Maintenance of the reference resistance standards of the Primary Electromagnetic Laboratory in Croatia

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Abstract – The past calibrations of the reference resistance standards of the Primary Electromagnetic Laboratory (PEL), the Leeds&Northrup (L&N) 1 Ω and 10 kΩ standards, carried out over a period of more than thirty years, have been analysed. The least-squares fitting was used and the regression coefficients of up to the 3rd order polynomial were calculated and evaluated, by which their resistances can be predicted for a moment of interest. Both standards have been maintaining into the self-developed oil ultrathermostat, where a stable temperature of 23 °C is maintained by the computer-based regulator within the limits of ±10 mK. Therefore, in the paper the analysis of the standards itself, as well as the achievement in the temperature regulation, as the basis of their use as reference standards, are described and pointed out.

I. Analysis of the long-term drift of reference resistance standards

The Primary Electromagnetic Laboratory is a part of the Faculty of Electrical Engineering and Computing of the University of Zagreb, as well as a part of the Croatian metrology system. Its main task is to maintain the reference standards in the field of electromagnetic quantities, and to ensure their traceability to the international level [1]. The basic quantities of the traceability chain of PEL are: voltage, capacitance, resistance, and frequency. Since the reference standards of voltage and capacitance are described elsewhere [2, 3], here we can concentrate on the analysis of the reference standards of resistance, placed into the special oil ultrathermostat UTEO-97, where the reference temperature of 23 °C is maintained [4]. The other resistance standards of PEL, as well as their maintenance and methods of comparison, were presented in [5].

Two reference resistance standards of PEL are: L&N 4210 1 Ω (S/N 1730950, in further text marked as RR1), and L&N 4040-B 10 kΩ (S/N 1703018, RR10k). For many years they had been used in PTB (Physikalisch-Technische Bundesanstalt, Braunschweig, Germany) and maintained at a temperature of 20 °C (R_{20}). During that period their calibration data were obtained and expressed for the same temperature, and the coefficients α_{20} and β_{20} were determined for both standards. In 1996 they were kindly donated to the PEL, where they have been maintained at a temperature of 23 °C, and even after regularly calibrated at PTB, but this time at a temperature of 23 °C. If we want to combine the calibration data obtained when the standards were kept and calibrated at different temperatures, it is necessary to express their resistances for the same temperature. Nowadays they have been maintaining at a temperature of 23 °C, and we choose that temperature to be a reference temperature. Using the known coefficients α_{20} and β_{20}, with $\Delta\vartheta = 3$ °C, the resistance at 23 °C (R_{23}) is equal to:

$$R_{23} = R_{20}\,(1 + \Delta\vartheta\,\alpha_{20} + \Delta\vartheta^2\beta_{20}). \quad (1)$$

Furthermore, to combine the results obtained before and after 1990, it is necessary to involve the correction of the Ω_{PTB} to the SI *ohm*, starting with January 1, 1990 [6, 7], and therefore the final values of resistances (R), taken for the further analysis, were calculated as follows:

$$R = R_{23}\,(1 - 0{,}56 \cdot 10^{-6}). \quad (2)$$

Except of the change of resistance due to the change of temperature, the coefficients α and β have temperature dependence, too, and for a reference temperature of 23 °C they are:

$$\alpha_{23} = (\alpha_{20} + 2\Delta\vartheta\,\beta_{20})\,/\,(1 + \Delta\vartheta\alpha_{20} + \Delta\vartheta^2\beta_{20}), \qquad \beta_{23} = \beta_{20}\,/\,(1 + \Delta\vartheta\,\alpha_{20} + \Delta\vartheta^2\beta_{20}). \quad (3)$$

Using the previous three equations we were able to calculate the corrected calibration data and coefficients for both resistance standards, and incorporate into the analysis of their long-term drift and prediction of their values in time, which is showed in the next few subsections.

