

ZBIGNIEW KŁOS

Wrocław University of Technology
Institute of Electrical Machines, Drives and Metrology

CALIBRATION OF EXTREMELY SENSITIVE RANGES OF DC PICOAMMETERS WITH ANALOG ACTIVE I/U CONVERSION

The paper deals with the problem of calibration of extremely sensitive picoammeters with measuring ranges close to limits of the measuring possibilities. The presented method of calibration bases on a resistive source of very low DC standard current, namely a current calibrator. Procedure for the design of a proper calibrator, criteria of optimal selection of high value resistance standards and a detailed analysis of the accuracy of obtained calibrating current have been described. The principle of evaluation of measurement uncertainty and method of assessment of combined uncertainty for calibrated picoammeters with analog and digital display have been introduced. It has been shown that in an averagely equipped measuring and research laboratory and using relatively simply accessible resistance standards with values of $10^{10} - 10^{11} \Omega$, it is possible to calibrate the most sensitive ranges of picoammeters with active i/u conversion.

Keywords: calibration, electrometric picoammeter, active i/u conversion, uncertainty of measurement

1. INTRODUCTION

Measurements of very low DC and low-frequency current appear in scientific research, i.e. on physical phenomena occurring in gaseous and solid bodies, environment protection, biophysics, etc. Also in many technical and industrial domains, measurements of some electrical and non-electrical signals, for instance vacuum control of many technological processes, are connected with the measurement and registration of extremely low DC and low-frequency currents which are often output signals from measuring sensors.

Because of the extreme character of these signals, the influence of occurring disturbances has to be taken into considerations. Moreover, very often disturbances are difficult to distinguish from defects of the measurement system. Therefore, in measurements of such a low currents it is necessary to calibrate and to test the indications of a picoammeter in normal operating conditions, apart from periodical calibration in the Measurement Office.

Only two legislative acts [1 - 4] deal with the calibration problem of all systems of current measurement. They contain only general hints about the choice of the control point on calibrated range in the measuring instrument and desired measurement accuracy relative to maximum errors of the calibrated instrument, whereas the choice of the measuring method and evaluation of the method's uncertainty has been left to the instrument user for individual solution. Moreover, the mentioned legislative acts have not taken into consideration the specific character of the extreme current measurements, which are on the limit of measuring possibilities.

The leading world enterprises producing electrometric measurement systems [5, 6], for comprehensible reasons, do not publish any important data about the calibration of their electrometers. The State Measurement Offices of the greatest countries of the world (USA, United Kingdom, Germany) do not publish detailed data about used methods of calibration but

they offer very expensive calibration stands and current calibrators, whose price is many times higher than the price of the used equipment.

However, the calibrating and testing measurements should be realized by a relatively simple method, unnecessarily with the highest accuracy, which use only a small number of the resistance standards and generic laboratory equipment. One of such calibration methods using a resistive current calibrator, which can be applied for typically used picoammeters with analog i/u conversion, will be presented in this publication.

The most popular type of modern extremely sensitive picoammeters are picoammeters with active i/u conversion. Such instruments have the lowest ranges up to 1 pA. The principle of operation of such picoammeters will be presented in Section 2. Section 3 will be devoted to a special calibration method of sensitive picoammeters based on a resistive current calibrator and to the procedure for the designing of the required current calibrator. In Section 4 an assessment of uncertainty of the proposed calibration method – evaluation of the calibrator's and picoammeter's uncertainties as well as determination of measurement errors at selected control points of a range will be introduced. Section 5 contains a description of the procedure for determination the expanded uncertainty of the instrument – a choice of the most probable error distribution and, consequently, a selection of a coverage probability and a coverage factor. Section 6 summarizes contents of the paper.

2. THE PRINCIPLE OF OPERATION OF THE PICOAMMETER

2.1. The idea of the measurement

The principle of operation of almost all picoammeters with extremely sensitive measuring ranges [5, 7, 8] equal to 1 - 10 pA, based on analog conversion of the measured current in active i/u converter into voltage is presented in Fig. 1.

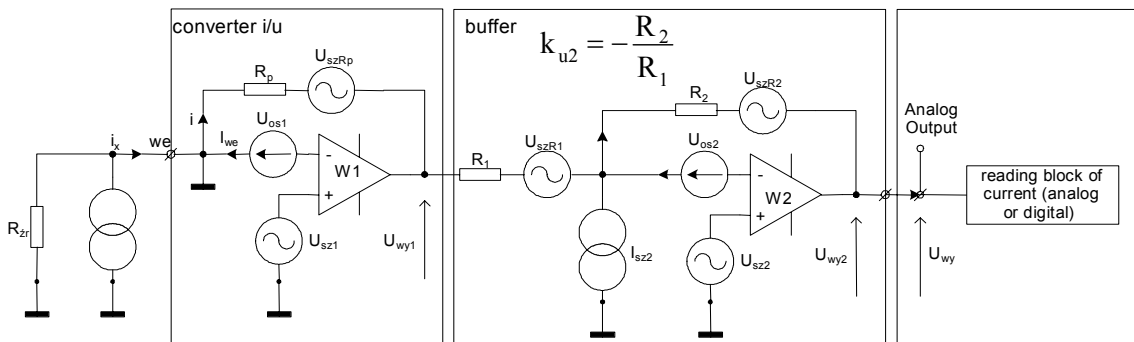


Fig. 1. Scheme of the picoammeter with active i/u converter and with sources of disturbances pointed out.

