

# TECHNICAL SPECIFICATION



**Nanomanufacturing – Key control characteristics –  
Part 6-20: Graphene-based material – Metallic impurity content: Inductively  
coupled plasma mass spectrometry**



## THIS PUBLICATION IS COPYRIGHT PROTECTED

Copyright © 2022 IEC, Geneva, Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either IEC or IEC's member National Committee in the country of the requester. If you have any questions about IEC copyright or have an enquiry about obtaining additional rights to this publication, please contact the address below or your local IEC member National Committee for further information.

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

Tel.: +41 22 919 02 11  
[info@iec.ch](mailto:info@iec.ch)  
[www.iec.ch](http://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](http://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 It also gives information on projects, replaced and withdrawn publications.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](http://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](http://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](mailto:sales@iec.ch).

#### IEC Products & Services Portal - [products.iec.ch](http://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](http://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.

Preview generated by EVS

# TECHNICAL SPECIFICATION



**Nanomanufacturing – Key control characteristics –  
Part 6-20: Graphene-based material – Metallic impurity content: Inductively  
coupled plasma mass spectrometry**

INTERNATIONAL  
ELECTROTECHNICAL  
COMMISSION

ICS 07.120

ISBN 978-2-8322-5732-6

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

## CONTENTS

FOREWORD.....	4
INTRODUCTION.....	6
1 Scope.....	7
2 Normative references .....	7
3 Terms and definitions .....	7
3.1 General terms .....	7
3.2 Key control characteristics measured in accordance with this document .....	9
4 General .....	9
4.1 Chemical reagents .....	9
4.2 Description of measurement instrument and apparatus .....	9
4.2.1 Measurement instrument .....	9
4.2.2 Sample pre-treatment apparatus.....	9
4.2.3 Other .....	9
4.3 Calibration standards.....	10
4.3.1 Standard stock solutions.....	10
4.3.2 Internal standard (IS) solutions .....	10
5 Sample preparation method.....	10
5.1 General.....	10
5.2 Sample pre-treatment procedure.....	10
6 Measurement procedure .....	12
6.1 Calibration of ICP-MS instrument.....	12
6.2 Quantitative measurement procedure.....	12
6.2.1 Whole element scanning.....	12
6.2.2 Quantitative measurement of metal impurities.....	12
6.2.3 Method recovery measurement.....	12
6.2.4 Standard recovery measurement .....	12
7 Data analysis.....	13
7.1 Content of metal impurities in test samples .....	13
7.2 Standard recovery.....	13
8 Measurement uncertainty estimation.....	13
9 Measurement report .....	14
9.1 General.....	14
9.2 Product or sample identification .....	14
9.3 Measurement conditions .....	14
9.4 Measurement specific information.....	14
9.5 Measurement results.....	14
Annex A (informative) Case study for FLG powder .....	16
A.1 Test sample .....	16
A.2 Sample pre-treatment .....	16
A.3 Instrument information .....	16
A.4 Standard calibration curve .....	16
A.4.1 Standard stock solutions.....	16
A.4.2 Standard calibration curve .....	17
A.5 Measurement procedure .....	17
A.6 Measurement results.....	17
Annex B (informative) Case study for rGO powder .....	19

