

Experimental determination of Boltzmann's constant

The international Boltzmann project – the contribution of the PTB

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Abstract

To support new determinations of the Boltzmann constant, which have been requested by the International Committee for Weights and Measures (CIPM) and which are necessary for preparative steps towards new definitions of the kilogram, the ampere, the kelvin and the mole, an iMERAPlus joint research project is coordinating the European activities in this field in Spain (CEM), Denmark (DFM), France (LNE-INM/CNAM, University Paris North), Italy (INRiM, Universities of Naples and Milan), United Kingdom (NPL), Germany (PTB) and in the European Institute for Reference Materials and Measurements (IRMM). In this major European research project, the Boltzmann constant will be determined with various methods. The aims and the progress to date of the PTB contribution are reviewed in this article. *To cite this article: B. Fellmuth et al., C. R. Physique 10 (2009).*

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Résumé

Le programme international de détermination de la constante de Boltzmann : contribution du Physikalisch–Technische Bundesanstalt. Le Comité international des poids et mesures (CIPM) a recommandé de procéder à de nouvelles déterminations de la constante de Boltzmann en vue de préparer les nouvelles définitions des unités liées au kilogramme, à l'ampère, au kelvin et à la mole. Un projet de recherche concerté iMERAPlus a été mis en place afin de coordonner les travaux engagés sur ce sujet par l'Espagne (CEM), le Danemark (DFM), la France (LNE-INM/CNAM, Université Paris-nord), l'Italie (INRiM, Universités de Naples et Milan), le Royaume-Uni (NPL), l'Allemagne (PTB) et l'Institut européen de matériaux de références et mesures (IRMM). Les travaux engagés par ces laboratoires reposent sur des principes diversifiés et mettent en oeuvre des techniques très variées. Cet article présente les objectifs, les progrès et l'état d'avancement de la contribution de la PTB. *Pour citer cet article : B. Fellmuth et al., C. R. Physique 10 (2009).*

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1. Overview of the project

The unit of temperature T , the kelvin, is presently defined by the temperature of the triple point of water. Thus, the kelvin is linked to a material property. Instead, it would be advantageous to proceed in the same way as with other units:

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Table 1

Relative uncertainty of determining the Boltzmann constant k applying different methods of primary thermometry and institutes developing these thermometers.

Method	2nd WS 2006	3rd WS 2008	2010 possibility	Institute
AGT	2 ppm	–	1 ppm	INRiM, NIST
QSCR	> 20 ppm	> 3 ppm	1 ppm	CEM, LNE, NPL
RIGT	300 ppm	9 ppm	5 ppm	NIST
DCGT	15 ppm	–	2 ppm	PTB
JNT	–	25 ppm	6 ppm	NIST
DBT	200 ppm	44 ppm	10 ppm	DFM, Uni Paris North, Uni Naples and Milan

to relate the unit to a fundamental constant and fix its value. By this no temperature value and no measurement method would be favoured. For the kelvin, the corresponding constant is the Boltzmann constant k , because temperature always appears as thermal energy kT in fundamental laws of physics. For fixing the value, the present value of k and its uncertainty need to be checked and improved using several independent measurement methods.

To encourage new determinations of the Boltzmann constant, the Consultative Committee for Thermometry recommended “that national laboratories initiate and continue experiments to determine values of thermodynamic temperature and the Boltzmann constant”, which is also asked for in the recent recommendation of the CIPM concerning preparative steps towards new definitions of the kilogram, the ampere, the kelvin and the mole [1].

As one result of a workshop held at PTB in 2006 [2], an iMERAPlus joint research project is now coordinating the European activities to determine the Boltzmann constant in Spain (Centro Español de Metrología, CEM), Denmark (Danish Fundamental Metrology, DFM), France (Institut National de Métrologie, LNE-INM/CNAM, and University Paris North), Italy (Istituto Nazionale di Ricerca Metrologica, INRiM, and Universities of Naples and Milan), United Kingdom (National Physical Laboratory, NPL), Germany (Physikalisch–Technische Bundesanstalt, PTB) and in the European Institute for Reference Materials and Measurements, IRMM [3]. In this major European research project, the partners use different methods to determine the Boltzmann constant aiming at a relative standard uncertainty on the level of a few ppm. These new values will be compared with the present CODATA value of k [4] determined to a very large extent by acoustic gas thermometry of the National Institute of Standards and Technology (NIST, USA) [5]. The recommended value has a relative uncertainty of $u_r(k) = 1.7 \times 10^{-6}$.

