Current Status of Quantum Hall System Implantation at the Inmetro

V C de Oliveira, H R Carvalho and R P Landim
Instituto Nacional de Metrologia, Qualidade e Tecnologia – Inmetro, Brazil

E-mail: vcoutinho@inmetro.gov.br

Abstract. This paper presents the activities currently being performed at the Inmetro with a view to obtaining the international recognition of its quantum Hall system as the primary standard of electrical resistance in Brazil. To enable it, the Inmetro is making arrangements to take part in the ongoing key comparison BIPM.EM-K12, which is an on-site comparison of quantum Hall effect resistance standards. Besides, as a prerequisite for joining in this key comparison, Inmetro is participating in the ongoing key comparison BIPM.EM-K13.a&b.

Keywords: key comparison; primary standard; electrical resistance; quantum Hall system.

1. Introduction
Since the installation of its primary resistance measurement system based on the quantum Hall effect (QHE) to the present day, Inmetro has been undertaking the necessary efforts to confidently operate its quantum Hall system (QHS). Inmetro aims at achieving the international recognition of the electrical resistance primary standardization based on the QHS and inserting this part of the scope into the Calibration and Measurement Capabilities (CMC) listed in the Appendix C of the Mutual Recognition Arrangement of the International Committee for Weights and Measures (CIPM) – nominated CIPM MRA [1].

The CIPM MRA objectives are to establish the degree of equivalence of national measurement standards maintained by National Metrology Institutes (NMIs); to provide for the mutual recognition of calibration and measurement certificates issued by NMIs; and to provide governments and other parties with a secure technical foundation for agreements related to international trade, commerce and regulatory affairs [2]. The process through which the CIPM MRA achieves these objectives involves two complementary paths: (a) technical requirements and (b) quality management system (QMS) requirements. Technical requirements are international interlaboratory comparison programs (named key comparisons), regional comparisons (named regional key comparisons) or other regional or bilateral comparison modalities (referred to as supplementary comparisons), where evidence of the proficiency of the participating NMI is provided; QMS requirements are related to the implementation and review of quality systems and demonstrations of competence by NMIs [2].

The line of conduct set out by Inmetro for reaching the desired goals regarding the recognition of its QHS involves the participation in the key comparison BIPM.EM-K12 (“quantum Hall resistance standards and their scaling to other resistance values”) [3]. BIPM.EM-K12 is run on-site in order to eliminate the limitation of transporting transfer standards between the Bureau International des Poids...
et Mesures (BIPM) and the participating NMI, which would lead to an increase of the comparison uncertainty. To this end, the BIPM has developed a transportable system that can be operated at the participant’s facilities to reproduce the ohm from a QHE reference at 100 Ω and also to scale this value to 1 Ω and 10 kΩ [4].

Looking toward to enhancing operation, procedures, and the measurement results provided by its QHS, Inmetro is carrying out several activities. For instance, Inmetro is currently participating in the key comparison BIPM.EM-K13.a&b (“Bi-lateral comparison of Electrical Resistance Standards using 1 Ω and 10 kΩ Resistors as Travelling Standards”) [5] [6]. Moreover, a technical mission to BIPM took place in September, 2018 and, as a result of it, various verifications and refinements are being performed. Additionally, the Quantum Electrical Metrology Laboratory (Lameq) was peer reviewed in 2019, and quantum Hall dc resistance quantity was included in the scope of assessment (albeit not yet required); such a strategy showed to be beneficial, since it produced a series of observations and improvement recommendations to be carefully followed. The objective of this paper is to discuss these activities as well as the preparatory measures for the BIPM.EM-K12.

2. Description of the Inmetro Quantum Hall System

Let us first shortly describe the Inmetro QHS. The system consists of two main modules: a cryostat for QHE measurement and a cryogenic current comparator (CCC) bridge.

