PD 6699-1:2007

PUBLISHED DOCUMENT

Nanotechnologies –

Part 1: Good practice guide for specifying manufactured nanomaterials

ICS 71.100.99

British Standards

Publishing and copyright information

ISBN 978 0 580 58723 8

The following BSI references relate to the work on this Published Document:
Committee reference NT/1

Publication history

First published December 2007

Amendments issued since publication

Amd. no.	Date	Text affected

Contents

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 16, an inside back cover and a back cover.

Foreword

Publishing information

This Published Document is published by BSI and came into effect on 31 December 2007. BSI Committee NTI/1, *Nanotechnologies*, takes collective responsibility for its preparation. The Committee wishes to acknowledge the contribution of Powdermatrix. A list of organizations represented on the Committee can be obtained on request to its secretary.

This Published Document was commissioned by the UK Department for Innovation, Universities and Skills (DIUS) to provide guidance for manufacturers and users of nanomaterials on the preparation of comprehensive technical specifications to help ensure reproducibility of delivered product properties.

Use of this document

As a guide, this Published Document takes the form of guidance and recommendations. It should not be quoted as if it were a specification and particular care should be taken to ensure that claims of compliance are not misleading.

It has been assumed in the preparation of this Published Document that the execution of its provisions will be entrusted to appropriately qualified and experienced people, for whose use it has been produced.

Presentational conventions

The provisions in this Published Document are presented in roman (i.e. upright) type. Its recommendations are expressed in sentences in which the principal auxiliary verb is "should".

The word "may" is used in the text to express permissibility, e.g. as an alternative to the primary recommendation of the clause. The word "can" is used to express possibility, e.g. a consequence of an action or an event.

Contractual and legal considerations

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

This Published Document is not to be regarded as a British Standard.

Introduction

The need for this guide arises from experience that, on occasions, agreed specifications between suppliers and users of manufactured nanomaterials have failed to ensure delivery of material, on a batch-to-batch basis, that responds consistently to downstream processing and/or generates consistent performance in the final product.

This observed inconsistent performance of batches of material, all apparently "in specification", has led to the conclusion that the cause has to be related to one or more of the following.

a) The specification agreed between user and supplier does not cover all material characteristics that have an influence on performance and/or processability or has been interpreted differently by the user and supplier.

b) One or more material characteristic is currently being measured by an inappropriate technique.

c) One or more measurement technique is being applied in an incorrect manner.

This guide is intended to address all of these issues.

Each broad category of manufactured nanomaterial has been considered in a separate clause; those at the nanoscale in all three dimensions, those at the nanoscale in two dimensions and those at the nanoscale in one dimension. As the most significant subset of two-dimensional nanomaterials, carbon nanotubes have been considered separately to other examples such as nanorods or nanofibres.

Through a process of discussion with an expert consultative group, for each category of manufactured nanomaterial:

- a list has been compiled of material characteristics believed to have a definite influence on product performance and/or downstream processing in all areas of application and which have therefore to be controlled in order to achieve product consistency;
- further material characteristics have been identified, which have a definite influence in certain areas of application;
- where the inclusion of all of these material characteristics in agreed specifications still does not ensure batch-to-batch consistency, a further list of characteristics is provided which could be investigated;
- for all identified material characteristics, appropriate measurement methods are recommended, which separate into two categories: those generally utilizing relatively low-cost equipment, which can be envisaged for use in routine batch quality control in an industrial environment, and those which require expensive equipment and which might therefore only be viable for use in less frequent audit checks;
- a brief description of the basis of each measurement method is provided and, wherever possible, reference is made to an appropriate source of guidance on best practice in carrying out the test (usually an established standard); and
- where no viable or validated measurement method can currently be identified, this is also stated.

1 Scope

This Published Document provides guidance on the preparation of comprehensive technical specifications for manufactured nanomaterials in order to ensure the delivery of product that behaves in a reproducible manner. The document includes guidance on specifying the physical and chemical characteristics of manufactured nanomaterials, which might affect performance or subsequent processing.

