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Quantum realisation of the SI ampere

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### 1 Overview

Single-electron sources that generate currents by transferring an exact number of electrons per cycle are the most direct methods for the realisation of the new revised ampere. Since they are particularly suited to generate small currents below 1 nA, these sources could enhance metrological capabilities for small current applications. This project developed semiconductor devices and instrumentation technologies which were required to implement direct and practical methods for the realisation of the new ampere, based on the fixed value of the elementary charge i.e. the magnitude of the electric charge carried by a single electron. The project developed high-precision measurement capability for ultra-small currents down to 1 fA and below, which is being used in many other areas of metrology and industrial applications, such as nuclear metrology, gas-particle metrology, semiconductor characterisation, and lighting manufacturing.

## 2 Need

Countless electrical measurements are performed as part of device and sensor operations in personal devices (smartphones, tablets, fitness monitors, etc.), medical devices, automotive technology, building management systems, manufacturing, environmental monitoring and security, etc. However, and despite the validity of these measurements relying on a consistent set of electrical standards, in the old International System of Units, SI, which was changed in May 2019, the SI ampere was not realised routinely in National Measurement Institute (NMI) laboratories. This was due to the technical difficulty and the experimental cost involved in its realisation. Instead, the chains of electrical calibration were served by the primary voltage and resistance standards that exploit two quantum effects, the Josephson effect and quantum Hall effect. The traceability to the SI units was maintained indirectly through the agreed values of two constants of proportionality that linked the quantum effects and the electrical units, the volt and the ohm. With the redefinition of the SI units in May 2019, the elementary charge and the Planck constant are fixed, which means volt and ohm can be directly realised by the two quantum effects. However, the ampere lacks a direct method for its realisation.

An electronic device that periodically transfers exactly one electron per cycle would produce an electric current that is equal to the operation frequency multiplied by the value of the elementary charge. Such a "single-electron" current source offers the possibility to directly realise the new ampere, and could serve as a primary standard for electric current. However, for this to become possible, the accuracy of electron transfer must be better than 1 part in 10<sup>7</sup> There are two main issues hampering this: i) accurate measurements are difficult to perform with the small electric currents (~100 pA) produced by single-electron sources are small so it is difficult to perform, and ii) the devices tend to produce higher counts of pump errors if the operation frequency is increased to achieve a larger current. In order to realise single-electron-based quantum current standards, the resolution of small-current measurement technologies must be at the attoampere (10<sup>-18</sup> A) level. Prior to the start of this project, the best resolution of NMIs' calibration and measurement capability was limited to several tens of atto-amperes.

For single-electron sources to be trusted as primary ampere standards, their accuracy of operations must be verified, which requires demonstrating their universality, robustness, and reproducibility. Additionally, strict guidelines on how to test/tune the devices need to be developed. Parts of these tests can be performed by comparing the current generated by single-electron sources to a current generated by a measurement system traceable to other quantum standards (by the Josephson and quantum Hall effects). In order to achieve the accuracy required as primary standards, the resolution of this traceable measurement system must be better than 10 aA (for a current of ~ 100 pA) or below, while the accuracy of traceability must be maintained at a level better than 1 part in 10<sup>7</sup>.

## 3 Objectives

The overall objective of this project was to develop a practical method for the realisation and dissemination of the future SI ampere using semiconductor-based single-electron current sources as primary standards and to develop novel types of portable reference sources as secondary standards.

This project addressed the following scientific and technical objectives:

1. To develop single-electron-based current sources reaching a current level of ≈1 nA with an uncertainty at, or below, 1 part in 10<sup>7</sup>. Parallelisation of single-electron sources will be developed and high-frequency



operation (above 1 GHz) investigated in order to extend the current level to ≈1 nA. The current quantisation accuracy will also be tested by the consortium's traceable small-current measurement systems.

- 2. To test the universality, robustness and reproducibility of single-electron-based current sources, which will be used for the practical realisation of the new SI ampere. Long-term stability and invariance in experimental parameters (e.g. RF amplitudes and magnetic fields) will be tested, and inter-device comparisons of current quantisation values performed, with a resolution better than 1 part in 10<sup>7</sup>. For this, single-electron sources fabricated both from the same material and from different materials (silicon and gallium arsenide) will be used.
- 3. To implement high-accuracy current measurement capability across NMIs, suitable for the testing of various types of single-electron devices. Multiple measurement systems, including ultra stable low-noise current amplifiers (ULCA) and null-detectors with calibrated resistors, will be developed and improved for traceable current measurements of single-electron sources (for use as a travelling standard). Multiple reference current sources that, in conjunction, cover a wide range from 1 fA to 10 mA will also be developed. These include a programmable quantum current generator (PQCG) directly realised by Josephson voltage standards and quantum-Hall resistance standards, as well as portable ultra-low DC current sources (ULCS) covering the range 1 fA 100 pA.
- 4. To develop guidelines for testing the accuracy of single-electron-based current standards. Towards a *mise en pratique* for the realisation of the new ampere, an agreed method for testing the current-quantisation accuracy will be developed and guidelines published for the verification of single-electron current sources.
- 5. To facilitate the take up of the technology and measurement infrastructure developed by the project by the measurement supply chain (accredited laboratories, instrumentation manufacturers) and end users (industries where small-current measurements at pico-ampere and femto-ampere levels are required). The consortium will demonstrate to the stakeholders, the calibration of their electrometers using the small-current measurement systems developed in the project and will give training on how to perform these calibrations, e.g. by inviting stakeholders to the partners' laboratories, or at stakeholders' premises using portable systems (the ULCA or ULCS). Some specific stakeholder needs, for example difficulties in making low-activity radiation measurements, have already been identified and will be addressed.

## 4 Results

The results of the project obtained towards each objective are described below.

## 4.1 Single-electron-based current sources reaching a current level of $\approx 1$ nA with an uncertainty at, or below, 1 part in $10^7$

Semiconductor single-electron sources (or single-electron pumps) transfer electrons one by one to produce a quantised current I = ef, where e is the elementary charge, which now takes a fixed value (1.602176634 × 10<sup>-19</sup> C) after the newly defined system of SI, and f is the frequency of electron transfer. Because the elementary charge (which is the magnitude of the charge of electrons) is small, the generated current by a single-electron source tends to be small. For example, with f = 1 GHz, the current produced is only  $I \approx 160$  pA (1 pA = 10<sup>-12</sup> A). There are two main problems with the smallness of the current. Firstly, it is difficult to perform a consistency test with the voltage and resistance standards (a so-called quantum metrological triangle), as resolving the current at the required uncertainty level (1 part in 10<sup>7</sup> or smaller) requires a long measurement time (typically 24 hours or longer) to average out the random noise. Also, if calibrated meters (such as transimpedance amplifiers) are used for the measurements of the current, then the stability of their calibration factors becomes an issue over such a timescale. Secondly, the small current limits the range of applications in which single-electron sources can be used. While this project has found increasing demand for traceable measurements in an ultra-small current range (100 pA and below), increasing the current level above 1 nA (= 10<sup>-9</sup> A) widens the area of applications (such as the calibration of high-value resistors). For this purpose, the consortium has set an objective for increasing the current level of single-electron sources with a target level of 1 nA.

One way to increase the generated current from single-electron sources would be to increase the operation frequency. Single-electron transfer at a repetition frequency approximately f = 6.24 GHz produces a current of 1 nA. However, there is an issue with simply increasing the device operation frequency. Errors of single-electron transfer (e.g. accidentally pumping extra electrons or no electrons in some cycles) tends to increase. It has been suggested that non-adiabatic effects or heating effects may be the source of the single-electron



transfer error under high-frequency operations, but the exact origin, which may vary between devices, is yet to be understood. At the time this project started, it was difficult to produce accurately quantised current beyond  $f \sim 500$  MHz. The smallest uncertainty achieved at f = 1 GHz was 1 part in 10<sup>6</sup>, which required a specially tailored waveform for pumping. Further enhancement in pumping accuracy required further optimisation in device structures and control parameters. Another way to increase pump current would be to operate a number of single-electron sources in parallel. A set of *N* parallel pumps operating at a frequency *f* would produce a total current *I* = *Nef*. 6 parallel pumps operating at 1 GHz would produce nearly 1 nA of current. There are two main challenges here. Firstly, despite the recent advances in semiconductor nanofabrication, achieving a high yield of working single-electron devices pose a challenge. A yield of 80 % (which is rather high) for each device, would result in a yield of 26 % (= 0.8<sup>6</sup>) for a set of 6 devices (assuming device failure mechanisms are not correlated between devices). Secondly, it is not enough that all devices in the set work at some level. They all need to operate accurately. A strategy of finding accurately-working devices out of a batch would not work here. A method of making all devices work accurately must be found in order to achieve accurate parallel pump operations.