A. Least-squares method used in analysis

The analysis for both resistance standards was performed using the least-squares method [8, 9], with weights $p = 1$ for all calibration data (due to the fact that the uncertainties of the older calibration data are not known), and for polynomials of the first, second and third order. So, for the fitting function

$$R(t) = K + at\,, \tag{4}$$

the following system of equations needs to be solved:

$$\begin{aligned} [p]K + [pt]a &= [pR] \\ [pt]K + [pt^2]a &= [ptR], \end{aligned} \tag{5}$$

where Gauss's notation is used for the sums, i.e. $[ptR] = \sum_{i=1}^{n} p_i t_i R_i$ with R_i as the resistance, calculated by (1) and (2) for a particular calibration day t_i, and n is the number of data. Similarly, for the function

$$R(t) = K + at + bt^2\,, \tag{6}$$

the system of equations is

$$\begin{aligned} [p]K + [pt]a + [pt^2]b &= [pR] \\ [pt]K + [pt^2]a + [pt^3]b &= [ptR] \\ [pt^2]K + [pt^3]a + [pt^4]b &= [pt^2R]. \end{aligned} \tag{7}$$

When the fitting function is the polynomial of the third order,

$$R(t) = K + at + bt^2 + ct^3\,, \tag{8}$$

the used system of equations is

$$\begin{aligned} [p]K + [pt]a + [pt^2]b + [pt^3]c &= [pR] \\ [pt]K + [pt^2]a + [pt^3]b + [pt^4]c &= [ptR] \\ [pt^2]K + [pt^3]a + [pt^4]b + [pt^5]c &= [pt^2R] \\ [pt^3]K + [pt^4]a + [pt^5]b + [pt^6]c &= [pt^3R]. \end{aligned} \tag{9}$$

If $v_i = R_i - R(t_i)$, i.e. the difference between the calibration and the regression value for a particular t_i, and k is the number of unknowns, the standard deviation of single data can be calculated as follows:

$$m = \sqrt{\frac{[pvv]}{n-k}}\,. \tag{10}$$

The systems (5), (7) and (9) can be expressed in the matrix form as $\boldsymbol{Nx} = \boldsymbol{n}$, where $\boldsymbol{N}$ is the matrix of coefficients of normal equations (sums on the left sides of systems of equations), $\boldsymbol{x}$ is the vector of unknowns and $\boldsymbol{n}$ is the vector of absolute members (sums on the right sides). If we calculate the inverse matrix of $\boldsymbol{N}$, i.e. $\boldsymbol{Q} = \boldsymbol{N}^{-1}$, the uncertainty of the predicted value of resistance for a particular day t_p can be calculated for the regression functions (4), (6) and (8), respectively:

$$u_\mathrm{p} = m\sqrt{Q_{11} + 2t_\mathrm{p}Q_{12} + t_\mathrm{p}^2 Q_{22}}\,, \tag{11}$$

$$u_\mathrm{p} = m\sqrt{Q_{11} + 2t_\mathrm{p}Q_{12} + t_\mathrm{p}^2(2Q_{13} + Q_{22}) + 2t_\mathrm{p}^3 Q_{23} + t_\mathrm{p}^4 Q_{33}}\,, \tag{12}$$

$$u_\mathrm{p} = m\sqrt{Q_{11} + 2t_\mathrm{p}Q_{12} + t_\mathrm{p}^2(2Q_{13} + Q_{22}) + 2t_\mathrm{p}^3(Q_{14} + Q_{23}) + t_\mathrm{p}^4(2Q_{24} + Q_{33}) + 2t_\mathrm{p}^5 Q_{34} + t_\mathrm{p}^6 Q_{44}}\,, \tag{13}$$

where $Q_{11} \ldots Q_{44}$ are the elements of matrix $\boldsymbol{Q}$, and m is calculated for a chosen regression function by (10). Equations (4) to (13) are the basis for the calculation of the parameters of interest, results of which for both standards are pointed out in the next two subsections.

B. Calibration data and their analysis for reference 1 Ω standard

Calibration data of RR1 are presented in Table I, as well as in Fig. 1. In the second column, the mean calibration date is given, followed by the associated day and relative deviation from the nominal value, where $\delta R = (R/R_n - 1)$ and $R_n = 1\ \Omega$. This relative deviation is given in ppm (parts per million).

Table I. Calibration data of RR1, expressed as relative deviations from the nominal value

