A modular, economic, portable quantised Hall resistance standard

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Abstract:

We outline the recent development of the “QuantΩ”, an affordable, portable quantised Hall resistance (QHR) standard that uses as its measuring component a room temperature dc current comparator bridge. We describe the characteristics of the QHR devices developed for this system, and give details of the refrigerator and integrated 8 T magnet which can be top-loaded as a single unit into a transport dewar. We discuss the measurements required to ensure an accurate transfer from a QHR device to a wire resistor, and show that the QuantΩ can be used to meet these requirements.

Even the best wire-wound resistors maintained in the most stable of conditions drift with time, and sometimes shows inexplicable step changes in value. Consequently the introduction at the beginning of this decade of the quantised Hall resistance as a representation of the ohm led to previously unattainable levels of repeatability and stability in resistance metrology. Comparisons between devices and between laboratories has lead to the conclusion that correct implementation of a QHR resistance standard yields a device-to-device agreement at the level of 2×10^{-10} [(1,2)] and interlaboratory agreement at the level of 5×10^{-9} or better [(3-5)].

However, with one or two notable exceptions all the QHR representations of the ohm in operation today are in the national laboratories of the more developed countries. The national measurement laboratories of some countries have in general found it to be technically or economically beyond their present means to develop such primary standards. Although commercial QHR systems based on cryogenic current comparator (CCC) technology have recently become available they are relatively expensive.

A few years ago we set out to develop at NRC an economical QHR primary standard which would be accurate at the level of < $1\text{x}10^{-7}$, and which would be both cryogenically and electrically simpler to operate than previous systems. It was thought that if such a system were economical to purchase and to operate then it would be an attractive option for some of the smaller national laboratories and perhaps also the larger industrial metrology laboratories.

The outcome was the QuantΩ, which we describe in this article. The QuantΩ is a fully self-contained primary standard of resistance comprising a QHR device, a cryogenic refrigerator and superconducting magnet, and a measuring system. It is modular in the sense that any of the three basic components could be purchased separately, and used independently – for example the dc current comparator bridge can be used as a high accuracy dc resistance ratio bridge in its own right. And although portability was not an initial design parameter, it is portable –a prototype was taken to the Conference on Precision Electromagnetic Measurements in Washington, D.C., in 1998 where it operated for a week in a hotel room. With the aid of a pair of extension cords we have operated the system at 1.2 K on the front lawn at NRC, in Ottawa. And we will be taking it to Charlotte, N.C., for the NCSL Workshop and Symposium in July this year.

1. QHR Devices.

The QH device parameters determine the flexibility one has with all the rest of the apparatus, so we started with device design. QH devices contain a two-dimensional layer of electrons (or 2-DEG), the behaviour of which yields the quantised Hall resistance. When the device is cooled to about 1 K and subjected to a magnetic field of several tesla perpendicular to 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 (V_{xx}) and across (V_{xy}) the device. The longitudinal and Hall resistances of the device are given by the ratios V_{xx}/I and V_{xy}/I, respectively.

In Figure 1 we show the way in which the two resistances vary with applied magnetic field. The “flat” parts of the V_{xy} vs. B curve, which occur where the V_{xx} vs. B curve go to zero, give constant values of resistance over a significant range of magnetic induction, and it is these plateaus which give the *quantised* values of the Hall resistance. The values of the resistance on the various plateaus are related by the relation

$$R_{xy}(i) = \mathrm{R}_{\mathbf{K\text{-}90}}/i \qquad (1)$$

where $\mathrm{R}_{\mathbf{K\text{-}90}}$ is the von Klitzing constant, defined to be 25 812.807 ohms, and i is an integer 1, 2, 3… In a well-quantised device the precision with which relation (1) holds is at least 2×10^{-9} for the plateaus of metrological interest [(2)].

The density of carriers in the 2-DEG determines the magnetic induction at which the Hall plateaus occur. We have developed devices which have step centres for i = 2 and i = 4 at about 7.5 T and 3.75 T respectively. The wafers were grown to our specifications, using molecular beam epitaxy by the Institute for Microstructural Sciences at the National Research Council, Canada, and devices were fabricated by standard photolithography and wet etching techniques. We have used either annealed tin balls or deposited and annealed multi-layer AuGeNi films for electrical contacts. For metrological use the contact resistance should be as small as possible, ideally of the order of mΩ, and we have found that either of our techniques leads to sufficiently good quality contacts [6].

