

3rd. Trinational Workshop on Standards for Nanotechnology

STATUS AND TRENDS OF NANOMETROLOGY AT CENAM

Speaker:

Rubén J. Lazos-Martínez
Centro Nacional de Metrología, México

Querétaro, México, February, 2008.



Mission

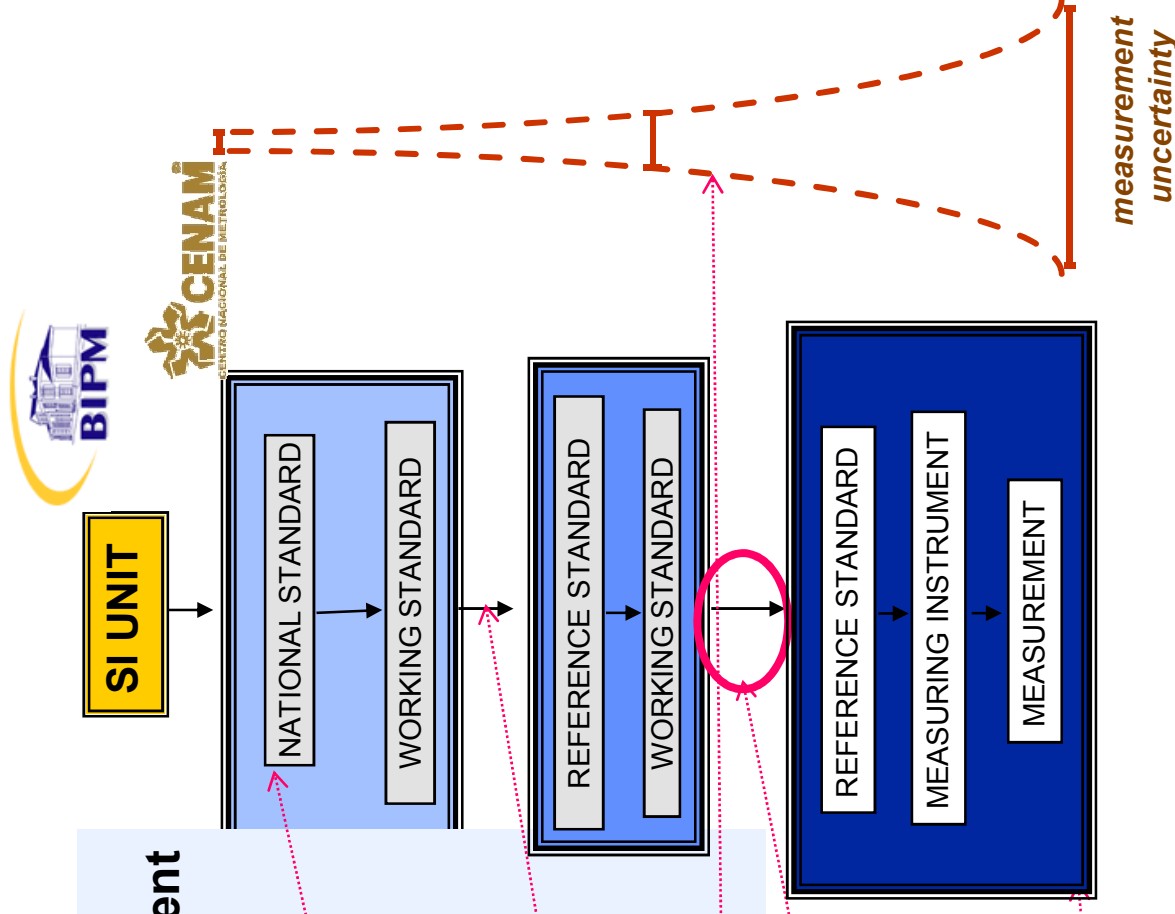
Support the various sectors of society in order to satisfy present and future demands in the field of metrology, establish national measurement standards, develop reference materials, disseminate their accuracy, provide technological services of the highest quality, aiming to increase the country's competitiveness, contribute to sustainable growth and improve the living standard of the population.

WHICH NEEDS IS CENAM SATISFYING?

