew-point hygrometer.

The travelling standard hygrometer was calibrated at INRIM humidity laboratory toward its primary frost-point generator. Then, the travelling standard was delivered to MIKES in order to proceed to a calibration by comparison against its standard frost point generator. The travelling standard hygrometer came back to INRIM in order to check for its stability and was, subsequently, delivered to LNE-CETIAT humidity laboratory where it was calibrated by comparison to the new frost-point generator "RBT". A final calibration was carried out at INRIM in order to check for stability and/or any potential drift of the travelling standard.

The measured quantity for the comparison was the resistance of one of the two PRTs embedded into the hygrometer mirror whose leads are directly connected to the instrument panel. For each measurement point, the mirror PRT resistance and the frost-point temperature with the associated uncertainty was recorded and, subsequently, analysed.

This work present the measurement protocol agreed among the participants, discusses the analysis method applied for the comparison and summarize the results of the exercise. As a conclusion, a bilateral degree of equivalence is estimated among the participants.

INTERCOMPARISON OF TIN AND ZINC FIXED-POINT CELLS USING STANDARD PLATINUM RESISTANCE THERMOMETER

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The objective of this EURAMET project (Bilateral comparison of Freezing point of Tin and Zinc) in the field of thermometry is to compare realization of Freezing point of Tin (231.928 °C) and Freezing point of Zinc (419.527°C) between the German national laboratory PTB and Croatian national laboratory HMI/FSB-LPM using long stem 25 ohm standard platinum resistance thermometer (SPRT).

As HMI/FSB-LPM is going through accreditation process for the calibration of SPRTs at the fixed points, comparison of the HMI/FSB-LPM fixed points against fixed points from PTB was needed.

Brand new quartz sheathed SPRT has been selected and calibrated at PTB, than it was carefully shipped to HMI/FSB-LPM where second set of measurement was made. In the comparison the W value of the SPRT has been used.

Results of the intercomparison showed satisfactory performance cells of the HMI/FSB-LPM.

INTERCOMPARISON OF SILVER AND COPPER FIXED-POINT CELLS USING STANDARD PLATINUM-PALLADIUM THERMOCOUPLE

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The objective of this EURAMET project (Bilateral comparison of Freezing point of Silver and Copper) in the field of thermometry is to compare realization of Freezing point of Silver (961.78 °C) and Freezing point of Copper (1084.62 °C) between the German national laboratory PTB and Croatian national laboratory HMI/FSB-LPM using long Platinum Palladium Thermocouple.

After the last comparison of the Cu cells (EUROMET 844) a crack of the quartz envelope of the HMI/FSB-LPM Copper cell occurred. As HMI/FSB-LPM in the process of accreditation for the calibration of calibration of Type S and pure metal thermocouple in fixed points, comparison against fixed points from PTB was needed.

Brand new Pt/Pd thermocouple has been selected and calibrated at PTB, than it was shipped to HMI/FSB-LPM where second set of measurement was made. Prior to every measurement thermocouple was annealed for 4 hours at 1000 °C.

Results of the intercomparison showed satisfactory performance cells of the HMI/FSB-LPM.

INTERCOMPARISON OF METEOROLOGICAL SERVICE CALIBRATION LABORATORIES IN THE SOUTH EASTERN EUROPE

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Intercomparison serves as a tool for comparison of measurement results performed by calibration laboratories in the relevant field of measurement. Intercomparison represents very effective means to demonstrate technical competence of the participant and is used as a technical base for accreditation. However, in the network of Meteorological Services calibration laboratories, intercomparisons among laboratories are still rare. Some of the laboratories are still not evaluating measurement uncertainty.

Environmental Agency of the Republic of Slovenia (EARS), serving in the frame of World Meteorological Organization (WMO) as a Regional Instrument Center (RIC), has organized a round robin intercomparison of calibration laboratories of Meteorological Services in the South-Eastern part of Europe using calibration kit. Calibration kit contains instruments for temperature, relative humidity and air pressure. Each involved laboratory had to calibrate set of instruments at defined calibration points, evaluate measurement uncertainty (if possible) and report the results.

