# IEC TS 62607-6-18

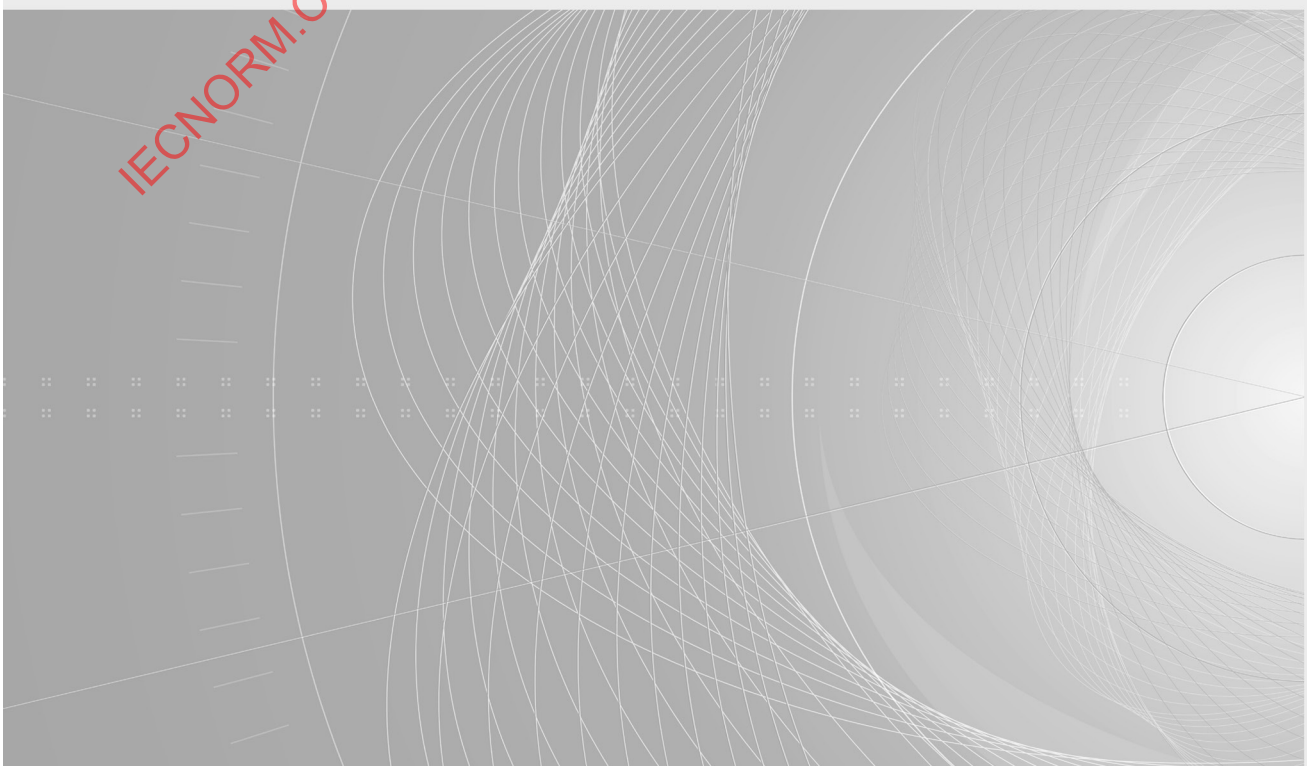
Edition 1.0 2022-12

# TECHNICAL SPECIFICATION

colour inside

## Nanomanufacturing – Key control characteristics – Part 6-18: Graphene-based material – Functional groups: TGA-FTIR





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## Nanomanufacturing – Key control characteristics – Part 6-18: Graphene-based material – Functional groups: TGA-FTIR

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# NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

# Part 6-18: Graphene-based material – Functional groups: TGA-FTIR

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

| Draft | Report on voting |
|---|---|
| 113/680/DTS | 113/706/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 in 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- withdrawn,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

