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Edition 1.0 2023-06

TECHNICAL SPECIFICATION

colour
inside

Nanomanufacturing – Key control characteristics –
Part 6-8: Graphene – Sheet resistance: In-line four-point probe

IEC TS 62607-6-8:2023-06(en)

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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NANOMANUFACTURING –
KEY CONTROL CHARACTERISTICS –

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

Draft	Report on voting
113/678/DTS	113/745/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

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- withdrawn,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, the sheet resistance, R_S, 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 in-line four-point probe method (4PP) allows the measurement of the sheet resistance of samples of arbitrary shape, with isotropic conductivity and uniform carrier density by performing four-terminal resistance measurements with electrical contact provided by a commercially available dedicated tool. The method is fast (it takes a few minutes) and easy to implement, since many commercial fixtures are available.

The four-terminal resistance measurements approach allows to minimize the effect of the contact resistance that appears between graphene and the measurement probes.

The 4PP method provides a certain degree of spatial resolution in principle, depending on the sampling plan adopted to map the sample area.

In this document it is explained how to specifically apply the 4PP method on chemical vapour deposited graphene on rigid insulating support and perform a reliable estimation of the sample KCC sheet resistance, R_S, also considering the non-ideal nature of commercial graphene.

NANOMANUFACTURING –
KEY CONTROL CHARACTERISTICS –

Part 6-8: Graphene – Sheet resistance: In-line four-point probe

1 Scope

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

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

by the

- in-line four-point probe method, 4PP.

The sheet resistance R_S is derived by measurements of four-terminal electrical resistance performed on four electrodes placed on the surface of the planar sample.

- 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 van der Pauw method (IEC 62607-6-7) for what concerns the measurement of the sheet resistance and can be useful when it is not possible to reliably place contacts on the sample boundary.

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.]

3.6 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 must 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 4PP method consists in placing four probes evenly spaced in a straight line on the sample to be measured. For ideal conditions no geometrical information is needed to measure R_S [1][1]. To achieve this the sample should be a 2D infinite layer, i.e. i) the distance between probes, s, should be ideally negligible compared to the dimensions of the sample and ii) the distance x between the probes and the sample edge should be large compared to s. To retrieve R_S, a current I is applied through the two external probes and the voltage V is measured between the internal probe pair. The measured I and V quantities are then used to calculate the sheet resistance using a formula. In practice, this formula can contain corrections, depending on, for example, sample dimensions, inter-probe distance, distance between the probes and the sample edge. Additional considerations for non-equidistant probes are given in Annex B.

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

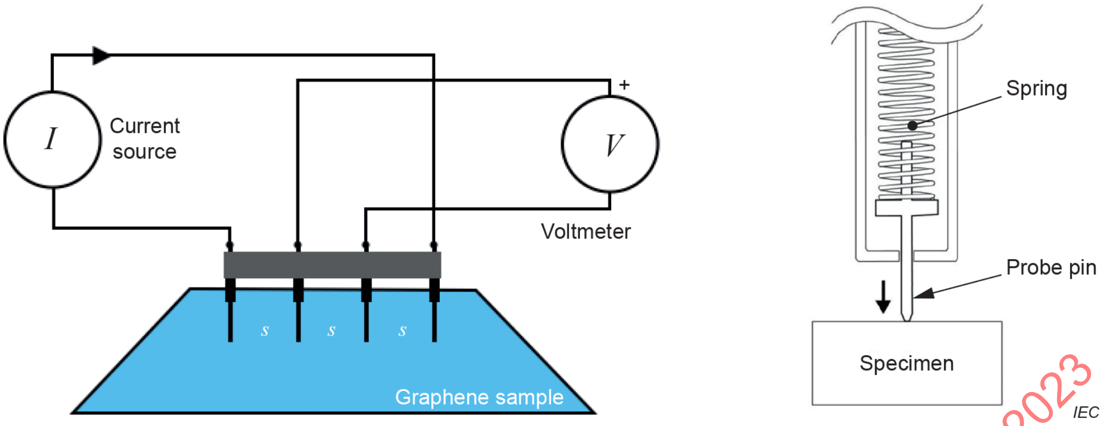


Figure 1 – Schematic representation of the four-point probe method (left) and detail of the structure of a spring-mounted probe (right)

4.2 Sample preparation method

The sample is measured as it is delivered by the supplier. No special sample preparation is required. The substrate supporting the graphene layer should be planar and insulating to prevent any contribution of the volume resistivity of the substrate. The sample shall be stored at the ambient conditions of 4.4 prior to the measurements.

4.3 Description of measurement apparatus

Figure 1 shows a schematic representation of the 4PP measurement setup, consisting of the 4PP tool (in contact with the sample), a current source and a voltmeter.

The measurement instrumentation necessary to carry out measurements with the in-line four-point probe method consists of a current source and a voltmeter. The source and measurement range of the instrumentation, for measuring monolayer CVD graphene, should be of 1 µA to 1 mA for the current source and 1 mV to 1 V for the voltmeter, considering typical five- or six-digit commercial instruments.

