IEC TS 62607-6-7

Edition 1.0 2023-06

TECHNICAL SPECIFICATION

colour
inside

Nanomanufacturing – Key control characteristics –
Part 6-7: Graphene – Sheet resistance: van der Pauw method

IEC TS 62607-6-7:2023-06(en)

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Part 6-7: Graphene – Sheet resistance: van der Pauw method

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 07.120

ISBN 978-2-8322-7114-8

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-7: Graphene – Sheet resistance: van der Pauw method

FOREWORD

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IEC TS 62607-6-7 has been prepared by IEC technical committee 113: Nanotechnology for electrotechnical products and systems. It is a Technical Specification.

The text of this Technical Specification is based on the following documents:

Draft	Report on voting
113/682/DTS	113/713/RVDTS

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this Technical Specification is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

A list of all parts of the IEC TS 62607 series, published under the general title *Nanomanufacturing – Key control characteristics,* can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

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INTRODUCTION

Graphene is a single layer of carbon atoms arranged in a honeycomb lattice. Graphene has shown many outstanding properties, among which is a high electrical conductivity. Nowadays graphene can be easily grown and transferred on large area (cm^2 to even m^2) and even roll-to-roll supports using chemical vapour deposition (CVD) techniques. This is already enabling its commercial applications in electrotechnical products.

Electrical conductivity of graphene samples can depend on many factors: structural quality, contamination, coupling with the physical support used for a given application to name a few. On practical grounds, sheet resistance is a quantity which can be used as global measure of the local conductivity of a sample with finite geometrical dimensions. In order to check the reproducibility of the electrical properties of graphene, the sheet resistance is clearly a key control characteristic for this material.

The van der Pauw method [1][1] allows the measurement of the sheet resistance of samples of arbitrary shape, with isotropic conductivity and uniform carrier density by performing a pair of four-terminal resistance measurements with electrical contacts placed at arbitrary positions on the sample's perimeter. The method is fast (it takes a few minutes) and easy to implement, since many commercial fixtures are available.

The four-terminal resistance measurements required to apply the method allow to minimize the effect of the contact resistance that appears between graphene and the measurement probes.

The van der Pauw method does not provide any spatial resolution in principle, but considerations about real samples' conductivity uniformity can be made.

In this document it is explained how to specifically apply the van der Pauw method on chemical vapour deposited graphene on rigid insulating support and perform a reliable estimation of the sample sheet resistance also considering the non-ideal nature of commercial graphene.

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[1] Numbers in square brackets refer to the Bibliography.

NANOMANUFACTURING –
KEY CONTROL CHARACTERISTICS –

Part 6-7: Graphene – Sheet resistance: van der Pauw method

1 Scope

This part of IEC TS 62607 establishes a method to determine the key control characteristics

- sheet resistance R_S [measured in ohm per square (Ω/sq)],

by the

- van der Pauw method, vdP.

The sheet resistance R_S is derived by measurements of four-terminal electrical resistance performed on four electrical contacts placed on the boundary of the planar sample and calculated with a mathematical expression involving the two resistance measurements.

- The measurement range for R_S of the graphene samples with the method described in this document goes from 10^{-2} Ω/sq to 10^4 Ω/sq.
- The method is applicable for CVD graphene provided it is transferred to quartz substrates or other insulating materials (quartz, SiO_2 on Si), as well as graphene grown from silicon carbide.
- The method is complementary to the in-line four-point-probe method (4PP, IEC 62607-6-8) for what concerns the measurement of the sheet resistance and can be applied when it is possible to reliably place contacts on the sample boundary, avoiding the sample being scratched by the 4PP.
- The outcome of the van der Pauw method is independent of the contact position provided the sample is uniform, which is typically not true for graphene at this stage. This document considers the case of samples with non-strictly uniform conductivity distribution and suggests a way to consider the sample inhomogeneity as a component of the uncertainty on R_S.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1 General terms

3.1.1

graphene
graphene layer
single-layer graphene
monolayer graphene

single layer of carbon atoms with each atom bound to three neighbours in a honeycomb structure

Note 1 to entry: It is an important building block of many carbon nano-objects.

Note 2 to entry: As graphene is a single layer, it is also sometimes called monolayer graphene or single-layer graphene and abbreviated as 1LG to distinguish it from bilayer graphene (2LG) and few-layer graphene (FLG).

Note 3 to entry: Graphene has edges and can have defects and grain boundaries where the bonding is disrupted.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.1]

3.1.2

bilayer graphene
2LG

two-dimensional material consisting of two well-defined stacked graphene layers

Note 1 to entry: If the stacking registry is known, it can be specified separately, for example, as "Bernal stacked bilayer graphene".

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.6]

3.1.3

few-layer graphene
FLG

two-dimensional material consisting of three to ten well-defined stacked graphene layers

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.10]

3.2

key control characteristic
KCC

product characteristic which can affect safety or compliance with regulations, fit, function, performance, quality, reliability or subsequent processing of the final product

Note 1 to entry: The measurement of a key control characteristic is described in a standardized measurement procedure with known accuracy and precision.

Note 2 to entry: It is possible to define more than one measurement method for a key control characteristic if the correlation of the results is well-defined and known.

[SOURCE: IEC TS 62565-1:2023, 3.1]

3.3
blank detail specification
BDS
structured generic specification providing a comprehensive set of key control characteristics which are needed to describe a specific product without assigning specific values or attributes

Note 1 to entry: Examples of nano-enabled products are: nanocomposites and nano-subassemblies.

