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Proposal for interlaboratory comparisons: Objective: Calibration of NTC-thermistors on the temperature domain of 100 to 400 K.

Temperature is the most important physical quantity in all human activities performed on the most common temperature domain of -50 to +100 ^oC corresponding to the natural distribution of temperature on the Earth. The latest version in force of International Temperature Scale (ITS-90) has only 3 fixed points as basic standards on this domain, namely [1]:

TP - Hg	-38.8344 ⁰ C
TP – Water	$0.01 {}^{0}C$
MP – Ga	29.7646 ⁰ C

(TP = triple point, MP = melting point).

On the other hand, Standard Platinum Resistance Thermometer (SPRT) actually used as the best working standard thermometer on this domain has a non-linear characteristic needing polynomial approximation for its calibration.

NIST has introduced new fixed points on this temperature domain by TP of the following standard reference materials (high purity) [2]:

Ethylene carbonate	36.3146 ⁰ C
Rb	39.265 ⁰ C
n-docosane	43.879 ⁰ C
succinonitrile	58.0642 ⁰ C.

Although, these new fixed points do not belong to ITS-90, they have the required characteristics for such basic standards for temperature, namely high repeatability and reproducibility.

It appears clearly that new physical phenomena are necessary in view to redefine old and new fixed points in a new ITS.

Asymptotic and smooth dependence of electric resistance of NTC-thermistors with temperature, R(T), can be a such phenomenon revealing a temperature driving solid-solid phase transition in these materials.

I have studied thoroughly R(T) dependences for a wide variety of commercial NTC-thermistors on the basis of topoenergetic principles [3] and a data base of Universal parameters was established with deep structural significances [4]. These results were the starting point of new measurements of R(T) on samples of NTC-thermistors in view to verify in more accurate conditions R(T) patterns and to reveal new aspects of this phase transition. Incidentally, our preliminary [5] and more accurate [6] measurements have evidenced a repeatable oscillatory

behavior of temperature deviation = difference between measured and calculated temperature according to both Steinhart-Hart and our Universal eqns. on the temperature domain of 0 to 100 0 C, so that the main conclusion was to consider this phase transition in NTC-thermistors and topoenergetic working principles as an opportunity to re-unite the old and new fixed points in a new better defined ITS for the domain of 100 to 400 K specific for a wide variety of commercial NTC-thermistors with stable, repeatable and reproducible R(T) patterns.

My long experience shows that small signal diodes made from silicon single crystals have linear and very stable temperature pattern on temperature domain of 0 to +100 ^oC and also small dimensions close to NTC-thermistors [7]. These are also important facts which must be considered in this project.

It is important to mention that I am available anytime to share my experience concerning control and measurement of absolute and differential temperature, retrieval of experimental data, design and construction of mechanical, thermal, electric and electronic parts, etc. in a fair cooperation for this project.

[1] BIPM, ITS-90: Procès-verbaux du Comité International des Poids et Mesures, 78th meeting, 1989.

[2] NIST thermometry group (<u>www.nist.gov</u>, <u>Gregory.Strouse@nist.gov</u>).

[3] Gh. Dragan, Gh. Dragan, "NTC-thermistors-1", <u>GDF Databanks Bull.</u>, Vol. 10, No. 1, pp. 1-36, 2006.

[4] Gh. Dragan, "TRESISTOR© - NTC-1: data bank of NTC thermistors", <u>GDF Databanks Bull.</u>, Vol.11, No. 3, pp.3-9, 2007.

[5] Gh. Dragan, "Temperature calibration of NTC-thermistors. 1. Preliminary results", <u>GDF Databanks Bull.</u>, Vol. 12, No. 4, pp. 3-10, 2008.

[6] Gh.Dragan, "Calibration of NTC-thermistors", The 14th International Metrology Congress, 22-25 June 2009, Paris, France; <u>GDF Databanks Bull.</u>, Vol. 13, No. 1, 2009.

[7] Gh.Dragan, "A new technique for temperature measurement and calibration", <u>GDF Databanks Bull.</u>, Vol. 9, No. 2, 2005.

