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INVESTIGATION OF THE LITHUANIAN NATIONAL STANDARD OF ELECTRIC RESISTANCE

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Abstract

The Lithuanian national standard of electric resistance is maintained as the basis for calibration and measurement capabilities published in the key comparison database of the International Bureau of Weights and Measures (BIPM). The stability and uncertainty of the resistance value measurements, performed since 2004 using the calibrated values of the standard resistors to predict their future behaviour as well as influence of environmental conditions, are discussed. Also discussed is the recovery of a standard resistor which had undergone a mechanical disturbance. It is concluded that the standard resistors operated by the Lithuanian National Electrical Standards Laboratory feature stable drift of resistance, which is well predicted by means of linear regression.

Keywords: measurement standards, electric resistance, standard resistor, calibration, uncertainty, prediction.

1. Introduction

Much work is devoted to electric resistance measurements, which is due to the fact that they are used in a variety of applications, such as sensing of temperature [1, 2] and gas [3, 4], and many others. To make a good measurement, we have to use an accurate instrument calibrated – through a chain of intermediate calibrations – to a national standard of the highest precision which is traceable to the SI unit of resistance.

Although resistance standards based upon the quantum Hall effect are used today by some *national metrology institutes* (NMIs), much cheaper conventional standard resistors are still being used as national standards by many NMIs, especially those of relatively small countries. Moreover, new designs and metal alloys for standard resistors are being elaborated in order to achieve as small as possible drift of resistance, low sensitivity to environmental conditions, and the highest possible stability [5–10]. Drift rates of the order of 10^{-9} (Ω/Ω)/year and stabilities of about $5 \cdot 10^{-8}$ Ω/Ω) have been reported [9, 10]. Graphene is also used as a material for standard resistors, and its noise properties are investigated [11].

Usually, the standard resistors are periodically calibrated at other NMIs operating their *quantum Hall resistance* (QHR) units. On the other hand, conventional standard resistors are widely used as the working standards and transfer standards during interlaboratory comparisons [12]. All this implies there is a need to investigate how the values changed with time and to predict the changes in the future. Usually, linear regression is used for that. Here we describe the results of a linear regression analysis of the resistance values for standard resistors performed by the *Center for Physical Sciences and Technology* (FTMC) – the Lithuanian NMI.

The national standards of electric resistance and DC voltage were developed and put to operation at the *Electrical Standards Laboratory* (ESL) of FTMC in 2003–2005, following an assignment from the government of the Republic of Lithuania.

2. The standard of electric resistance

The standard of electric resistance was recognized as the national standard in 2008; it is of the secondary metrological level. The standard values of resistance are realized by using eight standard resistors of 1 Ω and five standard resistors of 10 kΩ, and traceability to the SI unit of resistance is ensured by regular (once in three years) calibration of one resistor from each group with a QHR system at a foreign laboratory. On the other hand, the standard values are transferred to the reference standards – decade resistors for the range from 0.001 Ω to 1 TΩ – by means of direct comparisons. The relative uncertainty of the realized values of 1 Ω and 10 kΩ confirmed by international comparisons in 2007 does not exceed $2 \cdot 10^{-7}$.

The set of the resistance standards, besides the above-mentioned set of decade resistors, comprises also the resistance measurement bridges by *Measurements International* (models 6010Q and 6000B), data acquisition software, a computer, and equipment for maintaining stable ambient conditions. For extension of the range of resistance and transferring the standard resistance value, a 6010Q direct current comparator (for the values of up to 10 kΩ) and a 6000B potentiometric bridge (for the values of up to 1 TΩ) are used. The relative uncertainty of the bridges, $\Delta R/R$, is of the order of 10^{-8} to 10^{-7} and contributes to the overall uncertainty whenever the bridges are used. The structure of the Lithuanian national resistance standard is shown in Fig. 1.