The measured current i_x is applied to the input of the electrometric operating amplifier W1 characterized by very low bias current (input current) and in the scaling resistor R_p included in the negative feedback loop it is converted to the voltage drop U_{wy1} . This voltage is transmitted to the amplifier's output with negative polarization as follows

$$U_{wy1} = -(i_x + I_{we})R_p. \quad (1)$$

If the bias current of the amplifier is very low ($I_{we} \ll i_x$), then the equation (1) can be reduced to the form

$$U_{wy1} \cong -i_x R_p. \quad (2)$$

In the inverting buffer amplifier W2 the phase of the voltage is inverted. Moreover, this voltage is amplified with the gain $k_{u2} = -(1 - 10)$ V/V to the level of the (1 - 2) V/range.

$$U_{wy2} \cong k_{u2} R_p i_x. \quad (3)$$

In turn, the value of the measured current can be obtained from the approximate expression as

$$i_x = \frac{U_{wy2}}{k_{u2} R_p}. \quad (4)$$

The current can be read from the display of analog or digital voltmeter connected to the output of i/u converter. In such a case the used voltmeter should be scaled directly in current units and the scale of the picoammeter should be linear, because the voltage depends on the current in a linear way.

The output voltage U_{wy2} from the i/u converter, which is proportional to the measured current i_x , is connected with the external output, so-called 'analog output'. Terminals of the analog output can be connected to an additional external register. Apart from instrument reading, the analog output makes possible the continuous registration of the current value i_x . It finds also a practical application among others in calibration of the main functional blocks of the picoammeter - the i/u converter and the block of current readout.

2.2. Measurement ranges

It follows from equation (4) that the lowest measuring range of the picoammeter is limited by the minimal value of the output voltage U_{wy2} of the i/u converter, typically 0.1 - 1 V, and the maximal value of resistance of the precise scaling resistor R_p . Moreover, the lowest measuring range depends indirectly on internal disturbances coming from the active i/u converter, which have not to be indicated in the equation (4). These disturbances - input bias current, offset voltage, internal noise of the amplifier W1 and thermal noise of the resistor R_p - can be represented by the equivalent current sources and voltage sources connected to the circuit in the way presented in Fig.1.

The parasitic voltages of the i/u converter (of amplifier W1 and of the scaling resistor R_p) limit the minimal value of the voltage U_{wy2} to the level 0.1 - 1 V. The input bias current of the best electrometric operation amplifiers [5, 9] is not lesser than 10 - 50 fA, limiting the minimal range of the picoammeter to the level 1 - 10 pA, for which the influence of this current can be omitted ($I_{we} \ll i_x$). In such a situation the value of the scaling resistor R_p necessary to convert the current 1 - 10 pA onto the voltage 0.1 - 1 V is not higher than $10^{11} \Omega$. Unfortunately, resistors

with such large resistance values are not easily available and their accuracy is not good, in the range of 1 - 5%.

The influence of all the mentioned factors limits the most sensitive ranges of modern picoammeters to the level 1- 10 pA, with the accuracy not better than 2 - 5% [5]. Sometimes it is possible to achieve the range of 0.1 pA but only with a large increase of the measurement uncertainty.

2.3. Current measurement inaccuracy

The uncertainty of the current measurement carried out with the use of the indirect method of active i/u conversion can be roughly described by equation (4) which takes a specific form of a ratio of contributing quantities. Therefore, the most convenient way to calculate this uncertainty is as follows

$$\frac{u(i_x)}{i_x} = \sqrt{\left(\frac{u(U_{wy2})}{U_{wy2}}\right)^2 + \left(\frac{u(R_p)}{R_p}\right)^2 + \left(\frac{u(k_{u2})}{k_{u2}}\right)^2}, \quad (5)$$

where: $\frac{u(i_x)}{i_x}$ - relative standard uncertainty of the current i_x ,
 $\frac{u(U_{wy2})}{U_{wy2}}$ - relative standard uncertainty of the i/u converter's output voltage,
 $\frac{u(R_p)}{R_p} = \frac{kl_{Rp}}{\sqrt{3}}$ - relative standard uncertainty of the scaling resistor R_p ,
 kl_{Rp} - accuracy of the scaling resistor R_p ,
 $\frac{u(k_{u2})}{k_{u2}} = \frac{\delta k_{u2}}{\sqrt{3}}$ - relative standard uncertainty of the buffer's gain,
 δk_{u2} - relative admissible error of buffer's gain.

In the case of the most sensitive ranges, less than 100 pA, the influence of the input bias current of the amplifier W1 should be taken into account. As this current is of systematic nature, it should be considered as to be a random variable with rectangular probability density and standard deviation given by

$$S_{I_{we}} = \frac{I_{we}}{\sqrt{3}}, \quad (6)$$

where: I_{we} - maximum value of bias current (from manufacturer's specification), $S_{I_{we}}$ - standard deviation (uncertainty) of input bias current of amplifier.

The input bias current of the i/u converter having direct impact on the accuracy of conversion should be included in equation (4), which then takes the form

$$i_x = \frac{U_{wy2}}{k_{u2}R_p} - I_{we}. \quad (7)$$

For such a form of measurement function, the combined standard uncertainty of the measurement of the current i_x can be given by the general expression [10] as

$$u(i_x) = \sqrt{\left(\frac{\partial i_x}{\partial U_{wy2}}\right)^2 u^2(U_{wy2}) + \left(\frac{\partial i_x}{\partial R_p}\right)^2 u^2(R_p) + \left(\frac{\partial i_x}{\partial k_{u2}}\right)^2 u^2(k_{u2}) + \left(\frac{\partial i_x}{\partial I_{we}}\right)^2 u^2(I_{we})}. \quad (8)$$

After some calculations we get

$$u(i_x) = \sqrt{\left(\frac{1}{k_{u2}R_p}\right)^2 u^2(U_{wy2}) + \frac{1}{3} k I_{Rp}^2 \frac{U_{wy2}^2}{R_p^2 k_{u2}^2} + \frac{1}{3} \delta k_{u2}^2 \frac{U_{wy2}^2}{R_p^2 k_{u2}^2} + \frac{1}{3} I_{we}^2}. \quad (9)$$

The equation (9) has been obtained assuming that the quantities from equation (7) characterized by maximum error like resistance of scaling resistor R_p , gain of buffer k_{u2} and input current I_{we} of amplifier W1 have a rectangular probability distribution, whereas the calculation of uncertainty of output voltage U_{wy2} of the i/u converter is more complicated and it requires some additional explanations.