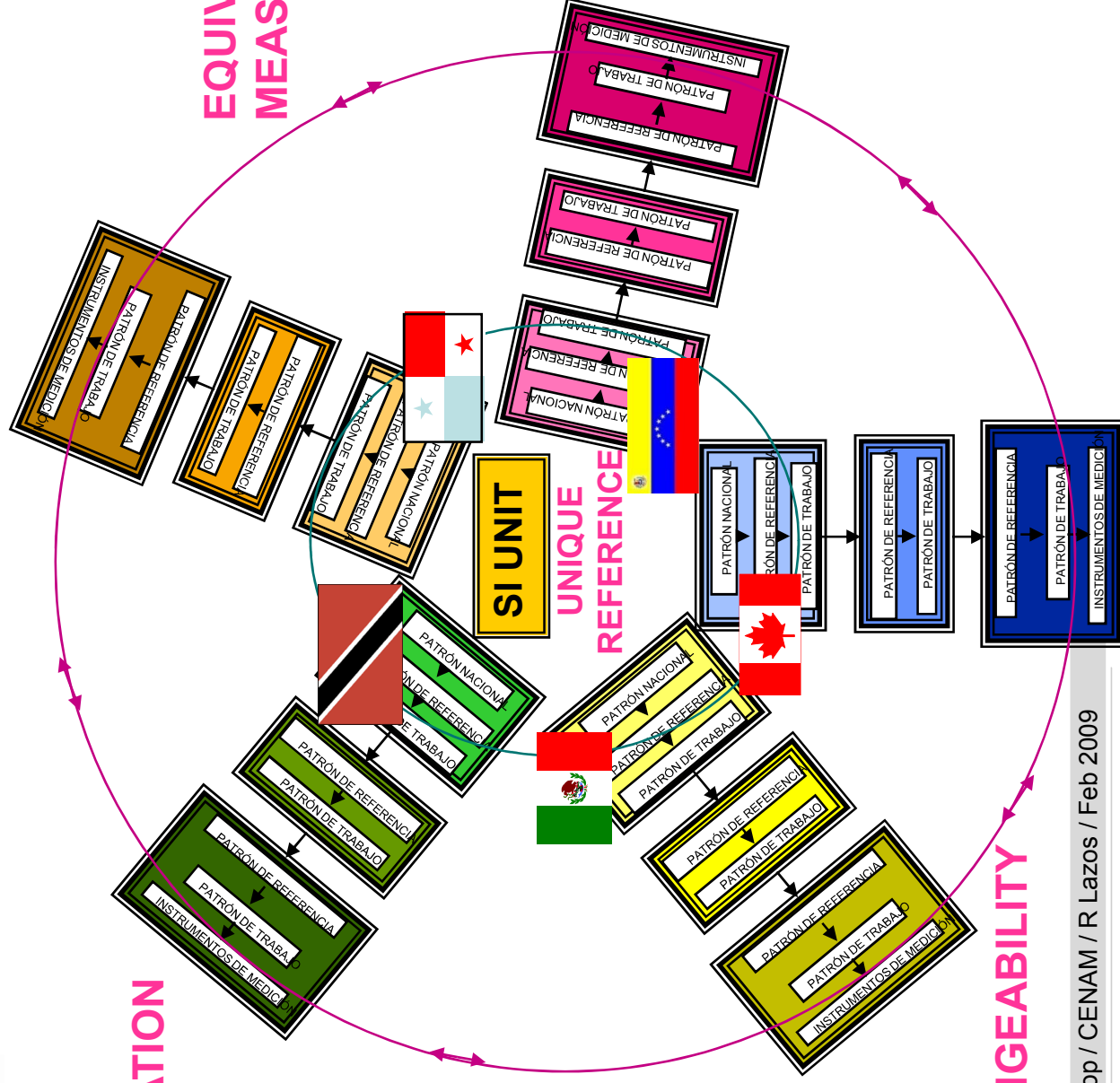
METROLOGICAL TRACEABILITY

• **Property of the result of a measurement** or the value of a standard whereby it can be **related to stated references**, usually national or international standards, through an **unbroken chain of comparisons** all having **stated uncertainties**

Methods for the comparisons have to be valid



METROLOGICAL TRACEABILITY



ISO TC 229 NANOTECHNOLOGIES

WG3 ENVIRONMENT, HEALTH AND SAFETY

- **Agglomeration / aggregation state** (fractal dimension, number particles in agglomerates binding force)
- **Composition** (chemical, crystal structure, purity)
- **Particle size and distribution** (equivalent diameter)
- **Shape** (macro-micro-meso shape descriptors)
- **Solubility** (hydrophobicity, water solubility)
- **Surface area**
- **Surface chemistry** (catalytic activity, chirality)
- **Surface charge**

OECD additional:

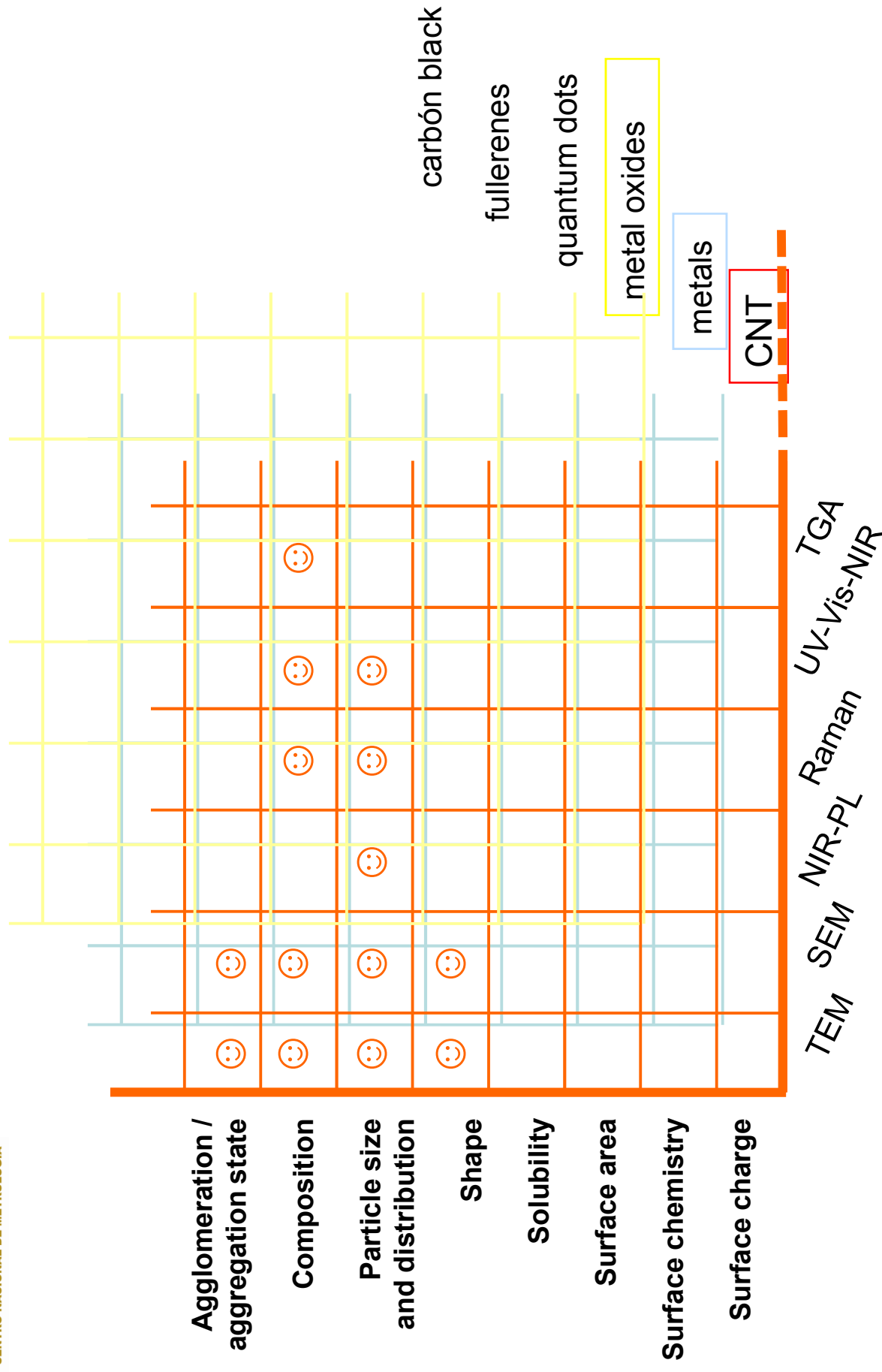
- Catalytic properties
- Concentration
- Crystalline phase
- Dustiness
- Grain size
- Length