The resistance of the standard resistors is affected by ambient temperature, air pressure, and relative humidity. Moreover, the resistance values drift due to aging. Sensitivity to environmental conditions and other properties of the standard resistors depend on materials and construction chosen by the manufacturer. Most of the standard resistors in our set (except for one of the 10 kΩ resistors, which is an SR104 by *Tegam*) have been manufactured by *Tinsley* from a special alloy. *Tinsley's* resistors feature relative resistance drift of about $2 \cdot 10^{-7}$/year, relative temperature dependence of resistance of about 10^{-6}/K, and relative pressure dependence of resistance of about $2 \cdot 10^{-9}$/hPa, while the *Tegam's* relative deviation of resistance from the nominal value due to temperature is described as $\Delta R/R = -2.8 \cdot 10^{-8}(T - 296\ \text{K})^2$; *Tegam's* sensitivity to pressure and humidity is not specified. The influence of ambient temperature is eliminated by keeping all the resistors with the resistance of up to 10 kΩ (except for the *Tegam's* 10 kΩ) in special oil baths at constant temperature (with the accuracy of ±0.005 °C), while the resistors of larger resistance are kept in thermostats (with the accuracy of ±0.1 °C). The *Tegam's* resistor, due to its construction, cannot be stored in an oil bath; on the other hand, it does not need additional thermostabilization: in the laboratory room with ambient temperature kept at 23 °C ±1 °C, deviation of the *Tegam's* resistance due to variation of temperature does not exceed 0.028 ppm.

All the *calibration and measurement capabilities* (CMCs) of the Lithuanian NMI in the area of electricity and magnetism now in effect are described in the key comparison database of the

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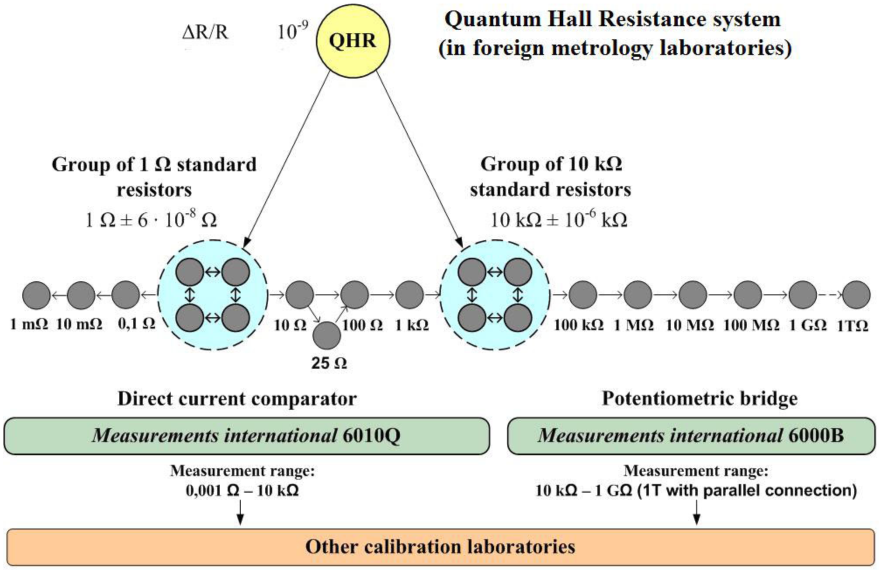

Fig. 1. The structure of the Lithuanian national resistance standard.

BIPM [13]. Here we consider the behavior and prediction of metrological characteristics of the resistance standard in the years 2003–2020.

By periodic calibration of the decade resistors, their histories are obtained. Between calibrations, a linear regression model is used to predict the resistance value of the standard resistors, which is justified as the drift is close to linear. Coefficients of the regression line (a and b) are computed by means of the conventional least-squares method, by minimizing a weighted mean standard deviation $\chi^2(a, b)$ of the line $a + bt$ from the resistance values $R_i(t_i)$ obtained at calibration times t_i:

$$\chi^2(a, b) = \sum_{i=1}^{N} \frac{[a + bt_i - R_i(t_i)]^2}{\sigma_i^2}, \tag{1}$$