EARS RIC has invited following National Hydrometeorological Services (NHMS) in the South Eastern part of Europe to take part in the intercomparison: Austria, Croatia, Hungary, Bosnia and Herzegovina, Serbia, Montenegro, Macedonia, Albania, Greece, Turkey, Romania, Bulgaria and Moldova. Additionally, one national laboratory - Laboratory of Metrology and Quality (MIRS/UL-FE/LMK- holder of a Slovenian national standard for temperature and relative humidity) was also invited to participate in intercomparison and data analysis. Results of MIRS/UL-FE/LMK were taken as reference value of intercomparison in the fields of temperature and humidity, while EARS results were taken as reference values in the field of pressure. All other data were compared against these results using En factor.

Fixed Points - M-C eutectics IV

Thursday
15:00 to 16:20
Emerald 1
Session Chairman: Yoshiro Yamada

THE EUTECTIC PT-C: DERIVING THE LIQUIDUS TEMPERATURE FROM FREEZING EXPERIMENTS BY EXTRAPOLATION TO ZERO FREEZING RATE

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Whereas usually melting is referred to when defining the eutectic transition temperature of high temperature eutectics here we study the feasibility of referring to freezing instead. To this end two cells Pt-5 and Pt-6 have been studied. Pt-5 is of the hybrid type, i.e. the ingot is protected by a sleeve wrapped around by two layers of graphite foil. The purity of the platinum is specified as 5N, crucible, sleeve and foil are of 5N purity. The cell is special in that the cavity is surrounded by a fragmented circular support of pure graphite protecting it against breakage by boyancy forces. Pt-6 is of the conventional sleeve type without foil or sheet around. The platinum for its ingot is of the same source as that of Pt-5 but partly from a different lot. In this study the effects of impurities will not be considered; it is just the liquidus temperature we are interested in.

Freezing and melting experiments have been done by varying the offset of the furnace temperature T_f with respect to the nominal eutectic temperature T_E during freezing: $(T_f-T_E)_{freeze}$ = -5, -10, -20, −40 K. Each freeze was followed by a melt at an offset $(T_f-T_E)_{melt}$ = +20 K. Stability tests over several days showed a good stability of freezing and melting curves around T_{max} and T_{infl}, respectively, for Pt-5, the duration of the melting curves remaining essentially unaffected. The stability of Pt-6 was worse however, the duration of the melting curve decreasing with time. Plots of T_{max} of the freezing curves and of the inflection temperature T_{infl} of the subsequent melting curves versus $(T_E-T_f)^{1/2}$, where (T_E-T_f) ¤ $(T_E-T_f)_{freeze}$, showed a linear behavior, the linearity for Pt-5 being however much better than that observed for Pt-6. The difference $T_{infl}(0)-T_{max}(0)$ amounted to about 5 mK for Pt-C and to about 20 mK for Pt-6. The difference $T_{infl}(0)_{Pt\text{-}5}$ $-T_{infl}(0)_{Pt\text{-}6}$ was about 100 mK. We think that part of the reason for the inferior behavior of Pt-6 is the absence of the temperature equalizing sheet, which for Pt-5 still resulted in a good temperature uniformity around the cell, even though the furnace-temperature uniformity was not optimum.

The superior behavior of Pt-5 triggered us starting the study, formulated in the title of the paper, not only for Pt-5 but also -to compare- for Pt-6, since it clearly shows the high potential of cells of type Pt-5. As a matter of fact cell Pt-5 will be at least duplicated to check for the repeatability of its quality. For both cells inflection temperatures T_{infl} will be converted to liquidus temperatures $T_{liq,melt}$ using the procedure reported earlier for Fe-C. Likewise maximum temperatures T_{max} will be converted to $T_{liq,freeze}$ following an analogue procedure, to be reported. Subsequently $T_{liq,\ freeze}(0)$ and $T_{liq,melt}(0)$ will be obtained by extrapolation of the respective plots against $(T_E\text{-}T_f)^{1/2}$ towards zero freezing rate and the difference $T_{liq,freeze}(0)$ $-T_{liq,\ melt}(0)$ determined together with its associated uncertainty.

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

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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

METAL(CARBIDE)-CARBON EUTECTIC HIGH TEMPERATURE FIXED-POINTS FOR DYNAMIC DIFFERENTIAL SCANNING CALORIMETRY

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Metal(Carbide)-Carbon (M(C)-C) eutectic fixed-points have been widely investigated for the application in thermometry and radiometry, expanding the range of the conventionally available high-temperature fixed-points presently used in the International Temperature Scale from the copper fixed-point at 1357.77 K to temperatures as high as 3500 K. However, up to now these fixed-points have only been used to improve the traceability of radiation thermometers and thermocouples using the conventional, large graphite crucibles containing a cavity as blackbody or as thermometer well for the contact thermocouple temperature sensor.

