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TECHNICAL GUIDELINES FOR RELIABLE MEASUREMENTS OF THE QUANTIZED HALL RESISTANCE

Document prepared by
the Working Group of the Comité Consultatif d'Électricité
on the Quantum Hall Effect

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This document integrates the main information and suggestions communicated to the CCE Working Group on the Quantum Hall Effect on the subject of reliable measurements of the quantized Hall resistance R_H for realizing laboratory representations of the ohm. (Throughout, the symbol R_K is used for the $i = 1$ plateau ≅ 25 812,8 Ω[+] as suggested by the Working Group in its report to the CCE. The symbol R_H is used for the quantized Hall resistance in general, *i.e.*, for any plateau.)

Its aim is not to recommend strict rules but rather to propose guidelines to serve as a reminder of the main tests and precautions necessary to assure reliable measurements of R_H at a relative accuracy of a few parts in 10^8.

Laboratories are strongly encouraged, when reporting their results, to describe their own tests and procedures related to the possible error sources addressed in these guidelines.

[+] In keeping with the preferred ISO usage, commas are used in this document to indicate decimal fractions.

1. Sample Choice

Metal-oxide-semiconductor field effect transistors (MOSFET's) or GaAs/GaAlAs devices (and possibly alternative hetero-structures) can be used for accurate measurement of the quantized Hall resistance R_H.

GaAs/GaAlAs devices have an important advantage over MOSFET's : at a temperature of 1,5 K or lower and for magnetic flux density B in the range ~ 6 to 12 T, they can possibly tolerate, without measurable dissipation in the longitudinal direction, a "source-drain" current I_{SD} of 20 to 50 μA which is significantly higher than that of MOSFET's (10 μA maximum). This allows the reduction of the random or Type A uncertainty in the measurement of R_H to 1 part in 10^8 for a reasonable measuring time.

On the other hand, MOSFET's may have decreased leakage current between contacts [1] and long life-time [2].

Further accurate comparisons, when possible, between MOSFET's and GaAs devices should be encouraged as a test of the independence of R_H on the type of sample used [3].

In the case of GaAs/GaAlAs devices, a mobility μ of ≈ 10 to 20 T^{-1} and a carrier concentration n in the range 3 to 6 × 10^{15} m^{-2} are suitable in order to obtain wide and well quantized i = 2 plateaux for the values of temperature mentioned above and B in the range 6 to 12 T [2]. Devices with higher n (from 6 to 8 × 10^{15} m^{-2}) can also yield good quantization conditions for their i = 4 plateaux in the range 6 to 8 T.

In the case of silicon MOSFET's a mobility μ of 1,5 to 4,5 T^{-1} is suitable in order to obtain wide and well quantized i = 2, 4 or 8 plateaux at a temperature of 0,5 K or lower and for B in the range 8 to 12 T [2].

The samples should be fitted with source and drain contacts SD (gate and substrate for MOSFET's), and with at least two, preferably three, pairs of Hall voltage contacts (Fig. 1).

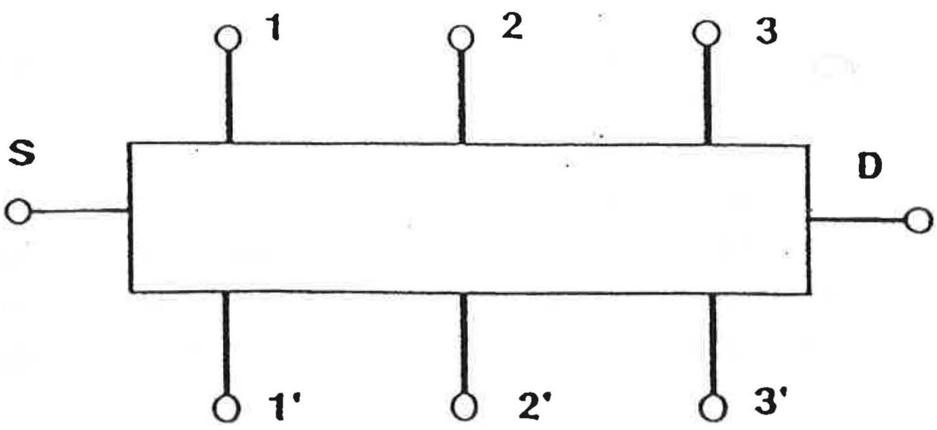

Fig. 1. Sample with three pairs of Hall voltage contacts.

