



TECHNICAL SPECIFICATION



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

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 07.120

ISBN 978-2-8322-6223-8

Warning! Make sure that you obtained this publication from an authorized distributor.

CONTENTS

FOREWORD.....	3
INTRODUCTION.....	5
1 Scope.....	6
2 Normative references	6
3 Terms and definitions	6
4 General	9
4.1 Measurement principle.....	9
4.2 Sample preparation method	9
4.3 Measurement system.....	9
4.4 Description of measurement equipment	9
4.5 Supporting materials	9
4.6 Ambient conditions during measurement.....	10
5 Measurement procedure	10
5.1 Calibration of measurement equipment	10
5.2 Detailed protocol of the measurement procedure	10
6 Data analysis.....	11
7 Results to be reported	12
7.1 General.....	12
7.2 Product or sample identification	12
7.3 Test conditions	12
7.4 Measurement specific information.....	12
7.5 Test results.....	12
Annex A (informative) Format of the test report.....	13
Annex B (informative) Case study: Data analysis	15
B.1 Confirmation of characteristic temperature points from TGA curve	15
B.2 Analysis of FTIR spectra obtained at different ashing temperatures	15
B.3 Analysis of FTIR spectra obtained by TGA-FTIR measurements	16
B.4 Confirmation and quantification of the functional groups	17
Bibliography.....	19
Figure 1 – Flow chart of data analysis.....	11
Figure B.1 – Weight loss curve (left) obtained from TGA-FTIR measurement and corresponding differential weight loss curve (right)	15
Figure B.2 – FTIR spectra corresponding to different ashing temperature points.....	15
Figure B.3 – A 3D FTIR spectrum obtained by one FTIR-TGA measurement.....	16
Figure B.4 – Absorption dynamics of each gas component.....	17
Figure B.5 – Absorption dynamics of each gas component.....	17
Figure B.6 – Weight loss dynamics of each gas component	18
Table A.1 – Product identification (in accordance with IEC 62565-3-1).....	13
Table A.2 – General material description (in accordance with IEC 62565-3-1).....	13
Table A.3 – Test information	14
Table A.4 – Measurement results.....	14

INTERNATIONAL ELECTROTECHNICAL COMMISSION

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –**Part 6-18: Graphene-based material – Functional groups: TGA-FTIR**

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 62607-6-18 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
113/680/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.

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

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 π 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.