Towards this objective, the consortium investigated two semiconductor nanodevice technologies, gallium arsenide (GaAs) based devices and silicon (Si) based devices. GaAs devices have advantages in the availability of high-mobility wafers and a long history of electron quantum transport study. The yield of singleelectron devices tends to be higher and the fabrication tends to require a shorter duration in comparison with Si devices. One disadvantage of GaAs devices is that they tend to require a low temperature T < 1 K and a high magnetic field (~ 10 T) for accurate operations. While the fabricate several thousand devices in one batch. This allows for design iterations with many varied parameters. Si devices also have an advantage in that it is possible to make the device dimensions smaller compared to GaAs devices. This in turn makes it possible to operate them at a higher temperature without magnetic fields. A disadvantage of Si devices is their yield. One failure out of the ~ 100 steps in fabrication process can ruin the whole batch of devices. We report the developments of our approach below.

#### (i) <u>Device fabrication and characterisation</u>

The project produced over 10,000 devices in total. The majority of these are Si devices due to their advantages in wafer-scale processing. Around 600 GaAs devices were fabricated through 30 design iterations by four partners, NPL, PTB, KRISS, and UCAM. The device designs by NPL, KRISS, and UCAM were based on gate-defined pumps, in which the confinement of electrons is created entirely by the electric fields from the surface gates. In PTB's devices, the side confinements were provided by wet etching to create a narrow channel. While NPL, PTB, and KRISS focused their effort on single-channel devices, UCAM worked on multiplexed and parallel devices. The multiplexer technology allows for a control of a number of devices with a limited number of control lines. UCAM created devices with 27 single-electron pumps on one chip that can be tested in one cool down. Strategies that enable parallel tests are very useful, because testing many cryogenic devices can be time-consuming and expensive as each one needs to be carefully cooled to its operating temperature and then warmed up. UCAM also fabricated devices with a set of 6 or 10 pumps in parallel. CEA and UoS fabricated Si devices. Around 2,000 devices were fabricated using electron-beam lithography, while over 10,000 devices were fabricated by industry-standard deep UV lithography process in 4 different fabrication runs. In addition, the consortium has gained a supply of around 30 Si devices by a collaborator (NTT: NTT Basic Research Laboratories, Japan) and a stakeholder (UNSW: University of New South Wales, Australia).

After fabrication, GaAs devices were tested at a liquid helium temperature of 4.2 K for initial characterisation. Si devices were tested at room temperature for basic transistor characteristics before low-temperature tests were conducted. More than 100 devices were tested at 4.2 K by NPL, PTB, KRISS, UCAM, UoS, CEA, and VTT, and Aalto. Over 20 devices have been selected and detailed tests of their current quantisation have been carried out at millikelvin temperatures and, in the case of GaAs devices, in strong magnetic fields. Following these characterisation results, design iterations (in some cases more than 3 iterations) were made to improve the device performance. Several devices were chosen to proceed to high-precision traceable measurements.

#### (ii) <u>Towards 1 nA current target</u>

The target current level of 1 nA was achieved by a Si device supplied by NTT. This particular device operated by transferring electrons through a trap state accidentally formed inside the entrance tunnel barrier. NPL, in collaboration with NTT, characterised this device and successfully increased the operation frequency up to 7.4 GHz, producing a current of 1.19 nA. The high-precision measurements performed at NPL showed that the generated current at 6.5 GHz was 2 parts in 10<sup>5</sup> smaller than the expected value of *ef.* It was suspected



that a finite rate to load the trap state leads to a loading error, causing a finite probability of missing an electron per cycle. Pump operations up to 2.5 GHz under a similar mechanism was achieved by Aalto in collaboration with UNSW. A disadvantage of these methods is that it is not yet possible to control the placement of the trap state. Roughly one in one hundred devices show such a pumping behaviour, limiting the availability of the device.

For the normal pumping mode (through a lithographically-defined quantum dot), the consortium has made a steady progress towards increasing the operation frequency. NPL and Aalto have operated Si pumps up to 2 GHz in collaboration with UNSW. KRISS studied the maximum frequency limit of pump operation up to 1 GHz. While more devices were made available through this project for high precision measurements in the GHz regime, the highest accuracy achieved so far is still limited to below 1 GHz.

Due to the difficulty in achieving high enough yield of accurate pumps, parallelising pumps to increase the generated current could not be achieved. However, the device fabrication method for parallel pumps established by UCAM should become applicable once the device design has been optimised for accurate pump operations in future.

#### (iii) <u>Towards the target uncertainty of 1 part in 10<sup>7</sup></u>

The lowest uncertainty of single-electron sources recorded with the target current level of 1 nA was 2 parts in 10<sup>5</sup>. This was achieved with a trap-based Si pump by NPL in collaboration with NTT. At the time the project was formulated, it was believed that a current level of at least 1 nA was necessary to achieve the target uncertainty of 1 part in 10<sup>7</sup>. However, the advances made by this project in the measurement of ultra-small currents, especially the development of the Ultrastable Low-noise Current Amplifiers (ULCAs) by PTB, has lowered the requirement for the current level to achieve our target uncertainty. PTB has achieved an uncertainty of 1.6 parts in 10<sup>7</sup> with a GaAs device operated at 600 MHz producing ~ 96 pA. This is the lowest uncertainty reported for single-electron sources so far. PTB developed a method for using two ULCAs to minimise the measurement integration time required to reduce the random measurement uncertainty. The voltage output readout of ULCAs were offset by a programmable Josephson voltage standard array to remove a requirement to calibrate the voltmeter to low uncertainty of 2.7 parts in 10<sup>7</sup> with a Si device operated at 1 GHz. Also, NPL, VTT and Aalto achieved an uncertainty level of 5 parts in 10<sup>7</sup> at 1 GHz with a Si device supplied by NTT. An important observation is that this device was operated at 4.2 K (and without magnetic fields), reducing the cost of cryogenics required.

#### (iv) Error correction and quantification protocols

NPL and PTB developed separate protocols to correct or quantify electron transport errors in GaAs devices. NPL's approach developed during this project removes unwanted electrons from the output of the pump using a precisely timed gate signal synchronised to the pump with a picosecond timing accuracy. This method directs one electron into an output in each pump cycle. The method has yielded current plateaus at 160 pA which are flat at a level of 4 parts in 10<sup>7</sup>, but offset from the expected current by 2 parts in 10<sup>6</sup>. It is not yet known whether the method can be refined to yield accuracy at the level of 1 part in 10<sup>7</sup>, but in principle it offers a route to correcting the errors from an electron pump driven to higher current levels.

At PTB, a protocol for in-situ validation by charge counting has been further developed. This employs a complex device-structure, which combines several electron pumps with charge detectors connected in series. It offers full counting statistics of the differential current between the pumps connected in series. This enables monitoring of the accumulated pump errors, provided they occur at a rate much slower than the inverse detector bandwidth. Generating 100 pA with in-situ validation seems feasible with available detector technology and may be achieved in the next 5 years. Increasing the current to 1 nA requires considerable improvement in the bandwidth of error detection.

#### **Conclusion**

While the two target numbers, 1 nA current level and 1 part in 10<sup>7</sup> target uncertainty could not be achieved simultaneously, the former was achieved, and an uncertainty level close to the target (only × 1.6 larger) was achieved in separate experiments. Also, in addition to GaAs device technology, the consortium has made Si device technology available for high-precision measurements. Even with the state-of-the-art device technology (1 GHz pump operation producing 160 pA) and ultra-small-current measurement technology (ULCAs) that this project has developed, it is still challenging to achieve the required uncertainty for the realisation of quantum current standards. However, the technology gap has considerably been narrowed by the collective effort of



consortium partners.

#### 4.2 Universality, robustness and reproducibility of single-electron-based current sources

Before single-electron sources can be trusted as primary standards for the new SI ampere in metrology laboratories, various tests including universality, robustness, and reproducibility need to be performed. Universality tests are required to demonstrate that the generated quantised current does not depend on individual devices and their material, or on the location and time when the measurements are made. Robustness tests are to test if the generated current is invariant under small fluctuations in realistic experimental environment, such as gate voltage noise or temperature fluctuations. Finally, reproducibility tests show that accurate current quantisation can be reproduced by multiple samples, and that the same device can be used repeatedly after thermal cycling (warmed up to the room temperature and cooled back down to a low temperature). These tests require collective efforts of many partners, and this project provided an ideal environment in which the leading experts in single-electron device technology and small-current measurement technology were involved as project partners and collaborators/stakeholders.