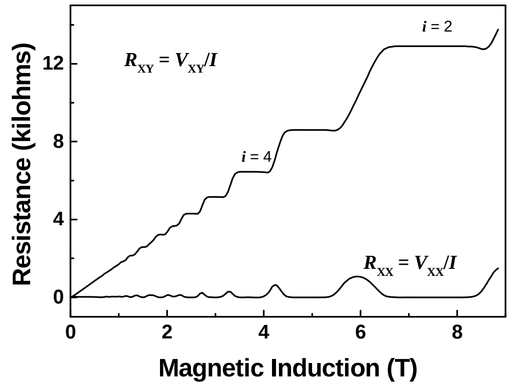

Figure 1: Variation of the Hall (R_{XY}) and the longitudinal (R_{XX}) resistances as a function of magnetic field. (Device NRC 1794B, 1.2 K, 50 μA)

Besides carrier density the principle parameter of importance in a QHR device is the critical current - the current at which sudden breakdown of the quantisation occurs. The structural factors affecting this parameter are not yet well understood, but it is always temperature dependent, increasing as the temperature decreases. Ideally one needs a useable current (40 μA or more) at 1.2 K, since this removes the need for a ^{3}He refrigerator. We would like to operate with a current of 77 μA at this temperature to allow us to transfer ultimately to a 10 kΩ resistor without being concerned about power corrections. Our devices will usually carry a current of 100 μA or more on step i = 2, at 1.2 K.

The advantage of these devices over most previously available devices is two-fold. Step i = 2 is at a low enough field that it allows us to use an 8 T magnet, which will fit into a transport dewar, but at the same time step i = 4 is at a high enough field (~ 3.75 T) that it is sufficiently quantised to also allow a precision measurement. Hence besides being able to transfer a resistance value to a working standard, one can also make a two-point check on the linearity of the measuring system.

2. Cryogenic components.

Because our QH devices operate with sufficient current at 1.2 K, we have been able to utilise simple cryogenic and magnet systems based on an 8 T magnet and a pumped ^{4}He refrigerator. The magnet measures 21.5 cm in length, with a diameter of 7 cm and a 2.6 cm bore. The field at the centre has ± 0.1% uniformity over 1 cm^3. A current of 56 A at 4.2 K a produces an induction of 8 T in the central region. In the prototype system the magnet is bolted to the outer tube wall of the ^{4}He refrigerator, and the whole can be inserted through the 7.6 cm opening in the top of a 60 L transport dewar.

The refrigerator (built by Cryo Industries of America) comprises a double-walled tube, of 2.5 cm outer diameter, the space between the two walls being evacuable to isolate the inner (sample-space) tube from the ^{4}He reservoir. An impedance connects the inner space with the ^{4}He reservoir, and pumping on this inner space both draws ^{4}He into the tube and lowers the temperature of the inner space to 1.2 K. 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 liquid nitrogen traps or baffles are required.

The QHR device is mounted on a standard TO-8 header, which plugs into a socket on the end of the probe. In the prototype the probe is inserted through an 'O'-ring at the top of the refrigerator, and can be adjusted vertically to optimize the field position of the sample. Because a standard TO-8 header is used, the sample is easily interchangeable. The probe also carries a thermometer and a heater. The present thermometer sensor is a GaAs diode, but we are investigating the use of ruthenium oxide sensors, which will go to a lower base temperature. The device wiring in the probe is Teflon insulated, with an impedance between wires or between wire and probe of $>10^{13}\ \Omega$.

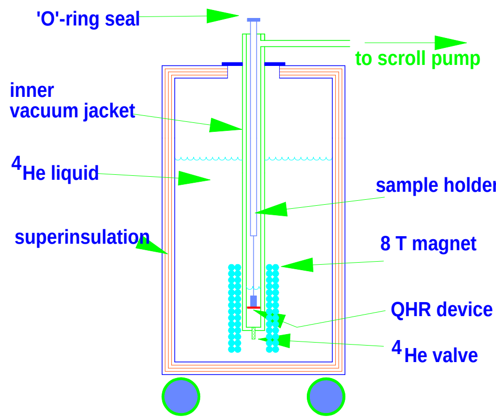

Figure 2: Schematic of the dewar, magnet and refrigerator.

A prototype system has been built, and is illustrated in Figure 2. It was shown and operated at the Conference on Precision Electrical Measurements in Washington DC, in July of last year, and will be operated at the National Conference of Standards Laboratories meeting in Charlotte, N.C., this summer. Operation is relatively simple. The dewar is sent out for filling, and on its return the fridge and magnet unit are inserted. This takes about an hour, if one wishes to avoid excessive boil-off. One can than measure for up to two days without refilling. This is sufficient time to complete all the necessary checks (see next Section) and to do a complete transfer from the QHR to working resistors. There is a fill-port in the top plate, so helium can be transferred in, to extend the run-time – we have kept the prototype cold for several weeks, on occasion.

