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Calibration and self-validation of thermistors for high-precision temperature measurements

Steffen Rudtsch *, Christoph von Rohden

Physikalisch-Technische Bundesanstalt (PTB), Braunschweig und Berlin, Germany

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ABSTRACT

In this paper we discuss influencing variables affecting the uncertainty of high-precision temperature measurements ($u < 1$ mK) by means of NTC thermistors. These are proper instrument settings, a suitable choice (distribution) of calibration temperatures in due consideration of uncertainties, self heating and the number of parameters in the calibration equation. Within this work we used 4 wire measurements to eliminate the influence of lead resistances, switched dc current to reduce errors by thermoelectric effects and amplifier offsets, a reference resistor with a nominal value close to the thermometer resistance to maximize the resolution and a maximum current consistent with the input voltage range and self heating of the thermistor.

We present results of high-precision calibrations of a so called super-stable thermistor and demonstrate the influence of changes of the calibration equation on the interpolation error. Our results confirm previous findings that the number of parameters in the interpolation equation can have a considerable influence on the interpolation error. It was confirmed that the Steinhart–Hart equation shows a poor performance and should be replaced by the more suitable models recommended in White et al. (2014). For the quantification of the long term stability of a calibration we recommend repeated single-point validations at the triple-point of water. If these are supplemented by measurements at the gallium fixed-point possible changes of the curvature of the characteristics can be detected.

1. Introduction

There is an increasing need for temperature measurements with uncertainties on a millikelvin-level in the temperature range between −20 °C and +50 °C. This is related to requirements in various fields such as ocean temperature measurements, air and artifact temperature monitoring in dimensional metrology laboratories but also to specific industrial applications in precision manufacturing, optics and semiconductor production. Compared with metrology-grade platinum resistance thermometers, thermistors have a higher sensitivity (up to ten times), are less sensitive to mechanical shock or vibrations and can be manufactured with smaller diameter. For so called ultra-stable thermistors, manufacturers claim accuracies better that 1 mK and drift rates less than 2 mK/year. This requires appropriate standards and techniques which can be used to (re-)calibrate sensors and to validate the uncertainty claims.

In the following we discuss methods for the quantification of the dominating uncertainty contributions and optimum instrument settings. Due to the strong non-linearity of the thermistor characteristics, the calibration equation should follow a suitable mathematical model with a specific number of individual parameters [1]. As a consequence

* Corresponding author. Tel.: +49 3034817650; fax: +49 3034817504.
E-mail address: steffen.rudtsch@ptb.de (S. Rudtsch).

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high-precision applications with thermistors require a calibration at a minimum number of 4–5 well distributed temperatures. These should always include the maximum and minimum application temperatures.

The calibration techniques are the so called comparison method and fixed-point calibrations. In the comparison method a calibrated Standard Platinum Resistance Thermometer (SPRT) is used as a reference for the calibration of a thermistor in a stirred liquid bath with excellent temperature stability and homogeneity. By means of this method typical uncertainties of (2–3) mK are achieved. For thermometer calibrations at the triple point of water (0.01 °C) uncertainties below 0.1 mK are state of the art, at the Ga- (29.7646 °C) and Hg-fixed points (−38.8344 °C) about 0.25 mK are possible.

Therefore, a thermistor calibration with smallest possible uncertainties requires the combination of both techniques, the comparison method for the characterization of the curvature and complementary measurements with lower uncertainties at selected fixed-points. Due to the required effort, repeated "calibrations" are in practice often carried out at only one or two temperatures.

A single-point test at only one fixed-point temperature is referred to as "validation". Repeated single-point validations allow a quantification of the drift with better accuracy than calibrations or validations by means of the comparison method. Although a drift of resistance based temperature sensors is mostly a shift of the calibration function (offset), some physical processes such as the so called poisoning by impurities can change the curvature of the characteristics. Therefore, an additional validation at another fixed-point temperature can indicate such variations and provides supplementary information about the mechanism of changes of the calibration results.

2. Experimental

In this work we exemplarily present calibration results for a MEAS type 46016 thermistor[1] with a nominal resistance value of about 10 kΩ at 25 °C. The sensor was assembled in a hermetically sealed stainless steel housing with 4-wire PTFE insulated cables. By investigations of other sensors of the same type it was checked whether the calibrated one showed a typical behavior. For the resistance measurements of the thermistor and Standard Platinum Resistance Thermometer (SPRT) an ISOTECH instrument type microK 70[1] was used. The microK uses a four-wire technique to eliminate lead resistances and a 10 Hz bipolar switched dc current to eliminate errors associated with thermoelectric effects and amplifier offsets. The calibrations were carried out in the resistance ratio mode by direct comparison with thermostated Wilkins-type standard resistors. Typical measurement currents were 1 mA for SPRTs and 10 μA for thermistors. Different currents have been applied to each sensor to allow a correction to zero self heating.

The determination of the characteristics of the thermistor is based on a calibration by means of the comparison method. For this purpose a calibrated SPRT was used as a reference thermometer in a thermostated and well circulated water bath (55 l) with a temperature stability and homogeneity of typically 0.5 mK. Two series of calibration were carried out (May 2014 and February 2015) in the temperature range between 5 °C and 60 °C. Between the calibrations the sensor was stored at room temperature. In order to reduce the uncertainties the results were validated by additional calibrations at the triple point of water and the Ga fixed point.

3. Results and discussion

3.1. Optimum instrument settings

In a first step the instrument settings were optimized. These improvements included four wire resistance measurements, specific shielding of cables, a suitable choice of integration (measurement) time but most important a proper selection of measurement current and reference resistor. Previous own investigations by means of a Hamon-type Resistance Bridge Calibrator have shown that an improper choice of the range used for the resistance ratio measurement can deteriorate the linearity by more than one order of magnitude. We found that for a microK-type of instrument, optimum linearity (about 0.04 ppm) is achieved in the resistance ratio range between 0.2 and 1.2. Because the microK measures ratios of voltage drops across the reference resistor and the unknown resistor by means of a specific substitution technique, a superior stability at unity ratio was expected. To maximize the resolution it is advisable to use a reference resistor close to the maximum resistance of the thermistor. The maximum current should be consistent with the input voltage range (125 mV or 500 mV) and the self-heating of the thermistor.

This was supported by the results of noise measurements. In Fig. 1 the noise for a 10 kΩ resistor (Vishay type VH102ZT) is exemplarily shown for two different setups, first (gray) with the internal 400 Ω reference resistor of the instrument and second (blue) with a 10 kΩ external Wilkins-type standard resistor as a reference.

The results demonstrate that by using a reference resistor of a similar nominal value the standard deviation is reduced by a factor of 3 (from 7.5 mΩ to 2.5 mΩ). For a thermistor with a resistance value of 10 kΩ at 25 °C this corresponds to a decrease in the temperature noise from 17 μK to 6 μK.

With a suitable shielding these small noise levels are also achieved using thermistors in fixed-point cells (Fig. 2).

Although the small variation of the fixed-point temperature after a sufficient run-in time of about 4 h indicates stability better than ±30 μK the pressure dependence of the fixed-point temperature and other contributions lead to typical uncertainties of about 250 μK for commercially available Ga fixed-point cells. Further improvements with uncertainties below 100 μK are possible with well characterized pressure controlled Ga fixed-point cells and specific preparation methods for the inner phase boundary.

[1] Certain commercial equipment, instruments or sensors are identified in this paper in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by PTB, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.