

Temperature control in the μK range for density measurements

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Abstract

A temperature controller for density measurements is presented that allows temperature stabilities of about $10\ \mu\text{K}$ to be achieved near $20\ ^\circ\text{C}$. The controller consists of a function generator, a thermistor in a Wheatstone bridge, a differential amplifier, a lock-in amplifier, a PC and a power source for the heater. Two of the controllers are also used to stabilize an electronic temperature fixed point at $20\ ^\circ\text{C}$ that will help to compare the realizations of the $20\ ^\circ\text{C}$ temperature in different laboratories with an uncertainty below $0.5\ \text{mK}$.

Keywords: Density measurements, temperature controlling, temperature regulation, electronic temperature fixed point.

1. Introduction

The temperature plays an important role in all density measurements. Today, 1-kg silicon spheres are used as density standards and have reached relative volume and density uncertainties below 1×10^{-7} (all uncertainties in this paper are standard uncertainties, i. e. for a coverage factor $k = 1$). Recently, the PTB quoted a relative uncertainty of 4×10^{-8} for the volume of the silicon sphere AVO#1 [1] and the NMIJ even published a relative uncertainty of 3.4×10^{-8} for the density of its silicon sphere S4 [2]. For the International Avogadro Project [3], a relative density uncertainty of only 1×10^{-8} is aimed at.

Simultaneously, the accuracy of density comparison measurements is improving. The hydrostatic weighing apparatus of NMIJ is now able to compare the density of 1 kg Si spheres with a relative uncertainty of 3.6×10^{-8} [4] and the pressure-of-flotation apparatus of PTB has an uncertainty of 2×10^{-8} [5]. In both methods, the spheres to be compared are measured alternately in a liquid which is, therefore, used as a transfer standard. The density of the liquid should be stable and homogeneous to about 1×10^{-8} . Since the expansion coefficient of the liquids is about $10^{-3}\ \text{K}^{-1}$, the temperature should be stable within $10\ \mu\text{K}$. Although already more than 20 years ago better temperature stabilities were realized [6, 7], such controllers are commercially not available. Here, the design of a temperature controller is presented that allows controlling the temperature of thermostats near $20\ ^\circ\text{C}$ with a stability of about $10\ \mu\text{K}$. The main parts of the controller were purchased; only rather simple circuits had to be self-made.

Whereas in density comparison measurements only the stability of the temperature is crucial, the absolute temperature is important for the interferometric volume determination of a silicon sphere. As silicon crystals have an expansion coefficient of about $8 \times 10^{-6}\ \text{K}^{-1}$, the temperature must be

known to 1 mK for a relative density uncertainty of 1×10^{-8} . The new PTB temperature controller was used to build an electronic temperature fixed point for 20 °C. After calibration in the temperature laboratory of PTB this can be used for a fast and simple check of the temperature measurement. Additionally, the realizations of the 20 °C temperature in different laboratories of the International Avogadro Project can be compared with an uncertainty below 0.5 mK.

2. Temperature Controller

2.1 Design

The PTB uses well-stirred water thermostats with a capacity of 70 l for density measurements by the hydrostatic weighing and flotation methods [8, 9, 10]. The thermostats are cooled by temperature-regulated pre-thermostats. In principle, the new temperature controller consists of a function generator, a thermistor in a Wheatstone bridge, a differential amplifier, a lock-in amplifier, a PC with a digital PID algorithm and a computer-controlled power supply for the heating resistance [11], see Fig. 1. A variable ac voltage compensates for larger ac output voltages of the bridge.

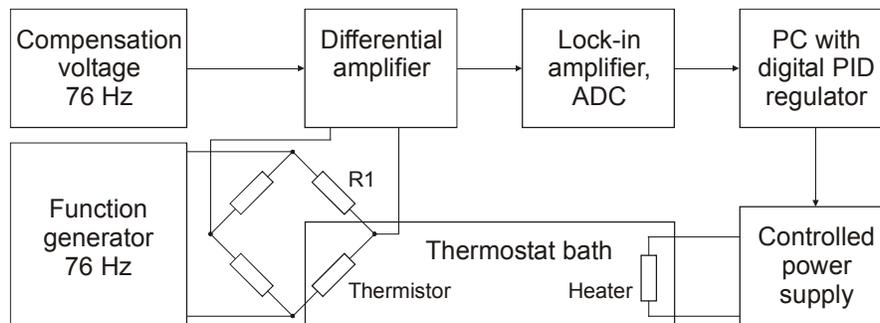


Fig. 1. Principle of the temperature regulation (ADC: analogue-to-digital converter).

