

This is a preview - [click here to buy the full publication](#)

# TECHNICAL SPECIFICATION

# ISO/TS 10797

First edition  
2012-06-01

---

---

## Nanotechnologies — Characterization of single-wall carbon nanotubes using transmission electron microscopy

*Nanotechnologies — Caractérisation des nanotubes de carbone  
monofeuillet par microscopie électronique à transmission*



Reference number  
ISO/TS 10797:2012(E)

© ISO 2012



## **COPYRIGHT PROTECTED DOCUMENT**

© ISO 2012

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 ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Case postale 56 • CH-1211 Geneva 20  
Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.org](mailto:copyright@iso.org)  
Web [www.iso.org](http://www.iso.org)

Published in Switzerland

<b>Contents</b>		Page
<b>Foreword</b> .....		iv
<b>Introduction</b> .....		v
<b>1 Scope</b> .....		1
<b>2 Normative references</b> .....		1
<b>3 Terms and definitions</b> .....		1
<b>4 General principles</b> .....		3
<b>4.1 TEM imaging and analysis</b> .....		3
<b>4.2 EDS analysis</b> .....		3
<b>4.3 Additional characterization methods</b> .....		3
<b>4.4 Applicability to multiwall carbon nanotube analysis</b> .....		3
<b>5 Sample preparation</b> .....		4
<b>5.1 General principles</b> .....		4
<b>5.2 Choice of TEM grid</b> .....		4
<b>5.3 Powder and film samples</b> .....		5
<b>5.4 Liquid suspension sample</b> .....		6
<b>5.5 Composite sample</b> .....		6
<b>6 Measurement procedures</b> .....		7
<b>6.1 TEM examination of an SWCNT sample</b> .....		7
<b>6.2 EDS analysis of an SWCNT sample</b> .....		7
<b>7 Data analysis, interpretation and reporting of results</b> .....		9
<b>7.1 General principles</b> .....		9
<b>7.2 Data analysis and interpretation of TEM results</b> .....		9
<b>7.3 Data analysis and interpretation of EDS results</b> .....		13
<b>Annex A (informative) Case studies</b> .....		14
<b>Annex B (informative) Additional information on sample preparation and experimental procedures</b> ....		20
<b>Annex C (informative) Additional information on observation of SWCNTs</b> .....		26
<b>Annex D (informative) Additional information on factors influencing the observation of SWCNTs</b> .....		30
<b>Bibliography</b> .....		33

## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of document:

- an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;
- an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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

ISO/TS 10797 was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

## Introduction

Carbon nanotubes (CNTs) are nanomaterials composed of concentric layers of graphene sheets in the form of cylindrical tubes placed along the longitudinal fibre axis. Single-wall carbon nanotubes (SWCNTs) are seamless cylinders derived from the honeycomb lattice representing just a single atomic layer of graphene sheet. The transmission electron microscope (TEM), and especially its high-resolution version (HRTEM), were the first instruments that revealed the unique structural features of carbon nanotubes. TEM/HRTEM has played an essential role in the research and development of carbon nanotube materials. It has the advantage of being a “direct” technique that avoids the imposition of physical or mathematical assumptions. At the same time, it provides a variety of experimental results and information-rich images that make the investigation of a wide variety of samples possible. Beyond imaging, TEM, along with other techniques described in this Technical Specification, can provide qualitative purity assessment of SWCNT samples. In addition, it can also reveal detailed morphological and structural features of carbon nanotubes such as graphene wall structure, defects, diameter, length, bundle size and orientation, and the existence of materials and nanoparticles<sup>[8]</sup> besides SWCNTs. In other operational modes, it is also possible to study the chirality and thermal and mechanical characteristics of individual nanotubes. It is important to develop a systematic protocol for using TEM in order to acquire reliable and comprehensive information about a sample containing SWCNTs.

The transmission electron microscope operates on similar basic principles as the optical microscope but uses electrons instead of light. A beam of electrons is focused onto a thin, electron-transparent sample, allowing an enlarged version to appear on a fluorescent screen, a layer of photographic film, or on an array detector that is sensitive to electrons. Modern instruments are equipped with a computer-linked digital imaging system that can also record real-time images.

The HRTEM can investigate crystal structure by phase contrast imaging, where images are formed due to differences in the phase of electron waves scattered through a thin sample. Resolution of the TEM is limited by spherical and chromatic aberrations, but new generations of instruments with advanced electron-optical columns have significantly lowered these aberrations. Software correction of spherical aberration has allowed the production of meaningful images with sufficient resolution at magnifications of many millions times. The ability to determine the positions of atoms within materials has made the HRTEM an indispensable tool for nanotechnology research and development.

[This is a preview - click here to buy the full publication](#)

# Nanotechnologies — Characterization of single-wall carbon nanotubes using transmission electron microscopy

## 1 Scope

This Technical Specification establishes methods for characterizing the morphology of single-wall carbon nanotubes (SWCNTs) and identifying the elemental composition of other materials in SWCNT samples, using transmission electron microscopy and chemical analysis by energy dispersive X-ray spectrometry.

## 2 Normative references

The following referenced documents are indispensable for the application 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 22493, *Microbeam analysis — Scanning electron microscopy — Vocabulary*

ISO 29301, *Microbeam analysis — Analytical transmission electron microscopy — Methods for calibrating image magnification by using reference materials having periodic structures*

ISO/TS 80004-3, *Nanotechnologies — Vocabulary — Part 3: Carbon nano-objects*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*