In the case of analog display the most dominant is the influence of systematic reading error coming from the inaccuracy of the analog display meter. Then the influence of disturbances of stochastic nature can be neglected and the reading error probability distribution can be regarded as rectangular.

On the contrary, when digital display is considered, the maximum error of a digital voltmeter is much lesser than that of an analog one, so that the effect of internal noise and offset voltage of the amplifier and also thermal noise of resistor R_p become more noticeable. A thorough analysis of this problem presented in [7, 8] shows that for the lowest ranges (1 - 10 pA) the measurement error of the voltage U_{wy2} (at the end of the range used) is 1 - 3% when analog display is used, and 0.1 - 0.5% in the case of digital display. Then the maximum error for the most sensitive ranges of picoammeters with active i/u converter is rarely lesser than 1 - 2%.

3. CALIBRATION OF A PICOAMMETER USING RESISTIVE STANDARD SOURCE OF LOW DC CURRENT

3.1. Principle of calibration

Due to lack of reference measuring devices and the unique character of electrometric equipment, calibration of picoammeters in the most sensitive ranges is not possible with the use of conventional methods which can be applied for medium or higher currents, like for example methods of accuracy verification using a standard reference meter. For calibration of the sensitive picoammeters it is required to design and build a dedicated test bed – an adjustable standard

source of very low DC current, so-called current calibrator. Alternatively, such a test bed can be set up using existing devices.

The simplest circuit of the standard source is a resistive current calibrator shown in Fig. 2a.

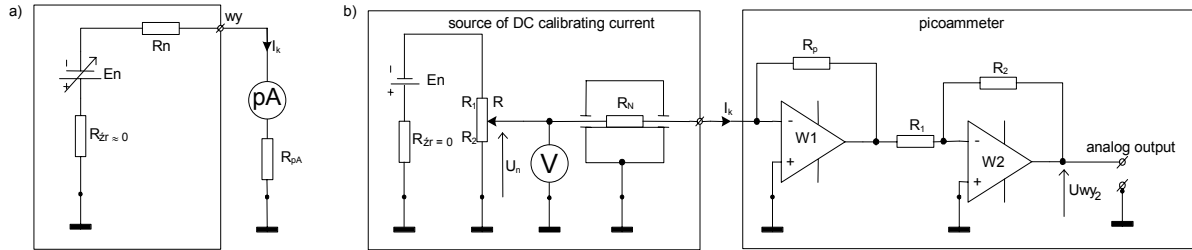


Fig. 2. Calibration of a picoammeter with active i/u converter using a standard source of low DC current: a) principle of operation, b) simplified schematic of source circuit and input circuit of the picoammeter.

The source of a standard current consists of a highly stable low resistance variable voltage source U_n and a high value standard resistor R_N connected in series with the source. The value of U_n is read with the use of a precise digital voltmeter. The calibrator's current is changed by adjustment of the voltage of the source assuming the value of R_N is kept constant. The nominal value of calibrator's current is defined in idle mode (i.e. when output terminals are shortened) by

$$I_{kn} = \frac{U_n}{R_N + R_{zr}} \approx \frac{U_n}{R_N}. \quad (10)$$

This value is assumed to be the reference value of the current feeding the input of the tested picoammeter. However, including picoammeter in measurement circuit makes nominal value of the calibrator's current lower, which results in a systematic error of the method given by

$$\delta_m \cong -\frac{R_{pA}}{R_N}. \quad (11)$$

This error can be taken into account in the form of a correction if only the input resistance of the picoammeter is known. Unfortunately, the input resistance of a picoammeter with an active i/u converter (Fig. 1) is not very precisely defined as

$$R_{pA} \cong -\frac{R_p}{k_{u0}}, \quad (12)$$

where k_{u0} - open loop gain of the operational amplifier W1. Therefore, the value of the resistor R_N should be chosen as high as possible to keep the method's error small enough with respect to the overall accuracy of the calibrator's current i.e.

$$R_N \geq \frac{1}{|\delta_m|} R_{pA}. \quad (13)$$

Additionally, if the internal resistance of the current source is low ($R_{zr} \ll R_N$), picoammeters with active i/u converter suffer from an undesired effect of offset voltage U_{os1} as well as internal noise U_{sz1} amplification (shown in Fig.1 in the form of an equivalent voltage source). The amplification factor of the active i/u converter presented in Fig.1 is defined as follows

$$k_{u/i} = 1 + \frac{R_p}{R_{zr}} \cong 1 + \frac{R_p}{R_N} . \quad (14)$$

Due to this fact, the value of internal resistance of the current calibrator from Fig. 2 described mainly by the value of the series resistor R_N should not be lesser than the value of the scaling resistor R_p in the picoammeter.

In practice, if the following relationship is faithful

$$R_N \geq (0.1 \div 1) R_p , \quad (15)$$

then $k_{u/i}$ is up to 10 times larger. However, even the tenfold increase of the gain $k_{u/i}$ does not cause a significant increase of internal disturbances' level of the i/u converter and, consequently, does not cause significant deviation of the tested picoammeter's reading.

3.2. Selection of resistor R_N

A key point in the design of the standard source of extremely low current for calibrated picoammeters with active i/u converter is the proper choice of the value of the high value standard resistor R_N . The basic criterion is minimization of errors caused by inclusion of the picoammeter in the measured circuit - the method's error as well as the amplification effect of internal disturbances of the i/u converter. Good design means minimization of these errors to a neglect-able level while keeping the resistance of resistor R_N as low as possible, because it is hardly available, very expensive and for values greater than $10^8 - 10^9 \Omega$ their accuracy dramatically falls [11, 12].

