EURAMET.EM-S38

(EURAMET Project 1260)

Supplementary Comparison of Small Current Sources

FINAL REPORT

Karl-Erik Rydler
SP Technical Research Institute of Sweden
Borås, Sweden

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1. Introduction

In the last years, there has been an increased need for calibrations of picoammeters, used e.g. in the field of dosimetry. Therefore, several European and non-European National Measurement Institutes (NMIs) have developed precision DC current sources to be used for traceable calibrations of picoammeters in the current range between about 100 fA and 100 pA. In the supplementary comparison EURAMET EM-S24, thirteen participants compared their calibration systems [1].

In this bilateral supplementary comparison EURAME.EM-S38 the calibration system of SP is compared to the one of PTB, the coordinator of the EURAMET.EM-S24.

2. Participants and organization of the comparison

2.1 Coordinator

The pilot and linking laboratory for the comparison was the Physikalisch-Technische Bundesanstalt (PTB), Germany.

Coordinator:

Dr. Gerd-Dietmar Willenberg
Tel.: +49 531 592 2141
E-mail: gerd-dietmar.willenberg@ptb.de

2.2 List of participants

There were two NMIs participating in this comparison; they are listed in Table 2.1.

Table 2.1: List of participants in alphabetical order

Acronym	Institute	Country
PTB (Pilot)	Physikalisch-Technische Bundesanstalt	Germany
SP	SP Technical Research Institute of Sweden	Sweden

2.3 Organization and comparison schedule

The comparison was organized in one loop. For each laboratory, a period of one month was scheduled including transportation time.

Table 2.2: Circulation time schedule

Institute	Country	Mean date of measurements	Label used in diagrams or used as an index
PTB (Pilot)	Germany	13.1.2010	PTB-1
SP	Sweden	14.3.2010	SP
PTB (Pilot)	Germany	13.4.2010	PTB-2

3. Travelling standards and measurement instructions

3.1 Description of the travelling standards

Two picoamperemeters were used as travelling standards:

- A modified commercial PTW Unidos E S/N T10008 Y-80130. It had been modified by the manufacturer in such a way that it provides a one digit higher resolution than the standard instrument.

- A commercial Keithley 6430 Source Meter S/N 1036593.

Both instruments were shipped in a single package together with their operating manuals. Since they had very special input connectors, they were accompanied by appropriate adapters with male BNC connectors.

As both instruments were commercial picoamperemeters which were not specifically designed as standards, in the following text they are preferably denoted as transfer instruments or travelling instruments instead of transfer standards or travelling standards.

3.2 Quantities to be measured and conditions of measurements

The measurements were carried out by calibrating the transfer instruments, i.e. by supplying a DC current specified by the participant's current source and recording the instruments' readings. The measurand was then the calibration factor "*Q*" of the transfer instrument, defined as the ratio of the reading of the transfer instrument over the supplied current: $Q = I_{readout} / I_{supplied}$.

The nominal values of the eight measuring points were: +100 fA, -100 fA, +1 pA, -1 pA, +10 pA, -10 pA, +100 pA, and -100 pA. Throughout this document, the current is defined as positive when it is flowing <u>into</u> the picoammeter and it is defined as negative when it is flowing <u>out of</u> the picoammeter. It is important to mention this definition because the measurement data of the Keithley 6430 show the reverse sign. Furthermore, throughout this paper the calibration factors "*Q*" are regarded as being positive, despite the fact that the numerical ratio of the value displayed by the Keithley 6430 over the supplied current always has a negative value.

The measurements were to be carried out at an ambient temperature of (23 ± 0.5) °C.

3.3 Measurement instructions

In order to take full advantage of the transfer instruments' resolution and to avoid internal range switching or overflows, the calibration points had to be slightly below the nominal values. Therefore, the calibration points were to be about 95 % of the nominal values, i.e. 95 fA, 0.95 pA, and so on. Only if for some technical reasons this was proved to be impossible, could the exact nominal values be used.

Both instruments, the Keithley 6430 as well as the modified PTW Unidos E, had to be operated remotely. Both instruments had RS-232 connectors, in addition, the Keithley had a GP-IB connector.

After transportation a minimum settling and warm-up time of one day had to be allowed for the instruments. Ambient temperature, pressure, and humidity had to be recorded and reported.

The transfer instruments had considerable time constants. To take these into account, a settling time of 15 s after each current change had to be allowed for.

3.4 Deviations from the protocol

There were no changes in the circulation time schedule.

3.5 Unexpected incidents and effects

3.5.1 PTW Unidos E (effects found during EURAMET EM-S24)

3.5.1.1 PTW Unidos E: locking effect

During the measurements at INRIM in EURAMET EM-S24, an irregular feature of the Unidos E was discovered: When the current was slowly varied, the instrument's readout stayed on certain preferred values. After reporting these findings to the pilot laboratory, the effect was verified at the pilot laboratory by performing special measurements where the input current was varied continuously. The results for input currents close to 95 pA are shown in Fig. 3.1. It can be seen that the current "I(readout)" indicated by the Unidos is not linear with respect to the supplied current "I(source)". Instead, the graph shows steps with a height of typically 6 in the last digit. This effect was found to be most pronounced at the high currents of nominally 100 pA, while at 10 pA it was smeared out to a larger extent. At currents below 10 pA it was smeared out completely.

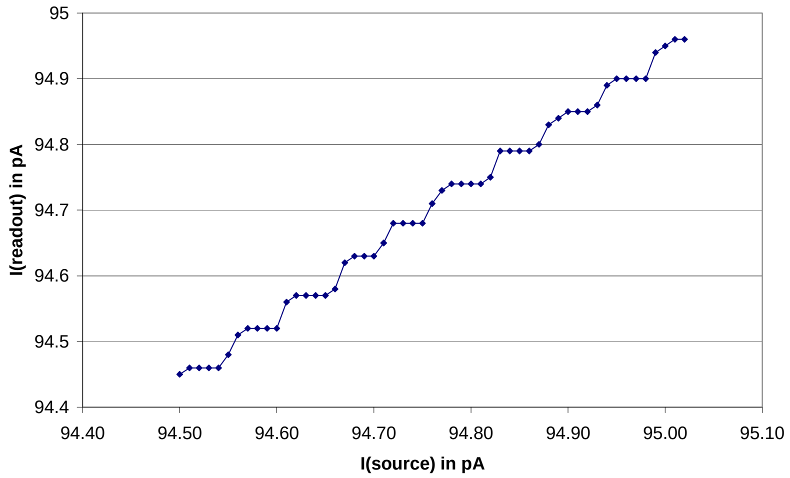

Fig. 3.1: Unidos E: indicated current versus applied current showing "locking" at certain steps

This behavior had not been recognized during the preparation of the comparison, probably because the calibration current was always sufficiently constant.

After contacting the instrument's manufacturer, the pilot laboratory received the explanation that the observed behavior is due to the instrument's specific electronics: the resolution of the analog-to-digital converter (ADC) is limited to about 0.05 % of the range's maximum value (112 pA in the "Low" range), the output of the ADC is then averaged over several measurements by software and the result is rounded to 4 digits. Therefore, the effect depends strongly on the presence of noise: at high currents there is a low relative noise level, hence, the reduced resolution is fully visible while at low currents the quantization is smeared out by a high relative noise level.

This behavior was communicated immediately to the participants in order to enable them to take this effect into account in their error budgets.

3.5.1.2 PTW Unidos E: zero-offset problem

During the compilation of the results, it was found that at nominal current values of 1 pA and above, the calibration factors Q_+ for the positive current direction and Q_- for the negative current direction vary strongly (see Figures 5.1 … 5.4 in the final report of EURAMET EM-S24), but a closer look revealed that they vary in a characteristic way: positive excursions of Q_+ correspond to negative excursions of Q_- of about the same amount and vice versa. This happens at least in situations where both values are determined from a simultaneously generated data set for positive and negative currents, which is generally the case when the calibration cycles are arranged as follows: zero current, positive current, zero current, negative current, zero current and so on. In this case, the variation of the mean calibration factor $Q_\pm$ defined as $Q_\pm = (Q_+ + Q_-)/2$ is much smaller than the variation of Q_+ or Q_- alone.

As has now been confirmed by the manufacturer, this behavior is explained as follows: the picoammeter uses - in addition to the three current ranges “High”, “Med” and “Low” being accessible from the front panel - four internal gain stages which are selected automatically and which cannot be controlled from the outside. This means that when the applied current is zero, the instrument internally switches to the highest gain stage which is in most cases not identical to the gain stage used for the previously or subsequently applied non-zero current. Therefore, the instrument’s zero-offset value is usually determined in the “wrong” gain stage. As a consequence, Q_+ might be too high by a certain amount and Q_- too low by the same amount, or vice versa.

The effect described above was found after receiving the results at the pilot institute and was not communicated to the participants before the end of the measurements. This effect might be responsible for some participants’ observation that the Unidos is far more unstable than the Keithley.

3.5.2 Unexpectedly large ac-dc difference of capacitances

The calibration method described below in Section 4.1 relies on charging a capacitor. After finishing all measuring loops in in EURAMET EM-S24 and -S38, it was found by NPL, PTB and Nick Fletcher from BIPM (which was not a participant of this comparison), that the dc capacitance effective for the calibration and the ac capacitance measured with an ac capacitance bridge differed more than might be expected before the beginning of the comparison [2]. This was communicated to the participants in spring 2010 and all participants had the opportunity to adjust their uncertainties in the light of these findings.

4. Calibration methods used by the participants

In this comparison, two calibrating methods were used by the participants:

4.1 Generating the calibrating current by charging/discharging a capacitor

The calibrating current *I* is generated by charging or discharging a gas-filled capacitor *C* with a linearly increasing or decreasing voltage of slope dV/dt. The calibrating current is then $I=C\cdot dV/dt$. Thus, it is traced back to the volt, the second and the farad. Typically, a trapezoidal voltage pattern symmetrical to zero voltage is used which allows the eliminating of linear drifts and the influence of leakage currents across the capacitor. This is discussed in more detail in [3].

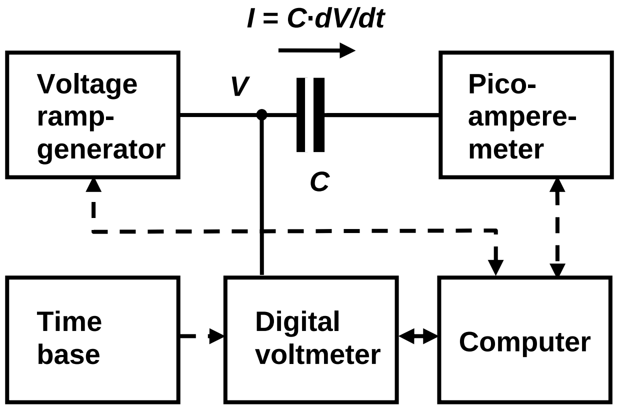

Fig. 4.1: Schematic calibration set-up for using the capacitor-charging method

This method was used by PTB and SP.

4.2 Generating the calibrating current by a voltage source and a resistor

The calibrating current I is generated by a voltage source V (e.g. a DC calibrator) and a resistor R:
It is then $I = V/R$. Thus, the current is traced back to the volt and the ohm.