This document does not include guidance on specifying the health and safety characteristics of manufactured nanomaterials, nor does it include guidance on specifying bulk materials containing nanosized phases.

2 Terms and definitions

For the purposes of this Published Document, the terms and definitions given in PAS 136 apply.

3 Specifying manufactured nanomaterials having all three dimensions at the nanoscale, i.e. nanopowders

3.1 Material characteristics with a definite influence on product performance and/or downstream processability

The following characteristics are essential in specifying manufactured nanopowders in all areas of application.

- Particle size distribution.
- Crystallite size distribution.
- Degree of agglomeration.
- Specific surface area.
- Bulk chemical composition.

3.2 Additional characteristics that have a definite influence in specific areas of application

Where manufactured nanopowders are to be used in specific areas of application, it will be essential to specify the following additional characteristics.

- Dispersibility in solid matrices – in specifying nanoscale reinforcements in composite materials.
- Dispersibility in liquids – polar and non-polar.
- Fuchs surface area – use of nanoparticles in aerosols.
- Composition across the particle cross-section – particles with a core shell structure.

3.3 Other material characteristics with a possible influence on product performance and/or downstream processability

To ensure reproducible behaviour in processing and consistent product performance, it might be necessary to specify other characteristics from the following list.

- Particle morphology.
- Flow characteristics.
- Tap density.
- Apparent density.
- Porosity.
- Surface chemical composition.
- Crystal structure.
- Moisture content.
- pH.
- Colour.
- Transparency.

4 Specifying manufactured nanomaterials having two dimensions at the nanoscale – carbon nanotubes

4.1 General

Carbon nanotubes have been identified as the most commercially significant example of nanomaterials with two dimensions at the nanoscale. For this reason, this clause is dedicated specifically to carbon nanotubes. Other types of two-dimensional nanomaterials are addressed in Clause **5**.

4.2 Material characteristics with a definite influence on product performance and/or downstream processability

The following characteristics are essential in specifying carbon nanotubes in all areas of application.

- Length distribution.
- Diameter distribution.
- Aspect ratio distribution.
- Wall thickness.
- Number of walls, i.e. single-walled, double-walled, or multi-walled.
- Chemical purity – presence of catalyst.
- Structural/Product purity – presence of other carbon-based materials.

4.3 Additional characteristics that have a definite influence in specific areas of application

Where nanotubes are to be used in specific areas of application, it will be essential to specify the following additional characteristic.

- Symmetry – for electrical properties.

4.4 Other material characteristics with a possible influence on product performance and/or downstream processability

To ensure reproducible behaviour in processing and consistent product performance, it might be necessary to specify other characteristics from the following list.

- Dispersibility.
- Degree of agglomeration.
- Surface chemical analysis.
- Surface functionalization.
- Structure at the ends, i.e. open or closed.

5 Specifying other manufactured nanomaterials with two dimensions at the nanoscale, i.e. nanofibres and nanorods

5.1 Materials characteristics with a definite influence on product performance and/or downstream processability

The following characteristics are essential in specifying manufactured nanofibres or nanorods in all areas of application.

- Size distribution.
- Length distribution.
- Diameter distribution.
- Aspect ratio distribution.
- Degree of agglomeration.
- Surface area.
- Porosity.
- Bulk chemical analysis.

5.2 Additional characteristics that have a definite influence in specific areas of application

Where nanofibres and nanorods are to be used in specific areas of application, it will be essential to specify the following additional characteristics.

- Dispersibility in solid matrices in specifying nanoscale reinforcements for composites.
- Strength of the interface with the matrix in specifying nanoscale reinforcements for composites.
- Crystallographic and mechanical anisotropy.