November, 2008

Matrix: Purity & Structural Properties, SWCNTs

Method							
Property Category	SEM/EDX (Lead: USA)	TEM (Lead: USA, Co-lead: Japan)	Raman Spectroscopy (Lead: USA)	UV-Vis-NIR Absorption (Lead: Japan)	NIR-PL/ Fluorescence (Lead: Japan)	TGA (Lead: USA, Co-lead: Korea)	TG-MS (Lead: Japan)
Morphology	Tube structure, bundle thickness, orientation	Wall structure, amorphous carbon, metal catalyst coatings					
Purity	Non-carbon impurities	Tube surface cleanliness	Nanotube and non-nanotube carbon	Carbonaceous content (Quantitative) (Lead: USA)		Non-carbon content (Quantitative) Non-CNT content (Quantitative)	Non-carbon content (Quantitative)
Length and Diameter	Length and diameter	Tube diameter, metal cluster size	Diameter (Lead: Japan)	Diameter (Lead: Japan)	Diameter		
Tube Type			Metallic/ Semiconducting	Metallic/ Semiconducting (Lead: USA, Co-lead: Korea)	Chirality (Semi conducting tubes)		
Dispersability/ Solubility	Tube bundling			Tube bundling or separation (solution)	Tube bundling		
Additional						Oxidation/transition temperatures	Oxidation/transiti on temperatures

HOW COMPLEX LOOKS THE MEASUREMENT HORIZON



HUMAN RESOURCES FOR NANOMETROLOGY

Personnel:

Norma González Riojano
Juan Antonio Guardado Pérez
José Antonio Salas Téllez
Froylán Martínez Suárez
José Manuel Juárez García
José Luis Cabrera Torres
Ma. Estela Ramírez Maldonado
Fernando Rosas Gutiérrez

Competences on:

non-destructive characterization
nano wires
polymers
ceramics
electrochemistry
length

Miguel Viliesid Alonso
Carlos Colín Castellanos
Eduardo Herrera Martínez
Rubén J. Lazos Martínez

metrology
production of certified reference materials

Potentially involved
Horacio Estrada Vázquez

EQUIPMENT FOR NANOMETROLOGY

- ✓ Scanning probe microscopes: High and low vacuum
- ✓ X-ray fluorescence
- ✓ X-ray diffractometer
- ✓ Fourier Transform infrared spectroscope
- ✓ Laser diffractometer
- ✓ Optical counter
- ✓ Atomic force microscope

- ✓ Metrological microscopes
- ✓ Stabilized lasers to I₂
- ✓ High accuracy angle measurement instruments
- ✓ Optical diffractometer

SEM JSM 6390



IF NECESSARY:

- ✓ Inductively Coupled Plasma – High resolution mass spectrometer
- ✓ Gas chromatograph – double mass spectrometer



