Comparison of quantum Hall effect resistance standards of the NIST and the BIPM

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Abstract. An on-site comparison of the quantum Hall effect (QHE) resistance standards of the National Institute of Standards and Technology (NIST) and of the Bureau International des Poids et Mesures (BIPM) was made in April 1999. Measurements of a 100 Ω standard in terms of the conventional value of the von Klitzing constant, $R_{K\text{-}90}$, agreed to 12 parts in 10^{10} with a relative combined standard uncertainty $u_c = 20 \times 10^{-10}$. Measurements of 10 000 Ω/100 Ω and 100 Ω/1 Ω ratios agreed to 59 parts in 10^{10} with $u_c = 55 \times 10^{-10}$ and to 38 parts in 10^{10} with $u_c = 31 \times 10^{-10}$, respectively.

1. Introduction

The comparison reported here is part of a BIPM programme to verify the international coherence of primary resistance standards by comparing QHE standards of the national laboratories with that of the BIPM. The procedure used for the present comparison is the same as that used previously for this programme [1-3]. The complete BIPM transportable QHE standard was shipped to the NIST and, from 14 to 22 April 1999, measurements of a 100 Ω resistance standard in terms of the conventional value of the von Klitzing constant, $R_{K\text{-}90}$, were carried out with the QHE standards of the two laboratories, and similar comparisons were made of 10 000 Ω/100 Ω and 100 Ω/1 Ω ratios. The BIPM measurements were made with 1 Hz ac and those of the NIST with dc. The 1 Hz ac-dc differences of the three resistance ratios were determined at the BIPM before the comparison. For this purpose, the three ratios were measured with the ac bridge at 1 Hz and with the BIPM cryogenic current comparator (CCC) bridge [4] operated with dc. The measured differences were applied as corrections to the BIPM ac measurements carried out at the NIST before comparing them with the corresponding NIST dc measurements, a procedure which has the effect of using the ac bridge as a transfer instrument referenced to the BIPM CCC.

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2. Equipment

2.1 QHE samples

The QHE standards were operated on the $i = 2$ plateau (12 906.4035 Ω), where the resistance of the i-th plateau, $R_H(i)$, is $R_{K\text{-}90}/i$. For this comparison the BIPM used two GaAs-based heterostructures fabricated by the Laboratoires d'Électronique Philips (LEP, Limeil-Brévannes, France) [5] and diced from an unprotected wafer (reference 900514). Samples from this wafer have mobilities of order 30 T^{-1} and carrier concentrations of order 5.1×10^{15} m^{-2}. The samples were operated at a temperature of 1.3 K, with a current of 40 μA, and with a magnetic flux density of about 10.5 T. The values of the longitudinal resistivity did not exceed 50 μΩ.

The NIST used a GaAs/$Al_xGa_{1-x}As$ heterostructure device grown in the early 1980s by molecular beam epitaxy. The electron density is near 5.6×10^{15} m^{-2} and the zero-field mobility at 4.2 K is near 11 T^{-1}. This device was operated on the $i = 2$ plateau at a temperature of 0.3 K, with a current of 40 μA, and with a magnetic flux density of about 11.5 T. The contacts on this device have exceptionally low resistance (< 0.03 Ω) and the longitudinal resistivity on the days of comparison was below 100 μΩ.

2.2 Measurement systems

The NIST measurement systems are based on the CCC bridge. Independent dc sources [6] provide two currents to the bridge in the approximate nominal ratio of the resistances, and are automatically controlled

using a computer. Between the dc measurements, the sources ramp the currents to zero over a period of 1 s (QHE:100 Ω bridge) or 4 s (100:1 bridge). The polarities of the currents are then reversed using relays, and the currents are ramped to the original level. An integrating feedback circuit keeps the ampere-turns product equal in the arms of the bridge and a second circuit balances the voltage across the resistors.

