Long-term research in Inmetro on samples of quantum Hall resistance standards made by PTB

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Abstract. This paper shows up to date results of Inmetro’s investigations on aging effects of quantum Hall samples fabricated by PTB in the frame of a mutual scientific agreement established between the Brazilian and German National Metrology Institutes.

1. Introduction
A mutual agreement on scientific cooperation in the frame of quantum Hall resistance measurements has been signed up between Inmetro and PTB on the 21st of December 2012 which will hold for the next five years. Related to this, there are two samples with appropriate semiconductor properties made by PTB team [1, 2]. These samples were sent to Inmetro in order to be periodically measured with its quantum Hall system (QHS). The resulting reports will be used by the PTB for improvement of quantum Hall samples manufacture, as well as by Inmetro for the fully characterization and knowledge of the behaviors of these two samples (which will stay at Inmetro after the end of the agreement). The two samples were labeled as P137-23 and P137-24 by PTB.

These samples were, prior to delivery, characterized by PTB at 2.2 K. This characterization and Inmetro’s up to date results are presented in this paper.

2. Quantum Hall measurements
The Quantum Electrical Metrology Laboratory (Lameq) contains two modules. One of them is responsible for making measurements and research with quantum Josephson Voltage Standards (conventional and programmable ones). The other one is responsible for making measurements and research with quantum Hall system (QHS) to provide, in the near future, traceability to SI ohm [3-5]. The tasks reported here take place in this second module.

It is equipped with two cryostats. One of them, called “Quantum Hall Sample Cryostat” or simply “Sample Cryostat”, is able to cool the sample down to 0.3 K (by pumping above liquid He-3) and enables magnetic field intensities of up to about 16 T. The equipment has a current source for the superconducting coil and a temperature control in three specific locations in cryostat.

The second Lameq’s cryostat, in this second module, is equipped with a cryogenic current comparator (CCC), which is used to compare resistors (including the quantum Hall samples) with a relative measurement uncertainty of about 10⁻⁸.

Additional instruments, common to QHS, were also used as an 8 ½ digit digital multimeter, a two-channel 7 ½ digit digital nanovoltmeter, a Zener voltage reference and a resistive decade.
A Labview application was developed in order to control the instruments and acquire measurements. It can change the field by controlling the coil current source and measures the Hall voltage ($V_{H}$) and the longitudinal voltage ($V_{xx}$) of the sample with the nanovoltmeter [6].

### 3. Measurements and results

#### 3.1. Contact resistance of both samples

All contact resistances of both samples were measured by “three-terminal measurement technique” [6]. First, a “short-circuit sample” was put instead of the actual samples in order to find the resistance of each wire. The wire resistance measurements were processed under conditions identical to the samples (temperature was 0.4 K and the magnetic field was the same as used in plateau 2). Then, the samples were placed and the value of wire resistance was subtracted. All measurements were carried out with current through the sample in both directions to eliminate offsets of the measuring equipment. The current value used was 9.91 $\mu$A, because this value does not exceed the maximum allowed for all contacts [6].

The contacts of both samples show very low resistances yielding values around $0 \Omega$ within a relative standard deviation of about $8 \times 10^{-6}$ (measurement limited by sensitivity of the nanovoltmeter).

#### 3.2. Results of the P137-23 plateaus scan

The graph of figure 1 shows three measurements ($R_{H}$ and $R_{xx}$) while scanning the magnetic field up to near 14 T. They are related to two P137-23 sample measurements made in Inmetro-Lameq in December 2013 and one made in PTB before shipping.

![Figure 1](image_url)  
**Figure 1.** Two P137-23 measurements made in December 2013 and one made in PTB.

The graph in figure 2 shows P137-23 measurements made under temperature of 0.4 K in April 2015.
Figure 2. P137-23 measurements in April 2015 (R_{H} above and R_{xx} below).

Figure 3 shows detail of R_{xx} graph in figure 2 at plateau 2 region for both current directions.

Figure 3. Graph of R_{xx} detail from P137-23 measurements (figure 2) at plateau 2 region in both current directions.

The measurement values between 10 T and 11.5 T are around zero and the standard deviation of these values is

\[ \sigma = 2.5 \times 10^{-2} \, \Omega. \]  

(1)

3.3. Results of the P137-24 Plateaus Scan

The same procedure was made with sample P137-24. First, figure 4 show the measurements made in December 2013 and one made in PTB before shipping.
Figure 4. Two P137-24 measurements made in December 2013 and one made in PTB.

The next graph shows the measurements in April 2015 (figure 5).

Figure 5. P137-24 measurements in April 2015 ($R_{\text{H}}$ above and $R_{\text{xx}}$ below).

The figure 6 shows detail of the $R_{\text{xx}}$ graph at plateau 2 region from figure 5.
In this case, the measurement values between 10 T and 11.5 T are also around zero and the standard deviation is

$$\sigma = 7.3 \times 10^{-2} \, \Omega.$$  \hspace{1cm} (2)

### 3.4. Comparison using CCC

In March 2015, these two samples were used to calibrate the standard resistor of 100 Ω using the CCC. Figure 7 shows these two measurements (the two points to the right) together with the historical evolution of this standard resistor (circle and square points and linear adjustment straight line) calibrated by using ordinary QHR samples.

The value of standard resistor predicted by historical for March 2015 and its expanded uncertainty are shown in (3) and (4).

$$R_{100\Omega} = 100.0001211 \, \Omega$$ \hspace{1cm} (3)

$$U_{100\Omega} = 4.2 \times 10^{-6} \, \Omega$$ \hspace{1cm} (4)

The value obtained by calibration, made in March 2015, of same standard resistor by using the P137-23 sample is shown in (5) and its expanded uncertainty is shown in (6).
The relative difference (RD) and normalized error (NE) [5] of measurements made by using samples P137-23 are shown in (9) and (10), respectively. The results from similar procedure, concerning to P137-24 sample, are shown in (11) and (12).

\[
RD_{100\Omega(23)} = \frac{R_{100\Omega(23)} - R_{100\Omega}}{R_{100\Omega}} = -3.5 \times 10^{-8}
\]  
(9)

\[
EN_{100\Omega(23)} = \frac{|R_{100\Omega(23)} - R_{100\Omega}|}{\sqrt{u^2_{100\Omega(23)} + u^2_{100\Omega}}} = 0.81
\]  
(10)

\[
RD_{100\Omega(24)} = -2.8 \times 10^{-8}
\]  
(11)

\[
EN_{100\Omega(24)} = 0.67
\]  
(12)

4. Conclusion
These results show that the two samples, made by PTB and sent to Inmetro according to agreement between these two institutes, which were measured, have worked as expected. and Lameq contributed to improve calibration procedures for both laboratories, allowing not only to reduce uncertainties as well as to increase the reliability of the measurement results.

The contact resistance is undetected as well as the Vxx on the plateaus region. The plateaus width increase when the temperature goes down and have not had perceptive changing during these three years.

The results of the standard resistor calibrations made by using the official laboratory quantum Hall sample were compared with the results of the same resistor calibrations made by using the PTB samples. The results, shown in this paper, indicate that both PTB samples have worked as expected and have similar behavior.

Acknowledgments
The authors would like to thank JT Janssen from NPL and Thomas Holubar from Cryogenic Ltd. to assist in improving our understanding about Hall cryostat and CCC and to recover some problems of these QHS elements during technical visits in our laboratory in the course of 2013. Furthermore, we would like to thank Waldemar G. K. Ihlenfeld and Robson Gomes to assist us in reviewing the text.

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