Note 2 to entry: Blank detail specifications are intended to be used by industrial users to prepare their detail specifications used in bilateral procurement contracts. A blank detail specification facilitates the comparison and benchmarking of different materials. Furthermore, a standardized format makes procurement more efficient and more error robust.

[SOURCE: IEC TS 62565-1:2023, 3.2]

3.4
detail specification
DS
specification based on a blank detail specification with assigned values and attributes

Note 1 to entry: The characteristics listed in the detail specification are usually a subset of the key control characteristics listed in the relevant blank detail specification. The industrial partners define only those characteristics which are required for the intended application.

Note 2 to entry: Detail specifications are defined by the industrial partners. Standards development organizations will be involved only if there is a general need for a detail specification in an industrial sector.

Note 3 to entry: The industrial partners may define additional key control characteristics if they are not listed in the blank detail specification.

[SOURCE: IEC TS 62565-1:2023, 3.3]

3.5 Key control characteristics measured in accordance with this standard

3.5.1
sheet resistance
R_S
electrical resistance of a conductor with a square shape (width equal to length) and thickness significantly smaller than the lateral dimensions (thickness much less than width and length)

Note 1 to entry: There is no definition of the unit ohm per square (Ω/sq) in the International System of units (SI). Nevertheless, R_S is a normalized quantity, in which the symbol represents the SI ohm. So there is no ambiguity concerning the traceability of measurements of R_S to the SI, provided the measurements are performed with calibrated instrumentation.

[SOURCE: IEC TS 61836:2007, 3.4.79, modified – The entry has been adapted to this document.]

3.5.2
drift mobility

μ

<of a charge carrier> quotient of the modulus of the mean velocity of the charge carriers in the direction of an electric field by the modulus of the field strength

Note 1 to entry: The SI unit of mobility is $\text{cm}^2/\text{V s}$.

Note 2 to entry: The drift mobility is here considered to be the fundamental, intrinsic (local) property. The Hall and field effect mobility are then the extrinsic (sample) electrical measurements, carried out to determine the intrinsic mobility.

Note 3 to entry: The drift mobility for electrons and holes can be very different, depending on the residual doping and scattering mechanisms for the given sample.

[SOURCE: IEC 60500-521:2002, 521-02-58, modified – The entry has been adapted to this document.]

3.6 Terms related to the measurement method

3.6.1
four-point probe method

4PP

method to measure electrical sheet resistance of thin films that uses separate pairs of current-carrying and voltage-sensing electrodes

Note 1 to entry: The method is local with a characteristic length scale defined by the probe distance, and generally requires the resistivity variations to be on a much larger scale than the probe spacing. Depending on the positions of the sample-probe contact of the four probe contacts with the surface, different geometrical factors need to be used to extract the sheet resistance.

[SOURCE: ISO/TS 80004-13:2017, 3.3.3.1, modified – The entry has been adapted to this document.]

3.6.2
in-line four-point probe method

type of four-point probe measurement where four-point electrodes are aligned in a row

Note 1 to entry: In this method, four probes contact the test sample in a linear arrangement. A voltage drop is measured between the two inner probes while a current source supplies current through the outer probes.

Note 2 to entry: The distance between the probes needs to be small compared to the lateral dimensions of the sample so that edge effects on the electric field in the sample can be neglected.

Note 3 to entry: The resistance of the sample can be calculated by Ohm's law. Geometrical factors can be used for corrections if the sample is too small or if the measurement is performed close to the edges of the sample.

[SOURCE: IEC TS 62607-6-9:2022, 3.2.3, modified – Note 2 to entry has been deleted.]

3.6.3
van der Pauw method

vdP

type of four probe measurement for samples of arbitrary shape

Note 1 to entry: The van der Pauw method requires four probes placed arbitrarily around the perimeter of the sample, in contrast to the linear four-point probe which is placed on the top of the sample.

Note 2 to entry: The van der Pauw method provides an average sheet resistivity of the sample.

[SOURCE: IEC TS 62607-6-9:2022, 3.2.4, modified – Notes 1 and 4 to entry have been deleted.]

4 General

4.1 Measurement principle

The measurement principle of the vdP method for the assessment of the sheet resistance R_S of graphene films is based on the measurement of two four-terminal resistances, or transresistances, performed on four contacts on the sample boundary. A mathematical theorem, derived from conformal mapping theory, relates the transresistance of the sample measured in two different configurations with its sheet resistance R_S [1]. The problem is practically solved with an explicit formula that has as input the two measured four-terminal resistances and as output R_S. The method is not sensitive to the sample shape and contacts positioning in principle; corrections for real cases exist.

4.2 Non-uniform samples

The van der Pauw method assumes homogeneous samples with uniform conductivity distribution. If these conditions are met, any measurement performed with the contacts in arbitrary positions along the sample's boundary should be identical within the measurement uncertainty. If the sample is not uniform the result of a single vdP measurement can substantially depend on the contacts position [2]. Since commercial graphene samples can be far from having a uniform conductivity, it is possible that the user is in this second scenario. This document gives indications to take into consideration this variability in the measurements of R_S which can be larger than the electrical measurements uncertainty itself and to provide a consistent expression of uncertainty on R_S.

4.3 Sample preparation method

The sample is measured as it is delivered by the supplier. No special sample preparation is required. An insulating, planar material shall support the graphene. The sample shall be stored at the ambient conditions of 4.5 prior to the measurements.