The magnet can be ramped to full field in about five minutes, although if data are being collected during the sweep it would be more normal to take perhaps half an hour or so. Once at field it can be put into persistent mode and the leads ramped down, to conserve helium. Although it was not originally intended that this unit be particularly portable we have found it to be relatively easy to move around, and to operate in environments outside of the laboratory.

With some months of experience behind us in operating the prototype system, we are now considering a variety of optional modifications. One is the addition of a load-lock with a sliding seal and gate valve, to allow quick sample changes for those who wish to do more than simply maintain a unit and transfer values to wire resistors. We are now looking at the possibility of using a "belly-style" dewar with integral magnet, which will considerably increase the measuring time available per fill of helium. It will also allow the use of a higher field magnet – this option could be offered with a 9T magnet. For the future we intend to investigate the possibility of cooling the magnet and sample space using a cryo-cooler, hence removing the dependence on a liquid helium supply.

3. Measuring system

Although potentiometric measurement systems have been used it is generally accepted that the “best” QHR measurements are made using a cryogenic current comparator. Such a device is difficult and time consuming to develop, and commercially available versions are prohibitively expensive for the smaller laboratory. At the start of this project it was assumed that the measuring system to be used would probably be based on an 8½-digit DVM. Such an approach has been studied in detail for use with the QHR [(7-9)]. The advantages of a DVM-based system are low cost and relative simplicity: The main disadvantage is that it cannot provide an accuracy of better than 1×10^{-7}. We have taken a middle road in both expense and complexity in pioneering the use of a commercial room-temperature dc current comparator bridge as the measuring component in the system described in this paper.

Our early attempts showed that such bridges could be used to transfer values from a QHR device on step $i = 2$ to a 1 kΩ resistor with an accuracy of a few parts in 10^8. However, in order to be confident in the accuracy of measurements made with a QHR device it is necessary to make a series of additional measurements of the device, over and above the comparison of R_{xy} with a wire resistor. In the order in which they might usually be carried out the set of required measurements is:

- a field sweep of the Hall and longitudinal resistances
- a three terminal measurement of the contact resistances
- precision checks on dissipation
- precision measurement of the Hall resistance, ideally using two different sets of contacts in turn

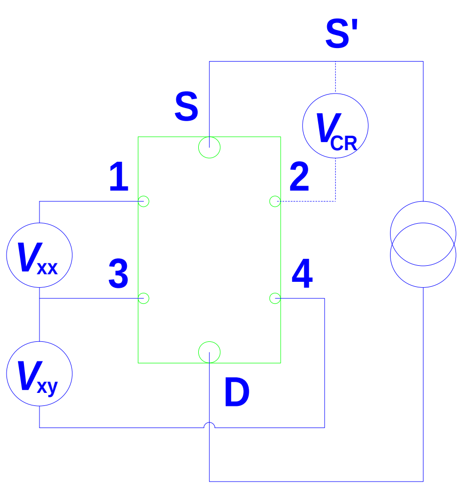

Figure 3: Schematic to indicate the measurements required to ensure an accurate value for the quantised Hall resistance.

In Figure 3 we show a schematic of a QHR device to illustrate what each of these measurements involves. The current is fed in and out of the ends of the rectangular device through terminals S and D, and potential differences can be measured between various points. The current is of course reversed repeatedly during the measurement. The magnetic field is applied perpendicularly to the device (perpendicularly to the page in this instance) and so the Hall potential differences, V_{xy}, are measured between terminals 1 and 2 or between 3 and 4. The potential difference along the device, the longitudinal potential difference V_{xx}, can be measured along each side, between terminals 1 and 3 (illustrated) and/or between 2 and 4. R_{xx} and R_{xy} are obtained as explained in Section 1, above.

Field sweeps are done after cooling the magnet and device, R_{xx} and R_{xy} being measured while the magnet is swept up to full field. This gives the characteristic sweep data sets shown in Figure 1, and requires the measurement of voltages of ±1 V with a precision of around 0.1%. The 6010Q Bridge has been modified so that one can access the microvoltmeter directly and use to measure these voltages (although not both voltages at once). The sweep data should look like that of Figure 1, with R_{xy} plateaus accompanied by zero values for R_{xx}. If the plateaus are not flat, or if the zero regions are not well aligned with the plateau regions (in terms of magnetic field range) then there is a problem with the sample, or the cooling-down of the sample, or both. One then sweeps to the centre of the appropriate plateau to make the rest of the measurements. At this point the magnet can be put into persistent mode, and the leads ramped down.