One of the most well-studied routes for the preparation of graphene is the oxidation and reduction process. The most cost-effective process to obtain graphene is the exfoliation of natural graphite layers after oxidation to get individual oxidized layers and then de-oxygenation (reduction) of these individual layers [1], [2] [1]. During the oxidation process, various functionalized groups (-OH, -O-, -COOH, C=O, etc.) go into the graphene skeleton, breaking the $\pi$ bond of graphene structure [3]. Oxygen attachment to graphene in any chemical form (epoxide, hydroxyl, carboxyl and ketonic-type functional groups) both on the basal plane and at the edges reduces electronic states at the Fermi level [4], [5], [6]. The type and content of functional groups affect the physiochemical properties of graphene. Therefore, the identification and quantification of functional groups on graphene powder is believed to be a key control characteristic for its production and application.

Coupling thermal gravimetric analysis (TGA) and Fourier transform infrared spectroscopy (FTIR) is an excellent solution to identify and quantify functional groups on graphene powder. In TGA-FTIR, while mass changes such as sample pyrolysis and vaporization that accompany changes in temperature are measured quantitatively by the TGA, qualitative analysis of the gaseous components can be conducted simultaneously by FTIR measurement of the obtained spectra. This document focuses on determining the type and content of functional groups (e.g. hydroxyl, amino, carboxyl, alkyl, carbonyl, sulfonic acid group) on graphene powder by coupling TGA and FTIR.



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

# NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

# Part 6-18: Graphene-based material – Functional groups: TGA-FTIR

## 1 Scope

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

- functional groups

for functionalized graphene-based material and graphene oxide by

- thermogravimetry analysis (TGA) coupled with Fourier transform infrared spectroscopy (FTIR), referred to as TGA-FTIR.

The content of functional groups is derived by changes in mass of the sample as a function of temperature using TGA. Materials evolved during these mass changes are then analysed using coupled FTIR to identify functional groups.

- The functional groups determined according to this document will be listed as a key control characteristic in the blank detail specification for graphene IEC 62565-3-1 for graphene powder.
- The method is applicable for functionalized graphene powder and graphene oxide that can be pyrolysed and gasified with elevated temperature during TGA.
- Typical application areas are quality control for graphene manufacturers, and product selection for downstream users.

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

Note 1 to entry: Graphene oxide is a single-layer material with a high oxygen content, typically characterized by C/O atomic ratios of approximately 2,0 depending on the method of synthesis.

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

**3.1.3**
**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.

**3.1.4**
**blank detail specification**
**BDS**
structured generic specification of the set of key control characteristics which are needed to describe a specific nano-enabled product without assigning specific values and/or attributes

Note 1 to entry: The templates defined in a blank detail specification list the key control characteristics for the nano-enabled material or product without assigning specific values to it.

Note 2 to entry: Examples of nano-enabled products are: nanomaterials, nanocomposites and nano-subassemblies.

Note 3 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.

**3.1.5**
**sectional blank detail specification**
**SBDS**
specification based on a blank detail specification adapted for a subgroup of the nano-enabled product

Note 1 to entry: In general the sectional blank detail specification contains a subset of those KCCs listed in the blank detail specification. In addition, sectional specific key control characteristics may be added if they are not listed in the blank detail specification.

Note 2 to entry: The templates defined in the sectional blank detail specification may contain key control characteristics with and without assigned values and attributes.

Note 3 to entry: The section can be defined by application, manufacturing method or general material properties.

**3.1.6**
**detail specification**
**DS**
specification based on a blank detail specification with assigned values and attributes

Note 1 to entry: The properties 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 properties which are required for the intended application.

Note 2 to entry: Detail specifications are defined by the industrial partners. SDOs 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.

**3.1.7**
**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 Key control characteristics measured in accordance with this document

**3.2.1**
**functional group**
atom or group of atoms that has similar chemical properties whenever it occurs in different compounds, which defines the characteristic physical and chemical properties of families of organic compounds

## 3.3 Terms related to the measurement method

**3.3.1**
**thermogravimetry analysis**
**TGA**
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/TS 80004-6:2021, 6.1.2, modified – The term has been changed from "thermogravimetry" to "thermogravimetry analysis".]