CENAM RESULTS ON NANOMETROLOGY

CERTIFIED REFERENCE MATERIALS PRODUCTION

SUPPORT TO NATIONAL INDUSTRY

INTERLABORATORY COMPARISONS

PARTICIPATION IN SPECIALIZED INTERNATIONAL COMMITTEES

CERTIFIED REFERENCE MATERIALS PRODUCTION

Mexican reference materials for size and size distribution



COMAR

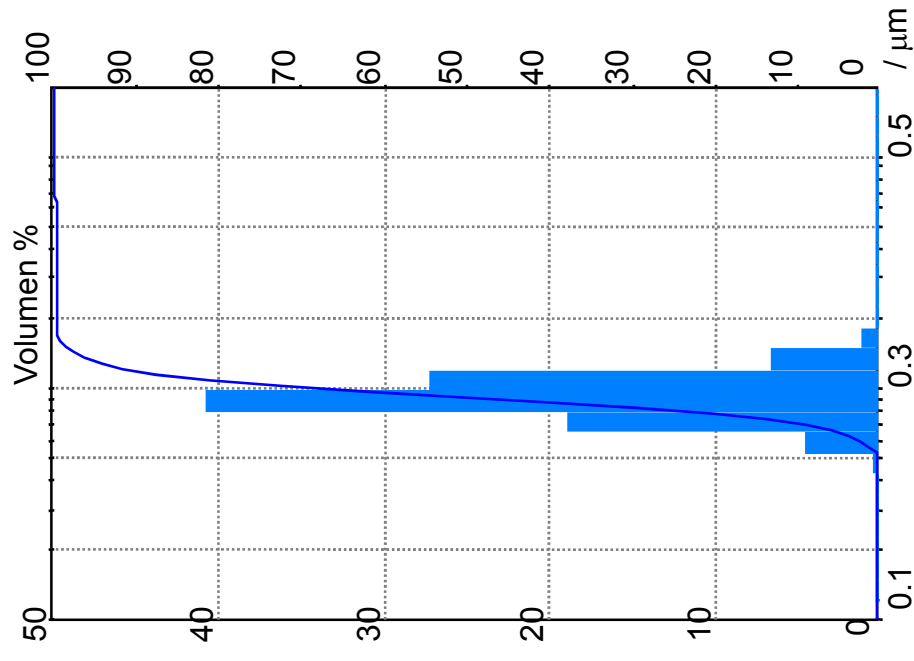


the international database for
certified reference materials

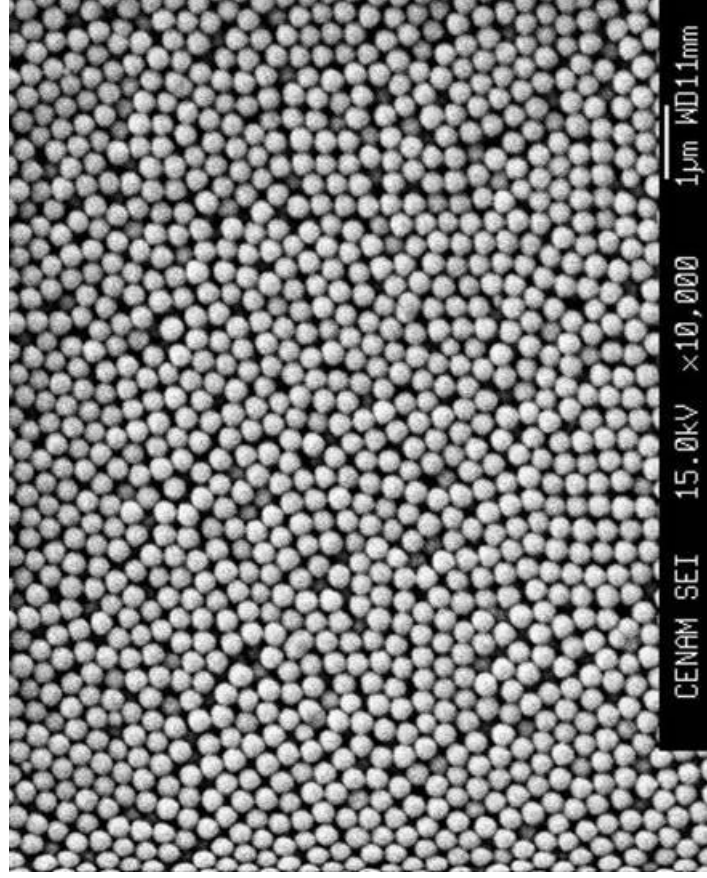
36	CRM Name	Status	Country
	DMR-92a	available	MEXICO
	Producer	Centro Nacional de Metrologia (CENAM)/Division de Materiales Ceramicos	
	Glass sphere		
	Glass spheres with the certified values of distribution particle size		
	Glass bottle with 50 g of glass sphere		
	Fields of Application	Industries -----other RM for Industry Physical Properties -----other Physical and Technological Properties	

