2020
Tingting Zhang, Xiaofeng Lu, Jianping Sun and Jiang Pan Calibration of a W/Re thermocouple using Pd-C, Pt-C and Ru-C eutectic cells at NIM 2020 Metrologia 57 (in press)
Abstract: In the paper, an in-house W/Re thermocouple was calibrated at the eutectic fixed points of Pd-C(1492 °C), Pt-C(1738 °C) and Ru-C(1953 °C). To correct the temperature error caused by the thermal conduction of the thermocouple sheath, a linear extrapolation method was developed to determine the emf of the thermocouple based on the melting and freezing values at the eutectic fixed points realized with different offset furnace temperatures. In a simpler approach, the average of the melting and freezing values was also used. The results showed that the temperatures corresponding to the emfs derived from the linear extrapolation and the average method agreed (1-2) °C with the standard reference values of type-C thermocouple in the temperature range studied

Abstract: The determination of the differences (T–T90) between the thermodynamic temperature T and the international temperature scale of 1990 (ITS-90) T90, is important for an evaluation of the approximation to T by T90. Such evaluations are necessary for the potential revision of ITS-90. A number of efforts have been devoted to the determination of (T–T90) by the acoustic gas thermometry (AGT) using spherical or quasi-spherical resonators. We report in this paper a new study in the temperature range from 234 K to 303 K using AGT in argon with a cylindrical resonator. Piezo-electric acoustic transducers were used to measure the acoustic resonant frequencies. The resonant frequencies of the transverse magnetic microwave modes of the cavity were measured using straight probe antennas. This work further illustrates the high performance of the microwave resonant procedure to measure the thermal expansion of a cylindrical cavity at different temperatures and pressures. The (T–T90) measurements, with standard uncertainties in the range from 0.5 mK to 0.8 mK, agree well with the existing data from AGT with spherical or quasi-spherical resonators. We believe that cylindrical acoustic gas thermometry, with an uncertainty comparable with that using spherical or quasi-spherical resonators but simpler mechanical assembly, has the potential for accurate measurements of (T–T90) at higher temperatures.

Abstract: A high-temperature tungsten-rhenium (W-Re) thermocouple is commonly used at temperatures up to 2000 °C for the high melting temperature of the thermoelement materials. When exposed to high temperatures, a thermocouple can show significant thermoelectric drift, which increases measurement uncertainty. The
uncontrolled drift may come from the change in the crystallographic structure, oxidization of the thermoelement materials, interaction between the thermoelement materials and insulator materials and some other unknown reasons. Therefore, periodic recalibration of the thermocouple should be performed; however, sometimes it is not possible to remove the sensor out of the process, especially in some special fields, such as a nuclear power plant. Self-validation methods for thermocouples provide a solution to avoid this drawback. In this paper, miniature eutectic fixed-point cells are presented for self-validation of W-Re thermocouples. To prevent the breakage of the graphite crucible, these cylindrical miniature fixed-point cells contain several small independent crucibles and one thermocouple well, which increase the robustness of the miniature cell. The melting temperature was assigned by a radiation thermometer traced to the primary radiation standard at NIM. The effect of the temperature offset and temperature ramping rate on the melting temperature were checked. The performance of the miniature fixed-point cells and type C thermocouple, including characterization of the stability and repeatability, is presented.

2019 Jifeng Qu, S. P. Benz, Horst Rogalla, Weston Tew, D. Rod White, Kunli Zhou. Johnson noise thermometry. Measurement Science and Technology, 2019, 30: 112001. Abstract: Johnson noise thermometers infer thermodynamic temperature from measurements of the thermally-induced current fluctuations that occur in all electrical conductors. This paper reviews the status of Johnson noise thermometry and its prospects for both metrological measurements and for practical applications in industry. The review begins with a brief description of the foundations and principles of Johnson noise thermometry before outlining the many different techniques and technological breakthroughs that have enabled the application of Johnson noise thermometry to high-accuracy, cryogenic, and industrial thermometry. Finally, the future of noise thermometry is considered. As the only purely electronic approach to thermodynamic temperature measurement, Johnson noise thermometry has appeal for metrological applications at temperatures ranging from below 1 mK up to 800 K. With the rapid advances in digital technologies, there are also expectations that noise thermometry will become a practical option for some industrial applications, perhaps reaching temperatures above 2000 K.

Wei Zheng and Xiaofeng Lu Temperature assignment of a Co–C eutectic fixed-point cell for thermocouple calibration 2019 Measurement Science and Technology 31: 1-6 The Co–C eutectic fixed point has been popular for thermocouple calibration since 2010. In this study, the ITS-90 temperature of a Co–C eutectic fixed-point cell intended for thermocouple calibration of noble thermocouples was measured using a radiation thermometer and a group of Pt/Pd and PtRh10%-Pt thermocouples. Satisfactory consistency was observed for measurements using both methods.

Xu-yao Song, Qing-duo Duanmu, Wei Dong, Zhi-bin Li, "Piecewise linear calibration

The spectral responsivity of Fourier Transform Infrared Spectrometer (FTIR) measurement system of high temperature blackbody infrared radiation characteristics is calibrated via ThermoGage HT9500 high temperature reference blackbody furnace from National Institute of Metrology, China (NIM). A calculation model of the spectral responsivity calibration of FTIR measurement system is established. The infrared spectrum of the blackbody radiation source is measured in the temperature range from 1273 K and 1973 K on the wavelength range from 1 μm to 14 μm. Calibration is carried out within the temperature range from 1373 K to 1873 K on the wavelength range between 1 μm and 13 μm. The infrared spectral radiation characteristics of ThermoGage HT9500 high temperature reference blackbody furnace are represented. The results indicated that the method of piecewise linear calibration was practicable. The measured infrared spectrum in the temperature range from 1373 K to 1873 K on the wavelength range between 1 μm and 13 μm was compared with the calculation which showed the signal divergence less than 1%. And the calculated temperature obtained by inverse calculation in this temperature region was compared with the actual temperature which showed the temperature divergence less than 0.45%.


Abstract: Taking Ga-In-Sn ternary alloy as the research object, a mini Ga-In-Sn eutectic cell which can be used for on-site and on-line calibration was developed, and the effect of three-ratio for the Ga-In-Sn on phase transition temperature and temperature plateau were investigated. The results show that the plateaus of three-ratio Ga-In-Sn realization last about 1.2 h to 2 h, the reproducibility is less than 4.5 mK and combined expanded uncertainty is 9.3 mK(k=2). The average value of the phase transition temperature is 10.748 ℃ and is not affected by the ratio of Ga-In-Sn ternary alloy. Changing the melt cooling rate can change the supercooling of the mini eutectic fixed point.


Abstract: Two types of high-temperature fixed points (HTFPs) were evaluated by VNIIOFI, NIM, KRISS and NMIJ. WC-C peritectic point cells manufactured independently in different National Metrology Institutes (NMIs) were compared for the first time, and agreement of the melting temperatures for three high-quality cells at the level of 0.05 degrees C was demonstrated. This confirms, in conjunction with previous results that verified their long-term stability, high repeatability and reproducibility, the high potential of the WC-C cell as the highest-temperature reference point for radiation thermometry.
The performance of the Ru-C eutectic fixed point was verified by evaluating Ru-C cells manufactured from 99.999% purity Ru materials from different manufacturers. These cells used considerably purer materials than those used in previous studies. New Ru-C cells were constructed from the best performing materials, and the t(90) values of these cells were measured at the four NMIs on their locally-realized ITS-90 scale. The values agreed within 0.25 degrees C among the four NMIs, and the t(90) value of Ru-C was determined to be 1953.64 degrees C with an expanded uncertainty of 0.20 degrees C. Both results confirmed that performance of WC-C and of Ru-C is comparable to or exceeds that of the HTFPs evaluated in the Consultative Committee for Thermometry's HTFP project, and both fixed points have capability as a reference fixed points to be included in the future MeP-K in terms of thermodynamic temperature T.

Abstract: The IR-RST standard radiation thermometers manufactured by CHINO Company, Limited, Japan, which cannot display the temperature in real-time because the display unit hadn’t been designed. Therefore, an electrical measurement display device was designed which can measure the voltage, display the temperature and supply 24 V DC voltage for this kind of infrared radiation thermometer. Calibrated against high-level voltage power source and a voltmeter, the correction coefficients of the display instrument were got, the stability was evaluated as well, the maximum relative error of voltage measurement can be achieved with in 0.03 %. Assembled with a 0.65 μm IR-RST radiation thermometer, it was calibrated against with a TG HT-9500 type hih-temperture furnace as the source, the accuracy was better than 0.03 % confirmed by the experimental results and the extended uncertainty of the calibration system was 0.62℃ at 1 300℃.

Abstract: The design of the acoustic waveguides is key to the signal-to-noise ratio of the acoustic resonances. Acoustic waveguides with larger inner diameter and shorter length is good for the signal transfer, but it will cause larger perturbation to the acoustic resonator. Here we propose a new kind of acoustic waveguides with variable dimensions to reduce energy loss along the waveguides as well as the perturbation from the ducts. We have developed a model of the energy loss and the perturbation for the new design and compared the sound attenuation and the perturbation to the acoustic resonance frequencies and half-widths for different dimensions of the waveguides. The optimized design of the acoustic waveguides can reduce the perturbation to the acoustic resonance frequency to below 3×10⁻⁵ for the first longitudinal non-degenerate mode of a cylindrical resonator with an inner length of 80 mm. This research contributes to the further study of high-temperature acoustic gas thermometry.

**Abstract:**
The refractive index of monatomic gases such as argon is an important parameter for the verification of the ab initio calculations based on quantum mechanics. The refractive index of argon from 234 K to 303 K and 0 kPa to 750 kPa was measured accurately using a cylindrical microwave resonator. Microwave resonance frequencies of four TM modes at different pressures in a cylindrical cavity were measured. After the correction of the non-ideal factors, the refractive index of argon was obtained by the comparison of the microwave resonance frequencies in vacuum and in gases. The uncertainty of microwave resonance frequency in the cylindrical cavity is $2 \times 10^{-8}$, and the inconsistence between the argon refractive index from four modes is less than $1 \times 10^{-6}$. The first dielectric virial coefficients of argon are obtained by calculating the refractive index of argon, and the results show a good agreement with the published results. The refractive index measurement of other gases can be carried out in the future using the experimental apparatus.


**Abstract**
The developed process of the gallium-based alloy miniature fixed points for on-orbit calibration and the quasi-adiabatic vacuum measurement system are introduced. Combined with cavity blackbody and non-proximal mounted temperature sensor, the repeatability of Ga-Sn and Ga-Zn alloy fixed points are measured, which was better than 2 mK during melting process. For Ga, the long-term stability is 2.1 mK. The temperature sensor at the bottom of cavity blackbody was indexed by the continuous melting plateau value measured by temperature sensor during gallium and two gallium-based alloy fixed points melting process in a specific thermal environment. The difference between the calibration result and the routine calibration method in the laboratory is less than 2 mK. The results are also shown that in the thermal environment conditions remaining unchanged, as the phase transformation time increases, the phase transition temperature is closer to the melting temperature of the phase change material, that is, the temperature difference between the fixed point and the temperature sensor hole is smaller. Meanwhile, for Ga-Sn and Ga-Zn, there is a linear relationship between the phase transition melting temperature and the single-point calibration temperature at zero power is 20.352 ℃ and 25.187 ℃.

2018
Abstract: High-temperature fixed points (HTFPs) have been thoroughly investigated, and the performance of variable temperature blackbodies (VTBB) has also improved rapidly. These two are beginning to be used in the calibration of pyrometers; however, tungsten strip lamps (STSL) still play a role in the dissemination of the high temperature scale in China. International Temperature Scale of 1990 values of HTFPs and the lamps were assigned on a primary standard pyrometer (PSP) and were traced to the primary standard of the high-temperature scale at the National Institute of Metrology. In this paper, two pyrometers calibrated by using extrapolation and interpolation methods are reported. The values of the calibration were compared against the STSL values and the PSP values on HTBB, and their uncertainties are calculated as well. Because the stability of the HTFPs was better than that of the lamps, the calibration chains based on the lamps are starting to be replaced by HTFPs and VTBBs in China.


Abstract: As infrared remote sensors are very important parts of Earth observation satellites, they must be calibrated based on the radiance temperature of a blackbody in a vacuum chamber prior to launch. The uncertainty of such temperature is thus an essential component of the sensors’ uncertainty. This paper describes the vacuum radiance-temperature standard facility (VRTSF) at the National Institute of Metrology of China, which will serve to calibrate infrared remote sensors on Chinese meteorological satellites. The VRTSF can be used to calibrate vacuum blackbody radiance temperature, including those used to calibrate infrared remote sensors. The components of the VRTSF are described in this paper, including the VMTBB, the LNBB, the FTIR spectrometer, the reduced-background optical system, the vacuum chamber used to calibrate customers’ blackbody, the vacuum-pumping system and the liquid nitrogen-support system. The experimental methods and results are expounded. The uncertainty of the radiance temperature of VMTBB is 0.026 °C at 30 °C over 10 μm.


Abstract: Based on the calibration requirements of vacuum low background aerospace infrared remote sensing radiance temperature, a high-precision vacuum blackbody (H500 type) is developed for the temperature range from −93 °C to +220 °C at the National Institute of Metrology, China. In this paper, the structure and the temperature control system of H500 are introduced, and its performance, such as heating rate and stabilization of temperature control, is tested under the vacuum and low-background condition (liquid-nitrogen-cooled shroud). At room temperature and atmospheric environment, the major technical parameters of this blackbody, such as emissivity and uniformity, are measured. The measurement principle of blackbody emissivity is based
on the control of surrounding radiation. Temperature uniformity at the cavity bottom is measured using a standard infrared radiation thermometer. When the heating rate is 1 °C min$^{-1}$, the time required for the temperature to stabilize is less than 50 min, and within 10 min, the variation in temperature is less than 0.01 °C. The emissivity value of the blackbody is higher than 0.996. Temperature uniformity at the bottom of the blackbody cavity is less than 0.03 °C. The uncertainty is less than 0.1 °C ($k=2$) over the temperature range from −93 °C to +67 °C.


Abstract A method of controlling the surrounding radiation, proposed to measure the emissivity of the sample with high accuracy, is introduced. Two disks at different temperatures are moved alternately in front of the sample for controlling the surrounding radiation of the sample. The emissivity of the sample is obtained from the relationship between the measured value of the radiation thermometer and the radiation from the sample. There are three samples, including the Japansensor JSC-3, Tempil Pyromark1200, and NEXTEL Velvet Coating 811-21 ultra-black coating, measured by this method. The uncertainty contributions of this method are analyzed, and the uncertainty of the emissivity measurement method is 0.54 % ($k=1$). The surface microstructure of the coatings is measured by scanning electron microscopy, and the relationship between emissivity and the surface properties is discussed.


Abstract: Johnson noise thermometry is of considerable interest at present due to the planned redefinition of the kelvin in 2019, and several determinations of the Boltzmann constant have recently been published in support of the redefinition. To determine the Boltzmann constant by noise thermometry, the thermal noise from a sensing resistor at the triple point of water is compared to a pseudo-random noise with a calculable power spectral density traceable to quantum electrical standards. In all the measurements to date, the two dominant sources of measurement uncertainty are strongly influenced by a single factor: the frequency-response mismatch between the sets of leads connecting the thermometer to the two noise sources. In the most recent determination at the National Institute of Metrology, China, substantial changes were made to the connecting leads to reduce the mismatch effects. The aims of this paper are, firstly, to describe and explain the rationale for the changes, and secondly, to better understand the effects of the least-squares fits and the bias-variance compromise in the analysis of measurements affected by the mismatch effects. While significant improvements can be made to the connecting leads to lessen the effects of the frequency-response mismatch, the efforts are unlikely to be rewarded by a significant increase in bandwidth or a significant reduction in uncertainty.

