Standards and their value in general and specifically for nanotechnology

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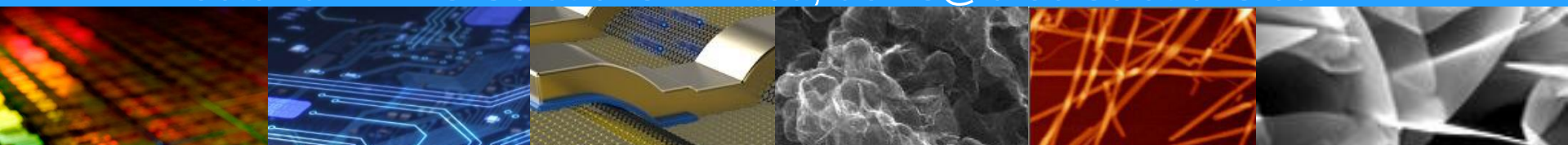


Who are we

NPL National Physical Laboratory

The UK's national standards laboratory world-leading **National Measurement Institute**

~750 staff; State-of-the-art laboratory facilities

Critical importance of **Partnership** with industry and academia

New model - NPL operates as a government company, wholly owned by BEIS.

We provide confidence in measurement with independence integrity and impartiality, and accelerate the application of Science & Technology

What is a standard?

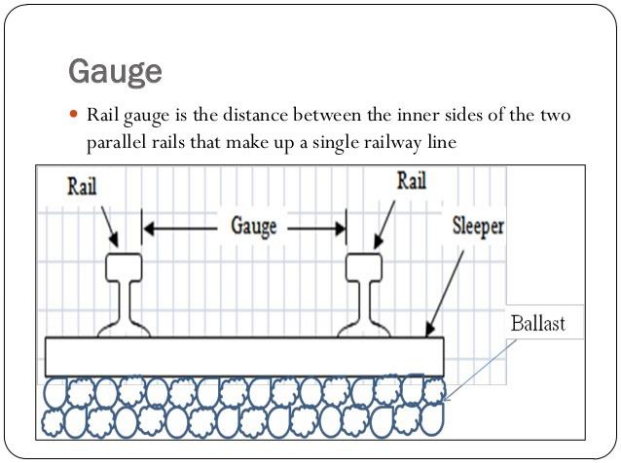
A standard is a …

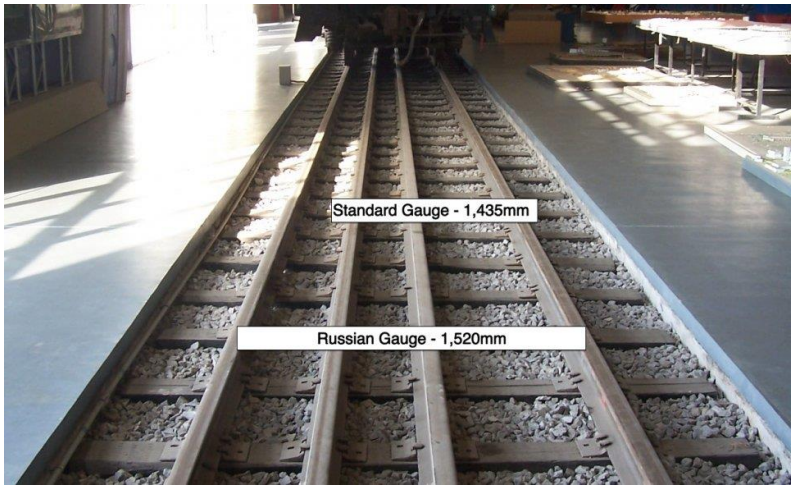
'document, established by consensus and approved by a recognized body, that provides, for common and repeated use, rules, guidelines or characteristics for activities or their results, aimed at the achievement of the optimum degree of order in a given context'

(ISO/IEC Guide 21-1:2005, 3.1)

Example of general use of standards

- Currently, most coal from Mongolian mines is transported to the Chinese border by trucks on paved roads
- Mongolia and China use two different rail gauges and requires wheel changes at the border.
- New railroad will use broad gauge and will cost $3 more per ton of coal than the standard gauge (which is approximately 7.5% of coal price), But save 65% compared with road travel