2. Sample Cool-Down and Handling

Samples should be cooled slowly, in the dark and in an environment which is shielded from RF radiation.

MOSFET's should be cooled with a gate voltage applied from the very beginning of cooling or alternatively with the gate short-circuited to the source or drain contact.

Output wires attached to the sample should be handled cautiously as connecting them to accidental environmental noise sources may induce longitudinal dissipation ($\rho_{xx} \neq 0$) in a sample previously in a dissipationless state ($\rho_{xx} \cong 0$). This is particularly true for MOSFET's but has also been observed on some occasions for GaAs devices. Restoration to a dissipationless state is often possible, however, by sweeping B through zero or by cycling the device to room temperature for a short time, in the case of GaAs devices, or for several weeks, in the case of MOSFET's.

3. Contact Resistance

Poor contact resistances are often the major sample limitation encountered by metrologists. The perturbing effects of poor contacts may have, at least, the following three characteristics :

- Poor source-drain contacts induce noise in I_{SD} despite the use of a current source with a relatively high (with respect to R_H) internal impedance. This noise often makes precise measurements impossible.

- Potential contacts may themselves generate excessive voltage noise when connected to a nanovoltmeter.

- Even in the case of an acceptable voltage noise level, imperfect potential contacts can generate DC offset voltages (possibly by a process of rectification of noise) which depend on the polarity of I_{SD} and which can introduce systematic errors in measurements of R_H.

It has been observed that the contact behavior can deteriorate with time over, say, several months. In addition, if, during an experiment, the device remains at low temperature for several days, or I_{SD} is increased to a value at which the current flow is no longer dissipationless [4], deterioration may also occur. In the latter two cases restoration is often possible by cycling the device to room temperature for a period of time which may depend on the sample used.

The following tests can be used to detect imperfect contacts. It is assumed that B (or the gate voltage) is first ajusted to a value corresponding to the center of a Hall plateau of resistance R_K/i.

- The resistance between any two contacts of the sample is determined by two-terminal measurements. The measured values depend on the sample material, the material and the thickness of the contacts and on the way they are made [4a]. For example, for alloyed Sn-contacts on GaAs/GaAlAs samples, the values should be ideally within 1×10^{-4} of $R_K/i + R_L$ [5], where R_L is the resistance of the leads, and independent of current polarity. For AuGeNi contacts on GaAs/GaAlAs samples values up to about 1 kΩ have been observed with no measurable effect upon $R_K/2$ [5a]. If higher values are measured, extra precautions should be taken to verify the absence of DC offset voltages generated in the contacts [6]. In any case the two-terminal resistances of different sets of contacts on the same sample, provided they have been prepared in the same way, should not differ considerably.

The problem of possible noise contamination, mentioned above, should be kept in mind while making this test. In particular, it is not recommended to connect a MOSFET sample to a mains-operated digital ohmeter. In all cases, the value of the measuring current delivered by the ohmeter should be low enough to avoid degrading the sample.

- The voltage noise across contact pairs (with $I_{SD} = 0$) is evaluated using a nanovoltmeter with a sufficiently high input resistance (> 10 kΩ), sufficiently low offset current (< 1 pA), and sufficiently low voltage noise for source impedances of the order of 10 kΩ in the frequency band 0 to 1 Hz.

The measured noise across pairs should be less than or equal to that observed with the meter's leads across the terminals of a good quality wire-wound resistor of resistance R_K/i at room temperature. A higher level of noise may be due to poor contacts, and also possibly to microphonic noise in the leads connected to the sample.