Worldwide many projects have been started to measure independently the value of the Boltzmann constant [6]. These are acoustic gas thermometry (AGT) with spherical or quasi-spherical cavity resonators (QSCRs) [5,7,8], dielectric-constant gas thermometry (DCGT) [9] and refractive index gas thermometry (RIGT) [10]. Other promising methods are Doppler-broadening thermometry (DBT) [11,12] and Johnson noise thermometry (JNT) [13]. At the National Institute of Metrology (NIM, China) and the National Research Council (NRC, Canada), AGT is under development. Table 1 gives a summary overview of the potential of the currently available relevant primary thermometers, as deduced from the 3rd workshop on determining k held in 2008 at LNE-INM/CNAM [14] and from the 2nd workshop held at PTB in 2006 [2]. Within the next two years, the possibility exists of achieving a relative uncertainty of order one part in 10^6 (1 ppm) based on measurements applying different methods. Thus, an improved value of k would ideally have been determined by at least the two different methods AGT and DCGT and be corroborated by other measurements as DBT with larger uncertainty.

Since primary thermometers are not applicable for most practical purposes, it will also be necessary in the future to refine the International Temperature Scale. Fixing the value of k means, on the other hand, that it is no longer possible to fix the temperature value of a phase transition used as a fixed point, e.g. the triple point of water (TPW). Then, all temperature reference values including that of the TPW have to be determined by primary thermometry (for details see [6]).

2. Dielectric-constant gas thermometry

The basic idea of DCGT pursued at PTB is more extensively discussed in [6]. In brief, the density in the well-known state equation of a gas is replaced by the dielectric constant ε and is measured by incorporating a capacitor in the gas bulb. The dielectric constant of an ideal gas is given by the relation $\varepsilon = \varepsilon_0 + \alpha_0 N/V$, where ε_0 is the exactly

known electric constant, α_0 is the static electric dipole polarizability of the atoms, and N/V is the number density, i.e. the state equation of an ideal gas can be written in the form $p = kT(\varepsilon - \varepsilon_0)/\alpha_0$. Absolute DCGT requires knowledge of the static electric dipole polarizability α_0 with the necessary accuracy. Nowadays this condition is fulfilled for ^4He . Recent progress has decreased the uncertainty of the ab initio value of α_0 well below one part in 10^6 [15]. The molar polarizability A_ε is defined as $A_\varepsilon = N_A\alpha_0/(3\varepsilon_0)$, thus the Boltzmann constant is related to α_0 , A_ε and the gas constant R by

$$k = \frac{R}{A_\varepsilon} \frac{\alpha_0}{3\varepsilon_0}$$

The measurement of the ratio A_ε/R of two macroscopic quantities allows, therefore, the determination of k . For a real gas, the interaction between the particles has to be considered by combining the virial expansions of the state equation and the Clausius–Mossotti equation [16]. For determining A_ε/R , isotherms have to be measured, i.e. the relative change in capacitance $(C(p) - C(0))/C(0) = \chi + (\varepsilon/\varepsilon_0)\kappa_{\text{eff}} p$ of the gas-filled capacitor is determined as a function of the pressure p of the gas (χ is the dielectric susceptibility and κ_{eff} is the effective compressibility): The capacitance $C(p)$ of the capacitor is measured with the space between its electrodes filled with the gas at various pressures and with the space evacuated so that $p = 0$ Pa. A polynomial fit to the resulting p versus $(C(p) - C(0))/C(0)$ data points, together with the knowledge of the pressure dependence of the dimensions of the capacitor (effective compressibility κ_{eff}), yields A_ε/R .