#### (i) <u>Universality tests</u>

The consortium partners NPL, PTB and Aalto published peer-reviewed journal papers on different singleelectron sources producing quantised currents equal to *ef* within measurement uncertainties of 1 part in 10<sup>6</sup> or less. Out of three, one device was based on GaAs technology fabricated by PTB, two devices were based on Si technology supplied by NTT and UNSW, one each. Prior to this project, there were four published results on the single-electron current quantisation with comparable measurement uncertainties. In these previous works, three devices were based on GaAs and one was based on Si. NPL, KRISS and Aalto have reviewed and analysed these seven published results and published the outcome of comparison in a review paper. This comparison showed that while the results are consistent with universality, more robust tests need to be introduced. They found that the methods used to assess the agreement of generated currents and *ef* varied slightly between studies, prompting the requirement for agreed guidelines to test single-electron sources.

A direct comparison between a GaAs device and a Si device was made by NPL. The GaAs device was fabricated by NPL, while the Si device was supplied by collaborator NTT. Both of these devices had previously been tested and verified for agreement with *ef* with a measurement uncertainty of 1 part in 10<sup>6</sup>. These devices were placed in separate cryostats and were operated simultaneously at a pumping frequency of 825 MHz. The difference between the currents generated by two devices was measured by a commercial ammeter. An agreement within 2 parts in 10<sup>6</sup> was achieved. Lower uncertainties should be possible, but in this experiment, there was unexpected time dependency of the current hampered further improvement. PTB performed a direct comparison of two GaAs pumps placed in the same cryostat. A variant of ULCA developed in this project was used to measure the difference of the currents produced by the two devices. An agreement of less than 1 part in 10<sup>6</sup> was found over a wide range of device parameters.

#### (ii) <u>Robustness tests</u>

During this project, the consortium performed four sets of studies on the robustness of the current quantisation by single-electron sources.

- PTB tested the robustness of a GaAs device fabricated in their laboratory. Two device control gates (that define entrance and exit barriers), source-drain bias voltage, externally applied magnetic field, and pump operation frequency were varied to see if the current quantisation condition was maintained. Uncertainties ranging from 3 parts in 10<sup>7</sup> to 5 parts in 10<sup>7</sup> were achieved in these tests.
- NPL and KRISS jointly tested the robustness of a GaAs device fabricated by KRISS. Three device control gates and the amplitude of the pump driving signal were varied for the test. Uncertainties ranging from 1 part in 10<sup>6</sup> to 2 parts in 10<sup>6</sup> were achieved in these tests.
- NPL performed robustness tests of a Si device supplied by NTT. The tests were made at a temperature of 4.2 K. Three device control gates and the pump driving signal were varied. Uncertainties of around 1 part in 10<sup>6</sup> were achieved.
- NPL tested the robustness of their own GaAs device using an error-correction protocol. Three control
  gates and timing of the error correcting gate was varied. Uncertainties of around 4 parts in 10<sup>7</sup> were
  achieved, but the plateau was offset by 2 parts in 10<sup>6</sup>.

There were other high-precision studies of single-electron sources made within this project, but their



robustness was tested to a less extent. NPL, KRISS, and Aalto published a review paper on the robustness tests of previous studies. The main conclusion was that much more rigorous investigations of robustness are required to demonstrate that current quantisation from single-electron sources can be trusted as primary current standards with an uncertainty level of 1 part in 10<sup>7</sup> or below. It is important to note that no single study has demonstrated robustness as a function of all the pump control parameters. Additionally, all the robustness studies to date have varied one control parameter at a time, with the other parameters fixed to their "optimal" settings. This only samples a small fraction of the multi-dimensional parameter space defined by the control parameters. In this project, VTT tested a randomised sampling of this parameter space, which may be the optimal way to perform a robustness test.

#### (iii) <u>Reproducibility tests</u>

The consortium has performed two types of reproducibility tests. One type was to verify that experimental results can be reproduced by multiple devices fabricated together (in the same batch) or by different batches from a repeated fabrication process. Another type was to test if the results can be reproduced from the same device after thermal cycling.

Multiple samples from the same batch or from a repeated fabrication process

- PTB performed high-precision measurements on the total of 13 GaAs-based single-electron sources. Seven devices were found to produce quantised currents that agreed with *ef* within a measurement uncertainty of 1 part in 10<sup>6</sup> or below.
- KRISS performed high-precision measurements on the total of 7 GaAs-based single-electron sources. Two devices have exhibited current quantisation that agreed with *ef* within a measurement uncertainty of 1 part in 10<sup>6</sup> or below.
- UCAM used their multiplexer technology to fabricate 27 nominally identical single-electron sources on one GaAs chip. They were tested at 4.2 K, and 21 of them were found to be working at some level and showed single-electron pumping behaviour. However, there was strong variability in the flatness of their current-quantisation plateau. No high-precision measurements were performed on these devices.

#### Repeated cool-downs of the same sample

- NPL performed measurements on one Si single-electron source supplied by a collaborator NTT in a total of 7 cool-downs from room temperature to 4.2 K spread over 7 months. The single-electron source was fabricated 18 years ago. High-precision measurements were performed during 3 cool-downs, and agreement with *ef* within a measurement uncertainty of 1 part in 10<sup>6</sup> was demonstrated. (High-precision measurements were not attempted in the other 4 cool-downs.) The lowest uncertainty achieved was 5 parts in 10<sup>7</sup>. This particular sample demonstrated an agreement with *ef* with a measurement uncertainty of 1 part in 10<sup>6</sup> at the temperature of 1.5 K in 2015, prior to this project.
- The same Si device mentioned above was transported to VTT and Aalto, where high-precision measurements were repeated with the device cooled down to 4.2 K. Both partners demonstrated agreements with *ef* within measurement uncertainties of 1 part in 10<sup>6</sup>. The overall characteristics of the sample was very similar to the behaviour observed at NPL, showing the advantage of NTT devices in the reproducibility between thermal cycling. Some small discrepancies discovered at the sub-ppm level are still the subject of analysis.
- In 2019, NPL cooled down one GaAs device that demonstrated agreement with *ef* with the measurement uncertainty at 1 part in 10<sup>6</sup> in 2011. The new measurements demonstrated a similar agreement with repeatable operating device-control parameters.
- PTB cooled down one GaAs single-electron source twice and demonstrated pumping accuracy better than 1 part in 10<sup>6</sup>.
- KRISS cooled down one GaAs single-electron source four times and demonstrated pumping accuracy better than 1 part in 10<sup>6</sup>.
- PTB demonstrated reproducible operations of one GaAs error-counting device in two cool-downs separated by 2 years.

Some general conclusions can be drawn regarding these reproducibility tests. Firstly, out of a fabricated batch of electron pumps, a small fraction, usually much less than half of the samples, demonstrate accurate pumping. The variation in pumping behaviour in nominally identical samples can be large, and this is a strong argument for developing sample architectures (such as the UCAM multiplexer) which allow high throughput testing of many devices. Secondly, pumps which demonstrated accurate pumping have also exhibited good stability and



robustness when tested on multiple cool-downs, in two cases over periods of several years. In particular, the Si device structure adopted by NTT showed remarkable reproducibility, where the device control parameters varied little between cool-downs. It also showed accurate pumping at a relatively high temperature of 4.2 K, and it survived transport between different labs. This device structure should be certainly considered for a candidate for future primary current standards.

#### **Conclusion**

The consortium has performed extensive tests regarding the universality, robustness, and reproducibility of single-electron sources. While the accuracy of current quantisation is still limited to above 1 part in 10<sup>7</sup>, the availability of devices that demonstrates accuracy at a level of 1 part in 10<sup>6</sup> or better has been improved by this project. The consortium has made both GaAs and Si devices available as accurate single-electron sources, which has made universality tests possible. The direct comparisons of two single-electron sources were also possible because of the improved availability. It is also worth mentioning that the successful circulation of a Si devices for high-precision measurements amongst three partner institutes marks an important step forward for single-electron sources to be used as practical primary current standards. On the other hand, the consortium's work highlighted that more rigorous tests would be needed to verify the accuracy of single-electron sources.

#### 4.3 Implementation of high-accuracy current measurement capability across NMIs

The need for accurate measurements of small currents (e.g. 1 nA and below) arises as industrial and scientific communities pursue higher sensitivity and traceability in sensing based on electrical measurements. The area of small current measurements is less well-developed in electrical metrology in comparison with well-established calibration chains in DC voltage and resistance based on quantum standards. The development of single-electron-based current standards also created a need for improved traceable measurement systems. On the other hand, if accurate single-electron sources can be found, then they can provide an ideal testing ground for the development of small-current measurement systems, as ideal single-electron sources are noiseless (the current output does not vary over time).

