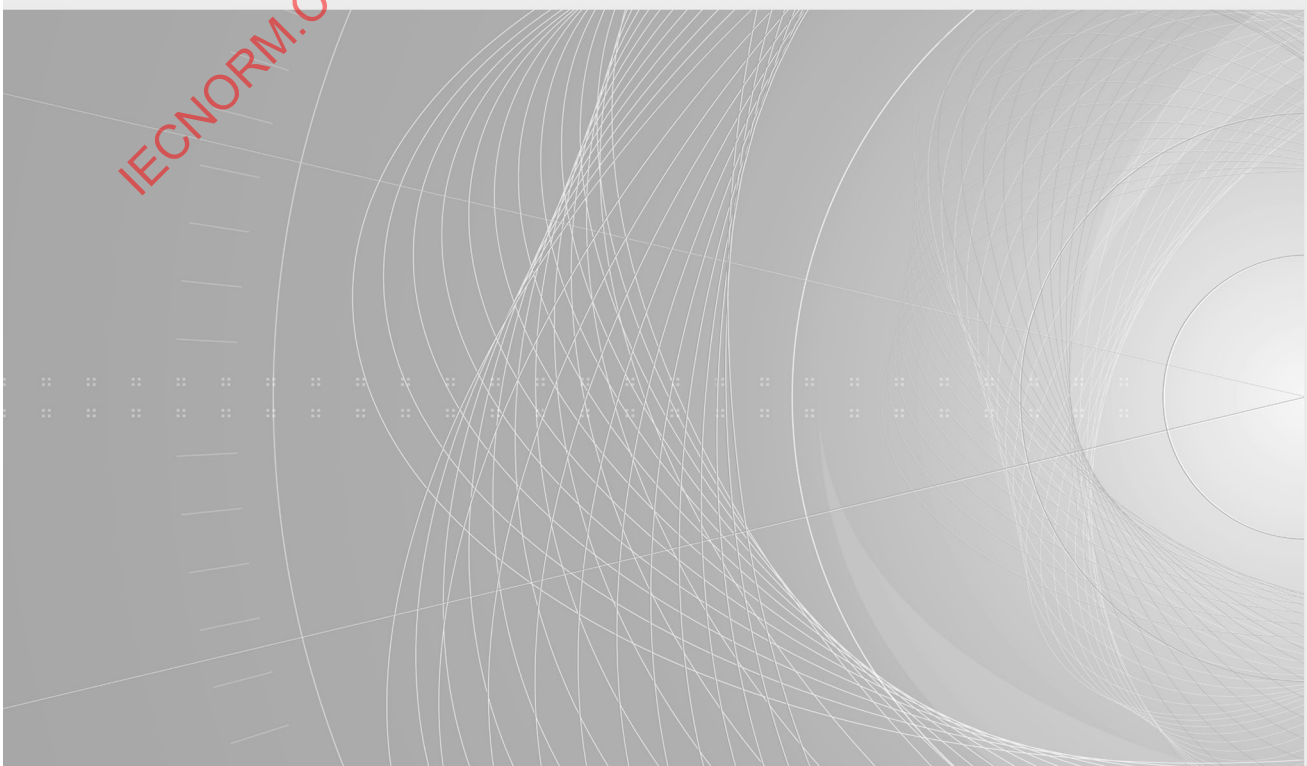
IEC TS 62607-6-22

Edition 1.0 2022-11

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 6-22: Graphene-based material – Ash content: incineration



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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-22: Graphene-based material – Ash content: incineration

FOREWORD

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IEC TS 62607-6-22 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
113/704/DTS	113/681/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- withdrawn,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

Impurity, which is inevitable because of the production process, often has significant influence on the performance of graphene in energy conversion and storage, electronics, composites and catalysis, etc. The ash content can quickly provide an indication of impurity to some extent.

Determination of ash content of graphene is essential for manufacturers to perform quality control. It is also important for users to choose suitable product.

Incineration, the most common method of testing ash content, is a low cost, good repeatable and easy to operate method. Some unique properties of graphene-based material, such as ultra-low bulk density, relative high oxygen content and thermal exfoliation, make it impossible to follow existing incineration standards to determine the ash content of graphene-based material correctly. With the development of the graphene industry, it is important to establish a specific standard method for graphene to determine the ash content correctly. In this method, the two key objectives are to increase the bulk density of ultra-low density reduced graphene oxide through press or impregnation and to avoid instant exfoliation of high oxygen content graphene oxide through low-speed heating during heating at 130 °C to 200 °C.