2.1. The cryostat

The cryostat, manufactured by Cryogenic Ltd., contains a probe to which is connected the quantum Hall device, a superconductive coil, and an auxiliary reservoir to store 3He gas. The quantum Hall device is a semiconductor sample composed of a GaAs/GaAlAs heterostructure, which is immersed in very low temperature liquid Hel (≈0.3 K when in 3He, or ≈0.8 K when in 4He) to allow the realization of the QHE. The superconductive coil enables the application of a magnetic field of up to 16 T to the quantum Hall device; QHE can be obtained on the $i = 2$ plateau for magnetic fields ranging from 9 T to 11 T, depending on the sample. Yet, the value of resistance on the $i = 2$ plateau does not depend on the sample. Figure 1 (left-hand side) shows the entire cryostat module.

It is important to note that cooling down the cryostat is no trivial operation. The cryogenic gases are costly, and specialized manpower is required. From the vacuuming to the cooling down and magnetic field application stages, the process demands a few weeks [7].

![Figure 1. The Inmetro cryostat module for QHE measurement (left) and the CCC module (right)](image)

2.2. The CCC bridge

The CCC is made by the National Physical Laboratory (NPL), UK [8]. It has a dewar with a probe, to which the CCC type II toroid [9] and a superconductive quantum interference device (SQUID) are
fixed. The probe must be immersed in a liquid $^4$He bath (~4.2 K) to let the superconductive state occur. The CCC also has a piece of electronics formed of four main units: two isolated dc current sources, named “master” and “slave”, a nanovoltmeter module (which includes an auxiliary current source named “balance”), and the control of the SQUID. A computer with specific software manages these units, reads the signals from the nanovoltmeter and the SQUID, adjusts the current values of the balance and slave sources appropriately (by means of digital servo controls) for balancing the CCC bridge, and gathers the data from the measurements. Figure 1 (right-hand side) shows the CCC module.

The CCC enables the calibration of the Lameq 100 $\Omega$-reference standards against the QHE device with an uncertainty of the order of 10 parts in $10^8$. The 100 $\Omega$-reference standards are compared to other resistors, also through the CCC on a scale, thus composing Lameq traceability chain. The temperature of the resistors is carefully controlled by means of air and oil baths.

3. **Brief history of the QHS at the Inmetro**

To facilitate understanding of the history of implantation of the QHS at the Inmetro, let us split the timeline into three major phases. “Phase I” starts with the purchase of the QHS in 2005; system components were delivered in 2007, and final assembly, commissioning and initial tests occurred in April, 2008 [7]. Until December, 2012, the system was powered on 10 times [10]. As previously explained, QHS operation is complex, which allows running the system no more than 3 times a year. During “Phase I”, the goal was to gain experience in operating the QHS, which involved performing measurements with the CCC and mastering cryogenic techniques.

“Phase II” starts in 2013, when the initial conversations with BIPM took place, after BIPM had modernized its transportable QHS; such conversations aimed at arranging the participation of Inmetro in the key comparison BIPM.EM-K12 in order to validate its implementation of the QHE for dc resistance traceability by comparison to the reference maintained at the BIPM. By 2013, the Inmetro had already carried out an unofficial comparison, taking by reference the results of a calibration performed by the BIPM in 2012 for a 10 k$\Omega$-reference resistor. The relative difference between the value obtained by the Lameq and the one provided by BIPM was 9.0 parts in $10^9$ [11].

In 2015, BIPM issued a questionnaire to the members of the Consultative Committee for Electricity and Magnetism (CCEM) to inquire their interest in participating in the on-site QHR comparison, to which Inmetro responded positively. Unfortunately, the comparison originally expected to occur in November, 2015 had to be delayed due to a series of circumstances, such as change of personnel in both Inmetro and BIPM. Meanwhile, Inmetro continued to investigate the minutiae of the system (for instance, the aging effects of quantum Hall samples [12]), explore possibilities of usage, and perform other informal comparisons, with small relative differences [13]. During 2017, the system underwent eventual maintenance.