4.4 Description of measurement equipment

The measurement setup for the measurement of R_S with the vdP method consists of:

- a source of DC current;
- a voltmeter;
- a contact fixture and sample holder;
- an optional switching system for measurement automation;
- an optional acquisition and data processing system.

A block schematic diagram of the typical vdP system is shown in Figure 1. The switch connects the current source to one pair of contacts, and the voltmeter to the other pair.

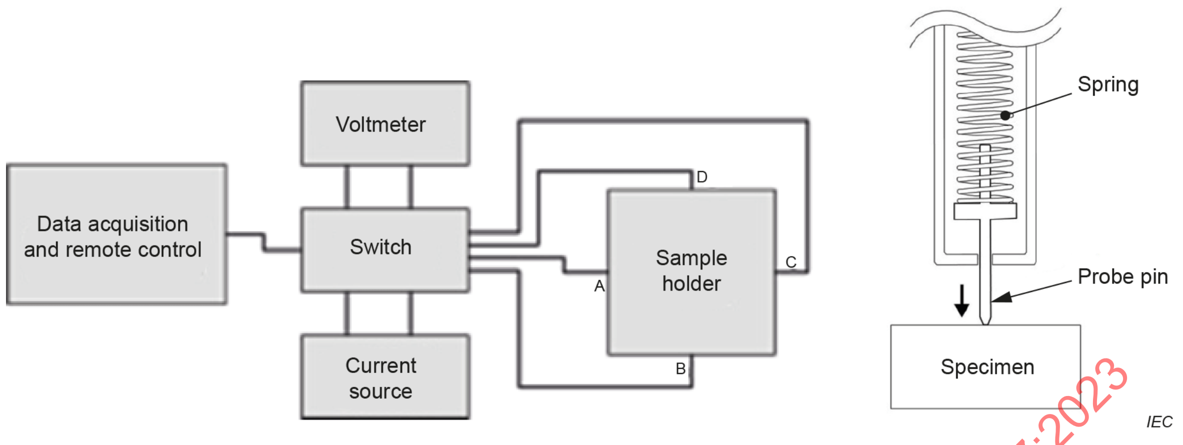


Figure 1 – Schematic view of a van der Pauw measurement setup, and a detail of typical spring-mounted probe

The current generator shall be able to supply current at least in the range 1 µA to 1 mA. The voltmeter shall be able to measure voltage at least in the range 1 mV to 1 V, considering typical five- or six-digit commercial instruments.

Furthermore, the system shall include a fixture to properly hold the sample and place the probes in position.

The sample holder fixture shall have purely mechanical contacts. This to avoid additional lithographic steps that inevitably would contaminate the sample surface, which could possibly alter its electrical properties. Contacts probe shall be adjustable in position and height. The probes shall have a very limited lateral swing in order not to scratch the graphene sample when placed in contact. If a custom fixture is used, the insulation resistance between the contacts shall be the highest possible, possibly of the order of 10^{10} Ω.

Point probes shall have internal springs to keep appropriate contact. An example of the internal structure around the probe pin is shown in Figure 1. Resistance is measured by pushing the probe in the direction of the arrow. A spring is installed in the root of the probe pin, and the force applied to the pin shall be controlled by an appropriate choice of the components or the commercial fixture.

The switch shall be a 4×4 matrix able to connect each terminal of the source and meter to each of the contacts on the sample.

4.5 Ambient conditions during measurement

Being a carbon monolayer, graphene electrical properties can be strongly affected by ambient conditions with strong variations induced by specific sample characteristics due to the growth process (see Annex A for details).

In order to obtain results which can be reasonably compared with other measurements performed in accordance with this document, the required ambient conditions are T = (23 ± 1) °C, RH = (50 ± 4) %.

4.6 Related standards

Annex C includes a list of related standards published by other standards development organizations.

5 Measurement procedure

5.1 Calibration of measurement equipment

The current source and the voltmeter shall be calibrated and within the specified calibration period.

5.2 Detailed protocol of the measurement procedure

A detailed example of the application of this procedure is given in Annex B.

Turn on the equipment for the time indicated in manuals before using it. Instrumentation shall be left to warm up in accordance with specifications.

Clean the measurement probes following the manufacturer instructions. If no indication is given, it is suggested to lower multiple times the probes on a soft cloth patch moistened with ethanol, placed at the sample seat.

Then place the sample in the proper position under the probes. If possible, place the contacts in positions which provide axial- or centre-symmetry with respect to the shape of the sample (the latter case is represented by Figure 2, with contacts on the edges; but placing contacts on the corner represents an equivalent solution). This is helpful to make some simplifications in the expression of uncertainty (discussed in Clause 6).

Measurements proceed in accordance with the following quantity definitions:

I_{ij}: current applied to a pair of contacts entering contact i, and exiting at contact j.

V_{kl}: voltage measured between contacts k and l.

$R_{ij,kl}$: four-terminal resistance calculated as I_{ij}/V_{kl}.