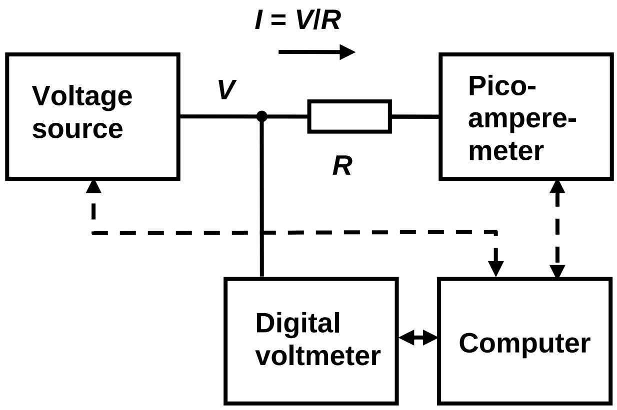

Fig. 4.2: Schematic calibration set-up for using the voltage-resistor method

This method was used by SP to evaluate the uncertainty contribution due to ac-dc difference of capacitors used.

5. Measurements and results of the participants

5.1 Measurements of the participants

In this section, the measuring set-up is briefly described for each laboratory in chronological order. Descriptions of the traceability chains are not given here, since traceability is not a matter of significance if viewed in the light of the uncertainties achieved.

5.1.1 Measurements of PTB (pilot institute)

The measurements were performed by using the capacitor charging method described in Section 4.1.

The voltage ramp was generated by a non-commercial ramp generator based on an electronic integrator, its nonlinearity being compensated by an analog feedback network as described in [4]. The

generator delivered a fixed voltage slope of 10 mV/s. The slope could be fine-tuned by using a Kelvin-Varley voltage divider and increased by a factor of ten using an additional amplifier stage.
During the time when this comparison was running, a new ramp generator based on digital-to-analog converters was developed [5], but, for reasons of continuity, it was not used for measurements in the framework of this comparison.
The ramp slope was measured using an Agilent 3458A multimeter (DMM). The DMM's triggering was performed by a precision time base consisting of an oven-stabilized 10 MHz quartz-oscillator and binary divider circuits.
For generating 100 pA and 10 pA, a commercial capacitor of the type GR1404 (1000 pF) was used, for generating 1 pA and 100 fA, two commercial capacitors of the types GR 1403 (100 pF and 10 pF) were used. Their capacitances were measured before and after each run using an Andeen-Hagerling AH2500 capacitance bridge. The whole set-up was situated in a temperature-controlled electrically shielded room. Humidity and air pressure were not stabilized.
For each current value, the measurement consisted of typically 60 cycles, each of them containing four phases: a) zero current, b) positive current, c) zero current, d) negative current. After the last cycle was completed, an additional zero-current phase was appended.

During the comparison, 11 sets of calibrations were carried out by PTB, denoted by "PTB-1" to "PTB-11".

5.1.2 Measurements of SP

The measurements were performed by using the capacitor charging method described in Section 4.1. The measuring system of SP is described in [6].

The setup consists of an arbitrary wave generator, AWG (National Instruments NI-PXI 5441), a digital multimeter, DMM (Agilent 3458A), hermetically sealed air gap capacitors (General Electric 1404, 10 pF – 1 nF), low pass filters (home built), the device under test, DUT, and a computer to communicate with and control the instruments. Coaxial cables (50 Ω) are used for all signal connections. The setup is operated in a climate controlled room with temperature 23±1°C and relative humidity 45±10%.
The measurement procedure consists of three steps. In the first step the output of the AWG is connected directly to the DMM and a number of voltage levels spanning the desired output range are measured. We used 63 levels in all these measurements spanning either -1V to +1V or -10V to +10V, depending on the current to be generated.
In the second step the measured levels are used to calculate a Δ-Σ modulated sequence which will generate the desired voltage ramp. The calculated sequence is transferred to the AWG.
In the third step the output of the AWG is connected through the low pass filters to the capacitor. The output of the capacitor is connected to the DUT.

We did the measurements three times in order to take into account medium term (weeks) stability of the DUTs. During the first measurements we had some problems with the climate control of the lab, which affected the measurements, and we decided to change the measurement procedure slightly for the second and third measurement. During the first measurements we generated the positive and negative ramps alternately without any waiting time between, and then measured the zero level afterwards. During the second and third measurements we added waiting time between each positive and negative ramp to measure the zero levels. This change resulted in modification of the uncertainty terms.

5.2 Behavior of the travelling standards

The behavior of the travelling instruments is based on the experience from the EURAMET EM-S24 and the two measurements of the pilot laboratory in this comparison. It must be mentioned here again that the travelling instruments used are commercial measuring instruments which were not designed for the use as standards. In these comparisons, they were used far beyond their specifications.

At the beginning of the comparison EURAMET EM-S24, it was intended to evaluate the temperature coefficients of the travelling instruments from the data obtained during the comparison, but this proved not to be feasible since any correlation with temperature was hidden by the scatter of the results.

Graphs showing the behavior of the travelling instruments can be found in Section 5.2.3 in the report on EURAMET EM-S24.

5.2.1 PTW Unidos E

As already discussed in Section 3.5.1, the Unidos E suffered from a low internal resolution and a problem with its zero-offset. This led to relatively large uncertainties and to differences in the results for positive and negative current directions. Due to the latter effect, the calibration factors Q_+ for positive current direction and Q_- for negative current direction show strong variations, whereas the variations are strongly reduced when the mean $Q_\pm = (Q_+ + Q_-)/2$ is calculated, at least when Q_+ and Q_- are measured in the same run.
Besides these properties, no further systematic effects were found.

During EURAMET EM-S24 MIKES reported a pressure dependence of the Unidos E. This dependence could not be supported by an analysis of the correlation of the participants' results with their associated ambient pressure data. If there were a noticeable pressure dependence, specifically the results of METAS and CEM, which are situated much higher above sea level than the other institutes, should differ significantly from the results of the remaining participants, but no such effect could be observed.

5.2.2 Keithley 6430

In EURAMET EM-S24 the results of the first PTB measurement "PTB-1" were considerably below the results of all succeeding measurements, which were attributed to some kind of aging of the instrument as already discussed in Section 3.5.2. Therefore, the whole first measuring loop containing the measurements of two participants was repeated at the end of the comparison.

Besides that irregular behavior at the first measurements, at current values of 1 pA, 10 pA and 100 pA a linear drift was observed superimposed by an additional wiggle, see eg Fig 5.8 in the report on EURAMET EM-S24.

6. Analysis of comparison data sets

The data sets reported by the participants (see Appendix A) mainly contains the calibration factors Q_+ (defined as the ratio of the transfer instrument's reading / supplied current) for a current flowing into the transfer instrument and Q_- for a current flowing out of the transfer instrument, accompanied by their standard uncertainties $u(Q_+)$ and $u(Q_-)$ and their effective degrees of freedom.

From these uncertainties and effective degrees of freedom, the pilot laboratory calculated coverage factors k corresponding to a 95% coverage and the corresponding expanded uncertainties $U(Q_+)$ and $U(Q_-)$. These data are also given in the tables of Appendix A.

As already discussed in Section 3.5.1.2, there was a zero-offset problem with the Unidos E leading to relatively large excursions of Q_+ and of Q_- of about the same amount, but in opposite directions. Due to this effect, the mean of Q_+ and Q_- is much more stable than the values themselves. Hence, it was found valuable to consider in addition to Q_+ and Q_- also their mean value $Q_\pm = (Q_+ + Q_-)/2$.

Since typically the sources for type B uncertainties are the same for Q_+ and Q_-, a high degree of correlation can be assumed and the uncertainty of $Q_\pm$ is approximated by $U(Q_\pm) = (U(Q_+) + U(Q_-))\,/\,2$. In reality, the correlation is not perfect and, therefore, this formula may overestimate the correct uncertainties, especially at the lower current values where the type A uncertainty components may contribute to a larger extent to the overall uncertainty than at the higher current values.

6.1 Method of analysis

The aim of the analysis is to establish, with PTB as linking laboratory, for each result of SP, Q_{SP}, a corresponding degree of equivalence $(d_{SP}, U(d_{SP}))$ to the reference value established in EURAMET.EM-S24. $U(d_{SP})$ being the expanded uncertainty of d_{SP} for a coverage of 95%.

6.1.1 Method of determining the degrees of equivalence

In this bilateral comparison PTB is acting as the linking laboratory to the reference value established in EURAMET.EM-S24. Due to the drift of one of the travelling instruments the value of the instrument as measured by PTB at the time of the measurement of SP, $Q_{PTB}(t_{SP})$, is estimated by interpolation between the two measurements of PTB, $Q_{PTB}(t_1)$ and $Q_{PTB}(t_2)$, assuming linear drift:

$$Q_{PTB}(t_{SP}) = Q_{PTB}(t_1) + (t_{SP} - t_1)\frac{Q_{PTB}(t_1) - Q_{PTB}(t_2)}{t_1 - t_2} \tag{1}$$

The standard uncertainty of the interpolated value is estimated as:

$$u(Q_{PTB}(t_{SP})) = \max\left[u(Q_{PTB}(t_1)); u(Q_{PTB}(t_2))\right] \tag{2}$$

No additional uncertainty is added for the interpolation. But a contribution for the instability of the travelling instruments is evaluated in EURAMET.EM-S24 and added below.

From of the results of SP, Q_{SP}, the degree of equivalence of SP, d_{SP}, to the reference value established in EURAMET.EM-S24 is determined as:

$$d_{SP} = Q_{SP} - Q_{PTB}(t_{SP}) + d_{PTB} \tag{3}$$

where the degree of equivalence of PTB, d_{PTB}, is determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24. The standard uncertainty of d_{PTB} is determined by pooling all the standard uncertainties. This standard uncertainty is not used in the analysis but the expanded uncertainty $U(d_{PTB})$ is reported for the different currents and travelling instruments in Section 6.2.

For the uncertainty analysis (3) is rewritten as:

$$d_{SP} = Q_{SP} - (Q_{PTB}(t_{SP}) + \Delta_{ts}) + Q_{PTB}(t_{S24}) - Q_{ref} \quad (4)$$

where Δ_{ts} is the correction of the interpolated value $Q_{PTB}(t_{SP})$ due to the instability of the travelling instrument , which is estimated to zero with a standard uncertainty u_{ts} evaluated in EURAMET.EM-S24. It is assumed that this uncertainty contribution is still representative for the travelling instruments. The standard uncertainty $u(d_{SP})$ can then be calculated as:

$$u^2(d_{SP}) = u^2(Q_{SP}) + u^2(Q_{PTB}(t_{S24}) - Q_{PTB}(t_{SP})) + u^2(Q_{ref}) + u_{ts}^2 \quad (5)$$

This expression is approximated to:

$$u^2(d_{SP}) = u^2(Q_{SP}) + u^2(Q_{PTB}(t_{SP})) + u^2(Q_{ref}) + u_{ts}^2 \quad (6)$$

which is an overestimation, although not large as the standard uncertainty of the measured values of PTB in most measuring points is less than half the standard uncertainty of the measured values of SP.

For each result Q_{SP}, the degree of equivalence is calculated as:

$$(d_{SP};\ U_{SP}) = (Q_{SP} - Q_{PTB} + d_{PTB};\ k \cdot u(d_{SP})) \quad (7)$$

with the coverage factor k=2.