where N is the total number of calibrations performed, with each calibration having its uncertainty σ_i. Usually, t_i is the number of days since the first calibration ($t_1 = 0$). Taking into account different calibration uncertainties (calibrations may have been made at different institutions using different equipment, *etc.*) yields a more reliable weighted prediction. Minimizing $\chi^2(a, b)$ means finding the values of a and b, where the appropriate derivatives are zero, *i.e.*:

$$\begin{cases} \dfrac{\partial \chi^2(a,b)}{\partial a} = 2\displaystyle\sum_{i=1}^{N} \frac{a + bt_i - R_i(t_i)}{\sigma_i^2} = 0 \\ \dfrac{\partial \chi^2(a,b)}{\partial b} = 2\displaystyle\sum_{i=1}^{N} \frac{a + bt_i - R_i(t_i)}{\sigma_i^2} t_i = 0 \end{cases}, \text{ or } \begin{cases} a\displaystyle\sum_{i=1}^{N} \frac{1}{\sigma_i^2} + b\sum_{i=1}^{N} \frac{t_i}{\sigma_i^2} = \sum_{i=1}^{N} \frac{R_i(t_i)}{\sigma_i^2} \\ a\displaystyle\sum_{i=1}^{N} \frac{t_i}{\sigma_i^2} + b\sum_{i=1}^{N} \frac{t_i^2}{\sigma_i^2} = \sum_{i=1}^{N} \frac{R_i(t_i)t_i}{\sigma_i^2} \end{cases}. \tag{2}$$

Solution of (2) is the following:

$$a = \frac{1}{\Delta}\left(\sum_{i=1}^{N}\frac{R_i(t_i)}{\sigma_i^2}\sum_{i=1}^{N}\frac{t_i^2}{\sigma_i^2} - \sum_{i=1}^{N}\frac{R_i(t_i)t_i}{\sigma_i^2}\sum_{i=1}^{N}\frac{t_i}{\sigma_i^2}\right),$$

$$b = \frac{1}{\Delta}\left(\sum_{i=1}^{N}\frac{R_i(t_i)t_i}{\sigma_i^2}\sum_{i=1}^{N}\frac{1}{\sigma_i^2} - \sum_{i=1}^{N}\frac{R_i(t_i)}{\sigma_i^2}\sum_{i=1}^{N}\frac{t_i}{\sigma_i^2}\right), \tag{3}$$

$$\text{where } \Delta = \sum_{i=1}^{N}\frac{1}{\sigma_i^2}\sum_{i=1}^{N}\frac{t_i^2}{\sigma_i^2} - \left(\sum_{i=1}^{N}\frac{t_i}{\sigma_i^2}\right)^2.$$

Having obtained a and b, the value of resistance R_{pred} for t_{pred} can be predicted at any time after the last calibration:

$$R_{pred}(t_{pred}) = a + bt_{pred}\,. \tag{4}$$

The reference time from which the time is counted can be chosen arbitrarily; the value of a obtained depends on the choice. From now on, the time of the first calibration will be considered as the reference time for both t_i and t_{pred}, and a day as a unit of time. Thus, the t_{pred} is the number of days elapsed since the first calibration until the day for which the resistance value is to be predicted.

The mean standard deviation of a single value measured from that predicted can be obtained using the expression derived in [14]:

$$s = \sigma_R\left(1 + \frac{1}{N} + \frac{(t_{pred} - \bar{t})^2}{\sum_{i=1}^{N}(t_i - \bar{t})^2}\right)^{1/2}, \tag{5}$$

where $\sigma_R = \sqrt{\frac{1}{N-2}\sum_{i=1}^{N}[a + bt_i - R_i(t_i)]^2}$ is the mean standard deviation of the resistance values obtained by the calibration from the regression line, and $\bar{t}$ is the average of the calibration times. Expanded uncertainty U of the resistance value predicted is obtained by multiplying the standard deviation by the t-factor $t_p(\nu)$ of Student's distribution, where the coverage probability p is 0.95 and the number of degrees of freedom ν is $N - 2$:

$$U = t_{0.95}(N-2)s \tag{6}$$

(the number of degrees of freedom is $N - 2$ because the two magnitudes, a and b, are computed from N calibration results; when N is large enough, $t_{0.95}(N - 2)$ is close to 2).