SUPPORT TO NATIONAL INDUSTRY

❖ **150 -300 nm (2005-2006)**



Distribución de diámetro de partícula



INTERLABORATORY COMPARISONS

comparisons on particle size

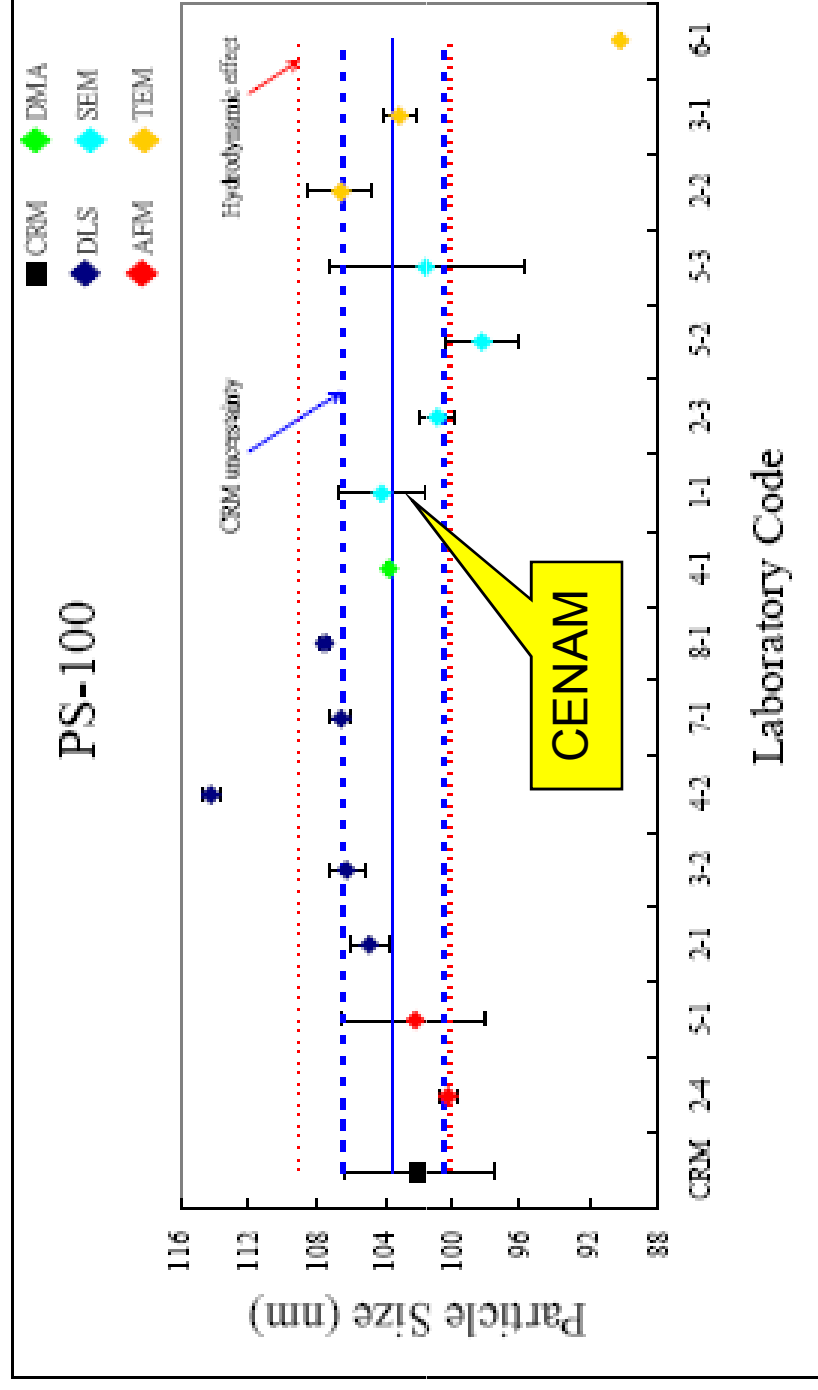


Figure 7: Measurement particle sizes from 15 participants

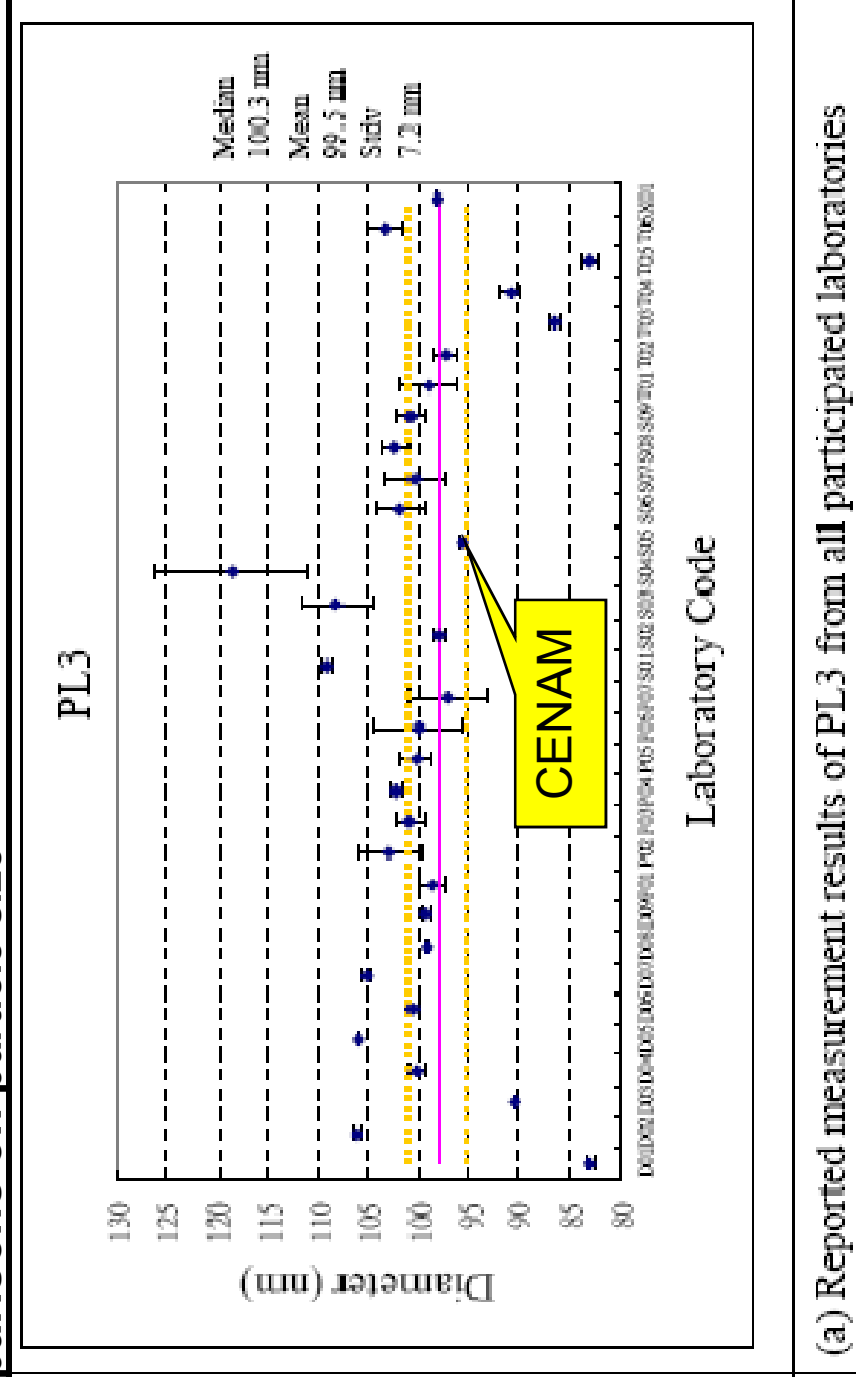
Ref: APEC Project: Preliminary Interlaboratory
 Comparison on Nanoparticle Size Characterization
 Comparison Report First Draft



**Asia-Pacific
Economic Cooperation**

INTERLABORATORY COMPARISONS

comparisons on particle size



Ref: APEC ISTWG Project Interlaboratory Comparison on
 Nanoparticle Size Characterization 2006 Report on
 Measurement Results