6.2 Results

6.2.1 Results for the PTW Unidos E

Results for the PTW Unidos E at 100 fA, positive current direction:

Reference value: Q_{ref} = 1.00097982, $u(Q_{ref})$ = 1.18·10^{-4}
Uncertainty describing the instability of the transfer standard: u_{TS} = 3.47·10^{-4}
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
d_{PTB} = -0.1·10^{-4}
$U(d_{PTB})$ = 8.8·10^{-4}

Table 6.1: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000859	3.5·10^{-4}		
SP	1.002800	66.8·10^{-4}	1.9·10^{-3}	6.7·10^{-3}
PTB-2	1.000880	4.4·10^{-4}		

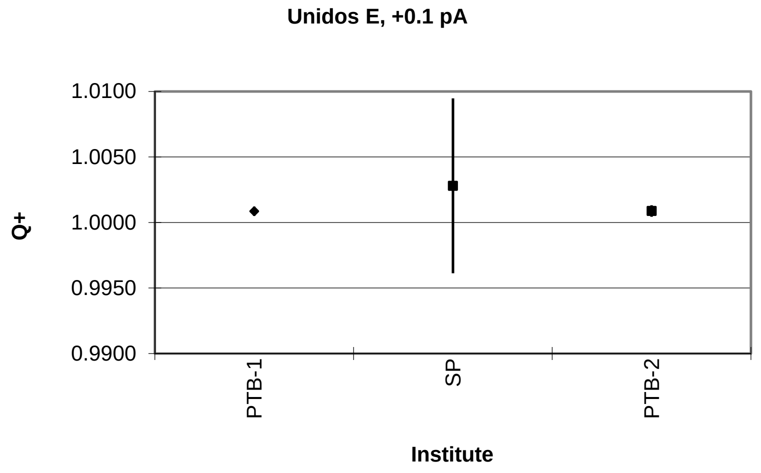

Fig. 6.1: Calibration factors for the Unidos at 100 fA for positive current direction.

Results for the PTW Unidos E at 100 fA, negative current direction:

Reference value: Q_{ref} = 1.00164994, $u(Q_{ref})$ = $1.20{\cdot}10^{-4}$
Uncertainty describing the instability of the transfer standard: u_{TS} = $3.47{\cdot}10^{-4}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
d_{PTB} = $1.6{\cdot}10^{-4}$
$U(d_{PTB})$ = $8.8{\cdot}10^{-4}$

Table 6.2: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1,001964	$3.5{\cdot}10^{-4}$		
SP	0,999600	$66.8{\cdot}10^{-4}$	$-2.3{\cdot}10^{-3}$	$6.7{\cdot}10^{-3}$
PTB-2	1,001620	$4.5{\cdot}10^{-4}$		

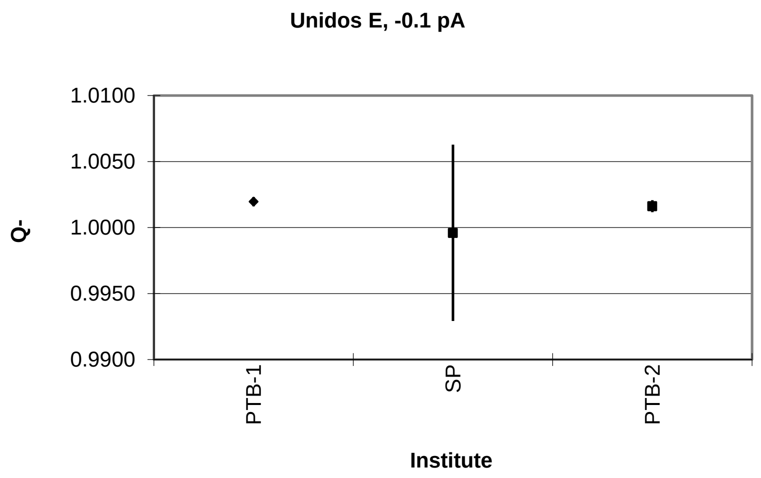

Fig. 6.2: Calibration factors for the Unidos at 100 fA for negative current direction.

Results for the PTW Unidos E at 100 fA, mean of both current directions:

Reference value: Q_{ref} = 1.00120970, $u(Q_{ref})$ = 1.08·10^{-4}
Uncertainty describing the instability of the transfer standard: u_{TS} = 3.47·10^{-4}
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
d_{PTB} = 1.8·10^{-4}
$U(d_{PTB})$ = 8.9·10^{-4}

Table 6.3: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1,001412	3.5·10^{-4}		
SP	1,001200	66.8·10^{-4}	-0.3·10^{-3}	6.7·10^{-3}
PTB-2	1,001250	4.5·10^{-4}		

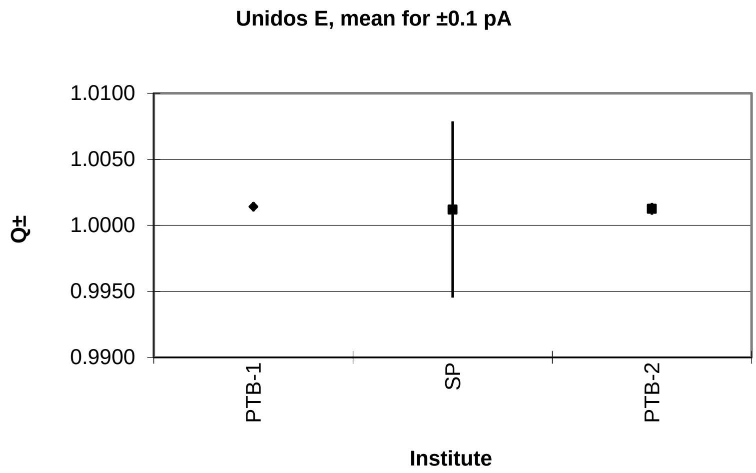

Fig. 6.3: Calibration factors for the Unidos at 100 fA for the mean of both current directions.

Results for the PTW Unidos E at 1 pA, positive current direction:

Reference value: Q_{ref} = 1.00053547, $u(Q_{ref})$ = 6.24·10^{-5}
Uncertainty describing the instability of the transfer standard: u_{TS} = 2.52·10^{-4}
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
d_{PTB} = -0.4·10^{-4}
$U(d_{PTB})$ = 5.1·10^{-4}

Table 6.4: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000590	1.4·10^{-4}		
SP	1.000750	10.2·10^{-4}	2.4·10^{-4}	11.5·10^{-4}
PTB-2	1.000396	1.3·10^{-4}		

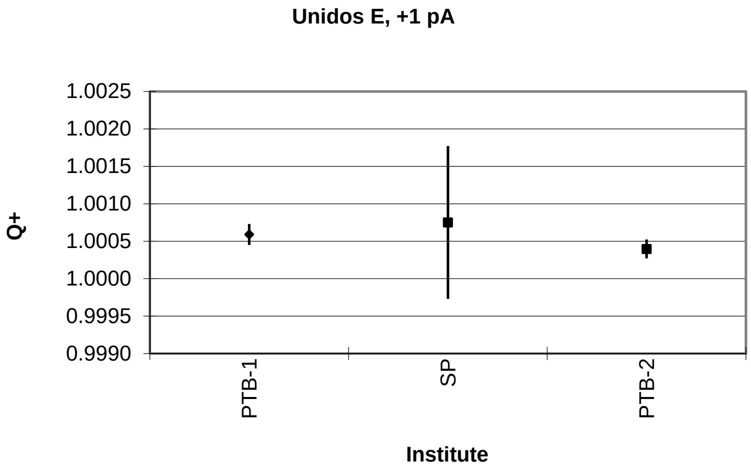

Fig. 6.4: Calibration factors for the Unidos at 1 pA for positive current direction.

Results for the PTW Unidos E at 1 pA, negative current direction:

Reference value: Q_{ref} = 1.00135313, $u(Q_{ref})$ = 6.45·10^{-5}
Uncertainty describing the instability of the transfer standard: u_{TS} = 2.52·10^{-4}
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
d_{PTB} = 0.8·10^{-4}
$U(d_{PTB})$ = 5.2·10^{-4}

Table 6.5: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.001461	1.4·10^{-4}		
SP	1.001400	10.2·10^{-4}	1.6·10^{-4}	11.5·10^{-4}
PTB-2	1.001259	1.3·10^{-4}		

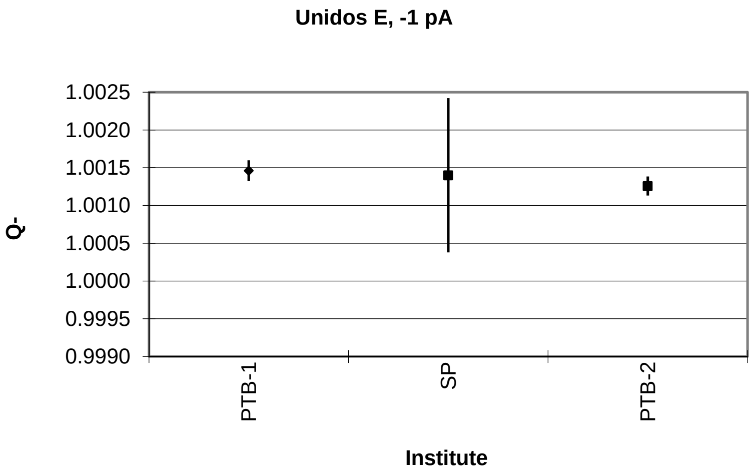

Fig. 6.5: Calibration factors for the Unidos at 1 pA for negative current direction.

Results for the PTW Unidos E at 1 pA, mean of both current directions:

Reference value: $Q_{ref} = 1.00090691$, $u(Q_{ref}) = 6.25\cdot10^{-5}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 2.52\cdot10^{-4}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = 0.6\cdot10^{-4}$
$U(d_{PTB}) = 5.2\cdot10^{-4}$

Table 6.6: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.001026	$1.4\cdot10^{-4}$		
SP	1.001075	$10.2\cdot10^{-4}$	$2.4\cdot10^{-4}$	$11.5\cdot10^{-4}$
PTB-2	1.000828	$1.3\cdot10^{-4}$		

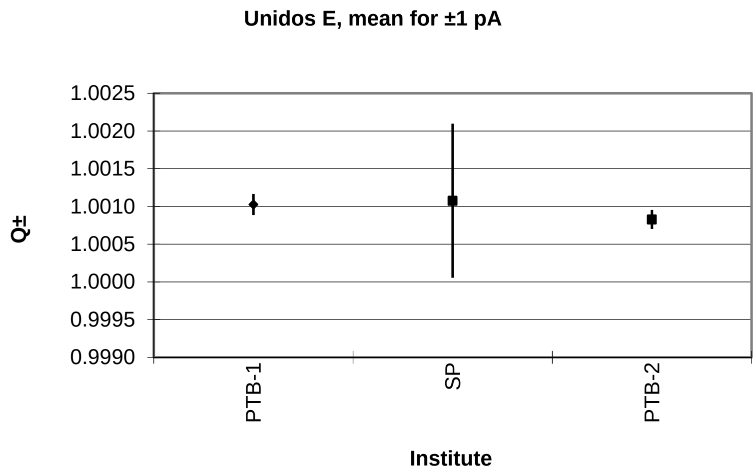

Fig. 6.6: Calibration factors for the Unidos at 1 pA for the mean of both current directions.