5.3 Other material characteristics with a possible influence on product performance and/or downstream processability

To ensure reproducible behaviour in processing and consistent product performance, it might be necessary to specify other characteristics from the following list.

- Compatibility with matrices.
- Dispersibility in liquid.
- Flow characteristics.
- Surface chemical analysis.
- Surface functionalization.

6 Specifying manufactured nanomaterials having only one dimension at the nanoscale, i.e. nanoscale thin films or coatings

6.1 General

The control of quality of nanoscale coatings is best achieved by defining optimum coating process conditions, initially by the measurement of agreed characteristics of the coating. Batch-to-batch control of coating quality is then achieved by monitoring and control of process conditions rather than by routine measurement of coating characteristics. The characteristics listed in **6.2** and **6.3** are those which should be considered in the initial definition of coating conditions. Relevant measurement methods for coating characteristics are therefore listed in Table 2 rather than Table 1.

6.2 Material characteristics with a definite influence on product performance

The following characteristics are essential in defining process conditions for the control of quality of nanoscale coatings.

- Coating thickness.
- Thickness uniformity.
- Chemical composition of the coating.
- Interfacial strength with the substrate.

6.3 Other material characteristics with a possible influence on product performance and/or downstream processability

To ensure consistent product performance from nanoscale coatings, it might be necessary to consider other characteristics from the following list.

- Porosity.
- Surface chemical composition.

7 Recommended measurement methods for determining the characteristics of manufactured nanomaterials identified as important for specification purposes

Appropriate measurement methods separate into two categories: those generally utilizing relatively low-cost equipment for use in routine batch quality control in an industrial environment (see Table 1), and those which require expensive equipment and which might therefore only be viable for use in less frequent audit checks (see Table 2).

Sampling of powders should be carried out according to BS 3406-1.

As many nanomaterials are reactive, their physical and chemical properties can be affected by the sampling point and their storage environment. Consequently, supplier and purchaser should agree the sampling point and storage of the samples for comparability of results.

Table 1 **Measurement methods for batch quality control**

Metric	Method	Guidance
1. Particle size distribution		Different measuring techniques might yield different results for the same material batch. In specifying the material, supplier and purchaser should agree the technique used to measure the particle size distribution.
	Light scattering methods	Applicable for particle diameters greater than about 40 nm, depending on the nature of the particle. Calibration of the equipment and measurement procedure should be in accordance with BS ISO 13320-1 or BS ISO 21501-2.
	Electron microscopy and image analysis	Guidelines for the calibration of image magnification can be found in BS ISO 16700. Guidance on static image analysis methods is available in BS ISO 13322-1.
	Electrokinetic sonic amplitude testing	A variant of zeta potential testing, in which an alternating voltage is applied to a suspension to produce a sound wave from the vibration induced in the suspended particles. Depending on the material properties, such as density, particle size distribution information for particles over 100 nm in diameter can be derived from the phase lag of the induced sound wave, whereas particle size distribution for finer nanoparticles (5 nm to 100 nm) can be derived from the attenuation of the amplitude of the sound wave.
2. Particle morphology, including aspect ratio	Electron microscopy and image analysis	Guidelines for the calibration of image magnification can be found in BS ISO 16700. Guidance on static image analysis methods is available in BS ISO 13322-1.

Table 1 **Measurement methods for batch quality control** (*continued*)

