
**Nanotechnologies — Carbon
nanotube suspensions —
Specification of characteristics and
measurement methods**

*Nanotechnologies — Suspensions de nanotube de carbone —
Spécification de caractéristiques et méthodes d'essais*

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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Contents

	Page
Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms, definitions and abbreviated terms	1
3.1 Terms and definitions	1
3.2 Abbreviated terms	2
4 Characteristics and measurement methods	3
5 Sampling	3
5.1 Sampling principle	3
5.2 Sampling devices	4
5.3 CNT retrieving	4
6 Description of characteristics and measurement methods	4
6.1 Outer diameter	4
6.2 Specific surface area	4
6.3 Homogeneity	5
6.4 Morphology	5
6.5 Dry solid content	5
6.6 CNT content	5
6.7 Viscosity	6
6.8 Fineness	6
6.9 Elemental impurity content	6
6.10 pH value	7
6.11 Water content	7
6.12 Thermal conductivity	7
6.13 Volume resistivity	7
6.14 Shelf life	8
7 Test report	8
Annex A (informative) Overview of CNT suspension characteristics	9
Annex B (informative) Case study of carbon nanotube dispersion	10
Bibliography	11

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.

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 ISO documents 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).

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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.

This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

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.

Introduction

Carbon nanotubes (CNT) have attracted great interest due to their wide scope of possible applications, such as composite reinforcement material, hydrogen containers, super-capacitors, molecular sensors and scanning probe tips. Offering attractive mechanical, electric and thermal properties, CNTs could achieve a significant improvement in bulk properties by adding low weight percentages.

The performance of nano-objects can be degraded with the formation of agglomerates or aggregates in post-processing. Suspensions of the appropriate fluids and additives will stabilize nano-objects, preventing agglomeration and reducing losses to the environment during handling. It is widespread practice in the manufacturing industry to pre-treat nano-objects by making suspensions before delivery to the downstream customers. Industrial products based on CNT suspensions are a good example.

Since CNT suspensions containing multi-walled carbon nanotubes (MWCNTs) are widely supplied nowadays, it is timely to develop appropriate specifications. Such specifications would facilitate the communication between interested parties and the commercialization of CNT suspensions, and help to generate consistent performance in the final products.

A number of characterization documents related to CNT have been developed by ISO/TC 229, in which measurement methods and procedures for characteristics including morphology, impurities, volatile components, etc. are specified. This document specifies the characteristics to be measured of CNT suspension samples and describes their measurement methods. ISO/TR 10929 describes the characteristics to be measured of bulk samples of MWCNTs and their measurement methods. ISO/TR 13097 provides guidelines on how to characterize the stability of suspensions. It includes general guidance on how to specify the suspension in terms of its physical and chemical characteristics, which might affect its performance or subsequent processing.

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Nanotechnologies — Carbon nanotube suspensions — Specification of characteristics and measurement methods

1 Scope

This document specifies the characteristics to be measured of suspensions containing multi-walled carbon nanotubes (CNT suspensions). It includes the essential and additional characteristics of the CNT suspension, and the corresponding measurement methods.

Characteristics specific to health, environmental and safety issues are excluded from this document.

WARNING — The use of this document can involve hazardous materials, operations and equipment. It does not purport to address all of the safety or environmental problems associated with its use. The execution of this document is entrusted to appropriately qualified and experienced people.

2 Normative references

There are no normative references in this document.

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1.1

agglomerate

collection of weakly or medium strongly bound particles where the resulting external surface area is similar to the sum of the surface areas of the individual components

[SOURCE: ISO/TS 80004-2:2015, 3.4, modified — Notes 1 and 2 to entry have been deleted.]

3.1.2

aggregate

particle comprising strongly bonded or fused particles where the resulting external surface area is significantly smaller than the sum of surface areas of the individual components

[SOURCE: ISO/TS 80004-2:2015, 3.5, modified — Notes 1 and 2 to entry have been deleted.]