Here, the objective was to implement new reliable small-current measurement systems and reference current sources at partner NMIs. These facilities in conjunction cover a wide current range from 1 fA to 10 mA. They are capable of testing various types of single-electron devices with an uncertainty at, or below, 1 part in 10<sup>7</sup>, as well as being used for applications in other areas of metrology and industry. In this project three types of new instrument were developed; (i) Ultrastable Low-noise Current Amplifier (ULCA), (ii) Programmable Quantum Current Generator (PQCG), and (iii) Ultra-Low DC Current Source (ULCS). Here, we first describe this project's development of these new instrumentation. Then, in section (iv) the assessment performed on the calibration and measurement capability (CMC) in small current will be described for each partner NMI.

#### (i) Ultrastable Low-noise Current Amplifier (ULCA)

The ULCA is a transimpedance amplifier that maintains an excellent gain stability within a level of 1 part in 10<sup>6</sup> over an extended period (of the order of months). The first version, or the standard version, of ULCA was developed by PTB in the EMRP project SIB07 Qu-Ampere, and has been made commercially available by Magnicon GmbH. During this project (EMPIR 15SIB08 e-SI-Amp), the consortium tested the capability of the ULCA as a travelling standard, as well as implementing them in their laboratories to boost their capability of small current measurements. PTB also developed new variants of ULCA with different gain and stability settings, two of which are available by Magnicon.

#### Stability test of ULCA under travelling

The ULCA is a good candidate to be used as a travelling standard in future international comparisons in the area of small-current and high-impedance metrology. In international comparisons, travelling standards (such as a high-value resistor) are circulated amongst participating NMIs. Because the purpose of these comparisons is to test the calibration capability of different laboratories, a drift of calibration values of the standards must be minimised. This means that the standards must be robust to mechanical shocks during transport and must have long-term stability (the whole comparison process may take many months).

In order to test ULCA's suitability as a transport standard, the consortium performed various on-site smallcurrent comparison experiments. ULCAs were shipped from PTB to five partner NMI institutes (KRISS, LNE, NPL, TUBITAK, and VTT), as well as one non-partner NMI (METAS). At each partner's site, the ULCAs' current



gain of the input stage, transresistance of the output stage, and their total resistance were calibrated. In total, four ULCA units were circulated. Three were standard version with 3 G $\Omega$ /3 M $\Omega$  resistor network of the input stage. One was a special "high accuracy" version of ULCA developed by PTB in this project, with an 864 M $\Omega$ /864 k $\Omega$  input resistor network covering a larger current range (50 nA in normal operation mode) than the standard ULCA (5nA). (Here, "high accuracy" refers to the fact that the calibration uncertainty achievable is lower by a factor of two compared to the standard version.)

The comparison results show that for each transport, the current gain of the input stage and the transresistance of the output stage typically change by no more than a few parts in  $10^7$ . Typical changes of the total transresistance were less than 1 part in  $10^6$ . There was one exception when the total transresistance was shifted by 1.8 parts in  $10^6$ . This was when the ULCA was sent to South Korea during winter, and it was exposed to a very low temperature of -4 °C.

#### Stability test of ULCA over time

The long-term stability of ULCAs was evaluated at PTB by monitoring their calibration values long periods. Five standard version ULCAs were monitored over 4 years (including 1.5 years before the start of this project). Two new-variant "noise-optimised" ULCAs developed within this project were monitored for about 1 year, starting late 2017. All monitored ULCA units showed consistent behaviour. The annual drift of the input current gain was in the range ±1  $\mu$ A/A. The drift of output transresistance was negative for all units at -1  $\mu$ D/ $\Omega$  per year. The annual drift of the total transresistance was in the range + 0.5 and -2  $\mu$ D/ $\Omega$ .

According to these excellent stabilities, we recommend the ULCA as robust travelling standard for future international comparisons in small current metrology.

#### (ii) Programmable Quantum Current Generator (PQCG)

In this project, LNE developed a programmable quantum current generator (PQCG). The PQCG provides a quantised current by a combination of Josephson voltage standard and quantum Hall resistance standard. This provides an alternative route to a realisation of the new SI ampere. The PQCG is more suitable in a larger current range, typically 1  $\mu$ A up to 10 mA. While the PQCG achieves uncertainties as low as 1 part in 10<sup>8</sup> in this range, the uncertainty at the lower current range increases above that of single-electron sources. This is partly because the PQCG requires an external (non-quantised) current source to supply the current to the load, adding noise to the generated current. These two technologies complement the shortfall of each other and will cover a wide range of current levels as primary standards.

LNE demonstrated operation of the PQCG at a current level between 0.7 mA and 2.2 mA, achieving the target uncertainty of 1 part in 10<sup>8</sup>. In the primary part of the PQCG circuit, a quantised current is generated by applying a quantised voltage by programmable Josephson voltage standard across a GaAs quantum Hall bar. While the size of the current is known, this current cannot be directly applied to a load, as the impedance of the load would change the series resistance of the circuit, and the current would deviate from the quantised condition. Instead, the current is fed into an arm of a cryogenic current comparator (CCC). The current through the other arm of the CCC is supplied by an external battery-powered low-noise current source. Servo-control of the output current is scaled exactly by the winding ratio of the CCC. LNE's result improved the current traceability in the mA range by two orders of magnitude. The uncertainty of PQCG in the lower current range was also evaluated. It is expected that the PQCG can maintain the uncertainty 1 part in 10<sup>8</sup> down to 1  $\mu$ A, below which it will rise to 1 part in 10<sup>6</sup> at 10 nA.

LNE then extended the current range of PQCG down to 50 nA. LNE and PTB made comparison studies of PQCG and ULCA by feeding the current from a PQCG to a ULCA. The "high-accuracy" version of new ULCA developed in this project was used in the "normal" mode at 50 nA, and in the "extended" mode at 50  $\mu$ A.

#### Measurement with the ULCA in the "extended" mode at 50 µA

ULCA's "extended" mode uses only the second stage of transimpedance gain with a nominal resistance of 100 k $\Omega$ . The measurements yielded a relative deviation (between the two current measurements) of 4 parts in 10<sup>7</sup> with a combined standard uncertainty of 3 parts in 10<sup>7</sup>. Here, the ULCA current value was estimated between two calibrations performed at PTB. We concluded that no significant deviation was measured within the expanded uncertainties (*k* = 2).



#### Measurement with the ULCA in the "normal" mode at 50 nA

For the measurements performed at 50 nA, the ULCA was operated under the normal mode, using both stages. At this current level, the stability of PQCG is reduced as the operation becomes very sensitive to noise, but it was possible to find a stable configuration. The measurement yielded a relative deviation of 1.4 parts in  $10^6$  with a combined standard uncertainty of 6.5 parts in  $10^7$ . The deviation was slightly larger than the expanded uncertainties (k = 2).

The performance of the PQCG also relies on the quantisation of quantum Hall and Josephson effects. Their verification would allow the confirmation of the quantisation of the generated current with the required uncertainty.

#### (iii) Ultra-Low DC Current Source (ULCS)

TUBITAK developed an Ultra Low DC Current Source (ULCS) in this project. The ULCS generates a current by applying a linear voltage ramp to capacitors. Four capacitors are kept in a temperature-controlled box. A relay card is used for automatic selection of capacitors, depending on the current range required. The operation of the ULCS is fully automated by computer control.

As part of the verification of the ULCS operation, this was compared to a standard version of ULCA supplied by PTB. The measurement was performed at ULCS current output levels of 1 pA, 10 pA, and 100 pA. Each measurement period was configured with four sections: hold (zero current), positive ramp, hold (zero current), and negative ramp. For each ramp section, the current values from 40% to 90% were evaluated. The current outside this section was discarded for current stabilisation. The agreement of the two currents within an uncertainty of 2-3 parts in 10<sup>5</sup> was found. The expanded uncertainty (k = 2) of the ULCS at the current level between 1 fA and 10 fA was evaluated as 2.5 parts in 10<sup>3</sup>, achieving the target uncertainty of 3 parts in 10<sup>3</sup>.

#### (iv) Calibration and Measurement Capability Assessment at partner NMIs

Following the development of small-current measurement capability at partner NMIs, the Calibration and Measurement Capability (CMC) for small current measurements, and high-impedance calibration in some cases, at each institute was re-evaluated. Below is the summary of activities at each partner.

