



BSI Standards Publication

Nanotechnologies — Characterization of single- wall carbon nanotubes using ultraviolet-visible-near infrared (UV-Vis-NIR) absorption spectroscopy

National foreword

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The UK participation in its preparation was entrusted to Technical Committee NTI/1, Nanotechnologies.

A list of organizations represented on this committee can be obtained on request to its secretary.

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**Nanotechnologies — Characterization of
single-wall carbon nanotubes using
ultraviolet-visible-near infrared
(UV-Vis-NIR) absorption spectroscopy**

*Nanotechnologies — Caractérisation des nanotubes à simple couche
de carbone par utilisation de la spectroscopie d'absorption UV-Vis-NIR*





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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 normative 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 10868 was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

Nanotechnologies — Characterization of single-wall carbon nanotubes using ultraviolet-visible-near infrared (UV-Vis-NIR) absorption spectroscopy

1 Scope

This Technical Specification provides guidelines for the characterization of compounds containing single-wall carbon nanotubes (SWCNTs), using optical absorption spectroscopy.

The purpose of this Technical Specification is to describe a measurement method for establishing the diameter, purity, and ratio of metallic SWCNTs to the total SWCNT content in the sample.

The analysis of the diameter is applicable to a diameter range of 1 nm to 2 nm.

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

3 Terms, definitions and abbreviated terms

For the purposes of this document, the terms and definitions given in ISO/TS 80004-3 and the following apply.

3.1 Terms and definitions

3.1.1

purity indicator

optically defined indicator of the ratio of the mass fraction of SWCNTs to the total carbonaceous content in a sample

NOTE Purity indicator is not “purity” itself, which is defined as the percentage of mass of SWCNTs to the total mass of the sample. This guideline cannot evaluate this general purity, because absorption spectroscopy cannot detect metallic impurities that are generally contained in any SWCNT sample. In order to characterize metal impurity content, see ISO/TS 11308, which addresses thermogravimetric analysis.

3.1.2

ratio of metallic SWCNTs

optically defined compositional ratio of metallic SWCNTs to the total SWCNTs contained in the sample

3.2 Abbreviated terms

CMC	sodium carboxymethylcellulose
DMF	dimethylformamide
DOS	density of states
NIR	near infrared
SC	sodium cholate
SDS	sodium dodecyl sulfate
SDBS	sodium dodecylbenzene sulfonate
SWCNT	single-wall carbon nanotube
TEM	transmission electron microscope
UV	ultraviolet
VHS	van Hove singularity
Vis	visible

4 Principle

4.1 General

All SWCNT samples contain both semiconducting and metallic SWCNTs, together with impurities consisting of carbon and other elements, unless the samples have been altered after production. UV-Vis-NIR absorption spectroscopy can be used for the measurement of interband optical transitions specific to SWCNTs. The analysis of these optical transitions provides qualitative and semi-quantitative information important for the characterization of SWCNT samples, such as mean diameter, purity, and the ratio of metallic SWCNTs to the total SWCNT content.