Figure 2 reveals calibration histories of two standard resistors: (a) 1 Ω model 5685A by *Tinsley* (s/n 279451); (b) 10 kΩ model SR104 by *Tegam* (s/n K201090230104). In the case of (a), calibration results (blue squares and red circles) were obtained by calibrating the standard 1 Ω resistor by *Tinsley* against a QHR (Quantum Hall Resistance) system at foreign laboratories – *Physikalisch-Technische Bundesanstalt* (PTB), Germany; *National Physical Laboratory* (NPL), United Kingdom; *Český metrologický institut* (CMI), Czech Republic – while the data obtained with the linear regression are shown with a line, which is dotted in the left area, and solid in the

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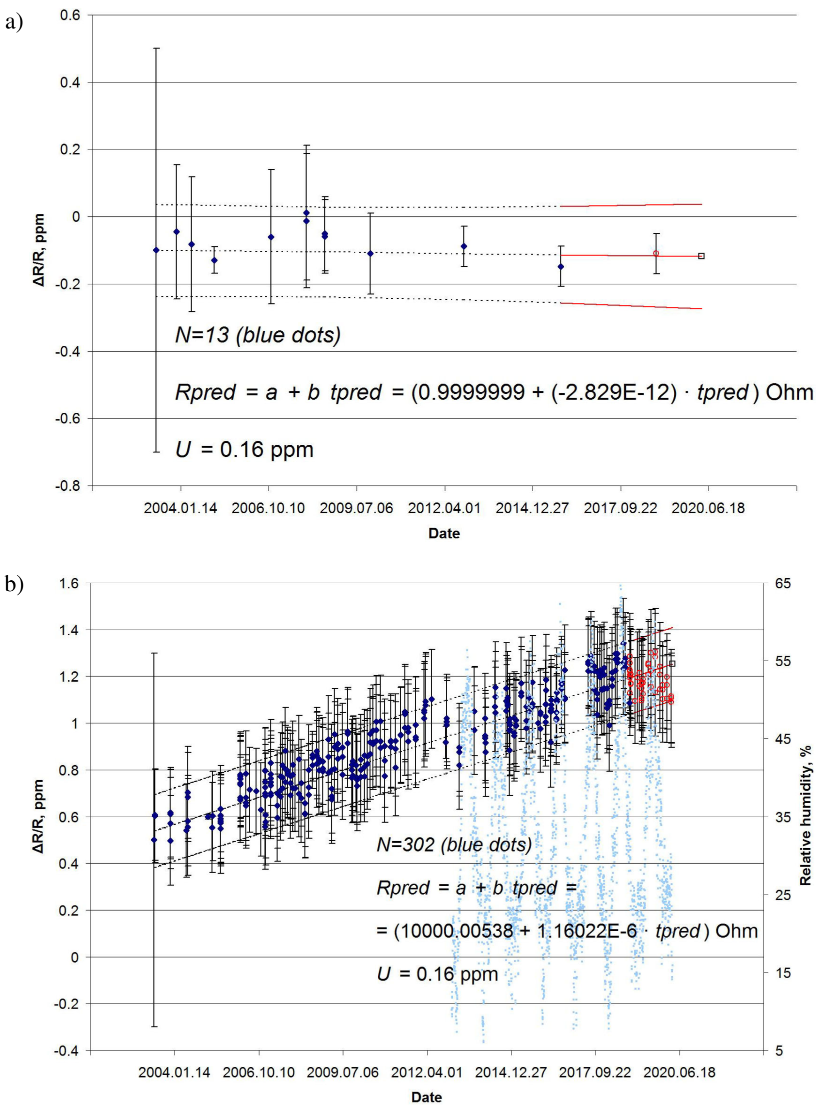

Fig. 2. Calibration histories with prediction lines and uncertainties: for a 1 Ω *Tinsley* 5685A standard resistor (a); for the 10 kΩ *Tegam* SR104 standard resistor (b). ΔR is the difference between the measured and the nominal value of the resistance; blue squares denote the calibrations used for the prediction, while red circles denote the calibrations performed later. Light blue dots in (b) denote relative humidity.