#### NPL

Since 2002, NPL has maintained routine traceability of decade-value standard resistors up to 1 G $\Omega$  using a custom-built CCC bridge. The CMC-declared relative standard uncertainty for 1 G $\Omega$  is 8 parts in 10<sup>7</sup>, and this has been a dominant contribution to the total uncertainty of small current measurements, such as those generated by single-electron sources. During this project, the uncertainty in calibration for 1 G $\Omega$  was re-evaluated. The hardware configuration has not been changed, but many tests were performed to understand the different components of uncertainty. The new uncertainty to be used in updated CMC declarations is 1 part in 10<sup>7</sup>. The uncertainty for 100 M $\Omega$  was also improved from 2 parts in 10<sup>7</sup> to 1 part in 10<sup>7</sup>. The improved uncertainty allowed an improvement of uncertainties in single-electron source measurements to 2.7 parts in 10<sup>7</sup> in this project. An unexpected and important finding from this work, is that commonly-used high-value standard resistors based on thick-film elements have drift with a 1/*f* character, on time-scales of hours to days. In practice, this limits the relative uncertainty achievable using this type of resistor to a few parts in 10<sup>7</sup> even if lower calibration uncertainties are available.

Another key activity for NPL towards this objective was a bilateral transfer of an ULCA unit with PTB. This was performed twice during this project. NPL has gained a capability to calibrate ULCA's input current gain and output transresistance gain, by adapting a CCC for towards this operation. The comparison study showed that the stability of ULCA at less than 2 parts in  $10^7$ . Also, during this ULCA transfer, NPL tested calibration of a 1 G $\Omega$  resistor with the ULCA. Agreement within 1 to 2 parts in  $10^7$  was found in comparison with CCC calibration. As a result of these activities, NPL bought a ULCA, which is now used for measurements of single-electron sources and other small-current activities.

#### <u>LNE</u>

Based on the knowledge gained in this project, LNE wrote a first draft of technical guidelines for calibrations based on the PQCG. This is a first step towards a declaration of new CMC in the area of current.



#### <u> PTB</u>

In order to test the potential of the ULCA as a reference device for future CMC determination in the area of small currents, PTB performed "triangle" comparisons involving two standard ULCAs. One ULCA was used as a current source and the other as the device under test (DUT) as an ammeter. The results showed improved uncertainties throughout the current range from 1 fA up to 1  $\mu$ A over the existing CMCs by PTB. PTB is currently working towards applying for improved small-current CMCs. PTB has also applied for a CMC entry for the CCC-based calibration of the ULCA for the BIPM KCDB in August 2017.

#### <u>TUBITAK</u>

The ULCS developed by TUBITAK in this project is being used as a reference current source for the EURAMET Project 1381 "Supplementary Comparison for Ultra-Low DC Current Sources". The comparison measurements are still on-going. After this comparison, TUBITAK plans to apply for new CMCs with extended current ranges and reduced uncertainty levels. The direct current range for the CMCs of TUBITAK will be extended from 2 pA down to 1 fA with considerably reduced uncertainties. For example, the uncertainty in the range 1 pA to 10 pA will improve from 1 part in 10<sup>2</sup> to 2 parts in 10<sup>4</sup>, and a new range 1 fA to 10 fA will be declared with an uncertainty of 4 parts in 10<sup>3</sup>.

#### VTT

VTT assessed potential CMC updates in the following three categories: ULCA calibrations, high resistances, and small currents.

VTT now performs ULCA calibrations routinely. Following the uncertainty updates and bilateral ULCA comparisons, VTT plans to declare a CMC of 5 parts in 10<sup>8</sup> for the input stage and 1 part in 10<sup>7</sup> for the output stage. There is a possibility to improve the uncertainty for output stage calibrations further. Currently, the output stage calibration is performed with a Magnicon CCC against stable secondary resistance standards. It is possible to use the primary quantum-Hall resistance standard, instead, for reduced uncertainty, although this option would be an expensive operation due to liquid helium and labour costs.

As part of this project, VTT has calibrated the national Finnish standards of resistances between 1 G $\Omega$  and 100 T $\Omega$  by using an ULCA. (We note that their official calibration certificates have not yet been finalised.) This ULCA method has dramatically shortened the calibration chain. Previously the high value resistors had to be calibrated decade by decade from a 1 M $\Omega$  resistor, which was directly calibrated against the quantum Hall resistance standard. VTT plans engage with the calibration customers to discuss their specific calibration needs and potential interests in improved calibrations. In principle, the ULCA would allow improving VTT's CMC values.

Two activities that VTT performed within this project yielded significant improvements in the measurement uncertainty of small currents. A stakeholder's ammeter was calibrated in ultra-small current range 0.1 fA to 1 pA, with attention was particularly paid on currents below 10 fA. In collaboration with NPL and NTT, VTT achieved an expanded uncertainty (k = 2) of 7 parts in 10<sup>7</sup> for the measurement of a Si single-electron source with a current level of 168 pA. Despite these improvements, measurements data for currents between 1 pA and 5  $\mu$ A are still scarce. CMC updates will be considered once enough data are accumulated through the regular internal calibrations in this range.

#### <u>KRISS</u>

The high resistances and low current CMCs of KRISS for the year 2017 are currently under review. For example, the relative expanded uncertainty (k = 2) for the calibration of a 1 M $\Omega$  fixed resistance with the 12-bit CCC bridge of KRISS is 1.2 parts in 10<sup>7</sup>. The relative expanded uncertainty (k = 2) for current calibrations with the "standard" ULCA of KRISS at 10 pA, 100 pA and 1 nA are 7 parts in 10<sup>5</sup>, 1.5 parts in 10<sup>5</sup>, and 7 parts in 10<sup>5</sup>, respectively.

#### **Conclusion**

The dissemination of measurement techniques based on the ULCA has resulted in a major improvement to small current measurement capability in European NMIs. Three partners (KRISS, VTT and NPL) have permanently adopted ULCA-based measurement capability, achieving measurement uncertainties significantly smaller than their capability before the project. Two partners, LNE and TUBITAK, used the ULCA to validate their measurement capability. Finally, PTB, the developer of the ULCA, has accumulated a large body of data demonstrating that the ULCA offers transportation stability at the level of 1 part in 10<sup>6</sup> or better,



and enables measurement uncertainties over a wide range of small currents that are at least a factor 10 more accurate than the best uncertainties available before the project.

## 4.4 Development of guidelines for testing the accuracy of single-electron-based current standards

Single-electron sources are usually characterized by classical current measurements, i.e. neglecting charge quantization and treating electric current as a classical variable. The state-of-the-art technology does not yet guarantee perfect charge quantisation, therefore current quantisation is not guaranteed. For this reason, presently-available single-electron sources are less accurate than quantum Hall resistance and Josephson voltage standards. One way to test the accuracy of single-electron sources is to perform current measurements traceable to the quantum Hall resistance and Josephson voltage standards. However, in this approach, single-electron sources approach that of quantum Hall or Josephson standard, the method of performing traceable current measurements becomes no longer valid in testing the accuracy. For single-electron sources to be used as primary standards, a method to tune the device to guarantee the generation of a quantised current without performing traceable measurements is required.

Under the present circumstances, researchers have been adopting different methods to claim the accuracy of their single-electron sources. A direct comparison of different experiments is often difficult. For this reason, clear and agreed guidelines for testing the accuracy of single-electron sources need to be developed. There are two types of guidelines that are required: (i) guidelines for testing the accuracy of single-electron sources in the context of research and development, and (ii) guidelines for using single-electron sources as primary current standards. In the first type, the device under test is not assumed to be accurate. The guidelines provide recommendation for procedures of tests that need to be performed on the device to assess its accuracy. Unified methods will make the comparison of different experiments easier and are expected to accelerate the future development of accurate single-electron sources. In the second type of guidelines, it is assumed that the device under test can produce accurately quantised current. The guidelines provide instructions on the tests that need to be performed to tune the device and guarantee accurate single-electron-transfer operations. This is in analogy to the guidelines for realising the SI ohm using quantum-Hall resistance standards. Since we do not have yet accurate single-electron sources that can be used as primary current standards, and we do not know yet what type of device architecture would yield accurate devices, it is not yet possible to formulate the second type of guidelines. Therefore, the consortium focused their effort on developing the first type of guidelines as detailed below. VTT led to produce these guidelines with contribution from NPL, Aalto, KRISS, and PTB.

#### Traceable current measurement techniques

In this project, the consortium has gained considerably expanded amount of experimental data on highprecision traceable measurements of the current generated by single-electron sources. Two measurement techniques were used for this purpose: (i) a current null detection between the pump current, a reference current source constructed by a traceable voltage applied across a traceable high-value resistor, and (ii) using ULCA's traceable transresistance to convert the current generated by a single-electron source to a voltage, which is measured with either a calibrated voltmeter or in a null measurement against a programmable Josephson voltage standard. While the experimental sets are different, conceptually they are equivalent in that the current from the single-electron source is measured traceably to the quantum Hall resistance standard and the Josephson voltage standard.