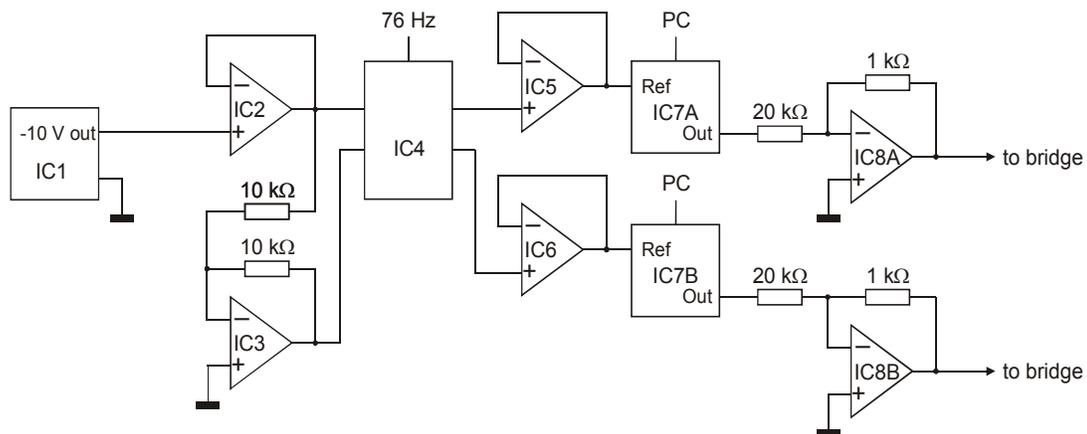


Fig. 2. Principle of the function generator for the bridge voltage. In the diagram, some condensators and resistors are omitted for clarity. Please contact the author for the full diagram. (IC1: MX2701L, IC2, IC3, IC5, IC6: LF355, IC4: MAX333AC, IC7: MAX532A, IC8: NE5532. All resistors 0.1% and 3 ppm/K temperature coefficient.)

Fig. 2 shows a simplified circuit diagram of the function generator. IC1 generates a very stable and temperature-independent voltage of -10 V that is decoupled by the voltage follower IC2 and inverted by IC3. From the output voltages of IC2 and IC3, the analogue switches of IC4 generate two ac voltages that are inverse to each other. The 75.68 Hz digital control signal for the analogue switches is provided by a 77.5 kHz quartz and ten binary dividers (not shown on the diagram). The multiplying digital-analogue converters (IC7, MDAC) are used to shift the ac voltages by digital control (PC) in order to centre the output voltage of the Wheatstone bridge on zero. The output voltages of the MDACs are scaled down by IC8 and IC9 to yield an amplitude of 0.5 V at the bridge.

A super-stable thermistor (Yellow Springs, type YSI 46046) of 12.5 k Ω (at 20 °C) is used for the temperature measurement. Since the other resistance R1 of the bridge is 10 k Ω , the current through the thermistor is 20 μ A, yielding a heating power of only 10 μ W for the thermistor and, thus, a very low self-heating. Whereas the resistance R1 is included in the temperature sensor together with the thermistor and, thus, is always thermostated, the other side of the bridge is housed in a box of its own and thermostated to a temperature of about 41 °C with a stability of 0.1 K by a commercial temperature regulator, designed for the use with quartz oscillators.

The resistors of the bridge are chosen to yield a zero differential output voltage at about 19 °C. In order to compensate for the “large” difference voltage of the bridge at 20 °C, a second ac voltage is realized (see Fig. 3) and subtracted. The amplitude of the compensation voltage is controlled by the PC using the MDAC IC10. The smallest amplitude change corresponds to a temperature change of 0.5 mK. As the MDAC has a range of 12 bit, the maximal range of the compensation voltage corresponds to a temperature range of 2 K.