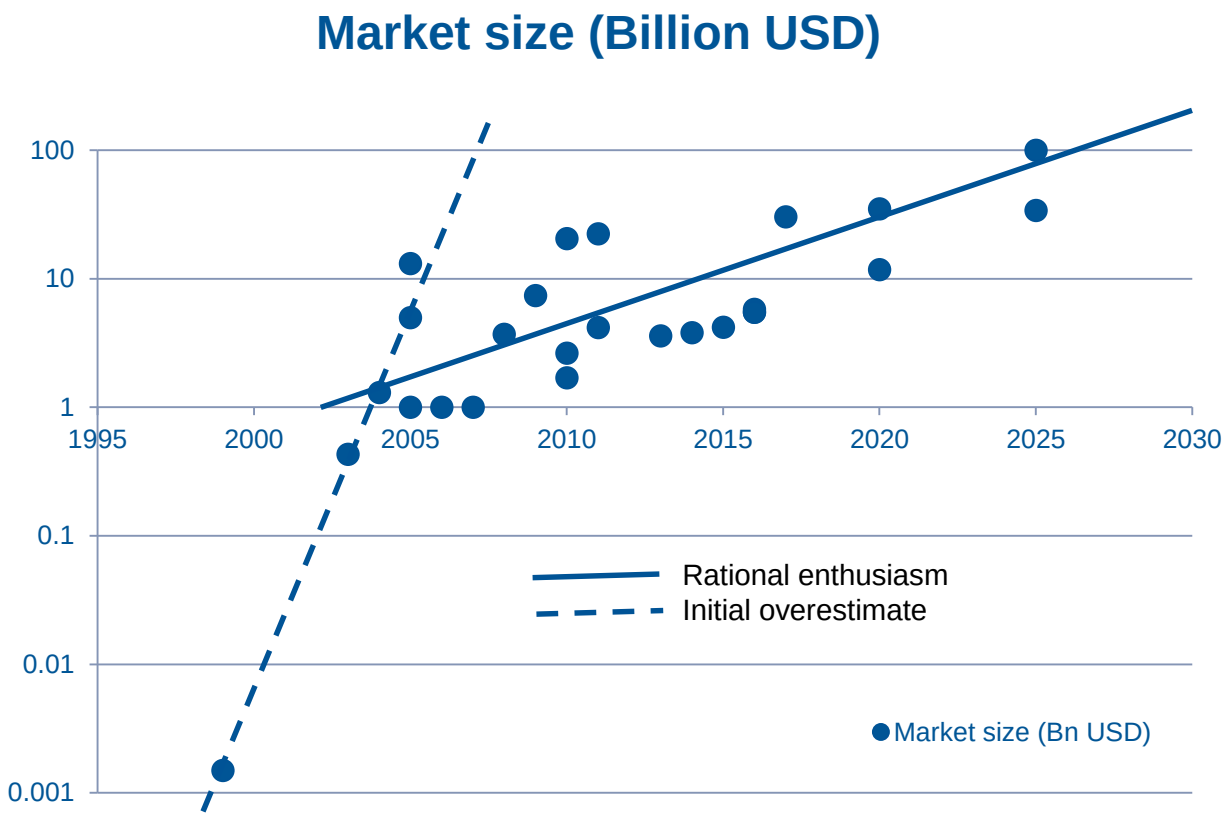
http://www.businessinsider.com/mongolia-extends-trans-border-railway-to-china-russia-2014-10?IR=T

Justification for following standards

- Standards represent the best practice
- Standards are made by consensus of all stakeholders
- Standards allow compatibility and intercomparison of products
- Safety standards for products facilitate consumer acceptance
- Standards form the basis of regulations
- While it may seem costly to comply at first, businesses save a lot of time and money in the long run

Developments in Nanotechnology

- Since 2000s, the development of nanotechnology has led to an enormous investment in nanomaterials production.
- Currently there is an oversupply situation with most nanomaterials and a corresponding downward pressure on prices.



