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Construction and Start-up of a Large-Volume Thermostat for Dielectric-Constant Gas Thermometry

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Abstract A liquid-bath thermostat with a volume of about 800 L was designed to provide a suitable thermal environment for a dielectric-constant gas thermometer (DCGT) in the range from the triple point of mercury to the melting point of gallium. In the article, results obtained with the unique, huge thermostat without the DCGT measuring chamber are reported to demonstrate the capability of controlling the temperature of very large systems at a metrological level. First tests showed that the bath together with its temperature controller provide a temperature variation of less than ±0.5 mK peak-to-peak. This temperature instability could be maintained over a period of several days. In the central working volume (diameter—500 mm, height—650 mm), in which the vacuum chamber containing the measuring system of the DCGT will be placed later, the temperature inhomogeneity has been demonstrated to be also well below 1 mK.

Keywords Boltzmann constant · DCGT · Definition of the kelvin · Dielectric-constant gas thermometry · Liquid-bath thermostat · Temperature metrology

1 Introduction

In response to the Comité International des Poids et Mesures (CIPM) proposal to give the Boltzmann constant k a fixed value for a redefinition of the base unit kelvin [1], many projects have been started to measure independently the value of k.

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Promising methods are dielectric-constant gas thermometry (DCGT) [2,3], acoustic gas thermometry (AGT) [4,5], and Doppler-broadening thermometry (DBT) [6,7]. An overview of these methods is given in a review article [8].

As one task of the Joint Research Project "Boltzmann constant" (JRP No. T1.J1.4) being part of iMERA-Plus, the execution phase of Implementing the Metrology European Research Area, within the European Community's Seventh Framework Programme, the German institute of metrology, "Physikalisch-Technische Bundesanstalt" (PTB), designed a new DCGT experimental setup. This decision has been taken in view of the excellent experimental DCGT results, which were obtained in the low-temperature range [2,9,10] and allowed one to set up a thermodynamic temperature scale between 3.7 K and 26 K.

DCGT is based on the idea to replace the density in the state equation of a gas by the dielectric constant ε. For an ideal gas, this yields the simple relation between the pressure p and ε: $p = kT(\varepsilon - \varepsilon_0)/\alpha_0$, where ε_0 is the exactly known electric constant and α_0 is the static electric dipole polarizability of a gas atom. 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 [2]. When neglecting higher-order terms and the very small dielectric virial coefficients, this yields

$$p \approx \frac{\chi}{\frac{3A_\varepsilon}{RT} + \kappa_{\text{eff}}} \left[1 + \frac{B(T)}{3A_\varepsilon}\chi + \frac{C(T)}{(3A_\varepsilon)^2}\chi^2 + \cdots \right], \tag{1}$$

where $\chi = \varepsilon/\varepsilon_0 - 1$ is the dielectric susceptibility, A_ε is the molar polarizability, $B(T)$ and $C(T)$ are the second and third density virial coefficients considering the pair and triplet interactions, respectively, and κ_{eff} is the effective compressibility of a suitable capacitor used to measure the susceptibility χ. For determining $3A_\varepsilon/(RT)$, isotherms have to be measured, i.e., the relative change in capacitance $(C(p) - C(0))/C(0)$ of the gas-filled capacitor has to be measured as a function of the pressure p of the gas. A polynomial fit to the resulting p versus $(C(p) - C(0))/C(0)$ data points, together with a knowledge of the pressure dependence of the dimensions of the capacitor, yields $3A_\varepsilon/(RT)$.

