EUROMET supplementary comparison EUROMET.EM-S38

"Comparison of small current sources"

Technical protocol

Content

1 Introduction

In the last few years several European institutes have independently developed current sources, based on the idea of applying a voltage ramp to a capacitor. These current sources are already or will be used in the near future as standards for calibrating commercial picoammeters for internal use or for customers. The currents generated by these sources are typically in the range from 100 fA to 100 pA, with corresponding typical uncertainties from a few aA to a few fA. In order to support their calibration and measurement capabilities (CMCs), the participants aim to compare their standards by means of this supplementary comparison.

The procedures outlined in this document are intended to allow for a clear and unequivocal comparison of the measurement results and to show the equivalence of measuring results obtained with various systems for generating small currents in different national institutes. This technical protocol was prepared following the EUROMET Guide no. 3 "EUROMET Guidelines on Conducting Comparisons".

2 The travelling standards

Two commercial picoamperemeters will be used as transfer instruments:

- A Keithley 6430 Source Meter S/N 1036593
- A PTW Unidos E (modified prototype) S/N T10008 Y-80130

In the following text they will be called travelling standards. They will be shipped together in one package. Since they have very special input connectors, they will be accompanied by appropriate adapters with male BNC connectors.

3 Organisation

This bilateral comparison is a follow-up of the EURAMET.EM-S24 supplementary comparison. The TC chairman of the EUROMET EM Working Group will be regularly informed about the progress of this comparison.

3.1 Participants

The address of the co-ordinator of the pilot laboratory is:

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A list of all participating institutes and contact persons with their addresses is enclosed in Annex A1

3.2 Time schedule

The time schedule for the comparison is given in Annex A2.

The comparison will be organized in several loops. In the last tree loops the package will be accompanied by an ATA carnet or by special customs documents.

For the measurements in most laboratories a period of four weeks is scheduled, including time necessary for transportation. It is intended to re-measure the traveling standards before and after the loops in the pilot laboratory to establish a drift rate for the standards and to detect transport problems.

In agreeing with the proposed circulation time schedule, each participating laboratory confirms that it is capable to perform the measurements in the limited time period allocated in the time schedule. If, for some reasons, the measurement facility is not ready or custom clearance should take too much time, the laboratory has to contact immediately the co-ordinator in the pilot laboratory. According to the arrangement made in this special case the travelling standards may be sent directly to the next participant before the measurements have been finished or even without performing any measurements. In such a case there will still be possibility for carrying out measurements once again at the end of the comparison.

If delay occurs the pilot laboratory will inform the participants and revise - if necessary - the time schedule, or skip one country and put it at the end of the circulation.

If an ATA carnet or other customs documents are used, they must be used properly. Upon each movement of the package the person organising the transit must ensure that the carnet is presented to customs on leaving the country, and upon its arrival in the country of destination. When the package is sent unaccompanied the carnet must be included with the other forwarding documents so that the handling agent can obtain customs clearance. <u>In no case should the carnet be packed with the devices in the package</u>. In some cases it is possible to attach the carnet to the package. The carnet must be saved in the laboratory very carefully because a loss of the carnet may cause a serious delay in the comparison schedule.

3.3 Transportation

Transportation is on each laboratory's own responsibility and cost. The instruments will be shipped in an appropriate box. Shipping using courier services is accepted as an alternative to transportation by hand. The shipment should be arranged in a way that the time for transport is as short as possible, that is day to day courier service. This means that customs procedures, where appropriate, have to be examined in advance of the transport. Particular care should be taken to avoid the shipping case being exposed to extreme temperatures, e.g. left standing on the airport.

After arrival of the package, the pilot laboratory has to be informed of this by completing and returning a form (confirmation note of receipt, Annex A5) by e-mail or fax.

As already mentioned in section 3.2 in one loop the package will be accompanied by an ATA carnet to accelerate customs procedures. The value of the package is about 25.000,-- €

Immediately after having completed the measurements, the package is to be transported to the next participant. It is advisable to prepare and organise this transportation beforehand. Please, inform the pilot laboratory again about the details of sending the package to the next participant (use the dispatch note Annex A6) - and also inform the next participant by e-mail or Fax.