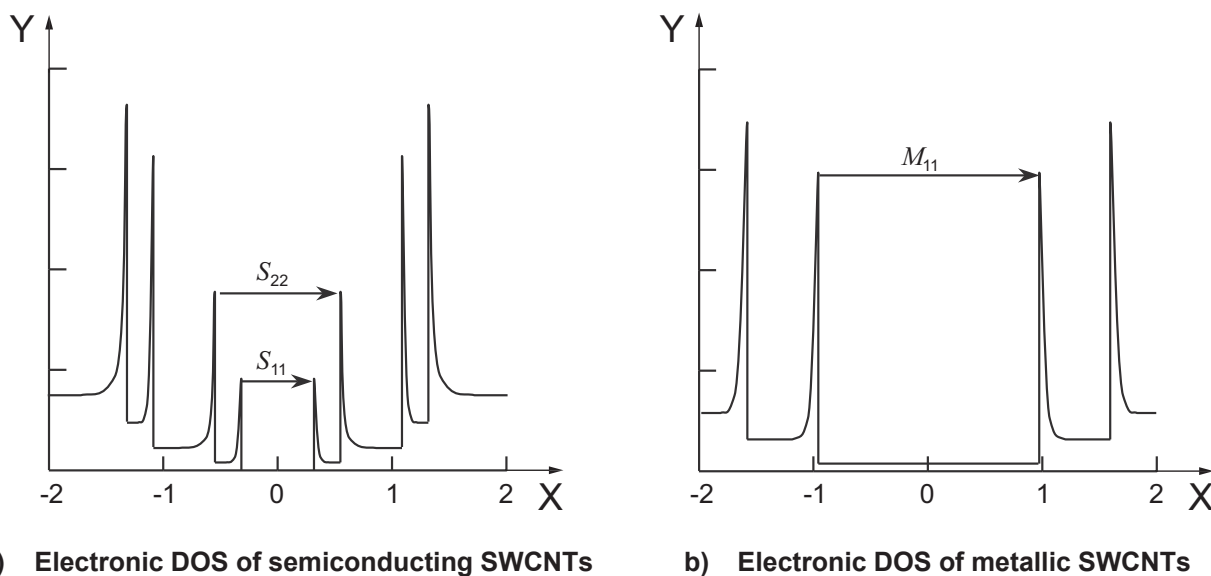
4.2 UV-Vis-NIR absorption spectroscopy

The intensity of light, I , passing at a specified wavelength, λ , through a specimen is measured and compared to the intensity of light before it passes through the specimen, I_0 . The ratio I/I_0 is called a transmittance. The absorbance, A , is expressed as $-\log(I/I_0)$. The plot of the absorbance against wavelength for a particular compound is referred to as an absorption spectrum.

NOTE The relationship between transmittance and absorbance is only rigorously correct when reflectance is negligible.

4.3 Optical absorption peaks of SWCNTs in the UV-Vis-NIR region

The shape of the electronic DOS of semiconducting and metallic SWCNTs shown in Figure 1 is a series of spikes that are referred to as VHS. The peaks observed in optical absorption spectra of SWCNTs are attributed to the electronic transitions between these VHSs as shown by arrows in Figure 1. S_{11} and S_{22} are used as the symbols of the absorption due to the first and second interband transitions of semiconducting SWCNTs, respectively [see Figure 1a)]. M_{11} represents the absorption arising from the first interband transition of metallic SWCNTs [see Figure 1b)].



Key

X Energy (eV)

Y Electronic DOS (arbitrary unit)

S_{11} The first interband optical transition attributed to semiconducting SWCNTs

S_{22} The second interband optical transition attributed to semiconducting SWCNTs

M_{11} The first interband optical transition attributed to metallic SWCNTs

NOTE See Reference^[2].

Figure 1 — Electronic DOS diagram of SWCNTs near the Fermi level

To interpret absorption spectra of SWCNTs, band structures calculated using the zone-folding method are frequently used. The electronic structure of a SWCNT is generally given by that of a two-dimensional graphite sheet expressed by the tight binding approximation as shown in Equation (1)^[3].

$$E_{2D} = \pm \gamma \left[1 \pm 4 \cos \left(\frac{\sqrt{3} k_x a}{2} \right) \cos \left(\frac{k_y a}{2} \right) + 4 \cos^2 \left(\frac{k_y a}{2} \right) \right]^{1/2} \quad (1)$$

where

E_{2D} is the two dimensional energy dispersion relation for a single graphene sheet;

a is the lattice parameter;

k_x and k_y are the components of the reciprocal unit vector;

γ is the overlap integral.

4.4 Relation between SWCNT diameter and optical absorption peaks

Within a simple tight-binding theory, in which the electronic band structure is assumed to arise from a pure p-orbital at each conjugated carbon atom, the low-energy band gap transitions take a simple analytical form. The energy gaps corresponding to the electron transitions are given by the following equations.

$$E_g(S_{11}) = \frac{2a\gamma}{d} \quad (2)$$