

International Standardization for Nanotechnologies

Dr Peter Hatto,
Director of Research, IonBond Ltd
Chairman ISO TC 229 and UK NTI/1 Nanotechnologies Standardization committees

Michigan State University

7th February 2007

East Lansing

Michigan, USA

Overview

- *International standards – what they are, what they do and how they are developed.*
- *International standardization for nanotechnologies – what needs doing, whose responsibility is it and when might it be done?*
- *International collaboration on standardization and regulation for nanotechnologies*

Standards

Two types:

- **Metrological standards: length, mass, time, quantity of matter – primary and secondary standards**
- **Written standards**

Written Standards provide agreed ways of:

- **Naming, describing and specifying things**
- **Measuring and testing things**
- **Managing things e.g. quality and environmental management: ISO 9001 and ISO 14000**
- **Reporting things as in e.g. proposed ISO 26000 (Social Responsibility)**

Can be NORMATIVE OR INFORMATIVE

Standards are voluntary unless called in a contract or regulation.

Standards provide a means of validated quantification

Standards

Standards are:

- **Ubiquitous** – shoe sizes, nuts and bolts, petrol grades, warning signs, pipes and fittings, fire extinguishers, gas cylinders, electrical sockets and plugs, steel specifications,.....
- **Virtually invisible** to “the man in the street” – there are over 15,000 International Standards, many with multiple parts;
- **But are absolutely critical** to our modern way of life – CDs/DVDs, internet protocols, credit cards, pin numbers, quality and environmental management,.....

and standardization

Standards can be:

- **FORMAL** – developed by independent experts working under the auspices of a National, Regional or International standards body
 - **AFNOR, ANSI, BSI, DIN, JIS,**
 - **CEN, CENELEC,.....**
 - **ISO, IEC & ITU**
- **INFORMAL** – developed by a SDO
 - **ASTM, IEEE, SAE, SEMI, VDI...(>600 SDOs IN US)**
- **PRIVATE** – developed by company or trade association

FORMAL standards are:

- **PROPOSED, DEVELOPED AND APPROVED** by the members of the standards body
- **Based on CONSENSUS**

Functions of standards

Standards can perform any of the following four functions:

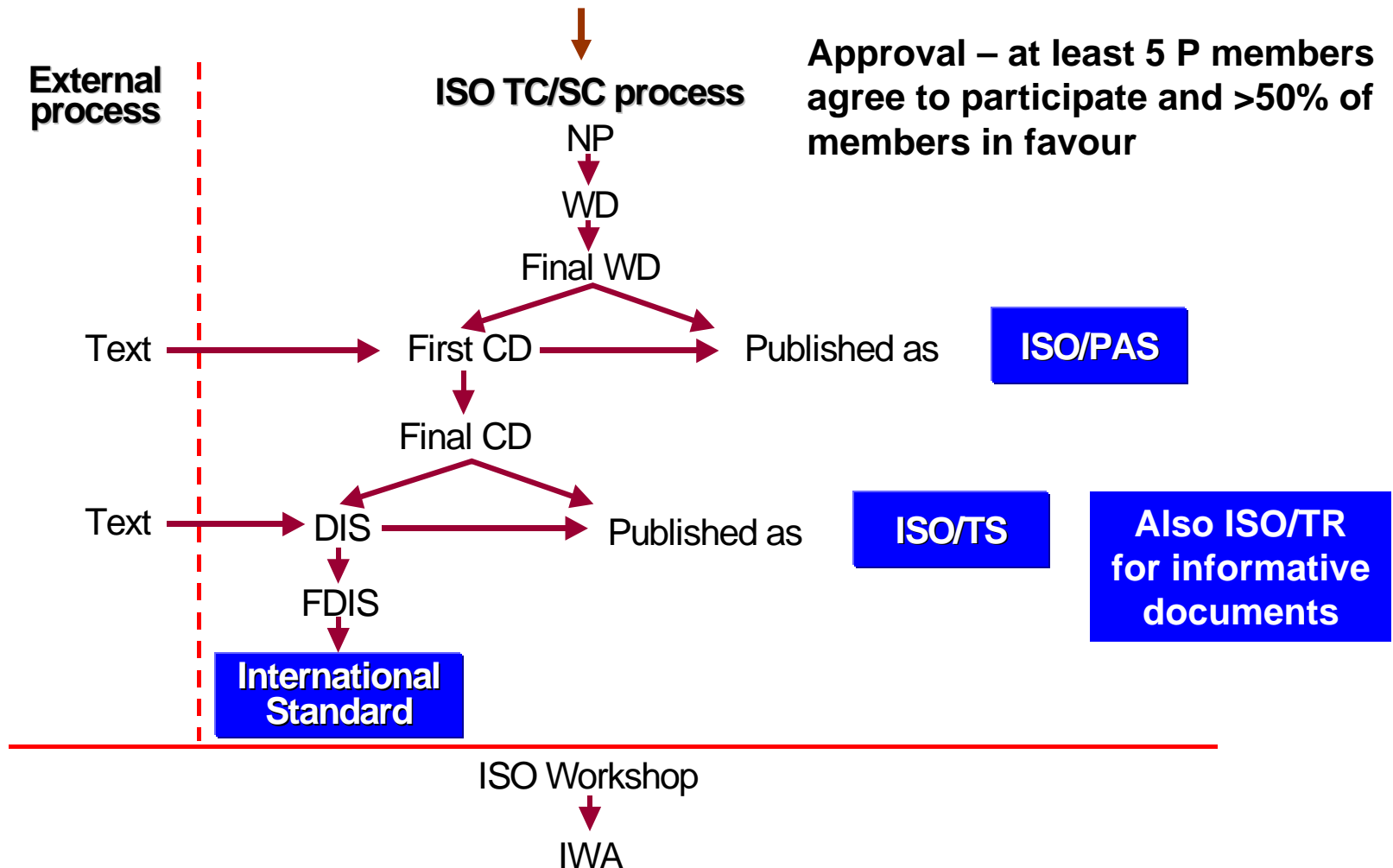
- **Interoperability/Compatibility**
 - as with e.g. nuts and bolts, railway gauges, electrical plugs and outlets, and interoperability standards for computers and telecommunications systems
- **Quality**
 - Fitness for purpose or safety
- **Variety reduction/optimization (based on best practice)**
 - E.g. shoe sizes, suit sizes – leading to mass production and price reduction
- **Information/Measurement**
 - Test and measurement methods for describing, quantifying and evaluating product attributes such as material, processes and functions