3.4 Unpacking, handling, packing

The package contain the following items:

Packing list:
- Keithley 6430 Source Meter S/N 1036593 without power cable
- Keithley 6430 Remote Preamplifier with cable
- Adapter: BNC male to Keithley 6430 input connector
- Operating Manual of Keithley 6430
- modified PTW Unidos E (prototype) S/N T10008 Y-80130 without power cable
- Adapter cable: BNC male to PTW Unidos E input connector
- Operating Manual for Unidos E
- RS-232 cable with male and female sub-D connectors (can be used for both instruments)

After the receipt of the package the box and the transfer instruments inside the box have to be inspected for any damage or dirt.

When the measurements have been finished ensure that the package is complete (see list above) before sending it in the original transportation box to the next participant.

3.5 Failure with a travelling standard

Should one of the travelling standards be damaged during the comparison the pilot laboratory must be informed immediately.

3.6 Financial aspects, insurance

Each participating laboratory covers the costs of the measurement, transportation and eventual customs formalities as well as for any damage that may have occurred within its country. The overall costs for the organisation of the comparison are covered by the organising pilot laboratory. The pilot laboratory has no insurance for any loss or damage of the travelling standards during transportation.

4 Measurement instructions

The measurements are 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 reading. The measurands are then the calibration factors of the transfer instruments, defined as the ratio of reading of the transfer instrument and supplied current.

The nominal values of the eight measuring points are +100 fA, -100 fA, +1 pA, -1 pA, +10 pA, -10 pA, + 100 pA, and -100 pA. In order to take full advantage of the transfer instruments' resolution and to avoid internal range switching the calibration points must be slightly below the nominal values. Therefore, the calibration points should be 0.95 times the nominal values, e. g. 95 fA, 0.95 pA, ... Only if for some technical reasons this might be impossible, the exact nominal values may be used.

Both instruments, the Keithley 6430 as well as the modified PTW Unidos E, can and should be operated remotely. Both instruments have RS-232 connectors, additionally the Keithley has a GP-IB connector. To become acquainted with the instrument, the instruction manuals will be supplied in advance in electronic form. In paper form, they will accompany the instruments.

After transportation a minimum settling and warm-up time of one day should be allowed for the instruments. The measurements should be carried out at a temperature of (23 ± 0.5) °C. Ambient temperature, pressure, and humidity should be recorded and reported.

The transfer instruments have considerable time constants. To take this into account, a settling time of 15 s after each current change must be allowed. Instructions specific for the instruments are given in the Annexes A3 and A4.

5   Uncertainty of measurement

Since this comparison is a Euromet supplementary comparison all participants must provide their results with the associated uncertainty of measurement and a complete uncertainty budget including the degrees of freedom (see Annex A8). The uncertainty must be evaluated at a level of <u>one</u> standard uncertainty. The uncertainty of measurement of the measuring results must be estimated according to the *ISO Guide to the Expression of Uncertainty in Measurement* (GUM). A model equation with all relevant quantities must be supplied. The evaluation of each uncertainty component has to be <u>detailed</u>.

For a simple example of an uncertainty budget see Annex A9. This example is only supplied to show which types of data are required. The example budget is not sufficiently detailed.

6   Measurement report

The individual results with date, temperature, air pressure, humidity, measurement current, measuring range of the instrument, readout, calibration factor, standard uncertainty, and degree of freedom will be reported to the pilot laboratory (please use the attached summary of results form, Annex A7). For each transfer standard and each nominal value a separate summary of results form has to be used.

For each nominal value the result (which may have been obtained by combining several measurements) has to be reported using one single line of sheet Annex A7. Only if a participant observes that for a nominal value the scatter of several independent measurements is incompatible with the uncertainty stated (maybe e.g. caused by drift or jumps of the transfer standard) then he should document this fact by using several lines in the summary of results sheet.

For each line used in the summary of results forms, a detailed evaluation of the uncertainty of measurement is required.