New DCGT measurements were performed at PTB in the temperature range from 2.4 K to 26 K in order to establish a temperature scale with reduced uncertainty [17] compared with the first DCGT scale of PTB described in [16]. Progress has been achieved concerning the measurement of capacitance changes, temperature and pressure that resulted in a reduction of the overall uncertainty by about a factor of two. For the capacitance measurements, a more symmetric setup has been realised. Further improvements concern the parasitic impedances of the capacitance bridge, its resolution and the data acquisition. The reduction of the uncertainty of temperature and pressure measurement is based on better thermal conditions in the new cryostat and a calibration of the measuring devices that is fully traceable to the national standards.

The dataset used for the DCGT2 evaluation of 2007 [17] was based on more than thirty DCGT isotherms measured in the temperature range from 2.4 K to 26 K. The reduced final DCGT2 dataset of 2008 [18] consists of 27 isotherms in the temperature range from 3.7 K to 26 K. A standard uncertainty of the established new DCGT temperature scale ranging from 0.15 mK at 4 K to 0.4 mK at 26 K has been achieved, applying improved mathematical methods, see the detailed discussion in [17]. The high-accuracy data allowed us to show that primary thermometry using DCGT with ^4He as measuring gas can be performed only at temperatures down to 3.7 K. At lower temperatures, special effects occur that are suspected to be caused by an additional bosonic interaction between the ^4He atoms [9]. The comparison of the temperature-measurement results with literature data corroborates the thermodynamic accuracy of the scale, see Fig. 1. It is noted that the difference between the two evaluations of the new DCGT data for ^4He performed in 2007 [17] and in 2008 [18], respectively, is connected with the special effects below 3.7 K. For the fermionic measuring gas ^3He , no problems were observed. It corroborates also the value for the triple-point temperature of hydrogen of 13.80365(50) K at a deuterium content of 35 $\mu\text{mol/mol}$ that is based on the first DCGT data set [16]. This value yields a triple-point temperature of 13.8039 K for the recently prescribed SLAP (Standard Light Antarctic Precipitation) deuterium concentration of about 89 $\mu\text{mol/mol}$. The insert in Fig. 1 illustrates another important result of the performed DCGT experiments. The agreement between the experimental and theoretical results for the ratio of the polarizabilities of ^3He and ^4He is excellent, see the detailed discussion in [9]. Furthermore, the differences between the results obtained for the virial-coefficients and new theoretical ab initio values are well within the uncertainty estimates [18]. All these results support the potential of the DCGT method for determining the Boltzmann constant at the triple-point of water with the claimed uncertainty [6] as a basis for the new definition of the base unit kelvin.

Compared with the activities at low temperatures, the uncertainty has to be reduced by an order of magnitude. The determination of the Boltzmann constant at the triple point of water requires essential progress in different directions:

- High-precision measurement of pressures up to 7 MPa with a relative uncertainty of 1 ppm, which corresponds to an essential reduction of one of the major uncertainty contributions, see the next section.
- Measurement of capacitance changes with a relative uncertainty of 1 ppb applying a special home-made bridge, the main part of which is a 1:1 high-precision inductive voltage divider (IVD). The IVD has been already produced