right area. The dotted part of the regression line is a linear approximation of the calibration results shown by the blue squares, while the solid part describes the predicted resistance behaviour for the near future, where calibration results are depicted with the red circles; these results were not taken into account while calculating the regression parameters. Thus, the solid part of the regression line and the lower and upper lines defining the prediction uncertainty interval indicate whether the prediction is adequate for a particular period. The uncertainty interval U depends not only on calibration uncertainty but also on the prediction date – the later the date, the wider the uncertainty interval (although sometimes this trend may be insignificant). The resistor is stable, with the prediction uncertainty $U < 0.16\ \mu\Omega/\Omega$, within a calibration interval since the last calibration; it is why its predicted resistance is ideally suitable for calibration of decade resistors (10 Ω, 100 Ω, 1 kΩ and 10 kΩ) which is based upon subsequent measurements of the ratio 10:1. While calibrating a 10 kΩ standard resistor, the four ratio measurements: $r_1 = \frac{10\ \Omega}{1\Omega}$, $r_2 = \frac{100\ \Omega}{10\ \Omega}$, $r_3 = \frac{1\ \text{k}\Omega}{100\ \Omega}$, and $r_4 = \frac{10\ \text{k}\Omega}{1\ \text{k}\Omega}$ – are made. Generally, the value R_c of the resistance calibrated is calculated as

$$R_c = R_{pred}\, r_1 r_2 \ldots r_n, \tag{7}$$

where R_{pred} is the resistance value of the 1 Ω standard resistor predicted for the day of calibration, and r_i is the ratio measured.

In a similar manner, the behaviour of the 10 kΩ standard resistor by *Tegam* was predicted (see Fig. 2b). Some of the calibration results depicted were obtained by calibrating against a QHR system abroad, while others were obtained by means of ratio measurements. Such a cross-calibration allows not only obtaining a more accurate prognosis, but also verifying the correctness of the ratio measured by the bridge. The prediction uncertainty U for this resistor changes little with time, and it is about $0.16\ \mu\Omega/\Omega$ within a time period much longer than a typical calibration interval since the last calibration; therefore, its predicted value is used for the calibration of the 10 kΩ group as well as decade resistors of up to 1 GΩ (or 1 TΩ, if a special method for a parallel connection is used). As Fig. 2 reveals, the regression curves obtained with the least-squares method fit the experimental results well. Although the drift of the resistance is significant, it is linear and stable, which allows a reliable prediction of the value of resistance between calibrations to be made which is an important feature of the standard.

Figure 3 reveals the behaviour of the 10 kΩ *Tegam* SR104 standard resistor and the variation of ambient air pressure and humidity in more detail. The data depicted imply that although there is some relation between the resistance and ambient conditions, it is not straightforward.

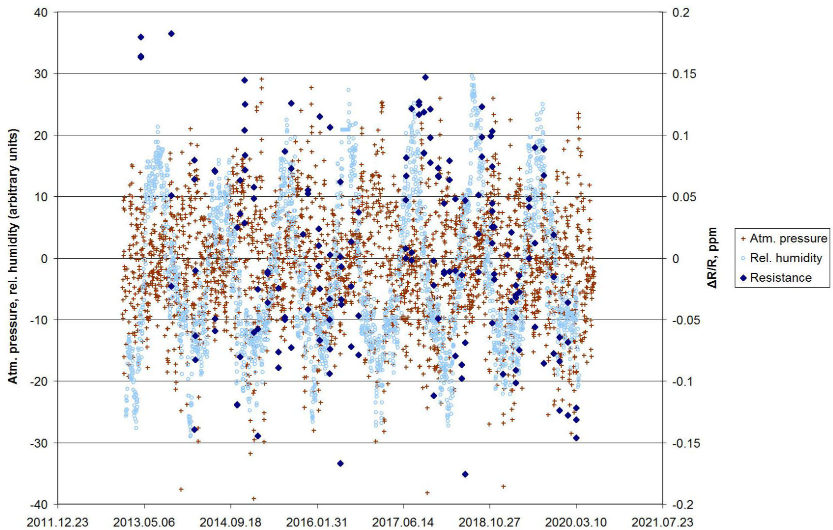

Fig. 3. Behaviour of the resistance of the 10 kΩ *Tegam* SR104 standard resistor and the variation of ambient air pressure and humidity with respect to their mean values. In this graph, ΔR is the difference between the measured and the predicted value.