“Phase III” starts in 2018, when the prospect of the realization of BIPM.EM-K12 was resumed. More specifically, the practical arrangements for BIPM.EM-K12, in cooperation with BIPM, started at April, 2018, when Inmetro was considered for inclusion in the BIPM.EM-K12 comparison schedule. As a prerequisite for joining in the on-site key comparison BIPM.EM-K12, Inmetro is participating in the key comparison BIPM.EM-K13.a&b. A technical mission sent to BIPM in 2018 is among the preparation activities. Additional details of these key comparisons and the technical mission, as well as the consequential actions, are presented in the following section of this paper.

4. **Current activities**

With a view to achieving the international recognition of its QHS as the primary standard of electrical resistance in Brazil and drawing up this part of the scope into the CMC of CIPM MRA, Inmetro has been carrying out several activities, as described below, to refine procedures and system operation, as well as to validate/improve the measurement results provided by its QHS.

4.1. **On-going key comparison BIPM.EM-K13.a&b**
For this comparison, the BIPM is providing two CSIRO type, 1 Ω-reference resistors and two TEGAM S104 type, 10 kΩ-reference resistors. The comparison is being carried out with a “BIPM – Inmetro – BIPM” pattern of measurements. The standards were to be calibrated first at the BIPM, then at the Inmetro, and again at the BIPM throughout 2020, but delays occurred due to the Coronavirus disease (COVID-19) pandemic. Measured resistance values provided by Inmetro to BIPM shall not be corrected for temperature or pressure, in compliance with BIPM.EM-K13.a&b technical protocol (TP) [14]. The final results of the comparisons are presented in the form of degrees of equivalence, that is, deviations between the Inmetro and the BIPM for values assigned to the resistance standards, and the associated uncertainties. The results from the Inmetro and the BIPM agree if the difference is within the relative expanded uncertainty.

4.2. Informal comparison of a 100 Ω resistor calibrated against QHS

Apart from the key comparison BIPM.EM-K13.a&b, BIPM and Inmetro agreed to exceptionally carry out an informal comparison of a 100 Ω-reference resistor provided by the Inmetro (WIKA/Tinsley model 5658A, serial number 3300/01, nicknamed “7E”). The 100 Ω-reference resistor was directly calibrated against both quantum Hall systems, following an “Inmetro – BIPM – Inmetro” pattern of measurements, during the second half of 2018.

Since it was an unofficial calibration, BIPM issued a study note instead of a certificate. Thorough details of this comparison are planned to be discussed in a future work.

4.3. Technical mission to BIPM in September, 2018

A technical mission to the Physical Metrology Department of BIPM took place in 2018. Some of the objectives of this mission were to clarify the BIPM scientists about the attributes of the Inmetro QHS, to know BIPM’s quantum Hall systems, and to discuss details about the BIPM.EM-K12 TP.

4.3.1. Attributes of the Inmetro CCC: discussions, studies to be conducted. Crucial information on how Lameq operates its CCC was exchanged. The consistency measurements the CCC can provide (available in a paper written by the very own designers of the system [8]) were presented to the BIPM colleagues. Not every resistance ratio can be realized in a direct fashion due to the system’s construction; so also the least value resistor should always be connected to the “master” source branch. It was explained that the top current this CCC can provide is 30 mA [8] – as opposed to higher currents that the BIPM uses –, and that it is not possible to apply any current of choice, for it depends on the resistance ratio and the number of turns of the coils, which is within a limited set (short-circuit, 1 turn, 2, 4, 8, 16, 32, 80, 320, 800, 1600, and 2065 turns). As a result from this discussion, Inmetro is going to conduct further studies on the possibility of usage of higher currents for some ratios, as well as power coefficient compensation.