No.	Date	t/day	δR/ppm	No.	Date	t/day	δR/ppm	No.	Date	t/day	δR/ppm
1	15.07.1969.	0	−6,780	12	23.03.1983.	4999	−7,275	22	27.10.1987.	6678	−7,454
2	15.05.1971.	669	−7,040	13	15.09.1983.	5175	−7,289	23	20.12.1987.	6732	−7,448
3	01.11.1972.	1204	−7,120	14	15.02.1984.	5328	−7,243	24	11.03.1988.	6814	−7,394
4	01.05.1976.	2482	−7,190	15	15.04.1984.	5388	−7,241	25	24.03.1988.	6827	−7,447
5	15.02.1978.	3137	−7,257	16	08.05.1985.	5776	−7,273	26	23.10.1988.	7040	−7,469
6	20.05.1978.	3231	−7,274	17	17.06.1986.	6181	−7,435	27	08.03.1989.	7176	−7,460
7	18.08.1978.	3321	−7,300	18	08.07.1986.	6202	−7,433	28	24.09.1996.	9933	−7,700
8	31.12.1978.	3456	−7,326	19	15.04.1987.	6483	−7,476	29	23.10.1998.	10692	−7,550
9	11.01.1980.	3832	−7,332	20	17.06.1987.	6546	−7,456	30	28.12.2000.	11489	−7,670
10	02.07.1980.	4005	−7,370	21	20.08.1987.	6610	−7,448	31	08.04.2003.	12320	−7,694
11	20.05.1982.	4692	−7,306								

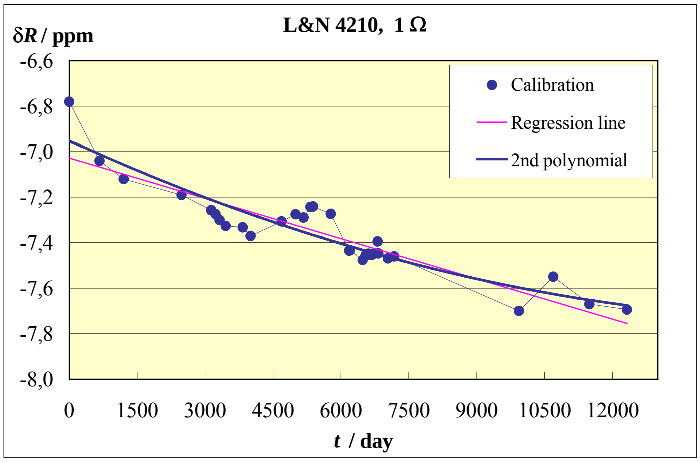

Fig. 1. Calibration data of RR1 (relative deviations from 1 Ω); the starting day is July 15, 1969

In Table II the parameters of fitting functions (4), (6) and (8) are pointed out, while in the last two rows of the same table the predicted values of resistance for September 30, 2004 ($t_p = 12861$ day), and their uncertainties, are presented as well. These values are calculated by expressions (4) to (13).

Table II. Calculated parameters of three fitting functions, as well as the predicted values of resistance on September 30, 2004

	Eq. (4)	Eq. (6)	Eq. (8)
K/Ω	0,999992972	0,999993048	0,999993107
$a/(\Omega/\text{day})$	−5,9011254E−11	−9,1024462E−11	−1,5214379E−10
$b/(\Omega/\text{day}^2)$	/	2,6171723E−15	1,5500006E−14
$c/(\Omega/\text{day}^3)$	/	/	−7,0353372E−19
m/Ω	7,71124E−08	7,17029E−08	6,85861E−08
m_K/Ω	3,06750E−08	4,30994E−08	5,15620E−08
$m_a/(\Omega/\text{day})$	4,86465E−12	1,43328E−11	3,49978E−11
$m_b/(\Omega/\text{day}^2)$	/	1,11186E−15	6,87014E−15
$m_c/(\Omega/\text{day}^3)$	/	/	3,70657E−19
δR/ppm	−7,787	−7,689	−7,782
u_p/ppm	0,038	0,054	0,071

Finally, we are able to evaluate the obtained results. To choose the best regression function one point of view could be the lowest value of u_p for the day of interest, and in that case function (4) would be the best choice. Another point of view could be the best approximation of the latest calibration data, i.e. the values that describe the change of resistance for the near past. Following this idea, function (6) has advantage because the predicted value of resistance for the chosen date is the closest to the last two calibration data (no. 30 and 31 in Table I) that can be easily espied in Fig. 1. Comparing the predicted values of resistance for the chosen day (Table II), functions (4) and (8) gave almost the same result, while the value reached by (6) is ≈0,1 ppm higher from both of them (exactly 0,098 ppm and 0,093 ppm, respectively). If we compare that difference with the calculated values of u_p, it is approximately 2,5 times u_p of (4), 1,8 times u_p of (6) and 1,35 times u_p of (8). This means that this difference is significant comparing to the calculated uncertainties of predicted resistances with different functions. It seems therefore that the function (6), i.e. *the 2nd order polynomial approximation* is the best choice to describe the change of its resistance with time and for the prediction of its value for the day of interest. Furthermore, the parameters calculated by (3) are: $\alpha_{23} = 4{,}28 \cdot 10^{-6}\ \mathrm{K}^{-1}$, and $\beta_{23} = -0{,}52 \cdot 10^{-6}\ \mathrm{K}^{-2}$.