For the measurement of thermophysical properties above 1300 K, methods of thermal analysis such as the dynamic differential scanning calorimetry (DSC), the laser-flash technique or dilatometry will significantly benefit from the in-situ application of these M(C)-C eutectic fixedpoints for a traceable temperature measurement.

In this paper we present the development and thorough investigation of in-situ high-temperature M(C)-C fixed-points for the application in DSC set-ups for temperatures up to 2300 K. In contrast to the application of M(C)-C fixed-points in contact thermometry or radiation thermometry only very small amounts of M(C)-C materials – several ten mg – are needed for fixed-points in thermal analysis. In a first step the stability and reproducibility of these small fixed-points has been tested for different crucible materials. To economically fabricate such fixed points a special manufacture method has been developed.

Resistance Thermometry III

Thursday
15:00 to 16:20
Emerald 2
Session Chairman: Carlos Nieto de Castro

A NEW THERMOMETER FOR THE COPPER POINT

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The ITS-90 has Silver (961.78 °C) as the upper limited for Contact Thermometry.

During the 1980's when ITS-90 was being designed a lot of work was done by researchers such as John Evans, Dr. Nubbermier and others to develop a High Temperature Thermometer to work up to the Copper Point (1084.62 °C). Their efforts failed because of unexpected contamination problems and so the scale was restricted to the Silver Point.

Up to, and including the Silver Point a ¼ ohm High Temperature Standard Platinum Resistance Thermometer (HTSPRT) exists with sub-mK reproducibility. The problem is to extend the design a further 120 °C to the copper point.

The main hurdle is to find a reliable sheath material. Quartz glass has an annealing temperature of 1050 °C and leaks ions which contaminate the platinum coil.

Recrystalised alumina is widely used at high temperatures, and is impervious to ions except chromium. Unfortunately alumina is not completely gas tight. A sapphire sheath would be perfect but will not accept even mild thermal shocks. Of eight sapphire sheathed HTSPRTs made by this author all eight fractured.

One solution could be to put a +Ve air pressure inside an alumina sheathed SPRT so that the very slow flux was outward through the sheath.

The slow flux of air would also keep the platinum windings under oxidizing conditions (platinum loves oxygen hates reducing atmospheres).

This article presents results from this thermometer design at both the Ag and Cu point.

INCONSISTENCY OF INDUSTRIAL PLATINUM RESISTANCE THERMOMETERS IN THE RANGE OF 0 ºC TO 420 ºC

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Calibration of Industrial Platinum Resistance Thermometers (IPRT) by fixed points is recommended to improve its uncertainty compared with that obtained by comparison. IPRTs present a regular and well-behaved deviation at the calibration points indicated by the International Temperature Scale of 1990 (ITS-90). However, it is necessary to evaluate its inconsistency when these instruments measure interpolated temperatures. For that evaluation some of the ITS-90 defining fixed points can be used as well as some secondary ones. Inconsistency values are used to assess values for the uncertainty budget of the calibration.

For the IPRT calibration in fixed points, we used smaller cells to those of conventional size, because the sheath length in most of these thermometers is about 30 cm. In this way the heat leaks through sheath are reduced.

We report the calibration results of several IPRT in the 0 ºC to 420 ºC range. These IPRTs were, calibrated at the tin and zinc points, as stated in the ITS-90 text. To evaluate the inconsistency below the tin point, they were measured at the indium and gallium point; for the Sn to Zn interval, inconsistency was evaluated at the cadmium freezing point (321.069 ºC), which is a secondary point of the ITS-90.

THE INVESTIGATION OF THE STABILITY OF THE INDUSTRIAL RESISTANCE THERMOMETERS IN REAL CONDITION

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The international standard IEC 751 for industrial platinum resistance sensors (IPRT) describing the behavior of the thermometers for industrial use in terms of Callendar – van Dusen equation. It is know very little about long term stability of these sensors after their exposition to higher temperatures (around 400 °C).

