Determination of the Boltzmann constant using a quasi-spherical acoustic resonator

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The paper reports a new experiment to determine the value of the Boltzmann constant, $k_B = 1.3806477(17) \times 10^{-23}$ J K$^{-1}$, with a relative standard uncertainty of 1.2 parts in 10$^6$. $k_B$ was deduced from measurements of the velocity of sound in argon, inside a closed quasi-spherical cavity at a temperature of the triple point of water. The shape of the cavity was achieved using an extremely accurate diamond turning process. The traceability of temperature measurements was ensured at the highest level of accuracy. The volume of the resonator was calculated from measurements of the resonance frequencies of microwave modes. The molar mass of the gas was determined by chemical and isotopic composition measurements with a mass spectrometer. Within combined uncertainties, our new value of $k_B$ is consistent with the 2006 Committee on Data for Science and Technology (CODATA) value: $(k_{B_{\text{new}}} / k_{B_{\text{CODATA}}}) - 1 = -1.96 \times 10^{-6}$, where the relative uncertainties are $u_r(k_{B_{\text{new}}}) = 1.2 \times 10^{-6}$ and $u_r(k_{B_{\text{CODATA}}}) = 1.7 \times 10^{-6}$. The new relative uncertainty approaches the target value of $1 \times 10^{-6}$ set by the Consultative Committee on Thermometry as a precondition for redefining the unit of the thermodynamic temperature, the kelvin.

Keywords: acoustic resonance; Boltzmann constant; International System of Units; kelvin redefinition; microwave resonance; quasi-sphere

1. Introduction

This paper reports a new experimental determination of the Boltzmann constant ($k_B$). The value is deduced from measurements of the velocity of sound in argon, as in the work performed by Moldover et al. at the National Institute of Standards and Technology (NIST) in 1988 [1]. However, several fundamental modifications and improvements have been made to measure and control the parameters that influence the measurement of $k_B$ [2,3].

The aim of this work was to determine the value of $k_B$ with an improved accuracy. By obtaining this value, a definition of the kelvin based on this fundamental constant is possible, instead of the temperature of the triple point of water ($T_{\text{TPW}}$) [4,5].

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Figure 1. Comparison of the recent determinations of \(k_B\) with the 2006 CODATA recommended value \(k_B_{\text{CODATA}}\) [6]. The shaded area spans the uncertainty \(u(k_B_{\text{CODATA}})\). The experimental determinations by Pitre et al. [7] in 2009, by Sutton et al. [2] in 2009 and by Gavioso et al. [8] in 2010 were, respectively, obtained at the Laboratoire National de Métrologie et d’Essai-Conservatoire National des Arts et Métiers (LNE–CNAM), at the National Physical Laboratory (NPL) and at the Istituto Nazionale di Ricerca Metrologica (INRiM), by measuring the speed of sound in He and Ar with quasi-spherical resonators. The value from the study of Zhang et al. [9] was obtained with a cylindrical acoustic resonator.

The experimental result is

\[
k_B = 1.3806477(17) \times 10^{-23} \text{ J K}^{-1}(1.2 \text{ ppm}).
\]

This value is consistent with both the previous determinations and the Committee on Data for Science and Technology (CODATA) value [6], as represented in figure 1.

2. Principle of measurement

The present method is based on the determination of the universal gas constant, \(R\), from a series of measurements of the speed of sound \(c_a\) in a monatomic gas. Measurements were performed in a 0.5 l quasi-spherical cavity made of copper, at different pressures, along an isotherm line close to the temperature of the triple point of water, \(T_{\text{TPW}} = 273.16\text{ K}\).

The measurements of \(c_a\) depend on the pressure, and \(k_B\) is calculated as follows:

\[
k_B = \frac{R}{N_A} = \frac{A_r(\text{Ar}) \cdot M_u}{(5/3)T_{\text{TPW}} N_A} \lim_{p\to0} c_a^2(p, T_{\text{TPW}}), \tag{2.1}
\]

where \(c_a^2(p, T_{\text{TPW}})\) is the square of the speed of sound at pressure \(p\) and temperature \(T_{\text{TPW}}\). \(A_r(\text{Ar})\) is the relative atomic mass of argon (alternatively, we can use helium, and this term becomes \(A_r(\text{^4He})\)), \(M_u\) is the molar mass.
constant, \( N_A \) is the Avogadro constant and the factor \( 5/3 \) is the ratio \( \gamma = C_p/C_V \) of the specific heat capacities for dilute monatomic gases. The term \( \lim_{p \to 0}^* \) indicates that we estimate the terms independent from the pressure in \( c_n^2(p, T_{TPW}) \).