On the most sensitive ranges, for example 1 pA, the required value of the scaling resistor R_p is no more than $10^{11} \Omega$ [5, 7, 9]. For a typical value of $k_{u0} = 10^5 - 10^6$, the internal resistance of the measurement device on this range is then $R_{pA} = 10^5 - 10^6 \Omega$. To lower the method's error to a level below $\delta_m = 0.1\%$ according to (11), it is sufficient to choose $R_N = 10^8 - 10^9 \Omega$, whereas to satisfy the criterion given by (15) a much higher value of R_N , reaching about $10^{10} - 10^{11} \Omega$, is required. It is clear because the criterion (15) is more critical than the criterion (11) of minimization of the method's error. Therefore, it is suggested to use equation (15) when choosing the value for R_N .

High value standard resistors of values in the range of $10^8 - 10^9 \Omega$ are made as wire-wound resistors of high accuracy, even 0.01 - 0.05%. Such standard resistors, manufactured mainly in the former USSR, exist as standard equipment of many domestic laboratories and can be successfully used to build the resistive current calibrator of DC currents up to 100 pA. For higher values up to $10^{11} - 10^{12} \Omega$ the resistance standards are made as composite resistors as well as oxide MOX resistors. Their accuracy is worse than the accuracy of wire-wound resistors and for the resistors of about $10^{11} \Omega$ reaches even 2% [11, 13]. The highest values up to $10^{14} - 10^{15} \Omega$ of

resistance standards are made as 3-terminal circuits (so called resistance substituters) [14, 15] but their usefulness for calibration of the most sensitive ranges of the picoammeter is problematical.

The choice of the value for resistor R_N in the designed calibrator of low DC currents is possible only if the value of the scaling resistor R_p is known for all the available ranges of the tested picoammeter. Unfortunately, manufacturers do not publish these data. It can be, however, estimated based on the general knowledge of the principle of operation of a picoammeter with active i/u converter using equation (2) rewritten in the form

$$R_p \cong \frac{U_{wy1}}{I_z}, \quad (16)$$

where: U_{wy1} - output voltage of the input block of the i/u converter,

I_z - maximum current of the calibrated range of picoammeter.

The voltage U_{wy1} for all the ranges is kept constant, usually about 0.1 - 0.2 V/range [5, 7, 9] for picoammeters with the lowest range of 1 - 10 pA. The resistance evaluated using (16) is close to the values of real resistors applied in a picoammeter. The value of resistor R_p can also be evaluated as

$$R_p = \frac{U_{wy2}}{|k_{u2}| I_z}, \quad (17)$$

what can be considered as to be a simplified method based on equation (4) in which we do not have to know the internal structure of the regarded instrument.

The voltage U_{wy2} available at terminals of the analog output in the picoammeter is always published by manufacturers and usually it is equal to 1 - 2 V at each range.

Usually, $|k_{u2}| \leq 10$, so the evaluated value of R_p is at most a range higher than the value found from the equation (16). Then the value of resistor R_p calculated from the equation (17) is at the most 10 times higher than that obtained from (16).

3.3. Selection of a standard voltage U_n

Current adjustment of the calibrator is driven by changes of a standard voltage U_n . The value of the standard voltage U_n obtained in the calibrator and shown in Fig. 2 is strictly related to the value of resistor R_N by Ohm's Law. For obtaining the standard current value I_{ki} needed for calibration of i th selected control point of the picoammeter's range, it is necessary to set the voltage U_n the value calculated as $U_{ni} = I_{ki} R_N$. The range of adjustment of the voltage U_n must be wide enough to cover the whole calibrated range ($I_{min} - I_{max}$) which is the requirement of the legislative acts [1, 2].

At the most sensitive range 1pA, the standard voltage U_n is not greater than (0.1 - 1.0) V for the formerly assumed value of $R_N = 10^{11} - 10^{12} \Omega$ evaluated from (15) and (17). A voltage of this level can be easily and accurately measured using a digital voltmeter that is part of the equipment of almost all scientific laboratories.

During calibration of the succeeding higher ranges of the picoammeter, above 1 - 10 pA, different intervals of required current are obtained by decreasing the value of R_N or by increasing

the interval of U_n with keeping the value of the resistor constant. For example, calibration of three succeeding ranges 1, 10 and 100 pA using the same resistor $R_N = 10^{11} \Omega$ requires the value of voltage U_n to be in intervals 0 - 0.1 V, 0 - 1 V and 0 - 10 V. Doing so, the number of required resistance standards can be considerably reduced.

4. ASSESSMENT OF UNCERTAINTY OF PICOAMMETER'S CALIBRATION

4.1. Calibration procedure

Following the definition, the goal of calibration is to map real values of current to the proper readings of a measurement device. Alternatively, it means finding corrections at particular points of a given range. To do this, due to the recommendations of the legislative acts [1, 2, 3, 16], it is required to determine the reading errors at least at five control points of the analog display meter including the lower and upper limit of a range. The uncertainty in determining the value of current regarded as the standard current should be basically no more than one tenth of the maximum error resulting from the accuracy of the calibrated instrument.