C. Calibration data and their analysis for reference 10 kΩ standard

Calibration data of RR10k, expressed as relative deviations from the nominal value, are presented in Table III, as well as in Fig. 2. The meanings of columns are the same as for Table I, and $R_n = 10$ kΩ is used in the calculation of δR.

Table III. Calibration data of RR10k, expressed as relative deviations from 10 kΩ

No.	Date	t/day	δR/ppm
1	15.06.1970.	0	–18,50
2	15.07.1971.	395	–17,40
3	02.08.1972.	2605	–13,05
4	19.08.1978.	2987	–12,24
5	15.02.1981.	3898	–11,33
6	15.01.1985.	5328	–8,20
7	24.09.1996.	9598	–5,40
8	23.10.1998.	10357	–4,05
9	27.12.2000.	11153	–3,59
10	03.04.2003.	11980	–3,12

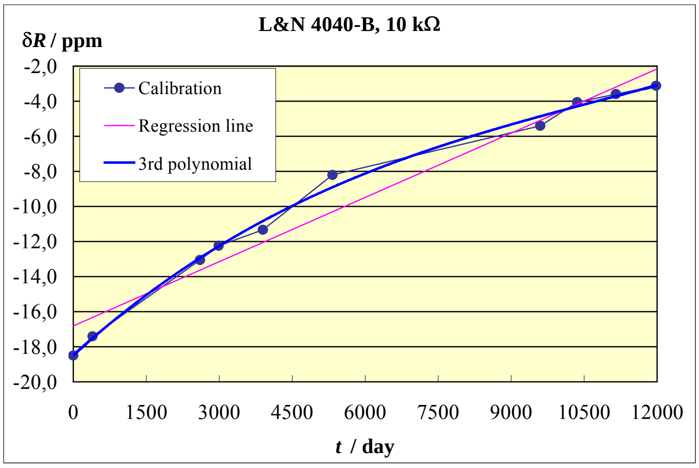

Fig. 2. Calibration data of RR10k; the starting day is June 15, 1970

As well as in Table II, in Table IV the parameters of fitting functions, the predicted values of resistance for September 30, 2004 ($t_p = 12526$ day), and their uncertainties are pointed out.

Table IV. Calculated parameters of three fitting functions, as well as the predicted values of resistance on September 30, 2004

	Eq. (4)	Eq. (6)	Eq. (8)
K / Ω	9999,8319	9999,8169	9999,8152
a / (Ω/day)	1,2221597E–05	2,2049594E–05	2,5144865E–05
b / (Ω/day^2)	/	–8,0337784E–10	–1,5977177E–09
c / (Ω/day^3)	/	/	4,7596707E–14
m / Ω	1,18750E–02	4,60651E–03	4,35396E–03
m_K / Ω	6,30512E–03	3,29298E–03	3,35597E–03
m_a / (Ω/day)	8,68746E–07	1,48523E–06	2,68142E–06
m_b / (Ω/day^2)	/	1,18242E–10	5,96852E–10
m_c / (Ω/day^3)	/	/	3,51307E–14

δR / ppm	−1,505	−3,298	−2,700
u_p / ppm	0,692	0,376	0,567

Similar analysis could be done for that standard, as we did in subsection I.B for the RR1. The presented results (Table IV and Fig. 2) clearly show that the regression line, i.e. function (4), is absolutely unsuitable for the approximation of the resistance change with time. For example, from calibration data no. 9 and 10 (Table III) follows the change of resistance of 0,47 ppm for a period of 827 days. However, the value predicted by (4) for the chosen day is 1,62 ppm higher than the last calibration data (no. 10) but, due to the fact that the according time interval is only 546 days, it should be higher for only 0,31 ppm. This means that we need to choose between the polynomial of the second and third order. The function (6) gave the smallest uncertainty u_p of all three functions for the chosen day (0,376 ppm), but the predicted value of resistance didn't suit the latest calibration data, or the change of resistance in time that follows from them. The obtained value of δR is −3,298 ppm, which lies between the data no. 9 and 10 in Table III. However, one can expect the rise of the resistance in time, and following that the predicted value should be somewhat higher than the last calibration data. On the other hand, function (8) gave a larger uncertainty u_p, but the predicted value was −2,7 ppm, which is in a better agreement with the real situation. Following the idea that the best approximation should fit match with the latest calibration data, the final conclusion is that the function (8), i.e. *the 3rd order polynomial approximation* is a better choice, although it gives larger uncertainty (for the presented example about 50 % than the second order polynomial). Finally, the parameters calculated by (3) for RR10k are: $\alpha_{23} = 6,28 \cdot 10^{-6}\ \mathrm{K}^{-1}$, $\beta_{23} = -0,58 \cdot 10^{-6}\ \mathrm{K}^{-2}$.