#### Eliminating drift of offset

In common with practically all measurement systems, both of these two methods have an offset that is far less stable than the transresistance gain (in the case of (i), this is the value of the high-value resistor, in the case of (ii), this is the total transresistance of the ULCA). Therefore, it is necessary to eliminate a drift of the offset either by periodically measuring the zero current in an "on-off" cycle or by reversing the polarity of single-electron current in a "forward-reverse" cycle. While it is in principle possible to reverse the polarity of single-electron current by changing the direction of pumping, it cannot be guaranteed that the same magnitude of the current is produced in the forward and reverse directions. Instead, PTB developed an alternative method towards the end of this project. They developed a novel polarity reversal technique based on cryogenic switches. In their setup, the single-electron source runs continuously in one direction, but the connection to the measurement instrument is reversed by the switches. This polarity reversal method is preferable in terms



of minimising the measurement time (the current is effectively twice larger). But most of published highprecision measurements so far have adopted "on-off" method.

#### Eliminating leakage currents

Semiconductor single-electron sources require application of gate voltages typically of the order of 1 V. In GaAs devices, gates are directly placed on the surface of the substrate and a Schottky barrier is formed. In the case of Si devices, a thin layer of silicon dioxide barrier is formed between gates and substrate. Gate leakage current flowing from the gates through these barriers into current contacts will offset the measured current. There can also be a leakage current between experimental wiring and sample packaging contacts. Typical resistance of combined leakage paths is  $10^{15} - 10^{16} \Omega$ , resulting in the leakage currents in the range 0.1 fA – 1 fA. This is roughly 1 – 10 parts in  $10^6$  of the pump current. The leakage currents can be subtracted by "on-off" cycle measurements if they do not change by turning on/off the pump current. In high-precision measurements of single-electron sources published so far, on-off cycles are usually implemented by turning on and off the AC drive signal applied to the pump entrance gate. However, the recent measurements on Si devices performed by Aalto, NPL, and VTT in this project suggest that the leakage current through the entrance gate changes over time, possible due to heating of the gate. An alternative method was developed to perform on-off" cycles by switching the exit gate voltage, keeping the AC pumping signal on. In this protocol, the change in the leakage current is typically a few parts in 10<sup>7</sup> of the pump current. This must be measured regularly, and a correction needs to be applied. Ideally, another method that does not require application of corrections, i.e. the leakage current does not change by on-off cycles, is desirable. PTB's forward-reverse cycle method mentioned above keeps the pump in a fixed state. There can be leakage current from the cryogenic switches, but these can be assessed independently of the pump device. However, the gate leakage from the pump gates into source and drain contacts can be different, and they cannot be cancelled by polarity switch. Nevertheless, this configuration allows for detailed study to identify the source of leakage currents. We note that the issue of leakage current is also present in the error-counting devices. We also note that these are preliminary conclusions and will be subject to further analysis.

#### Stability of instrumentation

In a typical measurement setup, the output voltage of ULCA, or the voltage applied across a high-value resistor in the case of a null measurement, is measured directly with a calibrated precision voltmeter. The voltmeter needs to be calibrated much more frequently than the ULCA, making the experiment impractical. In order to avoid this, PTB developed a method to use programmable Josephson voltage standards to almost null the ULCA output voltage. Only a small voltage difference needs to be measured, and therefore the stability of the voltmeter at a level of 1 part in 10<sup>7</sup> is not required. In this measurement setup, the ULCA is expected to be the only measurement device that requires a calibration that pauses the traceable measurements. The stability of ULCA is a complex issue. The test performed by PTB under this project shows that several ULCA units exhibited gain stability at a level of few parts in 10<sup>7</sup> on the time scale of one year. Single-electron-source measurements require stability better than one part in 10<sup>7</sup> on the time scale of one day. Further investigation is required to understand the ULCA's stability to meet this stability requirement.

#### Methodology

In this section, we discuss the methodology for testing the accuracy of single-electron sources using the traceable measurement technique described above. After the basic characterisation (e.g. to see if the device produces any quantised current plateaux) has been completed, the first high-precision test should be to demonstrate the robustness, i.e. to show that there is a regime in multi-dimensional space of control parameters where the output current is constant and independent of all control parameters. In other words, the current plateau must be flat within the target measurement uncertainty level. The robustness, or the flatness of the plateau, does not guarantee that the accuracy of the output current (whether it agrees with *ef*), but testing the accuracy of single-electron sources is meaningless unless the robustness is demonstrated (otherwise the current level changes if control parameters are varied).

#### Assessment of plateau flatness

During this project, the consortium found that the most reliable method in assessing the flatness (or robustness) of current quantisation plateau is to plot the absolute value of the deviation of the current  $l_p$  from the plateau value on the logarithmic scale, i.e.  $\log |l_p / ef - 1|$  (in the case of perfectly quantised plateau at *ef*), as a function of a control parameter. An exponential curve is usually a good approximation to the approach



of the current towards the plateau value. Therefore, on the log plot, the deviation of the current shows a linear behaviour. This linear behaviour stops when the deviation approaches measurement uncertainty, showing scatter of points mostly concentrated around the value of measurement uncertainty (type A). If there is an extended region where the data points scatter around the measurement uncertainty, we can conclude that the plateau is flat (or the pump current is robust) at the level of measurement uncertainty. If there is no flat region, the current deviation starts to deviate from the linear behaviour before reaching the value of measurement uncertainty, and then starts to increase again, showing a V-shape behaviour, as the sign of the current deviation reverses.

#### Random sampling in multi-dimensional parameter space

The method described above is effective if the parameter scan can be completed within the timescales for drift of measurement system calibration. However, single-electron sources have multiple control parameters, and it is usually not possible to complete measurements for the entire parameter space with sufficient resolutions within the drift-free period of the measurement system. If the data are taken in a sequential order, the time-correlation of neighbouring data induces a spurious space correlation, which could be misinterpreted as a parameter dependence to the pump current. In order to tackle such issue, VTT developed a random-order sampling method. In this method, a grid is defined in the parameter space (it can be of any number of parameters). A point in the grid is randomly selected and a measurement is performed. Random selection and measurements are repeated until the whole grid is covered, and ideally until each grid position is measured multiple times. It was found that, even without compensating for the drift of measurement zero offset by on-off or polarity reversal cycles, it was possible to study the flatness of plateau at a level of few parts in 10<sup>7</sup>.

#### Thermal cycling

The reproducibility of accurate single-electron transfer is not guaranteed after the device is warmed up to the room temperature and cooled back again. During this project, while the Si device supplied by NTT was found to maintain the accuracy at a level of 4 parts in 10<sup>7</sup> after 9 times of thermal cycling, a GaAs device showed high accuracy only 50% of times after multiple thermal cycling during an earlier part of the project when the experimental setup was less reliable. Therefore, devices should be tested for multiple thermal cycling to demonstrate the reproducibility.

#### **Conclusion**

The consortium developed guidelines for testing the accuracy of single-electron sources for the research and development purposes. Information on these guidelines are published in two papers. A third paper is being prepared for submission. We note that these guidelines are based on the present knowledge of state-of-the-art technologies. As the technology develops in future, we expect that modifications and improvement will be required. There is another type of guidelines that need to be developed in future. The guidelines for using single-electron sources as primary standards for the new SI ampere need to wait until accurate single-electron sources have been developed.

## 4.5 To facilitate the take up of the technology and measurement infrastructure developed by the project by the measurement supply chain

The ultra-small current measurement technologies developed by this project found applications across multiple sectors including healthcare, environmental monitoring, semiconductor fabrication and the lighting industry. As the project progressed, the consortium made contacts with stakeholders who were looking for solutions to their problems in practical measurements of small electrical currents. In some cases, the work was done under confidentiality agreement, and so it is not possible to describe the details here, but some examples of the activities performed by partners LNE, NPL, PTB, and VTT are listed below.

#### (i) LNE

LNE performed direct calibrations of multiple commercial ammeters with the Programmable Quantum Current Generator in the current range 1 µA to 10 mA. Highlights are listed below.

• Calibrations of a commercial digital ammeter on the ranges from 10 mA down to 1  $\mu$ A have been demonstrated. Relative uncertainties of ~ 2 parts in 10<sup>7</sup> have been achieved for measurements at the



top of all ranges. These measurements demonstrate better accuracies than those reported in the manufacturer specifications.

- In June 2018, another commercial ammeter was calibrated using the PQCG on the 2-mA range. The results of this calibration agreed with those of the usual calibration performed by the "Low Frequency" calibration department of LNE. This calibration was demonstrated to the chief corporate metrologist of the manufacturer.
- The European regional technical manager of a major commercial calibration service company attended a project meeting to witness the discussion of the project results. A plan was made to demonstrate the calibration of a digital ammeter to the representatives by the company.