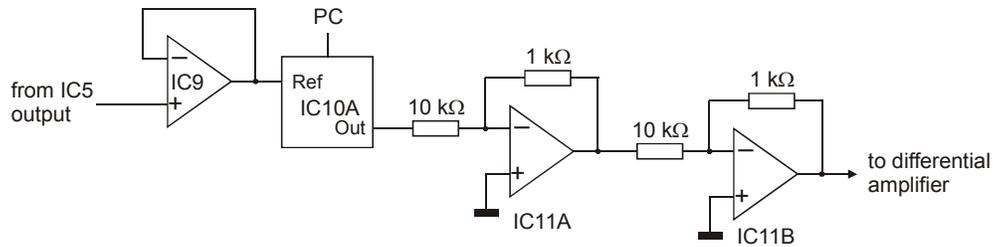


Fig. 3. Principle of the function generator for the compensation voltage. In the diagram, some condensators and resistors are omitted for clarity. Please contact the author for the full diagram. (IC9: LF355, IC10: MAX333AC, IC11: LF353.)

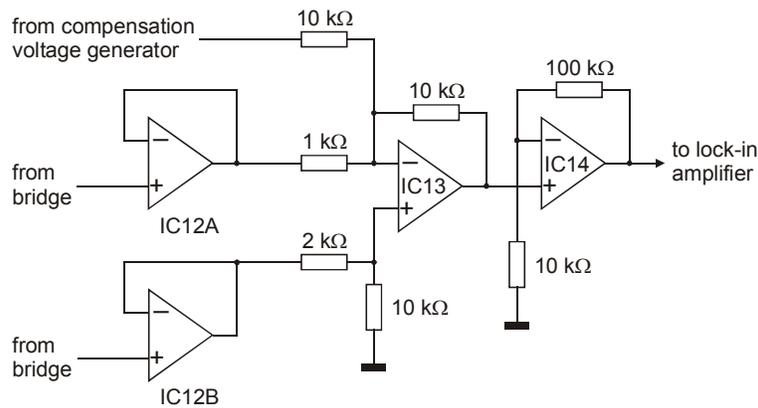


Fig. 4. Principle of the low-noise differential voltage amplifier. In the diagram, some condensators and resistors are omitted for clarity. Please contact the author for the full diagram. (IC12: OPA2107AP, IC13: AD743, IC14: LF355. All resistors 0.1% and 3 ppm/K temperature coefficient.)

Fig. 4 shows the design of the low-noise circuit that amplifies the difference of the bridge and compensation voltages. The output voltage is measured by a commercial lock-in amplifier. The PC reads the output of the lock-in amplifier and calculates the necessary heating power by a digital PID algorithm. For the determination of the PID parameters, first, temperature measurements of the system are made with deliberately changed heating power. In a linear fit the process function is determined [12] and simulation calculations are performed to determine the dependence of the standard deviation of the regulated temperature on a large variety of PID parameters. Then, parameters are chosen that provide a low standard deviation of the temperature and a large stability range of the regulation.

2.2 Performance

The temperature controller is used in the PTB's hydrostatic weighing and flotation apparatuses. Fig. 5 shows the temperature of the flotation apparatus, measured with a 25 Ω Platinum resistance thermometer (PRT) and a F900 resistance bridge. The standard deviation of the temperature values in the thermostat is less than 20 μ K.