DIN has reported that in Europe standardization adds approximately 1% to the value of gross domestic product and that the added value generated by standardisation is at least as important as the value generated by patents!

Development of International Standards

Process accommodates special needs

NWIP from member organisation



Why is nanotechnology important?

US Interagency Working Group on Nano Science, Engineering and Technology (IWGN) workshop on Nanotechnology Research Directions (Sept. '99):

“nanotechnology will be a strategic branch of science and engineering for the 21st century, one that will fundamentally restructure the technologies currently used for manufacturing, medicine, defence, energy production, environmental management, transportation, communication, computation and education.”

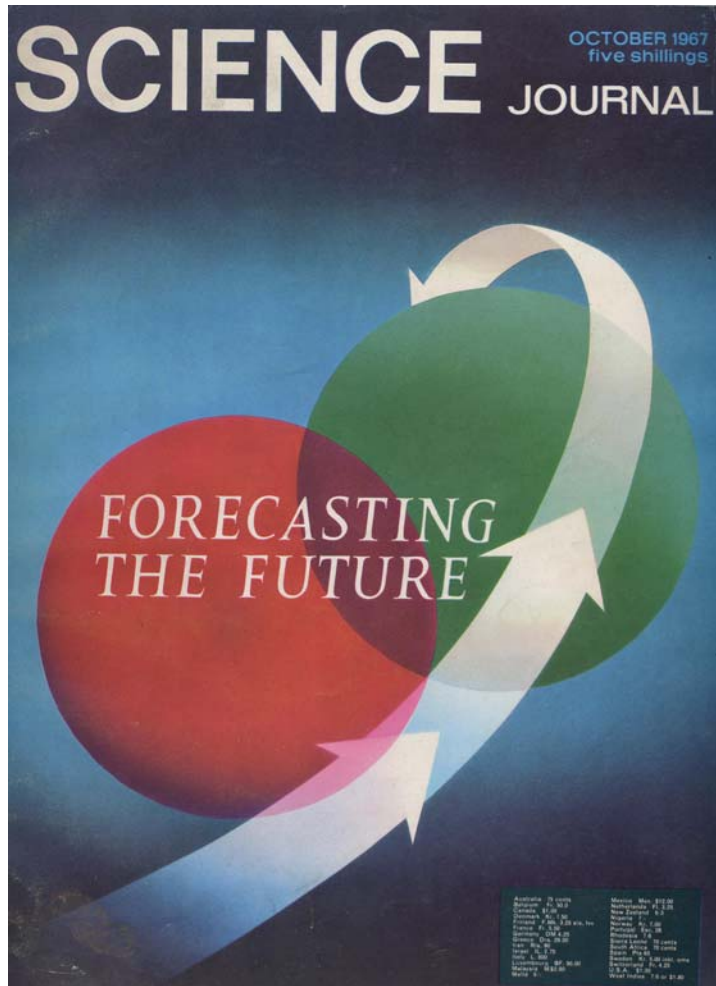
US NSF report on “SOCIETAL IMPLICATIONS OF NANOSCIENCE AND NANOTECHNOLOGY” March 2001:

“the impact of nanotechnology in the 21st century is likely to be at least as significant for health, wealth and security as the combined influences of antibiotics, integrated circuits and polymers.”

Projected world-wide market for n-t enabled products will be >\$1 trillion by 2015

“It is estimated that Nanotechnology is presently at a level of development similar to that of computer/information technology in the 1950s” (Nanostructure Science and Technology: A Worldwide Study, WTEC Panel report, 1999)

A word of Caution!



Transport forecasts:

By 2000:

- *Fly from London to Sydney in 50 minutes*
- *800km/hr trains in evacuated tubes*
- *Fully automatic “driving”*
- *Continuous integrated transport systems*

But the difference is:

- *there is a world-wide focus on nanotechnology, with >>\$5BN annual investment in R&D*
- *Timescale is significantly shorter!*