3.1.3

carbon nanotube

CNT

nanotube composed of carbon

Note 1 to entry: Carbon nanotubes usually consist of curved graphene layers, including single-wall carbon nanotubes and multi-walled carbon nanotubes.

[SOURCE: ISO/TS 80004-3:2010, 4.3]

3.1.4

carbon nanotube suspension

CNT suspension

suspension containing multi-walled carbon nanotubes

Note 1 to entry: Single-wall carbon nanotubes may be included in the suspension.

3.1.5

representative sample

random sample selected in such a way that the observed values have the same distributions in the sample as in the population

[SOURCE: ISO 3534-2:2006, 1.2.35, modified — EXAMPLE and Notes 1 and 2 to entry have been deleted.]

3.1.6

shelf life

recommended time period during which a product (CNT suspension) can be stored, throughout which the defined quality of a specified property of the product remains acceptable under expected (or specified) conditions of distribution, storage, display and usage

[SOURCE: ISO/TR 13097:2013, 2.14, modified — “(CNT suspension)” has replaced “(dispersion)”.]

3.1.7

dry solid content

mass fraction of substances remaining after completion of a specified heating process

Note 1 to entry: Adapted from ISO 13580:2005, 3.1.

3.1.8

suspension

heterogeneous mixture of materials comprising a liquid and a finely dispersed solid material

[SOURCE: ISO/TS 80004-6:2013, 2.13]

3.1.9

viscosity

measure of the internal friction of a fluid when caused to flow by an external force

[SOURCE: ISO 13503-1:2011, 2.14]

3.1.10

volume resistivity

quotient obtained when the potential gradient is divided by the current density

[SOURCE: ISO 472:2013/Amd.1:2018, 3.17, modified — Note 1 to entry has been deleted.]

3.2 Abbreviated terms

CNT	carbon nanotube
ICP-AES	inductively coupled plasma-atomic emission spectrometry
ICP-OES	inductively coupled plasma-optical emission spectrometry
ICP-MS	inductively coupled plasma-mass spectrometry
MWCNT	multi-walled carbon nanotube
SEM	scanning electron microscopy
TEM	transmission electron microscopy

4 Characteristics and measurement methods

Given the various applications of CNT suspension, the characteristics to be measured are different for desired applications. These characteristics in this document are classified into two categories:

- the essential characteristics of CNT suspension that shall be measured for all applications;
- the additional characteristics of CNT suspension that should be measured depending on specific applications.

The essential characteristics of both a CNT suspension and its constituent CNTs listed in [Table 1](#) shall be measured and reported to the buyer. The additional characteristics of CNT suspension listed in [Table 2](#) should be measured depending on applications.

The measurement method for each characteristic is provided in [Tables 1](#) and [2](#).

The CNT suspension characteristics are summarized in [Annex A](#).

Table 1 — Essential characteristics of CNT suspension and measurement methods

Characteristics	Measurement methods
Outer diameter	See 6.1
Specific surface area	See 6.2
Homogeneity	See 6.3
Morphology	See 6.4
Dry solid content	See 6.5
CNT content	See 6.6
Viscosity	See 6.7

Table 2 — Additional characteristics of CNT suspension and measurement methods

Characteristics	Measurement methods
Fineness	See 6.8
Elemental impurity content ^a	See 6.9
pH value ^{a,b}	See 6.10
Water content ^a	See 6.11
Thermal conductivity ^c	See 6.12
Volume resistivity ^{b,c}	See 6.13
Shelf life	See 6.14
^a Applicable to Li-ion battery application. ^b Applicable to composite material application. ^c Might be used in some adhesives, e.g. silicone.	

5 Sampling

5.1 Sampling principle

The representative sample should be taken from the CNT suspension and original CNT powder products. The amount of samples should be sufficient for the subsequent measurements. The detailed sampling method should conform to ISO 15528.

5.2 Sampling devices

When taking samples from a suspension product, the sampling devices should be able to take suspension samples from all layers of the product in the container (top, middle and bottom). Disposable pipettes are recommended so that different levels (top, middle and bottom) of the sample can be taken from the suspension product.