Metric	Method	Guidance
3. Crystallite size	X-ray diffraction line broadening (XRDLB)	A technique for measuring the size and strain of individual crystals under about 100 nm, where the Debye rings (X-ray lines) generated by the procedure are broadened. It should be noted that line broadening can be caused by strain in the material as well as changes in crystallite size. Guidance can be found in BS EN 13925-1, BS EN 13925-2 and BS EN 13925-3.
4. Degree of agglomeration	Scanning electron microscopy	Guidance on static image analysis methods is available in BS ISO 13322-1.
	Calculation of agglomeration index	Calculation from measurements of mean crystallite size, d, by X-ray diffraction line broadening (3) and mean particle size, D, by a light scattering method (1). The agglomeration index, T, is given by the ratio, D/d.
5. Surface area	BET analysis	A technique based on the model developed by Brunauer, Emmet and Teller that allows the surface area of powders to be determined by gas adsorption. Typically, nitrogen or carbon dioxide is used, but gases such as krypton or argon may be used for low surface area materials because of their sensitivity (mass gain per unit area). Specific surface area is the ratio of surface area to mass. Guidance on this method can be found in BS 4359-1:1996, ISO 9277:1995 and BS EN ISO 18757.
6. Porosity	Electron microscopy and image analysis	Guidelines for the calibration of image magnification can be found in BS ISO 16700. Guidance on static image analysis methods is available in BS ISO 13322-1.
	Pycnometry	Porosity can be measured using a pycnometer, a flask of known volume. A known weight and volume of powder is added to the pycnometer, which is then weighed, filled with a liquid or gas of known density, in which the powder is completely insoluble, and weighed again. The volume of the added liquid or gas can be determined and the difference between the combined volume of the powder and liquid/gas and the volume of the pycnometer gives the pore volume of the powder.
7. Crystal structure	X-ray diffraction	Guidance can be found in BS EN 13925-1, BS EN 13925-2 and BS EN 13925-3.
8. Compatibility with matrices	Raman spectroscopy	Spectroscopy in which the Raman effect is used to investigate molecular energy levels. The Raman effect involves the scattering of light with a change of frequency characteristic of the scattering material, representing a change in the vibrational, rotational or electronic energy of the substance that can be used to give information on the chemical bonding or mechanical stress state.
9. Dispersibility in liquids	Zeta potential methods	Zeta potential is the electrostatic potential at the slipping plane (which marks the region where the liquid molecules surrounding the particle first begin to move with respect to the surface) relative to the potential in the bulk solution. Guidance on measurement and characterization of particles by acoustic methods is available in BS ISO 20998-1.
10. Dispersibility in solid matrices	Electron microscopy	Guidelines for the calibration of image magnification can be found in BS ISO 16700. Guidance on static image analysis methods is available in BS ISO 13322-1.
	X-ray diffraction	Guidance can be found in BS EN 13925-1, BS EN 13925-2 and BS EN 13925-3.
	Rheometry	Guidelines on the determination of complex shear viscosity using a parallel-plate oscillatory rheometer are given in BS ISO 6721-10.

Table 1 **Measurement methods for batch quality control** (*continued*)