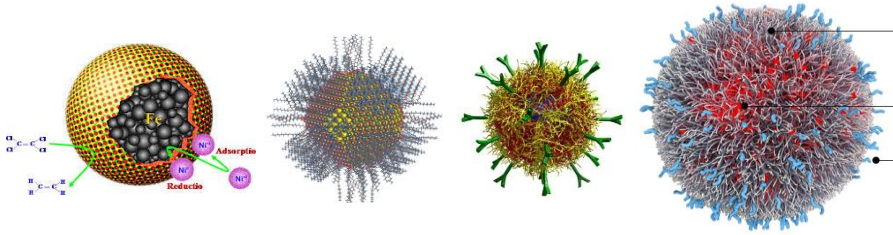
Plot is formed from market research predictions

Industrial Developments

High Volume, low Cost

- Production volume can outstrip application need
- Race to the lowest price
- Leads to poor quality
- Garbage in, Garbage out
- Standards necessary for quality and assurance !

Highly specialised



- Particle properties tailored to application
- Highly specific size, shape, chemistry, surface chemistry
- Need Standards!

Nanotechnology Standardisation

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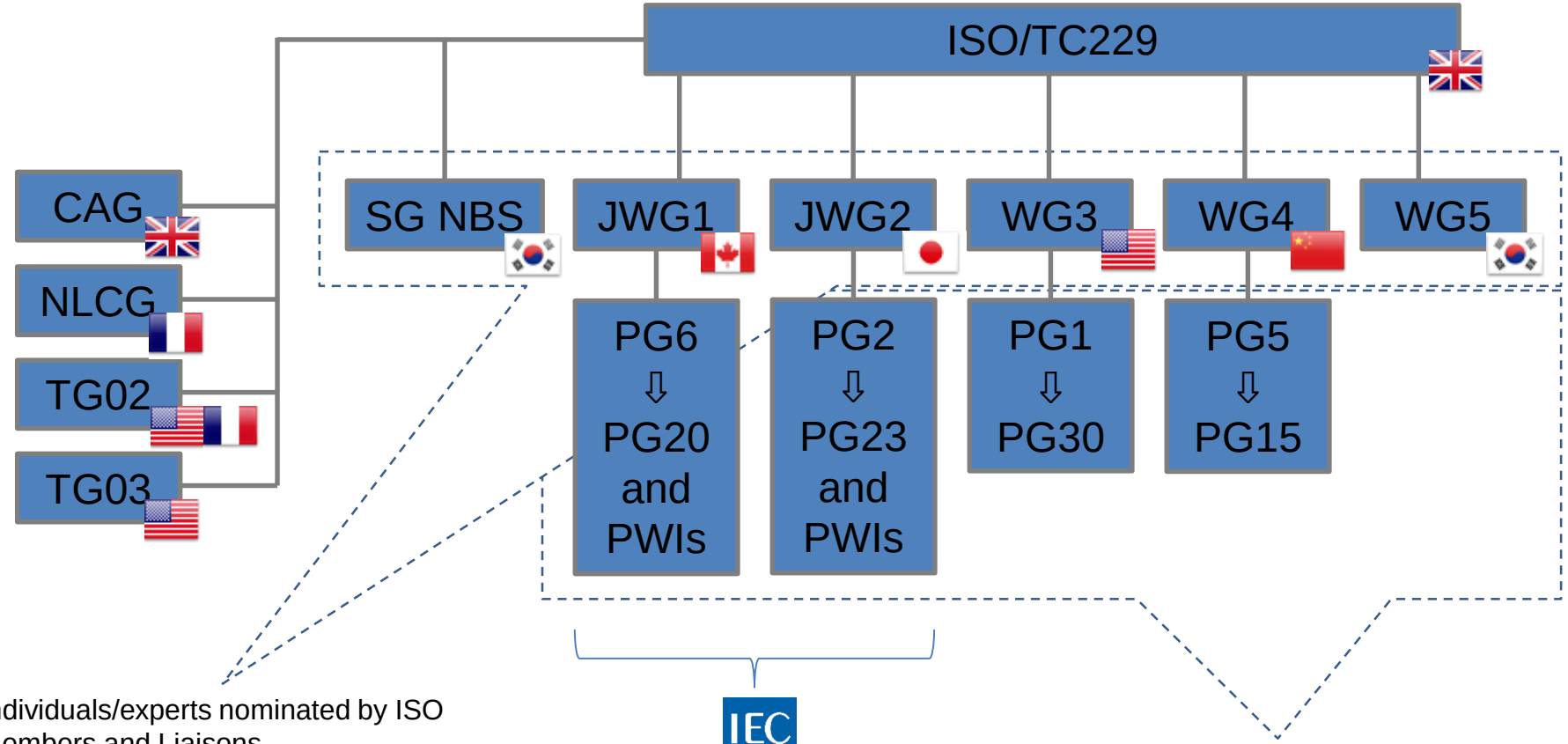
International Standardization Committees