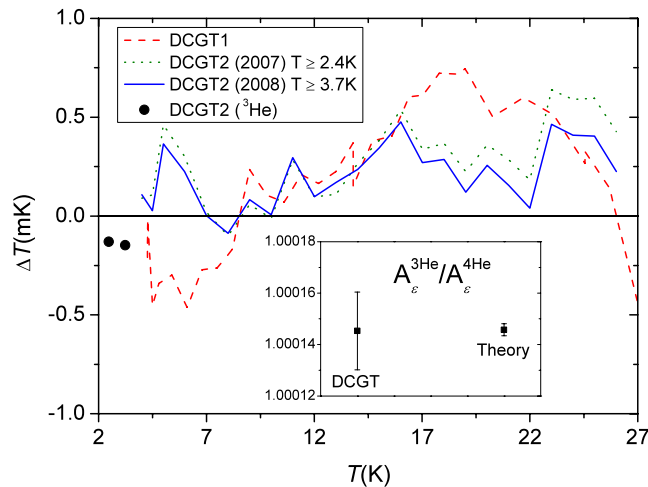


Fig. 1. Deviation of T_{fit} corresponding to different DCGT multi-isotherm fits from the ITS-90 ($\Delta T = T_{\text{fit}} - T_{90}$): Solid line: final results considering the new DCGT data for the measuring gas ^4He (DCGT2 [17]) in the range from 3.7 K to 26 K, see [18]. Dotted line: 2007 evaluation of all DCGT2 data as presented in [17]. Dashed line: first DCGT results (DCGT1) published in [16]. Thick dots: new data for ^3He . In the insert, experimental and theoretical results for the ratio of the polarizabilities of ^3He and ^4He are shown together with the combined standard uncertainties.

and tested. The tests yielded the needed parameters (absolute ratio-measurement in-phase error 6 ppb, out-of-phase error 1 ppm).

- Determination of the effective compressibility of the measuring capacitors on a corresponding level applying (i) novel methods for the characterisation of the properties of the capacitor materials (determination of the volume compressibility of the capacitor materials applying resonant ultrasound spectroscopy) and (ii) in-situ measurement of dimension changes as well as (iii) different new capacitor designs (special 10 pF cylindrical capacitors and multi-ring toroidal cross capacitors with up to 2×25 rings for 10 pF).
- Temperature measurement and control on a level of 0.1 mK in a very large measurement system having a high heat capacity and corresponding long thermal time constants.
- Application of high-purity gases (purity up to 99.99999%) including in-situ analysis by mass spectrometry.
- Check on the performance of the measurement equipment by primary measurements of the temperatures of other fixed points (triple point of mercury and melting point of gallium, the relation between the two fixed-point temperatures, which are very well known).
- Determination of the molar polarizability of alternative measuring gases as neon, argon and xenon against that of helium on an unprecedented level. Measurement of isotherms at the triple point of water using different gases.

3. Pressure measurement

Typical best pressure-measurement capabilities of recognised National Metrology Institutes (NMIs) were demonstrated e.g. in the key comparison CCM.P-K1.c [19], where differences between pressure balances of the participants of up to 25 ppm were observed in gauge operation mode. For this reason an absolute pressure measurement in the range up to 7 MPa with a relative standard uncertainty of 1 ppm, as required for the foreseen DCGT experiments, presents a challenging target. Pressure balances appear to be the only candidates for appropriate pressure standards. A feasibility analysis performed in [20] has identified the following major uncertainty sources: zero-pressure effective area (A_0), pressure distortion (λ) of pressure balances' piston–cylinder assemblies (PCAs), repeatability expressed in terms of the experimental standard deviation (type A uncertainty component).

From results of some NMIs reported for pressure balances at pressures around 0.1 MPa [21], the conclusion can be drawn that an essential improvement of cross-float measurements can be achieved when realising them in absolute mode. Therefore, a new concept of pressure balances has been developed that should allow measurements in absolute mode at pressures up to 7 MPa. These pressure balances will be equipped with remote piston loading systems enabling an adjustment of the desired pressure without breaking vacuum in a bell jar above the piston. Final small pressure differences between two cross-floated pressure balances during calibration by comparison will be measured

using accurate differential pressure transducers. Furthermore, high-precision dead weights will be used that meet the OIML R111-1 Class E₁ standard-weight requirements concerning shape, material's mechanic and magnetic properties, density and surface quality [22]. A total loading mass of 150 kg will allow PCAs with a relatively large nominal effective area of 2 cm² to be used. They will be traceable to 20 cm² PCAs, whose effective-area values will be based on dimensional metrology. Six PCAs in total, three of 20 cm² and three of 2 cm² nominal effective areas, will be used to provide redundant experimental information for demonstrating their equivalence within 1 ppm.