The tungsten–carbon (WC) peritectic, with a liquidus temperature near to 3020 K, shows promise as a high-temperature reference fixed point. It appears that the WC phase is a discrete phase with full stoichiometry of the components W and C, in which impurities cannot be dissolved. In this paper we describe the use of Thermo-Calc software to derive the liquidus and solidus temperatures versus the impurity concentration for the impurities V and Si. It is found that the liquidus is univariant, whereas the solidus is invariant. Additionally, the transition curves between the solidus and the liquidus are modeled. The liquid fraction at the liquidus point is found to be less than 1; in contrast, it is unity for metal–carbon eutectics. Measurements are presented for a series of melting and freezing curves for two WC fixed-point cells. The Gibbs phase rule requires segregation of a fourth compound phase, involving the impurity in question, at the solidus point. The observations confirm that during supercooling, impurities are trapped in compound phases consisting of the impurity together with W and/or C, encapsulating the impurity, and resulting in a flat freezing plateau characteristic of the pure system. Finally, an expression for the melting temperature, as a function of the liquid fraction, is derived, spanning the temperature range from an initial melting temperature to the liquidus temperature. Within this range just free impurities are involved.

J Fischer et al The Boltzmann project 2018 Metrologia 55: R1-R20

Abstract The International Committee for Weights and Measures (CIPM), at its meeting in October 2017, followed the recommendation of the Consultative Committee for Units (CCU) on the redefinition of the kilogram, ampere, kelvin and mole. For the redefinition of the kelvin, the Boltzmann constant will be fixed with the numerical value 1.380 649×10^{-23} J K^{-1}. The relative standard uncertainty to be transferred to the thermodynamic temperature value of the triple point of water will be 3.7×10^{-7}, corresponding to an uncertainty in temperature of 0.10 mK, sufficiently low for all practical purposes. With the redefinition of the kelvin, the broad research activities of the temperature community on the determination of the Boltzmann constant have been very successfully completed. In the following, a review of the determinations of the Boltzmann constant k, important for the new definition of the kelvin and performed in the last decade, is given.


Abstract: Using the microwave resonant method, the thermodynamic temperature is measured in the temperature range from 253 K to 303 K based on the gas refractive index primary thermometry by using the properties for argon from “ab initio” quantum mechanics calculation theory and experimental results and the microwave resonant method. Four transverse magnetic microwave resonance frequencies were measured.
to obtain the refractive index in argon at 700 kPa, the consistence of the refractive index from different microwave modes was better than 1×10⁻⁸. The thermodynamic temperature then was determined by combining the refractive index measurement results and the virial equation of state for argon. The uncertainty of the difference of the thermodynamic temperature \( T \) and the ITS-90 international temperature \( T_{90} \) was evaluated to be 11.6 mK. and the results agreed with the Consultative Committee for Thermometry (CCT) recommendation values well. The uncertainty from this method will be decreased with the development of the theoretical calculation for argon and the improvement of the pressure measurement in the next future.

2017


Abstract: We report a new determination of the Boltzmann constant \( k_B \) using a cylindrical acoustic gas thermometer. We determined the length of the copper cavity from measurements of its microwave resonance frequencies. This contrasts with our previous work (Zhang et al 2011 Int. J. Thermophys. 32 1297, Lin et al 2013 Metrologia 50 417, Feng et al 2015 Metrologia 52 S343) that determined the length of a different cavity using two-color optical interferometry. In this new study, the half-widths of the acoustic resonances are closer to their theoretical values than in our previous work. Despite significant changes in resonator design and the way in which the cylinder length is determined, the value of \( k_B \) is substantially unchanged. We combined this result with our four previous results to calculate a global weighted mean of our \( k_B \) determinations. The calculation follows CODATA’s method (Mohr and Taylor 2000 Rev. Mod. Phys. 72 351) for obtaining the weighted mean value of \( k_B \) that accounts for the correlations among the measured quantities in this work and in our four previous determinations of \( k_B \). The weighted mean \( \hat{k}_B \) is 1.380 6484(28) \times 10⁻²³ J K⁻¹ with the relative standard uncertainty of 2.0 \times 10⁻⁶. The corresponding value of the universal gas constant is 8.314 459(17) J K⁻¹ mol⁻¹ with the relative standard uncertainty of 2.0 \times 10⁻⁶.


Abstract: Recent measurements using acoustic gas thermometry have determined the value of the Boltzmann constant, \( k \), with a relative uncertainty less than 1×10⁻⁶. These results have been supported by a measurement with a relative uncertainty of 1.9×10⁻⁶ made with dielectric-constant gas thermometry. Together, the measurements meet the requirements of the International Committee for Weights and Measures and enable them to proceed with the redefinition of the kelvin in 2018. In further support, we provide a new determination of \( k \) using a purely electronic approach, Johnson noise thermometry, in which the thermal noise power generated by a sensing resistor immersed in a triple-point-of-water cell is compared to the noise power of a quantum-
accurate pseudo-random noise waveform of nominally equal noise power. The experimental setup differs from that of the 2015 determination in several respects: a 100 ohm resistor is used as the thermal noise source, identical thin coaxial cables made of solid beryllium-copper conductors and foam dielectrics are used to connect the thermal and quantum-accurate noise sources to the correlator so as to minimize the temperature and frequency sensitivity of the impedances in the connecting leads, and no trimming capacitors or inductors are inserted into the connecting leads. The combination of reduced uncertainty due to spectral mismatches in the connecting leads and reduced statistical uncertainty due to a longer integration period of 100 d results in an improved determination of $k = 1.3806497(37) \times 10^{-23}$ J K$^{-1}$ with a relative standard uncertainty of $2.7\times10^{-6}$ and a relative offset of $0.89\times10^{-6}$ from the CODATA 2014 recommended value. The most significant terms in the uncertainty budget, the statistical uncertainty and the spectral-mismatch uncertainty, are uncorrelated with the corresponding uncertainties in the 2015 measurements.


Abstract: In the electronic measurement of the Boltzmann constant based on Johnson noise thermometry, the ratio of the power spectral densities of thermal noise across a resistor at the triple point of water, and pseudo-random noise synthetically generated by a quantum-accurate voltage-noise source is constant to within 1 part in a billion for frequencies up to 1 GHz. Given knowledge of this ratio, and the values of other parameters that are known or measured, one can determine the Boltzmann constant. Due, in part, to mismatch between transmission lines, the experimental ratio spectrum varies with frequency. We model this spectrum as an even polynomial function of frequency where the constant term in the polynomial determines the Boltzmann constant. When determining this constant (offset) from experimental data, the assumed complexity of the ratio spectrum model and the maximum frequency analyzed (fitting bandwidth) dramatically affects results. Here, we select the complexity of the model by cross-validation—a data-driven statistical learning method. For each of many fitting bandwidths, we determine the component of uncertainty of the offset term that accounts for random and systematic effects associated with imperfect knowledge of model complexity. We select the fitting bandwidth that minimizes this uncertainty. In the most recent measurement of the Boltzmann constant, results were determined, in part, by application of an earlier version of the method described here. Here, we extend the earlier analysis by considering a broader range of fitting bandwidths and quantify an additional component of uncertainty that accounts for imperfect performance of our fitting bandwidth selection method. For idealized simulated data with additive noise similar to experimental data, our method correctly selects the true complexity of the ratio spectrum model for all cases considered. A new analysis of data from the recent experiment yields evidence for a temporal trend in the offset parameters.
X J Feng, J T Zhang, M R Moldover, I Yang, M D Plimmer and H Lin Determination of the molar mass of argon from high-precision acoustic comparisons 2017 Metrologia 54: 339–347

Abstract
This article describes the accurate determination of the molar mass M of a sample of argon gas used for the determination of the Boltzmann constant. The method of one of the authors (Moldover et al 1988 J. Res. Natl. Bur. Stand. 93 85–144) uses the ratio of the square speed of sound in the gas under analysis and in a reference sample of known molar mass. A sample of argon that was isotopically-enriched in 40Ar was used as the reference, whose unreactive impurities had been independently measured. The results for three gas samples are in good agreement with determinations by gravimetric mass spectrometry; (\frac{\text{Macoustic}}{\text{Mmass-spec}} - 1) = (-0.31 \pm 0.69) \times 10^{-6}, where the indicated uncertainty is one standard deviation that does not account for the uncertainties from the acoustic and mass-spectroscopy references.


Abstract
In the application of acoustic gas thermometry to determine the Boltzmann constant and thermodynamic temperatures using resonant cavities, the internal dimensions or the thermal expansion of the cavity have to be known accurately. For this purpose, measurement of the microwave resonances has proved to be an accurate and convenient experimental technique for dimensional measurement of acoustic resonators. We report measurements of the length and longitudinal thermal expansion of a prototype cylindrical cavity made of oxygen-free copper. We studied four non-degenerate transverse magnetic modes for three isotherms at 243, 258 and 273 K. Two procedures were investigated for calculating the length and longitudinal thermal expansion of the cavity at the temperatures examined. The results from both methods agree well. The relative standard uncertainties for the measurements of length and longitudinal thermal expansion are less than 0.47×10^{-6} and 0.04×10^{-6}, respectively, from 243 K to 273 K. The low uncertainty achieved here provides confidence to pursue a determination of the Boltzmann constant and thermodynamic temperature with a cylindrical cavity and microwave techniques.


Abstract: The knowledge of the liquidus slope of impurities in fixed-point metal defined by the International Temperature Scale of 1990 is important for the estimation of uncertainties and correction of fixed point with the sum of individual estimates method. Great attentions are paid to the effect of ultra-trace impurities on the freezing point of zinc in the National Institute of Metrology. In the present work, the liquidus slopes of
Ga-Zn, Ge-Zn were measured with the slim fixed-point cell developed through the doping experiments, and the temperature characteristics of the phase diagram of Fe-Zn were furthermore investigated. A quasi-adiabatic Zn fixed-point cell was developed with the thermometer well surrounded by the crucible with the pure metal, and the temperature uniformity of less than 20 mK in the region where the metal is located was obtained. The previous doping experiment of Pb-Zn with slim fixed point cell was checked with quasi-adiabatic Zn fixed-point cell, and the result supports the previous liquidus slope measured with the traditional fixed-point realization.


Abstract: Onsite thermometer calibration with temperature scale transfer technology based on fixed points can effectively improve the level of industrial temperature measurement and calibration. The present work performs an onsite calibration of a precision industrial platinum resistance thermometer near room temperature. The calibration is based on a series of small-size eutectic points, including Ga-In (15.7 degrees C), Ga-Sn (20.5 degrees C), Ga-Zn (25.2 degrees C), and a Ga fixed point (29.7 degrees C), developed in a portable multi-point automatic realization apparatus. The temperature plateaus of the Ga-In, Ga-Sn, and Ga-Zn eutectic points and the Ga fixed point last for longer than 2 h, and their reproducibility was better than 5 mK. The device is suitable for calibrating non-detachable temperature sensors in advanced environmental laboratories and industrial fields.


Abstract: Tungsten-rhenium thermocouples (type C thermocouples) are used to measure temperatures higher than 1500 degrees C under protective, inert, or vacuum conditions in a wide range of industries, such as metallurgy, power generation, and aerospace. Generally, the measurement uncertainty of a new tungsten-rhenium thermocouple is about 1%(20 degrees C at 2000 degrees C), and a significant drift is always observed above 1200 degrees C. Recently, the National Institute of Metrology, China, has spent great efforts to calibrate tungsten-rhenium thermocouples with high-temperature fixed points of up to 2000 degrees C. In the present work, three tungsten-rhenium thermocouples made by two manufacturers were calibrated at the Pt-C eutectic fixed point (1738 degrees C) and their stability was investigated. A linear fitting and extrapolation method was developed to determine the melting and freezing temperatures of the Pt-C eutectic fixed point for avoiding the effect of thermal resistance caused by the sheath and protection tube. The results show that the repeatability of the calibration is better than 0.9 degrees C from the melting curve of the Pt-C fixed point and better than 1.2 degrees C from the freezing curve of the Pt-C fixed point, and a good agreement was obtained for the calibration with the melting and
freezing temperature plateau through the linear fitting and extrapolation method. The calibration uncertainty of the thermocouples at the Pt-C eutectic fixed point was 3.1 degrees C ($k = 2$).


Abstract Miniature Ga and Ga–In alloy fixed points as temperature standards are developed at National Institute of Metrology (NIM), China for the in situ calibration of temperature sensors. A quasi-adiabatic vacuum measurement system is constructed to study the phase change plateaus of the fixed points. The system comprises a high-stability bath, a quasi-adiabatic vacuum chamber and a temperature control and measurement system. The melting plateau of the Ga fixed point is longer than 2 h at 0.008W. The standard deviation of the melting temperature of the Ga and Ga–In alloy fixed points is better than 2 mK. The results suggest that the melting temperature of the Ga or Ga–In alloy fixed points is linearly related with the heating power.


In high temperature fixed point research the so called furnace effect is where a larger furnace (or more properly high thermal inertia) appears to yield a higher temperature than a smaller furnace. In this paper we investigate the underlying cause of the furnace effect experimentally and give general arguments as to its origin.

We use three different furnaces to examine the melting behavior of a Co-C high temperature fixed point blackbody, especially as regards determining the point of inflection for melting where a clear furnace effect of a few 100 mK is observed.

We use general arguments, based on the physics of the situation to suggest that the origin of the furnace effect is the thermal response of the fixed point ingot to the way it is melted, a fast melt leading to a lower temperature and a slower melt to a higher temperature.


Abstract: Impurities represent the most significant source of uncertainty in most metal fixed points used for the realization of the International Temperature Scale of 1990 (ITS-90). There are a number of different methods for quantifying the effect of impurities on the freezing temperature of ITS-90 fixed points, many of which rely on an accurate knowledge of the liquidus slope in the limit of low concentration. A key method of determining the liquidus slope is to measure the freezing temperature of a fixed-point material as it is progressively doped with a known amount of impurity. Recently, a series of measurements of the freezing and melting temperature of ‘slim’ Zn fixed-point cells doped with Ag, Fe, Ni, and Pb were presented. Here, additional
measurements of the Zn-X system are presented using Ga as a dopant, and the data (Zn-Ag, Zn-Fe, Zn-Ni, Zn-Pb, and Zn-Ga) have been re-analyzed to demonstrate the use of a fitting method based on Scheil solidification which is applied to both melting and freezing curves. In addition, the utility of the Sum of Individual Estimates method is explored with these systems in the context of a recently enhanced database of liquidus slopes of impurities in Zn in the limit of low concentration.

Abstract: Blackbody radiation sources used in infrared temperature measurement require stable and consistent temperature control. A blackbody radiation source can produce a fixed temperature, which makes it the key element in the process of calibration. Effective control of its temperature is, therefore, crucial. In this study, a comparison is made between conventional PID control and a single neuron PID algorithm for temperature control of a blackbody radiation source. The single neuron PID control algorithm simulated in MATLAB demonstrates its feasibility and improved control performance over the conventional PID algorithm, with advantages of small overshoot, high precision, and strong anti-interference benefits, making it a strong prospect for practical applications.