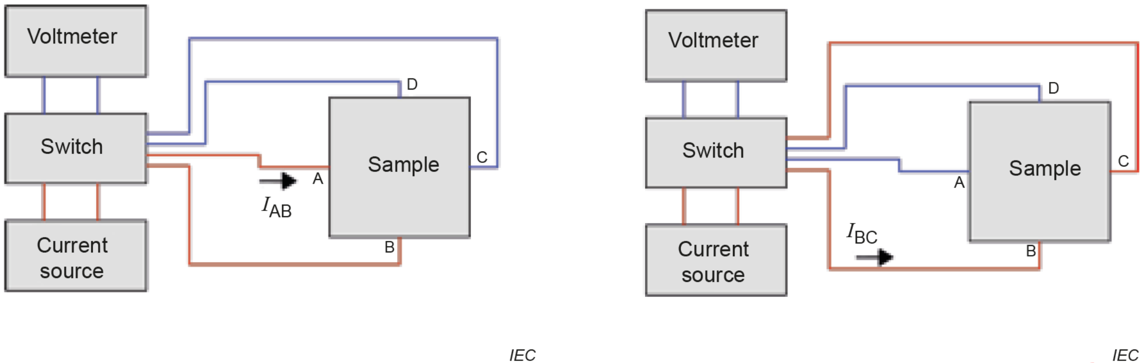
In the following, the four contacts are called A, B, C, and D. The contacts shall be placed on the sample in counter-clockwise order along the sample boundary (see contact labels in Figure 1 and Figure 2).

The transresistance measurements to be performed are

$$R_{\mathrm{AB,CD}} = \frac{V_{\mathrm{CD}}}{I_{\mathrm{AB}}} \tag{1}$$

$$R_{\mathrm{BC,DA}} = \frac{V_{\mathrm{DA}}}{I_{\mathrm{BC}}} \tag{2}$$

These are measured following the sequence reported in Figure 2 a) and b), respectively.



a) **for $R_{AB,CD}$** **b)** **for $R_{BC,DA}$**

Red leads carry the current I_{ij}, blue ones are used for the measurement of the corresponding V_{kl}.

Figure 2 – Schematic view of a typical vdP measurement setup and measurement sequence

The pair of transresistances $R_{\mathrm{AB,CD}}$ and $R_{\mathrm{BC,DA}}$ allow to calculate the sheet resistance R_{S} of the sample in the measurement configuration "p". Following the example of Figure 2, in the case of fixed contacts and a square sample, there are four possible measurement configurations p that can be obtained by rotating the sample by 90°.

As discussed in 4.2, even though the vdP method does not require the measurements to be performed in more than one configuration, in order to achieve a better estimation of R_{S}, considering possible sample inhomogeneity, more measurements should be performed, each in a different measurement configuration p.

The different pairs p of four-terminal resistance measurements labelled $R^{p}_{\mathrm{AB,CD}}$ and $R^{p}_{\mathrm{BC,DA}}$ can be stored to calculate the corresponding values of $R_{\mathrm{S}}(p)$.

5.3 Settings and precautions for the measurement of $R_{ij,kl}$

- The current source and the voltmeter shall be set in manual range mode[2].
- The magnitude of I_{ij} is a trade-off between the need to achieve a V_{kl} magnitude that can be measured with enough accuracy by the voltmeter, and the need to avoid sample heating on the contact point. Heating effects can be identified, by performing measurements at two different current levels (e.g. I and $2I$) and comparing the measurement outcome. High contact resistance can produce sample heating even at low currents. Heating of the sample exposed to the laboratory ambient can also cause sample oxidation which could alter irreversibly the sample's properties [3], [4].
- A too large magnitude of I_{ij} can also cause an abrupt breakdown of the sample. It has been reported that, for CVD graphene samples having sheet resistance in the range of 1 000 Ω the breakdown current is in the range of 10^7 A/cm^2 [3], [4]. A 100 μA current injected through a 50-μm-diameter needle probe would produce a current density of 4×10^4 A/cm^2, well below the reported breakdown level.
- The current source shall be switched on with zero current level before contacting the sample to avoid any voltage transient that could damage the sample.

---

[2] Instrumentation set "auto range" mode limits the expression of the type-B uncertainty (see Table 1), which is mandatory in precision measurements. In fact, if a source or measurement value magnitude is at the threshold between two instrument's ranges, electrical noise can induce abrupt switching between different ranges during the same measurement to which can correspond different accuracy specifications.

- Measurement accuracy is improved by performing two readings of $R_{ij,kl}$ with two currents equal in nominal magnitude and opposite in sign, and by averaging the results.
- Measurement accuracy is improved, and type A contribution to measurement uncertainty estimated, by recording time series of individual measurements and computing averages and standard deviations.

NOTE For a symmetric sample, in principle $R_{\mathrm{AB,CD}} = R_{\mathrm{BC,DA}}$, hence only a single measurement would be sufficient (e.g. $R_{\mathrm{AB,CD}}$). In practice, samples are never perfectly symmetric in shape and concerning the position of the contacts; therefore, a two-measurement protocol is suggested in the following.

5.4 Four-terminal resistance measurement accuracy

The accuracy of the sheet resistance calculated with the vdP method depends on the accuracy of the pair of four-terminal resistance measurements $R_{\mathrm{AB,CD}}$ and $R_{\mathrm{BC,DA}}$. For the definition of uncertainty and related terms used in the following, see [5], sec. 2.3.