The pressure-distortion coefficient λ , which describes the relative change of the effective area with pressure, becomes particularly important with increasing pressure. For free-deformation thick-wall PCAs made of tungsten carbide, λ has an order of 10^{-6} MPa⁻¹. Intensive research was carried out in the last years to develop models for the calculation of λ and the estimation of its uncertainty [23,24] that until now were applied to oil-operated PCAs only. The determination of λ in the case of gas-operated PCAs requires a new consideration because the pressure distribution along the piston–cylinder clearance differs significantly from that in the case of oil-operated PCAs. Four European NMIs, PTB, INRiM, LNE and CMI (Czech Metrology Institute), are working on the problem within the EURAMET joint research project No. 1039 [25]. The research includes a finite element analysis of the elastic distortion in PCAs using PCA materials' elastic constants, which are measured by resonance ultrasound spectroscopy, and the pressure distribution in the piston–cylinder clearance that is obtained by hydrodynamic calculations taking into account the real dimensions of the piston and cylinder.

The zero-pressure effective area A_0 is the main PCA property, whose uncertainty causes the biggest contribution to the pressure uncertainty. In the case of primary PCAs, it is determined from their dimensional properties. Usually this is done by applying the equations derived by Dadson et al. [26]. The uncertainty of A_0 obtained in this way depends directly on the uncertainty of the dimensional data, but it may be also in error due to the assumption of the theory that the fluid flow in the clearance is one-dimensional and viscous. The progress in dimensional measurement techniques [27,28] and in processing the obtained dimensional data [29] seems to allow a reduction of the uncertainty of A_0 to lower than 1 ppm. With a new two-dimensional flow model, which has been tested and will be published soon, it has become possible to exclude, compared with the uncertainty demands, the uncertainty caused by axial non-symmetry of PCAs. The effect of the gas-flow regime on A_0 is less evident. It is connected with the pressure distribution in the clearance and the resulting drag force acting on the piston. The literature information on this effect is inconsistent. From the problem analysis presented in [26,30], it follows that for an ideal cylindrical PCA with $h/r = 4 \times 10^{-5}$ ($h = R - r$, where r and R are the piston and cylinder bore radii along the clearance, respectively), the effect of the gas-flow regime on A_0 should not exceed 0.2 ppm. But in a later investigation [31], the effect evaluated for the same PCA parameters was found to be as high as 6 ppm in both absolute and gauge operation modes. In the same work, also experimental data are cited that seem to demonstrate the effect to be even higher: 35 ppm and 7 ppm for absolute and gauge operation mode, respectively.

Even although some of the assumptions used in the theory [31] appear to be not absolutely correct, we considered it because, among other theories, it furnishes the largest effect of the gas sort on the effective area, and thus yields the “worst-case estimate”. In addition, we extended this theory to PCAs of a real shape. The following equations for the axial pressure distribution in the annulus p_z and A_0 have been obtained:

$$p_z = p_1 + (p_2 - p_1) \frac{\int_0^z \frac{dx}{p_z h^3 + 8\eta c h^2}}{\int_0^l \frac{dx}{p_z h^3 + 8\eta c h^2}}$$

$$A_0 = \left[\pi(r_0^2 p_1 - r_l^2 p_2) + 2\pi r_0 \int_0^l p_z \frac{dr}{dz} dz + \pi r_0 \int_0^l h \frac{h p_z + 8\eta c}{h p_z + 3\pi \eta c} \left(-\frac{dp_z}{dz} \right) dz \right] / (p_1 - p_2)$$

In these equations, p_1 and p_2 are the pressures below and above the piston, η and c are the viscosity and mean molecular speed of the gas, r_0 is the piston radius at the lower end of the piston–cylinder assembly, l is the length of the assembly. As the integral expressions in the upper equation are dependent on p_z , the calculation of p_z has to be performed iteratively. The new approach was applied to a 10 cm² PCA, which is used at PTB as a primary gauge pressure standard in the range up to 1 MPa. In Fig. 2, the results of the calculation of A_0 performed for the purely viscous and molecular flow regimes as well as for the transition flow regime with the gases N₂, He and SF₆ are shown.