Abstract: In the paper, a 0.65μm and 0.9μm pyrometers were interpolated by three high temperature fixed points (HTFPs). The best uncertainty was estimated (0.3-1.2) oC (k=2) in (900-2500) oC for the 0.65μm pyrometer and (0.3-0.6) oC in (600-2000) oC for the 0.9μm one separately. The pyrometers were also verified by the other fixed points and high temperature blackbody against the primary pyrometer. The agreement was better than 0.1 oC. The HTFPs can be applied in the precise calibration of pyrometers to improve the level of measurement uncertainty in high temperature range.


2016
Abstract Relative primary acoustic gas thermometry (AGT) determines the ratios of thermodynamic temperatures from measured ratios of acoustic and microwave
resonance frequencies in a gas-filled metal cavity on isotherms of interest. When measured in a cavity with known dimensions, the frequencies of acoustic resonances in a gas determine the speed of sound, which is a known function of the thermodynamic temperature $T$. Changes in the dimensions of the cavity are measured using the frequencies of the cavity’s microwave resonances. We explored techniques and materials for AGT at high temperatures using a cylindrical cavity with remote acoustic transducers. We used gas-filled ducts as acoustic waveguides to transmit sound between the cavity at high temperatures and the acoustic transducers at room temperature. We measured non-degenerate acoustic modes in a cylindrical cavity in the range $295 \text{ K} < T < 797 \text{ K}$. The fractional uncertainty of the measured acoustic frequencies increased from $2 \times 10^{-6}$ at $295 \text{ K}$ to $5 \times 10^{-6}$ at $797 \text{ K}$. In addition, we measured the frequencies of several transverse magnetic (TM) microwave resonances up to $1000 \text{ K}$ in order to track changes in the cavity’s length $L$ and radius $R$. The fractional standard deviation of the values of $L$ deduced from three TM modes increased from $3 \times 10^{-6}$ for $T < 600 \text{ K}$ to $57 \times 10^{-6}$ at $1000 \text{ K}$. We observed similar inconsistencies in a previous study.

Lin H, Che J, Zhang J T, Feng X J Measurements of the viscosities of Kr and Xe by the two-capillary viscometry 2016 Fluid Phase Equilibria 418:198
Abstract Coiled two-capillary viscometers can measure gas viscosities with high accuracy over a wide temperature range. The authors built a two-capillary viscometer and used it to measure the zero-density viscosity of Kr and Xe at temperatures from $243.15 \text{ K}$ to $393.15 \text{ K}$ with relative standard uncertainties of 0.10 % and 0.11 %, respectively. Additional measurements on Ar were used to understand and avoid the error due to inadequate preheating or precooling of the gas entering the viscometer. The error, which occurred at volume flow rates above $4 \text{ m}^3\text{s}^{-1}$, was approximately proportional to the temperature difference between the reference capillary and the probe capillary.

Extensive studies of platinum–carbon eutectic alloy based high temperature fixed point cells have shown that this alloy has extremely good metrological potential as a temperature reference. However, it’s possible adoption as an accepted reference standard means that its eutectic temperature value will soon be agreed with an uncertainty less than most radiation thermometry scales at that temperature. Thus, it will lack credibility if used as a future scale comparison artefact. To avoid this, the fixed-point cell can be deliberately doped with an impurity to change its transition temperature by an amount sufficient to test the accuracy of the scales of the institutes involved in the comparison. In this study dopants of palladium and iridium were added to platinum–carbon to produce ternary alloy and quaternary alloy fixed-point cells. The stability of these artefacts was demonstrated and the fixed-point cells were used to compare the ITS-90 scales of NIM and NPL. It was found that the fixed point
temperatures could be changed by an appreciable amount while retaining the stability and repeatability required for comparison artefacts.


Abstract: The plateau of fixed-point realization is affected by many aspects in ITS-90, temperature uniformity is one of the most important factors. To improve the temperature field performance and reduce the influence caused by leakage, refer to the adiabatic principle which was widely used in the apparatus of low temperature fixed point, a zinc fixed point apparatus based on the quasi adiabatic is design. The temperature grads property 100 mK of the traditional fixed point is reduced to 20 mK, the usage amount of metal is also reduced from 2.0 kg to 200 g. The experimental results show that the temperature plateau sustainable long time, and the reproducibility of the apparatus can be reduced to 0.1 mK. The apparatus can greatly improve the properties of temperature field, reduce the factors of interference evaluation for effect of trace impurities in high purity metal on the phase transition temperature. The miniaturization of the device can be used repeatedly to achieve economic and practical purposes, and also give the impact assessment of trace impurities in high purity metal phase change temperature which provides the basis for reduction interference of the temperature field.

2015


Abstract: We report progress toward determining the Boltzmann constant $k_B$ using the concept of a virtual acoustic resonator, a hypothetical resonator that is mathematically equivalent to a cylindrical cavity with periodic boundary conditions. We derived the virtual resonator by combining the measured frequencies of the longitudinal acoustic modes of two argon-filled, cylindrical, cavity resonators in such a way as to minimize the effects of the cavities’ ends including transducers and ducts attached to the ends. The cavities had lengths of 80 mm and 160 mm and were operated in their longitudinal ($ℓ$,0,0) modes. We explored virtual resonators that combine modes of the two resonators that have the nearly the same frequencies. The virtual resonator formed from the (2,0,0) mode of the 80 mm resonator combined with the (4,0,0) mode of the 160 mm resonator yielded a value for $k_B$ that is, fractionally, only $(0.2 ± 1.5)\times10^{-6}$ larger than the 2010 CODATA-recommended value of $k_B$. (The estimated uncertainty is one standard uncertainty corresponding to 68 % confidence level.) The same virtual resonator yielded values of the pressure derivatives of the speed of sound $c$ in argon, $(\partial c^2/\partial p)_T$ and $(\partial c^2/\partial p^2)_T$, that differed from literature values by 1 % and 2 %, respectively. By comparison, when each cavity was considered separately, the values of $k_B$, $(\partial c^2/\partial p)_T$, and $(\partial c^2/\partial p^2)_T$ differed from literature values by up to 7 ppm, 10 %, and 5 %, respectively. However, combining the results from the (3,0,0) or (4,0,0)
modes of shorter resonator with the results from the (6,0,0) or (8,0,0) modes of the longer resonator yielded incorrect values of \( k_B \) that varied from run-to-run. We speculate that these puzzling results originated in an un-modeled coupling, either between the two cavities (that resonated at nearly identical resonance frequencies in the same pressure vessel) or between the cavities and modes of the pressure vessel.


Abstract: The unit of thermodynamic temperature, the kelvin, will be redefined in 2018 by fixing the value of the Boltzmann constant, \( k \). The present CODATA recommended value of \( k \) is determined predominantly by acoustic gas-thermometry results. To provide a value of \( k \) based on different physical principles, purely electronic measurements of \( k \) were performed by using a Johnson noise thermometer to compare the thermal noise power of a 200 ohm sensing resistor immersed in a triple-point-of-water cell to the noise power of a quantum-accurate pseudo-random noise waveform of nominally equal noise power. Measurements integrated over a bandwidth of 575 kHz and a total integration time of about 33 d gave a measured value of \( k = 1.3806513(53) \times 10^{-23} \) J K\(^{-1} \), for which the relative standard uncertainty is \( 3.9 \times 10^{-6} \) and the relative offset from the CODATA 2010 value is \(+1.8 \times 10^{-6}\).


Abstract: In the pulse-driven ac Josephson voltage standard, the low frequency (LF) component of a drive signal increases the system complexity and induces unwanted voltages through on-chip inductances. A novel zero compensation (ZC) method for pulse-driven ac waveform synthesis is presented in this paper. A pulse train is obtained through a two-level Delta-Sigma modulation and then reconstructed by replacing each pulse with three pulses of a negative-positive-negative pattern. The amplitudes of the reconstructed pulse train are carefully adjusted so that the positive pulses drive the Josephson junctions to the first quantum state, whereas the negative pulses keep the junctions on the zero quantum state and cancel the contribution of the positive pulses to the LF inductive voltage component. This new method eliminates the need for LF signal reinjection required by the conventional bias method and eliminates the need for a direct-current bias required by the recently proposed ZC method. An 8-kHz sinusoidal waveform with an amplitude of 1 mV rms is synthesized to demonstrate the feasibility of the approach. We also demonstrate that the inductive voltage error is significantly reduced for waveforms synthesized with the new method.

Inseok Yang et al Improving acoustic determinations of the Boltzmann constant with mass spectrometer measurements of the molar mass of argon 2015 Metrologia 52: S394–S409
Abstract We determined accurate values of ratios among the average molar masses $M_{Ar}$ of 9 argon samples using two completely-independent techniques: (1) mass spectrometry and (2) measured ratios of acoustic resonance frequencies. The two techniques yielded mutually consistent ratios (RMS deviation of $0.16 \times 10^{-6} M_{Ar}$ from the expected correlation) for the 9 samples of highly-purified, commercially-purchased argon with values of $M_{Ar}$ spanning a range of $2 \times 10^{-6} M_{Ar}$. Among the 9 argon samples, two were traceable to recent, accurate, argon-based measurements of the Boltzmann constant $kB$ using primary acoustic gas thermometers (AGT). Additionally we determined our absolute values of $M_{Ar}$ traceable to two, completely-independent, isotopic-reference standards; one standard was prepared gravimetrically at KRISS in 2006; the other standard was isotopically-enriched $40Ar$ that was used during NIST’s 1988 measurement of $kB$ and was sent to NIM for this research. The absolute values of $M_{Ar}$ determined using the KRISS standard have the relative standard uncertainty $ur(M_{Ar}) = 0.70 \times 10^{-6}$ (Uncertainties here are one standard uncertainty,); they agree with values of $M_{Ar}$ determined at NIM using an AGT within the uncertainty of the comparison $ur(M_{Ar}) = 0.93 \times 10^{-6}$. If our measurements of $M_{Ar}$ are accepted, the difference between two, recent, argon-based, AGT measurements of $kB$ decreases from $(2.77 \pm 1.43) \times 10^{-6}$ $kB$ to $(0.16 \pm 1.28) \times 10^{-6}$ $kB$. This decrease enables the calculation of a meaningful, weighted average value of $kB$ with a uncertainty $ur(kB) = 0.6 \times 10^{-6}$.


Abstract: A bilateral comparison of local realization of the International Temperature Scale of 1990 between the National Institute of Metrology (NIM) and National Metrology Centre (NMC) was carried out over the temperature range from 83.8058 K to 692.677 K. It involved six fixed points including the argon triple point, the mercury triple point, the triple point of water, the melting point of gallium, the freezing point of tin, and the freezing point of zinc. In 2009, NMC asked NIM to participate in a bilateral comparison to link the NMC results to the Consultative Committee for Thermometry Key Comparison 3 (CCT-K3) and facilitate the NMC's calibration and measurement capabilities submission. This comparison was agreed by NIM and Asia Pacific Metrology Programme in 2009, and registered in the Key Comparison Database in 2010 as CCT-K3.2. NMC supplied two fused silica sheath standard platinum resistance thermometers (SPRTs) as traveling standards. One of them was used at the Ga, Sn, and Zn fixed points, while the other one was used at the Ar and Hg fixed points. NMC measured them before and after NIM measured them. During the comparison, a criterion for the SPRT was set as the stability at the triple point of water to be less than 0.3 mK. The results for both laboratories are summarized. A proposal for linking the NMC’s comparison results to CCT-K3 is presented. The difference between NMC and NIM and the difference between NMC and the CCT-K3 average reference value using NIM as a link are reported with expanded uncertainties at each measured fixed point.

Abstract: Chemical analysis of old triple-point-of-water (TPW) cells has shown that the dominant impurities in the water are the principal constituents of borosilicate glass, which suggests the long-term downward drift observed in the realized triple-point temperature of TPW cells is caused by gradual dissolution of the glass containers. Because some of the glass constituents contribute to the electrical conductivity of the water, measurement of the conductivity provides a possible means for monitoring the long-term drift of TPW cells. This paper investigates the utility of a conductivity measurement, based on the measurement of a capacitor using the TPW cell as a dielectric, as a non-destructive means for monitoring the long-term stability of cells. The measurement exploits the cylindrical geometry of the cell and uses a two-terminal-pair coaxial electrical definition of capacitance. The results include estimates of the sensitivity of the method and examples of the long-term behavior of TPW cells, some as old as 38 years and monitored for periods of up to 10 years. It is found that the conductivity measurements correlate well with the cell age, with the drift rates increasing with time, as expected from the chemical model of glass dissolution. Measurements of temperature differences between cells show the technique can detect changes as small as 10 μK, and therefore the method is a useful means of monitoring TPW cells.


In this paper, we present the simulation of the eutectic phase transitions in the Pt–C system, in terms of both freezing and melting, using the multi-phase-field model. The experimentally obtained heat-extraction and injection rates associated with the induction of freezing and melting are converted into the corresponding rates for microstructure-scale simulations. In spite of the extreme differences in the volume fractions of the FCC–Pt-rich phase on the one hand and graphite (C) on the other, satisfactory results for the kinetics of solidification and melting have been obtained, involving reasonable offsets in temperature, inducing freezing and melting, with respect to the equilibrium eutectic temperature. For freezing in the simulations, the needle/rod-like morphology, as experimentally observed, was reproduced for different heat extraction rates. The seemingly anomalous peak characterizing the simulated freezing curves is ascribed to the speed up of the solidification process due to the curvature effect.
Similarly, a peak is observed in the experimental freezing curves, also showing up more clearly with increasing freezing rates. Melting was simulated starting from a frozen structure produced by a freezing simulation. The simulations reproduce the experimental melting curves and, together with the simulated freezing curves, help to understand the phase transition of the Pt–C eutectic. Finally, the effect of metallic impurities was studied. As shown for Au, impurities affect the morphology of the eutectic structure, their impact increasing with the impurity content, i.e., they can act as modifiers of the structure, as earlier reported for irregular eutectics.


The inflection point (POI) of the HTFP melting curve has been mainly applied as a reference point in comparisons of local temperature scales. It has also been proposed to serve as a lower limit to be set to the liquidus temperature. In this paper, we confine ourselves to the determination of the inflection points by a statistical method for the Co-Cinvolved in InK-WP1. Details of the method will be introduced in this paper.

ZHENG Wei, TANG Lei, MENG Xian-zhe, WU Shuang-shuang Statistic Inspection for the Thermoelectric Characteristic of Reference Pt/Rh10-Pt Thermocouples 2015 ACTA MEROLOGICA SINICA 36 6A: 41-44

The historical calibration datum of an amount of reference thermocouples of PtRh10-Pt from multiple institutes were inspected statistically. The thermocouples were observed of stable thermoelectric characteristic that the annual mean of the emf drift close to 0μV at freezing fixed points of Cu, Al, Zn with standard deviation 1.0μV, 0.6μV, and 0.4μV respectively. The histograms of datum at fixed points were nearly normal distribution; the annual emf drift characteristic of individual thermocouple at fixed point appeared in strong biserial correlation. The emf of those thermocouples was observed slightly increasing with time. The inspection demonstrated under the normal condition that no correlation existed between emf drift and frequency of using.


Abstract: An experimental system based on the differential time-of-flight method was built for the measurement of the speed of sound in liquids at high pressure. The double ultrasonic cavities were designed and assembled, the acoustic precision measurement system, liquid filling system with high pressure, pressure and temperature measurement system, and the data acquisition system were established. The speed of sound in pure water was measured at temperature from 303 K to 353 K and at pressure up to 10 MPa with a standard relative uncertainty of 0.018% (k=1). The measured speed of sound data
shows good agreement with the equation of state and published experimental data. This system can be used in the measurement of speed of sound in other liquid such as sea water and new fuels in the next future.