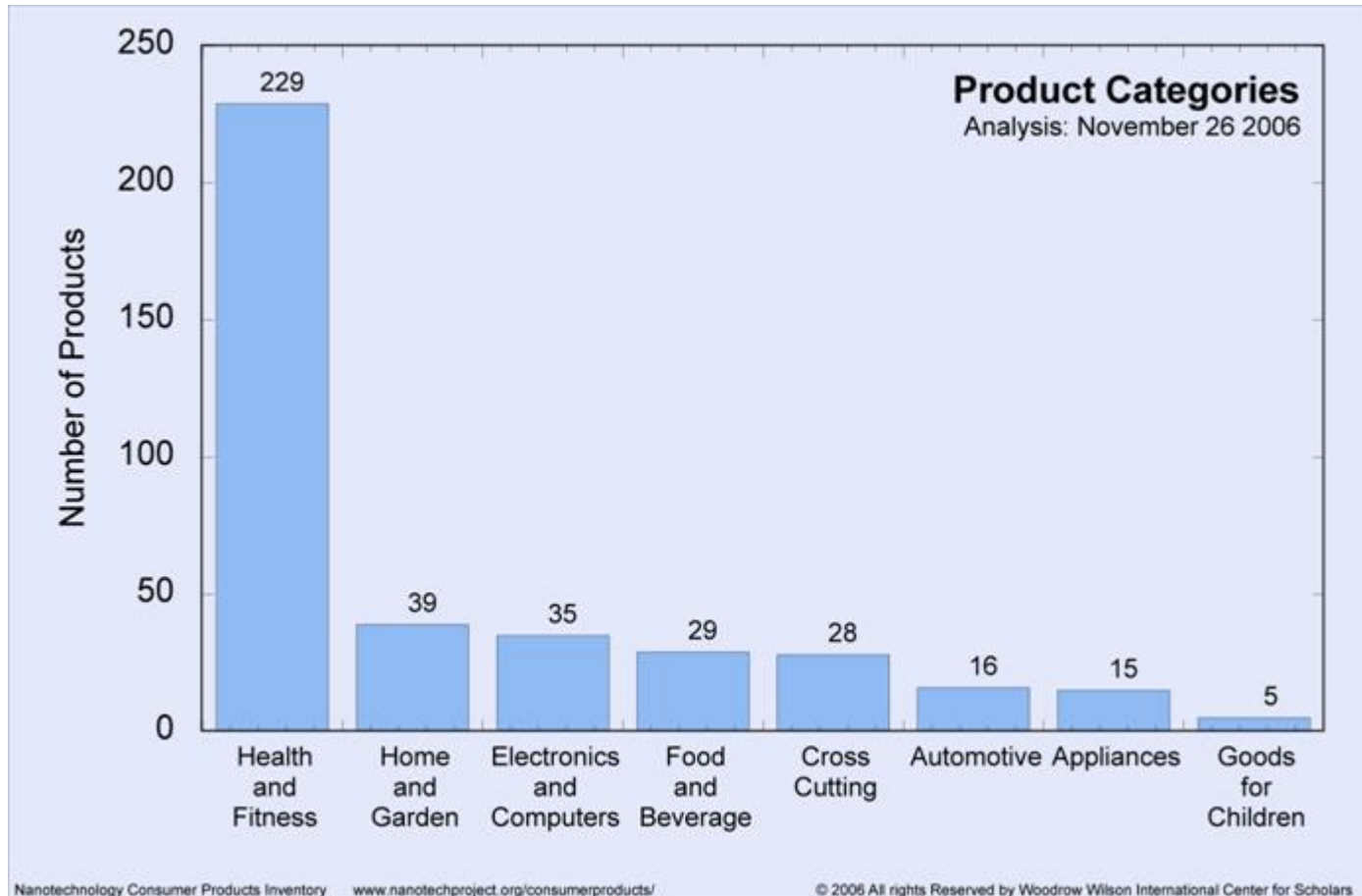
Needs for standardization

1. To support commercialisation and market development – Anticipatory standards
2. Provide a basis for procurement – technical/quality/environmental management
3. To support appropriate legislation/regulation – consumer and environmental protection

Currently there are:

- *No internationally agreed terminology/definitions for nanotechnology(ies).*
- *No internationally agreed protocols for toxicity testing of nanoparticles.*
- *No standardized protocols for evaluating environmental impact of nanoparticles.*
- *Existing “methods of test” may not be suitable for nanoscale devices and nanoscale dimensions.*
- *Measurement techniques and instruments need to be developed and/or standardized.*
- *New certified references materials are needed for validation of test instruments at the nanoscale.*
- *Multifunction nanotechnology systems and devices will need new standards.*
- *Some existing standards may be applicable e.g. for chemical analysis and imaging (ISO TCs 201 and 202) and particle detection/sizing (ISO TC 24)*

Nanotech consumer products today (356 to 26.11.06) – Woodrow Wilson Inventory



<http://www.nanotechproject.org/44>

A brief history of standardization for nanotechnologies

- 12/03: - China establishes United Working Group for Nanomaterials standardization
- 03/04: - UK Proposal to establish CEN BT WG to develop strategy for European standardization in nanotech approved (- CEN/BTWG 166)
- 05/04: UK establishes NTI/1 national committee
- 08/04: ANSI forms Nanotech Standards Panel in response to a request from OSTP
- 10/04: UK starts work on PAS 71 - vocabulary for nanoparticles
- 11/04: Japan establishes study group for nanotech. standardization
- 12/04: China publishes 7 national nanotech standards
- 01/05: UK submits proposal for an ISO committee
- 04/05: - China implements published nanotech standards
 - ASTM International approves establishment of E56 committee
 - ISO ballot on UK proposal approved – 30 votes to nil
- 06/05: - ISO establishes TC 229 – UK secretariat & chair.
 - UK publishes PAS 71, vocabulary – nanoparticles (free on www)
 - CEN/BT/WG 166 delivers European strategy to CEN/BT
- 11/05: Inaugural meeting of ISO TC 229 in London.
- 11/05: CEN establishes CEN/TC 352 – Nanotechnologies – UK Chair & Secretariat
- 03/06: IEC receives proposal for new TC for Nanotechnologies
- 04/06: First meeting of CEN/TC 352 (agreed to collaborate closely with ISO/TC 229)
- 05/06: IEC agrees to establish TC 113 in the field of nanotechnologies – issue of coordination with ISO/TC 229
- 06/06: 2nd meeting of ISO/TC 229, Tokyo, Japan
- 12/06: 3rd meeting of ISO/TC 229, Seoul, Korea
- 01/07: 6 NWIP submitted to TC 229.