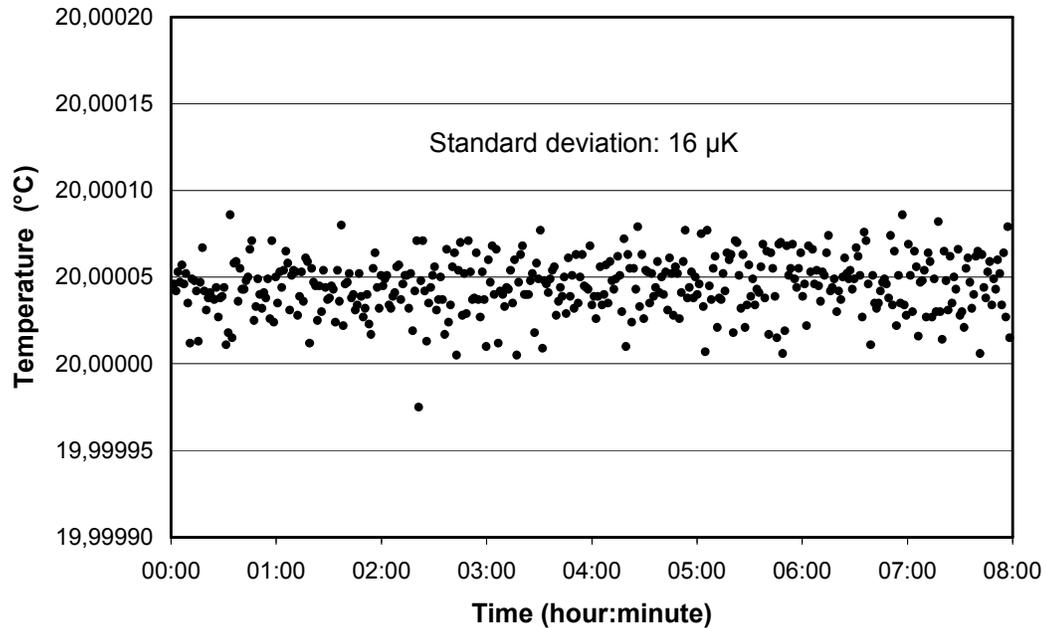


Fig. 5. Temperature stability in the water thermostat of the flotation apparatus. Each point is calculated from four bridge indications measured within 70 s.

3. Electronic Temperature Fixed Point

3.1 Design

The most accurate density measurements are usually performed only near 20 °C [1] or near 22.5 °C [2]. In the International Avogadro Project, it is important that the measurements of the silicon lattice parameter and the silicon-sphere diameter refer to the same 20 °C within 0.5 mK. Differences in the temperature may be due to differences in the fixed-points cells used for the calibration or due to the non-uniqueness of the PRTs [13]. Therefore, a transportable electronic temperature fixed point was designed to compare the 20 °C temperature realisations of the laboratories.

The fixed point is based on a dry block calibrator (DBC) type OCEANUS 6 Model 580 of ISOTECH. This DBC has a 300 mm deep well 51 mm in diameter with a stabilized temperature where usually a metal block with pockets for thermometers is inserted. For the fixed point instead of this block a newly designed temperature-regulated insert is used, see Fig. 6. It consists of a thick Teflon heat insulation at the top and a first copper part that is cooled by the DBC over a 1 mm air gap. Separated by thin Teflon plates, two regulated copper blocks follow. The air gap of the middle copper block to the DBC well is only 2.5 mm whereas the lowest block has 5.5 mm air isolation. The temperatures of the two lower copper blocks are measured by thermistors as above. Heating is provided by two wire resistances (Thermocoax, type 2NCNC). The well in the centre of the insert has an inner diameter of 10 mm and a depth of 335 mm, thus allowing a good immersion depth of the thermometer and providing a very good decoupling from the room temperature. Water is used as contact medium for the PRT.

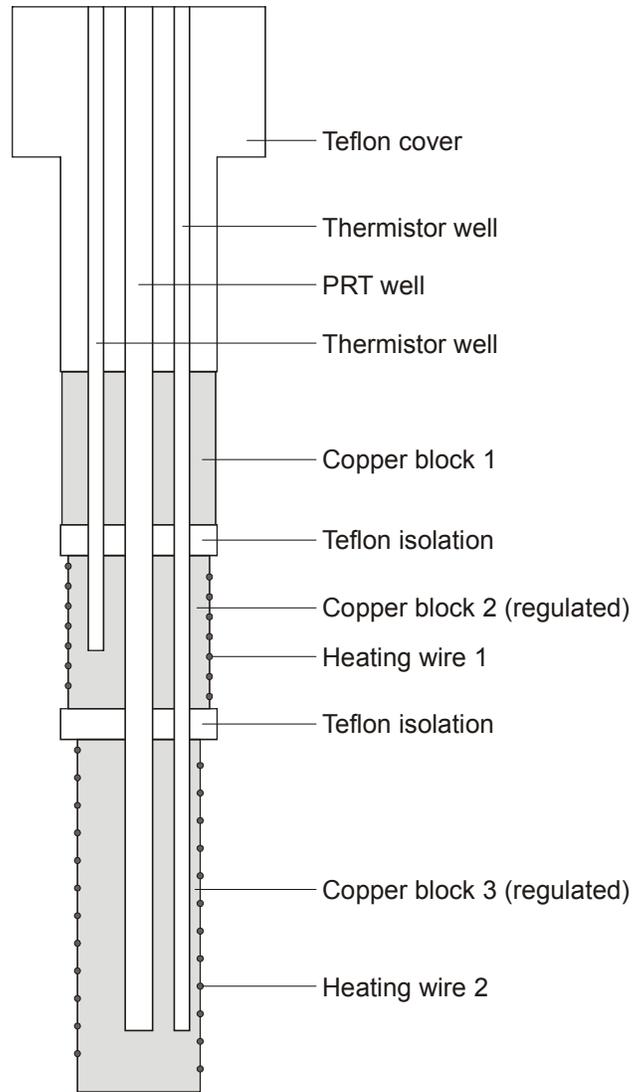


Fig. 6. Scheme of the temperature-controlled insert for the electronic temperature fixed point.