In some situations this ratio is allowed to be 1/4 or even 1/3 in the case of not very accurate measurements. On the other hand, a circuit including a calibrated instrument should be set up so that the effect of random factors determined by the uncertainty of type A (U_A) is essentially smaller than the uncertainty of systematic nature (rectangular distribution) of that instrument ($U_B \gg U_A$). In this case the readings' deviation is small enough (practically negligible) to determine the uncertainty as in the case of rectangular distribution, without the necessity of doing a series of measurements at each control point. Such calibration conditions can be ensured only in calibration of higher and middle ranges of the picoammeter. In the case of the most sensitive ranges of the picoammeter, especially a digital one, it appears as a significant reading deviation caused by internal disturbances of the picoammeter - drift of offset voltage, internal noise of an amplifier and the scaling resistor R_p as well as calibrator's setup i.e. random variations of the voltage U_n , thermal noise of the high value standard resistor R_N and also disturbances induced in the circuit by an external magnetic field. In such a situation it is difficult to determine the value of the displayed current and find the reading correction based on the result of a single measurement only. It is required then to do a series of measurements to find the mean value as the best estimate of the real value.

The reading error at all the control points of the calibrated range is determined by the following equation

$$\Delta I_x = I_x - I_N, \quad (18)$$

where: ΔI_x - absolute reading error,
 I_x - value indicated (read) by the instrument based on a single measurement (no reading deviation), or obtained as a mean value from the series of measurements (significant random errors) ,
 I_N - correct value of source current (standard value).

4.2. Uncertainty of determining the reading error (correction)

The uncertainty of determining the reading errors at all the particular control points of a range, is a resultant uncertainty of determining the standard value of calibrator's current and uncertainty of determining the reading. The combined standard uncertainty can be found [16, 4] as

$$u(\Delta I_x) = \sqrt{u^2(I_k) + u^2(I_x)}, \quad (19)$$

where: $u(\Delta I_x)$ - combined standard uncertainty of the reading error at the control point of the calibrated picoammeter's range,
 $u(I_k)$ - standard uncertainty of the calibrating standard current I_k ,
 $u(I_x)$ - standard uncertainty of picoammeter's readings.

Uncertainty of calibrator's current

The current value of the calibrator shown in Fig. 2 is determined in an indirect way, therefore its standard uncertainty can be calculated using the general expression

$$u(I_k) = \sqrt{\left(\frac{\partial I_k}{\partial U_n}\right)^2 u^2(U_n) + \left(\frac{\partial I_k}{\partial R_N}\right)^2 u^2(R_N)}, \quad (20)$$

where: $\frac{\partial I_k}{\partial U_n} = \frac{1}{R_N}$ - sensitivity coefficient describing the influence of the standard voltage U_n on the calibrator's current I_k ,
 $u(U_n)$ - uncertainty of a measurement of the U_n voltage,
 $\frac{\partial I_k}{\partial R_N} = -\frac{U_n}{R_N^2}$ - sensitivity coefficient describing the influence of standard resistor R_N on the calibrator's current I_k .
 $u(R_N)$ - uncertainty of resistance R_N .

The standard uncertainty of the calibrator's current I_k implied by uncertainty (by variations) of standard voltage is the resultant of few partial uncertainties

$$u(U_n) = \sqrt{u^2(kl_v) + u^2(U_{SZRN}) + u^2(U_{SZB}) + u^2(U_i)}, \quad (21)$$

where: $u(kl_v) = \frac{\Delta U_v}{\sqrt{3}}$ - standard uncertainty of the voltage U_n obtained from digital voltmeter readings,
 $\Delta U_v = aU_x + bU_z$ - maximum error of digital voltmeter's readings (multiplicative error aU_x - fraction of reading and additive error bU_z - fraction of range),

- $u(U_{SZRN}) = \sqrt{4kTR_N \Delta f}$ - standard uncertainty of the voltage U_n caused by thermal noise of resistor R_N and calculated for the picoammeter's pass-band Δf [17] (equals the root-mean-square of noise within this band),
- $u(U_{SZB}) = \frac{U_{SZBp-p}}{6}$ - standard uncertainty of voltage U_n caused by fluctuating changes of a battery source E,
- U_{SZBp-p} - peak-to-peak noise voltage value of the battery source E determined experimentally, for example by observing oscilloscope plots,
- $u(U_i) = \frac{U_{ip-p}}{\sqrt{3}}$ - standard uncertainty of voltage U_n caused by voltage disturbances U_{ip-p} induced in the measurement system by external electromagnetic fields,
- U_{ip-p} - peak-to-peak measured value of 50 Hz power network disturbing voltage.

The results of computations as well as experimental research show that if a measurement circuit is well shielded and is battery powered, it is still required to take into account not only the uncertainty of a digital voltmeter but also the influence of a noise caused by the resistor R_N and external disturbances with a similar level of values, $u(U_{SZRN}) \approx u(U_i) = 100 \mu\text{V}$, during calibration of the most sensitive ranges (1 - 100 pA). For higher ranges the influence of all the random factors can be omitted.

The standard uncertainty of the standard resistor R_N used in the construction of the current calibrator can be found with the use of the following expression

$$u(R_N) = \frac{1}{3} \sqrt{(kl_{RN} R_N)^2 + (TWR \Delta T R_N)^2 + (NWR \Delta U_p R_N)^2}, \quad (22)$$

where: kl_{RN} - accuracy of standard resistor R_N determined in reference conditions (usually $T = 23 \text{ }^\circ\text{C}$ and $U_p = 100 \text{ V}$) in which the last calibration has been carried out,

TWR, NWR - temperature and voltage resistance coefficient of the resistor R_N .

High value standard resistors with highest values, for example $R_N = 10^{11} \Omega$, required for calibration the most sensitive ranges of picoammeter, feature relatively low accuracy 0.5 - 2% as well as high temperature ($TWR = 500 \text{ ppm}/^\circ\text{C}$) and voltage ($NWR = 5 \text{ ppm}/\text{V}$) resistance coefficients, respectively. When small variations of nominal voltage and ambient temperature are considered, however, the uncertainty will be proportionally small regarding the systematic error determined by the accuracy of the resistor R_N .