To date national committees established in China, France, Germany, Iran, Italy, Japan, Korea (committee for CNTs), Russia, Singapore, Thailand, UK, US.....,

ISO/TC 229 - Nanotechnologies

- 28 “P” members
- 9 “O” members
- Liaisons with 15 ISO TC’s, CEN/TC 352, OECD (WPMN), EC JRC (Ispra – Health and Consumer Protection - and IRMM), VAMAS – liaison with ANF currently out for ballot.

ISO TC 229 adopted Scope:

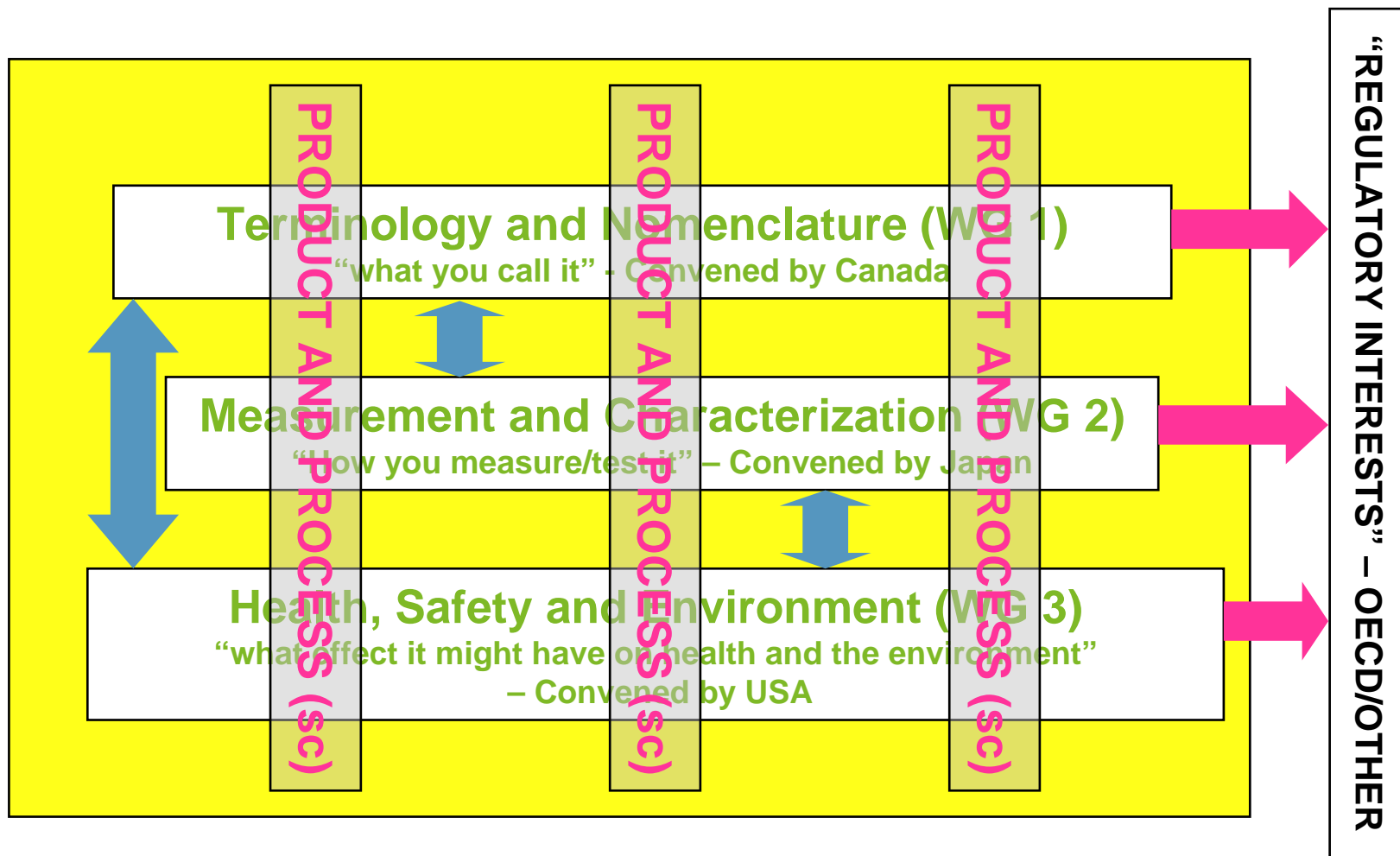
“Standardization in the field of nanotechnologies that includes either or both of the following:

- *Understanding and control of matter and processes at the nanoscale, typically, but not exclusively, below 100 nanometres in one or more dimensions where the onset of size-dependent phenomena usually enables novel applications;*
- *Utilizing the properties of nanoscale materials that differ from the properties of individual atoms, molecules, and bulk matter, to create improved materials, devices, and systems that exploit these new properties*

Specific tasks include developing standards for: *terminology and nomenclature; metrology and instrumentation, including specifications for reference materials; test methodologies; modelling and simulation; and science-based health, safety, and environmental practices.*”