**3.3.2**
**Fourier transform infrared spectroscopy**
**FTIR**
analytical chemical technique based on absorption of infrared radiation by chemical moieties in the specimen, used to identify and quantitate the absorbing chemical moieties

[SOURCE: ISO/TS 14101:2012, 3.3]

**3.3.3**
**attenuated total reflection mode**
**ATR Mode**
instrumental mode of operation in which the incident angle of IR light on the crystal is adjusted to be higher than the critical angle

Note 1 to entry: The light is completely reflected by the upper surface of the crystal, and the intensity of the light is attenuated through absorption by materials covering the upper surface of the crystal. The frequency of IR light absorbed is used to identify the absorbed chemical moiety, and the fraction of light that is absorbed is used to quantitate the amount of that moiety present.

[SOURCE: ISO/TS 14101:2012, 3.1]

**3.3.4**
**evolved gas analysis**
method in which the nature and/or amount of volatile product(s) released by a substance is (are) measured as a function of temperature while the substance is subjected to a controlled temperature programme.

[SOURCE: ISO/TS 80004-6:2021, 5.26]

## 4 General

### 4.1 Measurement principle

In TGA-FTIR, TGA is connected via heated and temperature-controlled transfer line. The samples are heated at a given heating rate from room temperature to the desired temperature in an inert gas such as ultra-purity nitrogen or helium. While mass changes such as sample pyrolysis and vaporization that accompany changes in temperature are measured quantitatively by the TGA, qualitative analysis of the evolved gases from pyrolysed materials can be monitored simultaneously by FTIR measurement of the obtained spectra.

### 4.2 Sample preparation method

For TGA measurements, tablet at least 5 mg of graphene powder for 1 min with pressure of 3 MPa to 4 MPa. The size of the sample tablet is controlled to be suitable for the pan of TGA.

For ATR measurements, gently press the graphene powder with a glass slide to ensure the sample contacting with ATR base is as smooth as possible, so that satisfactory spectral signals can be produced from ATR tests.

### 4.3 Measurement system

The heated gas cell is placed in the FTIR sample compartment for detection of the decomposition products. One end of the transfer line is interfaced to the inlet port of the gas cell and the other end to the TGA. TGA monitors sample weight loss caused by volatilization and pyrolysis often as a function of temperature ramping using a high-precision balance and furnace. Evolved sample gases originating from the TGA pass through the heated transfer line into the heated gas cell in the FTIR sample compartment. As these evolved gases travel through the gas cell, FTIR spectra are collected and stored for further processing to obtain qualitative and quantitative information.

### 4.4 Description of measurement equipment

The transfer line from the TGA to the gas cell needs to present an inert, nonporous surface to the evolved gas. Evolved gas transfer lines shall be heated to temperatures sufficient to prevent condensation of the evolved gas species. The temperature of the transfer line is normally held constant during an experiment at a level chosen to avoid both condensation and degradation of the evolved gases. Typical working temperatures have a range of 150 °C to 300 °C.

The gas cell usually is heated to a constant temperature at or slightly higher than the temperature of the transfer line, approximately 10 °C higher, to avoid condensation of the evolved gas. However, the maximum temperature recommended by the manufacturer should not be exceeded.

The ends of the gas cell are sealed with infrared transmitting windows or window and mirror combinations.

### 4.5 Supporting materials

During the analysis, reagents and materials are needed. Unless otherwise stated, use only reagents of recognized analytical grade.

**4.5.1** Helium gas or nitrogen gas, ultra-high purity, total impurity content 0,000 5 % (m/m).

**4.5.2** Cleaning solvent, suitable for cleaning pan for sample loading, for example ethanol absolute or acetone.

**4.5.3** Certified reference material, used for testing the instrument response and calibrating the measurement equipment.

### 4.6 Ambient conditions during measurement

TGA-FTIR measurements shall be carried out at room temperature and relative humidity below 60 %. Specific temperature and relative humidity are not required.