Committee	When Founded	Membership	Developed Standards	Standards[2] under development
ISO/TC 229 Nanotechnologies	2005	37 Participating countries 14 Observing countries	52	42
CEN/TC 352 Nanotechnologies	2005	34 Countries	14	13
IEC TC 113 Nanotechnology for electrotechnical products and systems	2006	15 Participating countries 19 Observer countries	27	37
ASTM International TC E56 Nanotechnology	2005	Over 180 members	18	8

[1] ISO - International Organization for Standardization, CEN - The European Committee for Standardization, IEC - International Electrotechnical Commission

[2] Term "standard" used here includes technical reports, technical specifications and standard test methods.

ISO/TC229 Structure



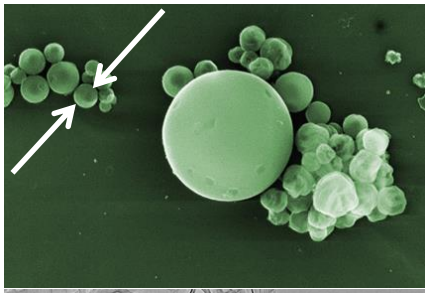
Individuals/experts nominated by ISO Members and Liaisons
Operate by consensus.
Make recommendations to parent body, ISO/TC 229, for Resolution.

Joint working groups
(Mode 5 cooperation).

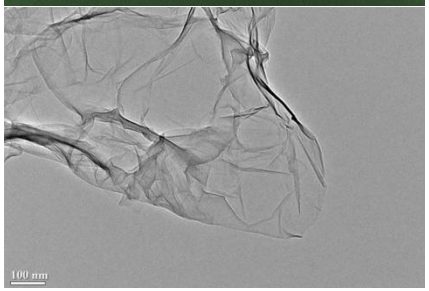
Individuals/experts nominated by ISO Members and Liaisons.
Operate by consensus.
Report to WG on document progress.

Terminology and nomenclature

- Clear description of nanomaterial or nanotechnology innovation is critical to the development of the entire industry.
- Clear terminology promotes clear communication, research, investment and regulation



nano-object
material with one, two or three external dimensions in the nanoscale
ISO/TS 80004-3:2010(en), 2.2



graphene
single layer of carbon atoms with each atom bound to three neighbours in a honeycomb structure
ISO/TS 80004-3:2010, 2.11, modified in 2017

European Commission

Nanomaterial' means a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50 % or more of the particles in the number size distribution, one or more external dimensions is in the size range 1 nm-100 nm.
EC 2011/696/EU

JWG1
Terminology and nomenclature

Scope:

Define and develop unambiguous and uniform terminology and nomenclature in the field of nanotechnologies to facilitate communication and to promote common understanding.

ISO Number	Title	Stage
TR 18401	Nanotechnology – Plain language guide – Explanation of core terms with examples	Under Publication
TS 20477	Standard terms and their definition for cellulose nanomaterial	Under Publication
TS 80004-11	Nanotechnologies - Vocabulary - Part 11: Nanolayer, nanocoating, nanofilm and related terms	Under Publication
TS 80004-4 (Rev)	Nanotechnologies — Vocabulary — Part 4: Nanostructured materials	Draft in Preparation
TS 80004-13	Nanotechnologies -- Vocabulary -- Part 13: Graphene and other two dimensional materials	Under Publication