- In the quantized regime and with a normal operating value of I_{SD}, the longitudinal voltage, V_x, between contacts on the same side of the sample, is determined for both directions ($I_{SD} = \pm I$) of the current. Both measured values should ideally be negligible within the resolution of the measuring instrument, *i.e.* :

$$\left| V_x(I_{SD} = +I) - V_x(I_{SD} = 0) \right| = \left| V_x(I_{SD} = -I) - V_x(I_{SD} = 0) \right| \cong 0.$$

This tests for the possible presence of offset voltages which depend on current polarity, and which could be caused by poor contact behavior or possibly other factors such as leakage currents.

4. Conditions of Quantization

The quantity to be measured, the quantized Hall resistance R_K/i, is believed to be the value of the Hall resistivity ρ_{xy} on a plateau of a two dimensional electron gas (2 DEG) in a dissipationless state, *i.e.*, with $\rho_{xx} = 0$, where ρ_{xx} is the longitudinal resistivity.

Under practical conditions of temperature and magnetic field, ρ_{xx} has either a finite minimum value ρ_{xx}^{min} or is "non-measurable" within the limit of resolution, which means that ρ_{xx}^{min} is lower than ≅ 0,1 to 0,5 mΩ.

Many laboratories have already investigated the possible corrections to be applied to ρ_{xy}, due to this residual value of ρ_{xx} [7, 8, 9].

4.1. Possible temperature dependence

Varying the temperature with I_{SD} held constant is an important test for the characterization of a sample. Its realization should be encouraged, at least once for a given sample.

Ideally ρ_{xy} should be invariant, within the limit of resolution of the measurements, over an appreciable range of temperature starting from the lowest temperature attainable with the cryogenic equipment used, T_1.

This is not always the case and, indeed, a sufficiently large increase in temperature produces an increase in ρ_{xx}^{min} and measurable variations of ρ_{xy}. The variation of ρ_{xy} as a function of ρ_{xx}^{min} can be quite different in magnitude, sign and character depending on the set of Hall contacts used, the magnetic field direction and the value of I_{SD}. It has been observed that often ρ_{xy} varies linearly with ρ_{xx}^{min}, at least for a limited range of temperature, and obeys the equation :

$$\Delta\rho_{xy} = \rho_{xy}(\rho_{xx}^{min}) - \rho_{xy}(0) = s\ \rho_{xx}^{min}$$

where s is a constant and $\rho_{xy}(0)$ the extrapolated value of ρ_{xy} at $\rho_{xx}^{min} = 0$, which is believed to be equal to R_K/i.

ρ_{xx}^{min} is evaluated by measuring the minimum voltage drop V_x^{min} between two V_x contacts and is given by $\rho_{xx}^{min} = \dfrac{V_x^{min}}{I_{SD}} \times \dfrac{w}{l}$ where w is the width of the sample and l the distance between the V_x contacts. (Note that this equation will always yield an approximate value for ρ_{xx} because of possible sample inhomogeneities and because w and l are never precisely defined.) The measured values of ρ_{xx}^{min} may vary for different pairs of V_x contacts. Consequently, s is a function of the chosen set of Hall and V_x contacts.

The measured values of s are usually < 1, possibly as low as 0,1. It should be remembered that s depends on the direction of B (its sign may change when the direction is reversed) and on the value of I_{SD}, as pointed out in the next paragraph on current dependence. Futhermore, the determination of s is very time-consuming and is not necessarily reproducible with thermal cycling.

As a consequence of this, a sample featuring a measurable temperature dependence of ρ_{xy} near T_1 can possibly be used for accurate measurements of R_H but only after it has been verified beforehand that s is reasonably reproducible. Futhermore, the relative value of the correction applied to ρ_{xy}, i.e., $-s\frac{\rho_{xx}^{min}}{\rho_{xy}}$, should not exceed a few parts in 10^8.