This article will be focused on real sensors used in the industrial environment of the Czech Republic. There will be both types of IPRT investigated (thin film and wounded). It will be shown the influence of long term exposition on higher temperatures to the calibration of the sensors. There will be more than 200 sensors of different design and suppliers showed the trends of their calibration coefficients.

The last part will be focused to showing of the differences of the different calibration points and different calibration equation to the calibration results of the IPRT with special case of heat flow sensors.

OPTICAL TEMPERATURE MEASUREMENT OF GLOWING MICROCOMPONENTS

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The temperature of a glowing body can be determined from the shape of the radiation spectrum using Planck's radiation law. A greybody assumption is often made for the emissivity, i.e., the emissivity of the material is assumed to be spectrally constant. This assumption is inaccurate when measuring the temperature of structures fabricated on silicon, because the partial transparency of thin material layers produces interference effects.

In this work, we present temperature measurements and measurement methods for a miniature emitter, known as microbridge. Microbridges are miniature light sources integrated on silicon. They are brought up to incandescence with electrical current. Operating temperatures range from 700 °C up to the melting point of silicon (1400 °C). Microbridges are made from highly doped single-crystal silicon and have a thin protecting layer of silicon dioxide on all sides. Highly doped silicon exhibits complicated behavior near intrinsic temperatures, where the absorptivity, and therefore emissivity, changes rapidly.

We first measured the extinction coefficient of highly doped silicon at high temperatures: a piece of a silicon-on-insulator wafer was heated to several temperatures in a high-temperature furnace, and the emitted spectra were measured using a spectroradiometer with focusing optics. The optical behavior of the sample was modeled with Fresnel equations. The extinction coefficient of silicon was obtained from the model, because other optical properties, the dimensions, and the temperature of the structure were known.

In the measurement of the microbridge, the measurement area is 40 µm in diameter, as determined by the properties of the microscope optics. The dimensions of the microbridge are 400 × 25 × 4 μm^3. The emissivity model was adapted for the microbridge with the known extinction coefficient values, which allows the temperature to be determined from the measured spectrum. We have shown that with the device and methodology developed, we can now measure the temperatures of the microbridges in the temperature range 600–1200 °C with an uncertainty of 100 °C. The optical method does not require mechanical contact with the sample, preserving the thermal equilibrium.

CALIBRATION OF LOW-TEMPERATURE INFRARED RADIATION THERMOMETERS

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Infrared radiation thermometry for near room temperature is getting become popular in a lot of industries, e.g. food, medical, security, manufacturing. Calibration of infrared radiation thermometers has been also of increasing significance for NMIs and calibration laboratories. NMIJ established radiance temperature scale traceable to the ITS-90 based on variable temperature blackbodies and started calibration cervices for industrial blackbodies with direct comparison using an InSb radiation thermometer as a comparator[1]. In the presnt study standard facilities consisting of a standard blackbody and an infrared radiation thermometers has been constructed for calibration of indutrial infrared thermometers in the range from –30 °C to 100 °C.

Basic configuration of the blackbody is a conventional one. A blackbody cavity made of copper is immersed horizontally in a temperature-controlled stirred fluid-bath in which the temperature of the fluid is measured with a reference PRT traceable to the ITS-90. The shape of the cavity is cylindro-conical with a circular aperture of 60 mm in diameter. To prevent possible water condensation and ice buildup inside the cavity at the temperatures below ambient, a specially designed gas-purging unit using a porous metal cylinder has been installed in front of the aperture of the cavity. The purge unit is performing successfully and the blackbody cavity having 60 mm aperture can be operate in good condition without any dew or ice on the surface in the temperature down to –30 °C. The blackbody cavity is also equipped with a temperature-controlled large area (500 mm x 500 mm) metal plate coated with a high emissive paint to compensate broadened filed of views of industrial infrared thermometers.

Previously NMIJ developed DC-operated infrared thermometers having the highest level of resolution using liquid nitrogen cooled detectors for research applications. In this study an infrared thermometer has been newly designed based on an industrial thermometer manufactured by CHINO for practical uses in calibration laboratories and industries. An optical system comprising a combination of a specially designed pyroelectric detector unit and a front chopping mechanism improves a short-term stability and a drift of offset signal of the infrared thermometer. Temperature scale of the infrared thermometer can be calibrated against the blackbody cavity with uncertainties better than 0.12 °C (k = 2) for the temperature range from –30 °C to 100 °C. This infrared thermometer is also a potential instrument for inter-laboratory comparisons in the low temperature range.

