Implementation of a Commercial Quantum Hall Resistance System

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This paper describes the process and implementation of a commercially available Quantum Hall Resistance System into a Primary Standards Calibration Laboratory. Topics include system design, component descriptions and verification of the sample operating characteristics, including the measurement of the QHR device in the MI 6800A system when compared to a cryogenic current comparator. (CCC) The system verification referenced is located at the Institute for National Measurement Standards (INMS) at the National Research Council (NRC), Canada. Also included is a brief description of the most recent on-site installation, the measurement results using the QHR 6800A system to establish the value of a 1kΩ resistor and the automated measurement process from the 1kΩ resistor up to the 10kΩ and down to the 1Ω value.

1.0 Introduction

In 1999 Measurements International released the prototype Quantum Hall Resistance Standard [1] with a working system on site at the CPEM in Washington, DC. Since then, there have been three successful installations in various laboratories around the world. Each successful installation brings to the customer the ability to have the Quantum Hall Resistance Standard as their representation of the ohm.

The QHR 6800A system, a fully self-contained primary standard of resistance, was introduced as a replacement for the larger, expensive and difficult to operate cryogenic current comparator systems. The QHR 6800A can be operated on an annual, semi-annual or quarterly basis to improve the accuracy in resistance metrology to national levels.
During the months of February and March of 2006, the QHR 6800A system was tested at the INMS laboratory of NRC, Canada. Tests proved the successful operation of the QHR device [2] and a measurement of the QHR 6800A against a 1kΩ as compared to the measurement of the Cryogenic Current Comparator (CCC) against the same 1kΩ resistor.

During the period of April 3-7, 2006, installation of the QHR 6800A system was completed at the US Navy Primary Standards Laboratory in San Diego, USA, with the same device characteristic tests completed to ensure proper sample operation and the measurement of the QHR Device against a 1kΩ on-site resistor. The automated measurements of the 1kΩ against a 10kΩ resistor were also tested.

2.0 QHR 6800 System Components

2.1 QHR Device

The QHR device parameters determine the flexibility with respect to rest of the system. The QHR device, which is a GaAs/AlGaAs heterostructure, was grown at the Institute for Microstructural Sciences at the National Research Council, Canada and devices were fabricated by standard photolithography and wet etching techniques. Tin ball contacts were annealed onto the device for electrical contacts.

The QHR device contains a two dimensional layer of electrons (2-Deg), the behavior of which yields the quantized hall resistance. When the device is cooled to about 1.2Kelvin and subjected to a magnetic field perpendicular the 2-Deg layer, then a current (I) passed through the 2-Deg will be diverted by the magnetic field, leading to measurable potential differences both along (Vxx) and across (Vxy) of the device. The longitudinal and Hall resistances of the device are given by the ratios Vxx/I and Vxy/I. [1]

Devices were first tested in the Quantum Hall System at the INMS of NRC, Canada. The device was tested at temperatures of both 0.3K and 1.2K. A device report was generated and supplied with the system.

The QHR 6800A operates on the step of (i) = 2 (12906.4035Ω) at a field of around 7.3 Telsa and uses a current of 77μA. This is due to the operation of the measuring equipment that will be discussed later in this article.

Before the values of the QHR step i=2 can be transferred to a 1kΩ resistor, a series of additional checks are necessary to ensure confidence in the accuracy of the QHR device. These measurements are as follows:

i) Sweep Check Measurement:

Feeding current of either 50μA or 77μA into the S & D of the sample and then reversing it allows measurement of the potential differences between various points. These potential differences can be measured at Hall resistances Vxy(1-2), Vxy(3-4), and the longitudinal resistance Vxx(1-3) and Vxx(2-4). Vxy(1-2) and Vxy(3-4) should be in close agreement with each other as should Vxx(1-3) and Vxx(2-4).

ii) Contact Resistance:

Large contact resistances can lead to errors in the QHR measurement. For this reason it is important to measure the contact resistance each time the QHR device is cycled to room temperature and re-cooled. The contact resistance can be calculated by making a three-probe measurement on each of the contacts in turn. The contact resistance is equal to V(cr) = resistance of wire + resistance of contact + resistance of 2-Deg. The contact resistance should be ideally less then 1Ω [2]

iii) Longitudinal Potential Difference:

For an accurate transfer of the QHR value it is also necessary to measure Vxx, the longitudinal potential difference. This can be accomplished by measuring between Vxx(1-3), and Vxx(2-4). This measurement is taken to verify that there is no dissipation in the 2-Deg. When the 2-Deg is quantized Vxx should go to 0 and should be < 2 x 10⁻⁸ of Vxy.