## 5 Measurement procedure

### 5.1 Calibration of measurement equipment

A standard test sample should be used on a regular basis to test the instrument response and indicate when problems have occurred with either the FTIR, the TGA or the optical interface. This test will also determine if the transfer line is obstructed, or if the gas flow has been interrupted in some other way. Polystyrene film with No. of GBW(E)130181 can be used for the calibration of FTIR. Pure metal (primary standard of Sn, Zn, Pb, Ni) can be used for calibration of TGA.

### 5.2 Detailed protocol of the measurement procedure

The tabletting sample with mass $m$ (at least 5 mg) is used for TGA-FTIR measurement. Firstly, set the temperature of infrared accessories, turn on the valve of nitrogen or helium purge gas to evacuate air and vapour in infrared accessories. After the pre-treatment, scan the infrared background with FTIR. Take the pan from the TGA furnace and clean it with appropriate solvents, and then load the sample. Set the TGA temperature from 25 °C to 1 200 °C at a heating rate of 5 °C/min and purified nitrogen or helium (99,999 %) at a flow rate of 50 mL/min to keep an inert atmosphere. The temperature of gas cell and transfer line is held at 300 °C and 280 °C, respectively. Once the measurement starts, the sample weight loss caused by volatilization and pyrolysis as a function of temperature is quantitatively monitored by TGA. The purge gas flow carries the evolved gas from the TGA to the gas cell installed in the FTIR sample compartment via the transfer line, and the spectrum is measured simultaneously by FTIR. The spectrum scope is in the range of 400 $cm^{-1}$ to 4 000 $cm^{-1}$ with a resolution factor of 8 $cm^{-1}$. After the TGA-FTIR measurement ends, TGA curve and 3D FTIR spectrum (intensity versus wavenumber versus time or temperature) can be obtained. The characteristic weight loss temperatures $T_i$ ($i$ = 1, 2, …, $n$) can be determined from the TGA curve.

Deposit graphene powder on the ATR base and gently press the powder with a glass slide. An FTIR spectrum can be obtained by conducting ATR measurement for the as-made graphene sample.

Take one tabletting sample with mass $m$ for TGA measurement. Set the TGA temperature from 25 °C to the characteristic weight loss temperature $T_1$ and heat the sample to $T_1$ at a heating rate of 5 °C/min. Then take the sample out for ATR measurement and an FTIR spectrum can be obtained for the graphene sample heated to $T_1$. Take one tabletting sample with mass $m$ for TGA measurement. Set the TGA temperature from 25 °C to the characteristic weight loss temperature $T_2$ and heat the sample to $T_2$ at a heating rate of 5 °C/min. Then take the sample out for ATR measurement and an FTIR spectrum can be obtained for the graphene sample heated to $T_2$. Repeat these steps until the sample is heated to $T_n$. After finishing these measurements, FTIR spectra can be obtained for graphene samples heated to different characteristic weight loss temperatures $T_i$ ($i$ = 1, 2, …, $n$).

## 6 Data analysis

Figure 1 shows the flow chart of data analysis, including identification of functional groups and quantification of functional groups.