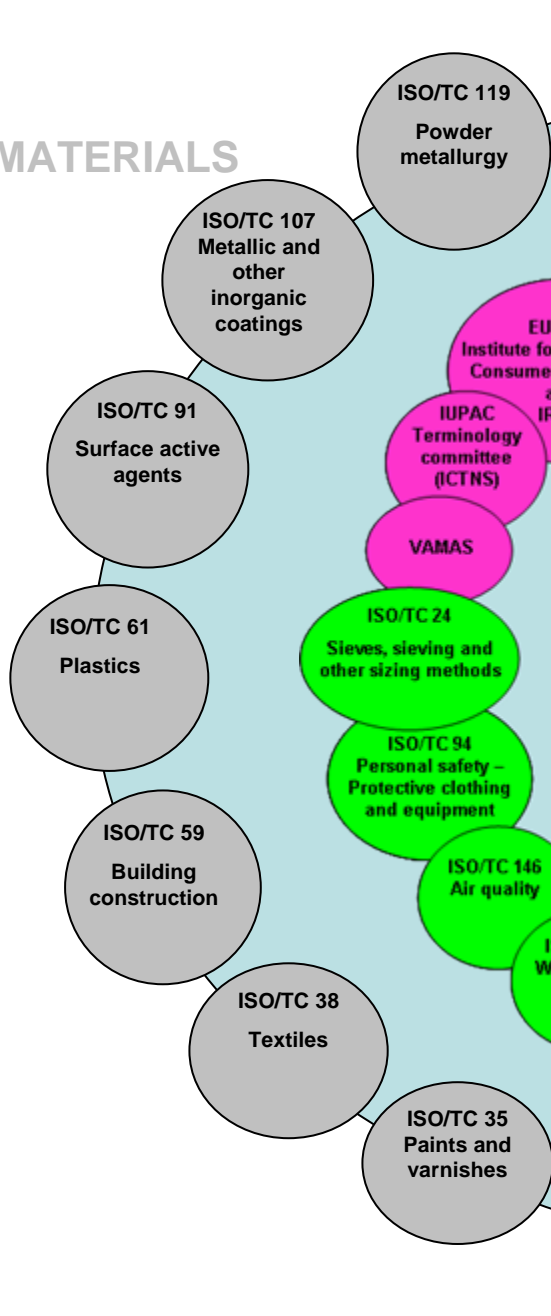
Note: Scope is further defined by the TC’s “Strategy Statement”

