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

IEC TS 62607-6-22:2022-11(en)

IEC Secretariat
3, rue de Varembé
CH-1211 Geneva 20
Switzerland

Tel.: +41 22 919 02 11
info@iec.ch
www.iec.ch

About the IEC
The International Electrotechnical Commission (IEC) is the leading global organization that prepares and publishes International Standards for all electrical, electronic and related technologies.

About IEC publications
The technical content of IEC publications is kept under constant review by the IEC. Please make sure that you have the latest edition, a corrigendum or an amendment might have been published.

IEC publications search - webstore.iec.ch/advsearchform
The advanced search enables to find IEC publications by a variety of criteria (reference number, text, technical committee, …). It also gives information on projects, replaced and withdrawn publications.

IEC Just Published - webstore.iec.ch/justpublished
Stay up to date on all new IEC publications. Just Published details all new publications released. Available online and once a month by email.

IEC Customer Service Centre - webstore.iec.ch/csc
If you wish to give us your feedback on this publication or need further assistance, please contact the Customer Service Centre: sales@iec.ch.

IEC Products & Services Portal - products.iec.ch
Discover our powerful search engine and read freely all the publications previews. With a subscription you will always have access to up to date content tailored to your needs.

Electropedia - www.electropedia.org
The world's leading online dictionary on electrotechnology, containing more than 22 300 terminological entries in English and French, with equivalent terms in 19 additional languages. Also known as the International Electrotechnical Vocabulary (IEV) online.

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

INTERNATIONAL ELECTROTECHNICAL COMMISSION

ICS 07.120

ISBN 978-2-8322-6003-6

CONTENTS

INTERNATIONAL ELECTROTECHNICAL COMMISSION

_____

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

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

FOREWORD

1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.
3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.
4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.
5) IEC itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.
6) All users should ensure that they have the latest edition of this publication.
7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.
8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.
9) Attention is drawn to the possibility that some of the elements of this IEC Publication may be the subject of patent rights. IEC shall not be held responsible for identifying any or all such patent rights.

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.

IMPORTANT – The "colour inside" logo on the cover page of this document indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

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.