Abstract: Based on the principle of ultrasonic resonance spectrum, a setup is developed including the ultrasonic signaling and detecting system, the thermostat and the data analysis software. The elastic modulus of three samples in different quality factors are measured at room temperature, the effects influencing the measurement are investigated. The resonant frequency matching error (RMS) of 0.04% has been reached with the measurements by the developed setup. The results agree well with those of the global advanced commercial instruments. In addition, bearing steel (9Cr18) are measured at temperature from 293 K to 353 K for isothermal compressibility. The system is capable to measure the elastic modulus of solids at temperature up to 500 K at anti-oxidation atmosphere. After redesigning the ultrasonic signaling and detection system, it is capable for measurements at higher temperature.

2014


Abstract We review the principles, techniques and results from primary acoustic gas thermometry (AGT). Since the establishment of ITS-90, the International Temperature Scale of 1990, spherical and quasi-spherical cavity resonators have been used to realize primary AGT in the temperature range 7 K to 552 K. Throughout the sub-range 90 K < T < 384 K, at least two laboratories measured (T−T90). (Here T is the thermodynamic temperature and T90 is the temperature on ITS-90.) With a minor exception, the resulting values of (T−T90) are mutually consistent within 3×10−6 T. These consistent measurements were obtained using helium and argon as thermometric gases inside cavities that had radii ranging from 40mm to 90mm and that had walls made of copper or aluminium or stainless steel. The AGT values of (T−T90) fall on a smooth curve that is outside ±u(T90), the estimated uncertainty of T90. Thus, the AGT results imply that ITS-90 has errors that could be reduced in a future temperature scale. Recently developed techniques imply that low-uncertainty AGT can be realized at temperatures up to 1350K or higher and also at temperatures in the liquid-helium range.


Abstract: Impurities are the most significant source of uncertainty in most metal fixed points for the realization of the International Temperature Scale of 1990 (ITS-90). The methods for the estimation of uncertainties and corrections of fixed-point temperatures
attributable to the influence of chemical impurities were summarized in 2005, and the sum of individual estimates (SIE) method was recommended to be used with the known concentration and liquidus slope of each impurity. This method requires the concentrations and the liquidus slopes of all impurities. For applying the SIE method, efforts still need to be made to solve a series of problems including the unsatisfactory chemical analysis, inadequate data of the liquidus slopes, and information about the dissolution and precipitation of impurities during the filling and the operation of a fixed-point cell. In the present work at the National Institute of Metrology (NIM), great attention is paid to the effect of ultra-trace impurities on the freezing point of zinc. Five slim graphite crucibles were filled with the same batch of zinc with a nominal purity of 6N for this research. One of them was used to investigate the concentration and distribution of the impurities in the freezing point of zinc by chemical analysis. The remaining crucibles were used to carry out the ultra-trace impurity doping experiments. The liquidus slopes of Ag-Zn, Pb-Zn, Fe-Zn, and Ni-Zn were measured. All results are reported and discussed.

Abstract: The use of melting or freezing temperatures of high-purity substances as thermometric fixed points requires knowledge of the binary phase diagrams of these substances and remaining impurities at very small impurity concentrations. In this paper, a calorimetric apparatus for the measurement of the change in liquidus temperature of fixed-point metals due to dissolved impurities at quasi-adiabatic conditions is presented. This approach combines advantages of the fixed-point method and adiabatic calorimetry. It is more efficient for the screening of a range of impurity concentrations than a fixed-point cell, requires less constructional and experimental expenditure compared with an adiabatic calorimeter, but provides similar small uncertainties on the millikelvin level. Measurements were carried out to determine the influence of lead on the melting temperature of indium at mass fractions up to 6.93%. The results are in very good agreement with previous measurements by means of slim fixed-point cells in the Physikalisch-Technische Bundesanstalt and confirm a minimum of the freezing point of -178 mK at a mass fraction of about 3.7%. It was demonstrated that this setup allows the investigation of binary phase diagrams with uncertainties less than 8 mK.

H. Lin X. J. Feng J. T. Zhang and Can Liu Using a two-capillary viscometer with preheating to measure the viscosity of dilute argon from 298.15 K to 653.15 K 2014 J. Chem. Phys. 141: 234311
Abstract Currently, there exists only one set of experimental results at temperatures up to 680 K with the claimed relative standard uncertainty of (0.15–0.20) %. This paper reports new experimental results using the two-capillary viscometer in the temperature range from 298.15 K to 653.15 K with the claimed relative standard uncertainty of 0.062 %. The new measurements agree with the existing high accuracy measurements and ab initio calculations in the overlapping temperature range within the extraordinary
low relative differences of ±0.08 %. The good agreement represents a robust proof of the potential models derived from the ab initio calculations, which play the powerful means in obtaining the thermophysical properties of dilute monoatomic gases over wide temperature ranges. In the experiments, the authors observed the occurrence of insufficient preheating existing with the two-capillary viscometer at high temperature.


Abstract: Since 2010 we have been developing a quantum-voltage-calibrated Johnson noise thermometer at NIM to measure the Boltzmann constant $k$. With recent improvements in grounding and shielding of the electronics, and matching of the noise sources and transmission lines, the effects of electromagnetic interference and variations of fitting parameters with different bandwidths were greatly reduced. By combining 14 measurements with about 10 hours integrating period for each measurement, the relative standard uncertainties of $6.5 \times 10^{-6}$ and $2.8 \times 10^{-6}$ were achieved from statistics and bandwidth fit variations, respectively.


Abstract: The high-temperature primary standard system was gradually improved through the continuing research work at the National Institute of Metrology (NIM) in China. Based on the developed primary standard pyrometer, an improved scheme, the “fixed-point blackbody - pyrometer” assembly, was applied to realize and disseminate the International Temperature Scale of 1990 (ITS-90) above the silver point. The new scheme can correct the drifts of the pyrometer and extend the highest temperature range from 2200 ºC to 2474 ºC. The expanded uncertainties of the scale were (0.08 - 0.62) ºC between the temperature range (961.78 - 2474) ºC. The un-certainties of the temperature scale are strongly supported by the international comparison of the high temperature points’ ITS-90 values among China, the U.K. and Spain in 2009. The new method could be not only applied in the highest level calibration of the precise pyrometers, but also extended in the calibration of the 900 nm infrared thermometers.


Abstract: Carbon dioxide (R744) and propene (R1270) are expected to be long-term working fluids for air conditioning systems, heat pumps, and organic Rankine cycles (ORCs) because of their low global warming potentials (GWP). The speed of sound was measured in gaseous carbon dioxide from (260 to 333) K and in gaseous propene from (260 to 330) K using a cylindrical resonator at pressures up to 1 MPa. The perturbations from the thermal and viscous boundary layers, the gas fill duct, the shell motion, and the vibrational relaxation were corrected in the frequency measurements. The uncertainties in the temperature, pressure, and speed of sound measurements were estimated to be less than 5 mK, 200 Pa, and 0.02 %. The ideal-gas heat capacities at constant pressure and the acoustic virial coefficients of carbon dioxide and propene were deduced from the measured speed-of-sound data. The second virial coefficients for carbon dioxide and propene were obtained from the acoustic data and the square-well intermolecular model.

Qiang Liu, Xiaojuan Feng, Kai Zhang, Baolin An and Yuanyuan Duan. Vapor pressure and gaseous speed of sound measurements for isobutane (R600a). *Fluid Phase Equilibria* 2014, 382: 260-269

Abstract: The vapor pressures of isobutane (R600a) were measured from (259 to 406) K. The uncertainty of the vapor pressures was estimated to be less than 300 Pa. The measured vapor pressures were correlated using a Wagner-type vapor pressure equation. The critical pressure, normal boiling point and acentric factor were then determined from the vapor pressure equation. The speed of sound in gaseous isobutane was also measured along seven isotherms from (270 to 330) K using a cylindrical resonator. The perturbations from the thermal and viscous boundary layer, the gas fill duct, the shell motion and the vibrational relaxation were corrected in the resonance frequency measurements. The longitudinal acoustic modes were used to determine the speed of sound. The relative standard uncertainty in the speed of sound was estimated to be less than 0.015%. The ideal gas heat capacities and the second acoustic virial coefficients for isobutane were deduced from the speed of sound data. The second virial coefficients were obtained from the measured acoustic data and the square-well intermolecular model.

2013

H Lin, X J Feng, K A Gillis, M R Moldover, J T Zhang, J P Sun and Y Y Duan Improved determination of the Boltzmann constant using a single, fixed-length cylindrical cavity *Metrologia* 2013 50: 417–432

Abstract: We report improvements to our previous (Zhang et al 2011 *Int. J. Thermophys.* 32 1297) determination of the Boltzmann constant k_B using a single 80mm long
cylindrical cavity. In this work, the shape of the gas-filled resonant cavity is closer to that of a perfect cylinder and the thermometry has been improved. We used two different grades of argon, each with measured relative isotopic abundances, and we used two different methods of supporting the resonator. The measurements with each gas and with each configuration were repeated several times for a total of 14 runs. We improved the analysis of the acoustic data by accounting for certain second-order perturbations to the frequencies from the thermo-viscous boundary layer. The weighted average of the data yielded $k_B = 1.3806476 \times 10^{-23} \text{JK}^{-1}$ with a relative standard uncertainty $u_r(k_B) = 3.7 \times 10^{-6}$. This result differs, fractionally, by $(-0.9\pm3.7) \times 10^{-6}$ from the value recommended by CODATA in 2010. In this work, the largest component of the relative uncertainty resulted from inconsistent values of $k_B$ determined with the various acoustic modes; it is $2.9 \times 10^{-6}$. In our previous work, this component was $7.6 \times 10^{-6}$.


Abstract: This work explores the feasibility of acoustic gas thermometry (AGT) in the range 700 K to the copper point (1358 K) in order to more accurately measure the differences between ITS-90 and the thermodynamic temperature. To test material suitability and stability, we investigated microwave resonances in argon-filled cylindrical cavities machined from a Ni–Cr–Fe alloy. We measured the frequencies of five non-degenerate microwave modes of one cavity at temperatures up to 1349 K using home-made coaxial cables and antennas. The short-term repeatability of both the measured frequencies $f_N$ and the scaled half-widths $g_N/f_N$ was better than $10^{-6} f_N$. Oxidation was not a problem while clean argon flowed through the cavity. The measurement techniques are compatible with highly accurate AGT and may be adaptable to refractive index gas thermometry.

J T Zhang, H Lin and J Che Effects of connecting tubing on a two-capillary viscometer 2013 *Metrologia* 50: 377–384

Abstract Two-capillary viscometry is useful for accurately measuring the temperature dependence of the viscosity of dilute gases. A number of publications show good agreement among measurements by two-capillary and primary viscometry, and ab initio calculation. To the authors’ best knowledge, there is no publication describing the effects of connecting tubing on two-capillary viscometry. We present in this paper an investigation by a two-capillary viscometer on the effect of connecting tubing, the available Dean number range and a different method for the stabilization of gas pressures. Our study demonstrates that the incompatibility of connecting tubing causes a systematic error in measurement of dilute gas viscosity. The error diminishes with decreasing Dean number. We discuss the cause of such a systematic error based on analysis of the experimental data. We observe that a Dean number not exceeding 17 is available for our two-capillary viscometer with capillaries made from electro-polished stainless steel. We observed that a piece of coiled connection duct can suppress the instability in the gas pressure by two orders. The results of the above experimental
investigations are reported in this paper.


Abstract: A new quantum-voltage-calibrated Johnson noise thermometer was developed at the National Institute of Metrology to demonstrate the electrical approach that determines Boltzmann's constant $k$, by comparing electrical and thermal noise power. A measurement with an integration period of 19 h and a bandwidth of 638 kHz results in a relative offset of $1 \times 10^{-6}$, from the current Committee on Data for Science and Technology value of $k$, and a type A relative standard uncertainty of $17 \times 10^{-6}$. Closely matched noise powers and transmission-line impedances were achieved, and consequently, the quadratic fitting parameters of the ratio spectrum show flat frequency responses with respect to the measurement bandwidth. This flat response produces a dramatically reduced systematic error compared to that of the National Institute of Standards and Technology measurement of $k$, in which the relative combined uncertainty was dominated by this error.

Xiaopeng Hao, Helen McEvoy, Graham Machin, Zundong Yuan, Tiejun Wang, A comparison of the In, Sn, Zn and Al fixed points by radiation thermometry between NIM and NPL and verification of the NPL blackbody reference sources from 156 °C to 1000 °C 2013 *Measurement Science and Technology* 24: 075004.

Abstract National Institute of Metrology, China (NIM) and National Physical Laboratory, UK (NPL) blackbody cavities based on the ITS-90 freezing points of In, Sn, Zn and Al have been compared by radiation thermometry. The average differences between In, Sn and Zn, measured by NPL InGaAs radiation thermometer, are 5.3, 2.2 and $-7.3$ mK, respectively, and the average differences between Sn, Zn and Al, measured by NIM InGaAs radiation thermometer, are 1.4, $-5.8$ and $13.7$ mK, respectively. These differences are well within the uncertainties of the comparison. The NPL blackbody reference sources from 156 °C to 1000 °C were verified using both the NPL and the NIM InGaAs thermometers calibrated at the fixed points. The differences between the radiance temperature of the sources and the temperature measured by the contact sensor within the source are generally within 0.1 K.

Pieter Bloembergen, DONG WEI, Zhang Hang, A new approach to the determination of the liquidus and solidus points associated with the melting curve of the eutectic Co–C, taking into account the thermal inertia of the furnace 2013 *Metrologia* 50:295~306

In the case of metal–carbon eutectics, with transition temperatures above 1000°C, the liquidus-point temperature, associated with melting, serves as a prime reference temperature. Factors influencing the shape of the melting curve and how to deal with them when specifying the liquidus temperature are the main subject of this paper. Influence factors investigated are the impurity factor, the furnace-temperature gradient, and a factor not considered before: the thermal inertia of the furnace and the associated
transient in furnace temperature during melting. These investigations resulted in an improved method for determining the liquidus point, demonstrated for Co–C, which implicitly corrects for the post-melting effect, if any. In addition, a new method, the step-shift method, is proposed for the determination of the solidus point by direct observation. As shown, there is a clear justification for establishing reference methods to determining the solidus and liquidus points of M–C eutectics along the lines set out in this paper.


Abstract: A quantum-voltage-calibrated Johnson-noise thermometer was developed at NIM, which measures the Boltzmann constant $k$ through comparing the thermal noise across a 100 $\Omega$ sense resistor at the temperature of the triple point water to the comb-like voltage waveform synthesized with a bipolar-pulse-driven quantum-voltage-noise source. A measurement with integration period of 10 hours and bandwidth of 640 kHz results in a relative offset of $0.5 \times 10^{-6}$ from the current CODATA value of $k$, and a type A relative standard uncertainty of $23 \times 10^{-6}$. Benefiting from closely matched noise power and transmission line impedances, small nonlinearities in the cross-correlation electronics, and some other possible reasons, the derived $k$ shows self-consistent values and standard uncertainties for different measurement bandwidths.