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Now, let us consider the case of the model 5685B 1 kΩ decade resistor by *Tinsley* (s/n 279757). At the beginning of 2011, a problem was identified with it – supposedly, due to a mechanical disturbance of contacts. When the contacts were recovered, a large jump of resistance occurred, and the calibration history was disrupted. Figure 4a reveals the behaviour of the resistor prior to the disruption, while Fig. 4b reveals the recovery afterwards.

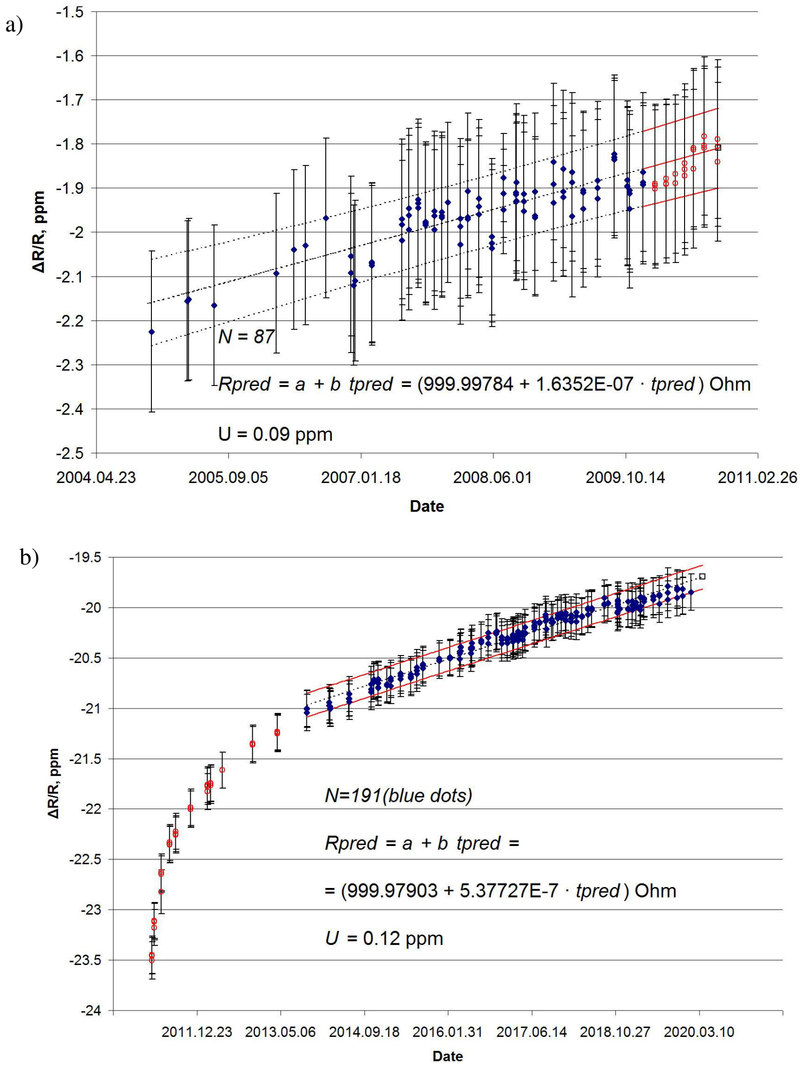

Fig. 4. Calibration histories with prediction lines and uncertainties for the model 5685B 1 kΩ decade resistor by Tinsley (s/n 279757) before (a) and after (b) the disruption due to mechanical disturbance. ΔR is the difference between a measured and the nominal resistance value. As in Fig. 2, the blue squares denote the calibrations used for the calculations, and the red circles denote the calibrations not taken into account.