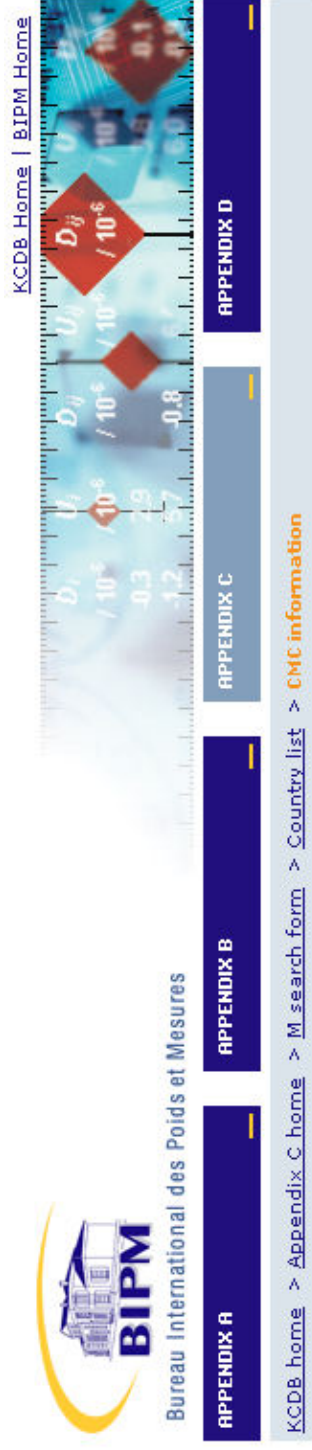
Asia-Pacific
Economic Cooperation

CCQM: Consultative Committee for Amount of Substance

CCL: Consultative Committee for Length

CENAM's CALIBRATION AND MEASUREMENT CAPABILITIES IN THE CIPM-MRA

465
CMCs



Appendix C - Result of the search

KCDB
[KCDB home](#)
[MRA](#)
[ICRB](#)
[Find my NMI](#)

Appendix C
[Appendix C home](#)
[Appendix C news](#)

Metrology area
[AUV](#)

Calibration and Measurement Capabilities Mass and related quantities

In the CMCs uncertainty statements, the notation $Q[a, b]$ stands for the root-sum-square of the terms between brackets: $Q[a, b] = [a^2 + b^2]^{1/2}$

➤ Result of the search

➔ **Your selection** : Mass and related quantities, Mass, mass standards, Mass

Mexico, CENAM (Centro Nacional de Metrologia)
[Complete CMCs in Mass and related quantities for Mexico \(.PDF file\)](#)

Mass: Mass standard, **1 mg**
 Absolute expanded uncertainty ($k = 2$, level of confidence 95%) in mg: **0.001**
 Subdivision method
 Temperature: 20 °C
 Humidity: 40% to 60%
 Internal NMI service identifier: CENAM/730-F008-C062



VAMAS

Versailles Project on Advanced Materials and Standards

VAMAS

Technical Working Areas

... international collaboration takes place to agree the technical basis for test method development and, using round robin intercomparisons, the test methods are checked to ensure that consistent results are obtainable at different laboratories, and provide precision (repeatability and reproducibility) data.

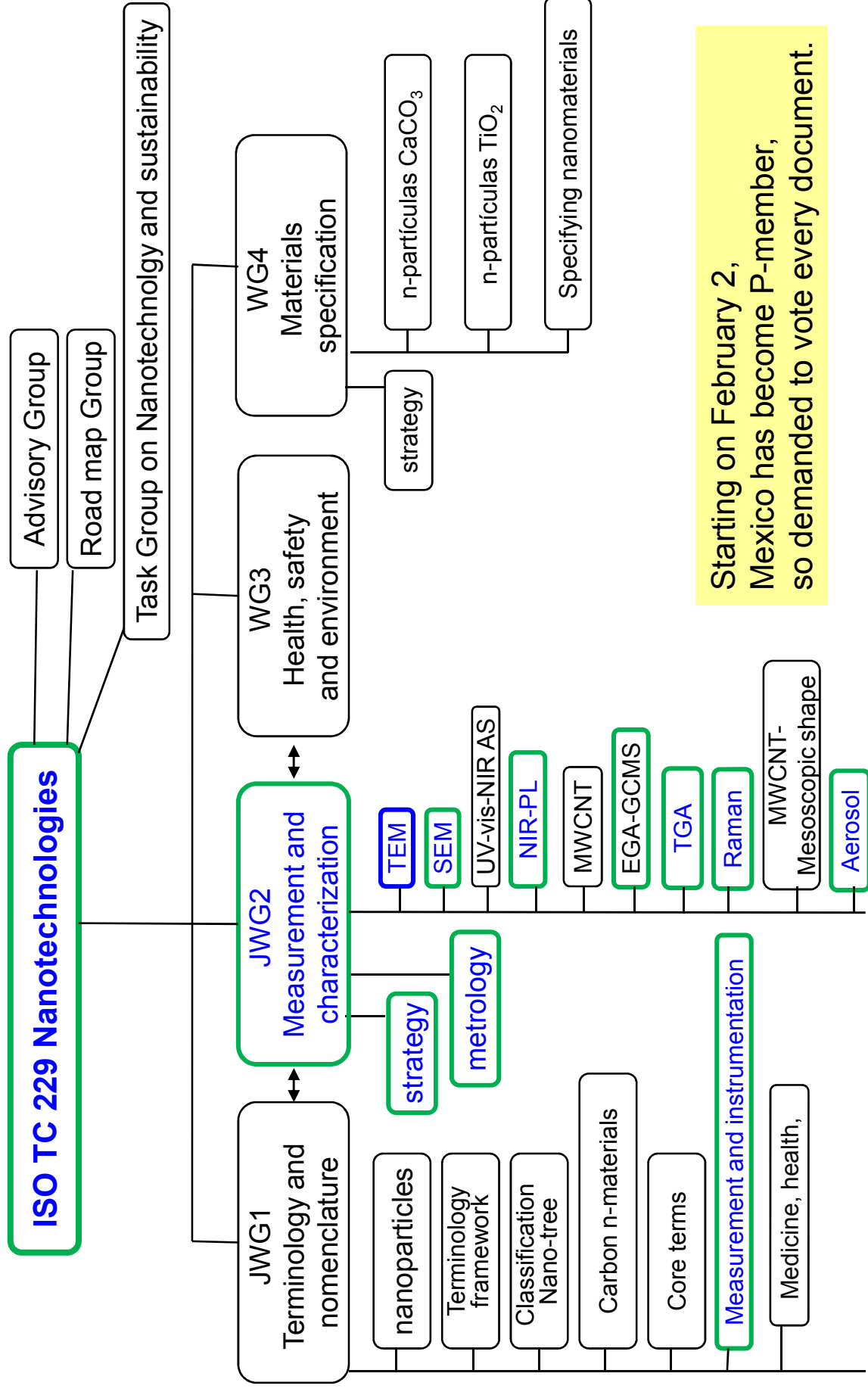
Nanoparticle Populations

... advance the measurement of nano-particles (NP) ...metal nano-wires, single-walled carbon nanotubes, magnetic nano-particles, nanopowders and quantum dots.