[1] J. Ishii and A. Ono, Temperature, vol. 7 pp. 657 – 662, AIP (2003)

DEVELOPMENT OF HIGH TEMPERATURE BLACKBODIES FOR RADIATION THERMOMETRY

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High temperature blackbodies are developed at VNIIOFI for more than twenty years. The pyrolytic graphite blackbodies of BB3200/3500 series are the most known ones and used now not only at VNIIOFI but also at several other metrological institutes all over the world. The main applications of them are radiometric such as spectral irradiance and spectral radiance. In the thermometry community BB3200 and BB3500 are used as furnaces for high temperature fixed points applications only. Due to their high emissivity, uniformity and stability they could also serve as high quality blackbody sources for calibration of radiation thermometers.

Two new blackbodies were developed and investigated on the base of experience with the BB3200/3500. The first is a graphite blackbody developed specially for pyrometer calibrations. It has a relatively large cavity with 40 mm opening and temperature range from 800 to 2000 °C. The second one is the smallest version of a pyrolytic graphite blackbody. Its cylindrical cavity is formed by set of pyrographite rings and has inner diameter of 20 mm, which could be increased up to 25 mm optionally. This blackbody has the high working temperature, up to 3000 °C and high emissivity, similar to BB3200/3500, but is less power-consuming that makes it simpler to operate.

The paper will describe the design of new blackbodies and results of their investigation.

CALCULATION OF THE TEMPERATURE DROP FOR HIGH TEMPERATURE FIXED POINTS FOR DIFFERENT FURNACE CONDITIONS

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High temperature fixed points (HTFPs) based on eutectic and peritectic reactions of metals and carbon are likely to become, in the near term, reference standards at high temperatures. Typically these HTFPs, for radiation thermometry applications, are formed of a graphite crucible, with a reentrant well, an included 120° cone and a nominal aperture of 3 mm. It is important to quantify the temperature-drop at the back-wall of the cavity, and to understand the influence of the furnace and furnace conditions on this drop.

We estimate the temperature drop for the following HTFPs, Co-C (1324 °C), Pd-C (1492 °C), Pt-C (1738 °C), Re-C (2474 °C) and WC-C (2749 °C), when realized in a Thermogage furnace. The following conditions are modeled:

- Ideal conditions: HTFP in uniform furnace at the HTFP temperature.
- Intermediate conditions: HTFP in furnace that has a sine temperature profile from the centre of the furnace to its aperture.
- Extreme conditions: Furnace has a linear gradient from the centre of the furnace to its aperture.
- Reference conditions: The bare HTFP crucible i.e. not in a furnace. This forms the upper bound of the temperature drop value.

In addition the influence of the thickness of the graphite back-wall on the value of the temperature drop is estimated for two HTFPs, Co-C and Re-C in the reference and ideal conditions. This parameter is often indeterminate due to erosion of the back-wall during filling. The calculations of this effect here enable boundaries to be placed on the effect of back-wall thickness variation on temperature drop.

In this paper we describe how these calculations were performed and a summary given of the calculations. Finally a discussion of the implications of these results for the use of HTFPs as temperature references is given.