5.3 CNT retrieving

When a CNT test specimen in powder form is needed for measuring the characteristics, the test specimen is retrieved from the suspension sample by heating the suspension to fully remove the liquid. In cases where the CNT test specimen in powder form cannot be retrieved from the CNT suspension sample, the original CNT powder sample before dispersion can be used for measuring the characteristics.

6 Description of characteristics and measurement methods

6.1 Outer diameter

The outer diameter of a fibrous object is the distance on a two-dimensional microscopic image between the two outer edges on a cross-sectional line orthogonal to the longitudinal direction of fibre. One datum of outer diameter is obtained for each fibrous object. Each diameter datum is randomly obtained from the fibrous object without deliberately measuring the narrower or wider area of the image of the fibrous object. The target fibrous objects to be measured shall be representative of the fibrous solid objects contained in a CNT suspension sample, i.e. all types of fibrous objects on an image shall be equally selected for measurements. The number of diameter data may be agreed between the interested parties.

The diameter shall be measured by TEM or SEM. SEM can be used when the spatial resolution is precise enough for the target CNT diameters. When a test specimen for measurement is prepared, the CNT concentration of the suspension is adjusted to be dilute enough.

The measurement results shall be displayed as a histogram of the number of fibrous objects versus diameter at the interval of 1 nm. Also, the average (median) of diameter data of fibrous objects shall be expressed in the unit of nm. It should be noted that the measurement results may be qualitative with increased uncertainty when the observed microscopic images lack the representativeness of sample.

ISO/TS 10797 and ISO/TS 10798 specify the protocols for diameter measurement of single-wall CNTs by TEM and SEM, respectively. These documents can be useful for MWCNTs.

6.2 Specific surface area

Specific surface area (SSA) refers to the absolute surface area of the sample divided by the sample mass. The SSA is one of the fundamental characteristics of CNTs and affects the performance of products containing CNTs.

The original CNT powder samples are preferably used for the SSA measurement, irrespective of 5.3. The SSA shall be measured using the gas adsorption method. The results of SSA measurements shall be expressed in the unit of m^2/g .

The analysis technique based on the model developed by Brunauer, Emmett and Teller (BET) allows the specific surface area of a powder to be estimated by measuring the amount of gas that is adsorbed. The BET analysis is the standard method for determining the specific surface area from nitrogen adsorption isotherms. ISO 9277 applies to the measurement of the specific surface area. This document specifies the measurement procedures for the overall specific external and internal surface areas (diameter > 2 nm) of disperse or porous solids by measuring the amount of physically adsorbed gas according to the BET analysis. ISO 18757 provides some useful detailed information concerning specific materials. Measurement instruments for the BET method are commercially available. Metrological

traceability should be maintained. Reference materials are available for the application of the BET analysis to nanoparticles in powder form.

6.3 Homogeneity

Homogeneity of a CNT suspension sample is the measure of how uniformly distributed constituents of the CNT suspension are throughout a larger sample, as determined by measuring representative smaller samples.

The homogeneity of CNT suspension shall be examined by visual observation. A sample for examination is taken from the CNT suspension product into a transparent container. The container with sample is left statically for more than 24 h before examination. It is examined whether the colour is black and uniform over the surfaces of suspension sample and whether there is a phase separation and sedimentation. The examination results shall be reported qualitatively.

6.4 Morphology

Morphology of a CNT suspension sample refers to the shapes and structures of CNTs and other solid objects contained in the suspension sample. The morphology shall be measured qualitatively to observe the presence of CNTs and other solid objects contained in a CNT suspension sample.

Microscopic images of solid objects in a CNT sample shall be obtained by TEM or SEM. SEM can be used when the spatial resolution is precise enough for the target CNT sizes.

