

**SINGAPORE STANDARD**

**Structural characterisation of graphene flakes –  
Part 1: Methods and sample preparation**

Published by



**Enterprise  
Singapore**

**SS 643 : Part 1 : 2019**  
(ICS 07.120; 59.100.20)

---

SINGAPORE STANDARD

**Structural characterisation of graphene flakes –  
Part 1: Methods and sample preparation**

---

All rights reserved. Unless otherwise specified, no part of this Singapore Standard may be reproduced or utilised in any form or by any means, electronic or mechanical, including photocopying and microfilming, without permission in writing from Enterprise Singapore. Request for permission can be sent to: [standards@enterprisesg.gov.sg](mailto:standards@enterprisesg.gov.sg).

ISBN 978-981-48-3564-0

This Singapore Standard was approved on 20 March 2019 by the Chemical Standards Committee under the purview of the Singapore Standards Council.

First published 2019.

The Chemical Standards Committee, appointed by the Standards Council, consists of the following members:

	<b>Name</b>	<b>Capacity</b>
<b>Chairman</b>	: Dr Keith Carpenter	<i>Individual Capacity</i>
<b>Deputy Chairman</b>	: Er. Lucas Ng	<i>Individual Capacity</i>
<b>Secretary 1</b>	: Ms Elane Ng	<i>Standards Development Organisation@Singapore Chemical Industry Council</i>
<b>Secretary 2</b>	: Ms Rosmalinda Tay	<i>Standards Development Organisation@Singapore Chemical Industry Council</i>
<b>Members</b>	: Mr Goh Tiak Boon	<i>Individual Capacity</i>
	Prof Alfred Huan	<i>Individual Capacity</i>
	Mr Khong Beng Wee	<i>Individual Capacity</i>
	Mr Terence Koh	<i>Singapore Chemical Industry Council Limited</i>
	Dr Leong Kwai Yin	<i>Individual Capacity</i>
	Dr Thomas Liew	<i>National Metrology Centre</i>
	Mr Alan Lim	<i>Maritime and Port Authority of Singapore</i>
	Mr Lim Eng Kiat	<i>Individual Capacity</i>
	Mdm Jamie Lim	<i>Ministry of Manpower</i>
	Mr Lim Kian Chye / Mr Ng Eng Fu	<i>Housing &amp; Development Board</i>
	Prof Loh Kian Ping	<i>National University of Singapore</i>
	Dr Loh Wah Sing	<i>Individual Capacity</i>
	Ms Pamela Phua	<i>Singapore Paint Industry Association</i>
	Mr Seah Khen Hee	<i>Individual Capacity</i>
	A/Prof Timothy Tan	<i>Nanyang Technological University</i>
	Dr Teo Tang Lin	<i>Chemical Metrology Division, Health Sciences Authority</i>
	Ms Suzanna Yap	<i>National Environment Agency</i>
<b>Co-opted Members</b>	: Ms Christina Loh	<i>Individual Capacity</i>
	Mr Pitt Kuan Wah	<i>Individual Capacity</i>

The Technical Committee on Nanotechnology, appointed by the Chemical Standards Committee and responsible for the preparation of this standard, consists of representatives from the following organisations:

	<b>Name</b>	<b>Capacity</b>
<b>Chairman</b>	: Prof Alfred Huan	<i>Individual Capacity</i>
<b>Deputy Chairman</b>	: A/Prof Lanry Yung	<i>Individual Capacity</i>
<b>Secretary</b>	: Ms Rosmalinda Tay	<i>Standards Development Organisation@Singapore Chemical Industry Council</i>
<b>Members</b>	: Dr Hengky Chang	<i>Nanyang Polytechnic</i>
	Dr Paul Chiew	<i>Agri-Food and Veterinary Authority of Singapore</i>
	Er. Veronica Chow	<i>Ministry of Manpower</i>
	A/Prof Duong Hai Minh	<i>National University of Singapore</i>
	Mr Foo Toon Fong	<i>GLOBALFOUNDRIES Singapore Pte Ltd</i>
	Dr Thomas Liew	<i>National Metrology Centre</i>
	Dr Lim Mong Hoo	<i>PUB, Singapore's National Water Agency</i>
	Dr Lerwen Liu	<i>NanoGlobe Pte Ltd</i>
	A/Prof Ng Kee Woei	<i>National Technological University</i>
	Ms Pamela Phua	<i>AkzoNobel Paints</i>
	Dr Allen Poh	<i>Ministry of the Environment and Water Resources</i>
	Mr Sachin Gupta	<i>Rolls-Royce Singapore Pte Ltd</i>
	Dr Junie Tok	<i>P &amp; G International Operations Pte Ltd</i>
	Ms Suzanna Yap	<i>National Environment Agency</i>
	Dr Yow Soh Zeom	<i>Health Sciences Authority</i>
<b>Co-opted Members</b>	: Dr Kurnia Wira	<i>Individual Capacity</i>
	Dr Leong Yew Wei	<i>Individual Capacity</i>
	Prof Ong Wei-Yi	<i>Individual Capacity</i>
	Dr Ramam Akkipeddi	<i>Individual Capacity</i>
	Dr Sanjay Thakur	<i>Individual Capacity</i>