The velocity of sound in a gas can be determined experimentally by measuring acoustic resonances in a spherical cavity of known radius. The method involves the measurement of the resonance frequencies of standing waves in cavities of simple geometry. The acoustic fields of high-quality, non-degenerate modes of such cavities are experimentally well-defined, theoretically well understood [10,11], and can be achieved with a mechanically simple apparatus [12]. The speed of sound is related to the following elements: resonance frequencies, \( f_{n,l}^A \), the corrections \( \Delta f_{n,l}^A \) taking into account the effects of the cavity and the non-ideality of the gas; and the eigenvalues \( Z_{n,l}^A \) for the wavenumbers \((n, l)\) in the acoustic cavity. In the particular case of a spherical cavity of radius \( a \), we have

\[
c_a(p, T_{TPW}) = \frac{f_{n,l}^A(p, T_{TPW}) + \Delta f_{n,l}^A(p, T_{TPW})}{Z_{n,l}^A} 2\pi a(p, T_{TPW}). \tag{2.2}
\]

A microwave technique can be used in combination with the acoustic measurements to determine the cavity dimensions as a function of temperature and pressure [7,13]. This experimental technique allows the measurement of the thermal expansion of spherical cavities in acoustic thermometry, and has successfully been applied in several experiments [14–19].

The radius \( a(p, T_{TPW}) \) of the cavity can be obtained using

\[
c(p, T_{TPW}) = \frac{f_{n,l}^{EM}(p, T_{TPW}) + \Delta f_{n,l}^{EM}(p, T_{TPW})}{Z_{n,l}^{EM}} 2\pi a(p, T_{TPW}), \tag{2.3}
\]

where \( c(p, T_{TPW}) \) is the speed of light in the gas at pressure \( p \) and 273.16 K, \( f_{n,l}^{EM} \) are the measured electromagnetic resonance frequencies and \( Z_{n,l}^{EM} \) are the electromagnetic eigenvalues for the wavenumbers \((n, l)\) in the cavity. \( \Delta f_{n,l}^{EM} \) are corrections related to the non-ideality of the resonator walls.

A quasi-spherical geometry, a triaxial ellipsoid, has been preferred in order to remove the degeneracy associated with the microwave eigenfunctions [12,20]. A second-order correction of microwave eigenvalues [21] has been computed to deduce the radius \( a(p, T_{TPW}) \). Microwave and acoustic measurements were carried out simultaneously, and \( k_B \) was determined by combining equations (2.2) and (2.3) with equation (2.1),

\[
k_B = \left\lfloor \frac{3}{5} \frac{m}{T_{TPW}} \cdot \lim_{p \to 0}^* \left( \frac{2\pi a}{Z_{n,l}^A} \right)^2 \left( \frac{(f_{n,l}^A + \Delta f_{n,l}^A)^2}{\langle f_{n,l}^A \rangle} \right) \right\rfloor
\]

\[
= \left\lfloor \frac{3}{5} \frac{m \cdot c_0^2}{T_{TPW}} \left( \frac{Z_{n,l}^{EM}}{Z_{n,l}^A} \right)^2 \lim_{p \to 0}^* \left( \frac{(f_{n,l}^{EM} + \Delta f_{n,l}^{EM})^2}{\langle f_{n,l}^{EM} + \Delta f_{n,l}^{EM} \rangle} \right) \right\rfloor, \tag{2.4}
\]

where \( m \) is the atomic mass of the gas and \( c_0 \) is the speed of light in vacuum.

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Figure 2. Schematic of the acoustic and electromagnetic acquisition systems.

Table 1. Overall relative standard uncertainty on the Boltzmann constant acoustic determination performed in argon at LNE-CNAM.