For the measurements with the CCC in the Lameq, 10 cycles (five forward and five reverse current cycles) are typically run. Each cycle consists of a 20 s guard interval, \( t_g \) (transition time + wait time), plus a 20 s interval valid for the measurements, \( t_s \). Figure 2 (left-hand side) depicts one forward and one reverse cycle. The transition time between forward and reverse modes is very short because it happens with the servo controls off. On the other hand, in the BIPM 8 cycles are run with a 100 s time interval valid for the measurements. Furthermore, the BIPM system needs a 36 s wait time, in which system currents are set to zero, to switch between modes, as illustrated in Figure 2 (right-hand side). Therefore, Inmetro is going to conduct studies to look into any possible effect that the interval duration may have on the measurement results. Preliminary data from \( t_s = 100 \) s measurements in the Lameq presented no significant influence on the type A uncertainty whatsoever.
4.3.2. Preparatory measures for the BIPM.EM-K12. The on-site key comparison pricing is based on a shared costs model; thus, the participant NMI is responsible for the accommodation costs of BIPM staff and for half of the shipment costs of the transportable system. It is pivotal for Inmetro to foresee it for the upcoming yearly budget, although the precise date of the BIPM.EM-K12 is still to be confirmed.

BIPM’s transportable QHS – comprised by a transportable Hall cryostat, plus a current comparator bridge operating at 1 Hz [4], which is simpler to operate than a CCC – is completely independent from the participating NMI’s facilities. For BIPM.EM-K12, Inmetro must only be prepared to provide liquid N₂, liquid He and gaseous He for the cryostat, besides some adapters for the transfer line.

4.4. Resulting actions from the peer reviews in May and June, 2019

Concerning the technical requirements for the CIPM MRA, Lameq was peer reviewed in May, 2019, and quantum Hall dc resistance quantity was included in the scope of assessment – notwithstanding the fact that this part of the scope is not yet in the CMC – with the intention to benefit from the external look of the reviewer in order to identify opportunities for improvement. Such a work produced a series of observations and recommendations to be carefully followed, e.g. adjusting the value of Lameq resistance standards for the von Klitzing constant valid from May 20th, 2019, \( R_K = 25\, 812.807\, 459\, 3045\, \Omega \) (with 15 digits, as described in the CCEM guidelines [15]) and auditing the computer programs and spreadsheets that use \( R_K \) for calculations accordingly, as well as documenting in more detail some operational procedures. Considering \( R_K \) updated value, the calibration values of the resistance standards shall be numerically corrected forthwith in accordance with equation (1):

\[
V_n = V_p (1 + 17.79 \times 10^{-9})
\]

(1)

where \( V_n \) is the numerically corrected calibration value and \( V_p \) is the previous calibration value for the resistance standard.

Another recommendation is to implement CCC consistency checks for metrological confirmation. Ways of implementing these consistency checks include (but are not limited to): (a) running 100 Ohm to 10 kOhm calibrations directly (using the CCC 1:100 ratio) and indirectly (with two 1:10 CCC ratios, from 100 Ohm to 1 kOhm, then 1 kOhm to 10 kOhm), and comparing both 10 kOhm measurement results; (b) performing a ladder check, where firstly resistor R2 is calibrated against resistor R1 (initial reference value of R1), then R1 is calibrated against R2 (the reference value to obtain this final value of R1 is the newly measured value of R2) and, finally, both initial and final values of R1 are compared; (c) carrying out a triangle consistency check with 3 (or more) resistances standards. The multiple values compared
should agree within the uncertainty of the measurement. It is advisable that Inmetro publishes the findings of these checks.

As for the QMS requirements, Inmetro has a policy to conduct yearly internal audits in order to assure that quality documents, records, etc. are up to date. In this regard, the separate peer review that took place in June, 2019 found that these requirements are being fulfilled.

4.5. Tests with a 1 kΩ resistor as the calibration standard for 10 kΩ resistors
Lameq is planning to perform a direct calibration of a 1 kΩ resistor against the QHS, then use it (instead of 100 Ω) as the calibration standard for 10 kΩ resistors. The objective is to examine possible decrease of the uncertainty through the adoption of a 1:10 resistance ratio in this particular case.

