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PUBLICLY AVAILABLE SPECIFICATION

PRE-STANDARD

colour inside

Nanomanufacturing – Material specifications –
Part 2-1: Single-wall carbon nanotubes – Blank detail specification

IEC/PAS 62565-2-1:2011(E)

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NANOMANUFACTURING – MATERIAL SPECIFICATIONS –

Part 2-1: Single-wall carbon nanotubes – Blank detail specification

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IEC-PAS 62565-2-1 has been processed by IEC technical committee 113: Nanotechnology standardization for electrical and electronic products and systems.

The text of this PAS is based on the following document:	This PAS was approved for publication by the P-members of the committee concerned as indicated in the following document
Draft PAS	**Report on voting**
113/100/PAS	113/105A/RVD

Following publication of this PAS, which is a pre-standard publication, the technical committee or subcommittee concerned may transform it into an International Standard.

This PAS shall remain valid for an initial maximum period of 3 years starting from the publication date. The validity may be extended for a single period up to a maximum of 3 years, at the end of which it shall be published as another type of normative document, or shall be withdrawn.

After publication of future IEC 62565-2-1, this IEC-PAS 62565-2-1 will be withdrawn.

A bilingual version of this publication may be issued at a later date.

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INTRODUCTION

This Publicly Available Specification is intended to provide guidance on how to list, illustrate and define various characteristics of single-wall carbon nanotubes (SWCNTs) for industrial use in electronic products, and how to incorporate these into a bilateral detail specification between vendor and user.

One particular point of interest is the fact that there are different modifications of carbon nanotubes. Subtle differences in the physical structure lead to marked differences in electrical, optical and chemical properties; therefore these characteristics need special attention.

To permit common processing equipment and common unit processes with predictable and reproducible results to be used in multiple fabrication lines, it is essential for the carbon nanotubes characteristics to be described and assessed in a standardized manner and to standardize the methods for quality control of the manufacturing processes.

To enable low-cost mass production (or production of pure fractions), a reliable, affordable means of preparing one type of carbon nanotubes (e.g. single-wall semiconducting carbon nanotubes, with a certain specified length) is necessary. To facilitate a reliable source of carbon nanotubes with tailored properties (length, diameter, purity, chirality, conduction type), it is necessary to specify the characteristics in a standardized way, stating the specification limits and the characterization methods to prove conformance. This does not only reduce transaction costs, but eliminates a major source of error, as explained below.

Accurately measuring and characterising the quality of nanotube-containing materials and the dispersion of nanotubes in liquids or polymers, are both considered crucial for the continued growth of applications incorporating single-wall carbon nanotubes. Significant differences in both methodology and interpretation continue to exist from one measurement laboratory to another. For this reason, comparison and specification of the quality of CNT materials is extremely difficult. While progress in these measurements is being been made, significant improvements are still needed to accurately measure and characterise the quality of carbon nanotube-containing materials and the protocol for doing so (e.g. how to describe / specify the characteristics relevant for the quality of the final nano-enabled product).

Furthermore, the development of reference materials is as important as improvements to measurement / characterization techniques. In addition, it is stressed that for any of the analysis methods, it is mandatory to specify the sample preparation method, sample size and the sampling method.

Experiences with this PAS should be reported to the Secretariat of IEC Technical Committee 113 to provide improvements for the future IEC 62565 International Standards under development in IEC/TC 113.

NANOMANUFACTURING – MATERIAL SPECIFICATIONS –

Part 2-1: Single-wall carbon nanotubes – Blank detail specification

1 Scope

This PAS establishes a blank detail specification for the essential electrical properties and certain other common characteristics including dimensional, structural and mechanical properties of single-wall carbon nanotubes.

This PAS provides a standardized format for detail specifications characterising essential basic properties of single-wall nanotubes and recommends measurement methods.

Single-wall carbon nanotubes with a chemical modification, dispersed into a solvent or grown on a substrate are included.

Properties and characteristics not of relevance for a specific application may be classified as not applicable or not specified.

NOTE 1 The present state of the art in manufacturing carbon nanotubes does not produce purely single-wall carbon nanotubes. The consequences are reflected in the requirements part.

NOTE 2 A revisable version of Tables 2 to 8 is attached to this file. These tables are intended to be used in the detail specification to be agreed between manufacturer and user of single-wall carbon nanotubes.