Metric	Method	Guidance
11. Flow characteristics of powders		The available methods are those already established for the characterization of flow of coarser powders.
	Annular shear cell	The annular shear cell measures the shear stress and dilatancy of a granular material or a powder.
	Hall flowmeter	This method is based on measurement of the time taken for a given mass of powder to flow through a conical funnel with a specified cone angle and aperture diameter. For metallic powders, guidance on funnel design and experimental procedure is given in BS EN ISO 4490.
	Hausner ratio and Carr index	These can be calculated from determined values of tap density and apparent density. Hausner ratio equals tap density/apparent density and high values of this ratio indicate poor flow behaviour. $$\text{Carr index} = 100 \times \frac{(\text{tap density} - \text{apparent density})}{\text{tap density}}$$ These indices are widely used measures of powder flow characteristics, particularly in the pharmaceutical sector.
	Jenike shear cell	The Jenike shear cell measures the unconfined strength, internal friction angle, effective internal angle of friction and wall friction angle as functions of consolidation or solid contact pressures.
12. Tap density of powders		The available method is that already established for the measurement of tap density of coarser powders. A relevant procedural standard is BS EN ISO 3953, which relates to metallic powders.
13. Apparent density of powders		The available methods are those already established for the measurement of apparent density of coarser powders. These methods involve the filling of a cup of known volume with powder and then weighing the powder collected in the cup. BS EN 23923 covers two procedures for the dispensing of the powder into the cup; the funnel method (BS EN 23923-1) and the Scott volumeter method (BS EN 23923-2).
14. Surface chemical analysis		There are no techniques currently available for surface chemical analysis of manufactured nanomaterials which can be recommended for batch quality control in an industrial environment. Several techniques which can be applied to the analysis of surface composition for audit check purposes are listed in Table 2 (5).
15. Bulk chemical analysis		Manufacturers should consider the expected composition of material and likely impurities when determining the most appropriate analytical approach to be adopted. Standards are published covering the many aspects of chemical analysis, such as sample preparation, quality of water and analytical reagents.
16. Surface roughness	Confocal microscopy	A confocal microscope system provides measurement of the variation in height of the sample (z-axis) as it is moved across the detection plane using an xy translation stage. Such a system uses point illumination and a pinhole in an optically conjugate plane in front of the detector to eliminate out-of-focus information. As only one point is illuminated at a time in confocal microscopy, 2D or 3D imaging requires scanning over a regular raster (i.e. a rectangular pattern of parallel scanning lines) in the specimen. The resolution of the measurements are <1 μm in x- and y-axes and <10 nm in the z-axis. Hence microfeatures and topographic variations can be monitored in detail. Sample sizes from a few mm^2 to 100 mm × 100 mm can be measured.
	Ellipsometry	Ellipsometry analyses the change of polarization of light, which is reflected off a sample, and may be used to assess surface uniformity and roughness.

Table 2 **Measurement methods for audit check purposes**

Metric	Method	Guidance
1. Particle size distribution		Different measuring techniques might yield different results for the same material batch. In specifying the material, supplier and purchaser should agree the technique used to measure the particle size distribution.
	Condensation particle counter	This is the most widely used type of instrument for detecting and counting nanoparticle aerosols. The instrument operates by condensing vapour, such as alcohol or water, onto sampled ultrafine particles to grow them to a size range that can be detected by a standard optical counter. The technique is applicable to the measurement of particle diameters of 100 nm or below.
	Scanning mobility particle sizer	This technique detects and counts nanoparticles and is applicable to the measurement of aerosol size distribution in the range 3 nm to 800 nm. The technique operates by charging particles and separating them based on their mobility when passing between electrodes.
	Photon correlation spectroscopy	This method measures diffusion diameter from Brownian motion. It is applicable to the measurement of particle diameters greater than 3 nm. Guidelines on the application of the method can be found in BS 3406-8:1997, ISO 13321:1996.
	X-Ray diffraction	Guidance can be found in BS EN 13925-1, BS EN 13925-2 and BS EN 13925-3.
2. Particle morphology	Electron microscopy	Particle morphology can be measured using scanning electron microscopy and transmission electron microscopy. Guidance on static image analysis methods is available in BS ISO 13322-1.
	Scanning probe microscopy	Scanning probe microscopy covers several related technologies for imaging and measuring surfaces on a fine scale. A sharp tip mounted on a flexible cantilever is scanned across the object profiling the surface. Atomic force microscopy, scanning tunnelling microscopy and near field scanning optical microscopy are types of scanning probe microscopy.
	Atomic force microscopy	Atomic force microscopy provides shape and structural information of nanoparticles. A very sharp tip (10 nm diameter apex) is moved across the surface in a raster-like fashion whilst being moved up and down (z direction) to maintain the force between tip and sample constant. The three motions provide a map of the surface being investigated. The technique provides direct measurement in the z direction of dimensions such as height, surface roughness and volume.