TC 229 – Structure/working areas

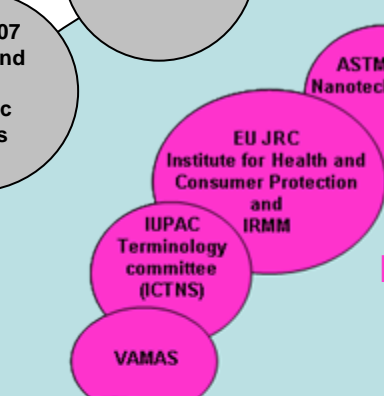


POSSIBLE LIAISONS

MATERIALS



EXTERNAL LIAISONS

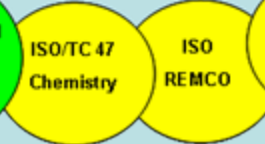


NANO-PARTICLES

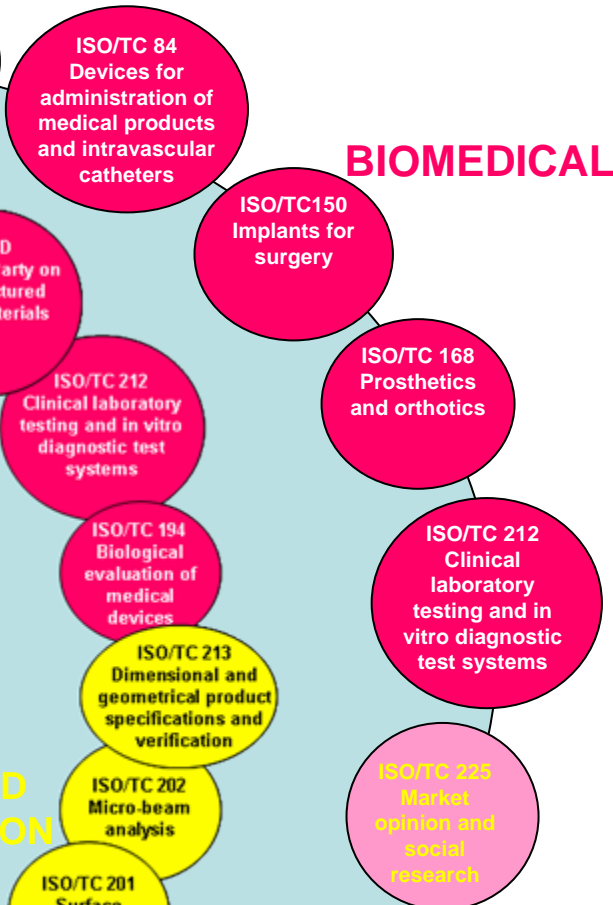
METROLOGY AND CHARACTERIZATION

ISO TC 229

RISK/HS&E

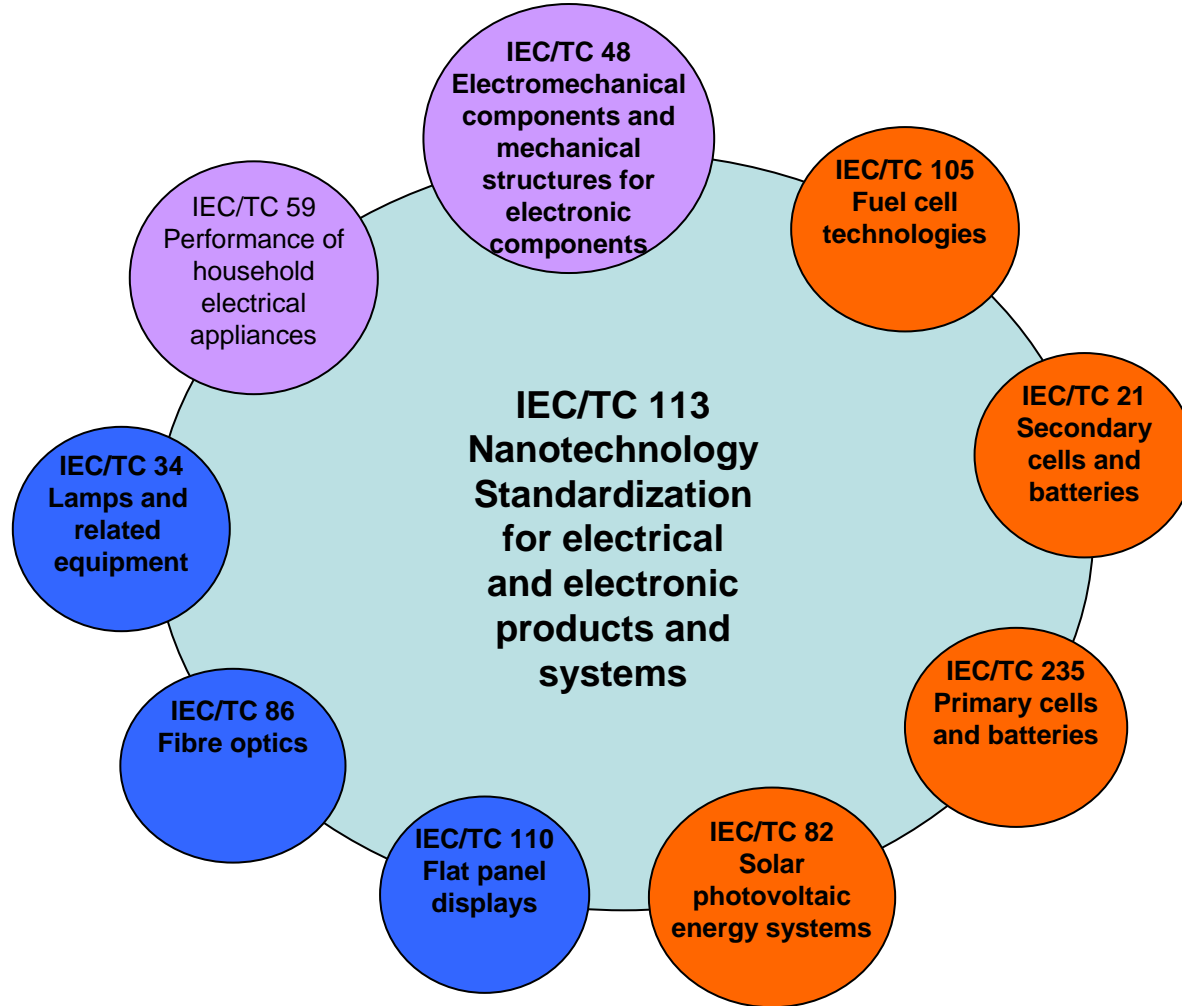


BIOMEDICAL



ENERGY

POTENTIAL LIAISONS WITHIN IEC



Current nanotechnology standards

- Only National standards + ASTM terminology to date:
 - China – National standards published Dec 2004, implemented Apr 2005:
 - **GB/T19619-2004 Terminology for nanomaterials**
 - Sizing:
 - **GB/T13221-2004 Nanometer powder - Determination of particle size distribution - Small angle X-ray scattering method (ISO/TS13762)**
 - **GB/T19587-2004 Determination of the specific surface area of solids by gas absorption using the BET method (ISO 9277:1999)**
 - **GB/T19627-2005 Particle size analysis - Photon correlation spectroscopy (ISO 13321:1996)**
 - Nano-material specifications:
 - **GB/T19588-2004 Nano-nickel powder**
 - **GB/T19589-2004 Nano-zinc oxide**
 - **GB/T19590-2004 Nano-calcium carbonate**
 - **GB/T19591-2004 Nano-titanium dioxide**
 - UK – PAS 71: 2005 – Vocabulary – Nanoparticles
 - ASTM E 2456-06 Terminology for Nanotechnology
- Several standards (International, regional and national) that also apply to the nanoscale

Current TC 229 work items

- Terminology and definitions for nanoparticles – to be published as a TS
- "Current safe practices in occupational settings relevant to nanotechnologies" – to be published as a TR
- NWI: Endotoxin test on nanomaterial samples for in vitro test systems

New Work Item Proposals:

- NWIP: the Use of Transmission Electron Microscopy in the Characterization of Single-walled Carbon Nanotubes
- NWIP: the use of Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Analysis (EDXA) of Single Wall Carbon Nanotubes (SWCNTs)
- NWIP: Technical Specification for the Use of UV-Vis-NIR absorption spectroscopy in the Characterization of Single-Walled Carbon Nanotubes (SWCNTs)
- Technical Specification for the use of NIR-Photoluminescence (NIR-PL) Spectroscopy in the Characterization of Single-Walled Carbon Nanotubes (SWCNTs)
- NWIP: generation of silver nanoparticles for inhalation toxicity testing
- NWIP: monitoring of silver nanoparticles in inhalation exposure chambers for inhalation toxicity testing

Standardization needs

ISO/TC 229 Survey of standardization needs:

- A total of 233 standardization needs were identified, including 21 in the area of materials specifications (new working group needed?) representing 77 materials and 8 additional classes of materials.
- Eliminating topics that <5 members identified as high priority leaves 111 topics including 5 materials specifications representing 40 separate materials and four “classes” of materials (2 each of “compound particles” and “functionalized particles”).
- Of the 111 topics that 5 or more members consider of high priority:
 - *2 are relevant to WG1*
 - *55 are relevant to WG2*
 - *31 are relevant to WG3*
 - *5 are relevant to a new WG for materials specifications*
 - *18 are relevant to other TCs (TC 24, TC 201, TC 202 and TC 213).*
- Of these 111, 28 have been identified by more members as being required on a 3-8 year timescale than on a 1-3 year timescale:
 - *12 are relevant to WG2*
 - *15 are relevant to WG3*
 - *1 is relevant to a new WG for materials specifications.*
 - *Majority of topics require either PNR or CNR*