Results for the PTW Unidos E at 10 pA, positive current direction:

Reference value: $Q_{ref} = 1.00038719$, $u(Q_{ref}) = 4.45\cdot10^{-5}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 1.38\cdot10^{-4}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = -0.6\cdot10^{-4}$
$U(d_{PTB}) = 4.3\cdot10^{-4}$

Table 6.7: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000481	$3.4\cdot10^{-4}$		
SP	1.000093	$6.0\cdot10^{-4}$	$-1.9\cdot10^{-4}$	$7.0\cdot10^{-4}$
PTB-2	1.000090	$3.4\cdot10^{-4}$		

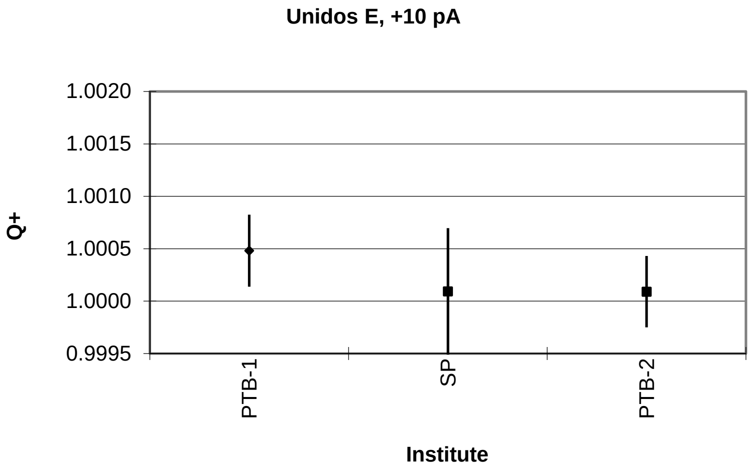

Fig. 6.7: Calibration factors for the Unidos at 10 pA for positive current direction.

Results for the PTW Unidos E at 10 pA, negative current direction:

Reference value: $Q_{ref} = 1.00123118$, $u(Q_{ref}) = 4.79 \cdot 10^{-5}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 1.38 \cdot 10^{-4}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = 0.7 \cdot 10^{-4}$
$U(d_{PTB}) = 4.3 \cdot 10^{-4}$

Table 6.8: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.001172	$3.4 \cdot 10^{-4}$		
SP	1.001497	$6.0 \cdot 10^{-4}$	$2.4 \cdot 10^{-4}$	$7.0 \cdot 10^{-4}$
PTB-2	1.001406	$3.4 \cdot 10^{-4}$		

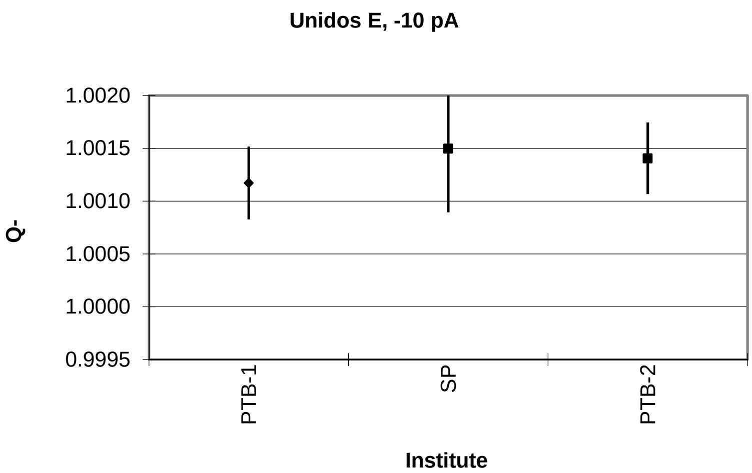

Fig. 6.8: Calibration factors for the Unidos at 10 pA for negative current direction.

Results for the PTW Unidos E at 10 pA, mean of both current directions:

Reference value: Q_{ref} = 1.00079514, $u(Q_{ref}) = 4.47 \cdot 10^{-5}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 1.38 \cdot 10^{-4}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = 0.2 \cdot 10^{-4}$
$U(d_{PTB}) = 4.3 \cdot 10^{-4}$

Table 6.9: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000827	$3.4 \cdot 10^{-4}$		
SP	1.000795	$6.0 \cdot 10^{-4}$	$0.4 \cdot 10^{-4}$	$7.0 \cdot 10^{-4}$
PTB-2	1.000748	$3.4 \cdot 10^{-4}$		

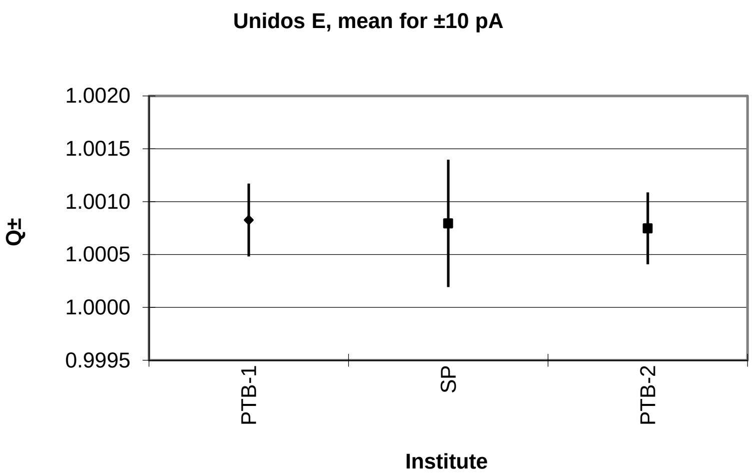

Fig. 6.9: : Calibration factors for the Unidos at 10 pA for the mean of both current directions.

Results for the PTW Unidos E at 100 pA, positive current direction:

Reference value: Q_{ref} = 1.00042401, $u(Q_{ref}) = 5.35 \cdot 10^{-5}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 2.0 \cdot 10^{-4}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = -0,5 \cdot 10^{-4}$
$U(d_{PTB}) = 5.1 \cdot 10^{-4}$

Table 6.10: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000584	$3.3 \cdot 10^{-4}$		
SP	1.000637	$5.8 \cdot 10^{-4}$	$-0.3 \cdot 10^{-4}$	$10.0 \cdot 10^{-4}$

PTB-2	1.000636	$3.5 \cdot 10^{-4}$		

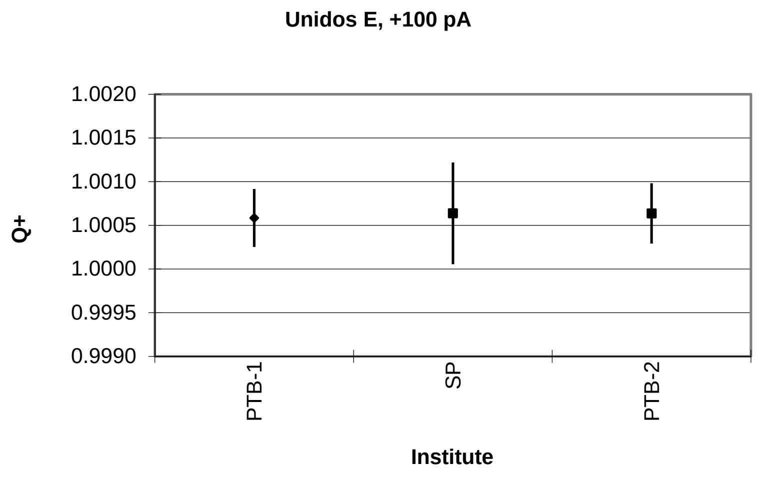

Fig. 6.10: Calibration factors for the Unidos at 100 pA for positive current direction.

Results for the PTW Unidos E at 100 pA, negative current direction:

Reference value: Q_{ref} = 1.00080189, $u(Q_{ref})$ = $5.42 \cdot 10^{-5}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 2.0 \cdot 10^{-4}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = 0.5 \cdot 10^{-4}$
$U(d_{PTB}) = 5.1 \cdot 10^{-4}$

Table 6.11: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000726	$3.3 \cdot 10^{-4}$		
SP	1.000564	$5.8 \cdot 10^{-4}$	$-0.5 \cdot 10^{-4}$	$10.0 \cdot 10^{-4}$
PTB-2	1.000636	$3.5 \cdot 10^{-4}$		

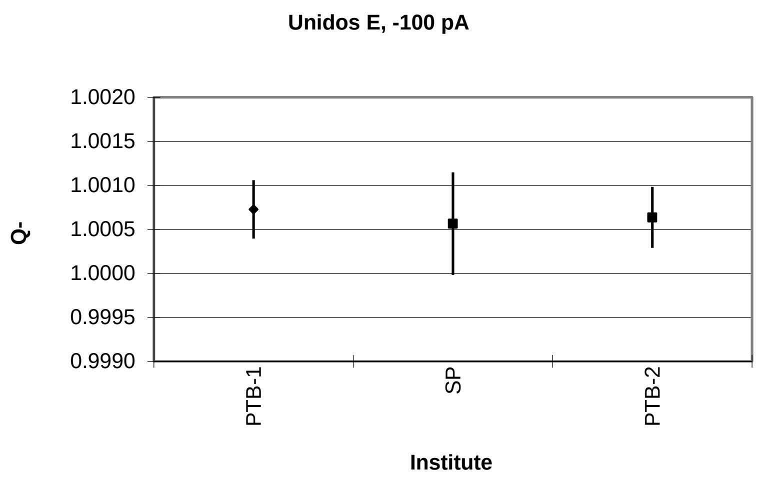

Fig. 6.11: Calibration factors for the Unidos at 100 pA for negative current direction.

Results for the PTW Unidos E at 100 pA, mean of both current direction:

Reference value: Q_{ref} = 1.00059677, $u(Q_{ref})$ = 5.37·10^{-5}
Uncertainty describing the instability of the transfer standard: u_{TS} = 2.0·10^{-4}
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
d_{PTB} = 0.1·10^{-4}
$U(d_{PTB})$ = 5.1·10^{-4}

Table 6.12: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000655	3.3·10^{-4}		
SP	1.000601	5.8·10^{-4}	-0.3·10^{-4}	10.0·10^{-4}
PTB-2	1.000636	3.5·10^{-4}		

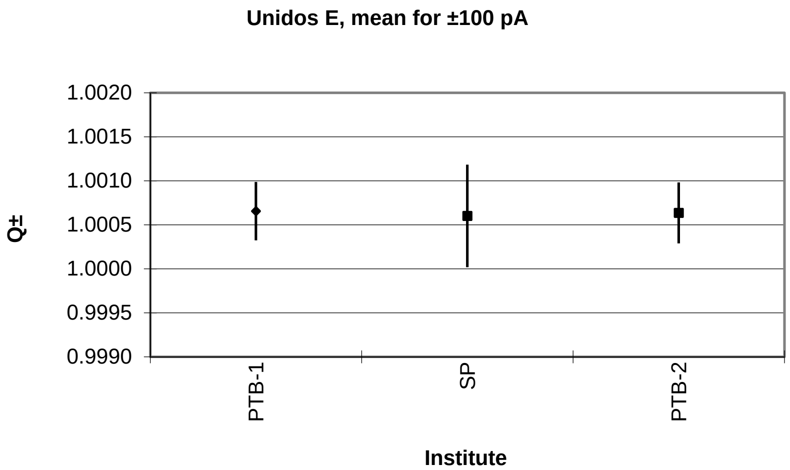

Fig. 6.12: Calibration factors for the Unidos at 100 pA for the mean of both current directions.