Calibration of NTC-thermistors

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<u>Résumé</u>

Les valeurs expérimentaux et calculées de température obtenues d'âpres la bien-connue eqn de Steinhart-Hart et la notre Universel eqn sont comparées aux cours d'étalonnage de NTC-thermistors sur l'intervalle de température 0 a +100 °C. La différence de ces valeurs a montrée un répétable comportement oscillatoire pour les deux eqns et aussi dans les 3 systèmes de mesure. La conclusion: ces expérimentes d'étalonnage doivent être prolonges sur l'intervalle 100 a 400 K comme comparaisons inter laboratoires pour redéfinir les points fixes actuels et aussi des nouvelles en considérant les significations structurel des paramètres Universels.

Abstract

Experimental and calculated temperature values obtained by multiple scan calibration of NTC-thermistors and by using the well known Steinhart-Hart eqn. and our own Universal eqn. thoroughly studied were compared on the temperature domain of 0 to +100 ^oC.A repeatable oscillatory behaviour of their difference was observed, for both eqns and in all 3 measuring systems. In conclusion: calibrations of large series of NTC-thermistors must be extended on the domain of 100 to 400 K as interlaboratory comparisons in view to redefine actual and new fixed points by considering the structural significance of Universal parameters.

Introduction

NTC-thermistors are materials with negative temperature coefficient (NTC) of electrical resistance, R, as revealed both in dc and ac measurements. There are several main companies producing very repeatable, stable and reproducible series of NTC-thermistors whose characteristics R(T) appears as a smooth exponential decreasing dependence from a threshold temperature, To, below which the electrical conduction is practically inhibited or has a different nature than over it where free electrons are responsible. This behavior is typical for systems experimented according to general principles of topoenergetic the procedure [1] and the obtained experimental results are retrieved according to the Universal eqn. This procedure was applied recently on series of NTC-thermistors produced by six companies taking into account R(T) values given in their websites [2]. The obtained results are discussed on the general structural meaning Universal parameters as they of were established during a long experience on a large variety and number of transforming systems. The resulted Universal parameters are available in the data bank called as TRESISTOR®-1 [3]. One of conclusions in this study was the fact that the given R(T) values are not the exact ones, but averaged values representing each type of NTC-thermistor in the series, so I decided to make exact measurements on representative samples.

Preliminary results were reported recently indicating an interesting oscillatory behaviour [4], so calibration experiments required more accurate experimental conditions.

In the present note the calibration results obtained by multiple scans on the temperature domain of 0 to +100 ⁰C are presented as the difference between experimental temperature, T, and calculated temperature, Tcalc, according to the Steinhart-Hart and Universal eqns. on a typical commercial NTC-thermistor (Bethatherm). Three different measuring systems and a digital diode thermometer (DDT) as standard thermometer are used. The

temperature stability and uncertainty was estimated under 0.01 ⁰C on this temperature domain. In all experimental conditions a very clear the oscillating behaviour of (T-Tcalc) vs T appeared, so I strongly sustain the extension of these calibration experiments by interlaboratory comparisons on the domain of 100 to 400 K specific to all commercial NTC-thermistors and defining their Universal behaviour. These facts will certainly allow at least redefining actual and new fixed points.

Experimental details

NTC-thermistor is type 30k6A1 (Betatherm). DDT as standard thermometer was thoroughly studied during several years on this temperature domain and it is considered as perfect linear [5]. It was calibrated several times on two points (triple point of water and a temperature close to 100 ⁰C comparing with a certified SPRT).

Ultrathermostate disposition is presented in Figures 1a and 1b. Due by their small and close sizes, NTC-thermistor and diode were cemented closely in a brass capsule with epoxy resin in view to minimize temperature gradients between them. Each temperature point was reached with 0.01 ⁰C stability in approximately 1 hour, but kept at least for 2 hours before to note the final Um(T) values.

Stability and uncertainty of experimental temperature was estimated under 0.01 ⁰C.

Table 1 shows the two measurement circuits used for electric resistance of NTC-thermistor (Th) by voltage Um measured by 4-wire procedure. The 10 μ A source was calibrated by using same circuits on standard resistors (100 to 10 MOhm). All voltage measurements were performed with the Digital Multimeter V563 (Meratronik, Poland) having 5 digit mantissa and separate exponent.

Experimental results

Table 2 shows details on experimental conditions denoted as A, B and C in which the values of Tcalc were obtained according to Steinhart-Hart and Universal eqns.