B.1	Test sample .....	19
B.2	Sample pre-treatment .....	19
B.3	Measurement instrument.....	19
B.4	Standard calibration curve .....	19
B.5	Measurement results.....	21
B.6	Standard recovery.....	22
Annex C (informative)	Comparison of different pre-treatment methods.....	24
C.1	Test sample .....	24
C.2	Comparison of different pre-treatment methods.....	24
C.2.1	GO test sample preparation .....	24
C.2.2	rGO test sample preparation.....	25
C.3	Comparison of different digestion conditions .....	25
Annex D (informative)	Results comparison of ICP-MS and ICP-OES.....	27
D.1	Test sample .....	27
D.2	Measurement results comparison between ICP-MS and ICP-OES.....	27
Bibliography	.....	28
Figure A.1	– Content distribution of metal impurities detected in FLG test sample.....	18
Figure B.1	– Standard calibration curves of several metal elements contained in rGO test sample .....	20
Figure B.2	– Content distribution of metal impurities detected in rGO test sample .....	22
Figure B.3	– Standard recovery of most species of metal impurities in rGO test sample.....	23
Figure C.1	– Result comparison of three pre-treatment methods for industrial GO powder.....	25
Figure C.2	– Result comparison of different digestion methods for industrial rGO powder.....	25
Figure C.3	– Content of metal impurities detected in rGO test sample using microwave-assisted digestion under different digestion conditions.....	26
Table 1	– Potential interferences for several typical elements in industrial graphene powder.....	12
Table A.1	– Content of all metal impurities detected in FLG test sample .....	17
Table B.1	– Content of all metal impurities detected in rGO test sample .....	21
Table D.1	– Measurement results comparison between ICP-MS and ICP-OES.....	27

## INTERNATIONAL ELECTROTECHNICAL COMMISSION

**NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –****Part 6-20: Graphene-based material – Metallic impurity content:  
Inductively coupled plasma mass spectrometry****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 62607-6-20 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
113/609/DTS	113/629/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](http://www.iec.ch/members_experts/refdocs). The main document types developed by IEC are described in greater detail at [www.iec.ch/standardsdev/publications](http://www.iec.ch/standardsdev/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](http://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

Graphene-based materials have wide potential applications because of their unique electrical, thermal and mechanical properties, especially in the electronics industry: batteries, integrated circuits, high-frequency electronics, displays, etc. [1], [2], [3]<sup>1</sup>. As industry uptake on graphene-based materials increases, international standardization is critical to enable the commercialization of graphene-based materials and related products. Metal impurities within graphene-based materials have significant impact on the electrical performance in the process of industrial application. Considering the multiple production routes and producers of graphene-based materials, in order to maintain product quality and reach a consensus between the supplier and the customer, there is no doubt that accurate, reliable and reproducible measurement methods for the key parameters of graphene-based materials need to be established.

Inductively coupled plasma mass spectrometry (ICP-MS) can carry out accurate detection of trace amounts of a variety of metal impurities simultaneously, obtain species and content of each metal impurity in graphene-based materials.

The purpose of this document is to enable accurate and quantitative determination of metal impurities using ICP-MS [4], through providing optimized digestion operation, preparation procedures for graphene-based materials in powder form, measurement method and data analysis. A similar document was published as ISO/TS 13278 for carbon nanotubes (CNTs) [5]; however, it is not suitable for graphene powder because of the noticeable difference between CNTs and graphene powder, especially in terms of sample preparation (including digestion technique and digestion procedure), the properties of test samples (many more species and much wider range of content of metal impurities in graphene powder), measurement procedure and so on. Therefore, this document has been developed for graphene powder; it is based on study in VAMAS Technical Working Area 41 (TWA 41).

---

<sup>1</sup> Numbers in square brackets refer to the Bibliography.

## NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

### Part 6-20: Graphene-based material – Metallic impurity content: Inductively coupled plasma mass spectrometry h

#### 1 Scope

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

- metallic impurity content

for powders of graphene-based materials by

- inductively coupled plasma mass spectrometry (ICP-MS).

The metallic impurity content is derived by the signal intensity of measured elements through MS spectrum of ICP-MS.

- The method is applicable for powder of graphene and related materials, including bilayer graphene (2LG), trilayer graphene (3LG), few-layer graphene (FLG), reduced graphene oxide (rGO) and graphene oxide (GO).
- The typical application area is in the microelectronics industry, e.g. conductive pastes, displays, etc., for manufacturers to guide material design, and for downstream users to select suitable products.

#### 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 [6], 3.1.2.1]