All of these tests have been described in detail in previous technical papers [1] [2] [3].

Upon completion of the checks on the sample, one can have confidence that the sample is operating correctly and that its value on step (i = 2) is 12906.4035Ω (exact).

2.2 Measuring Device

Past comparisons of a DC room temperature bridge (6010Q) against a Cryogenic Current Comparator have been completed with great success.
As a result of these experiments, it was decided to use a DC room temperature current comparator bridge in the Model 6800A system. The bridge, which is capable of ratio measurements up to 13:1, transfers the QHR device at step 1 = 2 (12906.4035ohms) to a 1kΩ resistor, with accuracies of < 0.02ppm. The 6010Q Bridge is independently calibrated over the range of 13:1, and 10:1 ratios from 1Ω to 10kΩ against the Cryogenic Current Comparator (CCC) at the INMS of NRC, Canada. The calibration report for the 6010Q is supplied with the system.

The QHR 6800A system also includes a model 4210A ten (10) channel matrix scanner which, when combined with software gives the user the ability to make automated measurements over the complete range from the QHR device down to 1Ω. The 6010Q has a built in microvolt meter and nano-volt meter to perform all of the needed checks on the sample to ensure proper operation.

2.3 Refrigeration with Integrated Magnet System

Because our QHR devices allow us to operate with sufficient current at 1.2K, we have been able to develop a simple cryogenic and magnet system based on an 8T magnet and a pumped 4He refrigerator. The magnet is removable from the Dewar and is built with a persistent switch, which allows the user to ramp the leads down to 0A to conserve liquid helium while maintaining a constant field.

The refrigerator (built by Cryo Industries of America) is a double walled tube, the space between being evacuable to isolate the inner (sample space) tube from the 4He reservoir. An impedance connects the sample space with the 4He reservoir, and pumping on the sample space both draws the 4He through the impedance into the inner space and lowers the temperature of the sample space down to 1.2K.

A scroll pump is used for the pumping. This provides sufficient pumping speed to cool to the desired temperature and has a low enough ultimate pressure to allow effective evacuation of the vacuum space between the double walls before cooling. Being a dry pump, no traps or baffles are required. The sample is mounted on a standard TO-8 header, which is easily interchangeable while the system is cold.

Also mounted near the sample are two temperature sensors. One sensor is a silicon diode, which is capable of reading down to 20Kelvin. The other sample is a ruthenium oxide sensor, which is used for temperatures below 20Kelvin down to 1Kelvin. Both sensors are read by a temperature controller to let the user know the temperature of the sample space.[2]

3.0 System Verification at INMS of NRC, Canada

During the months of February and March of 2006, the QHR 6800A system was tested as part of the system verification process. The system was tested on site at the INMS of NRC, Canada. The different areas of tests that were completed were an operational check on the QHR Device, as well as a measurement of the QHR Device against the CCC of INMS.

Figure 2 shows the data obtained during the testing process.

3.1 Contact Resistance Measurement

Poor contact resistance can lead to erroneous sample values in the measurement of the QHR value. Therefore, it is necessary to measure each contact resistance every time the sample is cooled from room temperature. Using the microvolt function of the 6010Q resistance bridge, the resistance of each contact was measured. This was accomplished by making a three-probe measurement on the sample contacts.

The resistance of each contact is equal to Contact Resistance = Resistance Total – Resistance Lead. A current of 77uA was used for all contacts in the measurement. For NRC sample Qi0502, each contact was verified to be < 1Ω.

<table>
<thead>
<tr>
<th>V(cr)</th>
<th>Resistance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Source</td>
<td>190mΩ</td>
</tr>
<tr>
<td>Drain</td>
<td>160mΩ</td>
</tr>
<tr>
<td>1</td>
<td>200mΩ</td>
</tr>
<tr>
<td>3</td>
<td>200mΩ</td>
</tr>
<tr>
<td>2</td>
<td>340mΩ</td>
</tr>
<tr>
<td>4</td>
<td>260mΩ</td>
</tr>
</tbody>
</table>

Table 1. NRC Sample Qi0502@1.3Kelvin. Field Set @ 7.28 Tesla.