J. T. Zhang, H. Lin, and X. J. Feng Fixed-path-length cylindrical cavities for redetermining the Boltzmann constant 2013 *AIP Conf. Proc.* 1552, 11-16

Abstract. In preparation for re-determining the Boltzmann constant $k_B$ at NIM, we report studies of the longitudinal acoustic resonances in five, argon-filled, fixed-path-length cylindrical cavities. These cavity resonators differ in lengths, method of support, and gas fill ducts. The square of the apparent speed of sound $c^2$ in each cavity deviates from reference values by linear functions of the pressure with mode-dependent, negative coefficients. Thus, all the resonators are more compliant than our models predict. The best-performing resonator was 80 mm long and 80 mm in diameter. For this resonator, the zero pressure limits of $c^2$ for the four modes had a fractional standard deviation from their mean of $3.6 \times 10^{-6}$. This suggests that we can reduce the uncertainty of our previous measurement of $k_B$ by a factor of two.


Abstract. Acoustic thermometers normally embed small acoustic transducers in the wall bounding a gas-filled cavity resonator. At high temperature, insulators of transducers loss electrical insulation and degrade the signal-to-noise ratio. One essential solution to this technical trouble is to couple sound by acoustic waveguides between resonator and transducers. But waveguide will break the ideal acoustic surface and bring perturbations($\Delta f^2+ig$) to the ideal resonance frequency. The perturbation model for
waveguides was developed based on the first-order acoustic theory in this paper. The frequency shift $\Delta f$ and half-width change $g$ caused by the position, length and radius of waveguides were analyzed using this model. Six different length of waveguides (52–1763 mm) were settled on the cylinder resonator and the perturbation ($\Delta f + ig$) were measured at $T=332$ K and $p=250–500$ kPa. The experiment results agreed with the theoretical prediction very well.


Abstract. The temperature-jump and slip-velocity at the resonator wall cause boundary perturbations to resonance frequencies. For cylindrical resonators, the perturbation contributes larger uncertainties to the Boltzmann constant than for spherical resonators. This report describes the analytical investigations on the shift of resonance frequency from the thermal and viscous accommodation effects. The expression for calculating the thermal accommodation coefficient from the speed of sound data using cylindrical resonators is presented. The thermal accommodation coefficient between argon gas and the bearing steel wall is estimated to be $0.97\pm0.03$. The squared speeds of sound at zero pressure $c_0^2$ in argon for various modes calculated from different thermal accommodation coefficients are compared. The 0.03 uncertainty for the thermal accommodation coefficient causes relative deviations of $c_0^2$ of $0.6 \times 10^{-6}$ and $0.8 \times 10^{-6}$ in cylindrical resonators with length of 130 mm and 65 mm, respectively. The effect on $c_0^2$ is reduced if the measurements from the two cylindrical resonators are analyzed together.

YAN Xiaoke, ZHANG Jintao, ANDREA Merlone, DUAN Yuning, WANG Wei. NIM Gas Controlled Sodium Heat Pipe 2013 AIP Conf. Proc. 1552: 834-839

Abstract: Gas controlled heat pipes (GCHPs) provide a uniform, stable and reproducible temperature zone to calibrate thermometers and thermocouples, and to realize defining fixed points using a calorimetric method. Therefore, to perform such investigations, a GCHP furnace using sodium as its working fluid was constructed at the National Institute of Metrology (NIM), China. Also, investigations into the thermal characteristics of the NIM gas controlled sodium heat pipe were carried out. The temperature stability over 5 hours was better than $\pm0.25$ mK while controlling the pressure at 111250 Pa. The temperature uniformity within 14 cm from the bottom of the thermometer well was within 0.3 mK. While keeping the pressure stable at the same value, 17 temperature determinations were performed over 14 days, obtaining a temperature reproducibility of 1.27 mK. Additionally, the NIM gas controlled sodium heat pipe was compared with the sodium heat pipe produced by INRiM. The temperature in the INRiM sodium heat pipe operating at 111250 Pa was determined, obtaining a difference of 21 mK with respect to the NIM GCHP. This difference was attributed to sodium impurities, pressure controller capabilities and reproducibility, and instabilities of high temperature standard platinum resistance thermometers (HTSPRTs). Further investigations will be carried out on extending the pressure/temperature range.
and connecting both GCHPs to the same pressure line.

Abstract: The temperature stability of standard resistors plays an important role in assuring the accuracy of resistance measurements that use resistance ratio bridges to calibrate standard platinum resistance thermometers (SPRTs) on the International Temperature Scale of 1990 (ITS-90). Therefore, during resistance measurements, the resistors are always kept in temperature controlled oil or air baths and enclosures to maintain a constant temperature. In order to achieve better temperature stability and reduce the effects of temperature fluctuations on the resistance of a standard resistor, an annular water heat pipe thermostat immersed in a mineral oil bath was developed to accommodate and stabilize a standard resistor. Also, the thermostat's performance was characterized. The results showed that the temperature stability of standard resistors was within 1 mK after using the heat pipe thermostat compared with that of 30 mK for the oil bath. Therefore, the temperature stability of standard resistors was improved by more than one order of magnitude, which was attributable to the good thermal self-regulating abilities of the water heat pipe.

Abstract: In order to improve the automation and convenience of the process involved in realizing the gallium fixed points, an automated apparatus, based on thermoelectric and heat pipe technologies, was designed and developed. This paper describes the apparatus design and procedures for freezing gallium mantles and realizing gallium melting and triple points. Also, investigations on the melting behavior of a gallium melting point cell and of gallium triple point cells were carried out while controlling the temperature outside the gallium point cells at 30 °C, 30.5 °C, 31 °C, and 31.5 °C. The obtained melting plateau curves show dentate temperature oscillations on the melting plateaus for the gallium point cells when thermal couplings occurred between the outer and inner liquid-solid interfaces. The maximum amplitude of the temperature fluctuations was about 1.5 mK. Therefore, the temperature oscillations can be used to indicate the ending of the equilibrium phase transitions. The duration and amplitude of such temperature oscillations depend on the temperature difference between the setting temperature and the gallium point temperature; the smaller the temperature difference, the longer the duration of both the melting plateaus and the temperature fluctuations.

Abstract: The influence of impurities on the shape of the freezing curves of the triple
point of water (TPW) in three small TPW cells was investigated using the freezing curve analysis method. We describe the procedure for preparing outer ice mantles in small TPW cells, for obtaining freezing plateaus, and for comparing the results between the old cell (s/n: 021) and the new cells (s/n: 001 and s/n: 008). The experimental results show that the maximum influence of impurities on the observed phase-transition temperature of water in the cell (s/n: 021) is approximately 0.2 mK below the peak temperature of the freezing plateau during freezing. Also, jagged temperature fluctuations were observed near the end of the freezing plateau in the old cell. However, these phenomena did not appear in the freezing plateaus of the new small cells. The equilibrium temperature realized with the old cell is 2.3 mK lower than that of the new cells, possibly due to excessive residual air. Therefore, assessing the effects of impurities on the TPW using an outer sheath method similar to that used in obtaining the fixed points of other metals is useful. Additionally, an estimated total mole fraction impurity concentration can be determined using Raoult's Law and the first cryoscopic constant for water.

J. Sun, J. T. Zhang, Q. Ping, Improvements in the realization of the ITS-90 over the temperature range from the melting point of gallium to the freezing point of silver at NIM 2013 AIP Conf. Proc. 1552: 277~282
Abstract: The temperature primary standard over the range from the melting point of gallium to the freezing point of silver in National Institute of Metrology (NIM), China, was established in the early 1990s. The performance of all of fixed-point furnaces degraded and needs to be updated due to many years of use. Nowadays, the satisfactory fixed point materials can be available with the development of the modern purification techniques. NIM plans to use a group of three cells for each defining fixed point temperature. In this way the eventual drift of individual cells can be evidenced by periodic intercomparison and this will increase the reliability in disseminating the ITS-90 in China. This article describes the recent improvements in realization of ITS-90 over temperature range from the melting point of gallium to the freezing point of silver at NIM. Taking advantages of the technological advances in the design and manufacture of furnaces, the new three-zone furnaces and the open-type fixed points were developed from the freezing point of indium to the freezing point of silver, and a furnace with the three-zone semiconductor cooling was designed to automatically realize the melting point of gallium. The reproducibility of the new melting point of gallium and the new open-type freezing points of In, Sn, Zn, Al and Ag is improved, especially the freezing points of Al and Ag with the reproducibility of 0.2mK and 0.5mK respectively. The expanded uncertainty in the realization of these defining fixed point temperatures is 0.34mK, 0.44mK, 0.54mK, 0.60mK, 1.30mK and 1.88mK respectively.

Abstract: This paper describes a comparison between PTB and NIM in the field of
absolute spectral-band radiometry and thermodynamic temperature measurement. For
the comparison a NIM made interference filter radiometer with a centre wavelength of
633 nm was taken to PTB. The filter radiometer was calibrated at NIM and PTB with
respect to spectral irradiance responsivity. For the integral value in the band-pass range
an agreement of 0.1% was observed in both calibrations. In a next step, the 633 nm
filter radiometer was used to measure the temperature of a high-temperature blackbody
in comparison to an 800 nm filter radiometer of PTB in the temperature range between
1400 K and 2750 K. The thermodynamic temperature measured by the two filter
radiometers agreed to within 0.2 K to 0.5 K with an estimated measurement uncertainty
ranging between 0.1 K and 0.4 K (k=1).

X. Hao, Z. Yuan, X. Lu, An InGaAs Detector Based Radiation Thermometer and Fixed-
1552,643.
Abstract. In this paper, we describe an InGaAs detector based radiation thermometer
(IRT) and new design of fixed-point blackbodies, including Sn, Zn, Al and Cu, for the
establishment of a temperature scale from 200 oC to 1085 oC at the National Institute
of Metrology of China. The construction and calibration of the IRT with the four fixed-
point blackbodies are described. Characteristics of the IRT, such as the size-of-source
effect, the amplifier performance and its stability are determined. The design of the four
fixed-points, with 10 mm diameter of aperture and 0.9999 emissivity, is described.
The uncertainty of the scale realization is elaborated.

X. Hao, Z. Yuan, X. Lu, Lens Transmission Measurement for an Absolute Radiation
Abstract. The lens transmission for the National Institute of Metrology of China
absolute radiation thermometer is measured by a hybrid method. The results of the lens
transmission measurements are 99.002% and 86.792% for filter radiometers with center
wavelengths 633 nm and 900 nm, respectively. These results, after correcting for
diffraction factors and the size-of-source effect when the lens is incorporated within the
radiometer, can be used for measurement of thermodynamic temperature. The expanded
uncertainty of the lens transmission measurement system has been evaluated. It is
1.3×10⁻³ at 633 nm and 900 nm, respectively.

High Temperature Fixed Points 2013 *AIP Conference Proceedings* 1552:386~391
The thermogauge furnace was commonly used in many NMIs as a blackbody source
for calibration of the radiation thermometer. It can also be used for realizing the high
temperature fixed point(HTFP). According to our experience, when realizing HTFP we
need the furnace provide relative good temperature uniformity to avoid the possible
damage to the HTFP. To improve temperature uniformity in the furnace, the furnace
tube was machined near the tube ends with a help of a simulation analysis by “ansys
workbench”. Temperature distributions before and after optimization were measured
and compared at 1300 °C, 1700°C, 2500 °C, which roughly correspond to Co-C(1324 °C), Pt-C(1738 °C) and Re-C(2474 °C), respectively. The results clearly indicate that through machining the tube the temperature uniformity of the Thermogage furnace can be remarkably improved. A Pt-C high temperature fixed point was realized in the modified Thermogauge furnace subsequently, the plateaus were compared with what obtained using old heater, and the results were presented in this paper.


Abstract Three WC-C peritectic fixed point cells, constructed from different sources of tungsten with different nominal purities, were measured at NIM and NMIJ. The three cells were constructed at NMIJ by NIM and NMIJ staffs, and $T_{90}$ values of the three cells were measured at NMIJ during the period 31 Aug. to 25 Dec. 2009. Thereafter, the three cells were then transported to NIM, and $T_{90}$ values of these cells were measured from 7 Dec. 2011 to 9 Jan. 2012. The results showed that $T_{90}$ values of the three cells measured at the two institutes agreed within 0.4 °C with the combined scale comparison uncertainty of 1.7 °C ($k = 2$). The main component of the uncertainty is not the uncertainty due to impurities of the cells but the scale uncertainty and the stability of the measurement system. From these results it can be concluded that the WC-C cell is stable enough to provide new means of international high-temperature scale comparison above 3000 K.


A Re-C fixed point was filled at the National Physical Laboratory (NPL), UK and its melting temperature compared to a fixed point that had been filled previously at NPL. Both fixed point cells were of the hybrid type and used a purified graphite foil between the sacrificial graphite sleeve and the outer crucible. The melting temperatures of these two fixed points were compared and found to agree within the comparison uncertainties. Another Re-C fixed point was filled at the National Research Council (NRC), Canada. This fixed point was also of the hybrid type but contained carbon-composite sheet as the liner between the sleeve and the outer crucible. The melting temperatures of the fixed point filled at NPL and the one filled at NRC were compared and found to agree within the uncertainties of the comparison. When the ITS-90 temperatures at the Re-C melting point (~ 2474 °C) measured at NPL were compared to those measured at NRC they were also found to agree within the uncertainties of their respective scales.

The WC-C peritectic fixed point, nominal melting and freezing temperature 2747 °C, shows extremely good metrological potential. Elsewhere, we published a prototype scale comparison of the ITS-90 between NPL, NIM and CEM, using high temperature eutectic fixed points (HTFPs) of Co-C (1324 °C), Pt-C (1738 °C), and Re-C (2474 °C). In this paper we present the further results of the bilateral comparison of the ITS-90 at an even higher temperature, 2747 °C, between NIM and NPL using WC-C peritectic fixed points. A NIM single zone high temperature furnace, model Chino IR-80, was modified to extend its temperature to 2800 °C. Then, an NPL researcher, on secondment to NIM, filled two WC-C cells in the modified furnace in a vertical position. The two WC-C cells were then realized in the same furnace, in an horizontal position. Their melting temperatures, defined by the inflection point of the melting curves, were measured by a linear pyrometer, model NIM-PSP. NIM’s ITS-90 scale was assigned to the two cells, which were then transported to NPL. The realization of NPL’s ITS-90 was then assigned to the two cells by using a model HT9500 Thermogauge furnace to realize the fixed points and a linear pyrometer, model LP3, to determine their temperature. The difference from the mean value of the NIM and NPL ITS-90 values for the WC-C points was derived. This allowed us to compare ITS-90 as realized by the two institutes and to determine the uncertainty in the scale comparison.


Nonlinearity of photoelectric pyrometer is one of the main uncertainty sources for realization of international temperature scale. A novel nonlinearity measurement system based on LED light sources and flux addition method was established. The high brightness LEDs with nominal 645 nm peak wavelength were utilized as light source to substitute conventional tungsten lamp. The switch of power supply for LEDs can realize flux addition, and the abandonment of optical strobe can avoid the nonideal result from repeated reflection in the optical path. The experimental research on the spectral radiation drift of LEDs under step-mode power supply was carried out to
validate the end time of nonlinear drift stage. The pulse-mode power supply of fixed phase shift for double LEDs and beam splitter were combined to attain flux addition. The linear drift stage of LEDs was chosen to measure the nonlinearity of pyrometer. The linear drift effect on the nonlinearity measurement was eliminated by exchange the measurement turn and experiments were carried out to validate the method. The nonlinearity of the standard radiation thermometer (model RT9032) was experimentally studied with the nonlinearity from $0.2 \times 10^{-4}$ to $1 \times 10^{-4}$, and the photocurrent ranged from $1.1 \times 10^{-11}$ to $2 \times 10^{-7}$ A. The standard uncertainty of nonlinearity was better than $0.7 \times 10^{-4}$ within the photocurrent range from $3.6 \times 10^{-11}$ to $4.4 \times 10^{-7}$ A, and $1.5 \times 10^{-4}$ for else.