The Working Group on Graphene, appointed by the Technical Committee to assist in the preparation of this standard, comprises the following experts who contribute in their *individual capacity*:

	<b>Name</b>
<b>Co-Convenors</b>	: Dr Ricardo Vinicius Oliveira
	: Dr Ramam Akkipeddi
<b>Secretary</b>	: Ms Rosmalinda Tay
<b>Members</b>	: Dr Fan Rongli
	Mr Ng Chun Tat
	Dr Vitali Lipik
	Dr Yu Shengkai

The organisations in which the experts of the Working Group are involved are:

*Housing & Development Board*

*Institute of Materials Research and Engineering*

*National Metrology Centre*

*NUS Centre for Advanced 2D Materials and Graphene Research Centre*

*PUB, Singapore's National Water Agency*

*Sportmaster*

**Contents**

	<b>Page</b>
Foreword _____	6
0 Introduction _____	7
1 Scope _____	7
2 Normative references _____	7
3 Terms and definitions _____	7
4 Overview of methods for characterisation of graphene _____	9
5 Sample preparation _____	10

**Table**

1 Summary of available national and international standards for characterisation of graphene _____	9
--	---

**Figures**

1 Laboratory vacuum filtration system with membrane _____	10
2 Systematic sample preparation for graphene / graphene oxide / reduced graphene oxide _____	12
3 Separation of solid from liquid medium _____	12
4 Dispersion methodology to prepare the sample suspension _____	14
5 Test sample preparation methodology _____	15
6 Examples of some morphology images _____	16
Bibliography _____	17

## Foreword

This Singapore Standard was prepared by the Working Group on Graphene appointed by the Technical Committee on Nanotechnology under the purview of the Chemical Standards Committee.

SS 643 consists of the following four parts, under the general title, 'Structural characterisation of graphene flakes':

Part 1: Methods and sample preparation

Part 2: Determination of lateral size of graphene flakes by optical microscopy

Part 3: Determination of level of defects on graphene flakes by Raman spectroscopy

Part 4: Determination of number of layers in graphene flakes by atomic force microscopy

Part 1 is essential for the implementation of the standard and is used with Parts 2, 3 or 4.

In preparing this standard, reference was made to the following publications:

ISO 18466:2016                      Stationary source emissions – Determination of the biogenic fraction in CO<sub>2</sub> in stack gas using the balance method

ISO/TS 80004-13:2017              Nanotechnologies – Vocabulary – Part 13: Graphene and other two-dimensional materials

Acknowledgement is made for the use of information from the above publications.

This standard is expected to be used by manufacturers, buyers, users and testing laboratories involved in the production and commercialisation of graphene, academia and IHLs in Singapore which have formed the nanotechnology research entities, relevant regulatory bodies and conformity assessment bodies.

Attention is drawn to the possibility that some of the elements of this Singapore Standard may be the subject of patent rights. Enterprise Singapore shall not be held responsible for identifying any or all of such patent rights.

### NOTE

1. *Singapore Standards (SSs) and Technical References (TRs) are reviewed periodically to keep abreast of technical changes, technological developments and industry practices. The changes are documented through the issue of either amendments or revisions.*
2. *An SS or TR is voluntary in nature except when it is made mandatory by a regulatory authority. It can also be cited in contracts making its application a business necessity. Users are advised to assess and determine whether the SS or TR is suitable for their intended use or purpose. If required, they should refer to the relevant professionals or experts for advice on the use of the document. Enterprise Singapore shall not be liable for any damages whether directly or indirectly suffered by anyone or any organisation as a result of the use of any SS or TR.*
3. *Compliance with a SS or TR does not exempt users from any legal obligations.*

## Structural characterisation of graphene flakes – Part 1: Methods and sample preparation

### 0 Introduction

Graphene is a single layer of carbon atoms with each atom bound to three neighbours in a honeycomb structure [1]. Since its discovery in 2004 by two researchers at the University of Manchester, graphene has been considered a wonder material with the potential to revolutionise industrial sectors, from coatings and composites to electronics and aerospace, due to its properties such as mechanical strength, high electrical and thermal conductivity, flexibility, impermeability, etc. The estimated global market for graphene is expected to reach a minimum of \$500 million by 2025 [2].

However, as in the case of any other revolutionary material, the development of these applications and their introduction into the market strongly depends on the quality of the graphene. There are currently no established international or national standards for graphene although there is currently ongoing work, detailed in ISO/TC 229 *Nanotechnologies* and IEC/TC 113 *Nanotechnology for electrotechnical products and systems*. The lack of an established standard leads to a plethora of producers selling “graphene products” which are essentially fine graphite, and not appropriate for the applications mentioned above.

The development of this Singapore Standard will help to provide manufacturers and users with standard methods for the characterisation of graphene. In addition, this standard is intended to be used to obtain certification of graphene materials.

### 1 Scope

This part of the standard provides an overview of graphene characterisation methods and procedures in sample preparation to improve the technology used to define properties.

Procedures for sample preparation from powders and suspensions for purposes of structural characterisation are defined in this standard.

### 2 Normative references

The following referenced documents are indispensable for the application of this standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696            Water for analytical laboratory use -- Specification and test methods