The focus ... is standardization and cross-comparison of measurement techniques for determining the defining dimensional, electronic, chemical, optical or magnetic characteristics of the nano-particles.



PARTICIPATION IN SPECIALIZED INTERNATIONAL COMMITTEES



Starting on February 2, Mexico has become P-member, so demanded to vote every document.

CENAM ACTION AXIS ON NANOMETROLOGY

Axis

Projects

I. Nano-objects in the air

Calibración de rejillas por difracción
Desarrollo de métodos para la caracterización de la morfología de nanopartículas en aire

II. Polymeric nanocomposites

Effects of polymeric nanocomposites in clays

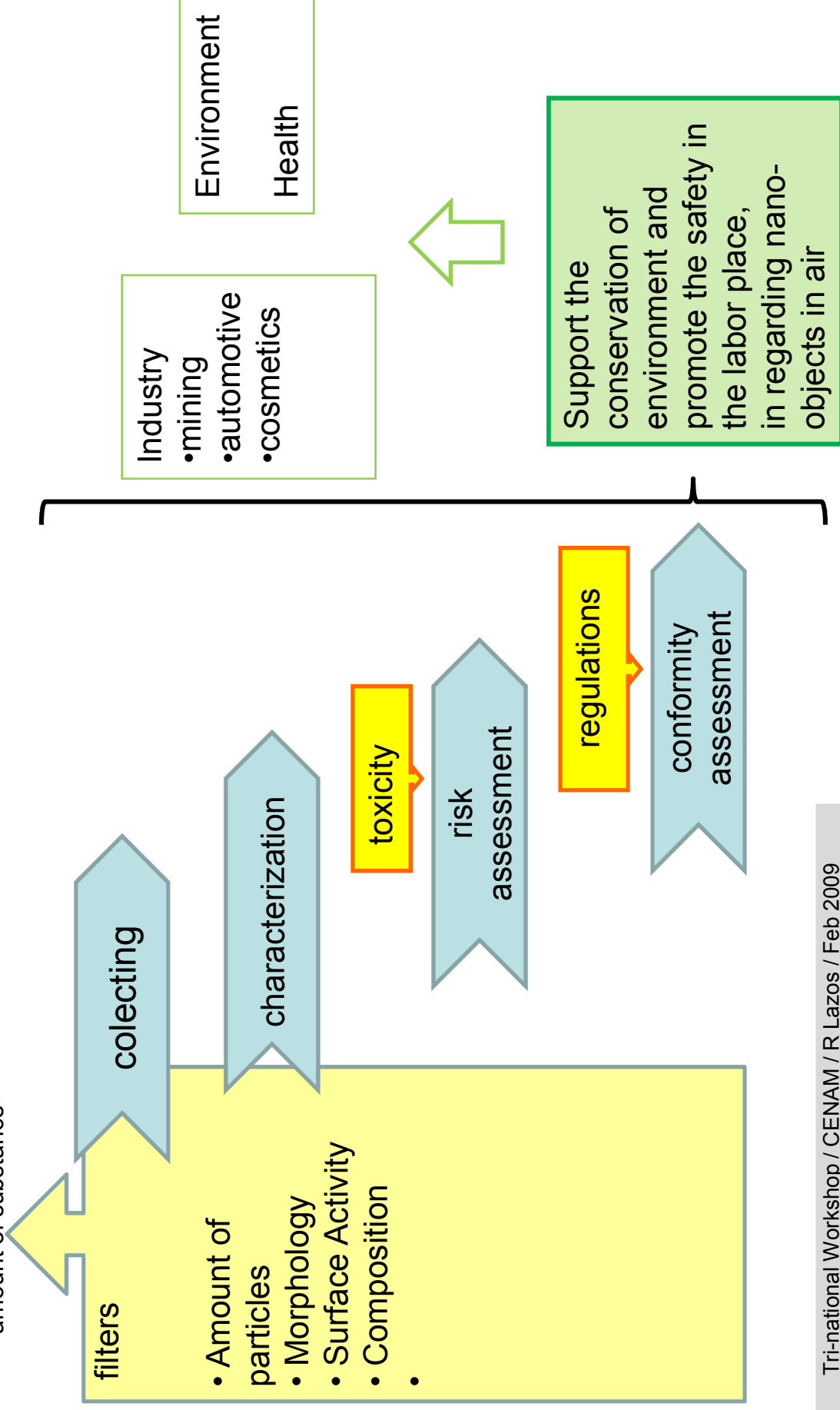
III. Physical properties of nanomaterials

Scanning probe microscopy for mechanical characterizations
Elasto-plastic properties of nanomaterials

Axis I. Nano-objects in air. Strategic framework

SI Units

- length
- amount of substance



Some benchmark:

NPL's first calibration service for airborne nanoparticle number concentration

Concern about the impact of nanotechnology on the environment and our health has emphasised the need to monitor our exposure to nanoparticles. In response to this, NPL has recently developed a calibration service for instruments that measure airborne nanoparticle number concentration, such as Condensation Particle Counters.

Measurement of particle number concentration is used in many applications, including ambient air monitoring and vehicle emissions assessments. These instruments have been increasingly used over the last 20 years to give a measure of the ultrafine fraction of particulate matter (typically below 100 nm in diameter). Their historical traceability to internationally recognised standards has been relatively weak, but the need to ensure worldwide

comparability has recently become much apparent.