The metal-carbon fixed point (M-C HTFP) used to thermometer calibration was star from Japan. The NIMs tried to establish the HTFP system recently for it could be adopted in next international temperature scale. In the paper, the Co-C cell was fabricated and tested in contact thermometer lab at NIM for thermocouple calibration. The reproducibility of melting point of CoC cell is 0.05°C (1σ) using Pt/Pt thermocouple, and cell was robust after 500h heat cycles above 1300°C.

Zheng Wei, Yang Zhen-hua, Xiang Ming-dong Study of Inhomogeneity on the Thermocouple in Liquid Bath 2013 ACTA MEROLOGICA SINICA 34(4): 356-359
The inhomogeneity of thermocouple was estimated by measuring the immersion characteristic in a liquid bath at 250°C. The emf. of thermocouple was recorded when it scanned was along its thermo-elements where great temperature gradient was in small gap above the bath. A group of fresh Pt10Rh-Pt thermocouple of and an used one were test. The repeatability of datum is satisfied, and the homogeneity of fresh thermocouple was better than the used one, which was concord to logical presumption.

Abstract: A new apparatus of the melting point of gallium is developed in National Institute of Metrology. The melting point of gallium can be realized automatically by the fixed point furnace with the three-zone freezing and heating technique. The excellent stability and uniformity of the fixed point furnace is obtained because of the precise temperature control and the reasonable design. The cell of the gallium melting point is filled with the 99.999 99% purity gallium that is purified by the present technique and improved perfusion technique. The experiment results show that the plateau of the apparatus lasts more than 70 hours, and the difference of the plateau from 20% to 80% is less than 0.15 mK. The reproducibility of the apparatus is 0.07 mK, nd the expanded uncertainty is 0.36 mK ($k=2$).

Abstract: Speed of sound is one of the most important thermophysical properties that can be measured precisely; it is also the important basic parameter in thermodynamic basic study and engineering applications. A new sound speed measurement system was built based on the cylindrical resonator method, which is the first national precision sound speed measurement system than can measure the sound speed at high pressure up to 6 MPa. The system solves the sound speed measurement problem at high pressure, greatly expands the measurement range (1 MPa) of the existing experiment system in China, and it is also a successful attempt in the world that uses cylindrical acoustic resonator at pressure above 1 MPa and achieves the uncertainty of 0.01%. The new built system includes the cylindrical acoustic resonator, high pressure vessel, frequency measurement system, temperature measurement system, pressure measurement system, gas filling system and vacuum system. Experimental results show that the system can be used to measure the speed of sound in gases at temperature from 220 K to 440 K and high pressure up to 6 MPa with an uncertainty less than 0.01%.

2012


Abstract: A high accuracy laser interferometric dilatometer with a resolution of less than 1 nm has been developed in the National Institute of Metrology, China. This instrument is based on a single frequency polarized interferometer. The disadvantage of the single frequency procedure is that the zero may drift with time. In order to improve the stability, we developed a procedure to correct the effect of this drift, to ensure the stability of the device within 1 nm. The performance of the new device has been examined by measuring some reference materials. The Standard Reference Material 738(austenitic stainless steel) from the National Institute of Standard and Technology was measured over the temperature range from 300 K to 770 K. The measured linear Expansion Coefficient agreed with the values referred by NIST of the relative deviation less than 1.7% with the combined standard uncertainty at room temperature of 3.4 x 10(-2)(k = 2). The silicon sample was measured over the range from 770 K to 1200 K, and the measurement results were in good agreement with the recommended values by the Committee on Data for Science and Technology (CODATA).

Wei Zheng, Mingdong Xiang Researching on the stability of Pt/Pd thermocouple in range of 960℃~ 1500℃ 2012 *ACTA MEROLOGICA SINICA* 33(6A): 66-69

The performance of Pt/Pd thermocouple was attained reputation in recent years in high temperature application. The skills of construction of thermocouple and test of
thermocouple in realization of Ag(961.78°C), Cu(1084.62°C) freezing point and Co-C(1324°C) Pd-C(1492°C) eutectic points were introduced in the paper. It was proved to be an ideal precise thermometer in high temperature scale by the stability of thermocouple. The mini-coil structure was helpful to solve the breakage bugs of measurement junction.

It was an improvement in temperature disseminate system in over 1100 ℃ when metal–carbon Eutectic Point was introduced in high temperature scale. The Co-C(1324°C) and Pd-C(1492°C) Eutectic points could filled the gap between Cu FP (1084.62°C) and Pd MP (1554.8°C) in thermocouple calibration interpolation. The process of construction and experiment were introduced in the paper. The reproducibility of melting point was 0.06 ℃, and a cell was not broken after 30 cooling and heating cycles over 100 h in high temperature.

A new bath was developed by using automatic freezing method for triple point of water (TPW) in this study. Compared with traditional manual method, the automatic method test data expressed that the TPW value is 0.3mK lower than primary standard after 1 hour, and 0.2mK small than primary standard after 3 hours. It proved that the automatic method could be used to measure and calibration platinum resistance thermometer.

A study on the development of calibration thermostat for temperature sensor pair of heat meter is described. The thermostat is designed of two tanks which liquid flows in horizontal pattern to reduce the heat convection. The cover has three layers which insure liquid flow placidly. The uniformity the the thermostat is measured by two thermometers in horizon position. The results showed that uniformity is better than 3mK at 3cm position below the liquid surface. The performance of the thermostat satisfied the requirement of shallow immersion calibration of the temperature sensor pair.


2011
J. T. Zhang · H. Lin · X. J. Feng · J. P. Sun · K. A. Gillis · M. R. Moldover · Y. Y. Duan
Progress Toward Redetermining the Boltzmann Constant with a Fixed-Path-Length Cylindrical Resonator 2011 Int J Thermophys32:1297–1329
Abstract A single, fixed-path-length cylindrical-cavity resonator was used to measure \( c_0 = (307.8252 \pm 0.0012) \text{ m} \cdot \text{s}^{-1} \), the zero-density limit of the speed of sound in pure argon at the temperature of the triple point of water. Three even and three odd longitudinal modes were used in this measurement. Based on the ratio \( M/\gamma_0 = (23.968644 \pm 0.000 033) \text{ g} \cdot \text{mol}^{-1} \), determined from an impurity and isotopic analysis of the argon used in this measurement and the measured \( c_0 \), the value \( k_B = 1.380 650 6 \times 10^{-23} \text{J} \cdot \text{K}^{-1} \) was obtained for the Boltzmann constant. This value of \( k_B \) has a relative uncertainty \( u_r(k_B) = 7.9 \times 10^{-6} \) and is fractionally, \((0.12 \pm 8.1) \times 10^{-6}\) larger than the value recommended by CODATA in 2006. (The uncertainty is one standard uncertainty.) Several, comparatively large imperfections of our prototype cavity affect the even longitudinal modes more than the odd modes. The models for these imperfections are approximate, but they suggest that an improved cavity will significantly reduce the uncertainty of \( c_0 \).

Abstract: Due to an excellent temperature flattening ability, annular sodium heat pipes operating in the 500 °C to 1200 °C temperature range have been widely used as liners for isothermal furnaces to provide uniform and stable temperature zones in the thermometry field. In order to develop the capabilities to fabricate liquid-metal heat pipes, apparatus for fabricating sodium heat pipes were set up at the National Institute of Metrology (NIM), China. In this paper, we describe the newly developed fabrication apparatus, the detailed procedures for manufacturing sodium heat pipes, the sodium heat pipes, the constructed furnaces for realizing the aluminum freezing point, and their isothermal characteristics. The experimental results showed that the biggest temperature differences within 150 mm from the bottom of the thermometer well in an aluminum fixed cell installed into the sodium heat pipe furnaces were better than 15 mK, when the temperatures of the furnaces were controlled at approximately 657 °C.

Abstract: An investigation into the thermal characteristics of glass-water heat pipes over the temperature range from 0 °C to 60 °C has been carried out at the National Institute of Metrology (NIM), China. In this paper, we describe a glass-water heat pipe with the four thermometer wells and corresponding studies. The experimental results
indicate that temperature stability and uniformity of the liquid bath could be obviously improved when the heat pipe immersed in 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. Also, over the temperature range of 0 °C to 30 °C, the glass-water heat pipes can still operate and the temperature stability of the thermometer well of the glass-water heat pipe was better than 0.1 mK for approximately 16 hours. Additionally, effects of the amount of the working fluid on the isothermal characteristics are conducted.


Abstract: In order to investigate mechanisms of phase transitions of supercooled water in a triple-point-of-water (TPW) cell when a mush method was used to create an ice mantle, an automated apparatus using small TPW cells was developed to obtain the TPW. In this article, the design principle, the apparatus, and the procedure for an automated formation of ice mantles in small TPW cells are described. Supercooled water in small TPW cells spontaneously transformed into uniform metastable dendritic crystals throughout the cells at supercoolings ranging from 5.85 °C to 8.77 °C and then changed into stable hexagonal closed-packed cellular crystals, forming an outer ice mantle from the outside inward. Some pertinent explanations based on thermodynamic solidification theory were used to describe the phase transition process in the mush method. In addition, the experimental results indicated that the realized temperatures of water in the small TPW cells were in good agreement within 0.1 mK approximately 6 h after the initial spontaneous crystallization had occurred. Finally, the small TPW cells (s/n 008 and s/n 001) were directly compared with a conventional TPW cell (s/n NIM-1-211); the temperature differences between the small TPW cells and the regular TPW cell were less than 0.21 mK.


Abstract: The length is one of the key parameters for a cylindrical acoustic resonator used for measurement of the Boltzmann constant. A research project has been conducted in the National Institute of Metrology (NIM), China, for the re-determination of the Boltzmann constant with a fixed-path cylindrical acoustic resonator. This paper describes the procedure for the length determination. The excess fraction method was applied to accurately obtain the length of the resonator. This method is performed in a two-step procedure. First, the length is coarsely determined as L 1 with an uncertainty of 1.5 μm in the length division of NIM. Second, the result of the coarse measurement is further interpolated by the dual wavelength laser interferometer with a resolution of 1 nm, which is composed of a 633 nm He–Ne laser and a 657 nm semiconductor laser. A Michelson wavemeter has been constructed for calibration of the wavelength of the
semiconductor laser. The length variation of the resonator has to be measured from room temperature to the triple point of water (TPW). As a result, the laser interferometer can be also used as a precise dilatometer. The result and the measurement uncertainty of the length measurement are given in this paper.


Abstract: At the National Institute of Metrology of China (NIM), silicon photodiode-based narrow-band interference filter radiometers (FRs) have been designed for the radiometric determination of the thermodynamic temperature. The FR calibrations were performed on a new spectral comparator with a trap detector which was calibrated against the cryogenic radiometer at several discrete laser lines. The new spectral comparator is constructed from two grating monochromators assembled to give lower stray light and higher transmitted flux. Applying a transmittance measurement of the filter in the out-of-band region and careful control of the temperature, the irradiance responsivity of a 633nm centered FR has been obtained over a dynamic range of nearly eight decades in the wavelength range from 450nm to 1200 nm. The relative standard uncertainty of the responsivity is also analyzed and estimated to be less than $7 \times 10^{-4}$ at the 1σ level.


Abstract: The size-of-source effect (SSE) is an important uncertainty source in radiation thermometry when radiation targets with different sizes are measured. In this article, a test pyrometer was established to study the way to reduce the SSE. The most dominant factors that influence the SSE, include the quality and surface condition of the objective lens, the placement and diameter of the aperture stop, and the inner baffles. Through an optimized lens and redesign of the aperture stop and baffles, the SSE of the test pyrometer is reduced to $2 \times 10^{-4}$ when a 50mm diameter radian source with a 4mm diameter central obscuration is used. The improvement method is applied to the primary standard pyrometer for which the SSE is reduced to less than $1 \times 10^{-4}$.


The feasibility of referring to freezing as an alternative to melting for defining the eutectic transition temperature has been studied using two Pt-C cells constructed at NIM; one of a sleeve type, and the other of a hybrid type, including support. Freezing and melting experiments have been done by varying the offset of the furnace temperature $T_{\text{furn}}$ with respect to the nominal eutectic temperature $T_E$ used to freeze the fixed point with offsets $(T_{\text{furn}}-T_E)_{\text{freeze}}$ from $-5$ to $-40$ K, followed by melting at a fixed offset $(T_{\text{furn}}-T_E)_{\text{melt}} = +20$ K. Plotting the liquidus temperatures $T_{\text{liq,freeze}}$ and $T_{\text{liq,melt}}$ obtained for freezing and melting against $(T_E-T_{\text{furn}})^{1/2}_{\text{freeze}}$ resulted in linear
relations for both cells, allowing extrapolation towards $T_{\text{liq,freeze}}(0)$ and $T_{\text{liq,melt}}(0)$. We found for the cells Pt-C5# and Pt-C6# in study: $T_{\text{liq,melt}}(0) - T_{\text{liq,freeze}}(0) = 10$ and $20$ mK, respectively, with a standard uncertainty of $30$ mK for both $T_{\text{liq,melt}}(0)$ and $T_{\text{liq,freeze}}(0)$. The coherence of the results obtained for melting and freezing indicates that freezing can be used, as an alternative to melting, to define the liquidus temperature $T_{\text{liq}}(0)$ of the eutectic Pt-C.


The non-linearity (NL) of radiation thermometers is critically involved when realizing ITS-90 above the silver point. It has to be corrected for, and its uncertainty should be adequately specified. In this article, results are presented of NL measurements based upon the superposition method and involving light emitting diodes (LEDs) with high radiance output, peaked at a wavelength of 645 nm. To this end, the two LEDs in question have been operated in the pulse mode with a fixed phase shift. Their spectral radiances have been measured by the radiation thermometers to be tested, separately or super imposed by means of a beamsplitter. Still the drift in the spectral radiance observed after switching on the LEDs has to be taken into account and corrected for, since this drift, interfering during the superposition procedure, could corrupt the sum rule for the fluxes involved, which is the crux of the superposition method. Therefore, experimental research has been carried out to characterize the drift of LEDs operated in the pulse mode with a fixed phase shift. The result indicated that the drift could be roughly specified in terms of characteristic time intervals: non-linear drift in the first tens of seconds followed by a quasi-linear drift in the subsequent time interval. The crossover time from non-linear to quasi-linear drift could be ascertained by experiment. In the course of the superposition process, switching of the LEDs was done in such a way that only the quasi-linear part of the drift was involved which allowed for the correction of the drift in the NL measurements, as will be reported. Two radiation thermometers were involved in the NL measurements at radiance temperatures between the silver point and 3153K: an LP-4, with a central wavelength of 650nm and 10nm bandwidth, manufactured by KE-Technologie and the primary standard pyrometer PSP, with a central wavelength of 660nm and 10nm bandwidth, developed by NIM.


The nonlinearity of filter radiometer is critically involved and has to be corrected for when realizing thermodynamic temperature above the silver point, and its uncertainty should be adequately specified. The nonlinearity is measured based upon the superposition method and involving two water-cooling LEDs with high radiance output as radiation source. A new method, the step-mode operation with fixed phase shift of double LEDs, is introduced. The junction temperature of LEDs was controlled by the
water bath. The system error caused by the differences of quasi-linear drift between the LEDs involved, is corrected by means of a model, described in the paper. The results of the nonlinearity and its associated uncertainty, derived for the filter radiometer, are reported.