The combined uncertainty u_{R_S} is obtained from the type-A and type-B uncertainty components of the measurement of $R_{ij,kl}$.

$$u_{R_{ij,kl}} = \sqrt{\left(u_{\mathrm{A},R_{ij,kl}}\right)^2 + \left(u_{\mathrm{B},R_{ij,kl}}\right)^2} \qquad (3)$$

Type-A uncertainty $u_{\mathrm{A},R_{ij,kl}}$ or standard uncertainty is in this case the standard deviation of the repeated series measurement. The type-B uncertainty component $u_{\mathrm{B},R_{ij,kl}}$ is calculated from the instruments' specifications and will depend on the absolute values of the measured transresistance $R_{ij,kl}$. In Table 1 are reported possible transresistance values and the current and voltage range needed to measure them with standard instrumentation. CVD graphene typically shows a sheet resistance within the range of 10 Ω to 100 Ω.

Table 1 – Example of measurable values for R_S, and the corresponding measurement settings and type-B uncertainty, when using a current source Keithley 2602B System SourceMeter® and a HP 34420 Nano Volt / Micro Ohm Meter[3] (1y calibration specifications)

Resistance, R	Current applied	Current range	Voltage measured	Voltage range	Relative type-B uncertainty of
Ω	A	µA	V	mV	R_S
1E-02	1,00E-04	100	1E-06	1	1,19E-02
1E-01	1,00E-05	10	1E-06	1	9,79E-03
1E+00	1,00E-05	10	1E-05	1	1,43E-03
1E+01[a]	1,00E-05	10	1E-04	1	5,93E-04
1E+02[a]	1,00E-05	10	1E-03	10	5,14E-04
1E+03	1,00E-05	10	1E-02	100	5,18E-04
1E+04	1,00E-05	10	1E-01	1 000	5,18E-04
1E+05	1,00E-06	1	1E-01	1 000	9,18E-04

[a] Typical graphene sheet resistance is within the range of 10 Ω to 100 Ω.

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[3] "2602B System SourceMeter®" and "HP 34420 Nano Volt / Micro Ohm Meter" are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of these products.

6 Data analysis and interpretation of results

6.1 General

6.1.1 Calculation of R_S

The sheet resistance $R_S(p)$ in each measurement configuration p, is calculated from $R^p_{AB,CD}$ and $R^p_{BC,DA}$ by means of the formula

$$R_S(p) = \frac{\pi}{\ln 2} \frac{R^p_{AB,CD} + R^p_{BC,DA}}{2} f \tag{4}$$

where then f is a numerical factor, function of the ratio $R_{AB,CD}/R_{BC,DA}$, defined in [1], and reported in table in ref [6], for which $f = 1$ holds when $R_{AB,CD}/R_{BC,DA} = 1$.

If more measurement configurations p were achievable, the corresponding values of $R_S(p)$ can be calculated separately following (4) and then R_S can be defined as

$$R_S \equiv \langle R_S \rangle_p = \frac{1}{p} \sum_p R_S(p) \tag{5}$$

where $\langle R_S \rangle_p$ is the configurational average of $R_S(p)$.

6.1.2 Further corrections

Depending on the contacts position (distance from physical edge) and size, further corrections can be applied [7], [8], [9], and if applied these shall be explicitly reported in the test report.

As an example of an acceptable non-ideal experimental situation, consider that for a square sample with contacts placed at the four corners for which

- contacts are placed within a distance d from the border for which the ratio d/D is 0,05, where D is the lateral size of the sample,
- the size of the contacts is such that the ratio β/D < 0,1, where β is the contact lateral size and D is the lateral size of the sample,

no further corrections need to be calculated, because their entity becomes negligible compared to i) the instrumental accuracy given in Table 1 and to ii) the possible variability between different $R_S(p)$. The above conditions are very easy to meet with commercial needle probe contacts.

6.1.3 Expression of uncertainty on R_S

In the general case, the uncertainty is calculated following the formula for the propagation of uncertainty [5]:

$$u_{g(x_i)} = \sqrt{\sum_i \left(\frac{\partial g(x_i)}{\partial x_i} u_{x_i} \right)^2} \tag{6}$$

where $g(x_i)$ is a function of several variables x_i, in this case the function in Formula (4), with relative uncertainty u_{x_i}. The factor f in Formula (4) is an implicit function of $R_{\mathrm{AB,CD}}$ and $R_{\mathrm{BC,DA}}$, and as a consequence, the application of Formula (6) to Formula (4) is not straightforward. Formula (6) can be more easily applied to the implicit form, equivalent to Formula (4), of the van der Pauw relation

$$e^{-\pi \frac{R_{\mathrm{AB,CD}}}{R_{\mathrm{S}}}} + e^{-\pi \frac{R_{\mathrm{BC,DA}}}{R_{\mathrm{S}}}} = 1 \tag{7}$$

Applying Formula (6) to Formula (7) gives

$$u_{R_{\mathrm{S}}} = K \times \sqrt{\left(e^{-\pi \frac{R_{\mathrm{AB,CD}}}{R_{\mathrm{S}}}} \times u_{R_{\mathrm{A}}} \right)^2 + \left(e^{-\pi \frac{R_{\mathrm{BC,DA}}}{R_{\mathrm{S}}}} \times u_{R_{\mathrm{B}}} \right)^2} \tag{8}$$

where

$$K = \frac{R_{\mathrm{S}}}{e^{-\pi \frac{R_{\mathrm{AB,CD}}}{R_{\mathrm{S}}}} + R_{\mathrm{B}} \times e^{-\pi \frac{R_{\mathrm{BC,DA}}}{R_{\mathrm{S}}}}}\ .$$

If the sample is contacted in symmetric positions, and $R_{\mathrm{AB,CD}} = R_{\mathrm{BC,DA}}$ then Formula (8) simplifies to

$$u_{R_{\mathrm{S}}} = \frac{\pi}{2\log(2)} \cdot \sqrt{\left(u_{R_{\mathrm{A}}} \right)^2 + \left(u_{R_{\mathrm{B}}} \right)^2} \tag{9}$$

which naturally follows from Formula (4) when $f = 1$ and Formula (6) is applied.