6.2.2 Results for the Keithley 6430

Results for the Keithley 6430 at 100 fA, positive current direction:

Reference value: Q_{ref} = 1.00093470, $u(Q_{ref})$ = 1.32·10^{-4}
Uncertainty describing the instability of the transfer standard: u_{TS} = 4.56·10^{-4}
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
d_{PTB} = -0.6·10^{-4}
$U(d_{PTB})$ = 10.8·10^{-4}

Table 6.13: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$

PTB-1	1.000216	$8.0 \cdot 10^{-4}$		
SP	1.002600	$81.9 \cdot 10^{-4}$	$2.2 \cdot 10^{-3}$	$8.3 \cdot 10^{-3}$
PTB-2	1.000631	$4.2 \cdot 10^{-4}$		

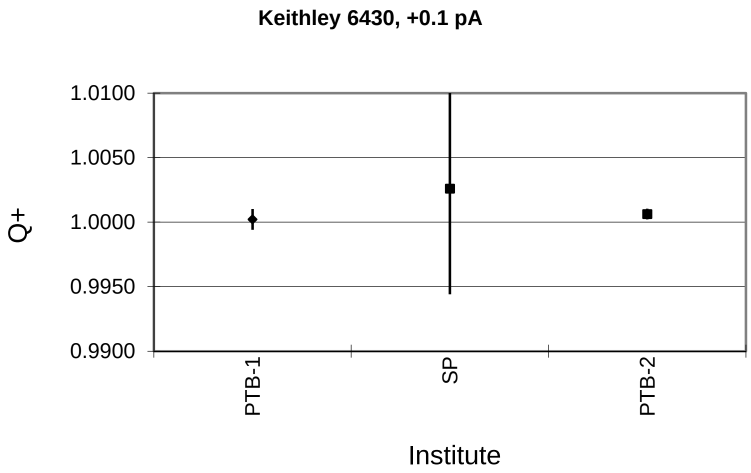

Fig. 6.13: Calibration factors for the Keithley at 100 fA for positive current direction.

Results for the Keithley 6430 at 100 fA, negative current direction:

Reference value: Q_{ref} = 1.00089524, $u(Q_{ref})$ = $1.37 \cdot 10^{-4}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 4.56 \cdot 10^{-4}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = 2.8 \cdot 10^{-4}$
$U(d_{PTB}) = 10.7 \cdot 10^{-4}$

Table 6.14: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000836	$5.9 \cdot 10^{-4}$		
SP	0.998600	$82.0 \cdot 10^{-4}$	$-2.3 \cdot 10^{-3}$	$8.3 \cdot 10^{-3}$
PTB-2	1.001284	$4.2 \cdot 10^{-4}$		

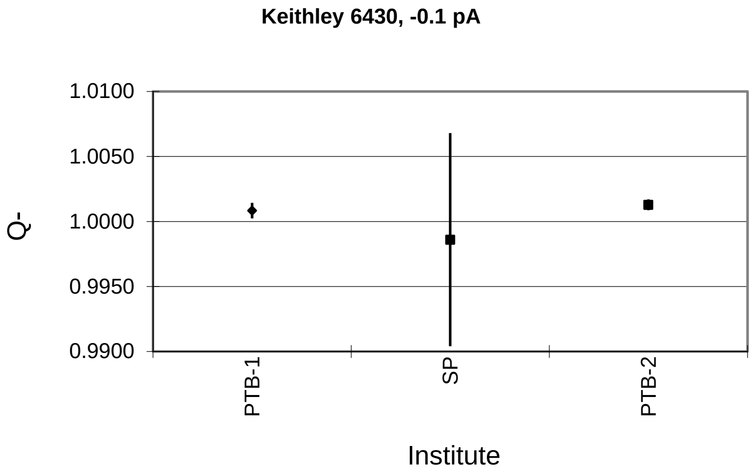

Fig. 6.14: Calibration factors for the Keithley at 100 pA for negative current direction.

Results for the Keithley 6430 at 100 fA, mean of both current directions:

Reference value: Q_{ref} = 1.00094466, $u(Q_{ref})$ = $1.25 \cdot 10^{-4}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 4.56 \cdot 10^{-4}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = 1.4 \cdot 10^{-4}$
$U(d_{PTB}) = 10.8 \cdot 10^{-4}$

Table 6.15: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000526	$7.0 \cdot 10^{-4}$		
SP	1.000600	$81.9 \cdot 10^{-4}$	$-0.7 \cdot 10^{-3}$	$8.3 \cdot 10^{-3}$
PTB-2	1.000958	$4.2 \cdot 10^{-4}$		

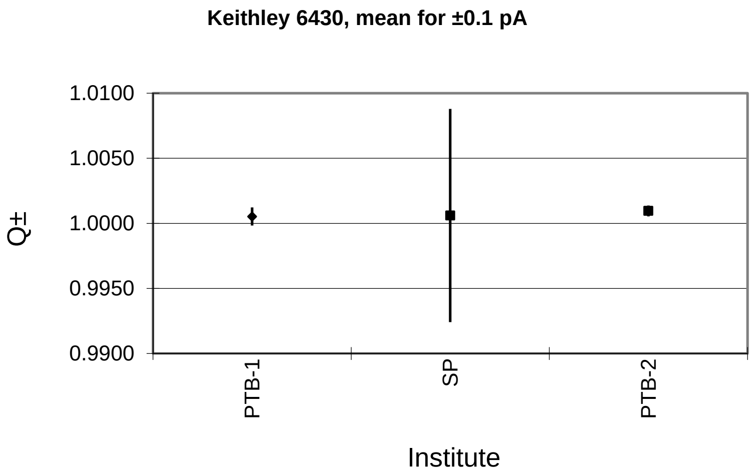

Fig. 6.15: Calibration factors for the Keithley at 100 fA for the mean of both current directions.

Results for the Keithley 6430 at 1 pA, positive current direction:

Reference value: $Q_{ref} = A + B \cdot t$, with:
$A = 1.00065496$, $u(A) = 3.03 \cdot 10^{-5}$
$B = 5.27 \cdot 10^{-8}$, $u(B) = 3.73 \cdot 10^{-8}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 9.37 \cdot 10^{-5}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = 0.0 \cdot 10^{-4}$
$U(d_{PTB}) = 2.2 \cdot 10^{-4}$

Table 6.16: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000448	$1.3 \cdot 10^{-4}$		
SP	1.000980	$7.1 \cdot 10^{-4}$	$2.8 \cdot 10^{-4}$	$7.5 \cdot 10^{-4}$
PTB-2	1.000827	$0.8 \cdot 10^{-4}$		

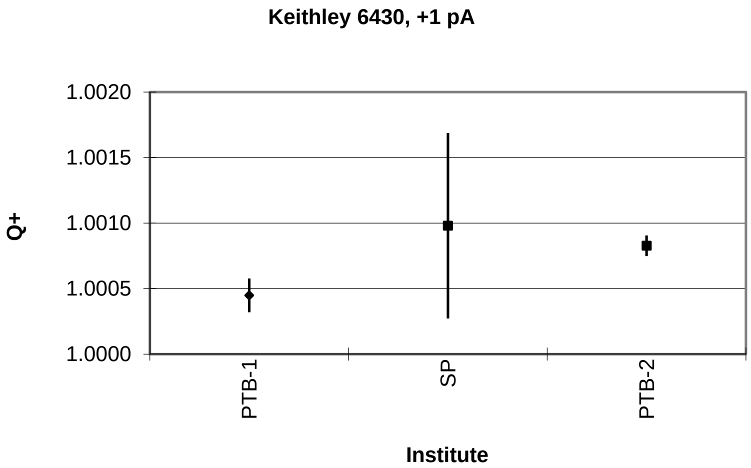

Fig. 6.16: Calibration factors for the Keithley at 1 pA for positive current direction.

Results for the Keithley 6430 at 1 pA, negative current direction:

Reference value: $Q_{ref} = A + B \cdot t$, with:
$A = 1.00063836$, $u(A) = 3.05 \cdot 10^{-5}$
$B = 5.27 \cdot 10^{-8}$, $u(B) = 3.73 \cdot 10^{-8}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 9.37 \cdot 10^{-5}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = -0.1 \cdot 10^{-4}$
$U(d_{PTB}) = 2.3 \cdot 10^{-4}$

Table 6.17: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000468	$1.3 \cdot 10^{-4}$		
SP	1.000640	$7.1 \cdot 10^{-4}$	$0.8 \cdot 10^{-4}$	$7.5 \cdot 10^{-4}$
PTB-2	1.000591	$0.8 \cdot 10^{-4}$		

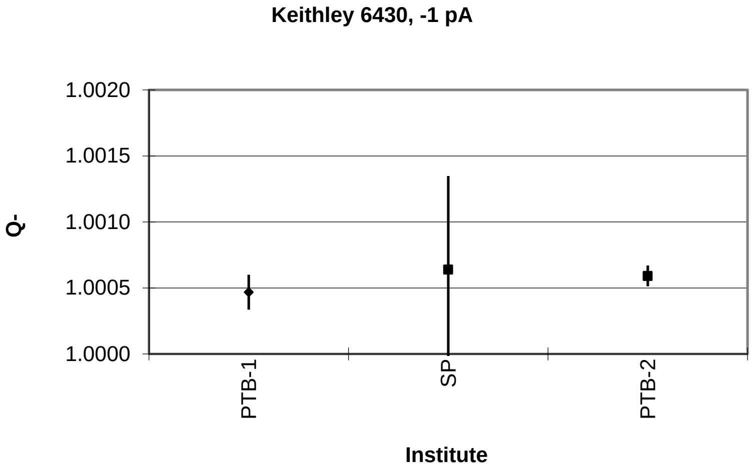

Fig. 6.17: Calibration factors for the Keithley at 1 pA for negative current direction.

Results for the Keithley 6430 at 1 pA, mean of both current directions:

Reference value: $Q_{ref} = A + B \cdot t$, with:
$A = 1.00066043$, $u(A) = 2.84 \cdot 10^{-5}$
$B = 5.27 \cdot 10^{-8}$, $u(B) = 3.73 \cdot 10^{-8}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 9.37 \cdot 10^{-5}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = -0.2 \cdot 10^{-4}$
$U(d_{PTB}) = 2.2 \cdot 10^{-4}$

Table 6.18: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000589	$1.3 \cdot 10^{-4}$		
SP	1.000810	$7.1 \cdot 10^{-4}$	$1.7 \cdot 10^{-4}$	$7.5 \cdot 10^{-4}$
PTB-2	1.000709	$0.8 \cdot 10^{-4}$		

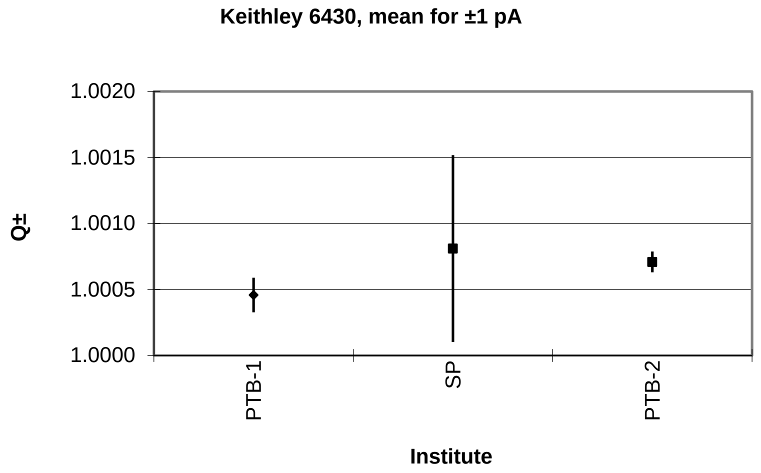

Fig. 6.18: Calibration factors for the Keithley at 1 pA for the mean of both current directions.