After taking into account all simplifications, the eventual expression determining the standard uncertainty of the calibrator's current can be given, using the proposed notation, in the following form

$$u(I_k) = \sqrt{\frac{1}{R_N^2} \left[\frac{1}{3} (aU_x + bU_z)^2 + U_{ip-p}^2 + 4kTR_N \Delta f \right] + kI_{RN}^2 \frac{U_n^2}{R_N^2}}. \quad (23)$$

It appears from the conducted considerations, that the relative standard uncertainty of 1 pA standard current of the calibrator presented in Fig. 2, built by the use of easily and widely available equipment - a digital voltmeter of 0.01 - 0.05% accuracy and a standard resistor $10^{11} \Omega$ of 0.5% accuracy - is about 0.30%. Such uncertainty is satisfactory for carrying out the calibration even of the most sensitive picoammeters, for example type 617 from Keithley, featuring the maximum error at the end of the scale, about 2 - 3%.

Uncertainty of picoammeter's readings

The way in which the uncertainty of readings of the calibrated picoammeter is determined, depends on the repeatability of succeeding measurements. A readings' deviation practically does not occur for medium and higher ranges, because the impact of all random factors in a picoammeter as well as in a standard current calibrator is negligibly low. In this case, the current resolution - the value of the lowest difference between readable values from the analog picoammeter's scale or the value of the unit of the last digit of the digital voltmeter display - should be taken as uncertainty of readings. On the other hand, for the most sensitive ranges a significant reading deviation appears as well as the uncertainty of calibrator's current increases. The increase of a calibrator's uncertainty is caused by a noise of the resistor R_N and by external disturbances of random nature. In that case, reading uncertainty of the calibrated picoammeter is determined using statistical methods based on a series of measurements. The systematic reading error is determined as the difference between the mean value of the obtained set of results (readings) of succeeding measurements and the value of calibrator's standard current. Standard deviation of the series of readings, however, can be regarded as the standard uncertainty of determining picoammeter readings. By applying the general laws of mathematical statistics and removing all the results from outside the $\pm 3\bar{S}_{\bar{I}_x}$ interval one can calculate the mean squared deviation of the mean value of a series as

$$\bar{S}_{\bar{I}_x} = \sqrt{\frac{1}{n(n-1)} \sum_{i=1}^n (\bar{I}_{xi} - \bar{I}_x)^2} \quad (24)$$

and on this basis determine the standard uncertainty $u(I_x)$ of picoammeter's readings ($u(I_x) = \bar{S}_{\bar{I}_x}$).

4.3. Expanded uncertainty of calibration of a picoammeter

The combined standard uncertainty u_c given by the equation (19) describes a quality of the calibration process of a picoammeter, whereas the expanded uncertainty enables to compare errors determined at particular control points of a given range to the maximum error given by the manufacturer. The expanded uncertainty is defined by

$$U(\Delta I_x) = k_p u_c, \quad (25)$$

where: $U(\Delta I_x)$ - expanded uncertainty of reading errors determined at control points of calibrated picoammeter's range,

k_p - coverage factor,

u_c - combined standard uncertainty of control measurements.

Taking the proper coverage factor is the most difficult problem when evaluating the expanded uncertainty. This factor depends on the coverage probability assumed and accurate determination of convolution of distributions of input quantities having a real impact on the results of the calibrating measurement.

When calibrating higher and middle ranges of picoammeter, where practically there is no reading deviation, the uncertainty of the standard resistor R_N determined by its accuracy predominantly influences the accuracy of the measurement. This uncertainty has a rectangular distribution. Far less impact comes from the uncertainty of the standard voltage U_n determined by digital voltmeter readings.

The maximum error of a digital voltmeter is the sum of two errors - a small additive error and a significantly higher multiplicative one which can be characterized by rectangular distribution. The other errors of calibration - reading error and the error coming from an internal noise of the picoammeter - are negligibly low and can be omitted. Hence, the resultant distribution of calibration uncertainty can be considered to be convergent to a dominant distribution, i.e. rectangular and then, assuming coverage probability $p = 0.95$, the coverage factor should be as follows

$$k_p = \sqrt{3}p = 1.65. \quad (26)$$

However, if the maximum error of the digital voltmeter used will be comparable to the accuracy of the standard resistor R_N , the distribution of uncertainty will be more complex. It is convenient then to assume, that the distribution is normal rather than rectangular and for $p = 0.95$ the coverage factor $k_p = 2$.

Calibrating the most sensitive ranges of picoammeter, very often limited by measurability, where the large deviation of readings is natural, it is required to perform a series of measurements (readings).

The uncertainty caused by factors coming from the picoammeter and the calibration circuit is comparable to uncertainties resulting from errors of systematic nature. It is then difficult to assume in advance which type of uncertainty will be predominant. In such a case, it is justified to take coverage factor $k_p = 2$ for the coverage probability $p = 0.95$ recommended by the international standard [4].

Another issue is obtaining a sufficient number of samples (results of measurement). Doing it manually is a strenuous work and therefore the number of samples is usually not too high, rarely above $n = 10$. The most suitable approximation of such a small set of samples is the t -Student distribution with effective number of degrees of freedom determined by the Welch-Satterwhite rule [3, 16, 18, 19]. For the number of degrees of freedom calculated in this way, the coverage factor k_p takes the standardized value of t -Student coefficient, i.e. $k_p = t$, which can be found in mathematical tables.

5. EVALUATION OF PICOAMMETER'S ACCURACY

The assessment of accuracy of the calibrated picoammeter is possible just after the evaluation of reading errors at particular control points of all ranges. The reading errors are calculated with the expanded uncertainty determining an interval covering the expected value of the measured quantity. The limits of the reading error should be expanded about the value of uncertainty of control measurement. For this reason, it is required that the calibration uncertainty be as small as possible [1, 2] - at least three times less than the basic error (maximum in a reference environment) of the calibrated instrument.