This document introduces a reliable method for determining the ash content of graphene with incineration. This document can be used as the reference for other carbonaceous materials, such as single-walled and multi-walled carbon nanotubes.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-22: Graphene-based material – Ash content: incineration

1 Scope

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

- ash content

  of powder and dispersion of graphene-based material by

- incineration.

The ash content is derived by residue obtained after incineration under the operating conditions specified in this document, being divided by the mass of the dried test portion.

- The method is applicable for graphene, graphene oxide and reduced graphene oxide in forms of both dry powder and dispersion. This document can be used as reference for graphite oxide and other modified graphene.
- Typical application areas of this method are research, manufacturer and downstream user to guide material processing and quality control.

2 Normative references

There are no normative references in this document.

3 Terms, definitions, symbols and abbreviated terms

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
graphene-based material
GBM
graphene material
grouping of carbon-based 2D materials that include one or more of graphene, bilayer graphene, few-layer graphene, graphene nanoplate and functionalized variations thereof as well as graphene oxide and reduced graphene oxide

Note 1 to entry: "Graphene material" is a short name for graphene-based material.

3.1.3
reduced graphene oxide
rGO
reduced oxygen content form of graphene oxide

[SOURCE: ISO/TS 80004-13:2017 [1], 3.1.2.14]

3.1.4
graphene oxide
GO
chemically modified graphene prepared by oxidation and exfoliation of graphite, causing extensive oxidative modification of the basal plane

[SOURCE: ISO/TS 80004-13:2017. 3.1.2.13]

3.1.5
graphite oxide
chemically modified graphite prepared by extensive oxidative modification of the basal planes

Note 1 to entry: The structure and properties of graphite oxide depend on the degree of oxidation and the particular synthesis method.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.12]

3.2 Key control characteristics measured in accordance with this document

3.2.1
key control characteristic
KCC
key performance indicator
material property or intermediate 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.

3.2.2
ash
residue obtained after incineration at a temperature of 650 °C under the operating conditions specified in this document, divided by the mass of the test portion

Note 1 to entry: The content of ash is usually expressed as a percentage.

3.2.3
thermal gravimetry
TG
method in which the change in mass of a sample is measured as a function of temperature while the sample is subjected to a controlled temperature programme

[SOURCE: ISO 80004-13:2017, 3.3.2.5]

3.2.4
differential scanning calorimetry
DSC
method in which the difference in energy inputs into a substance and a reference material is measured as a function of temperature while the substance and reference material are subjected to a controlled temperature programme

[SOURCE: ISO/TS 80004-6:2021, 6.2.1]

3.2.5
TG-DSC
combined technology of TG and DSC in which TG and DSC data of tested sample are obtained simultaneously

4 General

4.1 Measurement principle

4.1.1 Incineration principle

A test portion is pre-treated, dried and then incinerated at a controlled and programmed temperature in air, until complete disappearance of the carbon in the residue. After cooling to room temperature, the mass of the residue is determined.