3.2 Performance

The DBC is usually set to 19.95 °C, which provides enough cooling for the fixed point at 20 °C. Then, the heating powers for the regulated parts are about 10 mW. The temperature gradient in the well was checked by pulling the PRT 50 mm higher. A temperature lower by about 0.1 mK was observed which can be set to zero if the temperature of the upper regulated copper block is increased by a few mK. The self-heating of the PRT was determined to 0.17 mK for a measuring current of 1 mA. Fig. 7 shows the temperature of the fixed point measured with the PRT and the F900 over 9 hours after switching-on. The standard deviation of the temperature is about 40 μ K. During the first hours, the temperature drifts by about 0.1 mK. The reproducibility and long-term stability is shown in Fig. 8. All measured temperatures lie in a range of 0.6 mK.

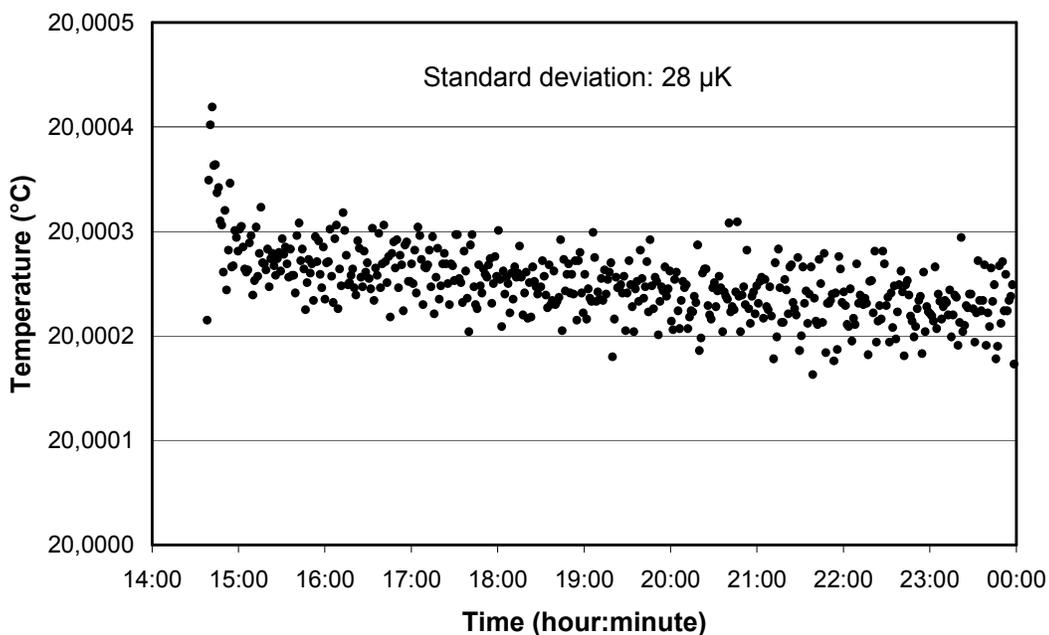


Fig. 7. Temperature stability of the electronic fixed point after switching-on.