A sample holder fitted with four needle spring-mounted probes is necessary to accommodate and contact the sample. The probes shall have a very limited lateral swing in order not to scratch the graphene sample when placed in contact. The outer contacts are used to inject the current while the inner contacts are used to probe the resulting voltage (commercial 4PP are already configured this way). If a custom fixture is used, the insulation resistance between the contacts shall be the highest possible, and at least 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.

4.4 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) %.

4.5 Related standards

Annex D 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.

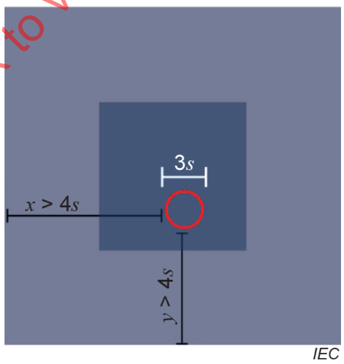
5.2 Detailed protocol of the measurement procedure

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

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 clean the probe by placing a piece of soft towel wetted with ethyl alcohol or isopropyl alcohol on a rigid support (in place of the sample), then lowering and raising the probes a few times to clean the tips.

Place the measuring probe aligned on the graphene sample within the safe area (as shown in Figure 2) and in accordance with a sampling plan that will be reported in the results. The distance s between the probes shall be known, as well as the distance between the current probes and the edge, x. If the probes are equidistant and if their positions meet the condition $x > 4s$, simpler calculations apply. Otherwise, correction factors shall be included, see [2] and [[3], Sec. 4].



Grey area represents the graphene sample. The blue area represents the available safe area to fulfil the "infinite sample" approximation detailed in 5.2, for which simpler calculations apply. Red circle represents the size and position of the 4PP tool.

Figure 2 – Schematic representation of a possible measurement location

The current I applied during the 4PP resistance measurement shall not damage the sample, but shall be large enough in order to get accurate readings from the voltmeter. Currents in the range 1 µA to 100 µA are typically suitable. Measurement accuracy is improved by performing two readings of each measurement of V with two currents equal in nominal magnitude and opposite in sign, and by averaging the results; this helps to reduce thermoelectric effects which introduce systematic errors.

Repeated measurement of V will allow to estimate the measurement standard deviation (measurement repeatability). A reasonable measurements' reading averaging will in general allow to keep the measurement standard deviation below 0 1 % for this kind of measurement with commercial laboratory grade instruments.

5.3 Measurement accuracy

For the definition of uncertainty and related terms used in the following, see Ref. [4], Sec. 2.3.

The 4PP measurement accuracy depends on:

- the measurement accuracy, resulting from the combination of the measurement's standard deviation (type-A uncertainty) and instruments accuracy specifications (type-B uncertainty), see Table 1;
- knowledge and stability of the ambient conditions;
- other knowledge about the measurement setup configuration, for example, probes distance and placement.

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 Nano Volt / Micro Ohm Meter[2] (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	1,00E-05	10	1E-04	1	5,93E-04
1E+02	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	1000	5,18E-04
1E+05	1,00E-06	1	1E-01	1000	9,18E-04

6 Data analysis and interpretation of results

In the case of measurements performed inside the safe area of the sample, the equations for R_S and its uncertainty are the following:

$$R_S = \frac{\pi}{\log(2)} \frac{V}{I} \tag{1}$$

$$u_{R_S} = \sqrt{u_A^2 + \sum u_B^2} \tag{2}$$

---

[2] "2602B System SourceMeter®" and "HP 34420 Nano Volt / Micro Ohm Meter" are examples of a 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.

where

u_A is the type-A uncertainty, estimated as the standard deviation of several calculated values of R_S;

u_B is the type-B uncertainty, estimated from the instrumentation specifications.

Hence more specifically, considering the current source and voltmeter type-B uncertainty, we have

$$u_{R_S} = \sqrt{u_A^2 + \sum u_B^2} = \sqrt{u_m^2 + u_I^2 + u_V^2} \qquad (3)$$

where

u_m is the uncertainty due to the spread of the voltage measurements (type-A);

u_I is the uncertainty due to the specifications of the current source (type-B);

u_V is the uncertainty due to the specifications of the voltmeter (type-B).

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 Product and 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 on Si 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 similar to the usual ones in a calibration laboratory:

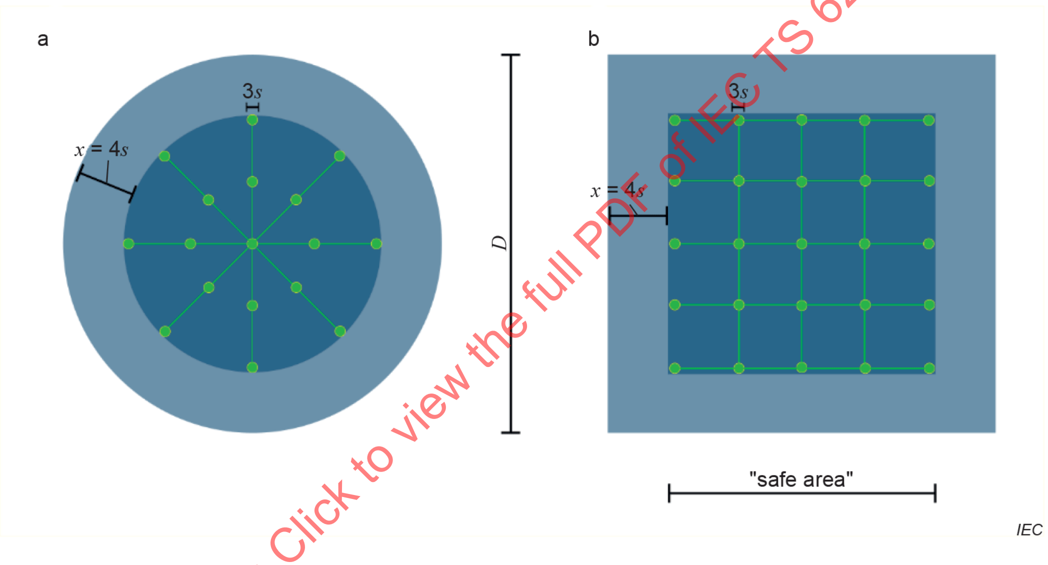
- Temperature range: T = (23 ± 1) °C.
- Relative humidity range: RH = (50 ± 4) %

7.4 Measurement results

- Sampling plan used: measurements shall be performed at different locations in order to include contributions from the spatial variability and the contact repeatability in the measurement accuracy. The sampling plan shall be consistent with the requirements in 5.2. Since the 4PP measurement can be destructive (the sample can be scratched by the probe tip's physical contact), the minimum distance between different locations shall be spaced in order for the probe not to touch the same area more than once.

- The sample orientation with respect to the 4PP tool during the measurements shall be identified in a way that is recognizable on the physical sample. See Figure 2 for a valid example of sample plan. The sampled locations shall be recorded with reasonable accuracy and reported in a table like Table 2.
- In case of small samples, where the sampling plan similar to those described above cannot be straightforwardly applied, the measurements can be performed by repeating the measurements around a single location, since if $3s \approx D$, with D the sample lateral dimension or diameter (see Figure 3), the spatial variability is already taken into account in the measurement. The location shall be anyway chosen in accordance with 5.2 (the centre of the sample will always fulfil those conditions). The measurements can be repeated a number of times by slightly changing the 4PP position in order to avoid contacting twice the same exact spot of the graphene layer.
- Sampling plans for circular and squared samples

  A schematic view of a circular sample (e.g. graphene transferred on a whole wafer) is shown in Figure 3 a). 4PP measurements shall be performed in accordance with the sampling plan such that the whole representative area of the sample is covered. A similar schematic view for a square sample is shown in Figure 3 b).



Safe area (darker colour) and 4PP size $3s$ (green dots) are shown.

Figure 3 – Schematic view of possible sample plans for a) circular and b) square substrates

Table 2 – Sampling plan for circular substrates

Sample number	**Location p (x,y)**				
	1	2	3	…	N
Sample 1	(x_1,y_1)	(x_2,y_2)	(x_3,y_3)		(x_n,y_n)
…					
…					
…					

Results of R_S measured in accordance with this document shall be reported in a table like Table 3.

Table 3 – Results of R_S measured in accordance with this document

Location, p	**R_S** Ω	**Type A $u(R_S)$** Ω	**Type B $u(R_S)$** Ω	**Combined $u(R_S)$** Ω
1	…	…	…	…
2				
3				

Annex A
(informative)

Effects of ambient conditions on graphene resistance measurements

A.1 General

A large body of literature reports about 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 [4], [5]. 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 [6] 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 [7], [8], [9].

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 [8], [10], [11].

Moreover, it has been observed that in presence of graphene characterized by increasing amount of multi-layer seeds and/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 [12].

Annex B
(normative)

More specific cases

B.1 Non-equidistant probes

In case of non-equidistant probes, separated by gaps s_1, s_2 and s_3, the expression for R_S is calculated as follows (adapted from [2], eq. 2.5])

$$R_S = \frac{V}{I}\left[\frac{2\pi}{\frac{1}{s_1}+\frac{1}{s_3}-\frac{1}{s_1+s_2}-\frac{1}{s_2+s_3}}\right] \tag{B.1}$$

For details about the expression of the uncertainty in this case, see [14].

B.2 Proximity to the edge

In case of proximity to the edge, but with equidistant probes, the expression for R_S – adapted from [15], eq. 6 – is as follows:

$$R_S = \frac{V}{I}\left[\frac{2\pi}{\frac{1}{s}-\frac{2}{\sqrt{(2s)^2+(2x)^2}}+\frac{2}{\sqrt{s^2+(2x)^2}}}\right] \tag{B.2}$$

where

s is the distance between the probes;

x is the distance between the current probes and the edge.

For details about the expression of the uncertainty in this case, see [14].

B.3 Non-equidistant probes and proximity to the edge

The case in which both these conditions are combined is even more complicated. See references [2] and [3] for more detail.

It is not worthwhile to follow this method in this case. It is better, if possible, to rely on the van der Pauw method.