The picoammeter's accuracy, just like that of many other instruments, is defined by the maximum error which is given in a form dependent on the type of the instrument - analog or digital - used to read the output voltage of the i/u converter.

5.1. Accuracy of an analog picoammeter

The accuracy of a picoammeter with analog display is described using one parameter only. This parameter, expressed in percents, is equal to the maximum error related to the nominal (maximum) value of a given range while the absolute value of the error resulting from the accuracy of an analog instrument is constant throughout the range, which means that its distribution is rectangular.

Hence, the largest value of a set of errors measured at the control points extended by the uncertainty interval with which these errors have been determined should be compared to the admissible error determined by the following expression, while assessing the accuracy of an analog picoammeter

$$\Delta_g I_z = \frac{kl I_z}{100}, \quad (27)$$

where $\Delta_g I_z$ - maximum systematic error of picoammeter's range,
 kl - accuracy of calibrated range (expressed by percents),
 I_z - nominal value of the calibrated picoammeter's range.

The value of error determined during the calibration should not be greater than the value of admissible error given by the manufacturer's specification at any control point within the range.

5.2. Accuracy of a digital picoammeter

The accuracy of a digital picoammeter is also determined by the maximum admissible error but is very often presented in the form of two components: a multiplicative error - a fraction of the reading - and an additive error - a fraction of the range, as follows

$$\Delta_g I_x = aI_x + bI_z, \quad (28)$$

where $\Delta_g I_x$ - absolute reading error at the control point of the calibrated range,
 a - admissible relative multiplicative error,
 b - admissible relative additive error,
 I_x - current value at the control point of the calibrated range,

I_z - range nominal value associated with the maximum value that can be read from the instrument's display on a given range.

The maximum error value varies through the range, so the measured value of that error should be compared to the admissible error calculated using the equation (28). The data needed for calculation of the maximum error of the calibrating instrument are given by the manufacturer.

It is a pretty good idea to present the results of calibration graphically, as shown in Fig. 3. Also, the intervals of the expanded uncertainty of measurement errors at all control points should be determined in the graph. It can be observed in Fig. 3 that the absolute error has a maximum value at the end of the range. It is worth noting that then the relative admissible error at the end of the considered range is the least.

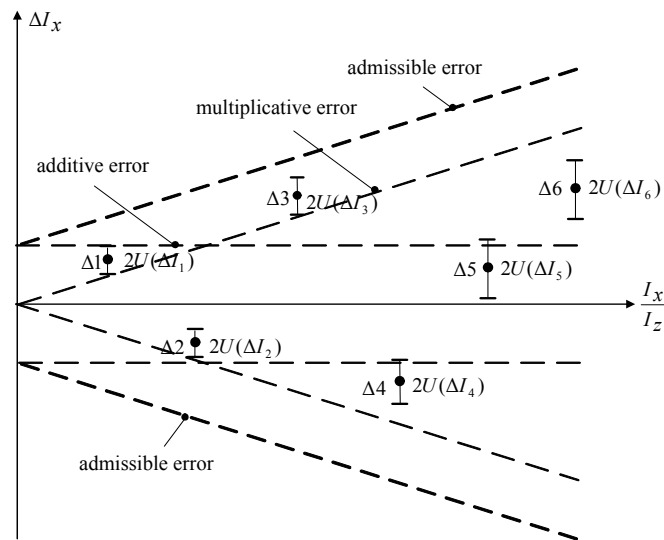


Fig. 3. Graphical exposition of digital picoammeter's calibration results on the lowest range: ΔI_i - mean value of measurement error at i -th control point of the range, $U(\Delta I_i)$ - expanded uncertainty of difference between the mean value of a series of measurements and the nominal value of the current calibrator.

This way of assessment of the picoammeter's accuracy is relatively simple but only for calibration of middle and higher ranges, where practically the reading deviation is not greater than the discretization error i.e. one unit of the last digit on the display panel. On the other hand, for the most sensitive ranges of the best picoammeters like, for example, 2 pA of type 617 from Keithley, the deviation caused by internal factors of only the instrument having 4.5 digits (20 000 units) reaches 66 units what makes 0.33% of the whole range.

Moreover, an additional deviation caused by disturbances induced in the measurement circuit (current calibrator) should be taken into account. Eventually, the deviation is quite significant causing an increase of uncertainty of reading's mean value, what leads to an extension of uncertainty of determining the reading error (correction).

Therefore, modern digital picoammeters, usually microprocessor-based [5, 7], are equipped with procedures of averaging the results, what makes it possible to gather a large number of samples and decrease the interval of uncertainty based on which the mean value of the acquired data is calculated (smaller standard deviation of mean value). Moreover, gathered data can be transmitted to a dedicated external computer and processed statistically.

This problem is not so important in the case of analog-reading picoammeters with similar sensitivity (measurement ranges) because an electromechanical analog display meter used to read the output voltage of an i/u converter features a big mechanical inertia which works like a low-pass filter or an averaging block attenuating fast changes of that voltage caused by noise and disturbances. As a result, the readings are proportional to a slightly averaged value of the real output voltage, which makes the reading deviation lower than the reading deviation of a digital picoammeter.

6. SUMMARY

The presented method allows calibration of active i/u conversion based picoammeters featuring very sensitive ranges and designed for measurement of extremely low DC currents - almost measurability limited. This method is relatively simple and can be carried out in almost all research or measurement laboratories, even those not very well equipped. Building a calibrator of very low (even less than 1 pA) DC currents in the user's own capacity depends on the availability of high value standard resistors within a not very demanding range of 10^{10} - 10^{11} Ω . Such standard resistors are readily available and relatively cheap and their accuracy is relatively high. Within this range of values, up to 10^{11} Ω , much cheaper precision resistors of type MOX (metal oxide resistors) produced with sufficient good accuracy of 0.5% and 1% [12, 13] can be used as standards as well.