The BIPM transportable measurement system includes a complete QHE resistance standard based on an ac-bridge operating at 1 Hz [7] as well as three conventional standard resistors of 1 Ω, 100 Ω and 10 000 Ω. Table 1 gives the BIPM uncertainty budget corresponding to the three resistance ratios that were the object of the comparison: $R_H(2)$/100 Ω, 10 kΩ/100 Ω and 100 Ω/1 Ω. The 1 Hz ac-dc differences of the three resistance ratios were measured at the BIPM before the comparison and are, in relative values, 44×10^{-10}, 60×10^{-10} and 176×10^{-10}, respectively. These 1 Hz ac-dc differences are reasonably stable: the present values differ from those measured in 1996 [3] by only 4×10^{-10}, 15×10^{-10} and 1×10^{-10}, respectively. The uncertainty associated with these 1 Hz ac-dc difference measurements is estimated to be about 10×10^{-10}.

The transatlantic shipment of the BIPM resistors was made on 16 March to allow time for the resistances to come to equilibrium at 25 °C and to recover from shipment. When the standard resistors arrived at the NIST and were connected to their temperature controllers, the initial temperatures were below 15 °C. Only the value of the 100 Ω resistor was significantly disturbed by shipment, showing an initial relative drift rate of more than 1×10^{-7} per day, which slowly dropped to about 5×10^{-9} per day at the time of the comparison. Relative drift rates for the 10 000 Ω and 1 Ω resistors were less than 2×10^{-9} per day.

Table 1. Type B standard uncertainties for BIPM measurements.

Resistance ratio	$R_H(2)$ /100 Ω	10 kΩ /100 Ω	100 Ω/1 Ω 50 mA
Type B uncertainties in parts in 10^{10}			
Reference CCC bridge			
CCC imperfect winding ratio	10	10	10
Resistive divider calibration	5	5	8
Leakage resistances	2	2	–
Statistical standard deviation of 1 Hz ac-dc difference measurement	10	10	15
RSS total in parts in 10^{10}	15	15	20

3. Comparison results

3.1 Measurements of the 100 Ω *resistance standard in terms of* $R_{K\text{-}90}$

Measurements of the resistance, R, of the 100 Ω standard in terms of $R_H(2)$ were carried out on 15, 16 and 19 April. Figure 1 shows the measurement results of 16 April 1999. The symbols correspond to data recorded over a period of approximately 3 min or 4 min by the NIST (symbol ○), or to a measurement carried out over a period of approximately 4 min by the BIPM (symbol ●). Here the NIST calculated each point as an average of four consecutive ratios, and in the last set of NIST data shown, 6 s were added to the delay used for settling after current reversal. The three sets of measurements carried out by the NIST correspond to the three different pairs of Hall voltage contacts of the sample used. The first three sets of BIPM measurements were carried out with the first LEP sample. The fourth set of BIPM measurements was obtained with the second LEP sample. For both LEP samples two different pairs of Hall voltage contacts were used, the first half of the measurements of a set corresponding to one pair and the second half to the other pair. The NIST noted no significant difference between results obtained with different contact pairs on its sample, nor did the BIPM note any significant difference between results from different pairs or from its two samples. For both laboratories the measuring current in the QHE samples was 40 μA to within a few percent. Similar measurement sequences were carried out on 15 and 19 April 1999.

The timing of the NIST measurements could have a residual effect owing to the low-frequency ac behaviour of the 100 Ω standard resistor. Also, an error in measurement could occur if the delay after the current reversal does not allow the feedback systems enough time to settle completely. Near the end of the day on 15 April, the delay was increased from 4 s to 10 s and the data showed a significant increase in the result for the 100 Ω resistor. Subsequently, over the three days, the delay was both reduced to 3 s and increased to 10 s in several measurements, and no such change was seen. In Figure 1, the third set of data was taken with a delay