In the general case of $R_{\mathrm{AB,CD}} / R_{\mathrm{BC,DA}} \neq 1$, Formula (8) shall be used. Consider though that if $R_{\mathrm{AB,CD}}$ and $R_{\mathrm{BC,DA}}$ are equal within 1 % the uncertainty on R_{S} can be calculated with the simplified Formula (9) because f of Formula (5) is equal to 1 within 0,001 % [10].

An additional observation can be made if the contacts A, B, C, D are placed in symmetric positions (e.g. on the four corners of a square sample, as suggested in 5.2). A value of $f < 1$ in this case indicates a substantial non-uniform conductivity distribution.

6.2 One measurement configuration

If only one measurement configuration is available ($p = 1$), the uncertainty $u_{R_{\mathrm{S}}}$ of $R_{\mathrm{S}} \equiv R_{\mathrm{S}}(1)$ will be estimated only on the basis of the measurement accuracy of $R_{\mathrm{AB,CD}}$ and $R_{\mathrm{BC,DA}}$.

6.3 Multiple measurement configurations

If more measurement configurations p are available, the uncertainty of each of the $R_\mathrm{S}(p)$ can be expressed following Formula (8) or Formula (9), then these will be combined with the standard deviation $s(<R_\mathrm{S}>_p)$ of the configurational average $<R_\mathrm{S}>_p$ of Formula (5). The uncertainty u_{R_S} on $R_\mathrm{S} \equiv <R_\mathrm{S}>_p$ is then

$$u_{R_\mathrm{S}} = \sqrt{s^2 + \sum_p u\left(R_\mathrm{S}(p)\right)^2} \tag{10}$$

Note that if $s < \sum_p u\left(R_\mathrm{S}(p)\right)$ the sample is reasonably uniform (i.e. the value of R_S does not substantially depend on the contact configuration, as assumed for uniform samples by the vdP method).

7 Results to be reported

7.1 Cover sheet

The results of the measurement shall be documented in a measurement report, including the date and time of the measurement as well as the name and signature of the person responsible for the accuracy of the report.

7.2 Sample identification

The report shall contain all information to identify the test sample and trace back the history of the sample.

- General procurement information, in accordance with the relevant blank detail specification.
- General material description in accordance with the relevant blank detail specification:
  - top view, indicating the inspected area and location of the measurement positions;
  - cross-section schematic diagram, showing the layer structure (e.g. graphene on SiO_2 or graphene on quartz).

NOTE A blank detail specification for graphene is under development (IEC 62565-3-1).

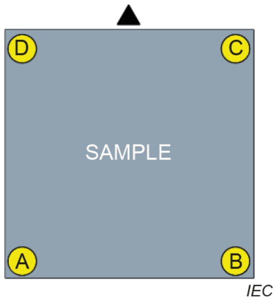
7.3 Measurement conditions

The ambient conditions during the test should be the usual ones in a calibration laboratory, and shall be reported, for example:

- Temperature range: T = (23 ± 1) °C
- Relative humidity range: RH = (50 ± 4) %

7.4 Measurement results

- Sampling plan used: Sample orientation, contacts positions and indexing and number of repeated measurements of $R_{ij,kl}$ need to be defined prior to the measurements and recorded, as shown in Figure 3.



Grey area represents the graphene sample on an insulating rigid support. Yellow dots represent the position of electrical contacts. Triangle marker indicates sample orientation.

Figure 3 – Schematic representation of a possible sampling plan representation for the vdP method

For each orientation of the sample with respect to the contact fixture, an index $p = 1,\dots,n$ shall be assigned to each measurement configuration.

- Results of $R_{\mathrm{AB,CD}}(p)$ and $R_{\mathrm{BC,DA}}(p)$ obtained in accordance with this document:

p	$R_{\mathrm{AB,CD}}$ Ω	$u(\mathrm{A},R_{\mathrm{AB,CD}})$ Ω	$u(\mathrm{B},R_{\mathrm{AB,CD}})$ Ω	$u(R_{\mathrm{AB,CD}})$ Ω	$R_{\mathrm{BC,DA}}$ Ω	$u(\mathrm{A},R_{\mathrm{BC,DA}})$ Ω	$u(\mathrm{B},R_{\mathrm{BC,DA}})$ Ω	$u(R_{\mathrm{BC,DA}})$ Ω
1	…	…	…	…	…	…	…	…
2	…	…	…	…	…	…	…	…
…	…	…	…	…	…	…	…	…

- Results of $R_{\mathrm{S}}(p)$ obtained in accordance with this document:

p	$R_{\mathrm{S}}(p)$ Ω	**Type A** $u(R_{\mathrm{S}}(p))$ Ω	**Type B** $u(R_{\mathrm{S}}(p))$ Ω	**Combined** $u(R_{\mathrm{S}}(p))$ Ω
1	…	…	…	…
2	…	…	…	…
…	…	…	…	…

- Results of R_{S} measured in accordance with this document:
  1) If only one value of $R_{\mathrm{S}}(p)$ is available, then $R_{\mathrm{S}} = R_{\mathrm{S}}(p)$ with combined uncertainty calculated following Formula (8) or Formula (9).
  2) If results obtained in more than one measurement configuration p are available, then $R_{\mathrm{S}} =< R_{\mathrm{S}}(p) >_p$ with combined uncertainty calculated following Formula (8) or Formula (9) and Formula (10).