6 documents in current work programme

Terminology freely available at ISO OBP

Characterisation

For any technical description of nanomaterial it is critical to be able to

- Detect
- Identify
- Characterise

nanomaterials in of various types, sizes, shapes and chemical functionality





Nano-object Phys-Chem characterisation needs

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Appearance: What does it look like?	NPL Equipment
Particle Size/Size Distribution	DLS, DCS, SEM, TEM, XPS, XRD
Aggregation/ Agglomeration State	SEM, TEM
Shape	AFM, SPM, SICM,EM
Surface Area	BET, NMR relaxation
What is it made of?	
Composition	ICP-MS, EDX, Uv-Vis, Flurometer, Raman,
Surface Chemistry	XPS, SIMS, Auger,
What else?	
Surface Charge	Zeta Potential
Photo Oxidation	Assays
Dispersibility, Solubility, Stability,	Turbidity
bio characterisation, toxicology	Super-res micro.,
Mass	MS, QCM

JWG2
Measurement and characterization

Scope:

The development of standards for measurement, characterization and test methods for nanotechnologies, taking into consideration needs for metrology and reference materials.

ISO Number	Title	Stage
TR 19733	Matrix of characterization and measurement methods for graphene	Draft due for ballot
ISO/NP 19749	Determination of size and size distribution of nano-objects by scanning electron microscopy	Draft in Preparation
TS 19805	On-line/Off-line techniques for characterizing size distribution of airborne nanoparticle populations	Draft due for ballot
TR 20489	Separation and size fractionation for the characterisation of metal-based nanoparticles in water samples	Draft due for ballot
ISO/NP TS 21361	Nanotechnologies -- Quantification of airborne nanoscale carbon black and amorphous silica in a manufacturing environment	NWIP ballot

20 documents in current work programme

Health and Safety

- One of the key questions asked of nanotechnology is “Is it safe?”. The effects of nanomaterials have been studied by academics, industry and regulators for over two decades.
- Sharing the best practice and minimum safety requirements for
  - Developing
  - Handling
  - Trading
  - Storing
  - Using
  - Disposing of

nanomaterials is critical for acceptable and sustainable development

WG3

Health, safety and environment

Scope:

The development of science-based standards in the areas of health, safety, and environmental aspects of nanotechnologies.

ISO Number	Title	Stage
TR12885:2008 (rev)	Nanotechnologies – Health and safety practices in occupational settings	Draft due for ballot
TS 18827	Nanotechnologies -- Comparing the toxic mechanism of synthesized zinc oxide nanomaterials by physicochemical characterization and reactive oxygen species properties	Under Publication
ISO 19007	Nanotechnologies – In vitro MTS assay for measuring the cytotoxic effect of nanoparticles	FDIS ballot due
TS 22082	Nanotechnologies --- In vivo toxicity assessment of nanomaterials using dechorionated zebrafish embryo	NWIP ballot
TR 22019	Considerations in performing toxicokinetic studies of nanomaterials	Draft in preparation.
TR 21386	Nanotechnologies -- Considerations for the measurement of nano-objects, and their aggregates and agglomerates (NOAA) in the environment	Draft in Preparation

16 documents in Programme of work

Materials Specifications

- Nanomaterials (NMs) supply is becoming mature and requires clear materials specifications based on materials characterisation evidence and using clear terminology.
- While some identify H&S as one of the biggest threats to NMs supply, it is the inaccurate and often exaggerated properties specified by suppliers that kill the market.
- Agreement between all stakeholders
    - Suppliers
    - Buyers
    - Traders
    - Academics (as both suppliers and buyers)
    - Regulators

is critical for global acceptance of NM specifications



WG4
Material specifications

Scope:

To specify relevant compositions and properties and characteristics of manufactured nanomaterials. Excluded are areas of duplication in other technical committees from ISO and IEC. NOTE These documents will aim to facilitate communications between buyers, sellers and regulators of raw and intermediate materials.

ISO Number	Title	Stage
TS 17200 (Rev)	Nanotechnology -- Nanoparticles in powder form -- Characteristics and measurements	Draft in Preparation
TS 19808	Specification for carbon nanotube dispersions	Draft in Preparation
TS 20660	Nanotechnologies -- Materials specification -- Antibacterial silver nanoparticles	Draft in Preparation
TS 21236	Nanotechnologies - Nanoclays: characteristics and measurements	Draft in Preparation

8 documents on Programme of work

How to get involved

BSI NTI/1 Committee - Nanotechnologies

- Join the UK nanotechnology standardisation committee
- ISO, IEC, CEN
- Free!
- Review standards, attend meetings, receive free standards
- Contact: charles.clifford@npl.co.uk or alex.price@bsigroup.com

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Standards under development…….