<table>
<thead>
<tr>
<th>Uncertainty Source</th>
<th>Uncertainty Contribution on $k_B$ (parts in $10^6$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature measurements</td>
<td>0.3</td>
</tr>
<tr>
<td>Volume electromagnetic measurements</td>
<td>0.6</td>
</tr>
<tr>
<td>Acoustic measurements</td>
<td>0.8</td>
</tr>
<tr>
<td>Molar mass and gas purity</td>
<td>0.6</td>
</tr>
<tr>
<td>Repeatability over two isotherms</td>
<td>0.3</td>
</tr>
<tr>
<td>Combined uncertainty</td>
<td>1.2</td>
</tr>
</tbody>
</table>

Figure 2 shows a schematic of the complete measurement system, allowing the realization of simultaneous acoustic and electromagnetic measurements.

Table 1 lists the different contributions to the evaluation of the uncertainty in the measured value of $k_B$. Each item is related to the different quantities measured in equation (2.4) and is described in detail in the subsequent sections of this paper.

The main improvements with respect to the previous determination [1] come from temperature measurements and molar mass of gas measurements. The most innovative technique is the measurement of the volume of the resonator using electromagnetic microwaves. The corresponding contribution to the uncertainty is of a completely different nature from that of the pyknometry used by Moldover et al. [1]. The microwave technique requires an accurate model involving the geometry of the resonator, the perturbations from antennas and the penetration of microwaves into the copper shell. Finally, this new determination of the Boltzmann constant has been made using a realization of the newly refined definition of the TPW that takes into account the influence of the isotopic composition.

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3. Temperature control on the acoustic resonator

Acoustic measurements for the determination of the Boltzmann constant are carried out at the $T_{\text{TPW}}$, which is the most accurately realized parameter up to now, according to the present definition of the kelvin.

The acoustic resonator (named BCU3) was installed in a temperature-controlled thermostat operating at temperatures near $T_{\text{TPW}}$ and was kept in isothermal conditions, so that thermal gradients over its surface were minimized during the experiment. The system was conceived like a quasi-adiabatic calorimeter, with a thermally shielded experimental chamber kept under vacuum. This ensured that temperature drifts were small enough to generate only negligible thermal gradients. Figure 3 shows the layout of the thermostat and details of the experimental chamber.

Four capsule-type standard platinum resistance thermometers, calibrated at $T_{\text{TPW}}$, were used to map the temperature distribution and to determine the average temperature of the resonator. Several configurations were tested and temperature measurement results are shown in figure 4. The positions of two thermometers (1551 and HS135 in figure 4) were swapped in different runs of the experiment (configurations named C1 and C2 in figure 4), while the two others (1825277 and 229073 in figure 4) were unchanged. This helped in discovering possible thermal gradients over the shell, but no gradients were detected. Different
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Figure 4. Plot of $\Delta T - \Delta \overline{T}$ values and the associated standard uncertainty bars $u(\Delta T)$ for the four thermometers. Nine thermal maps of the resonator are shown (Tm1–Tm9). Measurements are carried out in configuration C1 or C2, with either argon (Ar) or helium (He), at different resonator average temperatures, $\overline{T}$.

Gas pressures were applied in the resonator (not explicitly mentioned in figure 4), and thermal maps were studied at several temperatures within a few tens of millikelvins from $T_{TPW}$. Additional checks were performed in helium after the end of the experiments in argon (one point reported in figure 4).

The control of the temperature results in an overall relative standard uncertainty contribution of $0.3 \times 10^{-6}$ on $k_B$ from the measurements performed with BCU3 in argon. This is equivalent to one-third of the temperature uncertainty contribution in the previous determination of $k_B$ [1].

4. Precise measurement of the volume of the resonator

In 1986, Mehl & Moldover [13] used first-order perturbation theory to prove that the mean eigenfrequency of a multiplet was independent of volume-preserving deformations, and suggested that the volume of an imperfect spherical resonator could thus be determined from its microwave spectrum. Several of our earlier papers have set the groundwork for this determination [2,3,12,17,22].