4.1.2 Operation principle of this method

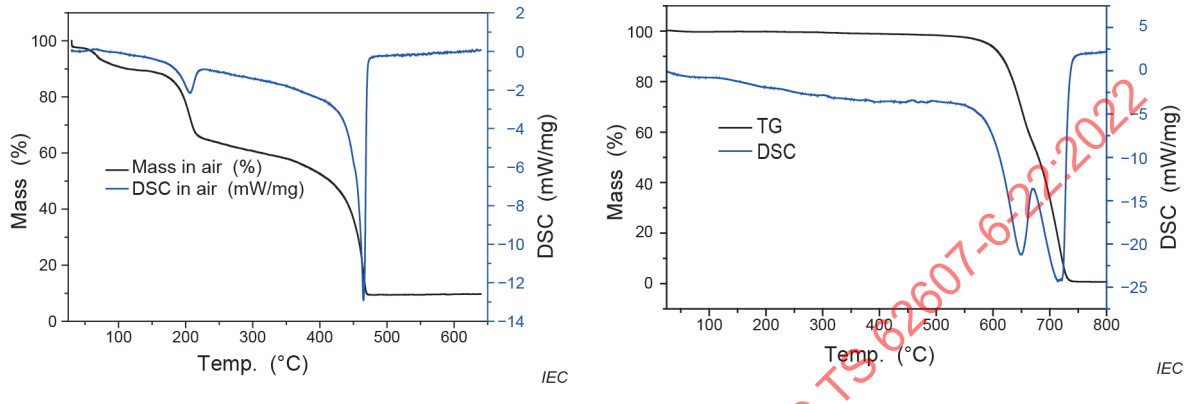
4.1.2.1 GO and graphite oxide

a) GO and graphite oxide have high content of oxygen functional groups. These oxygen functional groups can be decomposed at 130 °C to 200 °C to produce gas and heat, as shown in TG-DSC of graphite oxide (Figure 1a). Decomposition will happen at both outer surface and interior of sample at the same time.
   1) On one hand, the produced gas increases the pressure between graphene sheets inside the sample. When this pressure is larger than the total of Van der Waals force among graphene sheets and external pressure, the sample will be exfoliated instantly, expanding will happen and lead to a splash of sample.
   2) On the other hand, the produced heat will accelerate the decomposition itself, which will result in more gas and heat.
   3) Therefore, slow heating speed is needed during incineration at 130 °C to 200 °C to limit the decomposition speed. And to bring gas and heat out in a timely manner, the furnace should be with an appropriate air ventilation during incineration.

b) GO and graphite oxide will begin to be incinerated at around 400 °C. The incineration begins at the outer surface of sample and cannot lead to an increased pressure between graphene sheets, therefore the sample will not expand. Therefore, fast heating speed is needed during incineration at 200 °C to 650 °C to complete the oxidation.

4.1.2.2 Graphene and rGO

Graphene and rGO have been exfoliated and reduced. Decomposition at 130 °C to 200 °C produces very little gas and heat, as shown in TG-DSC of rGO (Figure 1b), and the produced gas and heat can be expelled in a timely manner, passing through the exfoliated graphene sheet. So, the fast heating speed during the whole incineration will not result in expansion of sample. Therefore, incineration of graphene and rGO can be conducted at a very fast heating speed directly from room temperature to 650 °C.



a) graphite oxide **b) rGO**

Figure 1 – TG-DSC of graphite oxide and rGO in air with 2 °C/min heating speed

4.2 Sample preparation method

The general analysis sample shall be mixed carefully before preparation.

4.3 Description of measurement equipment

Use laboratory apparatus and, in particular, the following.

4.3.1 Beaker.

4.3.2 Petri dishes.

4.3.3 Tablet press machine.

4.3.4 Square crucible, of inert material, such as porcelain, silica, platinum or any other material unaffected under the test conditions with a square flat base.

The size shall be 30 mm (W) × 60 mm (L) × 15 mm (H), of such a size that the sample loading does not exceed 0,08 g/cm^2 for graphite oxide and graphene oxide.

4.3.5 Desiccator and desiccant.

A desiccator with appropriate desiccant is required to prevent absorption of moisture from atmosphere by the test sample and ash.

4.3.6 Furnace, of muffle type or tube type.

The furnace shall be capable of providing a zone of uniform heat at the temperature required and reaching these temperatures within the specified times.

The air ventilation rate through the furnace shall be such as to give at least two air exchanges per hour.

4.3.7 Analytical balance, capable of reading to the nearest 0,1 mg.

4.3.8 Electric thermostatic drying oven.

4.4 Supporting materials

Use only reagents of recognized analytical grade, unless otherwise specified.

4.4.1 Water, complying with at least grade 3 in accordance with ISO 3696 [2][1].

4.4.2 Ethanol, analytical reagent.

4.5 Ambient conditions during measurement

The measurements can be performed under regular laboratory conditions without precise temperature and humidity control.

5 Measurement procedure

5.1 Pre-treatment of sample

5.1.1 Powder

5.1.1.1 GO and graphite oxide powder

Place the GO or graphite oxide powder sample in the electric thermostatic drying oven and heat at (105 ± 5) °C to remove the water completely. Cool to room temperature in the desiccator.