Optionally, a table with a summary of the uncertainty contributions can be placed as follows:

s Ω	$u(R_{\mathrm{S}}(1))$ Ω	$u(R_{\mathrm{S}}(2))$ Ω	$u(R_{\mathrm{S}}(3))$ Ω	$u(R_{\mathrm{S}}(4))$ Ω
…	…	…	…	…

Annex A
(informative)

Effects of ambient conditions on graphene resistance measurements

A.1 General

A large body of literature reports small temperature dependence and large humidity dependence of graphene conductivity.

A.2 Temperature (T)

CVD graphene shows a rather low dependence of resistivity on the temperature at low temperature (room temperature included). The temperature coefficient of CVD graphene resistivity transferred on SiO_2 is typically small, with experimentally reported changes of about 10 % in the wide temperature range 1 K to 300 K, which roughly translates in a temperature coefficient of 10^{-2} %/K [11], [12]. Anyway, the explicit relation in different temperature ranges is governed by different phenomena which depend on the sample characteristics such as carrier density, defect types and density, coupling with the support (see [13] parts 2-a and 2-b and references).

A.3 Relative humidity (RH)

Even restricting the discussion to the scope of this document (CVD graphene on insulating support), it is well documented how the resistivity change induced by adsorption of water on graphene can substantially differ from sample to sample due to local morphological and chemical properties of the sample itself and to the coupling with the substrate.

Wrinkles, grain boundaries, multi-layer seeds, defects, residue of fabrication chemicals, and other factors can strongly affect the interaction between water and charge carriers in graphene, leading to many different situations, as reported in the literature [14], [15], [16].

Typical experimental results show that the resistivity of CVD graphene SiO_2 is substantially affected by adsorbed water in the range 30 % to 70 % RH, with increasing conductivity with adsorbed water concentration on graphene. This change can easily be 10-fold depending on the sample properties.

The mechanism underpinning graphene's sensitivity to humidity is reported to be the result of the electrostatic interaction between the water and the graphene-support system [15], [17], [18].

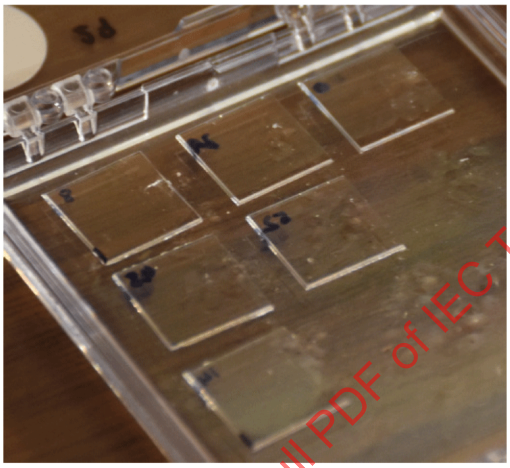
Moreover, it has been observed that in presence of graphene characterized by increasing amount of multi-layer seeds or islands, increasing, decreasing and the non-monotonous behaviours of resistance are possible depending on graphene structures. In particular, adsorption of water molecules at grain boundary defects or at the edge of multi-layer type defects can switch the water-resistivity relation [19].

Annex B
(informative)

Experimental example

B.1 Sample

The graphene sample used in this example is a square die of lateral size a = 10 mm cut from a four-inch wafer of CDV graphene transferred on quartz, provided by Graphenea SA. The measured sample was taken from the set shown in Figure B.1.



The samples are squares of lateral size 10 mm.

SOURCE: EMPIR 16NRM01 GRACE consortium. Reproduced with permission.

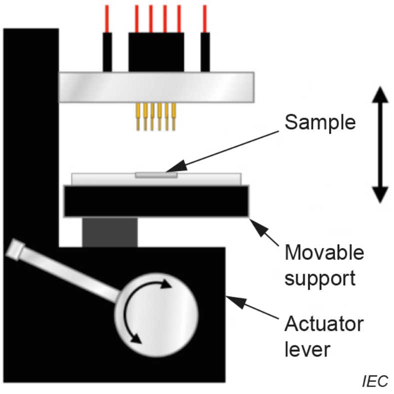
Figure B.1 – CVD graphene on quartz

B.2 Ambient conditions

Ambient conditions during the test were compatible with those reported in 7.3.

B.3 Instrumentation

Measurements were performed using a custom multi-terminal sample fixture of which a schematic view is shown in Figure B.2. See [20] for more detailed description.



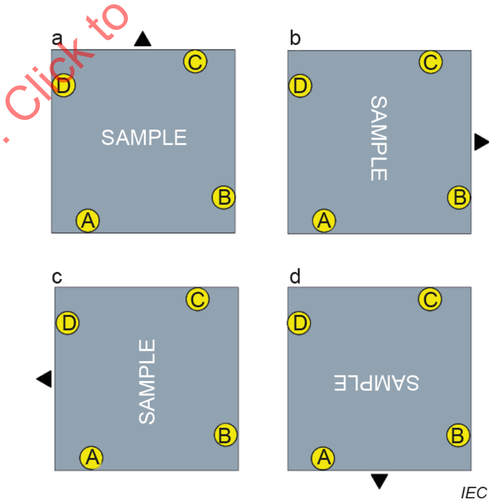
SOURCE: Cultrera, A. et al (2019) [20], modified. Reproduced with permission.