The dependences of (T-Tcalc) vs T according to these experimental conditions are graphically presented in Figures 2 - 4.

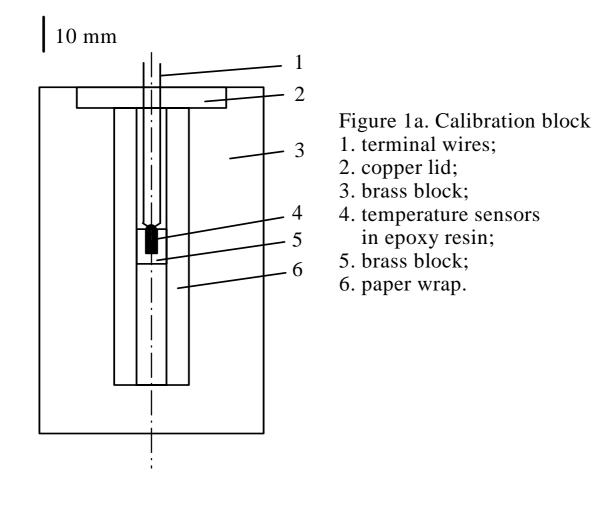
All measurement series show oscillatory behaviour of this dependence with some specific features. The main evidence consists in the fact that both Steinhart-Hart and Universal parameters are sensitive to the temperature domain on which they are determined, so that more realistic representations should result starting with temperature close to To (ranging between 80 and 160 K) up to approximately 400 K.

Conclusions

- 1. More accurate calibration experiments should be extended on the temperature domain of 100 to 400 K for a wide variety of NTC-thermistors as interlaboratory comparisons.
- 2. Dynamic procedures like differential scanning calorimetry and/or differential thermal analysis at different heating/cooling rates should be considered additionally.
- 3. Calibration of diodes should also be considered again in more accurate condition and on wider temperature range in connection with above mentioned calibrations.

References

[1] Gh. Dragan, "Solubility behaviour introducing topoenergetic principles", GDF Databanks Bull., Vol. 1, No. 1, pp. 5-12, 1997. Gh. Dragan, "NTC-thermistors-1", GDF [2] Databanks Bull., Vol. 10, No. 1, pp. 1-36, 2006. [3] Gh. Dragan, "TRESISTOR[®] - NTC -1: data bank of NTC thermistors", GDF Databanks Bull., Vol.11, No. 3, pp.3-9, 2007. [4] Gh. Dragan, "Temperature calibration of NTC-thermistors. 1. Preliminary results", GDF Databanks Bull., Vol. 12, No. 4, pp. 3-10, 2008. Gh.Dragan, "A new technique for [5] temperature measurement and calibration", GDF Databanks Bull., Vol. 9, No. 2, 2005.



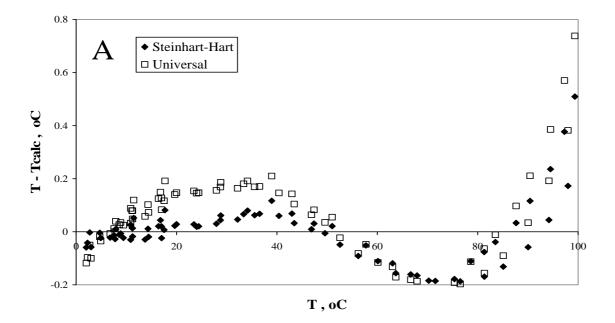


Figure 2. Temperature dependence of deviation values for the two representations in A experimental conditions.

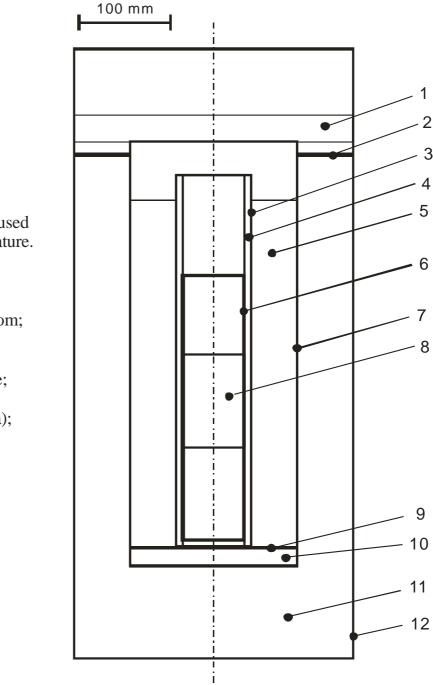
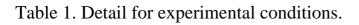


Figure 1b. Vessel assembly used under ambient temperature.