Figure 2. Sweep Check of QHR Device Qi0502 (at 77uA) @ 1.3Kelvin. These field sweeps are representative of the data sets from a correctly cooled two-dimensional electron gas (2-Deg).
3.2 Dissipation Measurement
The dissipation along the sample was measured with the 6010Q bridge in nano-volt mode. Measurements were done at the center field of the (i) = 2 plateau, 7.28 Tesla, with a current setting of 77μA for each measurement. Precision measurements were made along the sample Vxx(1-3) and Vxx(2-4). The dissipation through NRC sample Qk 0502 was found to be < 2.5 x 10^-8 of the total applied voltage to the device. These values of Vxx imply that the dissipation in the sample has no effect on the measured value of the Hall voltage, Vxy.

3.3 Calibration of Room Temperature Current Comparator Bridge (6010Q)
The National Research Council, Canada, did an independent calibration of the Measurements International 6010Q DC Room Temperature Bridge. Table 2 shows the results of measurements of the ratio's using the NRC CCC and the MI 6010Q Bridge. The values shown in the table are shown in ppm. All uncertainties in the table include a k = 2 coverage factor, and are based on a 95% confidence level.

<table>
<thead>
<tr>
<th>Resistance Ratio</th>
<th>6010Q Value</th>
<th>Uc MIL</th>
<th>NRC CCC Value</th>
<th>UC NRC</th>
<th>Difference (6010Q – CCC)</th>
<th>Uc Combined</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 Ω : 1 Ω</td>
<td>-25.158</td>
<td>0.006</td>
<td>-25.166</td>
<td>0.007</td>
<td>0.007</td>
<td>0.010</td>
</tr>
<tr>
<td>100 Ω : 10 Ω</td>
<td>116.655</td>
<td>0.004</td>
<td>116.634</td>
<td>0.003</td>
<td>0.020</td>
<td>0.005</td>
</tr>
<tr>
<td>1k Ω : 100 Ω</td>
<td>-89.951</td>
<td>0.008</td>
<td>-89.957</td>
<td>0.004</td>
<td>0.005</td>
<td>0.009</td>
</tr>
<tr>
<td>10k Ω : 1k Ω</td>
<td>-26.587</td>
<td>0.013</td>
<td>-26.594</td>
<td>0.004</td>
<td>0.007</td>
<td>0.014</td>
</tr>
<tr>
<td>13k Ω : 1k Ω</td>
<td>12.139</td>
<td>0.030</td>
<td>12.137</td>
<td>0.002</td>
<td>0.002</td>
<td>0.030</td>
</tr>
</tbody>
</table>

Table 2. Comparison of MI 6010Q Bridge and NRC Cryogenic Current Comparator.

3.4 Comparison of QHR 6800 Against the CCC by a 1kΩ Resistor
Table 3 shows the difference in value of a 1kΩ Tinsley resistor measured in an oil bath at 23 degrees Celsius. The measurements were made using the MI 6800A QHR Resistance System and the NRC QHR Primary Resistance Standard and CCC. Values are in the uΩ/Ω (ppm) deviation from the expected value. All uncertainties shown in this table are expanded uncertainties with k = 2 coverage factor and are approximately equal to 95% confidence interval.

<table>
<thead>
<tr>
<th>Resistance Ratio</th>
<th>6800A Value</th>
<th>Uc MIL</th>
<th>NRC CCC Value</th>
<th>UC NRC</th>
<th>Difference (6800A-NRC)</th>
<th>Uc Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>QHR(i=2): 1kΩ</td>
<td>14.626</td>
<td>0.038</td>
<td>14.641</td>
<td>0.025</td>
<td>-0.017</td>
<td>0.043</td>
</tr>
</tbody>
</table>

Table 3. Comparison of MI 6800A QHR Resistance System and NRC QHR Primary Resistance Standard.