5.1.1.2 rGO and graphene powder

5.1.1.2.1 rGO and graphene powder prepared by thermal exfoliation

a) Place about 0,2 g to 0,3 g of the rGO powder sample into the mould of the tablet press machine, then press and shape the sample into a cylindrical pellet by applying a pressure of about 2 MPa. Place the cylindrical pellet in the electric thermostatic drying oven and heat at (105 ± 5) °C to remove the moisture completely. Cool to room temperature in the desiccator.

b) Some rGO powder cannot be pressed and shaped into a cylindrical pellet. For these kinds of sample, the pre-treatment is to place about 0,6 g of sample into a clean beaker, add about 20 ml water and then use a glass rod to well mix the sample with water. If the sample is too hydrophobic to disperse in water, ethanol can be added to disperse well. Place the beaker in the electric thermostatic drying oven and heat at (105 ± 5) °C to remove the water completely. Cool to room temperature in the desiccator.

5.1.1.2.2 rGO and graphene powder prepared by other methods

Place these kinds of rGO or graphene powder sample in the electric thermostatic drying oven and heat at (105 ± 5) °C to remove the moisture completely. Cool to room temperature in the desiccator.

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

5.1.2 Dispersion

Place a certain amount of the dispersion containing 0,5 g to 0,6 g of the solid sample into a Petri dish and then place the Petri dishes in the electric thermostatic drying oven and heat at (105 ± 5) °C for water dispersion or (130 ± 5) °C for N-Methylpyrrolidone dispersion to remove the solvent completely. Cool to room temperature in the desiccator. After pre-treatment, graphene oxide dispersion (GO dispersion) becomes graphene oxide film (GO film) and graphene or rGO dispersion becomes graphene or rGO bulk.

The thickness of dried film will affect the thermal behaviour of the film. To control the thickness of film, it is recommended that the solid content of GO dispersion added to each Petri dish with a diameter of 10 cm is not more than 1 g.

5.2 Preparation of the crucible

Clean the crucible with tap water and then with distilled water at least three times.

Place the as-prepared crucible in the furnace and heat for at least 1 h at (850 ± 25) °C. Allow to cool to room temperature in the desiccator and then weigh to nearest 0,000 1 g and record the mass as m_1.

NOTE Several crucibles can be handled at the same time.

5.3 Incineration of sample

5.3.1 Graphene and rGO

Weigh rapidly, to the nearest 0,000 1 g, 0,5 g of the pre-treated sample at the bottom of the as-prepared crucible and spread in an even layer over the bottom surface, record the mass of crucible as m_1 and the mass of crucible plus the test portion as m_2.

Place the crucible in a room temperature furnace with air ventilation and heat the test portion in accordance with the following procedure.

- Raise the furnace temperature evenly from room temperature to (650±10) °C over a period of 30 minutes.
- Maintain the temperature at (650±10) °C for 120 minutes to complete incineration.

5.3.2 GO and graphite oxide

Weigh rapidly, to the nearest 0,000 1 g, 0,5 g of the pre-treated sample at the bottom of the as-prepared crucible and spread in an even layer over the bottom surface. Record the mass of crucible as m_1 and the mass of crucible plus the test portion as m_2.

Place the crucible in a room temperature furnace with air ventilation and heat the test portion in accordance with the following procedure.

- Raise the furnace temperature evenly from room temperature to (130 ± 5) °C over a period of 30 min (i.e. heating rate is 4 °C/min).
- Raise the furnace temperature evenly from (130 ± 5) °C to (200 ± 5) °C over a period of 150 min (i.e. heating rate is 0,4 °C/min).
- Maintain the temperature at 200 °C for 10 min.
- Raise the furnace temperature evenly from (200 ± 5) °C to (650 ± 10) °C over a period of 30 min.
- Maintain the temperature at (650 ± 10) °C for 120 min to complete incineration.

5.4 Weighing

Remove the crucible with its contents from the furnace and place a cover on the crucible. Allow the crucible and its contents to cool on a heat-resistant plate for 5 min and then transfer to a desiccator and allow to cool to ambient temperature. Weigh the crucible to the nearest 0,000 1 g as soon as ambient temperature is reached, and record the mass as m_3. Calculate the ash content of the test portion as detailed in Clause 6.

NOTE A cover is used while transferring the crucible to prevent the loss of sample and ash.

5.5 Completion of ashing

If there is any doubt of complete incineration (for instance presence of black sample at visual inspection), reload the crucible and its contents into the hot furnace [at (650 ± 10) °C] for 30 min periods until the change in mass does not exceed 0,5 mg.