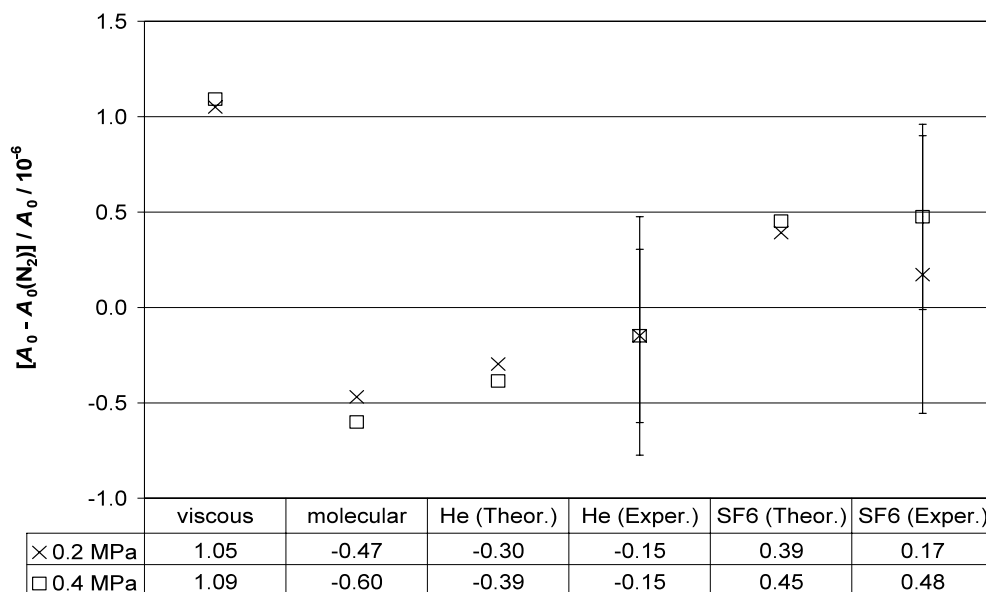


Fig. 2. Relative deviations of effective-area values (A_0), calculated for the purely viscous and molecular flow regimes as well as for the transient flow regime with He and SF₆, from the theoretical A_0 values obtained for N₂ ($A_0(\text{N}_2)$). Together with the standard confidence intervals, the relative deviations of experimental A_0 data measured with He and SF₆ from experimental values for N₂ ($A_0(\text{N}_2)$) are also shown.

The theoretical results obtained for N₂ are used as reference. For the comparison with experimental data, N₂ was also used as reference.

The theoretically calculated deviations have the same directions as the experimentally found ones, although the experimentally measured effects are insignificant compared with their uncertainties. These theoretically and experimentally found influences of the gas sort on the effective area are much lower than reported in [31], but they are comparable with the target uncertainty of 1 ppm. In fact, the differences in the effective area calculated for the molecular, viscous and transition flow regimes of real gases are expected to be much smaller than presented in Fig. 2. This follows from a new theory based on solutions of the rarefied gas dynamics to be published soon [32]. This new theory will be applied to the special 7 MPa pressure balances, which will be used in the DCGT experiments for the measurement of the Boltzmann constant.

4. Conclusions

Measurements of the thermodynamic temperature in the low-temperature range have impressively demonstrated the potential of the DCGT method for primary thermometry and thus for the determination of the Boltzmann constant. The high accuracy of the obtained results has been verified by comparison with literature data for the temperature scale as well as the virial coefficients and the polarizability of the measuring gas helium. The extreme sensitivity of the measurements allowed for the first time to verify a special behaviour of the bosonic ⁴He atoms below 3.7 K even in the gas phase. A detailed analysis of the experiments at low temperatures allowed us to define the directions, in which an essential progress is necessary for the determination of the Boltzmann constant at the triple point of water. First steps are outlined in the paper.

Pressure measurement causes a major uncertainty contribution in performing DCGT. With specially designed pressure balances and new techniques for measuring the dimensions of their piston–cylinder assemblies, preconditions have been created for absolute pressure measurements in the 7 MPa range with a relative standard uncertainty of about 1 ppm. The gas-sort influence on the effective area in the transient flow regime was found to be important, because it may reach the order of 1 ppm. The study of all effects should be extended to a pressure of 7 MPa in the absolute operation mode and supported by cross-float measurements between the new pressure balances with appropriate different effective areas.

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