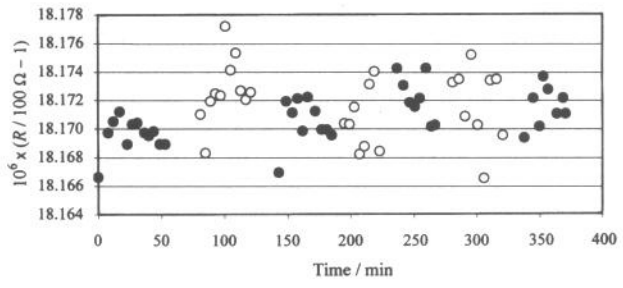

Figure 1. NIST and BIPM measurements of the 100 Ω resistance, R, in terms of $R_{K\text{-}90}$, on 16 April 1999. ●, BIPM; ○, NIST.

equal to 10 s, while the normal settling delay is 4 s, and no effect is found at the level of the random variations. To test that both the CCC SQUID magnetic detector and the nanovolt detector were maintained continuously at null output by the feedback, the NIST measured the direct output of these detectors during the comparison. The output of neither detector varied significantly upon current reversal.

A comparison result is obtained for each day by calculating the difference between the mean value of all NIST measurements and that of all BIPM measurements. The results of the three days, expressed as the relative difference between values R_{NIST} and R_{BIPM} attributed to R by the QHE standards of the two laboratories, are

$$(R_{NIST} - R_{BIPM})/R = 22 \times 10^{-10}$$
with $u_c = 25 \times 10^{-10}$ (1999-04-15),
$$(R_{NIST} - R_{BIPM})/R = 7 \times 10^{-10}$$
with $u_c = 21 \times 10^{-10}$ (1999-04-16),
$$(R_{NIST} - R_{BIPM})/R = 11 \times 10^{-10}$$
with $u_c = 20 \times 10^{-10}$ (1999-04-19).

The relative combined standard uncertainty u_c associated with each result is the square root of the sum of the squares (RSS) of the NIST and BIPM Type B standard uncertainties (10 parts in 10^{10} and 15 parts in 10^{10}, respectively; see Tables 1 and 2), of a standard uncertainty of 5 parts in 10^{10} due to residual power and temperature effects in the 100 Ω standard, and of the Type A standard uncertainty of the measurements (17 parts in 10^{10} on 15 April, 9 parts in 10^{10} on 16 April and 6 parts in 10^{10} on 19 April).

Using u_c^{-2} as the weight for each day's run, the weighted mean of the three results is

$$(R_{NIST} - R_{BIPM})/R = 12 \times 10^{-10}$$
with $u_c = 20 \times 10^{-10}$.

3.2 *Measurements of the* 10 000 Ω/100 Ω *ratio*

Measurements of the ratio, K, of the 10 000 Ω resistance to the 100 Ω resistance were carried out on 14 and 22 April. For both laboratories the measuring current in the 10 000 Ω standard was 50 μA to within a few percent. The insulation resistance of the 10 000 Ω resistor was measured on 22 April and a fractional correction of -6×10^{-10} was applied to account for the effect on the NIST CCC bridge of leakage in this resistor. The NIST measurements were made using a CCC bridge located in the NIST calibration laboratory adjacent to the QHR laboratory. This system is equipped with sets of shielded cables, 14 m long, which allow scaling comparisons to be made between the two laboratories. This eliminates the need for 100 Ω standards to be transferred between the laboratories during QHR scaling, and makes it possible to conduct frequent comparisons of 100/1 ratios.

The NIST has previously made tests indicating an effect due to capacitance between the measurement leads when using the 14 m long cables. Before starting this comparison the systematic effect was reduced to insignificant levels by increasing the speed of the SQUID feedback circuit. The basic cause of such a systematic effect is probably rectification in the SQUID, and this has not been eliminated. The corresponding uncertainty is indicated in Table 2. All of the NIST 10 000 Ω/100 Ω ratio measurements were made with a 7 s delay after current reversal.

Measurements on 14 April consisted of two sequences of data each for the BIPM and the NIST. Each BIPM data set consisted of five 4 min ratio measurements. The relative standard deviation of a single 4 min measurement was about 2×10^{-9}. Each NIST set consisted of ten ratio measurements, requiring about 90 s each. The relative standard deviation of a single measurement was between 5×10^{-9} and 8×10^{-9}. On 22 April much higher standard deviations were obtained in measurements using the same CCC bridge, with the NIST standard deviations ranging from 17×10^{-9} to 25×10^{-9}. The noisy conditions required much longer averaging times for the NIST measurements on 22 April, and may have given rise to a systematic error attributable to noise rectification in the SQUID detector.