Results for the Keithley 6430 at 10 pA, positive current direction:

Reference value: $Q_{ref} = A + B \cdot t$, with:
$A = 1.00001214$, $u(A) = 1.14 \cdot 10^{-5}$
$B = 1.03 \cdot 10^{-7}$, $u(B) = 2.23 \cdot 10^{-8}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 3.23 \cdot 10^{-5}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = -0.1 \cdot 10^{-5}$
$U(d_{PTB}) = 10.0 \cdot 10^{-5}$

Table 6.19: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000047	$7.2 \cdot 10^{-5}$		
SP	1.000052	$9.7 \cdot 10^{-5}$	$-4.1 \cdot 10^{-5}$	$13.9 \cdot 10^{-5}$
PTB-2	1.000095	$6.5 \cdot 10^{-5}$		

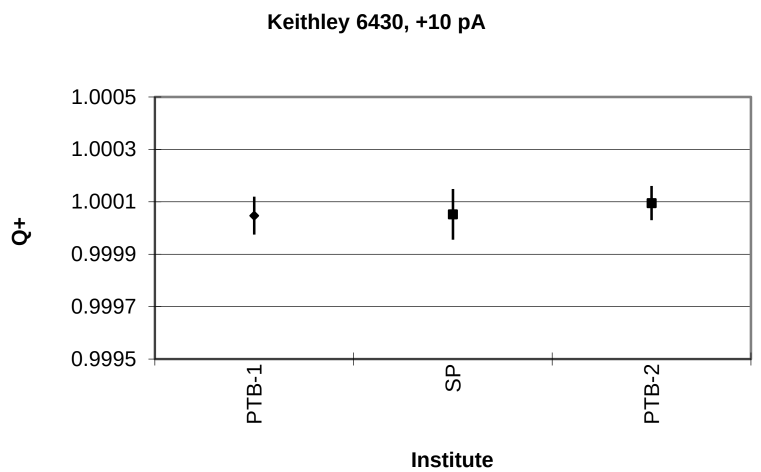

Fig. 6.19: Calibration factors for the Keithley at 10 pA for positive current direction.

Results for the Keithley 6430 at 10 pA, negative current direction:

Reference value: $Q_{ref} = A + B \cdot t$, with:
$A = 0.99986889$, $u(A) = 1.12 \cdot 10^{-5}$
$B = 1.03 \cdot 10^{-7}$, $u(B) = 2.23 \cdot 10^{-8}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 3.23 \cdot 10^{-5}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = -0.1 \cdot 10^{-5}$
$U(d_{PTB}) = 9.9 \cdot 10^{-5}$

Table 6.20: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	0.999927	$7.2 \cdot 10^{-5}$		
SP	0.999962	$9.6 \cdot 10^{-5}$	$0.2 \cdot 10^{-5}$	$13.8 \cdot 10^{-5}$
PTB-2	0.999966	$6.5 \cdot 10^{-5}$		

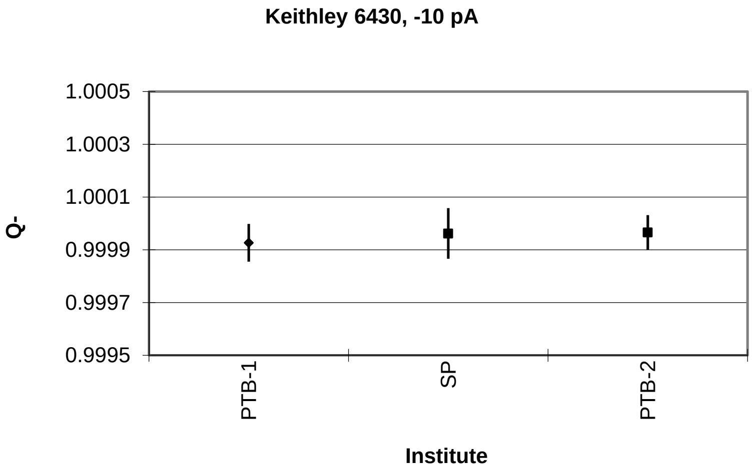

Fig. 6.20: Calibration factors for the Keithley at 10 pA for negative current direction.

Results for the Keithley 6430 at 10 pA, mean of both current directions:

Reference value: $Q_{ref} = A + B \cdot t$, with:
$A = 0.99994378$, $u(A) = 1.12 \cdot 10^{-5}$
$B = 1.03 \cdot 10^{-7}$, $u(B) = 2.23 \cdot 10^{-8}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 3.23 \cdot 10^{-5}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = -0.1 \cdot 10^{-5}$
$U(d_{PTB}) = 9.9 \cdot 10^{-5}$

Table 6.21: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	0.999987	$7.2\cdot10^{-5}$		
SP	1.000007	$9.6\cdot10^{-5}$	$-2.2\cdot10^{-5}$	$13.8\cdot10^{-5}$
PTB-2	1.000031	$6.5\cdot10^{-5}$		

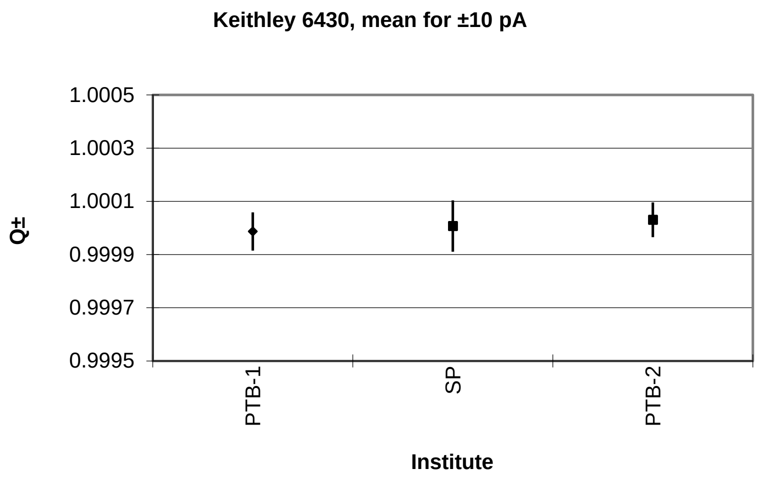

Fig. 6.21: Calibration factors for the Keithley at 10 pA for the mean of both current directions.

Results for the Keithley 6430 at 100 pA, positive current direction:

Reference value: $Q_{ref} = A + B \cdot t$, with:
$A = 1.00025007$, $u(A) = 7.33\cdot10^{-6}$
$B = 6.94\cdot10^{-8}$, $u(B) = 7.45\cdot10^{-9}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 2.67\cdot10^{-5}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = -0.3\cdot10^{-5}$
$U(d_{PTB}) = 5.8\cdot10^{-5}$

Table 6.22: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000270	$2.4\cdot10^{-5}$		
SP	1.000290	$5.5\cdot10^{-5}$	$-3.0\cdot10^{-5}$	$8.2\cdot10^{-5}$
PTB-2	1.000341	$2.4\cdot10^{-5}$		

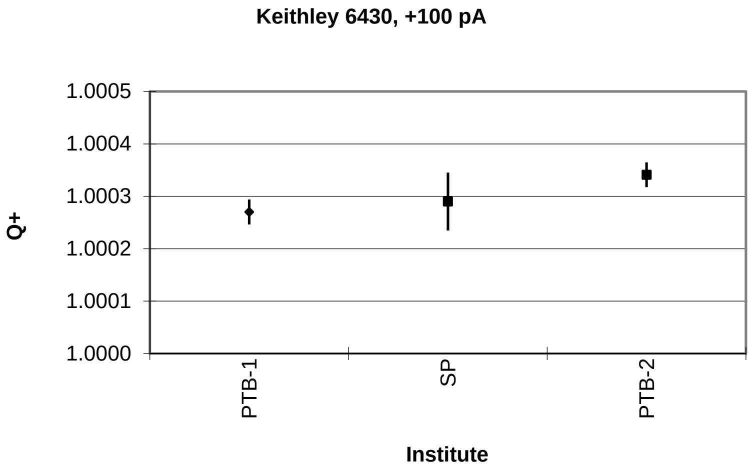

Fig. 6.22: Calibration factors for the Keithley at 100 pA for positive current direction.

Results for the Keithley 6430 at 100 pA, negative current direction:

Reference value: $Q_{ref} = A + B \cdot t$, with:
$A = 1.00026826$, $u(A) = 7.35 \cdot 10^{-6}$
$B = 6.94 \cdot 10^{-8}$, $u(B) = 7.45 \cdot 10^{-9}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 2.67 \cdot 10^{-5}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = -1.4 \cdot 10^{-5}$
$U(d_{PTB}) = 5.7 \cdot 10^{-5}$

Table 6.23: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000301	$2.4 \cdot 10^{-5}$		
SP	1.000308	$5.5 \cdot 10^{-5}$	$-4.4 \cdot 10^{-5}$	$8.2 \cdot 10^{-5}$
PTB-2	1.000357	$2.4 \cdot 10^{-5}$		

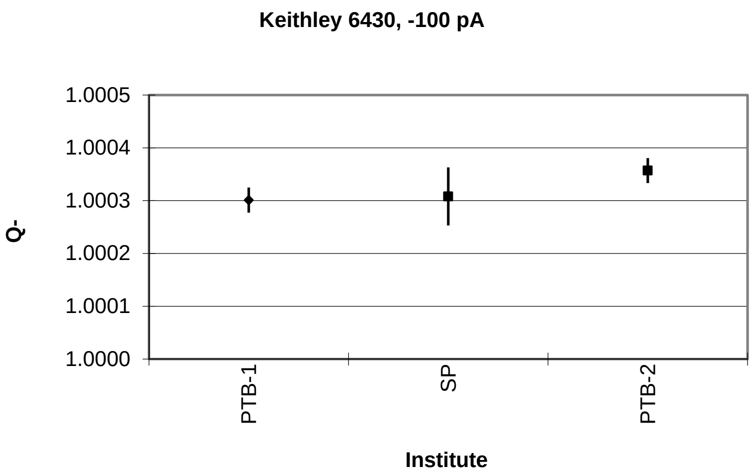

Fig. 6.23: Calibration factors for the Keithley at 100 pA for negative current direction.

Results for the Keithley 6430 at 100 pA, mean of both current directions:

Reference value: $Q_{ref} = A + B \cdot t$, with:
$A = 1.00025761$, $u(A) = 7.31 \cdot 10^{-6}$
$B = 6.94 \cdot 10^{-8}$, $u(B) = 7.45 \cdot 10^{-9}$
Uncertainty describing the instability of the transfer standard: $u_{TS} = 2.67 \cdot 10^{-5}$
Degree of equivalence (d_{PTB}; $U(d_{PTB})$) determined as the mean of all degree of equivalence of PTB for each current and travelling instrument in the EURAMET.EM-S24:
$d_{PTB} = -0.7 \cdot 10^{-5}$
$U(d_{PTB}) = 5.7 \cdot 10^{-5}$

Table 6.24: Summary of the results, containing each participant's result Q_i, its uncertainty $u(Q_i)$, the degree of equivalence (d_{SP}; $U(d_{SP})$) of SP calculated according to (7).

Institute	Q_i	$u(Q_i)$	d_i	$U(d_i)$
PTB-1	1.000286	$2.4 \cdot 10^{-5}$		
SP	1.000299	$5.5 \cdot 10^{-5}$	$-3.5 \cdot 10^{-5}$	$8.2 \cdot 10^{-5}$
PTB-2	1.000349	$2.4 \cdot 10^{-5}$		

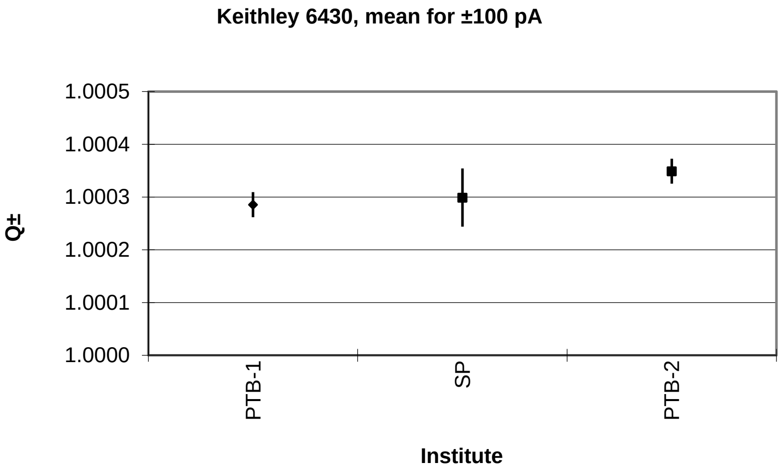

Fig. 6.24: Calibration factors for the Keithley at 100 pA for the mean of both current directions.