The drivers for this change are partly due to traditional mass-based techniques for particle measurements not being able to achieve the required sensitivity, as the amount of particulate matter from the latest generation of engines drops, and also the desire to understand the sources and behaviour of particulate matter in ambient air over the widest possible

size range. NPL's calibration service is designed to help both these and other industry sectors. NPL is the first laboratory in the UK, and to the best of our knowledge the whole world, to have developed a procedure that has been ISO 17025 (UKAS) assessed for this service.

For more information on calibration of these or related instruments, please contact
Richard Gilham 020 8943 6405
richard.gilham@npl.co.uk

REFERENCE MATERIAL CERTIFICATION

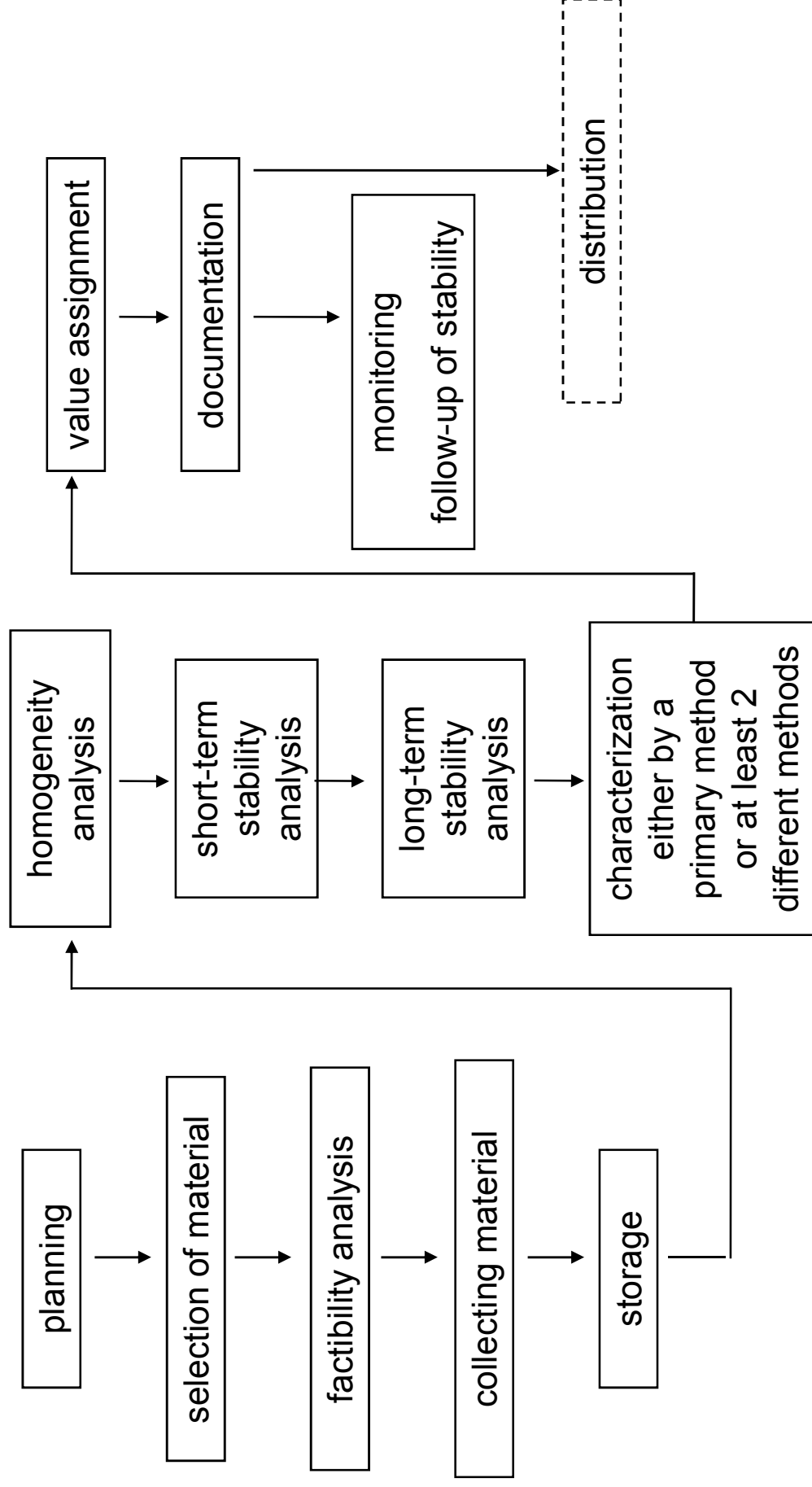
Production of certified reference materials is NOT a trivial task

Requires:

- A controlled production process for the material
- Material homogeneous and stable
- Characterization of the material
- Validated method for assigning values
- Metrological traceability

ISO Guide 34 General requirements for the competence of reference materials producers shall be complied with.

REFERENCE MATERIAL CERTIFICATION PROCESS



Adapted from: H. Emons, T.P.J. Lisinger, B.M. Gawlik, *Reference materials: terminology and use.*
 Can't one see the forest for the trees?, *Trends in Analytical Chemistry*, Vol.23, No.6, 2004.

FINAL REMARKS

- Nanomaterials are already around us
- Manufacturing, handling and using them with proper care is vital for society
- Measurements play an unavoidable role to take responsible care of nanomaterials
- Some reliable measurements are available in Mexico, but the horizon is still far away
- CENAM has knowledge, human resources, equipment and infrastructure to address some of the measurements needs derived from nanotechnologies.
- Mexico needs to cleverly use the resources, human and infrastructure, available

We appreciate your attention

Ruben J. Lazos Martínez
Centro Nacional de Metrología
MÉXICO

Phone: +52(442) 211 0575
e-mail: rlazos@cenam.mx