Microsoft Word Document

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.

IEC 62624, *Test methods for measurement of electrical properties of carbon nanotubes*

NOTE 1 Supplementary information is provided in the bibliography

NOTE 2 Terminology and nomenclature are under development in IEC/TC113/JWG 1 in cooperation with ISO/TC 229. Published terminology standards or specifications from this group will be incorporated into this document.

NOTE 3 Measurement and characterization are under development in IEC/TC113/JWG 2 in cooperation with ISO/TC 229. Published measurement standards or specifications from this group will be incorporated into this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

NOTE 1 Terminology and nomenclature are under development in IEC/TC113/JWG 1 in cooperation with ISO/TC 229. Published definitions from this group will be incorporated into this document. Not yet specified definitions are taken from the scientific literature.

NOTE 2 Measurement and characterization are under development in IEC/TC113/JWG 2 in cooperation with ISO/TC 229. Published definitions from this group will be incorporated into this document. Not yet specified measurement methods are taken from the scientific literature.

3.1
chirality

twist of carbon nanotubes, determined by the values of n and m for the chiral vector $\vec{C}_h$ according Figure 1

NOTE Chirality affects the conductance of the nanotube, density, lattice structure, and other properties. The chiral vector is defined in Table 1 in terms of the integers (n, m) and the basis vectors of the lattice, which are given in terms of rectangular coordinates.

3.2
diameter of single-wall carbon nanotubes

d_t

diameter uniquely determined by the integers (n, m)

3.3 Acronyms and abbreviations

AFM	Atomic Force Microscopy
BET	Brunauer-Emmett-Teller method to determine the surface area by gas absorption [2] 1)
CNT	Carbon Nanotube
CVD	Chemical Vapour Deposition
EDX	Energy Dispersive X-Ray Fluorescence Spectrometry
EFM	Electrostatic Force Microscopy
GPC	Gel Permeation Chromatography
HPLC	High Performance Liquid Chromatography
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
NIR	Near Infrared Spectroscopy
PL	Photoluminescence
Raman	Raman Spectroscopy
SWCNT	Single-wall Carbon Nanotube
MWCNT	Multiwall Carbon Nanotube
SEM	Scanning Electron Microscopy
SGM	Scanning Gate Microscopy
SPM	Scanning Probe Microscopy
SPS	Surface Photo Voltage Spectroscopy
STS	Scanning Tunnelling Spectroscopy
SThPM	Scanned Thermal Probe Microscopy

1) Numerals in square brackets refer to the Bibliography.

STM	Scanning Tunnelling Microscopy
TGA	Thermogravimetric Analysis
TG-MS	Thermogravimetry–Mass Spectrometry
TEM	Transmission Electron Microscopy
UV	Ultraviolet Spectroscopy
UV-vis-NIR	UV-vis-NIR Absorption Spectroscopy
XPM	X-ray Photoelectron Microscopy
XPS	X-ray Photoelectron Spectroscopy

4 Basic information

This clause summarizes the fundamental characteristics for single-wall carbon nanotubes, gives the basic relations governing these parameters and lists typical numeric values for these parameters.

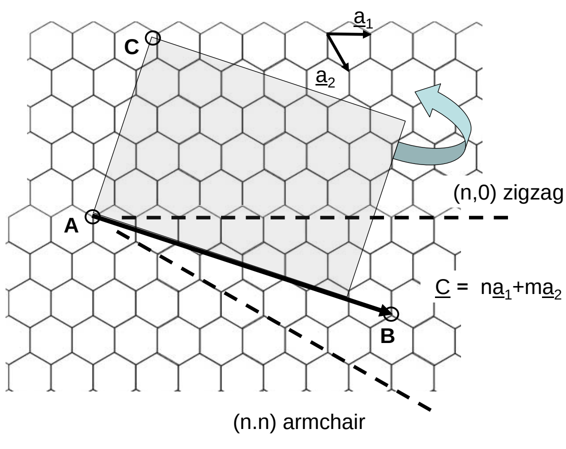

Figure 1 – Two-dimensional graphene sheet with vectors defining chirality

In single carbon layers of graphite each carbon atom is bound to three neighbours in a honeycomb structure. The chiral vector C_h is the connection between the points A and B, which coincide when the gray area is rolled up to form a short section of a carbon nanotube. The axis of the tube is parallel to the line AC. For the special case $n = m$ (as defined in Figure 1 and Table 1) the **armchair configuration** results, and for $m = 0$ the **zig zag configuration** results, see Figure 2 and Figure 3. Depending on the values of n and m the carbon nanotube is either semiconducting or metallic. The angle between the vector a_1 and the chiral vector C_h is defined as the chiral angle Θ. The length of chiral vector L is directly related to the tube diameter.