Table 2 **Measurement methods for audit check purposes** (*continued*)

Metric	Method	Guidance
2. Particle morphology *(continued)*	Scanning tunnelling microscopy (STM)	A technique for revealing the apparent electron-density-related atomic structure of surfaces, using a needle-like probe near the object under observation; a tunnelling current, which is measured, is generated by altering the potential at the tip of the probe. A 3D representation of the sample surface is generated by rastering the surface of the object and mapping the position of the tip for constant current level at various points.
	Near field scanning optical microscopy (NSOM)	A technique for imaging surfaces in transmission or reflection by mechanically scanning an optical probe much smaller than the wavelength of light over the surface whilst monitoring the transmitted or reflected light. The detector is placed very close to the specimen surface, which allows surface inspection with high spatial resolution.
3. Surface area	Aerosol diffusion charging	A method in which the Fuchs surface area of an aerosol is measured directly, by passing electrically neutral particles through a unipolar ion cloud and measuring the resulting aerosol charge. When the charging rate is low, aerosol charge is proportional to the Fuchs surface area.
	Epiphaniometer	Instrument used to measure the Fuchs surface area of aerosols directly. The aerosol is passed through a charging chamber where lead isotopes created from a decaying actinium source are attached to the particle surfaces. The particles are transported through a capillary to a collecting filter. The amount of radioactivity measured is proportional to the particle surface area.
4. Crystallite size	Electron backscatter diffraction (EBSD)	This technique can be used to measure crystallite size. EBSD is conducted using a scanning electron microscope (SEM) equipped with a backscatter diffraction camera. Electrons interact with the atomic lattice planes of the crystalline structure of the sample, satisfy Bragg conditions and undergo backscatter diffraction. Crystallite size is calculated from the resulting pattern.
	Transmission electron microscope diffraction	This technique can be used to measure crystallite size. Electrons interact with the atomic lattice planes of the crystalline structure of the sample, satisfy Bragg conditions and undergo diffraction. Different lattice orientations, corresponding with different crystallites, will result in different diffraction intensities. Crystallite size is calculated from the resulting pattern.

Table 2 **Measurement methods for audit check purposes** (*continued*)

Metric	Method	Guidance
5. Surface chemical analysis		With the exception of EELS and AES, these techniques have spatial resolution at the nanometre scale in one direction (z-axis), but can only resolve at the micrometre scale in the other two orthogonal directions (x- and y-axes). In the context of this guide, therefore, the techniques are applicable in the majority of cases to nanoscale thin films and coatings rather than 2D and 3D nanomaterials.
	Auger electron spectroscopy (AES)	A technique in which an electron spectrometer is used to measure the energy distribution of Auger electrons emitted from a surface. Guidance on the method can be found in PD ISO/TR 18394 and BS ISO 20903.
	Dynamic secondary ion mass spectrometry (D-SIMS)	This technique provides elemental and simple molecular information from the surface through to several micrometres. The technique can be used for profiling elemental or molecular distributions to sensitivities of ppb.
	Electron energy loss spectroscopy (EELS)	A technique where inelastic interaction of an electron beam with atoms in a sample results in an energy distribution spectrum that contains compositional and chemical bonding information. EELS provides resolution at the nanoscale in the x- and y-axes.
	Ion beam analysis (IBA)	A method to elucidate composition and structure of the outermost atomic layers of a solid material, in which principally monoenergetic, single-charged probe ions scattered from the surface, are detected and recorded as a function of their energy or angle of scattering or both.
	Secondary ion mass spectrometry (SIMS)	A method in which a mass spectrometer is used to measure the mass-to-charge quotient and abundance of secondary ions emitted from a sample as a result of bombardment by energetic ions. Guidance on the method can be found in BS ISO 22048 or BS ISO 18114.
	Time of flight secondary ion mass spectrometry (ToF-SIMS)	This method is highly surface sensitive where the sampling depth is only 1 to 2 monolayers (often < 2 nm). It is analytically very sensitive (often trace detection levels) but it is not quantitative. Its chemical specificity, however, may be exploited in a semi-quantitative manner where variations in the surface concentrations of different chemical species can be followed by monitoring changes in the relative intensities of their diagnostic ToF-SIMS signals.
	Total reflection X-ray fluorescence spectroscopy (TXRF)	A method in which an X-ray spectrometer is used to measure the energy distribution of fluorescence X-rays emitted from a surface irradiated by primary X-rays under the condition of total reflection.
	Ultra-violet photoelectron spectroscopy (UPS)	A method in which an electron spectrometer is used to measure the energy distribution of photoelectrons emitted from a surface irradiated by ultra-violet photons.
	X-ray photoelectron spectroscopy (XPS)	A method in which an electron spectrometer is used to measure the energy distribution of Auger and photoelectrons emitted from a surface irradiated by X-ray photons. It is an extremely surface sensitive non-destructive technique that provides quantitative surface chemical state information for all elements except hydrogen and helium. Guidance on the method can be found in BS ISO 20903.
6. Bulk chemical analysis	Glow discharge mass spectrometry (GDMS)	A method in which a mass spectrometer is used to measure the mass-to-charge quotient and abundance of ions from a glow discharge generated at a surface.
	Glow discharge optical emission spectrometry (GDOES)	A method in which an optical emission spectrometer is used to measure the wavelength and intensity of light emitted from a glow discharge generated at a surface. Guidelines of practices that should be followed in GD-OES analyses are described in BS ISO 14707.