The CNT suspension sample is sufficiently diluted by adding anhydrous ethanol at an adequate concentration for the TEM and SEM measurements. Each image shall be accurately representative of the solid objects contained in a CNT suspension sample and be taken so that CNTs can be clearly observed. The scale bar is shown on each image.

More than five microscopic images shall be taken and reported.

6.5 Dry solid content

A CNT suspension sample may contain solid components other than CNTs and dissolved materials. The dry solid content can be an indication of CNT content in the suspension sample when impurities are negligible. The dry solid content of CNT suspension sample is the ratio of the mass of the CNT suspension after drying to that of the CNT suspension before drying.

Dry solid content shall be measured by the oven-drying method, which consists of drying the suspension sample to constant mass at temperatures between 100 °C and 120 °C and weighing. The measurement results of dry solid content shall be expressed as the mass percentage, in % mass fraction.

NOTE When dissolved materials in a CNT suspension sample are negligible, the dry solid content is closely equal to the solid content of the sample.

6.6 CNT content

A CNT suspension sample is predominantly composed of carbonaceous materials, which can be removed by combustion at appropriately high temperatures. The sample may contain metallic and inorganic impurities, which are left as ash after combustion. The ash content is measured by weighing the residue after combustion at 800 °C ± 25 °C using an electric furnace.

The CNT content of a CNT suspension sample is the difference between the dry solid content and the ash content, provided that the suspension has negligible non-CNT carbon and non-volatile organic matters. The results of CNT content should be expressed as the mass percentage, in % mass fraction.

6.7 Viscosity

Viscosity is a rheological property of a fluid that expresses resistance to shearing flows. The viscosity of a CNT suspension is a fundamental characteristic for liquid handlings, and its data are usually taken at low shear rates, which are correlated with the length of CNTs and their dispersion state.

A test specimen is taken from the suspension sample and left statically for more than 10 h before measurement in order to avoid the influence resulted from the thixotropy of CNT suspension at the measurement starting point.

The viscosity shall be measured by rotational rheometry at one shear rate in the range between $0,2 \text{ s}^{-1}$ and 2 s^{-1} and can be optionally measured more at other shear rates.

The viscosity results at $25 \text{ }^{\circ}\text{C}$ shall be expressed in the unit of $\text{Pa}\cdot\text{s}$. The shear rates at which the viscosity measurements are taken, the dispersion medium and viscometer type used (e.g. single cylinder, concentric cylinder, cone and plate or others) shall be reported along with the viscosity results.

6.8 Fineness

The fineness is measured to evaluate the dispersion state of a CNT suspension. A case study of carbon nanotube dispersion is given in [Annex B](#).

The fineness refers to a reading obtained on a standard gauge under specified conditions of test, indicating the depth of the groove(s) of the gauge at which discrete solid particles in the product are readily discernible.

As-made CNT is usually in the form of agglomerate or aggregate, therefore, the fineness should be used to evaluate the extent of dispersion of CNT suspension by determining the maximum size of CNT agglomeration. If the maximum size is less than a certain size, usually $5 \text{ }\mu\text{m}$, it means CNT has been well dispersed.

The fineness of CNT suspension should be measured by use of a suitable gauge, graduated in micrometres. Typically, 1 or 2 drops of CNT suspension sample is taken with a pipette onto a standard fineness plate and scraped from top to bottom with a standard scraper, from which the size of the solid particles in the CNT suspension is visually determined. If the scrape is not uniform, an appropriate thinner or binder solution may be added to the sample by hand stirring, and the test is repeated.

The measurement results shall be displayed as a histogram of the number of test samples versus the maximum size. Also, the average (median) of the maximum size shall be expressed in the unit of μm .

The detailed measurement procedures of fineness of grind should conform to ISO 1524:2013.