II. Ultrathermostat UTEO-97

The resistance of RR1 and RR10k varies with the temperature for $\approx 1 \cdot 10^{-5}\ \mathrm{K}^{-1}$, and the associated relative uncertainty due to the changes of temperature will be less than $1 \cdot 10^{-7}$ if the temperature is maintained within ±10 mK. This is the basic requirement to be met by the regulator, although there are some examples that a better stability can be achieved [10]. Due to the position (near the floor) and ambient circumstances in the room in which the UTEO-97 is kept, the regulation is based only on heating up the oil within the ultrathermostat to a reference temperature of 23 °C. The basic parts of the UTEO-97 regulator (Fig. 3) are: a multichannel DMM, NTC resistors, a PC with a built-in GPIB card, and a D/A card as an output part. The measurement data are transferred to the computer, and within the control program, the voltage needed for the heating of oil is calculated. Via the D/A card, that voltage is led to the heater inside the ultrathermostat. The operation of the regulator and the changes of temperature are observed on the screen, whereas the measured data are stored on the disc.

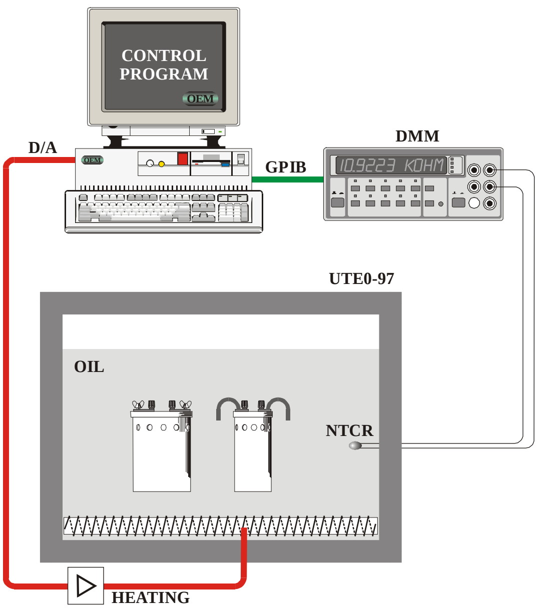

Fig. 3. Measuring system controlled by the computer for the measurement and regulation of the oil temperature inside the ultrathermostat UTEO-97

As temperature sensors we used the NTCRs of type UUA41J1 and UUA41J8 [11], with $R_N = 10\ \mathrm{k\Omega}$ at 25 °C and limits of error ±0,2 °C in the range (0 to 70) °C and (0 to 100) °C, respectively. To calculate the measured Celsius temperature ϑ_m from the measured resistance R_m, the following approximation curve is applied:

$$\vartheta_m = \frac{\ln(R_{T_0}/R_m)}{A - \alpha \ln(R_{T_0}/R_m)}, \qquad (14)$$

where the constants A and α should be determined for a particular NTCR if we want to achieve narrow limits of errors, and $R_{T_0} = 32650\ \Omega$ at temperature $T_0 = 273,15$ K; more details are presented in [12].

In Fig. 4 the temperature differences to a reference temperature of 23 °C are presented (in mK, left *y*-axis) as follows: a) for the channel used as the information for the regulator about the achieved temperature (mark "oil"), b) for RR1 ("1 ohm"), and c) for RR10k ("10 kiloohm"); right *y*-axis shows ambient temperature. The temperature stability can be as low as ±1 mK during a single day, or even through the longer period of time. Furthermore, since the temperatures are measured near by standards, their momentary values are known and can be used if corrections are needed, even within ±10 mK. In that way the influence of temperature instability become negligible, and uncertainty is determined by measurements of its absolute value by means of the used sensors, which is estimated to be 30 mK, mostly due to the uncertainty of their calibration.