7. Withdrawals or changes of results

There were no withdrawals or changes of results.

8. Summary and conclusions

A bilateral comparison has been made between SP and PTB, pilot laboratory, in the field of small DC currents below 1 nA. The technical protocol was similar to the one used in the supplementary comparison EURAMET.EM-S24. The aim of the comparison was to evaluate the capability of SP to traceably calibrate picoammeters. For that purpose, two different commercial picoammeters were used as travelling instruments. They were calibrated at current values of ±100 fA, ±1 pA, ±10 pA and ±100 pA. The agreement between the participants was good.

9. References

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Appendix A: Results of the participants in chronological order

PTB-1

Nominal Current	Date	Temp.	U (Temp)	Press.	U (Press)	Humid.	U (Humid)	Current	Range	Reading	Q	u(Q), k=1	eff. DOF	k	U=k*u(Q)
A		°C	°C	hPa	hPa	% R.H.	% R.H.	A	A	A					
K6430															
1,00E-13	2010-01-11	22,9	0,5	1005	2	30,6	1	9,6843E-14	1,0E-12	9,6864E-14	1,000216	3,9E-04	41	2,1	8,0E-04
-1,00E-13	2010-01-11	22,9	0,5	1005	2	30,6	1	-9,5453E-14	1,0E-12	-9,5533E-14	1,000836	2,9E-04	43	2,1	5,9E-04
1,00E-12	2010-01-11	22,9	0,5	1005	2	30,6	1	9,6724E-13	1,0E-12	9,6767E-13	1,000448	6,4E-05	269	2,0	1,3E-04
-1,00E-12	2010-01-11	22,9	0,5	1005	2	30,6	1	-9,5336E-13	1,0E-12	-9,5381E-13	1,000468	6,6E-05	237	2,0	1,3E-04
1,00E-11	2010-01-06	22,9	0,5	991	2	41,1	1	9,6724E-12	1,0E-11	9,6729E-12	1,000047	3,6E-05	1156	2,0	7,2E-05
-1,00E-11	2010-01-06	22,9	0,5	991	2	41,1	1	-9,5340E-12	1,0E-11	-9,5333E-12	0,999927	3,6E-05	1814	2,0	7,2E-05
1,00E-10	2010-01-07	22,8	0,5	1007	2	34,2	1	9,7019E-11	1,0E-10	9,7045E-11	1,000270	1,2E-05	inf	2,0	2,4E-05
-1,00E-10	2010-01-07	22,8	0,5	1007	2	34,2	1	-9,5681E-11	1,0E-10	-9,5710E-11	1,000301	1,2E-05	inf	2,0	2,4E-05
Unidos E															
1,00E-13	2010-01-13	22,9	0,5	998	2	29,1	1	9,6847E-14	LOW	9,6930E-14	1,000859	2,1E-04	95	2,0	3,5E-04
-1,00E-13	2010-01-13	22,9	0,5	998	2	29,1	1	-9,5436E-14	LOW	-9,5623E-14	1,001964	2,4E-04	90	2,0	3,5E-04
1,00E-12	2010-01-15	22,9	0,5	1010	2	37,1	1	9,6738E-13	LOW	9,6795E-13	1,000590	7,0E-05	597	2,0	1,4E-04
-1,00E-12	2010-01-15	22,9	0,5	1010	2	37,1	1	-9,5332E-13	LOW	-9,5471E-13	1,001461	6,9E-05	691	2,0	1,4E-04
1,00E-11	2010-01-15	23,4	0,5	1005	2	26,6	1	9,6737E-12	LOW	9,6784E-12	1,000481	1,7E-04	inf	2,0	3,4E-04
-1,00E-11	2010-01-15	23,4	0,5	1005	2	26,6	1	-9,5337E-12	LOW	-9,5449E-12	1,001172	1,7E-04	inf	2,0	3,4E-04
1,00E-10	2010-01-21	23,2	0,5	1012	2	17,3	1	9,7055E-11	LOW	9,7112E-11	1,000584	1,7E-04	inf	2,0	3,3E-04
-1,00E-10	2010-01-21	23,2	0,5	1012	2	17,3	1	-9,5673E-11	LOW	-9,5742E-11	1,000726	1,7E-04	inf	2,0	3,3E-04
mean		23,0		1004		30,8									
first day	2010-01-06														
last day	2010-01-21														
Median date of meas.	2010-01-13														

SP

Nominal Current	Date	Temp.	U (Temp)	Press.	U (Press)	Humid.	U (Humid)	Current	Range	Reading	Q	u(Q), k=1	eff. DOF	k	U=k*u(Q)
A		°C	°C	hPa	hPa	% R.H.	% R.H.	A	A	A					
K6430															
1,00E-13	2010-03-11	22,4	0,5	990	5	45,0	5	9,5000E-14	1,0E-12	9,5247E-14	1,002600	4,0E-03	54	2,0	8,2E-03
-1,00E-13	2010-03-11	22,4	0,5	990	5	45,0	5	-9,5000E-14	1,0E-12	-9,4867E-14	0,998600	4,0E-03	51	2,0	8,2E-03
1,00E-12	2010-03-06	22,4	0,5	990	5	45,0	5	9,5000E-13	1,0E-12	9,5093E-13	1,000980	3,5E-04	111	2,0	7,1E-04
-1,00E-12	2010-03-06	22,4	0,5	990	5	45,0	5	-9,5000E-13	1,0E-12	-9,5061E-13	1,000640	3,5E-04	112	2,0	7,1E-04
1,00E-11	2010-03-16	22,8	0,5	996	5	45,0	5	9,5000E-12	1,0E-11	9,5005E-12	1,000052	4,3E-05	11	2,3	9,7E-05
-1,00E-11	2010-03-16	22,8	0,5	996	5	45,0	5	-9,5000E-12	1,0E-11	-9,4996E-12	0,999962	4,3E-05	12	2,2	9,6E-05
1,00E-10	2010-03-22	22,4	0,5	995	5	47,0	5	9,5000E-11	1,0E-10	9,5028E-11	1,000290	2,6E-05	21	2,1	5,5E-05
-1,00E-10	2010-03-22	22,4	0,5	995	5	47,0	5	-9,5000E-11	1,0E-10	-9,5029E-11	1,000308	2,6E-05	22	2,1	5,5E-05
Unidos E															
1,00E-13	2010-03-15	22,5	0,5	983	5	46,0	5	9,5000E-14	LOW	9,5266E-14	1,002800	3,3E-03	106	2,0	6,7E-03
-1,00E-13	2010-03-15	22,5	0,5	983	5	46,0	5	-9,5000E-13	LOW	-9,4962E-13	0,999600	3,3E-03	106	2,0	6,7E-03
1,00E-12	2010-03-15	22,5	0,5	985	5	44,0	5	9,5000E-11	LOW	9,5071E-11	1,000750	5,0E-04	60	2,0	1,0E-03
-1,00E-12	2010-03-15	22,5	0,5	985	5	44,0	5	-9,5000E-13	LOW	-9,5133E-13	1,001400	5,0E-04	60	2,0	1,0E-03
1,00E-11	2010-03-17	22,4	0,5	995	5	45,0	5	9,5000E-12	LOW	9,5009E-12	1,000093	3,0E-04	300	2,0	6,0E-04
-1,00E-11	2010-03-17	22,4	0,5	995	5	45,0	5	-9,5000E-12	LOW	-9,5142E-12	1,001497	3,0E-04	300	2,0	6,0E-04
1,00E-10	2010-03-17	22,4	0,5	996	5	44,0	5	9,5000E-11	LOW	9,5061E-11	1,000637	2,9E-04	300	2,0	5,8E-04
-1,00E-10	2010-03-17	22,4	0,5	996	5	44,0	5	-9,5000E-11	LOW	-9,5054E-11	1,000564	2,9E-04	300	2,0	5,8E-04
mean		22,5		991		45,1									
first day	2010-03-06														
last day	2010-03-22														
Median date of meas.	2010-03-14														

PTB-2

Nominal Current	**Date**	**Temp.**	**U (Temp)**	**Press.**	**U (Press)**	**Humid.**	**U (Humid)**	**Current**	**Range**	**Reading**	**Q**	**u(Q), k=1**	**eff. DOF**	**k**	**U=k*u(Q)**
A		°C	°C	hPa	hPa	% R.H.	% R.H.	A	A	A					
K6430															
1,00E-13	2010-04-14	23,1	0,5	1006	2	28,7	1	9,4615E-14	1,0E-12	9,4675E-14	1,000631	2,1E-04	189	2,0	4,2E-04
-1,00E-13	2010-04-14	23,1	0,5	1006	2	28,7	1	-9,4615E-14	1,0E-12	-9,4736E-14	1,001284	2,1E-04	189	2,0	4,2E-04
1,00E-12	2010-04-15	23,0	0,5	1007	2	24,0	1	9,4511E-13	1,0E-12	9,4589E-13	1,000827	4,0E-05	585	2,0	7,9E-05
-1,00E-12	2010-04-15	23,0	0,5	1007	2	24,0	1	-9,4511E-13	1,0E-12	-9,4567E-13	1,000591	3,9E-05	606	2,0	7,9E-05
1,00E-11	2010-04-13	22,9	0,5	1009	2	27,6	1	9,4510E-12	1,0E-11	9,4519E-12	1,000095	3,3E-05	inf	2	6,5E-05
-1,00E-11	2010-04-13	22,9	0,5	1009	2	27,6	1	-9,4511E-12	1,0E-11	-9,4508E-12	0,999966	3,3E-05	inf	2	6,5E-05
1,00E-10	2010-04-12	23,0	0,5	1012	2	28,2	1	-9,4536E-11	1,0E-10	-9,4568E-11	1,000341	1,2E-05	inf	2	2,4E-05
-1,00E-10	2010-04-12	23,0	0,5	1012	2	28,2	1	9,4535E-11	1,0E-10	9,4569E-11	1,000357	1,2E-05	inf	2	2,4E-05
Unidos E															
1,00E-13	2010-04-15	23,0	0,5	1007	2	24,0	1	9,4615E-14	LOW	9,4698E-14	1,000880	2,2E-04	94	2,0	4,4E-04
-1,00E-13	2010-04-15	23,0	0,5	1007	2	24,0	1	-9,4615E-14	LOW	-9,4768E-14	1,001620	2,2E-04	93	2,0	4,5E-04
1,00E-12	2010-04-14	23,1	0,5	1006	2	28,7	1	9,4511E-13	LOW	9,4548E-13	1,000396	6,3E-05	7205	2,0	1,3E-04
-1,00E-12	2010-04-14	23,1	0,5	1006	2	28,7	1	-9,4511E-13	LOW	-9,4630E-13	1,001259	6,3E-05	7069	2,0	1,3E-04
1,00E-11	2010-04-13	22,9	0,5	1009	2	27,6	1	9,4510E-12	LOW	9,4519E-12	1,000090	1,7E-04	inf	2	3,4E-04
-1,00E-11	2010-04-13	22,9	0,5	1009	2	27,6	1	-9,4511E-12	LOW	-9,4644E-12	1,001406	1,7E-04	inf	2	3,4E-04
1,00E-10	2010-04-12	23,0	0,5	1012	2	28,2	1	9,4535E-11	LOW	9,4595E-11	1,000636	1,7E-04	inf	2	3,5E-04
-1,00E-10	2010-04-12	23,0	0,5	1012	2	28,2	1	-9,4536E-11	LOW	-9,4596E-11	1,000636	1,7E-04	inf	2	3,5E-04
mean		23,0		1009		27,1									
first day	2010-04-12														
last day	2010-04-15														
Median date of meas.	2010-04-13														

Appendix B: Uncertainty budgets of the participants

It is clear, that in this report it is impossible to present all uncertainty budgets. Instead, for each participant only two error budgets will be presented, namely those for the Keithley 6430 at the current values +100 fA and +100 pA. This choice was made because these current values are at the extremes and because the Keithley 6430 was the more stable instrument causing fewer problems (see also Section 3.5).