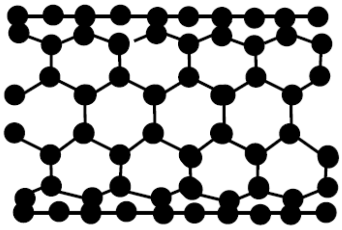

Figure 2 – Example of armchair tube (θ = 30° direction, θ as defined in Table 1)
(view perpendicular to the CNT axis)

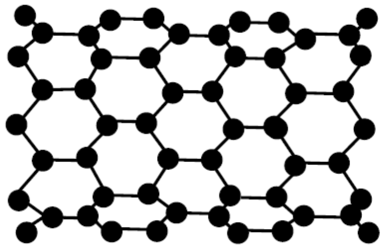

Figure 3 – Example of zigzag tube (*θ* = 0° direction, θ as defined in Table 1),
(view perpendicular to the CNT axis)

Table 1 – Parameters of single-wall carbon nanotubes [1]

Symbol	Name	Formula	Value
$a_{\text{C-C}}$	Carbon-Carbon distance sp^2		0,1421 nm for graphene
A	Length of unit vector	$\sqrt{3}a_{C-C}$	in (x, y) coordinates
$\underline{a}_1, \underline{a}_2$	Unit vectors	$\left(\sqrt{3}/2, \frac{1}{2}\right)a, \left(\sqrt{3}/2, -\frac{1}{2}\right)a$	in (x, y) coordinates
$\underline{C}_h$	Chiral vector	$n \cdot \underline{a}_1 + m \cdot \underline{a}_2 \equiv (n, m)$	n, m: integers
L	Circumference of nanotube	$a\sqrt{n^2 + m^2 + nm} = \lvert \underline{C}_h \rvert$	$0 \leq \lvert m \rvert \leq n$
d_{t}	Diameter of nanotube	$\frac{L}{\pi}$	
Θ	Chiral angle (inner wall)	$\tan\theta = \frac{\sqrt{3}m}{2n+m}$	$0 \leq \lvert \theta \rvert \leq 30°$

5 General introduction regarding measurement methods

The specification of material parameters of carbon nanotubes has to refer to measurement methods for which currently no standards exist. Standardized methods for the characterization of carbon nanotubes are under development. For reasons of practicality for industrial use in manufacturing of electronic products, this PAS recommends only one measurement method for each material parameter.

In the absence of adequate standardized method for industrial use, the user shall fulfil the following documentation requirements:

– description of the sample preparation;
– measurement procedure;
– sample size and statistical significance;
– description how the original measurement data are converted to the specified material parameter(s).

The choice of the measurement methods and procedures has to be carried out with respect to the application of the material. Furthermore, it has to take into account cost, robustness and efficiency of the method.

6 Basic specification requirements

A basic specification is one that describes a commercially and technically appropriate single wall-carbon nanotube product having stable quality and parametric control. Single-wall carbon nanotubes produced to fulfil this specification shall be qualified through routine process checks (in the manufacturing process of the carbon nanotubes), demonstrating that the process is in a state of control.

The list of characteristics provided in the Table 3 should be used as the basic specification requirement.

7 Recommended single-wall carbon nanotubes specification format

7.1 General procurement information

The state of the art in manufacturing carbon nanotubes does not produce purely single-wall carbon nanotubes. Therefore the general procurement information as detailed in Table 2 shall be given by the supplier.

Table 2 – Format for general information

Item	Information	Date
General specification number		
Revision level		
Part number / Revision		
Growth method	[ ] Laser ablation; [ ] High pressure carbon monoxide process; [ ] CVD; [ ] Arc synthesis; **[ ] Combustion**[ ] Other (specify):	
Functionalization (details to be provided)	Covalent [ ] non-covalent functionalization [ ] end / tip functionalization [ ] side wall functionalization [ ]	
Dispersion agent		

Item	Information	Date
Dispersion Method		

7.2 Single-wall carbon nanotubes characterization

7.2.1 General characteristics

General characteristics as detailed in Table 3 shall be agreed between manufacturer and user. Properties and characteristics not of relevance for the application may be classified as not applicable or not specified.