Equation 1 shows that a DCGT measurement can be used to determine the Boltzmann constant k at a known temperature applying the relation $k = (\alpha_0/\varepsilon_0)/(3A_\varepsilon/R)$ [3]. For the redefinition of the base unit kelvin, the known temperature must be that of the triple point of water (TPW), which is at present fixed. As a consequence of this, the current design of the DCGT experiment performed at PTB in the low-temperature range loses accuracy due to decrease of sensitivity at higher temperatures. In the pressure range around 0.1 MPa utilized up to now, the susceptibility of helium is too small for DCGT measurements at temperatures near the TPW. To give an example, at a pressure of 0.1 MPa the susceptibility of helium has a value of only 7×10^{-5}. Therefore, in the new experimental setup it is absolutely necessary to perform DCGT measurements at higher pressures (up to about 7 MPa), which must be accompanied by new, larger-sized capacitor designs, which reduce the uncertainty component due to the effective compressibility.

Compared with the DCGT setup for low temperatures, the capacitors of the new design described in [11] require an essentially larger experimental space. Thus, larger dimensions of the thermostat are necessary for realizing a highly stable and homogeneous temperature environment inside the measuring chamber. Results of the investigation of the thermal conditions within the measuring chamber with outer dimensions of 500 mm in diameter and 750 mm in height are described in [11]. In the central working volume of the thermostat, in which the measuring chamber will be located, the temperature instability and non-uniformity must be well below 1 mK. The temperature inside the measuring chamber will be controlled separately at a level of the order of 0.1 mK; see [11]. In this article, the construction and the parameters of the huge thermostat are described in detail.

2 Thermostat Design Considerations and Construction

To fulfill the requirements for the DCGT experiments reported above, a project was started in 2008 as a research cooperation between PTB and the Italian institute of metrology "Istituto Nazionale di Ricerca Metrologica" (INRiM). Previous scientific and technical experience has been achieved at INRiM, where several liquid thermostats have been constructed for different purposes [12,13], including the INRiM AGT experiment. Satisfactory results have been achieved in terms of temperature inhomogeneity and instability; in all cases both parameters were reduced to less than 1 mK. To reach such capabilities, the solution consists of placing the central working volume, where the experiment is contained, in a temperature-controlled bath and surrounding it by the main control volume, which will intercept most of the heat exchange with the surroundings.

In all previous thermostats made at INRiM, the dimensions of the measuring volume are less than those for the DCGT one, being in particular for the AGT experiment a volume of 160 L and about 20 L for the smaller thermostat. Despite the different dimensions, the main principle adopted is the same: the region devoted to the temperature measurement must be separated from that containing the heaters and coolers for temperature control. The motion of the liquid surrounding the measuring chamber (a vessel housing the experiments or a block for performing calibration by comparison for other realizations) is kept in the laminar flow regime. For the DCGT thermostat, the same principle has been applied, even if the total liquid volume is close to 1000 L. A sketch of the thermostat is shown in Fig. 1. The heat exchange between the fluid surface and the ambient temperature is reduced using a double-walled container made of stainless steel, with the space between the two walls packed with a foam insulating material (Armaflex).

Inside the thermostat a cylindrical shield (inner cylinder) of stainless steel is placed, separating the liquid into two cylinders. Inside the shield the DCGT experiment vessel is situated (central working volume). The thermostat fluid continuously circulates from the outer cylinder to the inner one. The liquid is forced to flow from the bottom through a diffuser to the upper free surface, where a sort of "counter-diffuser" returns the liquid to the outer cylinder.

A stable starting temperature of the liquid bath is provided by the circulation of a liquid in a heat-exchanger helix tube fed by an external commercial liquid circulator with a temperature instability of ±10 mK. The temperature of the commercial liquid

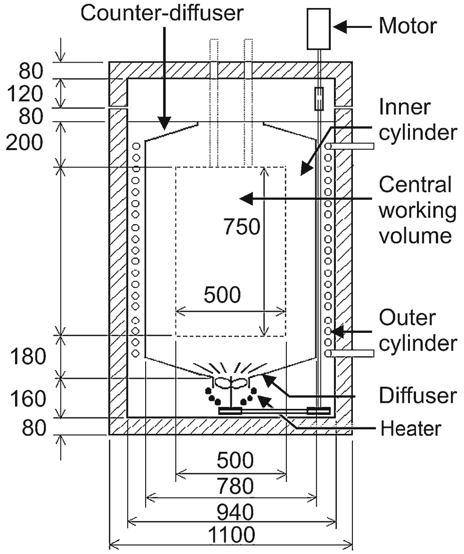

Fig. 1 Schematic design of the thermostat (dimensions in mm)

circulator was set a few tenths of a kelvin below the desired temperature of the central working volume.