4.0 Installation at the Navy Primary Standards Lab
During the week of April 3 to April 8, 2006 the QHR 6800A was installed and tested at the Navy Primary Standards Laboratory, San Diego. On the first day of installation the system was unpacked and setup. This process was completed in 2.5 hours. In the afternoon the system was cooled down slowly with liquid helium. Fill-up was completed in 4 hours. The reason for the long period of time was due to first pre-cooling the system with cold helium gas. This was to avoid a large amount of liquid helium loss as the system had to be cooled down from room temperature. Following the fill up the system was left overnight to allow the sample space to cool down to the needed temperature.

On the second day of installation the system was refilled to full capacity, as there was some helium loss overnight. Once the system was refilled, testing of the probe space was completed and measured at 1.3 Kelvin.

4.1 Sweep Check Measurement
Sweep check measurement results are shown in Figure 3. These field sweeps are representative of the data sets from a correctly cooled two-dimensional electron gas (2-Deg).

4.2 Contact Resistance Measurement
The contact resistance test is a three-probe measurement on all of the sample contacts to measure the contact resistance. The contact resistance of each contact (measured by the 6010Q) is equal to $V_{CR} = \text{Resistance Total} - \text{Resistance Lead}$. A current of 77μA was used for all contacts in the measurement. For NRC sample Qk 0502, each contact was verified to be << 1Ω.

<table>
<thead>
<tr>
<th>Contact</th>
<th>Resistance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Source</td>
<td>190mΩ</td>
</tr>
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<tr>
<td>1</td>
<td>150mΩ</td>
</tr>
<tr>
<td>2</td>
<td>200mΩ</td>
</tr>
<tr>
<td>3</td>
<td>290mΩ</td>
</tr>
<tr>
<td>4</td>
<td>260mΩ</td>
</tr>
</tbody>
</table>

Table 4. NRC Sample Qk 0502 @ 1.3 Kelvin. Field Set @ 7.28 Tesla

4.3 Dissipation Measurement
The dissipation along the sample was measured with the 6010Q Bridge in nano-volt mode. Measurements were done at the center field of the (i) = 2 plateau, 7.28 Tesla, with a current...
setting of 77uA for each measurement. Precision measurements were made along the sample Vxx(1-3) and Vxx(2-4).

The dissipation through NRC sample Q60502 was found to be < 2.8 x 10⁻⁸ of the total applied voltage to the device. These values of Vxx imply that the dissipation in the sample has no effect on the measured value of the Hall voltage, Vxy.

4.4 Measurement of Q60502 Device against 1kΩ On-site Resistor

Once all of the checks on the sample were completed and the sample was verified as operating correctly, the next step was to measure the QHR against a 1kΩ on-site resistor. The 6010Q software was set-up to measure the QHR standard against the 1kΩ resistor. When the measurement of the QHR (i=2) against the 1kΩ was completed, the 1kΩ was then measured against the 10kΩ to determine the value of the 10kΩ.

Table 5 is a summary of the measurements taken at the Navy Primary Standard Laboratories. Values are in the uΩ/Ω (ppm) deviation from the expected value. All uncertainties shown in this table are expanded uncertainties with k=2 coverage factor and are approximately equal to 95% confidence interval.

5.0 Conclusion

Described was the implementation of a commercial available Quantum Hall Resistance System. There were four main areas covered:

1. System components, which include the QHR device, the refrigeration and magnet system, and the measuring device (MI 6010Q)
2. Calibration of the DC Room Temperature Current Comparator Bridge (6010Q) against the Cryogenic Current Comparator (CCC) at the Institute for National Measurement Standards (INMS), of the National Research Council, Canada (NRCC) which was accurate to a few parts in 10⁻⁸, with a measurement uncertainty of a few parts in 10⁻⁸
3. System verification by process of a 1kΩ comparison between the Measurements International Model 6800 Quantum Hall Resistance System and the INMS, of NRCC, Cryogenic Current Comparator System, with the Model 6800 being accurate to a few parts in 10⁻⁸

<table>
<thead>
<tr>
<th>Resistance Ratio</th>
<th>6800A Value</th>
<th>Uc MIL</th>
<th>NPSL Value</th>
<th>Difference (6800A-NPSL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10kΩ: 1kΩ</td>
<td>4.78</td>
<td>0.04</td>
<td>4.82</td>
<td>-0.04</td>
</tr>
</tbody>
</table>

Table 5. Comparison of MI 6800A QHR against 1kΩ resistor.

*All values shown in uΩ/Ω (ppm)