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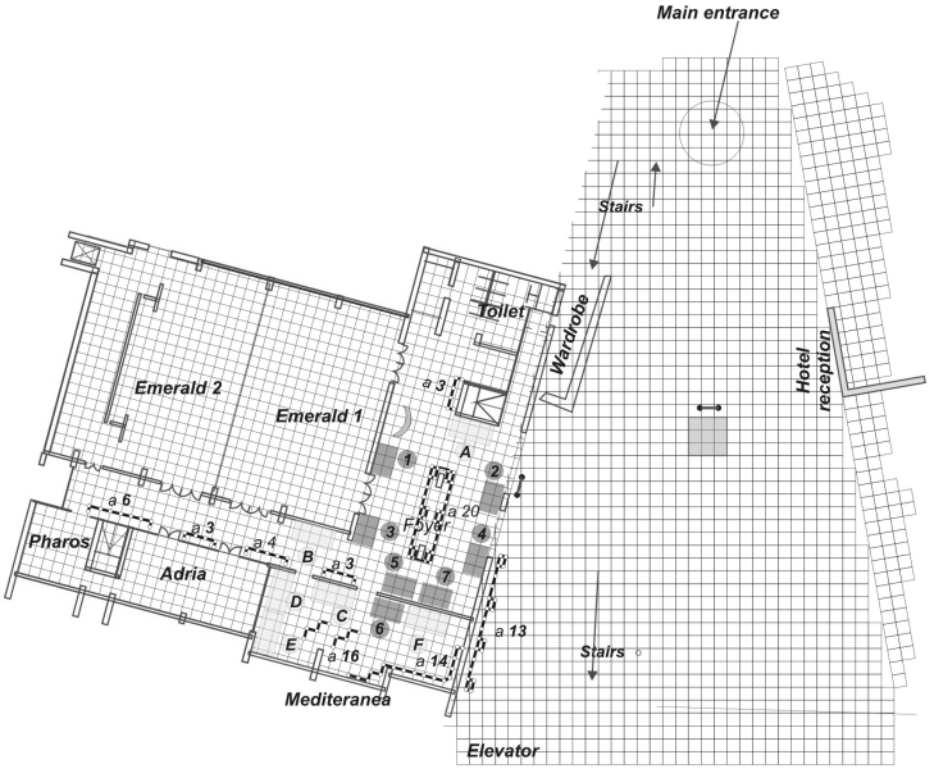

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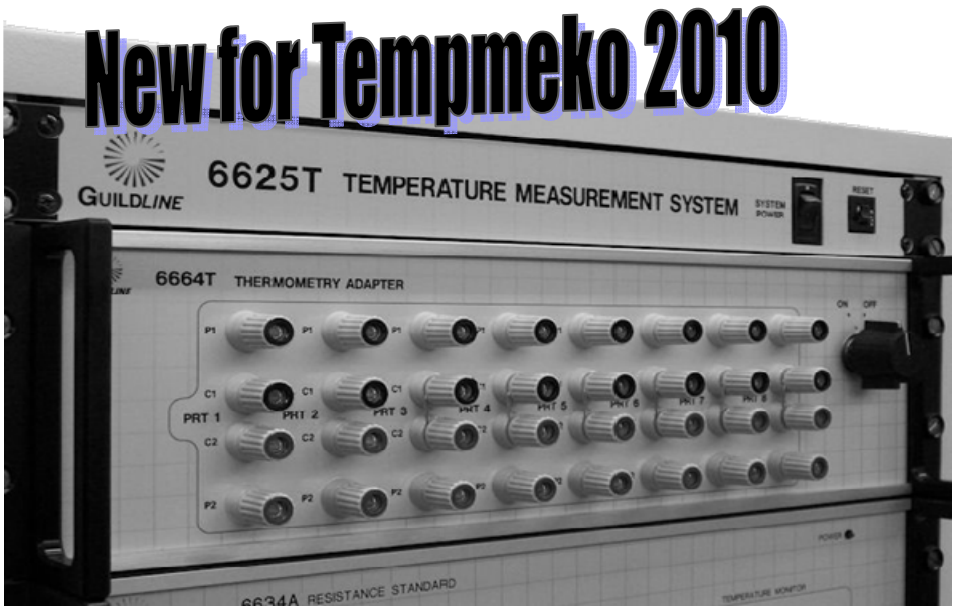

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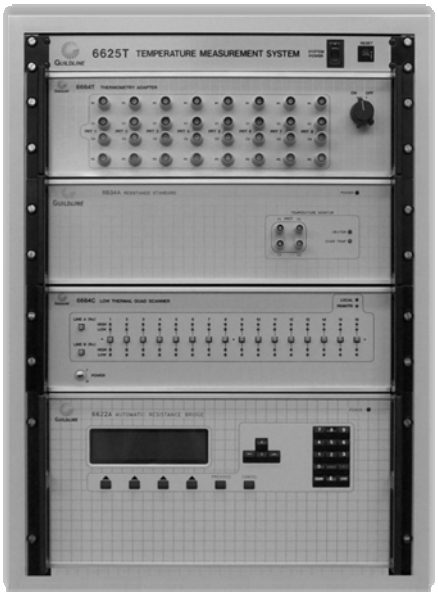

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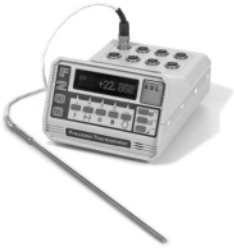

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