The temperature control of the liquid bath is achieved with a 100 Ω platinum resistance thermometer (PRT) placed at the bottom of the volume within the inner cylinder, directly above the diffuser, to quickly detect the temperature changes induced by the action of a 17 Ω heater below. The PRT provides the input signal for a temperature controller, which drives this heater made of sheathed nickel–chromium (Ni/Cr Thermocoax) wire (1.5 mm in diameter). The ring-shaped heater is placed below the diffuser in such a way that the heated liquid is forced through the diffuser.

Furthermore, the heater is placed close to a stirrer to obtain a fast response time and consequently to reduce the time lag in the control loop. The stirrer is a 19cm diameter propeller driven by an ac motor with adjustable frequency of rotation. The motor is placed on the top cover of the thermostat and a series of shafts, belt, and junction allow the motion to be transmitted to the stirrer. The transmission ratio from the motor to the stirrer is 1:1.5, and the frequency of the motor may vary from 0 up to 50 Hz. Usually the rotation frequency of the motor is set between 24 Hz and 32 Hz. The propeller forces the liquid with a spin through the diffuser upwards into the inner cylinder. Outside the cylinder the liquid flows down along the cooling helix tube. The diffuser, which realizes the laminar flow in the central working volume, is supported by vertical baffles placed close to the stirrer to minimize rotational movement in the fluid. The flow obtained is constant and uniform along the whole cross section of the measuring volume. Figure 2 shows the thermostat together with the handling equipment, and Tables 1 and 2 summarize its main features.

Fig. 2 Thermostat with cover opened and the special structure dedicated to handling its main parts

In our experimental setup, a programmable, microprocessor-based, digital temperature controller PTC10[1] from Stanford Research Systems (SRS), Inc. was implemented. It has a specified instability of 1 mK and can communicate via IEEE 488 bus with a personal computer. All parameters may be varied and heater-current values can be recorded. The control-loop parameters are programmable and may be found by the controller itself in a self-optimizing auto-calibration run. The controller is a modular system that can be configured to be compatible with a wide range of applications. For this experiment, a PTC321 input card was used, which supports up to four 100 Ω PRT sensors. The PTC321 allows the use of a four-wire technique to suppress cable-resistant impact on the measurement. The PTC420 output card used is a heater driver that switches up to 5 A of 120 VAC line current with a solid-state relay.

3 First Results

The liquid bath (tap water) has been operated at a temperature approximately 15 °C below room temperature. In all tests, the temperature of the room was maintained constant within ±0.5 K. Measurements of the bath temperature were performed with

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[1] Identification of commercial equipment and materials in this article neither imply recommendation or endorsement by the PTB, nor does it imply that the equipment and materials identified are necessarily the best available for the purpose.

Table 1 Structure of the thermostat

General	
Insulating material	Armaflex
External wall	Stainless steel 2 mm thickness
Internal wall	Stainless steel 3 mm thickness
Insulated wall thickness	80 mm
Total height	1650 mm
External diameter	1100 mm
Total volume	1.568 m^3
Outer chamber	
Height	1490 mm
Inner diameter	940 mm
Inner volume	1.034 m^3
Shield	
Height	1130 mm
Diameter	780 mm
Inner volume	0.54 m^3
Structure	Stainless steel 1.5 mm thickness
Central working volume	
Height	750 mm
Diameter	500 mm
Inner volume	0.15 m^3