The second check is to check the contact resistances. It is known that a large contact resistance in a QHR device can also lead to erroneous values for the QHR [10]. The contact resistance of a QHR device is in practice rather difficult to measure unambiguously. It is usually considered sufficient to establish an upper limit by making a three-probe measurement on each of the contacts in turn, in the manner illustrated in Figure 3 for contact S. In the case shown the current is passed through contacts S and D, and the voltage drop measured between the lead into S (point S' in the diagram) and contact 2. This gives the total resistance between S' and 2. The resistance of S' – S can be determined in a separate experiment. If the device is well quantised then the resistance within the device between S and contact 2 will essentially be zero, so one can determine an upper limit for the contact resistance of contact S. Again this measurement is made with the 6010Q in microvoltmeter mode.

One can rotate the three-probe connection around the sample, feeding current into each contact in turn and hence determining an upper limit for the resistance of each contact. Contacts S and D are tested using the full measurement current – typically 77 μA – but the potential contacts can be tested at lower currents, 10 μA or less. For sample NRC 1794A for example, the resistance of (lead+contact+sample) for $V_{(S'-2)}$ (the case in Fig. 5) is 0.85 Ω at 50 μA. Since the lead resistance is 0.77 Ω the contact resistance is $\leq$ 0.08 Ω. This value is typical for all the contacts on this particular sample.

The third “check” measurement, made using the nanovoltmeter mode of he 6010Q, is the measurement of dissipation in the sample. This is done at the plateau centre. If the 2-DEG is properly quantised there will effectively be no longitudinal potential difference between terminals 1 and 3, or between 2 and 4. If there is a measurable potential drop the 2-DEG is resistive, and will not give an accurate value for the Hall resistance. Here one requires a resolution of a nanovolt or so.

When the above checks show all to be in order one proceeds to the measurement of the quantised Hall resistance. Here the normal procedure is to use the 6010Q as a ratio bridge at 13:1, to compare the QHR with a 1 kΩ wire resistor. It is advisable to check that both pairs of terminals – 1and 2, and 3 and 4 in Figure 3 - give the same value. Furthermore, the potential differences around the loop (1-2-3-4) should sum to zero. If there is a leakage current into or out of the sampled region – for example, through the cap layer - this leakage current could lead to an erroneous QHR value [11].

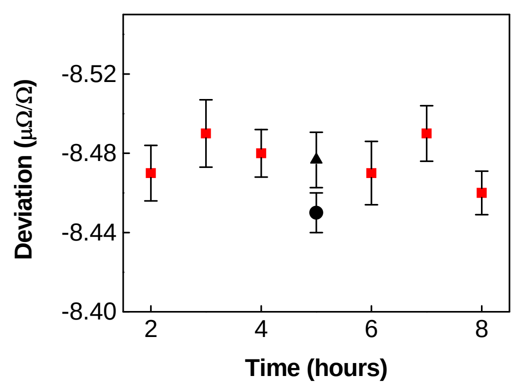

Figure 4: The deviation from nominal of the ratio of a QHR on step i = 2 compared with a 1000 ohm wire resistor. (MIL6010Q – squares; MIL mean – triangle; NRC CCC – circle)

In Figure 4 we show data for the deviation from nominal of the ratio between a QHR device (NRC 1794B) on step i = 2 and a Tinsley 1 kΩ wire resistor. The squares show values obtained using an MIL 6010Q, the triangle representing the mean "MIL" value at the mid-time. The circle is the mid-time value expected for the ratio, from measurements made using the NRC cryogenic current comparator. The agreement is good, with an offset of 2.5 x 10^{-8} between the two data sets. We continue to investigate this offset with a view to eliminating it, although for the present we simply treat it as a calibration offset.

Ultimately a scanner will be added to the instrumentation, so that field ramping and setting and the complete sequence of checks and measurements required for an accurate QHR transfer will be made under computer control.

Conclusion

We have described the development of a simple and relatively economical quantised Hall resistance standard. It comprises three main elements:

- a quantised Hall device which operates on step i = 2 at 1.2 K at fields between 7 T and 8 T and carries a dissipationless current of 77uA or more

- a pumped ^{4}He refrigerator with integral magnet which top-loads, complete, into a transport dewar with a 7.5 cm diameter neck, to give magnetic fields to 8 T and sample temperatures down to 1.2 K

- a dc room temperature current-comparator bridge which is accurate to a few parts in 10^{-8}, with a measurement uncertainty of 1 x 10^{-8}. Magnetic field sweep measurements of V_{xx} and V_{xy}, precision V_{xx} measurements and contact resistance measurements can all be made using this same bridge.

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