Formal pair-wise temperature differences, a comparison reference value (CRV) and degree of equivalence, between the UK (NPL), Chinese (NIM) and Spanish (CEM) national measurement institutes (NMIs), are calculated from the results of a prototype key comparison of the ITS-90 above the silver point using high temperature fixed points (HTFPs) of Co–C (1324°C), Pt–C (1738°C) and Re–C (2474°C). The local realizations of ITS-90 temperatures were assigned by NPL, NIM and CEM to their individual sets of HTFPs. NIM and CEM then transported their cells to NPL and the temperature was realized by all the cells determined in quasi-identical measurement conditions using a linear pyrometer. From these results the pair-wise temperature differences between the three institutes were established. In addition a non-weighted and a weighted CRV were calculated for the comparison and degree of equivalence estimated. The results show very close agreement of the ITS-90 as realized by the three institutes. It is argued that the ease of the comparison and the low uncertainties attained strongly suggest that HTFPs should be the scale comparison artefacts of choice when comparing primary realizations either of the ITS-90 or thermodynamic temperature.


In this paper, impurity parameters \( m_i \) and \( k_i \) have been calculated for a range of impurities I as detected in the eutectics Co–C and Pt–C, by means of the software package Thermo-Calc within the ternary phase spaces Co–C–I and Pt–C–I. The choice of the impurities is based upon a selection out of the results of impurity analyses performed for a representative set of samples for each of the eutectics in study. The analyses in question are glow discharge mass spectrometry (GDMS) or inductively coupled plasma mass spectrometry (ICP-mass). Tables and plots of the impurity parameters against the atomic number \( Z_i \) of the impurities will be presented, as well as plots demonstrating the validity of van’t Hoff’s law, the cornerstone to this study, for both eutectics. For the eutectics in question, the uncertainty \( u_{TE-Tliq} \) in the correction \( TE – Tliq \) will be derived, where TE and Tliq refer to the transition temperature of the pure system and to the liquidus temperature in the limit of zero growth rate of the solid phase during solidification of the actual system, respectively. Uncertainty estimates based upon the current scheme SIE–OME, combining the sum of individual estimates (SIE) and the overall maximum estimate (OME) are compared with two alternative
schemes proposed in this paper, designated as IE–IRE, combining individual estimates (IE) and individual random estimates (IRE), and the hybrid scheme SIE–IE–IRE, combining SIE, IE, and IRE.


The stability of the Co–C and Pt–C eutectic fixed points is investigated in this article. Six cells were employed for the testing. Two existing cells, a Co–C cell with a C/C sheet incorporated and a Pt–C cell equipped with a graphite sleeve that had been previously aged, were studied to determine the presence of anomalous drifts or degrading robustness that may possibly appear when the testing period is extended. The stability of four cells of a new design, a Co–C cell and three Pt–C cells, which were constructed in a hybrid manner, to include a sleeve, foil lining, and two of them with a cavity support, were likewise tested. Results show that different designs of Pt–C cells can exhibit good stability. However, for Co–C cells, the cavity rift remains a problem. The possible reasons for this rift and methods for increasing cell life are discussed.


Temperature Standard instruments were studied for the temperature sensor pair of heat meter. Two Grade B industrial platinum resistance thermometers (IPRTs) were tested. The experimental results showed that the stability of high precise IPRTs was 3mk in the temperature range from 0℃ to 90℃ per year. The resistance drifts of the IPRTs were eliminated by using W value calculation and the accuracy of the calibration was improved. This type IPRT can be used as the temperature standard for calibration.


Boping Wang, Wei Dong, Minchen Guo, Zundong Yuan. Numerical Simulation and Experimental Validation of The Four Channel Hot Air Gun Heating System. ICMMME
In the spectral emissivity measurement of material, one domain uncertainty source is the measurement of the actual sample surface temperature. This paper presents a novel method on heating the sample—the four channel hot air heating method, with the purpose of making sure that the heat flux is nearly equal to zero inside the sample, which is essential for the low thermal conductive materials. So the temperature difference between the sample surface and inside can be neglected. The FEA (Finite Element Analysis) method, through using FLUENT, was adopted to numerically simulate the temperature profile of the new heating system. The simulation results showed that temperature profile at the direction of X in ±15 mm ranges at the central area of exits of the air-guide duct is almost at ±5°C and at the direction of Y in ±15 mm ranges is nearly ±10°C. The temperature profile was experimentally studied by using thermocouples, the numerical simulation and experimental results were in good coherence.


Abstract: The spectral responsivity of the filter radiometer (FR) is an essential parameter which needs to be measured accurately in the radiometric determination of the thermodynamic temperature. In this paper the facility of the spectral responsivity measurement and a 900 nm centered FR are studied. The influence issues of the facility and the FR, including the central region, wings and the wavelength step, are discussed. The measurement repeatability of the integration of spectral responsivity is estimated 0.03%, which is suitable for the requirement.


Abstract The size-of-source effect (SSE) of radiation thermometers is generally determined using direct and indirect methods. The present paper describes the difference in the SSE obtained using the two methods. Three thermometers at different SSE levels, namely, $1.5 \times 10^{-4}$, $1.05 \times 10^{-3}$, and $2.44 \times 10^{-3}$, are selected. SSEs with 5mm to 96mm aperture diameters are higher in the indirect than in the direct method. Three factors contribute to this difference: the light reflection from the inside face and edge of the aperture to the black spot, the relay reflections from the thermometer and background, and the decreased radiance caused by obstruction of light by a black spot. Thus, the difference in the SSEs results from the difference in the principle of the two methods and not from the SSE levels of the thermometers themselves. The difference is negligible to be considered in a number of radiation thermometers, but they must be taken into consideration in top-level inter comparisons.

F. Edler, W. Zheng Investigation of different designed Pd-C eutectic fixed-point cells for the calibration of thermocouples 2011 Metrologia 48:275-282
An intercomparison of the melting temperatures of three palladium-carbon (Pd-C) eutectic fixed-point cells of different designs and usable for the calibration of thermocouples was performed at NIM. Two Pt/Pd thermocouples were constructed for this particular purpose. One of the Pd-C eutectic cells is of conventional design and size; the two other cells are miniature fixed-point cells of different design. The melting temperatures of two Pd-C cells agree within their measurement uncertainties for \( k = 2 \) of about 180 mK, the temperature of the second miniature cell was slightly lower than the melting temperature of the conventional cell by about 265 mK. Furthermore, a new aspect is presented of leakage effects often observed in using Pd-C cells.


Abstract Inconsistencies in the ITS-90 interpolations above 0.01 °C have been investigated. Thirty-five standard platinum resistance thermometers are used to determine the magnitude of inconsistencies in this range with the exception of the overlap region of the water–zinc and water–aluminum sub-ranges. The results show inconsistencies that are less 4.1 mK. Simpler representations of ten of the fifteen inconsistency functions above 0.01°C are presented by means of factoring and extremum analysis.


Abstract: Argon triple-point is not only one of the required most important reference fixed points on International Temperature Scale of 1990(ITS-90), but also one of the national primary fixed points of temperature. Argon triple-point device is the basic instrument in realizing argon triple-point. A new open-type argon triple-point device for calibrating long-stem standard platinum resistance thermometers(SPRTs) in quasi-adiabatic condition has been fabricated in National Institute of Metrology(NIM). A long plateau time of argon triple-point is achieved because there is sufficient argon gas injection and enough amount argon curing in the device. The new device successfully solves the problems of short plateau time and low resolution that exist in the design using continuous heat flux method. The measurement results obtained using a high accuracy temperature-measuring bridge show that, the plateau time at argon triple-point is over 26 hours, and the temperature variation is within 0.2 mK during a period of 16 hours. The reproducibility in the order of 0.04 mK is achieved. According to the measurement results of two long-stem SPRTs, the standard deviation is less than 0.05 mK and the uncertainty is better than \( 2 \times 10^{-7} \). This research improves the national level of argon triple-point reproducibility, and has great significance for the transfer of primary value of temperature and international comparison.

2010

Abstract: The subrange inconsistency is a significant factor to uncertainty in the standard platinum resistance thermometer (SPRT) subranges of the International Temperature Scale of 1990 (ITS-90). This paper investigated the subrange inconsistency between the water–zinc and water–aluminum subranges. The calibration data for 60 SPRTs from four manufacturers were analyzed, and the result confirms that the coefficient $c$ in the interpolation of ITS-90 is available to determine the subrange inconsistency in this temperature range again. The inconsistency, $\Delta t$, can be simply equal to $59.83c$.


Abstract Piezoelectric ceramics mounted on the endplates of a cylindrical resonator were used as the source and detector for speed-of-sound measurements. The perturbations of the longitudinal gas modes of the cavity due to the compliance of the diaphragms (10 mm diameter, 0.3 mm thick) and the attached transducers were estimated from first-order perturbation theory. The fractional shift of the resonance frequencies in argon caused by the source and detector was $0.03 \times 10^{-6}$ at 0.1 MPa and 273.16 K. The high signal-to-noise ratio (up to $1 \times 10^4$ with a 6 s integration time) that was obtained with these transducers makes them suitable for acoustic thermometry. The heat dissipation in the source transducer was measured to be only $0.7 \mu$W at the working voltage (7 V) and frequency (1 kHz).


Abstract The progress towards re-determining the Boltzmann constant $k_B$ using two fixed-path, gas-filled, cylindrical, acoustic cavity resonators is described. The difference in the lengths of the cavities is measured using optical interferometry. Thus, a literature value for the density of mercury is not used, in contrast with the presently accepted determination of $k_B$. The longitudinal acoustic resonance modes of a cylindrical cavity have lower quality factors $Q$ than the radial modes of gas-filled, spherical cavities, of equal volume. The lower $Q$s result in lower signal-to-noise ratios and wider, asymmetric resonances. To improve signal-to-noise ratios, conventional capacitance microphones were replaced with 6.3 mm diameter piezoelectric transducers (PZTs) installed on the outer surfaces of each resonator and coupled to the cavity by diaphragms. This arrangement preserved the shape of the cylindrical cavity, prevented contamination of the gas inside the cavity, and enabled us to measure the longitudinal resonance frequencies with a relative standard uncertainty of $0.2 \times 10^{-6}$. The lengths of the cavities and the modes studied will be chosen to reduce the acoustic perturbations due to non-zero boundary admittances at the endplates, e.g., from endplate bending and
ducts and/or transducers installed in the endplates. Alternatively, the acoustic perturbations generated by the viscous and thermal boundary layers at the gas–solid boundary can be reduced. Using the techniques outlined here, $k_B$ can be re-determined with an estimated relative standard uncertainty of $1.5 \times 10^{-6}$.


A prototype comparison of the ITS-90, as realized by NPL, NIM, and CEM, using high-temperature fixed points (HTFPs) of Co-C (1324°C), Pt-C (1738°C), and Re-C (2474°C), is reported. The local realizations of ITS-90 temperatures were assigned by NPL, NIM, and CEM to their own set of HTFPs. NIM and CEM then transported their cells to NPL, and the ITS-90 temperatures of all three sets of cells were measured using a linear pyrometer. From these measurements, a comparison reference value (CRV) was derived. At the Co-C and Pt-C points, the deviation from the CRV was <0.1°C for all three institutes; at the Re-C point, the deviation was <0.4°C. These deviations are significantly less than the scale realization uncertainties ascribed by the individual institutes indicating that these uncertainty estimates are conservative and could be revised to smaller values. In addition, thermodynamic temperatures were determined for these HTFPs using the current value of the thermodynamic temperature for the copper point, namely, 1357.82K. Given the consistent performance of the HTFPs, they should be seriously considered as scale comparison artifacts of choice when comparing primary realizations of the ITS-90 and of the thermodynamic temperature.


Abstract This article proposes a correlation relation between the resistance ratios of the triple points of argon and mercury. By this relation, the resistance ratio of the triple point of argon can be extrapolated from that of mercury, and a deviation function which is defined in the range from 83.8058 K to 273.16 K can be determined from only the calibration values at the triple points of water and mercury. It is a close approximation to the ITS-90 deviation function in the subrange. Using it, the calibration at the triple point of argon can be saved. Twenty-five standard platinum resistance thermometers are used to check the function. The errors are less than 5 mK. It is sufficient for secondary measurements.


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. The results are compared with the melting temperatures measured using non-contact radiation 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, the uncertainties are comparable to those of non-contact radiation 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 radiation thermometry measurements.


Nonlinearity of photoelectric pyrometer is one of the main uncertainty sources for realization of international temperature scale. A novel nonlinearity measurement system based on LED light sources and flux addition method was established. The high brightness LEDs with nominal 645 nm peak wavelength were utilized as light source to substitute conventional tungsten lamp. The switch of power supply for LEDs can realize flux addition, and the abandonment of optical strobe can avoid the nonideal result from repeated reflection in the optical path. The experimental research on the spectral radiation drift of LEDs under step-mode power supply was carried out to validate the end time of nonlinear drift stage. The pulse-mode power supply of fixed phase shift for double LEDs and beam splitter were combined to attain flux addition. The linear drift stage of LEDs was chosen to measure the nonlinearity of pyrometer. The linear drift effect on the nonlinearity measurement was eliminated by exchange the measurement turn and experiments were carried out to validate the method. The nonlinearity of the standard radiation thermometer (model RT9032) was experimentally studied with the nonlinearity from 0.2×10^{-4} to 1×10^{-4}, and the photocurrent ranged from 1.1×10^{-11} to 2×10^{-7} A. The standard uncertainty of nonlinearity was better than 0.7×10^{-4} within the photocurrent range from 3.6×10^{-11} to 4.4×10^{-7} A, and 1.5×10^{-4} for else.

Wei Zheng , Xiang Ming-dong, Yu Deng-feng The Research on the Calibration
Abstract: The “mini-coil” method of preparing, and purity of Pd, melting rate of wire and oxidation of Pd as an important factor which could be influence the accurate of temperature measurement is considerate. The measurement repeatability of 0.6μV with the “mini-coil” method is better than traditional wire-bridge method. It shows about 1.5 ℃ lower of temperature measurement in oxidation than in Ar2 protection environment. The expanded uncertainty of measurement in calibration PtRh thermocouple is a round 0.8℃ (k=2).


Abstract: To obtain monochromatic light with high spectral efficiency and low level of stray-light, using TRIAX190 as short focal with Model207 as long focal length monochromator coupled in series connection in absolute spectral responsivity measurement facility of filter radiometer. The stray light’s level of this system can achieve 10-8, efficiency of spectral luminous reach 3%. Wavelength calibration for 207 monochromator which played a major role in characteristics of series connection double-monochromator. The experiments’ results show that the uncertainty of the monochromometer wavelength is less than 0.02nm and the repeatability of 0.01nm.

Yang Jiajian, Lu Xiaofeng, Feng Xiaodong, Yuan Zundong, Wavelength calibration system of the grating monochromator based on LabVIEW, 2010 ELECTRONIC MEASUREMENT TECHNOLOGY, 33(4): 78~81

Abstract: The new wavelength calibration system of the grating monochromator was described to research the wavelength characteristic of Model 207 monochromator which was used in the spectral responsivity measurement. The automatic parts of the facility were all connected with the computer by RS232 or IEEE-488. The wavelength calibration could he carried out automatically, and the monochromometer’s performance was also tested. At fast the method of processing acquisition data was introduced in the program. The experiment results show that the accuracy of the monochromometer wavelength is 0.02 nm.