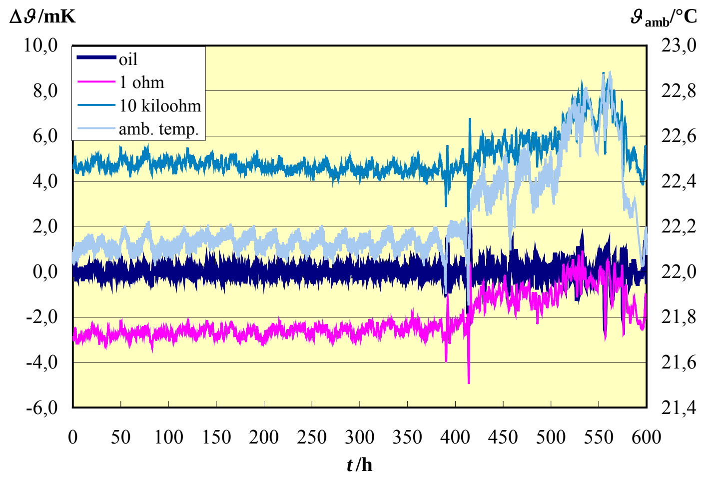

Fig. 4. The changes of temperature inside the ultrathermostat as well as of ambient temperature for a period of 25 days

III. Conclusion

The presented analysis of the RR1 and RR10k shows that the uncertainty of the predicted resistance for a particular day depends of the used regression function. If we choose a day two years after the last calibration date (as the worst case between two calibrations), the following values of u_p for the best approximation function of each standard will be obtained: 0,057 ppm and 0,644 ppm, respectively. However, another contribution to the overall uncertainty is due to the temperature (0,3 ppm for both standards), and the combined uncertainties could be 0,4 ppm and 0,8 ppm for RR1 and RR10k. Obviously, to achieve a lower combined uncertainty, a better measurement of temperature is needed.

References

[1] J. Butorac, V. Bego and D. Ilić, "The basis of Croatian electromagnetic metrology", *CPEM ’96 Digest*, pp. 41–42, Braunschweig, June 17–20, 1996.

[2] D. Ilić and D. Vujević, "Analysis of the long-term stability of the Zener-based reference voltage standard", *Digest of the HMD 18th Metrology Symposium*, pp. 5−9, Cavtat, October 8−10, 2001.

[3] D. Ilić, J. Butorac and B. Ferković, "Long-term stability of 100 pF capacitance standards", *Digest of the HMD 18th Metrology Symposium*, pp. 32–35, Cavtat, October 8–10, 2001.

[4] V. Bego, J. Butorac and D. Ilić, "PC-based regulator for oil ultrathermostat", *Proc. of the 10th IMEKO TC-4 Symposium*, vol. I, pp. 201–204, Naples, September 17–18, 1998.

[5] V. Bego, J. Butorac and R. Malarić, "Maintenance of Croatian resistance standards", *Proc. of the XIV IMEKO World Congress*, vol. IVA, pp. 161−166, Tampere, June 1−6, 1997.

[6] ***, Changing to the 1990 Volt and Ohm, Application Note, John Fluke Mfg. Co., Inc., 1989.

[7] B.N. Taylor and T.J. Witt, "New international electrical reference standards based on the Josephson and quantum Hall effects", *Metrologia*, vol. 26, pp. 47–62, 1989.

[8] N. Čubranić, *Theory of errors* (in Croatian), Tehnička knjiga, Zagreb, 1967.

[9] B.E. Cooper: *Statistics for Experimentalists*, Pergamon Press, Oxford, New York, Toronto, Sydney, Paris, Braunschweig, 1969.

[10] H. Leontiew: "Flüssigkeitsthermostat sehr hoher Temperaturkonstanz mit neu entwickelter Temperaturregelung", *PTB-Mitteilungen*, 98. Jahrgang, Heft 6, S. 392–394, 1988.

[11] Fenwal Electronics: Thermistoren UNI Kurve (Widerstands-Temperatur-Tabellen); Standard Typ: UUA41J1, UUA41J8; 10000 ohm bei 25 °C.
[12] V. Bego, D. Ilić and N. Hlupić: "Temperature measurement by means of NTC resistors", *Proc. of the 10th IMEKO TC-4 Symposium,* vol. II, pp. 783–787, Naples, September 17–18, 1998.