5. Conclusions
This paper presented the activities that are either being executed or prepared at the Inmetro with the aim to obtaining recognition of its quantum Hall system as the primary standard of electrical resistance in Brazil. These activities intend the enhancement of procedures and system operation, as well as the validation/improvement of the measurement results provided by the QHS. Inmetro is participating in the key comparison BIPM.EM-K13.a&b (1 Ω & 10 kΩ travelling standards); the results and reports of such will be published in the BIPM key comparison database. An informal comparison of a 100 Ω resistor calibrated against the BIPM QHS was also performed and will be scrutinized in a future work. In addition, technical studies are going to be carried out to assess the effects of varying nominal currents, measurement cycle times and resistance ratios. Lately, Lameq was peer reviewed, and CCC consistency checks for metrological confirmation will be implemented by recommendation of the reviewer. Finally, the participation in the key comparison BIPM.EM-K12 is expected to occur in the near future, which requires suitable budget allocation.

Acknowledgments
The authors would like to thank Dr. Pierre Gournay and Mr. Benjamin Rolland, from the BIPM, for the close cooperation and for kindly allowing us to use the measurement cycle times image. We also wish to express our deepest gratitude to Dr. Alain Rüfenacht, from the NIST, whose contributions provided during the peer review were valuable. The authors also thank the colleagues from the Electrical Standardization Metrology Laboratory (Lampe) at the Inmetro for the constant technical support.

References
[1] Comité International des Poids et Mesures 1999 Mutual recognition of national measurement standards and of calibration and measurement certificates issued by national metrology institutes (Paris, France: BIPM)
[2] Bruce S 2018 Guide to the implementation of the CIPM MRA - CIPM MRA-G-01 Version 2.0 (Paris, France: BIPM)
[3] Bureau International des Poids et Mesures 2002 BIPM.EM-K12 Information Available at https://www.bipm.org/kcdb/comparison?id=430
[4] Delahaye F, Witt T J, Piquemal F and Genevks G 1995 IEEE Trans. Instrum. Meas. 44(2) 258–61
[5] Bureau International des Poids et Mesures 2002 BIPM.EM-K13.a Information Available at https://www.bipm.org/kcdb/comparison?id=26
[6] Bureau International des Poids et Mesures 2002 BIPM.EM-K13.b Information Available at https://www.bipm.org/kcdb/comparison?id=438
[7] da Silva J R B, de Brito J P and Briones R E M 2011 Condicionamento para Operação de um Sistema Hall Quântico Proc. IX Semetro - 9th International Congress on Electrical Metrology (Natal, RN, Brazil) 90186
[8] Williams J M, Janssen T J B M, Rietveld G and Houtzager E 2010 Metrologia 47(3) 167–74
[9] Grohmann K, Hahlbohm H D, Lübbig H and Ramin H 1974 *IEEE Trans. Instrum. Meas.* **23**(4) 261–3
[10] Carvalho H R, da Silva J R B and Briones R E M 2013 Estabelecimento da Base da Escala do Ohm no Inmetro a Partir da Padronização Quântica de Resistência *Proc. X Semetro - 10th International Congress on Electrical Metrology* (Buenos Aires, Argentina) ID033
[11] Carvalho H R, Briones R E M and da Silva J R B 2015 *J. Phys.: Conf. Ser.* **575** 012014
[12] Carvalho H R, Briones R E M, Pierz K and Götz M 2016 *J. Phys.: Conf. Ser.* **733** 012069
[13] da Silva M C, Carvalho H R and Vasconcellos R T B 2016 *J. Phys.: Conf. Ser.* **733** 012074
[14] Goebel R 2015 *Technical protocol for BIPM.EM-K13a&b comparisons* (Paris, France: BIPM) Available at https://www.bipm.org/kcdb/comparison/doc/download/26/bipm.em-k13_tp.pdf
[15] Consultative Committee for Electricity and Magnetism 2017 *CCEM Guidelines for Implementation of the ‘Revised SI’* (Paris, France: BIPM)