Table 3 – Format for general characteristics [1]

Item		Specification	Recommended method(s)	Other measurement methods
3-1	Orientation	[ ] armchair; [ ] zigzag; [ ] chiral Chiral vector (specify): n = [ ]; m = [ ]	Raman, TEM	Fluorescence spectroscopy
3-2	External diameter	[ ] Nominal [ ] ± Tolerance [ ] nm	TEM	AFM, Fluorescence, SEM; SPM; Raman; PL
3-3	Length	[ ] Nominal [ ] ± Tolerance [ ] µm	SEM	TEM; SPM; Raman;
3-4	SWCNT content	Greater than: [ ] wt %	TGA	NIR; Raman; ICP-MS;
3-5	Other carbon content	Not greater than: [ ] wt%	TGA	NIR; ICP-MS; Raman: XPM
3-6	Metal content	Not greater than: [ ] wt%	ICP-MS (ISO/TS 13278)	TGA; NIR; XRF, XPS
3-7	Other impurities	Not greater than: [ ] wt%	ICP-MS (ISO/TS 13278	XPS

7.2.2 Electrical characteristics

Electrical characteristics as detailed in Table 4 to Table 6 shall be agreed between manufacturer and user. Properties and characteristics not of relevance for the application may be classified as not applicable or not specified.

NOTE 1 The measured values of the electrical characteristics are in many cases dependent on the type and properties of the contacts made to the nanotube(s). The values also depend on whether one or more tubes are being measured. That needs to be accounted for in the specifications, where appropriate.

NOTE 2 Electrical characteristics listed in Tables 4 -6 are supposed to be related to measurements on single-wall carbon nanotubes. It should be documented in the report how the tubes are sampled and whether the sample is representative of the distribution of types of nanotubes in the measured batch of material.

NOTE 3 For further information on electrical characteristics see [3] through [8].

Table 4 – Format for electrical characteristics

Item		Specification	Single CNT or batch or both	Specified method	Other measurement methods
4-1	Conductivity type	[ ] Percent metallic [ ] type 1 wide band [ ] type 2 narrow band	Batch	ISO/TS 10812 ISO/TS 10868	EFM; SGM; STM; STS

Table 5 – Format for electrical characteristics, metallic single-wall CNTs

Item		Specification	Single CNT or batch or both	Specified method	Other measurement methods
5-1	Resistivity	[ ] ± x% Ω*m at 20°C; [ ] Nominal [ ] ± Tolerance [ ] Ω*m	Both	IEC 62624	EFM; SGM; STM; STS;
5-2	Maximum current density	[ ] ± *x*% A/m^2; [ ] Nominal [ ] ± Tolerance [ ] A/m^2	Both	IEC 62624	EFM; SGM; STM; STS;

Table 6 – Format for electrical characteristics, semiconducting single-wall CNTs

Item		Specification	Single CNT or batch or both	Specified method	Other measurement methods
6-1	Mobility	[ ] ± *x*% m^2/Vs; [ ] Nominal [ ] ± Tolerance [ ] m^2/Vs	Both	Specify, no recommendation yet	
6-2	Energy band gap between filled and empty electron states	E_g = Nominal [ ] ± Tolerance [ ] eV	Single CNT	Specify, no recommendation yet	
6-3	Carrier type	[ ] n [ ] p	Single CNT	Specify, no recommendation yet	
6-4	Dopant concentration	[ ] Nominal [ ] ±Tolerance [ ] m^{-3}	Both	Specify, no recommendation yet	
6-5	Other electrical characteristics (as required)				

7.2.3 Optical characteristics

Optical characteristics as detailed in Table 7 shall be agreed between manufacturer and user. Properties and characteristics not of relevance for the application may be classified as not applicable or not specified

Table 7 – Format for optical characteristics

Item		Specification	Single CNT or batch or both	Specified method NOTE	Other measurement methods
7-1	Absorption spectrum	To be determined	Both	Optical absorbance spectrum	
7-2	Others (as required)				

7.2.4 Mechanical and dimensional characteristics

Mechanical and dimensional characteristics as detailed in Table 8 shall be agreed between manufacturer and user. Properties and characteristics not of relevance for the application may be classified as not applicable or not specified.