Furthermore, a short description of the measuring set-up used and the raw data are to be reported. The raw data may be supplied as a simple text-file, an excel-file or written on paper. The source of traceability has to be stated, since this may be a potential source of correlation. Preliminary results can be sent by email. In any case, a printed and signed report of the results must be sent by mail. In case of any differences, the paper forms are considered to be the valid version.

<u>The reports should be sent to the pilot laboratory no later than six weeks after the measurements have been completed</u>. No information about differences of the reported results with respect to others will be communicated before the completion of the comparison, unless very suspicious larger deviations of particular laboratories results and the preliminary reference results obtained by the pilot laboratory have been observed. In this case the laboratory in question will be contacted.

7   Report of the comparison

Within 3 months after completion of the circulation, the organisation group will prepare a first draft report (draft A) and send it to the participants for comments. In this report an overview about the different measuring systems and a proposed supplementary comparison reference value will be included. Reference values will be prepared according the procedures outlined in: M. G. Cox, "The evaluation of key comparison data", Metrologia, 2002, vol. 39, 589-595. It is planned that the participants discuss the draft A report at the "EUROMET meeting of experts on Quantum Metrology" in Espoo, Finland, in 2007. Subsequently, the procedure outlined in the Euromet Guidelines will be followed.

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Circulation Time Schedule (Preliminary) **Annex A2**

Supplementary Comparison EUROMET EM-S38

Institution	Country	Start date	End date
PTB (initial meas.)	Germany	2010-01-05	2010-02-05
SP	Sweden	2010-02-15	2010-03-15
PTB (final meas.)	Germany	2010-04-01	2010-05-01

The preamplifier is already connected to the instrument using the appropriate cable. The preamplifier's input connector to be used for the calibration is marked as "IN/OUT HIGH". The "SENSE" input is not used.

The instrument is designed as a source-meter. Therefore it has much more functions than needed for the comparison. Especially, it is able to generate a current flowing out of the terminal and to measure simultaneously the voltage at the terminal as well as it is able to generate a voltage at the terminal and to measure simultaneously the current flowing out of the terminal. In our case, both generator functions must be disabled and only the current measurement function enabled. Don't be confused by the fact that currents flowing into the instrument are displayed as negative currents and currents flowing out of the instrument are displayed as positive currents. This is due to its design as a source-meter: its point of view is the source inside the instrument. Nevertheless, with respect to the comparison currents flowing into the instrument are to be counted positive and currents flowing out of the instrument are to be counted negative. Therefore, the displayed current values must be multiplied by a factor of -1.

The instrument can be operated remotely via GP-IB interface or via RS-232 serial interface with the following parameters:

RS-232 parameters: 9600 Baud, 8 bits, no parity, 1 stop-bit

Many filter functions are available. To avoid correlation effects please use only the repeat filter which implements an arithmetic averaging algorithm which is equivalent to extending the integration time. To assure that all participants get comparable results the following commands (below in bold-face) must be sent to the instrument via GP-IB or RS-232 interface (Using these commands all filters except the repeat filter which may be adjusted by each participant are switched off):

```
*RST
:SYSTEM:CLEAR
:SYSTEM:PRESET
:SYSTEM:LFR 50                  ! Line frequency 50 Hz
!************************* Config Volt ********************************
:SOUR:FUNC VOLT
:SOUR:VOLT:MODE FIXED
:SOUR:VOLT:RANG MIN
:SOUR:VOLT:LEV 0
!************************* Config Current *****************************
:CONF:CURR
:FORM:ELEM CURR
:FUNC 'CURR'
!************************* Filter settings*****************************
```

```
 :AVER:AUTO OFF              ! Auto Filter off
 :AVER:REP:COUN 3            ! REPEAT FILTER: REP =3. PLEASE INSERT HERE
                            ! THE NUMBER OF YOUR CHOICE !
 :AVER:REP ON               ! REP ON
 :MED OFF                   ! MEDIAN FILTER OFF
 :AVER:ADV OFF              ! ADVANCED FILTER OFF
 :AVER OFF                  ! MOVING FILTER OFF
!*********************** Misc ***************************************
 :SENS:CURR:NPLC 10         ! meas. rate 10*0.02 s
 :SENS:CURR:RANGE 1E-12     ! meas. range for 1E pA and 100 fA
                            ! CHANGE THIS FOR THE HIGHER CURRENTS:  1E-11 and 1E-10
 :SYST:AZER:STAT ON         ! Auto Zero ON  (side effect: this doubles the measurement time!)
```

The data transfer to the computer is initiated by the command:

:READ?