#### (ii) NPL

NPL undertook a number of activities disseminating small current measurement expertise to stakeholders, both in other measurement departments within NPL and also externally.

#### Ionising radiation measurement (internal within NPL)

- An extensive collaboration was undertaken with the NPL Medical Radiation Physics Group. This group performs high-throughput calibrations of the activity of radioactive sources using ionisation chambers, mostly for the medical sector. The ionisation chamber current is presently measured with custom-made integrating electrometers, which are more than 30 years old and in need of replacement. Some of the small-current measurement expertise developed within this project was applied to investigating the most fit-for-purpose solution for replacing the old electrometers. The investigation also clarified some general aspects of ion chamber current readout such as the relationship between measurement time and uncertainty, and the excess noise contributed by the high-voltage supply. The results of the investigation were published in a peer-reviewed journal.
- A smaller collaboration was undertaken with another NPL group, the Medical Radiation Science Group. This group also performs calibrations of radioactive sources and operates a large number of ionisation chambers with associated readout electrometers. An ULCA was used to calibrate a transportable reference current standard and monitor its stability. The very good stability of the ULCA allowed the verification of the stability of the transportable reference at the 10-ppm level.
- Preliminary test measurements were undertaken to investigate the feasibility of an accurate, direct
  measurement of the half-life of carbon-14, used for the dating of historical artefacts. This possibility is
  enabled by the very good gain stability of the ULCA, introduced to the radiation measurement community
  during the project. At the project end date, a small feasibility study (partly funded by the UK National
  Measurement System) is underway at the low-background underground facility in Boulby, UK.

#### Nanoparticle concentration measurement (internal within NPL)

• Measuring the concentration of particulates in air is a growth area of metrology, driven by environmental legislation. The NPL Nanoparticles Group maintains a reference electrometer which is used to calibrate customer's instruments. The reference electrometer has been calibrated annually since 2012, at currents in the range 0 pA to 1 pA. Prior to this project, the calibrations were quite disruptive as they required the electrometer to be moved to the electrical laboratory for a period of several days. During the project, an ULCA (initially on loan from PTB, but since 2018 NPL's own ULCA unit) was taken to the nanoparticles lab to calibrate the electrometer in situ, and the calibration time was reduced to 12 hours. This is of obvious benefit to the Nanoparticles Group, who have recently bought a second reference electrometer and expanded their range of services.

#### Evaluation of standard resistors (external companies)

The improved accuracy in making high resistance measurements, developed as part of the project, revealed that commercially available high resistance standards are not stable at uncertainty levels much below 1 part in 10<sup>6</sup>. One manufacturer of standard resistors supplied a sample of their 1 GΩ resistor to NPL for evaluation of its stability. The results were communicated to the company and presented at the 2018 CPEM conference. In common with resistors from other suppliers, their resistor exhibited instability at the level of a few parts in 10<sup>7</sup>, which is below the uncertainty level at which most customers operate. The company was happy with the result, as a quantitative verification that their product meets most of



their customer's needs. NPL also had discussions with another manufacturer of standard resistors about the stability of their resistors which NPL maintains as internal reference standards.

#### Measurement of ion beam current (external company and university)

The promise offered by quantum information processing (QIP) is driving development of single-ion implantation techniques for the fabrication of precisely engineered nanostructures. A UK company had installed a single-ion implantation machine at the site of a UK university, but did not have confidence in their measurement of the ion-beam current. This current is in the range 10 fA – 100 fA. NPL performed a traceable measurement of the ion beam current in situ at a university site. The result gave the company confidence that their measurement of the current, although noisy, was correct.

#### Probe-station characterisation of semiconductors (external company)

 Internet-of-things (IOT) applications are driving the development of low-power semiconductor electronics. NPL performed characterisation of Peta-Ohm (10<sup>15</sup> Ohm)-level leakage resistances on test chips for one semiconductor company using a probe station. This required measurement of fA-level currents. The traceable (ULCA-based) calibration of the measuring instruments, and the methodology for analysing the raw data, were developed in this project.

#### Measurement of resistivity (external companies)

NPL undertook two investigations of resistivity for commercial customers during this project. One of
them was concerned with the electrical insulation of test samples. The other was interested in using
surface resistivity as a probe of surface treatments of composite materials for aircraft engines. Both
investigations employed a commercial resistivity cell and readout instrument, but the traceable
calibration of the instrument on its low-current ranges was performed using ULCA-based traceability
techniques developed in this project.

#### (iii) PTB

PTB undertook several activities for the dissemination and demonstration of small-current/high-resistance calibration and measurement expertise, mainly centred around the application of measurement techniques and setups enabled by the ULCA, including special ULCA versions developed within the project. These activities were involving stakeholders from instrumentation manufacturer industry, industrial calibration laboratories, national metrology institutes and the BIPM.

Interactions with instrumentation manufacturer industry and industrial calibration laboratories

- PTB is in contact with a major commercial calibration service company in Germany, communicating and consulting on the application of the ULCA for small-current and high-resistance calibrations. This company now owns an ULCA and is among Europe's leading metrology laboratories with core competency in industrial calibration for test and measurement equipment.
- Communication and consultation occurred with the central laboratory for light measurements of a major lighting manufacture on the application of the ULCA for the calibration of small-current meters for traceable photometric and radiometric light measurements. The accredited reference laboratory owned an ULCA until 2018.
- Experts from instrumentation fabrication companies and accredited calibration service providers have visited PTB. They requested and received information on the ULCA and on related calibration and measurement techniques. These companies include a calibration laboratory and developer of dosimetry instrumentation and ULCA owner since 2018, and a developer of aerosol measurement instrumentation.

#### Interactions with other national metrology institutes, inside PTB, and with BIPM

- During a site visit of PTB delegates at METAS, training on the ULCA and related measurement techniques was given to METAS staff. This work was undertaken in the frame of EURAMET TC-EM project 1409 "Calibration strategies of a dual-stage transimpedance amplifier (ULCA) with 1 GΩ nominal overall transresistance".
- During a site visit of a NIST delegate at PTB, training on the application of the ULCA for small-current



and high-resistance measurements was provided by PTB. NIST owns and uses several ULCA units.

- A three-months site visit of a PTB expert at KRISS was dedicated to training KRISS staff on the CCCbased calibration of the ULCA, and on the application of the ULCA for single-electron-pump measurements.
- On occasion of several visits at PTB, technical and scientific staff from INRiM (ULCA owner and user) received training on the CCC-based calibration of the ULCA and on ULCA-related measurement techniques. This included a working visit of a PhD student from INRiM over a period of 10 months at PTB.
- Remote communication with NMIJ/AIST (ULCA owner) enabled detailed discussion of the ULCA and related calibration and measurement techniques.
- During a guest working period of several weeks at PTB, a scientific staff member from MSL (NMI in New Zealand) received training on the CCC-based calibration of the ULCA and on ULCA-related measurement techniques.
- PTB department "Direct Current and Low Frequency" represents PTB's calibration and measurement capabilities (CMC) in the fields of (small) direct current and (high) resistance calibrations and provides the corresponding calibration services offered by PTB according to the KCDB of the BIPM. The department owns and uses several ULCA units and currently works towards providing calibration and measurement services in the fields of small currents/high resistances based on the ULCA. This has been continuously supported by consultation of ULCA experts/developers from the PTB departments "Electrical Quantum Metrology" and "Cryosensors".
- PTB department "Radioactivity" provides the realization of the legal unit Becquerel and its dissemination, achieved by the provision of activity standards or by the calibration of radioactive sources. Research institutes, industrial companies as well as measurement laboratories for monitoring of radioactivity in the environment benefit from these services. The work also focuses on radionuclides being used for therapy and diagnostics in medicine. The measurement techniques involve ionisation chambers combined with picoammeters measuring the small ionisation currents, which are subject to continuous improvements. During this project, ULCA experts from the PTB departments "Electrical Quantum Metrology" and "Cryosensors" supported the establishment of the ULCA for small-current metrology on ionisation chambers and provide general expertise and training on small-current measurement techniques in the department "Radioactivity" of PTB.
- Regarding the future application of ULCA-based measurement and calibration techniques for radionuclide metrology, PTB currently is in contact with the department "Ionising Radiation" of BIPM, preparing first on-site test measurements at BIPM and providing general expertise and training on small-current measurement techniques.