Table 2 **Measurement methods for audit check purposes** (*continued*)

Metric	Method	Guidance
7. Single agglomerate crushing strength		Techniques to measure the crushing strength of a single agglomerate are under development. For example, a relatively large (greater than 63 μm diameter) single agglomerate is placed on a transverse beam and the load versus deformation curve is measured until the agglomerate breaks into pieces. However, no crushing strength tests have been identified for agglomerates with diameters of 63 μm or less.
8. Coating thickness	Atomic force microscopy (AFM)	A technique for imaging surfaces by mechanically scanning their surface contours using a microfabricated probe, in which the deflection of a sharp tip sensing the surface forces, mounted on a soft cantilever, is monitored as the tip is moved across the surface. The specimen should have a small area of substrate exposed with a sharp step between the film surface and the substrate. The thickness is determined by monitoring the displacement of the probe as it passes over the edge of the step.
	Electron microscopy	Guidelines for the calibration of image magnification can be found in BS ISO 16700.
	Confocal microscopy	A confocal microscope system provides measurement of the variation in height of the sample (z-axis) as it is moved across the detection plane using an xy translation stage. The systems use point illumination and a pinhole in an optically conjugate plane in front of the detector to eliminate out-of-focus information. As only one point is illuminated at a time in confocal microscopy, 2D or 3D imaging requires scanning over a regular raster (i.e. a rectangular pattern of parallel scanning lines) in the specimen. The resolution of the measurements are <1 μm in x- and y-axes and <10 nm in the z-axis. Hence microfeatures and topographic variations can be monitored in detail. Sample sizes from a few mm^2 to 100 mm $\times$ 100 mm can be measured.
	Nano indentation testing machine	A nano indentation testing machine can determine film thickness. The specimen should have a small area of substrate exposed with a sharp step between the film surface and the substrate. The thickness is determined by monitoring the displacement of a probe as it passes over the edge of the step.
	Ellipsometry	Ellipsometry analyses the change of polarization of light, which is reflected off a sample, and may be used to assess surface uniformity and roughness.
9. Strength of interface with substrate (for thin film coatings)	Scratch testing	A nano indentation machine can be used to characterize coating to substrate systems and quantify parameters such as friction force. A sharp metal or diamond tip is drawn across the coated surface with an increasing load resulting in failure of the coating at a critical load, which indicates the strength of adhesion of the coating to the substrate.
10. Surface morphology of coatings	Atomic force microscopy (AFM)	A technique for imaging surfaces by mechanically scanning their contours using a microfabricated probe, in which the deflection of a sharp tip sensing the surface forces, mounted on a soft cantilever, is monitored as the tip is moved across the surface.
	Near field scanning optical microscopy (NSOM)	A technique for imaging surfaces in transmission or reflection by mechanically scanning an optical probe much smaller than the wavelength of light over the surface whilst monitoring the transmitted or reflected light. The detector is placed very close to the specimen surface, which allows surface inspection with high spatial resolution.
	Scanning tunnelling microscopy (STM)	A technique for revealing the apparent electron-density-related atomic structure of surfaces, using a needle-like probe near the object under observation; a tunnelling current, which is measured, is generated by altering the potential at the tip of the probe. A 3D representation of the sample surface is generated by rastering the surface of the object and mapping the distance for constant current level at various points.