Table 8 – Format for mechanical and dimensional characteristics

Item		Specification	Single CNT or batch or both	Specified method	Other measurement methods
8-1	Young's modulus	[ ] Nominal [ ] ± Tolerance [ ] Pa	Single tube	Specify, no recommendation yet	
8-2	Maximum tensile strength	[ ] Nominal [ ] ± Tolerance [ ] Pa	Single tube	Specify, no recommendation yet	
8-3	Thermal conductivity	[ ] ± *x*% W/mK; [ ] Nominal [ ] ± Tolerance [ ] W/mK	Both	SThM	AFM
8-4	Specific surface area	Nominal [ ] ± Tolerance [ ] m^2/g	Batch	BET	XPM

8 Test methods overview

The most extensively utilized techniques are TGA, SEM, TEM, Raman and UV-vis-NIR spectroscopy. AFM and other SPM methods are also being used.

TGA quantitatively determines the amounts of carbon and non-carbon materials in bulk samples, as well as nanotube homogeneity and thermal stability representing a good indicator of nanotube quality.

SEM images give a rough idea of quality, while higher-resolution TEM images can monitor the surface texture of individual tubes and help characterise metals and non-tubular carbon impurities.

Raman spectroscopy offers a quick test for the determination of single-wall carbon nanotubes content. It also can qualitatively estimate carbon content, distinguish the types of nanotubes present, and corroborate SEM and TEM data, which due to the nature of microscopy can only characterise a very small sample of the material for which inhomogeneous materials may not be representative of the total material.

UV-vis-NIR spectroscopy can be used to characterise the carbon present in a sample and identify the presence of individual and bundles of single-wall carbon nanotubes.

For a qualitative analysis of a sample containing single-wall CNTs, TEM and SEM should be used. The TEM image demonstrating the existence of a significant quantity of single-wall CNTs is an important measure of the quality of the material. The methodology by which TEM and SEM images are selected should be specified. For quantitative estimation, a combination of TGA, Raman, and NIR methods is recommended.

PL and TG-MS can be used for the characterization of SWCNTs.

For measurements of the electrical resistivity, IEC 62624:2009, *Test methods for measurement of electrical properties of carbon nanotubes* is recommended.

Table 9 – Summary of test methods

Property/ Category	Method						
	SEM/EDX	TEM	Raman Spectroscopy	UV-vis-NIR Absorption	NIR-PL/ Fluorescence	TGA	TG-MS
Morphology	Tube structure, bundle, thickness, orientation	Wall structure, amorphous carbon, metal catalyst coatings					
Purity	Non-carbon impurities	Tube surface cleanliness	CNT and non-CNT carbon	Carbonaceous content (Quantitative)		Non- carbon content (Quantitative) Non- CNT content (Quantitative)	Non- carbon content (Quantitative)
Length and diameter	Length and diameter	Tube diameter, metal cluster size	Diameter	Diameter	Diameter		
Tube Type			Metallic / Semi-conducting	Metallic / Semi-conducting	Chirality; (Semi-conducting)		
Dispersa-bility/ Solubility	Tube bundling			Tube bundling or separation (dispersion)	Tube bundling		
Additional						Oxidation/ transition temperatures	Oxidation/ transition temperatures
Property/ Category	Method						
	AFM	SGM	SPM	STS	SThPM	STM	
Morphology	Tbd	Tbd	Tbd	Tbd	Tbd	Tbd	
Purity	Tbd	Tbd	Tbd	Tbd	Tbd	Tbd	
Length and diameter	Tbd	Tbd	Tbd	Tbd	Tbd	Tbd	
Tube Type	Tbd	Tbd	Tbd	Tbd	Tbd	Tbd	
Dispersa-bility/ Solubility	Tbd	Tbd	Tbd	Tbd	Tbd	Tbd	
Additional	Tbd	Tbd	Tbd	Tbd	Tbd	Tbd	

NOTE 1 Tbd = to be determined in Joint Working Group 2 of IEC/TC113 and ISO/TC229.

NOTE 2 The definition of the properties of Column 1 are still work in progress in Joint Working Group 1 of IEC/TC113 and ISO/TC229.

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