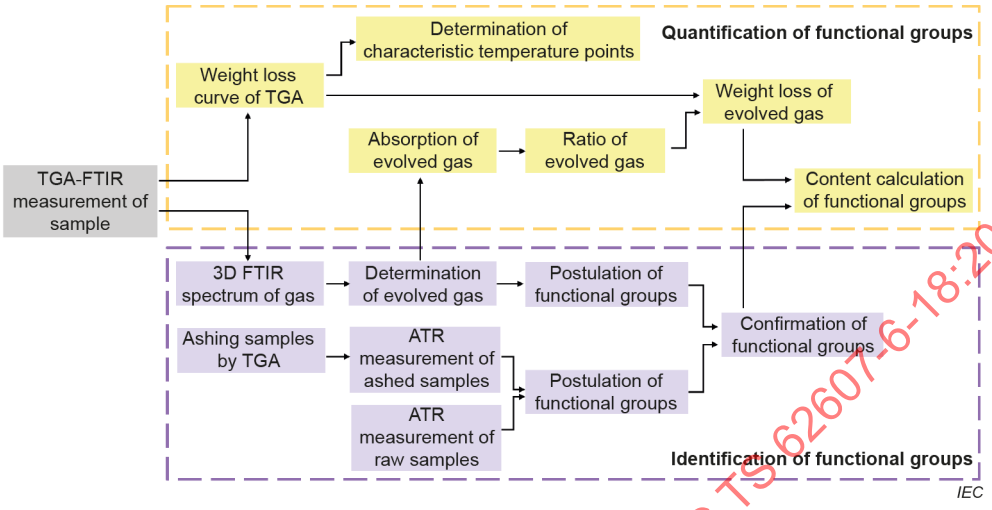


**Figure 1 – Flow chart of data analysis**

Firstly, analyse TGA data from one FTIR-TGA measurement of sample with mass $m$. From the TGA curve, it is possible to determine the characteristic weight loss temperature points $T_i$ ($i$ = 1, 2, …, $n$) at which significant weight loss of sample occurs and the mass difference between the starting point and the corresponding temperature, written as $\Delta m_{Ti}$ ($i$ = 1, 2, …, $n$).

Secondly, postulate the functional group types in sample from ATR measurements. Process the FTIR spectra of as-made graphene sample and graphene samples heated to different $T_i$ ($i$ = 1, 2, …, $n$) with the same methods, including background removal, noise removal and baseline normalizing. Analyse the difference between the FTIR spectrum of as-made graphene sample and the FTIR spectra of ashed graphene samples heated to $T_i$ ($i$ = 1, 2, …, $n$). Based on the infrared spectrum database of solid, it is possible to postulate the functional group types corresponding to $T_i$ ($i$ = 1, 2, …, $n$), written as set(1). In this step, vibration peak C=O can be used as an internal reference.

Thirdly, postulate the functional group types in sample from TGA-FTIR measurements. A 3D FTIR spectrum of gas can be extracted from the measurement. Based on the infrared spectrum database of combustion gas, it is possible to postulate the functional group types corresponding to $T_i$ ($i$ = 1, 2, …, $n$) based on the evolved gases, written as set(2). In this step, $CO_2$ with known quantity can be introduced during measurements. Vibration peak C=O of $CO_2$ can be used as an external reference.

Fourthly, determine the functional group types in sample. Comparing the set(1) and set(2) of functional groups, the functional group types can be determined from their intersection.

Finally, calculate functional group contents in sample. Find peaks for each gas component from the 3D FTIR spectrum and calculate the peak ratios at the intensity where the principal component of each gas is the highest. One can obtain the ratios of different gas types $G_{Ti}$ ($i$ = 1, 2, …, $n$) corresponding to $\Delta m_{Ti}$ ($i$ = 1, 2, …, $n$). Sum up the product of $G_{Ti}$ ($i$ = 1, 2, …, $n$) and $\Delta m_{Ti}$ ($i$ = 1, 2, …, $n$) from 1 to $n$, and divide by the sample mass $m$, then the mass fraction of functional group content F(%) can be obtained, $\mathrm{F}(\%) = \dfrac{\sum_{i=1}^{n} G_{Ti} \times \Delta m_{Ti}}{m}$.

A case study about data analysis can be found in Annex B.

# 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: 18 °C < $T$ < 30 °C.
- Range of relative humidity: 40 % < RH < 65 %.

## 7.4 Measurement specific information

- Calibration status of equipment.
- Manufacturers' names and model numbers for the complete TGA-FTIR system, and the individual components.
- Gas cell temperature.
- Window material used in the gas cell.
- Transfer line temperature.
- Type of detector.
- TGA purge gas and flow rate.
- TGA temperature and elevated rate.
- Sample mass.
- Spectral resolution.

## 7.5 Test results

Results of types and contents or functional groups measured according to this document.