It is, of course, much better to use a sample for which ρ_{xy} is invariant with respect to a significant increase of the temperature above T_1. This is usually associated with a non-measurable value of ρ_{xx}^{min} at T_1. A knowledge of s is not necessary for such a sample, when the ρ_{xy} measurements are made at T_1.

4.2. Possible current dependence

The check of the invariance of ρ_{xy} with respect to significant changes in I_{SD} is also important as it may reveal imperfect quantization. It is also a good test to detect the possible effects of leakage currents.

It should be noted that, when observed, the current-induced variations of ρ_{xx}^{min} result in variations $\Delta\rho_{xy}$ of the Hall resistance that may differ from those associated with temperature-induced variations of ρ_{xx}^{min}. This has been observed for GaAs devices [10, 11] but not, however, in the experiments on MOSFET's described in reference [7].

For this reason I_{SD} should be held constant when ρ_{xy} and ρ_{xx}^{min} are measured at different temperatures to evaluate the slope s. This slope may depend on the value of I_{SD} used. For instance, it has been found in reference [11] that, for a particular GaAs/GaAlAs sample, the value of s measured with I_{SD} = 40 μA was approximately twice the value measured with I_{SD} = 10 μA.

4.3. Possible magnetic field (or gate voltage) dependence

The flatness of the Hall plateau should be verified, at least once for a given sample, by making measurements of ρ_{xy} not only at the center of the plateau but at a few points on either side of the center. Flatness is necessary for useful measurements but does not mean that the correction due to finite ρ_{xx}^{min} is negligible. Also, ρ_{xx}^{min} should occur at the same value of B on both sides of the sample. If ρ_{xx}^{min} does not occur at the center of the plateau, ρ_{xy} should be determined at the value of B for which ρ_{xx}^{min} occurs, but extra precautions should be taken to demonstrate the flatness of the plateau.

Another test that should be made at least once is that of the invariance of ρ_{xy} with respect to the direction of B, as failure of this test also reveals imperfect quantization.

4.4. Possible geometric dependence

To ensure that the finite aspect ratio of the quantum Hall effect device does not cause a significant error in the determination of R_H [13], R_H measurements at all three Hall contact pairs along the length of the device (or at least the pair on the center and one pair on the end) should yield the same value.

5. Measurement of R_H

All the tests mentioned above are very time-consuming and cannot be repeated each time a measurement of R_H is made.

This section suggests the minimum measurements that might be made, in one day, during a particular determination of R_H. It is assumed that the sample used has already been thoroughly characterized and is known to be usually free from significant corrections due to a finite value of ρ_{xx}^{min}, at the temperature of the measurements (usually T_1) and for the value of I_{SD} used.

5.1. Fast check of the contacts

Two-terminal measurements between contact pairs, which are not time-consuming, can easily be made before each R_H measurement. To avoid any possible problem due to noise contamination, a battery-operated ohmeter or controller, even of modest resolution, may be used.

5.2. Measurement of ρ_{xx}^{min}

ρ_{xx}^{min} should be evaluated on both sides of the channel, using two opposite pairs of V_x contacts, for instance 1-3 and 1'-3' (Fig. 1) [12]. This checks that the area of the channel delimited by these pairs has reasonably homogeneous ρ_{xx}^{min} characteristics. The measured values should ideally be limited by the measurement resolution ($\angle$ 0,2 mΩ) and at least similar to those obtained previously with this sample.

It is a good precaution to repeat the ρ_{xx}^{min} evaluation, on both sides of the sample, immediately after the precise ρ_{xy} measurements to check that no accidental change occurred during the experiment.

5.3. Measurement of ρ_{xy}

Whenever possible the ρ_{xy} measurements should be made on two different pairs of Hall contacts. These two pairs should be either those delimiting the area mentioned in 5.2 and sharing their contacts with the V_x pairs, i.e., 1-1' and 3-3', or a delimiting pair (1-1' or 3-3') and a central one (2-2'), if any. However, it may be argued that to unequivocally ensure contact reliability, the two pairs of V_x contacts should involve the same pads as the two V_H pairs. Additionally, the voltages of the two V_x pairs and those of the two V_H pairs should sum to zero around the loop (to within the random uncertainties).