The comparison results are

$$(K_{NIST} - K_{BIPM})/K = 23 \times 10^{-10}$$
with $u_c = 55 \times 10^{-10}$ (1999-04-14),
$$(K_{NIST} - K_{BIPM})/K = 94 \times 10^{-10}$$
with $u_c = 56 \times 10^{-10}$ (1999-04-22).

The relative combined standard uncertainty, u_c, associated with each result is the square root of

Table 2. Type B standard uncertainties for NIST measurements.

Resistance ratio	$R_H(2)$ /100 Ω	10 kΩ /100 Ω	100 Ω/1 Ω	
			50 mA	100 mA
Type B uncertainties in parts in 10^{10}				
Insulation resistance	4	4	0	0
CCC imperfect winding ratio	3	6	6	6
Null detector balance errors	4	6	12	6
SQUID rectification errors	2	50	10	5
Primary current measurement	2	8	8	6
Output signal gain measurement	3	8	8	6
QHR device imperfect quantization	7	–	–	–
RSS total in parts in 10^{10}	10	52	20	13

the sum of the squares of the NIST and BIPM Type B standard uncertainties (see Tables 1 and 2), of a standard uncertainty of 5 parts in 10^{10} arising from residual power and temperature effects in the 10 000 Ω and 100 Ω standards, and of the Type A standard uncertainty of the measurements (8 parts in 10^{10} on 14 April, 14 parts in 10^{10} on 22 April).

The mean of the two results is

$$(K_{\mathrm{NIST}} - K_{\mathrm{BIPM}})/K = 59 \times 10^{-10}$$
with $u_c = 55 \times 10^{-10}$.

3.3 *Measurements of the* 100 Ω/1 Ω *ratio*

Measurements of the ratio, K', of the 100 Ω resistance to the 1 Ω resistance were carried out on 20 and 21 April. Both the BIPM and NIST used a measuring current of 50 mA in the 1 Ω standard on the first day. On the second day, the NIST used a measuring current of 50 mA or 100 mA and no significant difference was found between values of K' measured with these two currents. The NIST used the calibration laboratory CCC system as described above, with settling delays of 7 s and 15 s in the measurements at each current level. The comparison results are

$$(K'_{\mathrm{NIST}} - K'_{\mathrm{BIPM}})/K' = 44 \times 10^{-10}$$
with $u_c = 32 \times 10^{-10}$ (1999-04-20),
$$(K'_{\mathrm{NIST}} - K'_{\mathrm{BIPM}})/K' = 32 \times 10^{-10}$$
with $u_c = 29 \times 10^{-10}$ (1999-04-21).

The relative combined standard uncertainty u_c is the square root of the sum of the squares of the NIST (20 parts in 10^{10} at 50 mA, 13 parts in 10^{10} at 100 mA) and the BIPM (20 parts in 10^{10}) Type B standard uncertainties, of a standard uncertainty of 10 parts in 10^{10} resulting from residual power and temperature effects in the resistances, and of the Type A standard uncertainty of the measurements (12 parts in 10^{10} for both measurements).

The mean of the two results is

$$(K'_{\mathrm{NIST}} - K'_{\mathrm{BIPM}})/K' = 38 \times 10^{-10}$$
with $u_c = 31 \times 10^{-10}$.

4. Conclusions

The comparison results demonstrate excellent agreement between the NIST and the BIPM for measurements of a 100 Ω resistance standard in terms of $R_H(2)$ and very good agreement for measurements of 10 000 Ω/100 Ω and 100 Ω/1 Ω ratios. In all three cases the measured differences do not significantly exceed the total standard uncertainty of the differences, 20 parts in 10^{10}, 55 parts in 10^{10}, and 31 parts in 10^{10}, respectively.

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