- 1 rigid polystyrene foam;
- 2 PCV lid;
- 3 PCV pipe with tight bottom;
- 4 cardboard shield;
- 5 water;
- 6 controlled heating mantle;
- 7 brass vessel;
- 8 calibration block (Fig. 1a);
- 9 rigid sieve;
- 10 ice and salt mixture;
- 11 polyurethane foam;
- 12 external vessel.



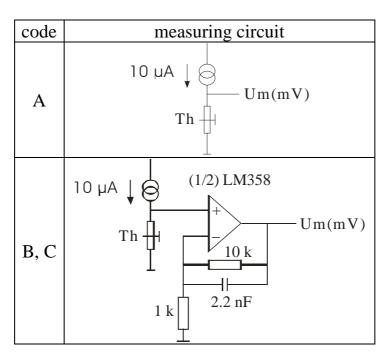


Table 2. Values of parameters obtained by non-linear regression of calibration values (T, Um) for NTC-thermistor (30k6A1, Betatherm) in different experimental conditions denoted as A, B and C. Confidence level of 68.3%; T in K, Um in mV;

C(Mfr) – takes into account data on R(T) reported by Betatherm on the range of -80 to +150 ^{0}C ;

C- takes into account the same experimental data as in B, but optimizing in Universal eq. only parameter M and imposing (N, To) parameters from C(Mfr).

Steinhart-Hart: $(1/T) = a + b*\ln(Um) + c*(\ln(Um))^3$

	А	В	C (Mfr)
a	(2.036 ± 0.004) E-3	(1.511 ± 0.01) E-3	(2.046 ± 0.002) E-3
b	(2.222 ± 0.01) E-4	(2.151 ± 0.03) E-4	(2.24 ± 0.004) E-4
c	(2.53 ± 0.2) E-7	(1.74 ± 0.2) E-7	(1.57 ± 0.03) E-7

Universal: $\ln(Um) = N*\ln(T-To) + M$, or: $T = To + exp((\ln(Um) - M)/N)$

	А	В	С	C(Mfr)
Ν	$-(8.13 \pm 0.2)$	$-(7.52 \pm 0.2)$	$-(8.822 \pm 0.2)$	$-(8.74 \pm 0.1)$
М	47.8 ± 1	39.7 ± 1	47.65 ± 0.01	56.3 ± 3
To,K	120 ± 4	132 ± 4	105.6 ± 3	106.94 ± 0.9

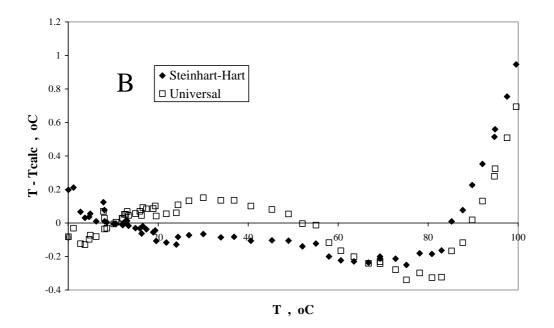


Figure 3. Temperature dependence of deviation values for the two representations in B experimental conditions.

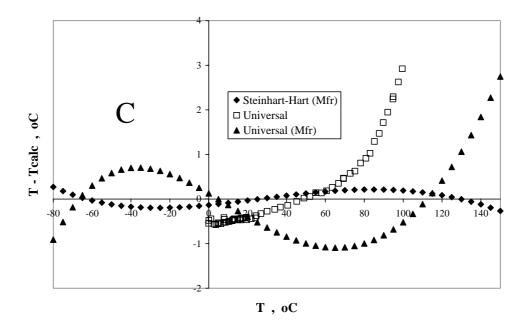


Figure 4. Temperature dependence of deviation values for the two representations in C experimental conditions.

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