6 Data analysis

6.1 Ash content

The ash content on dry basis, A_d, of the sample expressed as a percentage by mass on a dry basis, is given by the formula

$$A_d = \frac{m_3 - m_1}{m_2 - m_1}$$

where

m_1 is the mass of empty crucible, in grams;

m_2 is the mass of crucible plus the test portion, in grams;

m_3 is the mass of crucible plus ash, in grams.

The result shall be calculated to two decimal places and the average value shall be rounded to the nearest 0,01 % for reporting. Take the arithmetic average of the values obtained in two determinations as the result, provided that the requirement for repeatability is fulfilled. The worked examples for determination of different GBM are shown in Annex B.

6.2 Repeatability

When the ash content is less than or equal to 1 %, the absolute error between the values obtained in two determinations, carried out simultaneously or in rapid succession by the same analyst on the same sample, shall not exceed 0,10 %.

When the ash content is greater than 1 %, the relative error between the values obtained in two determinations, carried out simultaneously or in rapid succession by the same analyst on the same sample, shall not exceed 10,0 %.

If the difference exceeds these limits, two further determinations shall be carried out.

7 Results to be reported

7.1 General

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. Guidelines are given in Annex A.

7.2 Product or 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.

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

7.3 Test conditions

The laboratory ambient conditions during the test:

- temperature range;
- range of relative humidity.

Desiccation condition:

- heating temperature and duration.

Ashing condition:

- ramp cycles of ashing.

7.4 Test results

Results of ash content measured in accordance with this document.

Annex A
(informative)

Format of the test report

Table A.1, Table A.2, Table A.3 and Table A.4 are guidelines to write the report and can be modified to fulfil the requirements of the involved parties.

Table A.1 – Product identification (in accordance with the relevant blank detail specification)

Item No.	Item		Information
1.1	Supplier		
1.2	Trade name		
1.3	ID number		
1.4	Typical batch quantity	Number of wafers	
1.5	Traceability requirements	☐ Batch number ☐ Serial number ☐ Others, specify ……	
		Manufacturing date	
1.6	Specification	Number	
		Revision level	
		Date of issue	
1.7	Material Safety Data Sheet (MSDS) available	☐ No	
		☐ Yes Reference	

Table A.2 – General material description (in accordance with the relevant blank detail specification)

Item No.	Item	Information
2.1	Material type	
2.2	Manufacturing method	
2.3	Shelf life	
2.4	Typical batch size	

Table A.3 – Measurement related information and results

Item No.	Item	Information
3.1	The photo of sample after incineration	
3.2	Environmental humidity (mean)	
3.3	Environmental temperature (mean)	
3.4	Reference document	IEC TS 62607-6-22
3.5	Desiccation condition	
3.6	Ashing condition	

Table A.4 – Measurement results

Measurement	Mass of crucible, m_1 (g)	Mass of crucible plus sample, m_2 (g)	Mass of crucible plus ash, m_3 (g)	Ash content (%)	Average ash content (%)	Relative or absolute error
1						
2						

Annex B
(informative)

Worked examples

B.1 rGO powder prepared by thermal exfoliation of graphite oxide: sample 1

Place about 0,4 g of sample 1 into the mould of the tablet press machine, then press and shape sample 1 into a cylindrical pellet by applying 2 MPa pressure, as shown in Figure B.1. Press three pellets. Place the cylindrical shaped pellet sample in the electric thermostatic drying oven and heat at 105 °C to remove the moisture completely. Cool to room temperature in the desiccator.