Example 1
Graphene Standardisation

Charles Clifford and Andy Pollard

Need

- 100+ companies sell “graphene”
- Is it really graphene?
- **Need standardised measurement and characterisation protocol**

- **Overall protocol for specific series of complimentary techniques to determine the different material properties**
- **Many types of characteristics are required** for these materials

- Need most time effective and information effective method to analyse the samples

Nanotechnologies - Structural Characterisation of Graphene

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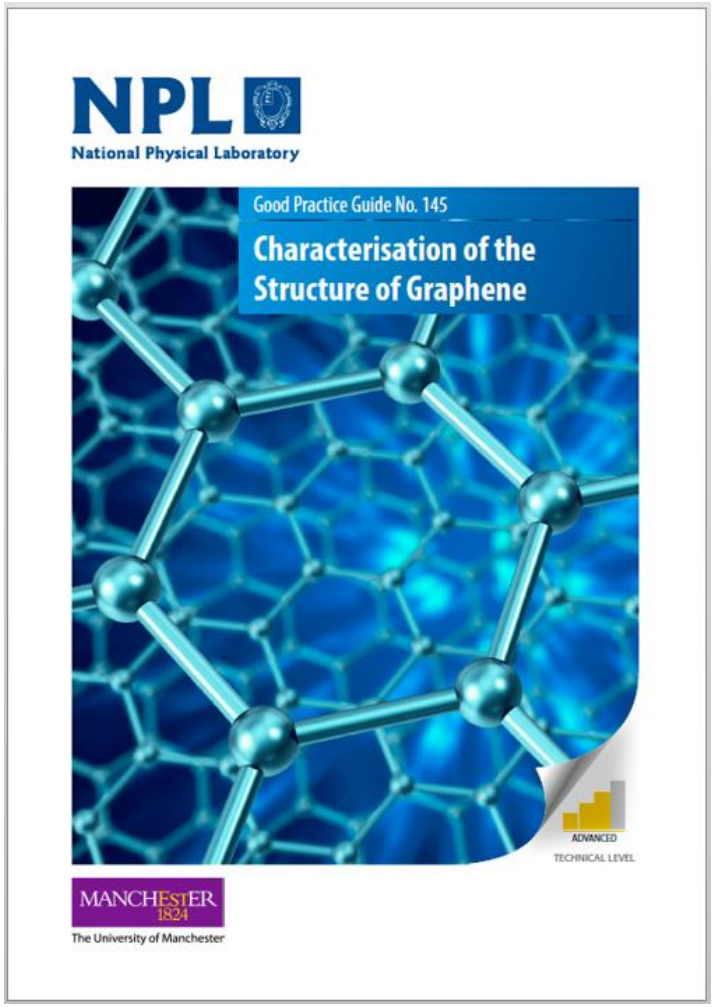
Proposed Scope:

This technical specification specifies methods for characterising the structural properties of graphene using a range of measurement techniques. The properties covered are the number of layers (thickness), lateral flake size, lateral domain size, the level of disorder, layer stacking and surface area. Measurement protocols for both samples consisting of layers of graphene on a substrate and graphene in dispersion/powder form are detailed. The sample preparation required to enable measurements using different techniques is included, as well basic data analysis.

NPL Good Practice Guide



- Joint NPL and NGI (Manchester)
- Technique, issues, protocol, data analysis
- Optical, Raman, SEM, TEM, AFM,
- Publication due 2017
- Will be used as basis for TS and protocols for ILS
- Free to download

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Example 2
Interlaboratory Studies
Pre-standardisation

Measurement of number concentration of nanoparticles

Caterina Minelli, Alex Shard, Charles Clifford

Measurement of number concentration of nanoparticles

NPL National Physical Laboratory

Need:.

- Growing use of nanoparticles
- significant concerns about reproducibility, measurement and regulation.

COMMISSION RECOMMENDATION

of 18 October 2011

on the definition of nanomaterial

'Nanomaterial' means a natural, incidental or manufactured material containing particles…where, for **50 %** or more of the particles in the **number size distribution**, one or more external dimensions is in the size range 1 nm-100 nm.