Uncertainty budgets of PTB (pilot institute)

The uncertainty budgets were calculated using the following model equation:

$$Q_{\pm} = \frac{k_{a1} + k_{a2} + k_{a3}}{k_{Cacdc} + k_c + k_{us} + k_{ul} + k_t} \cdot \overline{Q_{\pm}}$$

where eight "k" parameters with values equal to one are introduced in order to describe the different sources of type B uncertainties:

k_{a1}	describes the limited resolution of the transfer instrument in the zero-phase before the current phase.
k_{a2}	describes the limited resolution of the transfer instrument in the current phase.
k_{a3}	describes the limited resolution of the transfer instrument in the zero-phase following the current phase.
k_C	describes the uncertainty of the capacitance.
k_{Cacdc}	describes the uncertainty of the capacitance due to ac-dc differences.
k_{us}	describes the short-term uncertainty of the DMM.
k_{ul}	describes the long-term uncertainty of the DMM.
k_t	describes the timing uncertainty of the slope measurement.

Generally, k_{a1} and k_{a3} are identical. k_{a1}, k_{a2} and k_{a3} are negligible for the Keithley 6430 while the limited resolution and the "locking effect" of the Unidos E were taken into account by using the one-sigma-uncertainties given in the following table:

Current:	**100 fA**	**1 pA**	**10 pA**	**100 pA**
$u(k_{a1}) = u(k_{a3})$	$1.5\cdot10^{-5}$	$1.5\cdot10^{-6}$	$1.5\cdot10^{-7}$	$1.5\cdot10^{-8}$
$u(k_{a2})$	$3.0\cdot10^{-5}$	$3.0\cdot10^{-5}$	$1.6\cdot10^{-4}$	$1.6\cdot10^{-4}$

In the following, only two uncertainty budgets are presented, namely those for the last one of the eleven regular PTB runs ("PTB-11").

Example of an uncertainty budget of PTB for the Keithley 6430 at a current of 95 fA:

Quantity	Estimate	Standard uncertainty	Prob. dist.	Type	Sensitivity coefficient	Uncertainty contribution	Degrees of freedom
$\overline{Q}_+$	1.000163	$2.7\cdot10^{-4}$	N	A	1	$2.7\cdot10^{-4}$	79
k_{a1}	1	$1.5\cdot10^{-5}$	R	B	1	$1.5\cdot10^{-5}$	∞
k_{a2}	1	$3.0\cdot10^{-5}$	R	B	1	$3.0\cdot10^{-5}$	∞
k_{a3}	1	$1.5\cdot10^{-5}$	R	B	1	$1.5\cdot10^{-5}$	∞
k_C	1	$2.3\cdot10^{-6}$	R	B	-1	$-2.3\cdot10^{-6}$	∞
k_{Cacdc}	1	$4.0\cdot10^{-5}$	R	B	-1	$-4.0\cdot10^{-5}$	∞
k_{us}	1	$2.4\cdot10^{-5}$	R	B	-1	$-2.4\cdot10^{-5}$	∞
k_{ul}	1	$2.4\cdot10^{-6}$	R	B	-1	$-2.4\cdot10^{-6}$	∞
k_t	1	$5.8\cdot10^{-8}$	R	B	-1	$-5.8\cdot10^{-8}$	∞
$\boldsymbol{Q_+}$	**1.000163**					$\mathbf{2.7\cdot10^{-4}}$	**87**

Example of an uncertainty budget of PTB for the Keithley 6430 at a current of 95 pA:

Quantity	Estimate	Standard uncertainty	Prob. dist.	Type	Sensitivity coefficient	Uncertainty contribution	Degrees of freedom
$\overline{Q}_+$	1.000282	$2.6\cdot10^{-6}$	N	A	1	$2.6\cdot10^{-6}$	59
k_{a1}	1	$1.5\cdot10^{-8}$	R	B	1	$1.5\cdot10^{-8}$	∞
k_{a2}	1	$3.0\cdot10^{-8}$	R	B	1	$3.0\cdot10^{-8}$	∞
k_{a3}	1	$1.5\cdot10^{-8}$	R	B	1	$1.5\cdot10^{-8}$	∞
k_C	1	$2.3\cdot10^{-6}$	R	B	-1	$-2.3\cdot10^{-6}$	∞
k_{Cacdc}	1	$1.0\cdot10^{-5}$	R	B	-1	$1.0\cdot10^{-5}$	∞
k_{us}	1	$5.1\cdot10^{-6}$	R	B	-1	$-5.1\cdot10^{-6}$	∞
k_{ul}	1	$2.4\cdot10^{-6}$	R	B	-1	$-2.4\cdot10^{-6}$	∞
k_t	1	$5.8\cdot10^{-8}$	R	B	-1	$-5.8\cdot10^{-8}$	∞
$\boldsymbol{Q_+}$	**1.000282**					$\mathbf{1.2\cdot10^{-5}}$	∞

Uncertainty budgets of SP

The measured calibration factor Q is calculated using the model equation:

$$Q = \frac{I_i}{I_s} = \frac{I_i}{C\dfrac{dV}{dt}} \tag{B1}$$

where I_i is the current indicated by the device under test and I_s is the current applied by the current source standard. *C* is the value of the capacitor and *dV/dt* is rate of voltage change.

We can write the relative standard uncertainty of the calibration factor *u(Q)/Q* as:

$$\frac{u^2(Q)}{Q^2} = \frac{u^2(I_i)}{I_i^2} + \frac{u^2(C)}{C^2} + \frac{u^2\left(\dfrac{dV}{dt}\right)}{\left(\dfrac{dV}{dt}\right)^2} \tag{B2}$$

where

$$u^2(I_i) = u^2(I_r) + u^2(I_{res}) + u^2(I_L) \tag{B3}$$

$$u^2(C) = u^2(C_{cal}) + u^2(C_{ac-dc}) \tag{B4}$$

$$\frac{u^2\left(\dfrac{dV}{dt}\right)}{\left(\dfrac{dV}{dt}\right)^2} = \frac{u^2(V_{\mathrm{cal}}) + u^2(V_{\mathrm{d}})}{V_{\mathrm{max}}^2} \tag{B5}$$

Here $u(C_{cal})$ and $u(V_{cal})$ are the standard uncertainties of the capacitance and the DMM voltage calibration respectively, $u(I_r)$ is the standard deviation of the mean of the difference in reading of DUT between applied current and zero current, $u(I_{res})$ is the standard deviation due to the resolution of the reading of DUT, $u(I_L)$is the standard uncertainty due to leakage in the capacitor, $u(C_{ac\text{-}dc})$ is the standard uncertainty of the correction due to ac-dc difference of the capacitance, $u(V_d)$ is the standard uncertainty due to the stability of the measured ramp and V_{max} is the maximum voltage of the ramp.

We can then rewrite the relative standard uncertainty of the measurand as:

$$\frac{u^2(Q)}{Q^2} = \frac{u^2(I_{\mathrm{r}})}{I_r^2} + \frac{u^2(I_{\mathrm{res}})}{I_r^2} + \frac{u^2(I_{\mathrm{L}})}{I_r^2} + \frac{u^2(C)}{C^2} + \frac{u^2(V_{\mathrm{cal}})}{V_{\mathrm{max}}^2} + \frac{u^2(V_{\mathrm{d}})}{V_{\mathrm{max}}^2} \tag{B6}$$

The standard uncertainty of the calibration of the capacitance at 1 kHz, $u(C_{cal})$, is negligible compared to ac-dc difference of the capacitance, $u(C_{ac\text{-}dc})$. Also other sources of error are negligible, like the error due to voltage burden of the DUT and timing of the voltage ramp. For the Keithley 6430 the standard uncertainty due to resolution is negligible.

The uncertainty budget below is an example for one of the measurements made at SP (measurement No. 2) and not for the mean of all three measurements. The reason for this is that the measuring method was slightly changed after the first measurement, as mentioned earlier. Also, the value of the measured current is normalized to an ideal ramp.

Note: when measuring low currents one need to be careful when determining the standard deviation of the mean. A minimum value is set by the Allen deviation.

Example of uncertainty budget of SP for the Keithley 6430 at a current of 95 fA:

Quantity	Value	Standard uncertainty	Distribution	Sensitivity coefficient	Uncertainty contribution	Degrees of freedom
I_r	$95.027 \cdot 10^{-15}$ A	$0.195 \cdot 10^{-15}$ A	normal	$10.5 \cdot 10^{12}$	$20 \cdot 10^{-4}$	7
I_L	0.0 A	$5.0 \cdot 10^{-18}$ A	rectangular	$10.5 \cdot 10^{12}$	$0.53 \cdot 10^{-4}$	100
C	$10.00000 \cdot 10^{-12}$ F	$0.14 \cdot 10^{-15}$ F	normal	$-100 \cdot 10^{9}$	$0.14 \cdot 10^{-4}$	100
dV_{cal}/dt	$9.5 \cdot 10^{-3}$ V/s	$25 \cdot 10^{-9}$ V/s	normal	-105 s/V	$0.03 \cdot 10^{-4}$	100
dV_d/dt	0.0 V/s	$100 \cdot 10^{-9}$ V/s	rectangular	-105 s/V	$0.1 \cdot 10^{-4}$	100
$\boldsymbol{Q}$	**1.0003**				$\mathbf{20 \cdot 10^{-4}}$	**7**

Example of uncertainty budget of SP for the Keithley 6430 at a current of 95 pA:

Quantity	Value	Standard uncertainty	Distribution	Sensitivity coefficient	Uncertainty contribution	Degrees of freedom
I_r	$95.02654 \cdot 10^{-12}$ A	$0.626 \cdot 10^{-15}$ A	normal	$10.5 \cdot 10^{9}$	$6.6 \cdot 10^{-6}$	7
I_L	0.0 A	$5.0 \cdot 10^{-18}$ A	rectangular	$10.5 \cdot 10^{9}$	$0.05 \cdot 10^{-6}$	100
C	$1.00000 \cdot 10^{-9}$ F	$0.14 \cdot 10^{-15}$ F	normal	$-1 \cdot 10^{9}$	$14 \cdot 10^{-6}$	100
dV_{cal}/dt	$95 \cdot 10^{-3}$ V/s	$0.25 \cdot 10^{-6}$ V/s	normal	-10.5 s/V	$2.6 \cdot 10^{-6}$	100
dV_d/dt	0.0 V/s	$1 \cdot 10^{-6}$ V/s	rectangular	-10.5 s/V	$10.5 \cdot 10^{-6}$	100
$\boldsymbol{Q}$	**1.000279**				$\mathbf{19 \cdot 10^{-6}}$	**159**