6.9 Elemental impurity content

Elemental impurities are likely introduced into a CNT suspension sample during the manufacturing processes of CNTs. The elemental impurity content of a CNT suspension sample is the ratio of the mass of elemental impurities included in the sample to that of the sample. The elemental impurity content should be measured by wet chemical digestion and ICP-AES/OES or ICP-MS. A dried test specimen in powder form is first prepared from the suspension sample, followed by digesting them in the mixture of nitric acid, sulfuric acid and perchloric acid in the ratio of 3:3:2. Elements in the digested solution were finally tested by ICP-AES/OES or ICP-MS with corresponding working curves. The measurement results should be expressed as mass percentage, in % mass fraction, for each element present.

ISO/TS 13278 specifies the protocols for elemental impurity content measurements of single-wall CNTs and MWCNTs by ICP-MS. ISO 22036 specifies the protocols for elemental impurity content measurements of soils by ICP-AES/OES that can be useful for CNT suspension measurements.

6.10 pH value

pH value is a measure of the concentration of acidity or alkalinity of a material in suspension. It is one of the fundamental characteristics of the suspension. The pH of the CNT suspension sample should be quantitatively measured by a pH meter. The result should be expressed as a number between 0 and 14. ISO 787-9 specifies a general method of test for determining the pH value of an aqueous suspension of pigment or extender. It can be useful for CNT suspension measurements.

6.11 Water content

Water content refers to the quantity of water contained in a material. For CNT non-aqueous suspensions, the water content of the suspensions is needed to strictly control for certain applications, e.g. lithium ion battery application.

The water content of the CNT suspension should be measured upon the agreements between the interested parties. The Karl Fischer titration method is recommended for the measurement. The measurement shall be conducted performed immediately after the sample was unsealed to avoid moisture absorption from the environment. The relative humidity of the measurement environment does not exceed 80 % and ambient temperature shall be approximately 5 °C to 30 °C.

The CNT suspension sample is sufficiently diluted by adding ethyl acetate removed of water with molecular sieves at an adequate concentration. Then the mixture is stirred and centrifuged, and the supernatant is taken for water content measurement. The result shall be expressed as mass percentage, in % mass fraction.

6.12 Thermal conductivity

Thermal conductivity refers to the heat-transfer rate per unit area of a material divided by the temperature gradient causing the heat transfer. It is usually temperature-dependent, i.e. it varies with temperature.

A test specimen for measurements is prepared by drying from the CNT suspension sample. The thermal conductivity should be measured using the transient plane heat source (hot disc) method. The experimental arrangement can be designed to match different specimen sizes. Measurements can be made in gaseous and vacuum environments at a range of temperatures and pressures. The results of thermal conductivity measurement should be expressed in the unit of W/(m·K).

ISO 22007-2 specifies a method for the determination of the thermal conductivity and thermal diffusivity, and hence the specific heat capacity per unit volume of plastics. This method is suitable for testing homogeneous and isotropic materials, as well as anisotropic materials with a uniaxial structure. ISO 22007-2 can be useful to the thermal conductivity measurement procedure for the sample dried from the CNT suspension.

6.13 Volume resistivity

Volume resistivity is used to evaluate the electrical performance of a material. Generally, the less the volume resistivity is, the better the electric performance of the CNT suspension.

There are two options for sample preparation for volume resistivity, which should be agreed between the interested parties. One is to dry CNT suspension samples to solids and compress them in a pellet form. The other is to mix CNT suspension samples with a non-conductive material, e.g. TiO₂, followed by heating and compressing in a pellet form. For the first option, the pressure when preparing the pellet test specimen shall be reported together with the measurement results and the pellet density. For the second option, the type and quality of non-conductive material and its mass fraction should be reported in order to produce compatible measurement results of volume resistivity for CNT suspension samples.

The volume resistivity of CNT suspension should be measured using four-point probe resistivity meter.

The measurement results should be expressed in the unit of $\Omega \cdot m$.

The detailed measurement procedures should conform to IEC 62899-202.

6.14 Shelf life

The shelf life of a CNT suspension product should be given by the manufacturer. It is verified by observing homogeneity and measuring dry solid content and viscosity. If during the given time period there is a phase separation and sedimentation by visual observation, or the dry solid contents of the upper, middle and lower layers or the viscosity of the suspension sample exceeds the specified ranges of deviation from the initial value that are agreed by the buyer and the seller, then the product has expired. An acceleration test method for shelf life and corresponding storage conditions may be agreed by the interested parties.