Nanoparticulates – Generic standards

| |
|--|
| nomenclature |
| sampling methods for characterization |
| characterization protocols for |
| size |
| size distribution |
| shape |
| degree of agglomeration /aggregation |
| chemical purity |
| specific area |
| chemical functionality |
| chemical structure |
| core composition |
| porosity |
| physical properties - electrical |
| strength of agglomerate/aggregates (3-8 years) |
| dispersability (3-8 years) |
| degree of functionalization (3-8 years) |

Nanoparticulates and other nanoscale materials - Health Safety and Environment Standards – 1 to 3 years

Standard Methods for Toxicological Screening of Nanomaterials

Standard Methods for Determining Relative Toxicity/Hazard Potential of Nanomaterials

Standard Guide for Controlling Occupational Exposures to Nanomaterials

Standard Template for Material Safety Data Sheet (MSDS) for Products Containing Nanomaterials

Nanomaterial Product Information For Use In Determining Health & Safety Precautions

Standard Method for Selection of Personal Protective Equipment (PPE) for Use With Nanomaterials

Standard method for determining physical hazards of nanomaterials (i.e. explosive, flammability, water reactivity, etc.)

Standard Method to Establish Occupational Exposure Limits for Nanomaterials

Standard Methods to Assess Exposure to Nanomaterials During Consumer Product Use

Standard methods for determining nanoparticle concentrations in air and water

Nanoparticulates and other nanoscale materials - Health Safety and Environment Standards – 3 to 8 years

Standard Methods for Measuring Personal Exposure to Nanomaterials in Occupational Settings

Standard Method for Performing Risk Assessments on Use of Nanomaterials

Product Safety Standards for Consumer Products Containing Nanomaterials

Standard Methods to Determine Environmental Toxicity of Nanomaterials

Standard Method to Assess Product Degradation and Release of Nanomaterials from Consumer Products

Standard Method to Develop Nanomaterial Product Labelling

Standard Method to Assess Emissions from Handling, or Machining of Nanomaterial Containing Products

Standard Method for Reporting Toxicity of Nanomaterials in Consumer Products

Standard Methods to Determine Exposure to Nanomaterials in Food

Methodology to Determine effectiveness of Filtration Media against Nanomaterials

Standard Method of Life Cycle Analysis for Consumer Products Containing Nanomaterials

Standard test methods for measurement of nanomaterials in manufacturing discharges

Nanotubes – generic standards

| |
|--|
| nomenclature |
| sampling methods for characterization |
| characterization protocols for: |
| diameter distribution |
| length distribution |
| batch quality |
| chemical structure |
| determination of nature and concentration of contaminants |
| mechanical properties |
| dispersability |
| protocols for: |
| inhalation testing |
| toxicology testing |
| safe handling |
| exposure determination – ambient air |
| exposure determination – water |
| safe disposal including destruction |
| eco-toxicology testing (3-8 years) |
| exposure determination - food (3-8 years) |
| exposure determination - cosmetics and other skin contact products (3-8 years) |

Material-specific test methods: CNTs

Carbon nanotubes: Sampling methods

Carbon nanotubes: batch purity – SWCNT

Carbon nanotubes: batch purity - MWCNT

Carbon nanotubes: purification methods - SWCNT

Carbon nanotubes: purification methods - MWCNT

Carbon nanotubes: dispersability, generic requirements for test (3-8 years)

Nanoscale coatings/films – generic standards

| measurement/characterization protocols for: |
|--|
| thickness |
| composition |
| scratch resistance |
| chemical structure |
| porosity |
| physical properties - optical |
| physical properties - mechanical |
| chemical functionality |
| friction coefficient |
| ageing / particles release |
| barrier properties |
| physical structure |
| physical properties - electrical (3-8 years) |

Nanostructured materials– generic standards

Nanoporous/mesoporous materials– generic standards

Specific area

Porosity

physical properties – mechanical (3-8 years)

Nanocomposites– generic standards

Dispersion of nanoscale component

Volume fraction of nanoscale component

physical properties - mechanical

ageing / particle release (3-8 years)

Nanoparticles/nanotubes – material specifications

metallic nano-particles: Ag, Al, Au, Co, Cu, Ni, Pd, Pt, Ru, Zn + Shape Memory and Magnetostrictive

metal oxides: ATO, Al₂O₃, CeO₂, CuO, Fe₃O₄, ITO, NiO, SiO₂, TiO₂, WO₃, ZnO

other compound particles: AgCu, BaTiO₃, CuNi, MoS₂, WS₂, WSe₂ + "Polymers" and "Organic"

functionalized particles: Au, Functionalized Fe oxides, SAM functionalized particles

quantum dots (3-8 years): CdS, CdSe, CdTe, GaAs, GaN, InAs, InP, Si based

Equipment – scanning probe microscopes/Analytical Electron microscopy

| |
|--|
| calibration |
| probe characterisation |
| Reference Materials |
| data interpretation |
| terminology |
| cantilever properties/Spec. |
| data acquisition |
| sample preparation |
| Equipment– analytical electron microscopy |
| magnification calibration |
| sample preparation |
| EELS characterization |
| detector energy-scale calibration |
| detector wave-length scale calibration |
| Reference Materials |

Metrological measurements at the nanoscale

length

surface roughness

mass

surface area

hardness

force (3-8 years)

porosity (3-8 years)

artefacts including CRMs

grids, gratings and scales for length measurement at the nanoscale

reference powders for instrument testing/calibration

Standardization needs survey - subject areas relevant to regulation for consumer products (1-3 years)

NANOPARTICLES

Standard Methods for Toxicological Screening of Nanomaterials

Standard Methods for Determining Relative Toxicity/Hazard Potential of Nanomaterials

Standard method for determining physical hazards of nanomaterials (i.e. explosive, flammability, water reactivity, etc.)