# Annex A
(informative)

## Format of the test report

The form of the report is oriented on the relevant material specification,[2] a related sectional blank detail specification or detail specification. Table A.1, Table A.2, Table A.3 and Table A.4 are guidelines to write the report and can be customized to fulfil the requirements of the involved parties.

**Table A.1 – Product identification (in accordance with 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 | Mass [g] | |
| 1.5 | Traceability requirements | ☐ Batch number<br>☐ Serial number<br>☐ 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 in accordance with the relevant blank detail specification)**

| Item No. | Item | Information |
|---|---|---|
| 2.1 | Material type | |
| 2.2 | Manufacturing method | |
| 2.3 | Physical form | |
| 2.4 | Shelf life | |
| 2.5 | Typical batch size | |



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

**Table A.3 – Test information**

| Item No. | Item | | Information |
|---|---|---|---|
| 3.1 | Instruments | Name | |
| | | Manufacturer | |
| | | Model | |
| 3.2 | Temperature range of TGA | | |
| 3.3 | Elevating rate of temperature of TGA | | |
| 3.4 | Purge gas atmosphere | | |
| 3.5 | Sample mass | | |
| 3.6 | Number of spectra or measurements | | |
| 3.7 | Temperature for transfer line | | |
| 3.8 | Temperature for gas cell | | |
| 3.9 | Window material of gas cell | | |
| 3.10 | Type of detector | | |
| 3.11 | Spectral resolution | | |
| 3.12 | Environmental temperature | | |
| 3.13 | Environmental relative humidity | | |

**Table A.4 – Measurement results**

| Item | Types of functional groups | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| Mass fraction (%) of first test | | | | | | | | |
| Mass fraction (%) of second test | | | | | | | | |
| Mass fraction (%) of third test | | | | | | | | |
| Average | | | | | | | | |
| Standard deviation | | | | | | | | |

# Annex B
(informative)

# Case study: Data analysis

## B.1 Confirmation of characteristic temperature points from TGA curve

A graph of weight plotted against temperature can be obtained from the TGA-FTIR measurement, as shown on the left side of Figure B.1. The right side of Figure B.1 represents the corresponding differential curve of weight loss, from which the characteristic weight loss temperature points can be determined to be 75 °C, 200 °C, 230 °C, 570 °C and 1 000 °C.

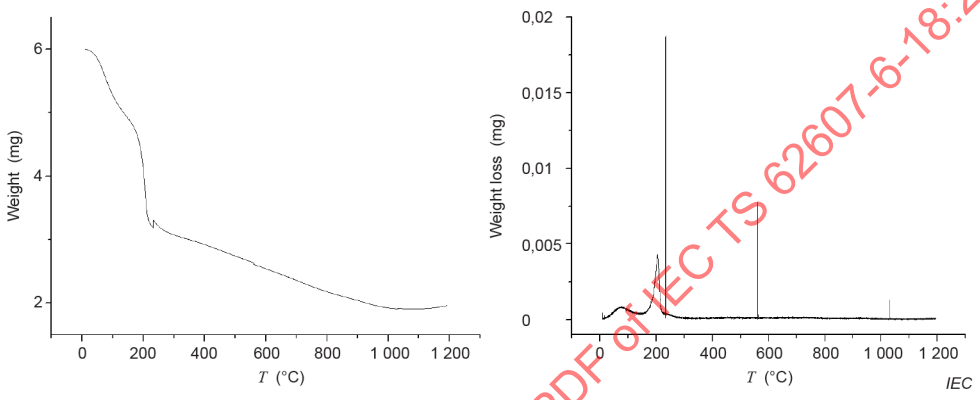


**Figure B.1 – Weight loss curve (left) obtained from TGA-FTIR measurement and corresponding differential weight loss curve (right)**

## B.2 Analysis of FTIR spectra obtained at different ashing temperatures

Conduct ATR measurements for samples ashed at different temperatures including the above-mentioned characteristic temperature points. And then plot all the obtained FTIR spectra together, as shown in Figure B.2.