In this work, the average radius of BCU3 has been determined from measurements of the frequencies and half-widths of nine microwave triplets, corrected to take into account three effects: (i) the microwave penetration depth using measured half-widths of the resonances, as described by Sutton et al. [2], (ii) the inlet and outlet gas ducts and the two microwave antennas using the extensive study of the effects of probes and holes in a quasi-spherical resonator (QSR) performed by Underwood et al. [22], and (iii) the shape of the QSR, using second-order theory [21] and our measurements of the frequency splitting of the microwave triplets.
The determination of the equivalent radius and its uncertainty budget was accomplished in four steps.

— At 20°C, with flowing argon and using straight microwave antennas (probes), we measured the frequencies of five transverse magnetic (TM) mode microwave triplets while the microphone ports were closed with blank copper plugs. We corrected these frequencies to account for shape and probe perturbations using the models and the results in Underwood et al. [22].

— With loop antennas, we determined the electrical conductivity of the copper surface and established a bound on any dielectric layer on the surface, in the same experimental conditions (20°C and flowing argon) as those specified for the previous step.

— After having replaced the blank ports, we obtained the final equivalent radius of the QSR with the acoustic microphones and loop antennas installed on it (figure 5).

— We estimated the resonator’s compressibility on isotherms and measured the resonator’s thermal contraction upon cooling from 20°C to $T_{TPW}$. These determinations give access to the thermal expansion coefficient and to the compressibility of the resonator, which are useful to calculate the change in the resonator radius as a function of temperature and pressure.

The agreement obtained in QSR radius measurements between straight and loop antennas was the order of 0.05 parts in $10^6$.

Table 2 summarizes the budget of the uncertainties associated with the determination of the equivalent radius of the resonator, and hence that associated with the volume determination.

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In 1988, Mehl & Moldover [13] suggested that the volume of an imperfect spherical resonator could be determined from its microwave spectrum, although they could not achieve accurate measurements at that time. By applying
Table 3. Uncertainty budget associated with acoustic measurements for the determination of the Boltzmann constant. Details on each component are provided by Pitre et al. [23].

<table>
<thead>
<tr>
<th>uncertainty source</th>
<th>effect on $k_B$ (parts in $10^6$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>scatter among modes</td>
<td>0.35</td>
</tr>
<tr>
<td>thermal conductivity of argon</td>
<td>0.02</td>
</tr>
<tr>
<td>dispersion related to $1/p$ term in $c_a^2(p, T_{TPW})$</td>
<td>0.53</td>
</tr>
<tr>
<td>functional form of $c_a^2(p)$</td>
<td>0.39</td>
</tr>
<tr>
<td>shell perturbation</td>
<td>0.14</td>
</tr>
<tr>
<td>$A_3$ uncertainty</td>
<td>0.07</td>
</tr>
<tr>
<td>tubing acoustic impedance</td>
<td>0.23</td>
</tr>
<tr>
<td>flow effect</td>
<td>0.11</td>
</tr>
<tr>
<td>microphone impedance effect</td>
<td>0.05</td>
</tr>
<tr>
<td>pressure uncertainty</td>
<td>0.08</td>
</tr>
<tr>
<td>combined uncertainty</td>
<td>0.80</td>
</tr>
</tbody>
</table>

this technique now in our experiment, we have reduced the uncertainty associated with the determination of the volume involved in the present experiment by nearly a factor of 1.7 with respect to Moldover et al. [1].

5. Measurements and analysis of the acoustic data

For the acoustic measurements, the experimental techniques and data analysis methods are built on foundations established over two decades [1,11], and subsequently applied to temperature measurements from 7 to 552 K [17,24,25].

In our experiment, we measured the velocity of sound in argon inside our cavity from the measurement of the acoustic resonance frequencies, $f_{A,0,n}$. The wavenumbers $(0, n)$ refer to acoustic resonances having a radial symmetry. Nonlinear effects were negligible at the low sound pressure levels used in the experiment. A detailed description of the experiment will be published elsewhere [23].