#### ULCA workshop (March 2019)

The workshop "Ultrastable Low-Noise Current Amplifier (ULCA) in Laboratory Practice" was held at PTB on March 20/21, 2019. Sixteen participants from national metrology institutes (NIST (USA), INRiM (IT), NPL (UK), KRISS (KR), TUBITAK (TR)), the BIPM, as well as from five German industrial calibration laboratories and manufacturers of small-current instrumentation attended the workshop. By practical demonstrations and lectures the workshop participants were made familiar with the most important application aspects of the ULCA for small current/high-resistance measurements and calibrations.

#### (iv) VTT

VTT, working with PTB, has assessed and improved the small-currents measurement technique and the traceability of the Single Charge Aerosol Reference (SCAR) located at Tampere University of Technology (TUT), a stakeholder group member of this project.

Aerosol particles are considered to play an important role in global climate change. Fine and ultrafine particles have also found to cause severe effects on human health. Regulation and control of aerosols requires robust techniques for monitoring and quantifying particle concentrations. One way of measuring aerosol concentration in a gas sample is to charge up the particles and detect the resulting electrical current when they are accumulated in a 'Faraday cup' electrode. If the particle charging is accurately controlled, the electric current represents precise information on the particle concentration and can be used as an accurate reference to calibrate the sensitivity of other instrument types.



TUT have developed SCAR as a novel reference standard to calibrate aerosol measurement devices. Its traceability is based on verifying aerosol charging and flow division with traceable current readout in the femtoampere (10<sup>-15</sup> A) range. Measurements of such small currents is difficult, so that at low aerosol concentrations the uncertainty of the SCAR is dominated by the noise of the current measurement and the calibration uncertainty of the instrumentation involved.

As part of this project, VTT demonstrated the use of noise-optimized version of ULCA in the SCAR setup. Noise measurements and the demonstrating the Allan deviation as an analysis tool helped to identify and suppress triboelectric noise in SCAR's Faraday cups. Furthermore, VTT used the standard ULCA for an improved calibration of the electrometer that TUT normally uses in the SCAR setup. Because of the significant input voltage and voltage noise of this electrometer, the calibration was performed indirectly by using the ULCA for improved calibrations of VTT's high-value resistance standards ranging between 1 T $\Omega$  and 100 T $\Omega$  at low voltages. A calibrated voltage source and these resistors were then used to calibrate the electrometer. This work demonstrated a significant improvement compared to calibration results before this project has started.

#### Conclusion

The consortium partners have performed a number of activities to disseminate the ultra-small current measurement capability developed in this project. A wide area of small-current metrology was covered by these activities ranging from internal NMI groups to the commercial calibration service providers, lighting manufactures, semiconductor device manufactures, and so on. The extent to which these activities were made was limited by the amount of available time that could be spent to identify stakeholders' needs in small current measurements. It is very likely that there are many other areas to which the small-current technology developed by this project can provide practical solutions.

### 5 Impact

The consortium has disseminated the results of the project through different routes. 27 papers have been published in peer-reviewed journals. In addition, 8 articles were published in trade/popular press and 64 conference presentations and posters have been given. 14 in-depth training sessions were organised and held to train stakeholders in small-current measurement techniques. In addition, the partners have been engaging in a significant number of activities to promote the uptake of the project's outputs and improve the small-current measurement capability of our stakeholders. In order to provide the stakeholders and the general public information on the importance of small-current metrology, a short video has been created and is available on website (http://www.e-si-amp.eu/) the project and also on YouTube (https://www.youtube.com/watch?v=5WQP0yv-IM4). The Helmholtz Prize 2018, considered the most prestigious award in the field of metrology, in the category "Application" was awarded to a partner NMI for ground-breaking improvements in the field of high-precision traceable measurement and generation of small electrical currents. A contribution from a partner NMI won "the Best Paper of the Conference" award at a major conference for the test and measurement industry (NCSLI, August 2017) and a consortium's paper on the robustness of single-electron pumps at sub-ppm current accuracy level was selected by the journal editors for the category "Highlights of 2017". One paper on the new variant of ULCA was selected as one of the most read editors' highlights of 2017 in the Review of Scientific Instruments. Another paper published in the same journal, studying the noise optimised version of ULCA, was selected as an editor's pick. A paper on the electron counting capacitance standard was selected for "2017 Highlights of Metrologia".

#### Impact on industrial and other user communities

The manufacturers of instruments that require small current measurements, such as particle counters or radiation dose meters, have benefited from the availability of high accuracy portable current measurement/source systems. The uncertainties of electrical units that propagate through the calibration chains have been improved, and therefore more accurate electrical units have become available to end users. Training and demonstration courses have been held for the stakeholders to exploit the enhanced small-current measurement facilities at partner NMIs and portable current measurement/source systems.

A partner has agreed with a manufacturer to commercialise two versions of the ULCA variants developed in this project. Also, after the distribution of ULCAs within the consortium, the partners are exploiting their improved small-current measurement capability to engage with stakeholders in a wide range of areas. These activities resulted in an improvement to the traceability of ionisation chambers for medical radiation metrology



and nuclear metrology, and also an improvement to the traceability of single-charge aerosol references that are used in the area of health and environment. We have tested the stability of a high-value resistor for its manufacturer. We demonstrated PQCG's capability to a multinational calibration and asset management supplier and made a plan for calibrations of their instruments and later demonstrated a calibration of their multimeter in the presence of the manufacturer's chief corporate metrologist. Also, we have started a new measurement service performing electrical current measurements on prototype semiconductor devices from external customers. Our work with a manufacturer of bespoke cryogenic components and electronics has resulted in a sample enclosure, which is now commercially available from the manufacturer. We demonstrated a calibration of a multimeter using the PQCG in the presence of the manufacturer's chief corporate metrologist. We worked with a large lighting manufacturer to implement a ULCA for optical calibration measurements in their reference laboratory.

#### Impact on the metrology and scientific communities

The enhanced capability of small-current measurements is expected to have a direct impact on the Calibration and Measurement Capabilities (CMC) of NMIs. Up to one order of magnitude improvement in the range 1 pA – 100 pA has been achieved. In addition, high-resistance (e.g. 100 M $\Omega$  and 1 G $\Omega$ ) calibration has been improved. For scientific communities, the development of single-electron devices will benefit the development of nano-device fabrication technology and the understanding of mesoscopic device physics. The training and demonstration courses described in the "industrial and other user communities" section have been also provided to the metrology and scientific communities.

Four non-partner NMIs have been trained for ULCA usage. Six guest students/scientists from outside the consortium received in-depth training on small-current measurements and electron-pump experiments. We have re-evaluated the uncertainty of the resistance calibration chain to 1 GΩ. A partner NMI had a discussion with a university laboratory working on quantum-limited measurements on ways to control vibration-induced noise in cryogenic systems. Another partner NMI has applied for a CMC entry for the ULCA calibration using cryogenic current comparators. Several international NMIs in Europe, Asia, and America are already using the measurement and calibration capabilities offered by the commercially available ULCA for improved processes or services. Three partners (LNE, VTT and NPL) are preparing to revise CMCs in the high-value resistance and small-value currents following the development of small-current measurement capabilities using ULCA, PQCG, ULCS, and the standard resistor with improved traceability. An international small current comparison (TC Project EM-1380) is taking place between two e-SI-Amp project partners and 5 other European NMIs. A new EMPIR project 17FUN04 SEQUOIA has started, which exploits some of the single-electron device technologies that this project has developed, for applications in quantum information and quantum sensing.

#### Impact on relevant standards

The progress and results of this project have been reported to European and international committees on electrical measurements such as the EURAMET Technical Committee for Electricity and Magnetism (TC-EM), and Consultative Committee for Electricity and Magnetism Working Groups (CCEM WGs) on Low-Frequency Quantities and on Proposed Modification to the SI.

The consortium has a member of the working group of the CCEM on implementing the revised SI (WGSI), and the project had good visibility in the most relevant standards committees. Two members of partner institutes have given presentations to the CCEM committee, and five partners presented work at two TC-EM DC QM subcommittee meetings. The outcomes of this project will have an impact on international standardisation of electrical units. Improved accuracy and scalability will pave the way for the use of single-electron current sources as primary current standards.

#### Longer-term economic, social and environmental impacts

In the long term, the improvements in small-current measurement capability will have high impact on society and the environment. The sensitivity of sensors used in environmental or health monitoring, and manufacturing relies on the detection of a small current signal. We have demonstrated that the technology developed in this project equips metrological and industrial laboratories with higher sensitivities in fields such as ionising radiation, particle counting, optical measurements, and material characterisation. The areas covered are likely to expand in the future. The effect of these improvements will eventually permeate into wider society, where more efficient and sensitive devices will allow for more accurate and precise control in products and services, such as environmental protection, medical care, and commercial production of goods.



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