two 100 Ω long-stem industrial platinum resistance thermometers (LIPRTs). The thermometers were calibrated at the triple point of water and the melting point of gallium as well as additionally by comparison with a 10 Ω standard platinum-resistance thermometer in a stirred liquid bath at five temperatures. The calibration data were fitted applying the reference and two-parameter deviation functions of the ITS-90. The redundancy of the calibration data allowed a check of its quality. But for the results presented in this section, the calibration accuracy is of minor interest. The short-term stability of the LIPRTs is more important that has been checked carefully by repeated measurements at the TPW. The so-called self-heating effect amounted to a few mK temperature equivalent at a measuring current of 0.5 mA. Its stability during the experiments has also been verified as necessary. The resistances of the thermometers were measured by means of an automatic resistance bridge, type microK 400[1] from Isothermal Technology Ltd., equipped with the channel expander microsKanner. The nominal uncertainty at the temperatures of interest here amounts to a temperature equivalent of 0.1 mK, and the resolution to 0.01 mK. A personal computer drives the bridge for automatic measurement and digital data recording.

The results of the temperature recording over a ten-day period are shown in Fig. 3. The positions of the two LIPRTs were not changed during this measurement period. The data shown in Fig. 3 demonstrate that the long-term instability is very good, within ±0.5 mK peak-to-peak scatter, while the mean value is even more stable. For the ten-day period, the value of the estimated standard deviation of the temperature

Table 2 Details of the control system for the thermostat

Auxiliary controlling line (heat-exchanger helix tube)	
Material	Copper pipeline
Tube diameter	16 mm
Total length	20 m
Pitch	16 mm
PID	
Instrument	SRS PTC10 Temperature controller
PRT	25 Ω ITS-68 SPRT or 100 Ω PRT
Stirrer	
Diameter	190 mm
Channel height	95 mm
Engine	
Type	AC Siemens Sinamics G110
Power	120 W to 3 kW
Heater	
Material	3.55 Ω/m Ni/Cr Thermocoax wire
External sheath	3.2 mm diameter Inconel
Length	4.8 m
Total resistance	17 Ω
Max power	450 W

reading of the two LIPRTs is better than 0.25 mK. A significant instability has not been experienced throughout the 10 days of control. The temperature recording showed no monotonic long-term drift, but a small oscillation of the mean temperature with a period duration of around 136 h and an amplitude of around 0.1 mK is visible. This oscillation was caused by the resolution limit of the temperature measurement of the digital temperature controller PTC10.

The presence of gradients within the central working volume of the bath is an important feature for the environment of the DCGT measuring chamber. Temperature gradients along the vertical axis of the inner cylinder were expected to be small because of the effective separation of the cooling and heating regions from the main control volume. The temperature gradients in the bath have been investigated by placing the two LIPRTs at various positions.

As expected from the symmetry of the water flow, measurements at different positions at the same height yielded similar results for the temperature. Vertical movements of the two LIPRTs caused also no significant difference in the thermometer reading as shown in Fig. 4. Furthermore, a change in the stirring frequency has likewise no detectable influence on the temperature gradient, as reported in Fig. 5. The rotational speed can, therefore, be a useful option to speed up temperature changes, without disturbing the uniformity.

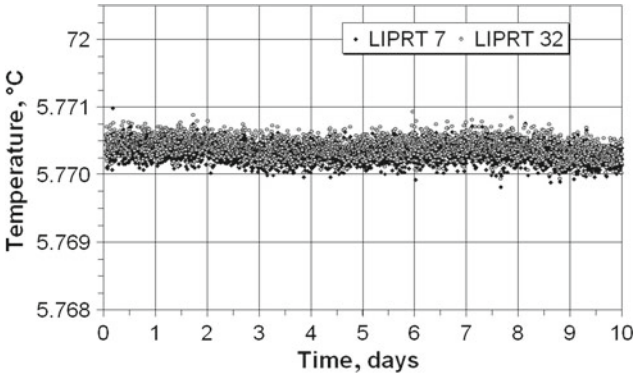

Fig. 3 Long-term measurement of the temperature in the central working volume of the liquid bath with two LIPRTs. The vertical and horizontal distances between the two LIPRTs were approximately 150 mm. The data were recorded over a 5 min averaging time for one measurement point. A measurement period of 10 days is shown. The stability of the temperature demonstrated here is maintainable for many weeks