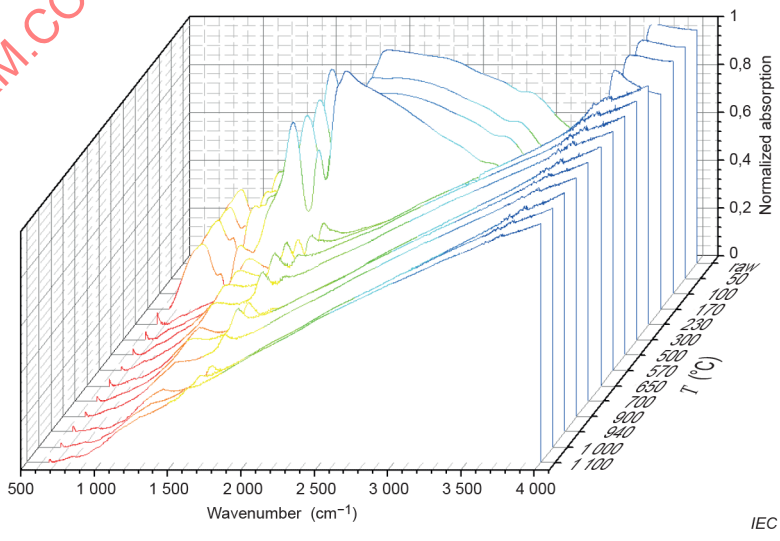


**Figure B.2 – FTIR spectra corresponding to different ashing temperature points**

From Figure B.2, in the temperature range of 25 °C to 170 °C, it can be postulated that the functional groups are (-OH), (-COOH), (C-O-C) and (C=O). In the temperature range of 170 °C to 700 °C, it can be postulated that the functional groups are (-OH) and (C=O). In the temperature range of 700 °C to 1 100 °C, it can be postulated that the released functional groups are (-OH) and edge (C-O-C) [7].

## B.3 Analysis of FTIR spectra obtained by TGA-FTIR measurements

A 3D FTIR spectrum can be obtained from the TGA-FTIR measurement. Comparing it with standard FTIR gas spectra source, it is safe to draw the conclusion that near 2 000 $cm^{-1}$, 2 500 $cm^{-1}$ and 4 000 $cm^{-1}$, the released gas is mainly CO, $CO_2$, and $H_2O$, respectively. Furthermore, it can be postulated that in the temperature range of 25 °C to 170 °C, the released functional groups are (-OH), (-COOH) and (C=O). In the temperature range of 170 °C to 700 °C, it can be postulated that the functional groups are (-OH), (C=O) and (C-O-C). In the temperature range of 700 °C to 1 100 °C, it can be postulated that the released functional groups are (-OH) and edge (C-O-C).