Figure B.2 – Schematic lateral view of the multi-terminal fixture

A Keithley 2602B was used as a current source. Given the very good stability of this instrument the source current I_{ij} was treated as nominal. An HP 34461 voltmeter was used for the measurement of the voltages V_{kl}. The four-terminal resistance measurements were performed with the circuit of Figure 1, consisting of the current source and the voltmeter connected to the electrode array of the contact fixture through a relay scanner.

B.4 Sampling plan

The sample was measured averaging $R_{ij,kl}$ 10 times for each of the four p measurement configurations corresponding to the four possible rotations of the sample on the used contact fixture shown in Figure B.3 a), b), c) and d). The 10 repetitions of the transresistance measurements in each configuration account for the measurement repeatability, while spatial variability is taken into account by the four sample orientations.



Four sample orientations correspond to the measurement's configurations in subfigures a), b), c) and d).

The triangular marker represents the sample orientation with respect to the contacts array.

Figure B.3 – Sampling plan used for the present example

B.5 Measurement procedure

Instrumentation was switched on 2 h prior to the measurements. Current source K2602B output was set at 0 A. The sample was then contacted in the first sampling position (Figure B.3 a). Transresistance measurements $R_{\mathrm{AB,CD}}$ and $R_{\mathrm{BC,DA}}$ were performed with current I_{ij} = 100 µA and the voltmeter HP 34461 set in the range of 100 mV and integration time 20 ms (in number of power line cycles NPLC = 1). Measurements were performed following the sampling plan. The contacts were then raised, and the sample sequentially rotated to the next positions (Figure B.3 b), c), d) to perform the other corresponding pairs of transresistance measurements. $R_{ij,kl}$ values were calculated following Formula (1) and Formula (2).

B.6 Results

Table B.1 reports the measurements of $R_{\mathrm{AB,CD}}$ and $R_{\mathrm{BC,DA}}$ in each of the p configurations that were performed following the sampling plan described in Clause B.4. The type-A and type-B uncertainty values were used to calculate the combined uncertainties $u\left(R_{\mathrm{AB,CD}}\right)$ and $u\left(R_{\mathrm{BC,DA}}\right)$ of $R_{\mathrm{AB,CD}}$ and $R_{\mathrm{BC,DA}}$, respectively, using Formula (3).

Table B.1 – Measured values for $R_{\mathrm{AB,CD}}(p)$ and $R_{\mathrm{BC,DA}}(p)$ using a current source Keithley 2602B and a voltmeter HP 34461 (1y stability specifications)

p	$R_{\mathrm{AB,CD}}$ Ω	$u\left(\mathrm{A},R_{\mathrm{AB,CD}}\right)$ Ω	$u\left(\mathrm{B},R_{\mathrm{AB,CD}}\right)$ Ω	$u\left(R_{\mathrm{AB,CD}}\right)$ Ω	$R_{\mathrm{BC,DA}}$ Ω	$u\left(\mathrm{A},R_{\mathrm{BC,DA}}\right)$ Ω	$u\left(\mathrm{B},R_{\mathrm{BC,DA}}\right)$ Ω	$u\left(R_{\mathrm{BC,DA}}\right)$ Ω
1	21,128 8	0,006 3	0,000 75	0,006 3	191,303 40	0,015	0,006 8	0,015
2	25,475 5	0,008 0	0,000 90	0,008 1	172,418 31	0,034	0,006 1	0,034
3	146,90 1	0,011	0,005 2	0,012	35,185 486	0,010	0,001 2	0,011
4	136,103 7	0,009 7	0,004 8	0,011	37,703 61	0,011	0,001 3	0,012

The sheet resistance of the sample for each measurement configuration $R_{\mathrm{S}}\left(p\right)$ was calculated following Formula (4) and R_{S} following Formula (5); the corresponding uncertainty was calculated using the values of $u\left(R_{\mathrm{AB,CD}}\right)$ and $u\left(R_{\mathrm{BC,DA}}\right)$ reported in Table B.1. Table B.2 reports the calculated values of $R_{\mathrm{S}}(p)$ obtained applying the sampling plan described in Clause B.4.

Table B.2 – Measured values for $R_{\mathrm{S}}(p)$ and corresponding uncertainty values using a current source Keithley 2602B and a voltmeter HP 34461 (1y stability specifications)

p	$R_{\mathrm{S}}(p)$ Ω	**Type A** $u(R_{\mathrm{S}}(p))$ Ω	**Type B** $u(R_{\mathrm{S}}(p))$ Ω	**Combined** $u(R_{\mathrm{S}}(p))$ Ω
1	344,679	0,040	0,009 0	0,041
2	344,075	0,058	0,009 0	0,060
3	351,828	0,042	0,009 0	0,043
4	345,431	0,043	0,008 8	0,044

Since a total of four measurement configurations p were available, the configurational average $< R_{\mathrm{S}} >_p$ was calculated as the average of the four values of $R_{\mathrm{S}}\left(p\right)$ in the contact configurations p = 1, 2, 3, 4.