The examination results should be reported.

7 Test report

The test report shall contain at least the following information:

- reference to this document, i.e. ISO/TS 19808;
- identification (product name, chemical name);
- sample description:
 - supplier name of a CNT suspension product;
 - batch number or lot number;
 - chemical name of solvent;
 - chemical name of additives as listed in the (materials) safety data sheet;
 - manufacturing method of original CNT powder, e.g. arc, CVD;
 - sampling; from a stirred product or from a layer (top/middle/bottom) of the product;
- results:
 - measurement results of characteristics, with the name of them and the measurement methods used in [Tables 1](#) and [2](#) when requested;
 - measurement dates and names of measurement laboratories for individual characteristics;
 - source of a powder test specimen, retrieved from the suspension sample or taken from the original CNT powder sample;
 - uncertainty (subject to the agreement between users, suppliers and regulators);
- shelf life guaranteed by manufacturer, if requested, and the measurement results of homogeneity, dry solid content and viscosity at the end of shelf life with the agreed storage conditions and acceleration test method, if any;
- deviation from this document;
- additional information.

Annex A (informative)

Overview of CNT suspension characteristics

An overview of CNT suspension characteristics is given [Table A.1](#).

Table A.1 — Overview of CNT suspension characteristics

CNT characteristics	Medium characteristics	Suspension characteristics	Application specific characteristics
Morphology ^a Outer diameter ^a Specific surface area ^a Fineness ^b	Chemical name of solvent ^c Chemical name of additives ^c	Homogeneity ^a Visual appearance ^c Colour ^c Dry solid content ^a CNT content ^a Viscosity ^a	Elemental impurity content ^b pH value ^b Water content ^b Thermal conductivity ^b Volume resistivity ^b Shelf life ^b
^a Essential characteristics. ^b Additional characteristics. ^c Other related characteristics.			

Annex B **(informative)**

Case study of carbon nanotube dispersion

B.1 General

CNTs, as made, often exist as entangled agglomerates. In order to fully utilize the unique properties of this special material, it is important to apply appropriate dispersion procedures. This annex provides a few case studies that can help to achieve this goal.

B.2 Carbon nanotube dispersion — High viscosity material

There are basically three ways of incorporating CNTs into high viscosity melts:

- melt impregnation;
- solvent impregnation;
- in situ polymerization.

Melt impregnation is generally accepted as the easiest approach. In order to achieve an even distribution of CNTs, agglomerates of CNTs are dispersed by high shear forces.

However, care is taken in order to achieve the optimum dispersion of CNTs while minimizing any potential breakage or degradation of the conductive fillers.

Excellent dispersion results can be achieved by processing the thermoplastic materials in a twin screw extruder. It can be used for the production of compounds as well as for masterbatches containing a high concentration of CNTs in a polymer matrix, which can be diluted in either single screw or twin screw extruders.

For rubber materials, the use of standard Banbury equipment or three roll mill are often used.

B.3 Carbon nanotube dispersion — Medium viscosity material

CNTs can be dispersed in medium viscosity media such as polyols or epoxide prepolymers by processing them in a three roll mill or a torus mill. Ultrasonic treatment is also suitable if the viscosity of the matrix is low and the concentration of CNTs is not too high (e.g. < 2 %). It has been found that CNT agglomerates cannot be dispersed by using high speed stirrers or dissolver discs.

B.4 Carbon nanotube dispersion — Low viscosity material

CNTs can be dispersed in low viscosity media such as water and organic solvents by using a jet disperser. Ultrasonic treatment is also suitable. Stabilizers (surfactant) need to be added to aqueous dispersions, in particular, to ensure that the nanotubes do not re-agglomerate. Examples of suitable surfactants include sodium dedocyl sulfate, dodecylbenzene sulfonate, etc.