TECHNICAL SPECIFICATION

# ISO/TS 21356-1

First edition
2021-03

# Nanotechnologies — Structural characterization of graphene —

## Part 1: Graphene from powders and dispersions

*Nanotechnologies — Caractérisation structurelle du graphène —*

*Partie 1: Graphène issu de poudres et de dispersions*

Reference number
ISO/TS 21356-1:2021(E)

© ISO 2021

**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2021

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

# Contents

Page

**Foreword** iv
**Introduction** v
**1 Scope** 1
**2 Normative references** 1
**3 Terms and definitions** 1
**4 Abbreviated terms** 3
**5 Sequence of measurement methods** 3
**6 Rapid test for graphitic material using Raman spectroscopy** 5
**7 Preparing a liquid dispersion** 7
7.1 General 7
7.2 Preparing a dispersion of the correct concentration 7
7.2.1 Powder samples 7
7.2.2 Samples already in a dispersion 8
**8 Determination of methods** 8
**9 Structural characterization using optical microscopy, SEM, AFM and Raman spectroscopy** 8
**10 Structural characterization using TEM** 9
**11 Surface area determination using the BET method** 10
**12 Graphene lateral size and number fraction calculation** 10
**Annex A** (informative) **Rapid test for graphitic material using Raman spectroscopy** 11
**Annex B** (informative) **Structural characterization protocol using SEM, AFM and Raman spectroscopy** 14
**Annex C** (informative) **Structural characterization using TEM** 29
**Annex D** (informative) **Lateral size and number fraction calculation** 36
**Annex E** (informative) **Brunauer–Emmett–Teller method** 43
**Annex F** (informative) **Additional sample preparation protocols — Silicon dioxide on silicon wafer preparation and cleaning** 47
**Bibliography** 48

© ISO 2021 – All rights reserved 

# Foreword

ISO (the International Organization for Standardization) and IEC (the International Electrotechnical Commission) form the specialized system for worldwide standardization. National bodies that are members of ISO or IEC participate in the development of International Standards through technical committees established by the respective organization to deal with particular fields of technical activity. ISO and IEC technical committees collaborate in fields of mutual interest. Other international organizations, governmental and non-governmental, in liaison with ISO and IEC, also take part in the work.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives or www.iec.ch/members_experts/refdocs).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO and IEC shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents) or the IEC list of patent declarations received (see patents.iec.ch).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html. In the IEC, see www.iec.ch/understanding-standards.

This document was prepared jointly by Technical Committee ISO/TC 229, *Nanotechnologies*, and Technical Committee IEC/TC 113, *Nanotechnology for electrotechnical products and systems*.

A list of all parts in the ISO/IEC 21356 series can be found on the ISO and IEC websites.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html and www.iec.ch/national-committees.

 © ISO 2021 – All rights reserved

# Introduction

Due to the many superlative properties of graphene and related 2D materials, there are many application areas where these nanomaterials could be disruptive, areas such as flexible electronics, nanocomposites, sensing, filtration membranes and energy storage.

There are barriers to commercialisation that are impeding the progress of products containing graphene, which need to be overcome. One of these crucial barriers is answering the question "What is my material?". End-users of the raw materials containing graphene should be able to rely on the advertised properties of the commercial graphene on the global market, instilling trust and allowing worldwide trade. Reliable and repeatable measurement protocols are required to address this challenge.

This document provides a set of flow-charts for analysts to follow in order to determine the structural properties of graphene from powders and liquid dispersions (suspensions). Initially, a quick check should be undertaken to determine if graphene and/or graphitic material is present. If it is, then further detailed analysis is required to determine if the samples contain a mixture of single-layer graphene, bilayer graphene, few-layer graphene, graphene nanoplatelets and graphite particles. Depending on the methods used, the samples are typically analysed after deposition on a substrate. The document describes how to assess what measurements are required depending on the type of sample and includes decision trees and flow diagrams to aid the user. This document describes a selected set of measurands that are needed, namely:

a) the number of layers/thickness of the flakes;

b) the lateral dimensions of flakes;

c) layer alignment;

d) the level of disorder;

e) the estimated number fraction of graphene or few-layer graphene;

f) the specific surface area of the powder containing graphene.

The above physical properties of the material can change during its processing and lifetime, for example, the samples can become more agglomerated, obtain different surface functionalities. The above measurand list for the initial material defines their inherent characteristics that, along with the chosen manufacturing processes, will determine the performance of real-world products. Generally, different material properties can be important in different application areas, depending on the functional role of the material.

The document provides methods for structural characterization of individual flakes of graphene, bilayer graphene, graphene nanoplatelets and graphite particles isolated from powders and/or liquid dispersions. It does not provide methods for determination of whether the powders and/or dispersions are composed solely of these materials. No recommendation is provided as to when or how often to measure samples, although it is not expected this would be for every batch of the same material. It is up to the user to determine when, how often and which characterization routes described in this document to take. As with all microscopical investigations, care is needed in drawing statistical conclusions dependant on representative sampling.

A set of annexes provide example protocols on how to prepare and analyse the samples, sources of uncertainty and how to analyse the data. The methods used are Raman spectroscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM), transmission electron microscopy (TEM) and the BET (Brunauer–Emmett–Teller) method.

© ISO 2021 – All rights reserved

# Nanotechnologies — Structural characterization of graphene —

## Part 1: Graphene from powders and dispersions

## 1 Scope

This document specifies the sequence of methods for characterizing the structural properties of graphene, bilayer graphene and graphene nanoplatelets from powders and liquid dispersions using a range of measurement techniques typically after the isolation of individual flakes on a substrate. The properties covered are the number of layers/thickness, the lateral flake size, the level of disorder, layer alignment and the specific surface area. Suggested measurement protocols, sample preparation routines and data analysis for the characterization of graphene from powders and dispersions are given.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/TS 80004-1:2015, *Nanotechnologies — Vocabulary — Part 1: Core terms*

ISO/TS 80004-2:2015, *Nanotechnologies — Vocabulary — Part 2: Nano-objects*

ISO/TS 80004-6:2021, *Nanotechnologies — Vocabulary — Part 6: Nano-object characterization*

ISO/TS 80004-13:2017, *Nanotechnologies — Vocabulary — Part 13: Graphene and related two-dimensional (2D) materials*

© ISO 2021 – All rights reserved