Good agreement between the values of ρ_{xy} obtained with two different pairs is a confirmation that there is no significant problem due to the contacts.

6. Consistency of R_H Measurements for Different Samples and Different Quantum Numbers

A last but essential criteria for judging a particular measurement of R_H is its agreement with measurements made on other samples, preferably from different wafers, and with different quantum numbers.

Such comparisons test for shunting resistances (parallel conduction) across the sample, especially those between source and drain which are not revealed by ρ_{xx}^{min} measurements. In the case of different quantum numbers, they are also an excellent test for leakage resistances in the measuring equipment. (For example, compare R_K values as obtained from the i = 2 and i = 4 plateaux on the same sample and during the same run.)

7. Comments on the Measuring Equipment

- All electronic apparatus used in the experiment should introduce a minimum amount of extraneous electrical noise to prevent possible noise rectification and dammage to the sample.

- All the components of the equipment used to measure R_H, including the sample holder, should have leakage resistances as high as possible.

Normally 10^{12} Ω is a minimum value but for gated samples, where a relatively high voltage (compared with the Hall voltage) has to be applied to the gate, the minimum leakage resistance for lines connected to the gate should be 10^{14} Ω [10]. Other voltage sources (usually batteries) incorporated in the equipment and which may drive excessive leakage current should also have leakage resistances of the order of 10^{14} Ω.

Guarding techniques limiting the effect of the leakage currents can be designed but if care is not taken, the guard circuits themselves can inject currents into the measurement system and these are difficult to detect.

It is a necessary precaution to check the leakage resistances or currents before each precise measurement of $\underline{R}_H$, as they may deteriorate with time.

- Many laboratories use, for the first step of the scaling process from $\underline{R}_H$ to 1 Ω standards, a potentiometric method whereby $\underline{R}_H$ is placed in series with a standard resistor $\underline{R}$ having either the same nominal value as $\underline{R}_H$ or a value of 10 kΩ.

Significant "interchange errors" have been reported [10] for such potentiometers, which means that the measured ratio $\underline{R}_H/\underline{R}$ depends on the relative position of these two resistors in the series circuit.

Whenever possible, this test should be carried out and the results reported.

- If the two resistors in the series circuit, $\underline{R}_H$ and $\underline{R}$, do not have the same nominal value (for instance, if $\underline{R}$ = 10 kΩ), the voltage drops accross them are significantly different. Consequently, the linearity errors of the potentiometers used to measure them must be carefully checked. In particular, commercially available potentiometers that use a fixed internal resistor to generate the output voltage may introduce significant errors due to the power coefficient of this resistor.

Generally speaking, the effect of the power coefficient of any standard resistor used at different power levels in the course of measurements of $\underline{R}_H$ or in the scaling-down process should be checked.

Even if $\underline{R}_H$ and $\underline{R}$ differ by only a few parts in 10^6, it is necessary to calibrate the linearity of the null detector used in the measurements. Since the calibration curve for the detector is likely to depend upon time, ambient temperature, and the particular characteristics of the digital voltmeter used to read the output of the detector, its calibration requires careful attention.

8. Reporting Results

In order to compare values of R_H obtained in different laboratories, the assigned uncertainties must be estimated in a similar manner. This should be done following Recommendation INC-1 (1980) of the CIPM Working Group on the Statement of Uncertainties [14] and Recommendation 1 (CI-1986) of the CIPM [15] advocating that the uncertainties be expressed as one-standard-deviation estimates. In particular, a detailed and complete listing of the Type A and Type B uncertainties should be given, along with measurement dates, the number of measurements made, a clear statement as to the units in which the result is being reported, and other useful information. As noted above, special attention should be given to describing the characteristics of the devices used and the tests and procedures employed to address the possible sources of error discussed in these guidelines.

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