Figure 6 shows the results obtained in two runs of the same experiment: one performed in May 2009 and one in July 2009. Acoustic modes from $(0,2)$ to $(0,9)$ were measured (modes $(0,6)$ and $(0,7)$ were perturbed by the coupling between the shell’s motion and acoustic resonances of the gas [25], and were not used in the determination of $k_B$; modes $(0,8)$ and $(0,9)$ were too noisy and unusable in the measurement of July 2009, and were discarded as well).

The relative standard uncertainty of $k_B$ related to the acoustic measurements is $0.80 \times 10^{-6}$, as detailed in Table 3. This uncertainty value is the same as that obtained in [1], but with a 0.5l cavity instead of a 3l cavity.

The main uncertainty component in Table 3 is the dispersion of the acoustic modes related to the term $1/p$ in the acoustic virial expansion of the squared speed of sound $c_a^2(p, T_{TPW})$ [23]. Physically, this effect is generally associated with the heat flux across the interface between the gas and the walls of the resonator [1], but it can also be related to other effects, such as impurities in the gas.
6. Control of the purity and measurement of the molar mass of the gas in the resonator

The composition of the gas used in this experiment was analysed by mass spectrometry at the Institute for Reference Material and Measurements (IRMM) of the European Joint Research Centre [26]. The analysis provided us with information on the isotopic ratio and the residual impurities in the gas.

Gas in the resonator is supplied by a controlled continuous flow from a gas-handling system (shown in figure 7). The continuous flow ensured that all the impurities, trapped inside the inner surface of the resonator and continuously desorbed during the experiment, are promptly evacuated and did not affect the gas composition. Flow and pressure control were regulated by mass flow controllers. Table 4 shows the properties of the gas.

The gas-handling system includes a purification step that consists of passing the gas through a cold trap and a getter. The argon flows from the manufacturer’s cylinder through the cold trap at a temperature near 100 K. The getter is a Valco helium purifier (HP2). The manufacturer claimed that if the total concentration of impurities at the inlet were less than 10 parts in 10^6 by mole fraction, then the concentration of impurities at the outlet would be less than 10 parts in 10^9 for H_2O, H_2, O_2, N_2, NO, NH_3, CO, CO_2 and CH_4. Other impurities removed include CF_4, CCl_4, SiH_4 and light hydrocarbons. The 100 K cold trap is used to remove some noble gases such as Kr and Xe. This purification system cannot remove neon and helium from the argon because these noble gases do not react with the getter and the temperature of the cold trap is too high to condense them.

Helium and neon are elements lighter than argon and they contribute only to a diminution of the average molar mass. As a result, the upper and lower bounds of the interval in which the best estimate of the molar mass lies are

Figure 7. Schematic of the gas-handling system.
gas properties

Table 4. Gas composition.

<table>
<thead>
<tr>
<th>isotopic ratios$^a$</th>
<th>standard uncertainty on isotopic ratios$^a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$r^{(36\text{Ar}/40\text{Ar})}$</td>
<td>0.0033460</td>
</tr>
<tr>
<td>$r^{(38\text{Ar}/40\text{Ar})}$</td>
<td>0.00063477</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>atomic mass$^b$</th>
<th>standard uncertainty on atomic mass$^b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$M_{40}$</td>
<td>39.962383123</td>
</tr>
<tr>
<td>$M_{36}$</td>
<td>35.9675451</td>
</tr>
<tr>
<td>$M_{38}$</td>
<td>37.9627324</td>
</tr>
<tr>
<td>total: $M_{\text{Ar}}^c$</td>
<td>39.9478051</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>impurities$^a$</th>
<th>standard uncertainty on impurities$^a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>N$_2$</td>
<td>&lt;2.00</td>
</tr>
<tr>
<td>O$_2$</td>
<td>&lt;0.150</td>
</tr>
<tr>
<td>H$_2$O</td>
<td>&lt;0.50</td>
</tr>
<tr>
<td>CO$_2$</td>
<td>&lt;0.50</td>
</tr>
<tr>
<td>H$_2$</td>
<td>not measured</td>
</tr>
<tr>
<td>THC</td>
<td>not measured</td>
</tr>
<tr>
<td>He</td>
<td>&lt;1.00</td>
</tr>
<tr>
<td>Ne</td>
<td>&lt;1.00</td>
</tr>
<tr>
<td>Kr</td>
<td>not measured</td>
</tr>
<tr>
<td>Xe</td>
<td>not measured</td>
</tr>
</tbody>
</table>