The drawback of the presented method is that the high-value resistor used to build the calibrator of low DC current works under low voltage ranging from 0 to 0.1 V or 0 to 1 V. Under such conditions, high value resistors feature relatively higher non-linearity than in the range of medium or higher voltages. Unfortunately, the manufacturers do not define this non-linearity but nevertheless precise high value resistors can be used as standards because the voltage changes are small. Detailed information about this phenomenon can be found in [12, 20].

This paper contains all the information and required procedures allowing the proper selection of calibrator's components as well as detailed analysis of current's uncertainty resulting from errors of instruments used (standard resistor, digital voltmeter) as well as factors of random nature - thermal noise of the high-value resistor, fluctuation of the standard voltage source and also external disturbances. The method of statistical data processing has been given as well the method of assessment of uncertainty done especially on the most sensitive ranges of the picoammeter, where a significant reading deviation can be observed. Also, the method of evaluation of the final accuracy of the calibrated instrument (electrometric picoammeter) has been presented.

REFERENCES

1. Order of the President of the Head Office of Measures of October 14th, 1996, No.156 on the instruction of checking analog voltage, current and power meters – and analog universal meters and multimeters. Regulations Gazette of Measures and Assays, 26: 161, (in Polish).
2. Order of the President of the Head Office of Measures of October 14th, 1996, No. 156 on the introduction of the instruction of checking voltage, current and resistance calibrators, digital voltage and current meters, digital multimeters and RCL bridges. Regulations Gazette of Measures and Assays, 26: 163, (in Polish).
3. *Guide to the Expression of Uncertainty in Measurement*. ISO 1993, 1995. Główny Urząd Miar 1999, (in Polish).
4. *Expression of Uncertainty in Measurement in Calibration*. Polish edition EA-4/02. Główny Urząd Miar, 2001, (in Polish).
5. Keithley Instruments Inc. *Test & Measurement: Catalog and Reference Guide*, 1999/2000.
6. Hewlett-Packard. *Test & Measurement Catalog*, 1993.
7. Z. Kłos: *Microprocessor - based picoammeter, type PA-101*. Pomiary, Automatyka, Kontrola, no. 11, pp. 268-270, 1993, (in Polish).
8. Z. Kłos: *Analog circuit for the measurement of very small direct currents*. Pomiary, Automatyka, Kontrola, no. 5, pp. 101-103, 1992, (in Polish).
9. Burr-Brown. *Integrated Circuits Data Book - Linear Products*, 1997.
10. J. Jaworski: *Inaccuracy, error and uncertainty of measurement*. Pomiary, Automatyka, Robotyka, no. 7-8, pp. 12-16, 35-38, 1999, (in Polish).
11. Victoreen. *Mini - MOX Resistors*, 1984.
12. Z. Kłos: *Properties of high-resistance oxide resistors, type MOX*. Normalizacja, no. 12, pp. 12-15, 1992, (in Polish).
13. Z. Kłos: *Control system of high-resistance standards, type ZKRW-2*. Pomiary, Automatyka, Kontrola, no. 10, pp. 6-8, 2000, (in Polish).
14. Guildline Instruments Ltd. *High value resistance standards 9336 series and ultra high value resistance standards 9336 series*, 2003.
15. Z. Kłos, P. Madej: *Imitated large-resistance standard, type IZWR-2*. Pomiary, Automatyka, Kontrola, no. 9, pp. 15-17, 2001, (in Polish).
16. J. Arendarski: *Uncertainty of measurements*. Oficyna Wydawnicza Politechniki Warszawskiej, Warszawa, 2003, (in Polish).
17. L. Hasse, L. Spiralski: *Noise in electronic elements and circuits*. Wydawnictwa Naukowo-Techniczne, Warszawa, 1981, (in Polish).
18. B. Kalus - Jęcek, Z. Kuśmirek: *Standards of electrical quantities and estimation of measurement uncertainty* Wydawnictwo Politechniki Łódzkiej, Łódź, 2000, (in Polish).
19. J. Jaworski: *Error and uncertainty of measuring instruments*. Pomiary, Automatyka, Robotyka, no. 11, pp. 5-9, 1999, (in Polish).
20. Z. Kłos: *Problems with calibration of electrometric apparatus*. Oficyna Wydawnicza Politechniki Wrocławskiej, Wrocław, 2004, (in Polish).

WZORCOWANIE EKSTREMALNIE CZUŁYCH ZAKRESÓW MIERNIKÓW MAŁYCH PRĄDÓW DC Z ANALOGOWYM AKTYWNYM PRZETWARZANIEM I/U

Streszczenie

W artykule przedstawiono metodę wzorcowania ekstremalnie czułych pikoamperomierzy o najniższych zakresach pomiarowych bliskich aktualnej granicy mierzalności, wykorzystującą rezystancyjne źródło wzorcowe małych prądów DC - kalibrator prądu. Podano procedurę projektowania kalibratora, kryteria optymalnego doboru wysokoomowych wzorców rezystancji oraz szczegółową analizę niepewności wytworzonego prądu wzorcowego użytego do wzorcowania najniższych zakresów pikoamperomierza. Przedstawiono metodę szacowania niepewności pomiarów kontrolnych oraz procedurę oceny końcowej niepewności sprawdzanych pikoamperomierzy z odczytem analogowym i cyfrowym. Wykazano, że w warunkach przeciętnie wyposażonego laboratorium pomiarowego lub badawczego i przy użyciu stosunkowo łatwo dostępnych wzorców rezystancji o wartościach nie przekraczających $10^{10} - 10^{11} \Omega$ możliwe jest wzorcowanie najczulszych nawet pikoamperomierzy.