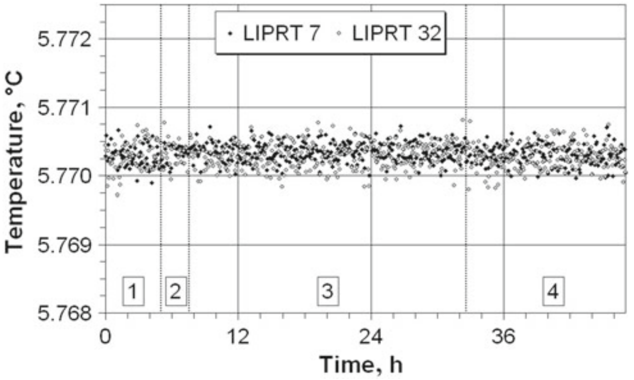

Fig. 4 Measurement of the temperature in the central working volume of the liquid bath with two LIPRTs. Each data point was obtained by averaging the LIPRT readings over a period of 5 min. During the measurement, the vertical position of the LIPRTs was changed in steps of 10 cm. The distances from the top surface of the bath for position numbers 1, 2, 3, and 4 given in the diagram were 50 cm, 40 cm, 30 cm, and 20 cm, respectively. The horizontal distance of the two LIPRTs was not changed during the measurement and was approximately 150 mm

4 Outlook

It has been demonstrated that the described temperature-controlled liquid-bath thermostat provides very good stability for long time periods. The original goal of realizing temperature instability and inhomogeneity in the central working volume well within ±1 mK has been achieved. Measurements of temperature gradients demonstrated that the temperature signal was sufficiently independent of the sensor position. However, the performance of the thermostat may be increased further using a thermistor as a sensing element for the controller together with more sophisticated electronics.

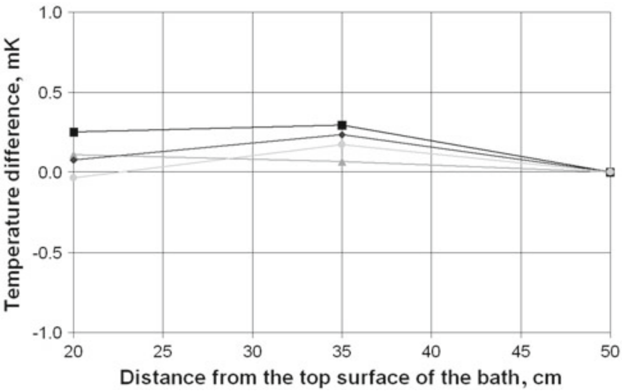

Fig. 5 Temperature gradient in the top 30 cm of the central working volume at different stirrer rotation frequencies. The upper end of this volume is located 20 cm below the top surface of the bath. The *lines* are added only as guides for the eyes. The *symbols* indicate the rotational speed as follows: *filled triangles* 36 Hz, *filled diamonds* 39 Hz, *filled squares* 42 Hz, and *filled circles* 45 Hz

The measurement of temperature gradients in the central working volume of the bath with a finer mesh of sensing elements at appropriate positions is one of the future tasks. Such a mesh is under construction. It will allow mounting of 12 thermometers. Nevertheless, the main work in the future will be the characterization of the thermostat with the DCGT measurement chamber placed in the central working volume. Though the chamber has a convex bottom plate, it will essentially change the flow of the thermostat liquid.

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