Standard Methods to Assess Exposure to Nanomaterials During Consumer Product Use

Standard methods for determining nanoparticle concentrations in air and water

NANOTUBES

inhalation testing

toxicology testing

Standardization needs survey - subject areas relevant to regulation for consumer products (3-8 years)

NANOPARTICLES

Standard Method for Performing Risk Assessments on Use of Nanomaterials

Product Safety Standards for Consumer Products Containing Nanomaterials

Standard Methods to Determine Environmental Toxicity of Nanomaterials

Standard Method to Assess Product Degradation and Release of Nanomaterials from Consumer Products

Standard Method to Develop Nanomaterial Product Labelling

Standard Method for Reporting Toxicity of Nanomaterials in Consumer Products

Standard Methods to Determine Exposure to Nanomaterials in Food

Standard Method of Life Cycle Analysis for Consumer Products Containing Nanomaterials

NANOTUBES

eco-toxicology testing

exposure determination - food

exposure determination - cosmetics and other skin contact products

Terminology and nomenclature for nanotechnologies

- One current document specific to nanoparticles:
 - UK PAS 71 - available for free download at <http://www.bsi-global.com/Manufacturing/Nano/Download.xalter>
- For fullerenes (nanoparticles or molecular structures???), there is a provisional nomenclature developed by IUPAC - <http://www.chem.qmul.ac.uk/iupac/fullerene/#r3>
- Chinese National standard for nanomaterials: GB/T19619-2004 Terminology for nanomaterials
- ASTM E56 Terminology for Nanotechnology <http://www.astm.org/COMMIT/COMMITTEE/E56.htm>
- Approach recommended by UK:
 - Develop series of sector specific terminologies/vocabularies, which together will form a terminology for nanotechnologies. This will :
 - allow consensus to be achieved more easily, allowing earlier publication;
 - enable changes in one area to be implemented without altering a substantial document;
 - allow documents to be developed in tandem with technology developments.
- ISO TC 229 “Terminology and definitions for nanoparticles” will be CD balloted in March.

Current UK activities in terminology

Following the success of PAS 71 (>750 downloads to date), the UK has commenced work on 6 new PAS for free distribution on the www, and submission to ISO/TC 229 as NWIPs:

- **Terminology for Medical, Health and Personal Care Applications of Nanotechnologies**
- **Terminology for the Bio-Nano Interface**
- **Terminology for Common Nanoscale Measurement Terms Including Instrumentation**
- **Terminology for Carbon nanostructures**
- **Terminology for Nanofabrication**
- **Terminology for Nanomaterials**
- **All will be published before the end of 2007**

Other current UK activities

Preparation of three guides for publication in 2007:

- **Guide to Safe Handling and Disposal of Free Engineered Nanomaterials (PD)**
- **Guide to Specifying Nanomaterials (PD)**
- **Good Practice Guide for Labelling of nanoparticles and products containing nanoparticles (PAS)**

International collaboration on “regulation” of nanotechnologies

- **OECD Working Party on Manufactured Nanomaterials (established Sept 06) has six projects:**
 - **Development of a Database on Human Health and Environmental Safety Research (EHS)**
 - **Human Health and Environmental Safety (EHS) Research Strategies on Manufactured Nanomaterials**
 - **Definition and test of a representative set of nanoparticles using standard test methods**
 - **Manufactured Nanomaterials and Test Guidelines**
 - **Co-operation on Voluntary Schemes/ Programmes**
 - **Co-operation on Risk Assessments and exposure measurements.**
- **IRGC has been undertaking a project on Risk Governance of Nanotechnology**
- **International Dialogue on Responsible Research and Development for Nanotechnology**
- **European Framework RTD projects in FP6 and FP7 – Nanosafe2, Nanoderm, Nanopathology, Nanotox, etc – opportunities in FP7.**

Ethical implications of nanotechnology

On the legal front, EGE (European Group on Ethics) does not believe that structures set up specifically to deal with nanomedicine are needed right now. However, they suggest monitoring existing regulatory systems to ensure they do address all nanomedical products.

Nanomaterial test methods needed for risk/regulation

Particle detection and measurement

- Fast, accurate methods for particle “size” measurement in air, water, food and the environment:
 - Specific Surface Area (SSA)
 - Particle size distribution
 - Shape factor
 - Particulate density/Exposure
- Work relevant to ISO TC 24 SC4 - Sieves, sieving and other sizing methods: Particle Characterisation, Size, Surface area and Zeta potential

Identification of composition and surface functionalities, including any catalytic and enzymatic characteristics

- “bulk” or “individual particle” measurements?
- Work relevant to ISO TC 201 – Surface Chemical Analysis and ISO TC 202 – microbeam analysis

May be that only particulate density and SSA + “chemistry” or SSA + oxidative potential will be needed as HS&E particle measures?

But shape also appears to be important (Chithrani, et al, *Nano Lett.* (2006) 6, 662).

Role of ISO TC 229 will be to identify requirements in cooperation with stakeholders, including industry, governments, regulators, OECD, the European Commission, and the public, to coordinate standards development with relevant TCs, and to develop standards where no TC exists, or the existing TC does not have the necessary resources.

Thank you for your attention