AIMS

(1) Comparative evaluation of the techniques and methods for the number concentration measurement of colloidal nanoparticles.

(2) Dissemination of best practice for accurate sample preparation and measurement.

FUTURE

Reference materials

Standards

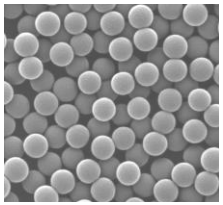
Participants welcome email: caterina.minelli@npl.co.uk

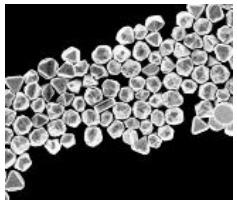
Relevant techniques include:

Small angle X-ray scattering (SAXS),

Single-particle - Inductively Coupled Plasma Mass Spectroscopy (sp-ICP-MS),

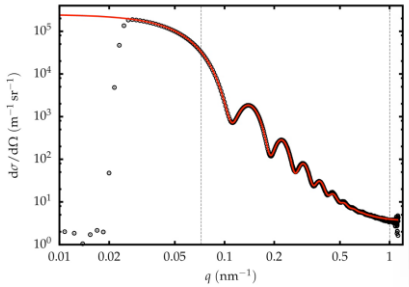
Particle Tracking Analysis (PTA), Tunable Resistive Pulse Sensing (TRPS), Condensation Particle Counter (CPC), Centrifugal Liquid Sedimentation (CLS), Dynamic Light Scattering (DLS) and UV-Vis spectroscopy





10 to 600 nm
Au, silica, polystyrene

Accurate methods:



SAXS

SP-ICPMS

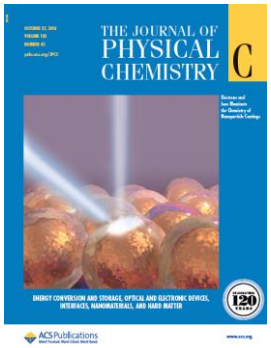
VAMAS TWA34

ISO

VAMAS Interlaboratory Studies

National Physical Laboratory

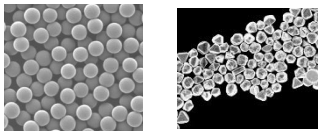
Nanoparticle surface chemistry:



VAMAS TWA2

ISO TC201

Number Concentration:

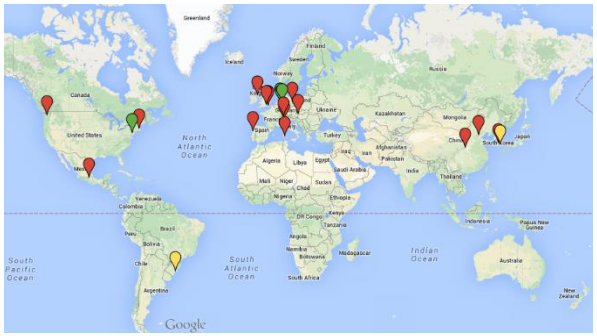


10 to 600 nm
Au, silica, polystyrene

VAMAS TWA34

ISO

Participants:



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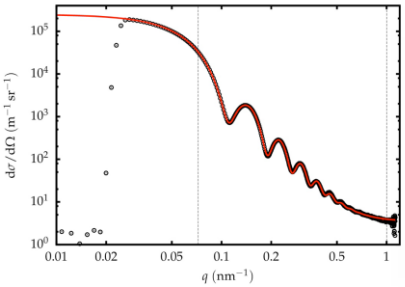

SAXS

SP-ICPMS

Findings:

Current practice provides shell thicknesses >100% in error.
Major error sources identified and methods to reduce precision to ~10% published.

Aim of the study:

Understand current practice and sources of measurement uncertainty.
Calibration methods for lab-based instruments: DLS, CLS, PTA, TRPS, etc…

Summary

- Standards allow comparability and intercomparison
- NPL develops standards of industrial importance
- Standards in Terminology, specific measurement methods, H&S, material specification
- Validate methods using interlaboratory studies where appropriate
- Valid characterisation of 2D materials and nano-objects essential

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