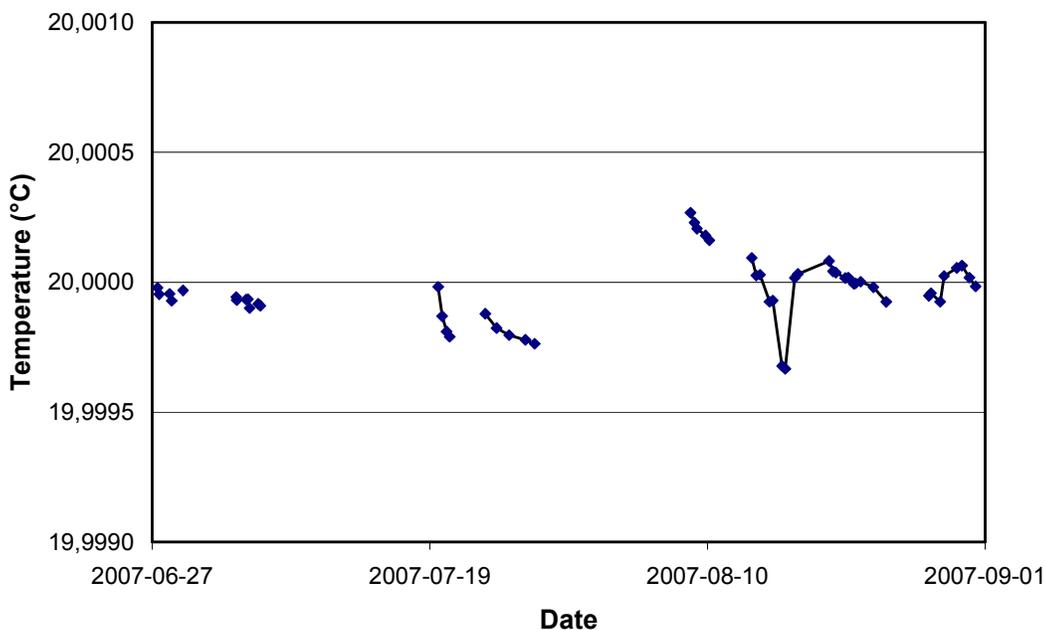


Fig. 8. Reproducibility of the temperature over 2 months. Points that are connected with a line belong to the same switching-on of DBC and fixed-point insert.

A small influence on the fixed point temperature comes from the room temperature, see Fig. 9. Raising the room temperature from 22 °C to 25.7 °C decreased the temperature of the fixed point by 0.13 mK. Simultaneously, the PID controller increased the heating power of the regulated parts from 10 mW

to 20 mW. Apparently, the temperature of the DBC decreases with increasing room temperature. At 11:20 the DBC was set to 20.00 °C to reduce the heating power. This did not shift of the temperature of the fixed point.

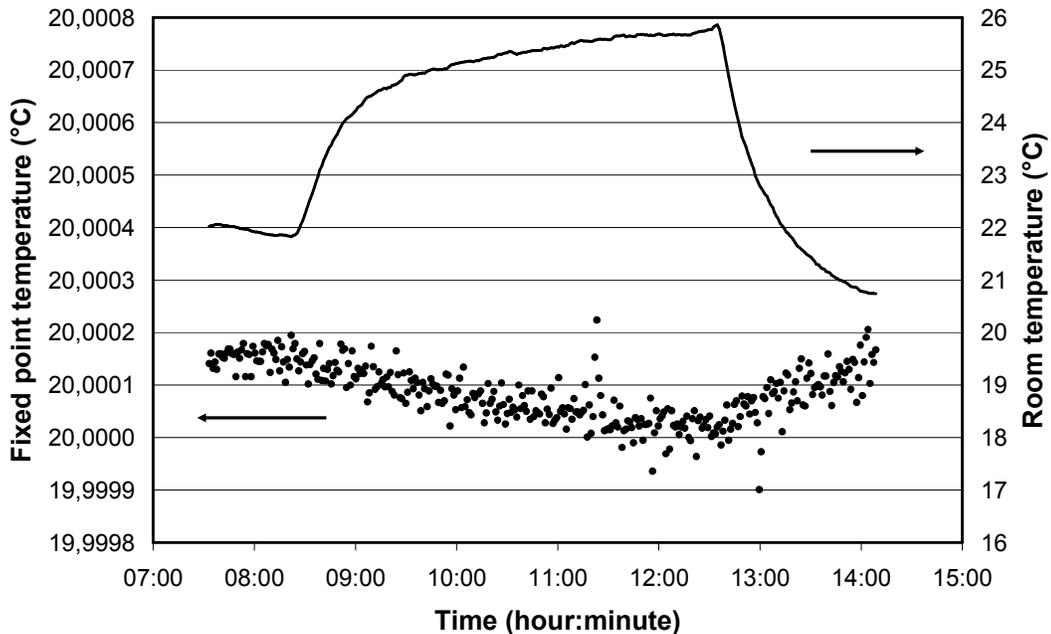


Fig. 9. Influence of the room temperature (line and right scale) on the electronic fixed point temperature (points and left scale).

4. Conclusion

The new temperature controller allows stabilizing the temperature of water thermostats to about 10 μ K. The electronic temperature fixed points realizes the 20 °C temperature with a reproducibility below 0.5 mK. Calibrations in the temperature laboratory of the PTB will assure absolute accuracy. A rather large experimental set-up was used to investigate the performance of the fixed point. A compact controller will be built that includes not only the circuits shown above, but also the lock-in amplifiers, the computer-controlled power supplies and a microcontroller for controlling.

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