$^a$Measured by the IRMM.
$^b$Values and uncertainties from CODATA 2006 [6].
$^c$Calculated by combining isotopic ratio and atomic mass data.

not symmetric, and the evaluation of the final combined uncertainty cannot be performed with conventional uncertainty propagation techniques. Nevertheless, tools are available to take these components into account [27], especially when there is not enough information to choose an appropriate probability distribution. To evaluate the uncertainty on $k_B$ related to the presence of neon and helium, we used approximate evaluation techniques, after having considered the following elements.

— In similar measurements performed in the past [1,28], concentrations of neon and helium as high as the bounds in Table 4 were not observed. The NPL group intends to measure the presence of neon and helium with a detection level below $0.3 \times 10^{-6}$. To date, they have shown $x_{\text{Ne}} \leq 0.3 \times 10^{-6}$ [28].

— Our experiments performed with the getter and the cold trap show that the impurity bounds provided by the IRMM are overestimated by almost a factor 10 [23].

— The argon is produced by an air-liquefaction process, where helium and neon are present only in sub-part-per-million levels. During the production process, light impurities such as oxygen and nitrogen are
Table 5. Uncertainty contributions from gas properties on the determination of the Boltzmann constant. Details on each component are provided in Pitre et al. [23].

<table>
<thead>
<tr>
<th>uncertainty source</th>
<th>relative standard uncertainty on $k_B$ (parts in 10^6)</th>
</tr>
</thead>
<tbody>
<tr>
<td>isotopic composition</td>
<td>0.15</td>
</tr>
<tr>
<td>Avogadro constant</td>
<td>0.05</td>
</tr>
<tr>
<td>getter Allan deviation</td>
<td>0.02</td>
</tr>
<tr>
<td>cold trap Allan deviation</td>
<td>0.06</td>
</tr>
<tr>
<td>subtotal (combined expression)</td>
<td>0.16</td>
</tr>
<tr>
<td>presence of He</td>
<td>0.52</td>
</tr>
<tr>
<td>presence of Ne</td>
<td>0.29</td>
</tr>
<tr>
<td>combined uncertainty</td>
<td>0.60</td>
</tr>
</tbody>
</table>

removed; therefore, it is extremely plausible that most of the helium and neon are removed at the same time.

Finally, we applied flow rates ranging from 0.05 to $1.33 \text{ cm}^3 \text{s}^{-1}$ in order to detect possible outgassing in the sphere. No effects related to outgassing or to the presence of water were observed.

Table 5 summarizes the contributions of the gas properties with respect to the uncertainty on the determination of $k_B$. Controlled gas flow and gas filtering operated with our gas-handling system led us to an uncertainty value nearly identical to Moldover et al. [1].

7. Conclusion

We have reported on isothermal acoustic measurements carried out at LNE-CNAM (LCM) in a copper triaxial ellipsoid resonator of 0.5l filled with argon, yielding a new value of the Boltzmann constant. The average experimental value for the Boltzmann constant, with its associated combined standard uncertainty is

$$k_B = 1.3806477(17) \times 10^{-23} \text{ JK}^{-1}.$$  

The combined standard uncertainty corresponds to a relative combined standard uncertainty of 1.2 parts in 10^6, better than the previous 2006 CODATA recommended value [6]. Furthermore, this value of $k_B$ lies 1.9 parts in 10^6 below the CODATA value, and is consistent with it, as the fractional standard uncertainty of the CODATA value is $1.7 \times 10^{-6}$.

The value for the universal gas constant, $R$, deduced from our measurement is $8.3144562(99) \text{ J mol}^{-1} \text{ K}^{-1}$.

This measurement has been achieved using a 0.51 resonator. Several new experimental techniques were implemented, and we improved our knowledge and experience on existing theories and technologies. We will apply this experience for future measurements using a 3.11 QSR. As proved by the present result and by

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numerous pending works on the determination of $k_B$, the final aim of reducing the uncertainty below 1 part in $10^6$, which appears to be suitable for a new definition of the kelvin, is likely to be achieved.

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