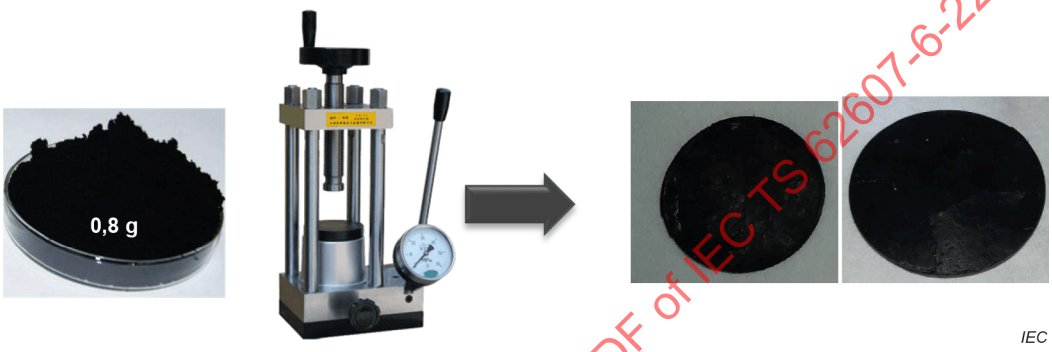


Figure B.1 – Press and shape the sample 1 powder into a cylindrical pellet

Weigh rapidly 0,500 9 g and 0,499 6 g of the pre-treated sample 1 in two crucibles, the crucibles having been pre-treated in accordance with 5.2.

Place the crucibles in a cold furnace with air ventilation rate at 150 mL/min and heat the test portion in accordance with the following procedure.

- Raise the furnace temperature evenly to 650 °C over a period of 30 min.
- Maintain the temperature at 650 °C for 120 min to completely incinerate.

Remove the crucibles with their contents from the furnace and place a cover on the crucibles, as shown in Figure B.2. Allow these two crucibles and their contents to cool on a heat-resistant plate for 5 min and then transfer to a desiccator and allow to cool to ambient temperature. Weigh these two crucibles with a balance to the nearest 0,000 1 g as soon as ambient temperature is reached and record the mass as m_3. Calculate the ash content of the test portion. The result is shown in Table B.1.

Figure B.2 – Incineration of sample 1

Table B.1 – Measurement results of sample 1

Measurement	Mass of crucible, m_1 (g)	Mass of crucible plus sample, m_2 (g)	Mass of crucible plus ash, m_3 (g)	Ash content (%)	Average ash content (%)	Absolute error (%)
1	15,143 7	15,644 6	15,144 5	0,16	0,17	0,01
2	16,458 1	16,957 7	16,458 9	0,17		

B.2 rGO powder prepared by thermal exfoliation of graphite oxide: sample 2

Place about 1 g of sample 2 into a clean Petri dish, add 5 mL ethanol to infiltrate the sample, and then add about 20 mL water and use a glass rod to well mix the sample.

Place the beaker in the electric thermostatic drying oven and heat at 105 °C to remove the water completely, as shown in Figure B.3. Cool to room temperature in the desiccator.

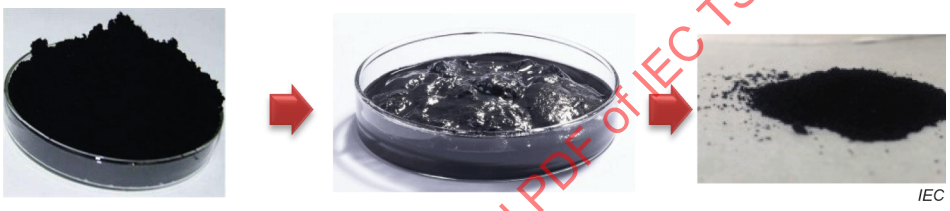


Figure B.3 – Pre-treatment of sample 2

Weigh rapidly 0,501 9 g and 0,500 5 g of pre-treated sample 2 at the bottom of two crucibles and spread in an even layer over the bottom surface of each. Record the mass of crucibles as m_1 and the mass of crucible plus the test portion as m_2.

Place the crucibles in a cold furnace with air ventilation rate at 150 mL/min and heat the test portion in accordance with the following procedure.

- Raise the furnace temperature evenly to 650 °C over a period of 30 min.
- Maintain the temperature at 650 °C for 120 min to completely incinerate.

Remove the crucibles with their contents from the furnace and place a cover on the crucibles. Allow the crucibles and their contents to cool on a heat resistant plate for 5 min and then transfer to a desiccator and allow to cool to ambient temperature. Weigh each crucible with ash to the nearest 0,000 1 g as soon as ambient temperature is reached and record the mass as m_3. Calculate the ash content. The result is shown in Table B.2.

Table B.2 – Measurement results of sample 2

Measurement	Mass of crucible, m_1 (g)	Mass of crucible plus sample, m_2 (g)	Mass of crucible plus ash, m_3 (g)	Ash content (%)	Average ash content (%)	Relative error (%)
1	15,456 2	15,958 1	15,466 0	1,95	1,91	1,65
2	16,891 2	17,391 7	16,900 6	1,88		