The behaviour of the resistor stabilized fully only in the end of 2013, and the calibration history recorded since that time allows predicting the resistance value with the uncertainty $U \approx 0.12\ \mu\Omega/\Omega$.

Calibration of the USSR-made 1 GΩ standard resistor model P4083 (s/n 3450) is performed through measuring five ratios – $\frac{100\ \text{k}\Omega}{10\ \text{k}\Omega}, \frac{1\ \text{M}\Omega}{100\ \text{k}\Omega}, \ldots, \frac{1\ \text{G}\Omega}{100\ \text{M}\Omega}$. The resistance value is calculated as in (6), where R_{pred} is the resistance value of the 10 kΩ standard resistor predicted for the day of calibration. Similarly, the behaviour of the 1 GΩ standard resistor was predicted (see Fig. 5). The prediction uncertainty U for this resistor is about 7 μΩ/Ω. Resistance of such a large-value resistor, at least in some cases, tends to decrease with the relative humidity increasing.

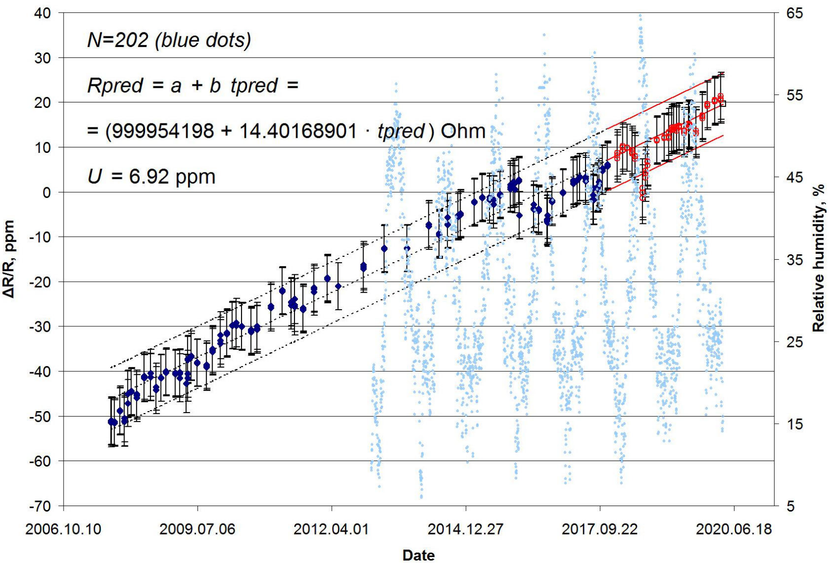

Fig. 5. Calibration history with prediction lines and uncertainty for the USSR-made model P4083 1 GΩ decade resistor (s/n 3450). ΔR is the difference between the measured and the nominal resistance value. As in Figs. 2 and 4a, the blue squares denote the calibrations used for the calculations, and the red circles denote the calibrations not taken into account. Small light blue circles denote relative humidity.

As a result of our work, the Lithuanian National Electrical Standards Laboratory has declared the uncertainties of the resistance values as follows: 0.3 μΩ/Ω for 1 Ω, 0.4 μΩ/Ω for 1 kΩ, 0.3 μΩ/Ω for 10 kΩ, and 7.1 μΩ/Ω for 1 GΩ [13].

3. Conclusions

The standard resistors operated by the Lithuanian National Electrical Standards feature stable drift of resistance which is well predicted by means of linear regression. The uncertainties of resistance values obtained taking into account the drift are well below those stated by the laboratory in the CMC published by the BIPM.

Even the Tinsley's model 5685B 1 kΩ decade resistor, which has undergone mechanical disturbance, after a period of recovery, features good predictability, although the drift rate is almost four times higher than that before the disturbance.

A resistor's resistance is affected by ambient humidity if the resistor is not kept in the oil bath. This becomes more obvious at high resistance values. Therefore, controlling humidity in the laboratory may yield a slight improvement in stability and predictability of those resistors.

The results presented are consistent with the results of the key comparisons [15, 16].

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