The instrument will respond with a text string giving the measured current in ampere.

The instrument was designed for use as a dosimeter and then modified. Therefore it is has some more functions than needed for the comparison. It can measure electrical charges as well as electrical currents. In our case only the current range is used. The instrument may be programmed remotely using the RS-232 interface.

RS-232 parameters: 9600 Baud, 8 bits, no parity, 1 stop-bit.

As manual range setting is quite simple, only this method will be described here:

Key **[MOD]** :   toggles between the modes **Charge** and **Current**.

Key **[RGE]** :   toggles between the ranges **Low** (max. 112 pA),

                                                 Med (max. 12.5 nA)

                              and   **High** (max. 1.2µA).

Key **[NUL]** :   starts a zeroing procedure which takes 130 s.

For all current values the correct setting is: **"Current"** and **"Low"**.

After warming up, the zeroing procedure must be started.

The instrument has a built-in filter algorithm which can be adjusted only via the RS-232 interface by the undocumented parameter "CUA".
The setting after power-on is "CUA 10". This leads to the fact that successive measurements are strongly correlated. To circumvent this, the undocumented command

           CUA 1

must be sent to the instrument via the RS-232-interface.
The instrument will respond with **CUA 1** at the RS-232-interface and a "**1**" will appear at the front display (in the middle of the upper horizontal black line).
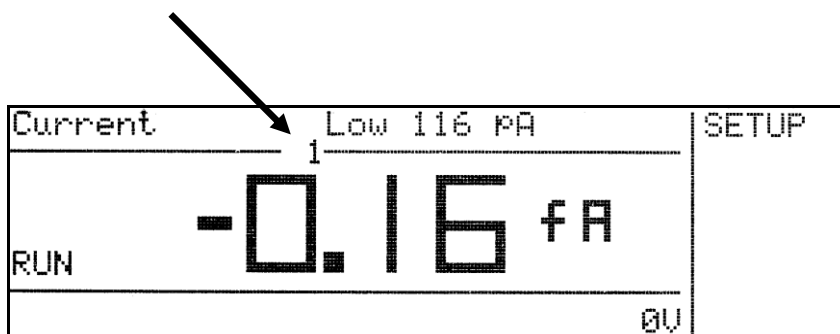
After this procedure, the correlation time is below 1 s.

The data transfer to the computer is initiated by the command:

D

The instrument will respond with a text string which is separated into 8 data fields by semicolons. The value in the 6th field is the measured current in ampere.

Euromet supplementary comparison receipt form   **Annex A5**

T e l e f a x   T e l e f a x   T e l e f a x

(Please pass on immediately!)

To   Physikalisch-Technische Bundesanstalt (PTB)

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          Post Box 3345   D-38023 Braunschweig   GERMANY

          FAX No. : +49 531 592 2105

e-mail: gerd-dietmar.willenberg@ptb.de

From: (participating laboratory):

Fax: International +

Pages (total): 1

In the case of faulty reproduction, please call:

Re: **Euromet supplementary comparison Euromet.EM-S38 - Receipt of travelling standards**

Date:

We confirm having received the travelling standards of the Euromet.EM-S24 supplementary comparison

on....................................

After visual inspection:

☐ No damage of the suitcase and the travelling standards has been noticed

☐ the following damage(s) must be reported(if possible add a picture):

....

....

Date: Signature:

Euromet supplementary comparison sending form **Annex A6**

T e l e f a x   T e l e f a x   T e l e f a x

(Please pass on immediately!)