Table 2 **Measurement methods for audit check purposes** (*continued*)

Metric	Method	Guidance
11. Diameter of carbon nanotubes	Raman spectroscopy	Spectroscopy in which the Raman effect is used to investigate molecular energy levels. The Raman effect involves the scattering of light with a change of frequency characteristic of the scattering substance, representing a change in the vibrational, rotational or electronic energy of the substance. The magnitude of the effect can be used to interpolate the diameter of carbon nanotubes.
	Scanning tunnelling microscopy	The technique can be used to measure the diameter of individual carbon nanotubes.
12. Fibre length	Scanning electron microscopy	Fibre length can be measured by imaging from fibres spun coated onto silicon wafers. Guidelines for the calibration of image magnification can be found in BS ISO 16700.
13. Aspect ratio		The aspect ratio of fibre is the ratio of length to diameter.
14. Wall thickness of carbon nanotubes	Transmission electron microscopy	Wall thickness can be measured by imaging cross sections of fibres.
15. Symmetry		Measurement techniques for symmetry in carbon nanotubes are under development. For example, researchers report using scanning tunnelling microscopy and electron backscatter diffraction, but there is no established, widely applied technique.

8 Possible impacts of contamination on the properties and performance of manufactured nanomaterials and their mitigation

By their nature many nanomaterials are very reactive and consequently their physical and chemical properties can be deleteriously affected by their environment and atmospheric by contamination. The mechanism and rate of deterioration will depend on several variables, including the composition of the nanomaterial, its morphology, the storage time and storage conditions.

Consequently mitigation techniques should be specified considering the tendency of the material to deteriorate. Packing and storage conditions should be negotiated between the supplier and purchaser of the material.

Specification of product storage conditions should consider the tendency of the material to be degraded by moisture or heat and the material's reactivity with other materials being stored in the same area.

9 Packaging, labelling and transport

9.1 Packaging

Supplier and purchaser should negotiate the packing manner and specifications, with consideration of the applicable statutory regulations.

9.2 Labelling

In Great Britain, the two major areas of legislation that impact on the provision of chemical hazards information are the Chemicals (Hazard Information and Packaging for Supply) Regulations 2002, as amended [1], and the Carriage of Dangerous Goods and Use of Transportable Pressure Equipment Regulations 2004, as amended [2]. Similar, but separate, regulations apply in Northern Ireland. The supply regulations deal with the duty of the supplier to the customer and the transport regulations with the duty of both the consignor and the carrier in the carriage of chemicals.

9.3 Transport

In a broader context, exporters of chemicals need to be aware of the various transport regulations, ADR [3] (road), RID [4] (rail), IMDG Code [5] (sea) and ICAO Technical Instructions/IATA [6 and 7] (air), and, when exporting outside the EU, of the presence of local regulations concerning such matters as safety data sheets and local chemical inventories, and the obligations placed on exporters by international agreements such as EC Regulation No. 304/2003 [8], which deals with the export of chemicals to countries outside the EC, and the Chemical Weapons Convention [9].

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BS EN 23923-2:1993, ISO 3923-2:1981, *Metallic powders – Determination of apparent density – Part 2: Scott volumeter method*

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