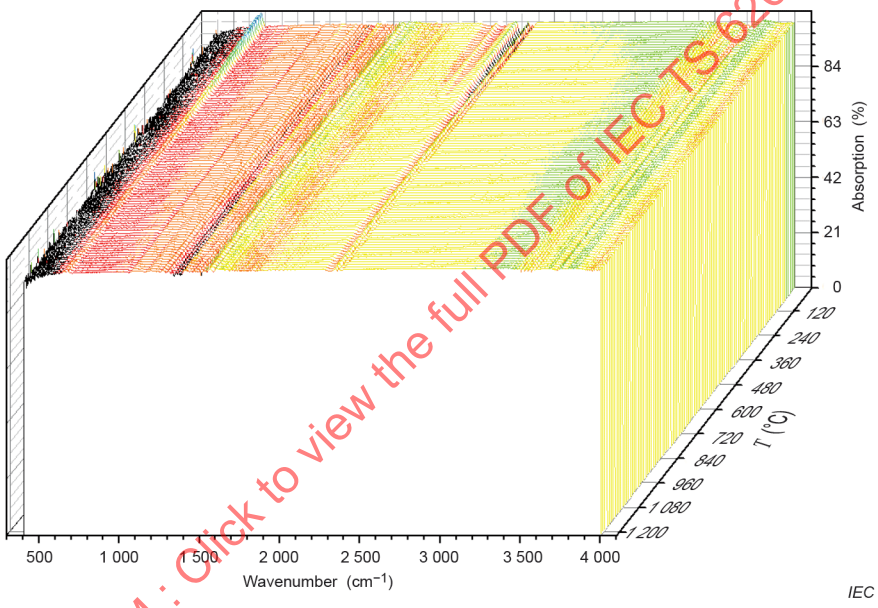


**Figure B.3 – A 3D FTIR spectrum obtained by one FTIR-TGA measurement**

From Figure B.3, it is possible to trace the absorption peaks where the intensity is the highest for each gas component (CO, $CO_2$ and $H_2O$) at different temperatures. The corresponding curves are plotted in Figure B.4.

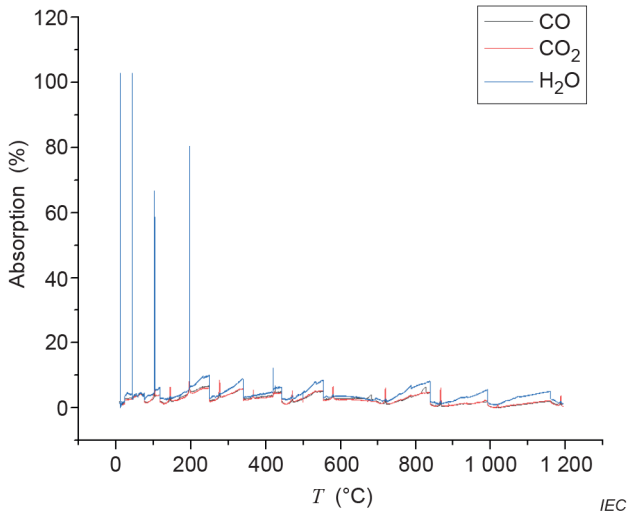


**Figure B.4 – Absorption dynamics of each gas component**

Then the peak ratios of each gas component can be calculated, and the result plotted in Figure B.5.

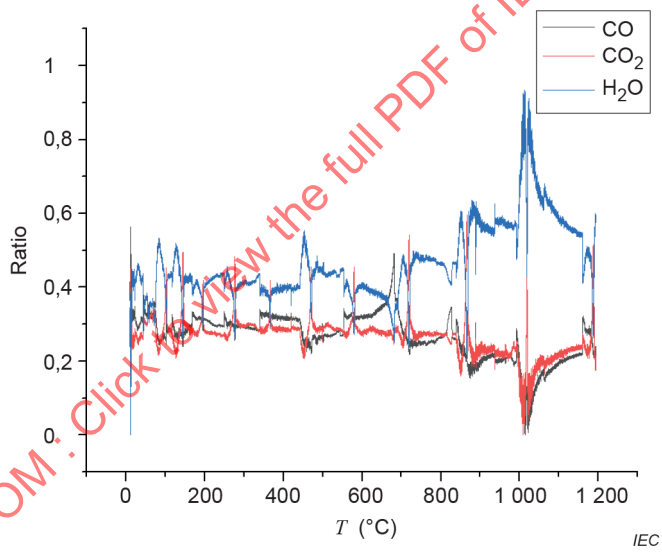


**Figure B.5 – Absorption dynamics of each gas component**



## B.4 Confirmation and quantification of the functional groups

One can confirm the functional groups by the intersection set of the postulated functional groups in Clauses B.2 and B.3. Therefore, in the temperature range of 25 °C to 170 °C, the functional groups are (-OH), (-COOH) and (C=O). In the temperature range of 170 °C to 700 °C, the functional groups are (-OH) and (C=O). In the temperature range of 700 °C to 1 100 °C, the functional groups are (-OH) and (C-O-C).

The weight loss curve due to the release of CO, $CO_2$ and $H_2O$ is shown in Figure B.6. Such a result is derived from Figure B.1 and Figure B.5.