To: Physikalisch-Technische Bundesanstalt (PTB)

Working Group 2.63   att.: Dr. Gerd-Dietmar Willenberg

Post Box 3345   D-38023 Braunschweig   GERMANY

FAX No. : +49 531 592 2105

e-mail: gerd-dietmar.willenberg@ptb.de

From: (participating laboratory):

Fax: International +

Pages (total): 1

In the case of faulty reproduction, please call:

Re: **Euromet supplementary comparison Euromet.EM-S38 - Sending off of travelling standards**

Date:

We have informed the next participant on.............................that we will send the travelling standards to them next time.

We confirm having sent the travelling standards of the Euromet.EM-S24 supplementary comparison on.................................to the next participant.

 Additional informations:

....

....

Date:.......................... Signature:...

Summary of results form (one sheet per standard and nominal current)   **Annex A7**

Standard: Nominal Current:

Date of measurement	Ambient temperature	Uncertainty of ambient temperature	Ambient pressure	Uncertainty of ambient pressure	Ambient humidity	Uncertainty of ambient humidity	Supplied Current	Transfer instrument's measuring range	Reading of transfer instrument	Ratio (Measure-ment result)	Standard uncertainty of measurement (combined type A and B)	Degree of freedom

Proposed scheme for an uncertainty budget

Quantity X_i	Estimate x_i	Standard uncertainty $u(x_i)$	Probability distribution / method of evaluation(A,B)	Sensitivity coefficient c_i	Uncertainty contribution u_i	Degree of freedom v_i
Calibration factor						*effective degree of freedom* $v_{\text{eff}} =$

The following uncertainty budget is fictitious. It is supplied here to shows which types data are required. The model used is not exhaustive.

The example budget is developed for the very simplest case that a picoamperemeter is calibrated using a voltage source with a known voltage $V = 1$ V and a resistor with known resistance $R = 1$ TΩ. The uncertainty components are exaggerated.

The result Q (ratio of readout and applied current) is calculated according to the model equation

$$Q = I \cdot k \cdot R / U.$$

The parameters used are:

I: the picoamperemeter's readout, the average is $1.01 \cdot 10^{-12}$ A.

k: a factor with nominal value of 1, its uncertainty describes the limited resolution of the digital display.

R: resistance of 1 TΩ.

U: voltage of 1 V.

The corresponding fictitious uncertainties are:

$u(I)$: the readout is averaged over 11 measurements. The standard uncertainty of the average is $u(I) = 1.5 \cdot 10^{-15}$ A,

$u(k)$: in the 1 pA-range a resolution of 3 ½ digits gives: $u(k) = 0.0005 / \sqrt{3} = 2.9 \cdot 10^{-4}$,

$u(R)$: the resistance is known within the limits of ±0.2%: $u(R) = 1 \cdot 10^{12} \Omega \cdot 0.002 / \sqrt{3} = 1.2 \cdot 10^{9}$ Ω,

$u(U)$: the voltage is known within the limits of ±0.1%: $u(U) = 1$ V $\cdot 0.001 / \sqrt{3} = 5.8 \cdot 10^{-4}$ V.

Uncertainty budget:

Quantity X_i	Estimate x_i	Standard uncertainty $u(x_i)$	Probability distribution / method of evaluation(A,B)	Sensitivity coefficient c_i	Uncertainty contribution u_i	Degree of freedom v_i
I	$1.01 \cdot 10^{-12}$ A	$1.5 \cdot 10^{-14}$ A	normal / A	$1.0 \cdot 10^{12}$ A^{-1}	$1.5 \cdot 10^{-3}$	10
k	1.0	$2.9 \cdot 10^{-4}$	rectangular / B	1.0	$2.9 \cdot 10^{-4}$	∞
R	$1.0 \cdot 10^{12}$ Ω	$1.2 \cdot 10^{9}$ Ω	rectangular / B	10^{-12} Ω^{-1}	$1.2 \cdot 10^{-3}$	∞
U	1.0 V	$5.8 \cdot 10^{-4}$ V	rectangular / B	-1 V^{-1}	$-5.8 \cdot 10^{-4}$	∞
Calibration factor Q	1.010				$2.0 \cdot 10^{-3}$	*effective degree of freedom* $v_{\mathrm{eff}} = 32$