2009


Abstract We examined the perturbations of modes of an acoustic thermometer caused by circular ducts used either for gas flow or as acoustic waveguides coupled to remote transducers. We calculate the acoustic admittance of circular ducts using a model based on transmission line theory. The admittance is used to calculate the perturbations to the resonance frequencies and half-widths of the modes of spherical and cylindrical acoustic resonators as functions of the duct’s radius, length, and the locations of the
transducers along the duct's length. To verify the model, we measured the complex acoustic admittances of a series of circular tubes as a function of length between 200Hz and 10kHz using a three-port acoustic coupler. The absolute magnitude of the specific acoustic admittance is approximately one. For a 1.4mm inside-diameter, 1.4m long tube, the root mean square difference between the measured and modeled specific admittances (both real and imaginary parts) over this frequency range was 0.018. We conclude by presenting design considerations for ducts connected to acoustic thermometers.


Abstract National Institute of Metrology (NIM) (China) and National Physical Laboratory (NPL) (UK) have collaborated to construct metal-carbon eutectic alloy fixed points at NPL. A modified NPL Thermogaue furnace was vertically used to construct fixed points of Pd–C, Pt–C, Ru–C, and Ir–C. Breakage of Pd–C and Ru–C cells was traced to changes in furnace temperature gradients resulting from changing from horizontal to vertical operation. Subsequently, it was found that positioning the cell being filled so that the metal melting always starts from the top and freezing from the bottom to solve this problem. The constructed Pt–C cell was then compared to a Pt–C fixed point previously constructed by NIM. The results indicate that the two cells made independently agreed to be better than 40mK.


Abstract: The continuous heat flux method for realizing the aluminum freezing point using a sodium heat pipe furnace is described. The depression of aluminum freezing temperature is less than 0.6 mK within three hours after the freezing temperature arrive at the maximum. Also, it is necessary to form one inner and one outer solid-liquid interface when realizing the aluminum freezing point at the highest
level of accuracy. The realized freezing temperature plateau will fluctuate if the inner solid—liquid interface is destroyed. Moreover, it is very helpful to postpone this phenomenon by inserting a silica glass tube at the room temperature into the thermometer well.

Yan Xiaoke, Lv Zhuofan, Ma Chongfang, Li Jie, Duan Yuning. High Precise Sodium Heat-Pipe Fixed Point Furnaces and Their Isothermal Characteristics ACTA METROLOGICA SINICA 2009 30(6): 489-492
Abstract: Two sodium heat-pipe fixed point furnaces (SHPF-1, SHPF-2) developed at the National Institute of Metrology (NIM) are described, and their vertical temperature uniformities are measured. When the temperatures of the these two furnaces are controlled about 2 °C and 3 °C respectively, below the freezing point of the aluminum, the largest temperature differences did not exceed 15 mK and 11 mK in a distance about 150mm along the reentrant well of the aluminum point cell, respectively. These temperature uniformities are compared with those of foreign similar sodium heat pipe furnaces. Additionally, factors influencing isothermal characteristics of fixed point furnaces are analyzed.

2008
J T Zhang1 and S Q Xue Investigation of the imperfection effect of correlation on Johnson noise thermometry 2008 Metrologia 45: 436–441
Abstract Johnson noise thermometry comes with many imperfection effects that arise primarily from electromagnetic interference, the non-linearity of the electronic system, the transmission line error, etc. Current Johnson noise thermometers (JNTs) operate according to the principle of correlation by which interference noises would be rejected completely. Unfortunately, correlators of JNTs are observed to perform imperfectly in the elimination of these imperfection effects. This paper presents the direct imperfection effect measurements of the JNT of the National Institute of Metrology at the melting point of gallium. The result of these measurements implied that the imperfection effect of correlation acted as an equivalent input noise equally distributed on both probes of the JNT. A relation was derived in this paper to treat this equivalent input noise as a systematic error to Johnson noise thermometry. As a result, the systematic error arising from the measured imperfection effect was corrected from the measured thermodynamic temperature of the melting point of gallium.

Abstract In 2005, an agreement was reached on how to estimate uncertainties and how to correct fixed-point temperatures for the influence of chemical impurities. Although the general procedure is now specified, some problems remain. The slope of the liquidus line at very low-impurity concentrations must be extracted either from binary-phase diagrams or from doping experiments. Apart from this, there is little experimental evidence to prove that the models used to characterize the freezing and the melting
plateaux are adequate, especially for impurities that increase the fixed-point temperature. Therefore, a series of measurements were carried out using a tin fixed-point cell doped with antimony. By varying the freezing and the melting conditions, some useful experimental data were collected.


Abstract The formation of crystallization centers from extremely pure molten tin is normally associated with deep supercooling. This deep supercooling is inconvenient for the operation of tin freezing-point cells, especially for sealed tin fixed-point cells without a holder to facilitate removal from the furnace. Researchers of the National Institute of Metrology (NIM) intended and succeeded in reducing this deep supercooling by adding fine and pure graphite powders to tin fixed-point cells without influencing the fixed-point temperature, but the mechanism is yet to be properly clarified. The principle of heterogeneous nucleation indicates that a decrease of the contact angle of the crystalline nucleus on the substrate surface results in a significant reduction of supercooling required for initiation of nucleation. The heterogeneous theory is utilized by the authors of this paper to give a reasonable description of the mechanism of supercooling reduction by addition of graphite powder. It is demonstrated that the freezing plateau can be realized by the natural cooling of the tin cell within the furnace without using the ‘outside nucleation’ technique. The maximum temperature of the freezing curves of the tin cell with graphite powder agrees well with the reference tin cell without the graphite powder, and the cells with graphite powder show good consistency.


Abstract: As the triple point of water is of great importance for the International Temperature Scale of 1990 (ITS-90) and for the definition of the unit of thermodynamic temperature, its long-term stability has attracted a great deal of attention. In a study of long-term stability, a mystery has been uncovered. Some triple-point-of-water cells remain stable for many decades, while others decrease with increasing age of the cells, which is called long-term drift. To investigate this mystery, we used cells with different manufacture dates ranging from 1974 to 2002 and compared their analyses, which were done in 1984 and 2003. Using the same model of long-term drift as that used by Hill, the long-term drift rates of the two data sets are $-4.7 \mu \text{K}\cdot\text{year}^{-1}$ and $-9.2 \mu \text{K}\cdot\text{year}^{-1}$, respectively. One is consistent with the observed depression of about $-4 \mu \text{K}\cdot\text{year}^{-1}$ measured by Hill, whereas the other differs greatly from Hill’s result. In addition, corresponding factors influencing long-term drift are discussed in this paper.

Abstract: An investigation into the effects of isotopic composition on the triple point temperature of water has been carried out at the National Institute of Metrology (NIM), China, since redefinition of the kelvin with respect to Vienna Standard Mean Ocean Water (V-SMOW) was officially proposed by the Consultative Committee for Thermometry (CCT) in 2005. In this paper, a comparison of four cells with isotopic analyses and relevant results corrected for isotopic composition, employing the isotope correction algorithm recommended by the CCT, is described. The results indicate that, after application of the corrections, the maximum temperature difference between the cells drops from 0.10mK to 0.02mK and that these cells are in good agreement within 0.02mK. Also, temperature deviations arising from isotopic variations fall in the range from −55.9μK to +40.7μK. We consider that the distillation temperature and degassing time of the production procedure lead to isotopic variations.

W Zheng , Y Yamada, Y Wang The experimental investigation of Cr_3C_2-C Peritectic fixed point 2008 International Journal of Thermophysics 29:935-943

The authors have recently reported the very first radiometric plateau observation of high-temperature fixed points of metal-carbide carbon peritectics. These act similar to the metal (carbide)-carbon eutectic points in the sense that they can be used at high temperature without reacting with the graphite crucibles. The performance seems similar, or maybe even better, in terms of repeatability and plateau shape. The temperature range coincides with some of the metal (carbide)-carbon eutectics. In this paper, we show results of further study to understand the melting and freezing involved in these fixed points, with focus on the Cr_3C_2-C peritectic point. Due to low surface energy of the molten metal, we encountered difficulty in filling an ingot without voids. To overcome this, we devised a filling technique utilizing capillary effect. We compared plateau shapes of various filling methods, in conjunction with microstructure observation with EPMA. The observation of two fixed point plateaus, one at the Cr_3C_2-C peritectic point and the other at a lower temperature of the Cr_7C_3-Cr_3C_2 eutectic point, is seen to correspond to two different domains observed. The graphite crucible is shown to play an essential role in realizing peritectic plateaus of good quality.


In previous investigations, it was shown that the shape of the melting curve for Fe-C eutectic fixed points depends not only on impurity content but also on freezing and annealing conditions. The change of eutectic structure would be correlated with the change in shape of the melting curve. From this point of view, the cross-section of the eutectic structure of Fe-C was analysed in 2D using the image analysis software “Image-J” for sixteen Fe-C eutectic ingots that were produced from a selected combination of four different materials with different impurity contents and five different freezing and annealing conditions. We found that the structure of Fe-C eutectic depends on freezing rate and annealing time. Average distance between graphite
particles $\lambda$ versus $(T_E - T_{\text{furnace}})^{-1/2}$ and $\lambda$ versus $v^{-1/2}$ were compared with theoretical prediction, where $T_E$ is the eutectic transition temperature, $T_{\text{furnace}}$ is the furnace set point during the freeze, and $v$ is the velocity.

YAN Xiaoke, WU Helian, ZHANG Jintao, MA Chongfang, DUAN Yuning. Influences of Water Purification on the Triple Point Temperature of Water 2008 ACTA METROLOGICA SINICA 29 (5A): 155-159
Abstract: To investigate the effects of water purification on the triple point temperature of water, three TPW cells having different numbers of repeat distillations of de-ionized water were fabricated in light of our usual production process. Also, we carried out the direct comparison experiments among the cells, adopting the same freezing method and measurement procedure. The results show that triply-distilled water caused the realized triple point temperature to drop as much as 0.10mK. Additionally, based on our analysis and the results of the Consultative Committee for Thermometry (CCT) key comparison of TPW cells (CCT-K7), it can be concluded that once-distilled water used during manufacture of the cells is adequate to ensure a high quality of the triple point of water cells.

Abstract: This paper presents an automated ice mantle maker based on heat pipe technology and the procedure for automatic forming an ice mantle. Also, we investigated effects of this method on the temporal behavior of the triple point temperatures. Our results indicate that beyond 24 hours after preparation of the ice mantle, the temperatures of triple point of water (TPW) measured during six days are in good agreement within 0.05mK. Therefore, this new method is capable of satisfying the demands for the most accurate realization of TPW. Moreover, compared with the others, it can be concluded that strains introduced during preparation of the ice mantle are rather smaller.

Abstract: The size-of-source effect (SSE) describes the contribution to the measured signal from the surroundings of target area. It’s one of the most significant and difficultly determined properties of radiation thermometers. The objective lens is one of the dominant effects that influence SSE. In this paper, the characteristics, cleanliness and placement of objective lenses were mainly studied. We describe the efforts how to choose and clean the objective lens for the less SSE. The relationship between the aperture stop and objective lens is also discussed. It’s shown that SSE can be reduced to $2 \times 10^{-4}$ in our radiation thermometers as measured using a 50mm diameter radiance source with a 4mm diameter central obscuration.
Wei Dong, Tiejun Wang, Chengyu Bai, Yuanyuan Duan. Experimental research on the melting behavior of Pt-C eutectic fixed point affected by thermal history. *ACTA Metrologica Sinica*, 2008, 29(5A): 129-133.

Previous research indicates that melting behavior of the metal carbon eutectic fixed points (MCs) can be affected to different extent by thermal history such as previous freezing rate of the cooling process and the pre-annealing condition. This paper deals with the experimental research on the melting behavior of different structural Pt-C cells affected by the thermal history. Two Pt-C fixed point cells were chosen, the “sleeve” type cell (named Pt-C 2#) and the “two layer C/C sheet + sleeve” type cell (named Pt-C 4#). Least square fit of 5 order polynomials were employed to characterize the melting plateaus and to obtain TE (the point of inflection of the melting plateau). The effect of thermal history on the subsequent melting plateaus was distinct for the different structural Pt-C cells, for the “sleeve” type cell the pre-freezing rate effect was more remarkable. The different structure might account for the variation.

The pre-freezing rate varied from (TE-5) oC, (TE -10) oC, (TE -20) oC to (TE -40) oC, and the subsequent melting rate was set at the same (TE+20) oC, the maximal change of the melting temperature was 43 mK for the “sleeve” type cell and 12 mK for the “two layer C/C sheet + sleeve” type cell. The 4 h pre-annealing at (TE -20) oC flattened and prolonged the melting plateau for both the two type cells, with TE increased 45mK for “sleeve” type cell and no obvious change for the “two layer C/C sheet + sleeve” type cell.


2007


Abstract: The spectral responsivity is the primary parameter of photoelectric pyrometer and other radiation thermometry. A new setup of spectral responsivity measurement at NIM was described. Through the ports of RS-232 or IEEE-488 the automatic parts of
the facility were connected with the computer. The wavelength calibration and spectral responsivity measurement could be carried out automatically by the program written in VB. The spectral responsivity of photoelectric pyrometer in the peak zone was measured.

Abstract: The spectral responsivity of the primary standard pyrometer (PSP) is an important parameter for realizing ITS-90 above silver point. An improved monochrometer-based facility for measuring spectral responsivity of PSP has been developed at the National Institute of Metrology (NIM). The spectral responsivity of PSP is measured as a system. The wavelength calibration and the spectral responsivity measurement can be completed automatically. The spectral responsivity from 600 nm up to 1200 nm of PSP was measured in this article. The effective wavelength of PSP was calculated and its uncertainty evaluation was described.

Qiu Ping, Sun Jianping, Duan Yuning The Development of Thermal Diffusivity Measurement Facility Using the Laser Flash Method 2007 ACTA METROLOGIA SINICA 28(3A): 82-85
The development of the thermal diffusivity facility and its measurement method is described. By comparison with the reference material, the expanded uncertainty is not more than 5%.

Qiu Ping, WANG Yulan Evaluation of CCT Comparison of Aluminium and Silver Freezing Point Cells 2007 ACTA METROLOGIA SINICA 28(4): 400-403
The CCT key comparison 4 of aluminium and silver freezing points is introduced. The comparison was carried out by means of direct comparison of fixed point cells. Eleven national laboratories participated the comparison. The organization, procedure and determination of key comparison value are presented.

2006
J T Zhang1 and S Xue A noise thermometry investigation of the melting point of gallium at the NIM 2006 Metrologia 43: 273–277
Abstract This paper describes a study of the melting point of gallium with the new NIM Johnson noise thermometer (JNT). The new thermometer adopts the structure of switching correlator and commutator with the reference resistor maintained at the triple point of water. The electronic system of the new thermometer is basically the same as the current JNT, but the preamplifiers have been improved slightly. This study demonstrates that examining the characteristics of the noise signals in the frequency domain is of critical importance in constructing an improved new thermometer, where a power spectral analysis is found to be critical in establishing appropriate grounding for the new thermometer. The new JNT is tested on measurements of the
thermodynamic temperature of the melting point of gallium, which give the thermodynamic temperature of 302.9160 K, with an overall integration time of 190h and a combined standard uncertainty of 9.4 mK. The uncertainty analysis indicates that a standard combined uncertainty of 3 mK could be achieved with the new thermometer over an integration period of 1750 h.