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Institute for National Measurement Standards

Recent Developments in Canadian Nanotechnology Measurement Science and ISO Standards Initiatives

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Tri-National Workshop on Standards for Nanotechnology

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National Research Council Canada | Conseil national de recherches Canada

Canada

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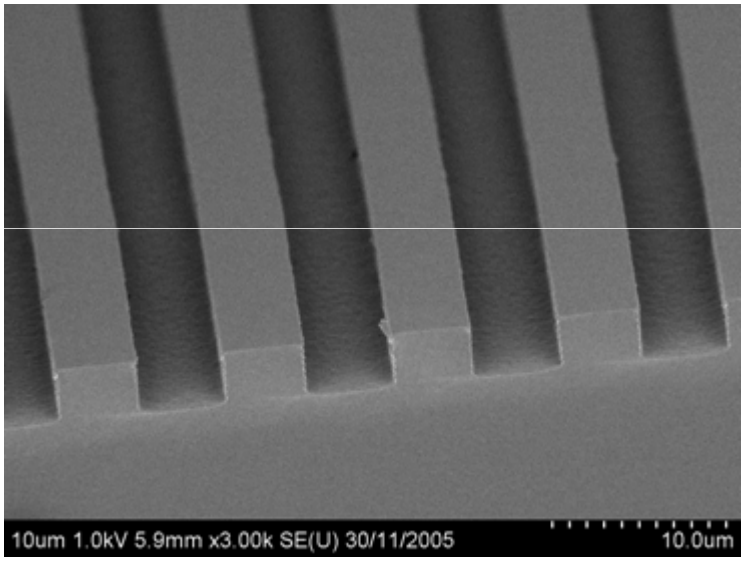
Introduction

- Elements required to enable the platform of nanotechnologies:
  - Traceability to the SI
  - Reference Materials/Artefacts
  - Documentary Standards



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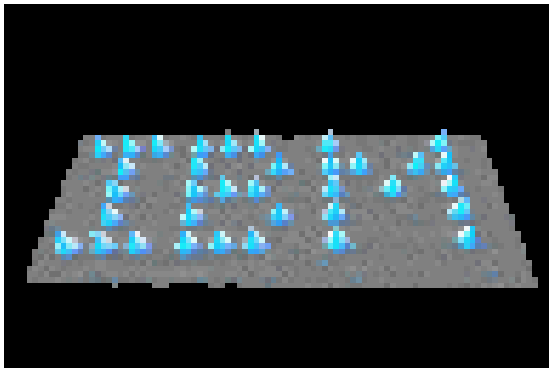
Nanotechnology

Norio Taniguchi, Tokyo Science University

– First to use the term in 1974, *College International pour la Recherche en Productique (CIRP)*

"Nano-technology mainly consists of the processing of separation, consolidation, and deformation of materials by one atom or one molecule."

D.M. Eigler, E.K. Schweizer
"Positioning single atoms with a scanning tunneling microscope"
Nature ***344*** *524-526 (1990)*



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Nanotechnologies

- >800 nano- Consumer Products
  - *Nano-objects include: particles, tubes, ropes, surfaces. . .*
  - *OECD 'short list' of 14 manufactured nanomaterials*

- How to characterize them?
  - *Select some out of many scientific methods*

Nanotechnology Consumer Products Inventory, Woodrow Wilson International Center for Scholars

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R&D + Standards for Managing Risk

- *Unique identification* of these nanomaterials is *essential* for *reliable* scientific investigation

1. Establish a *name* for the unique nano-object (ISO/TC229 JWG1, JWG2)
2. Establish a *body of R&D* (identification, characterization, predictive toxicology, etc.) to indicate if this object is safe or toxic (international R&D cooperation, OECD)
3. Establish *standard methods* to identify and report the characteristics of this entity (ISO)
4. *International consensus* agreement on *testing protocols* is required for trade (ISO/TC229 WG3, OECD)

Documentary Standards

- Standards promote levels of quality, safety, reliability, efficiency and interchangeability

- ISO/TC229, IEC/TC113 Nanotechnologies
  - JWG1 Terminology & Nomenclature: Canada
  - JWG2 Measurement & Characterization: Japan
  - WG3 Health, Safety, Environment: USA
  - WG4 Product Specifications: China

ISO International Organization for Standardization

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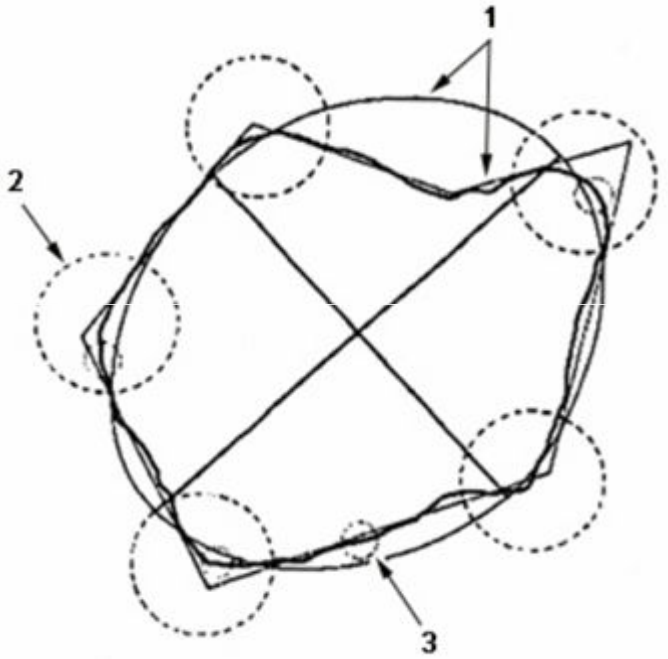
Identification of a Measurand

- **ISO/TC 229 WG 3/PG 5 Project: Guidance on physico-chemical characterization of engineered nano-objects for toxicologic assessment**
  Leader: Dr. Richard Pleus (USA)

- Purpose: What is the intended application of the measurement?

- Measurements to be compared with each other should be traceable to the same reference (SI units)

- Available measuring instruments and reference materials



ISO FDIS 9276-6 Descriptive and quantitative representation of particle shape and morphology

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Recent Developments

- **Metrological Content ISO/TC229 Standards:** Two documents to steer towards more comprehensive metrological content
  - Check List: Used prior to accepting draft standard for ballot
  - Guidance on Metrological Content: harmonize content & terminology

- Category A Liaison with BIPM
  - Definition of the base SI units + realization at the nanoscale
  - International comparisons for validation of measurements

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Line Scales: IEC/TC113

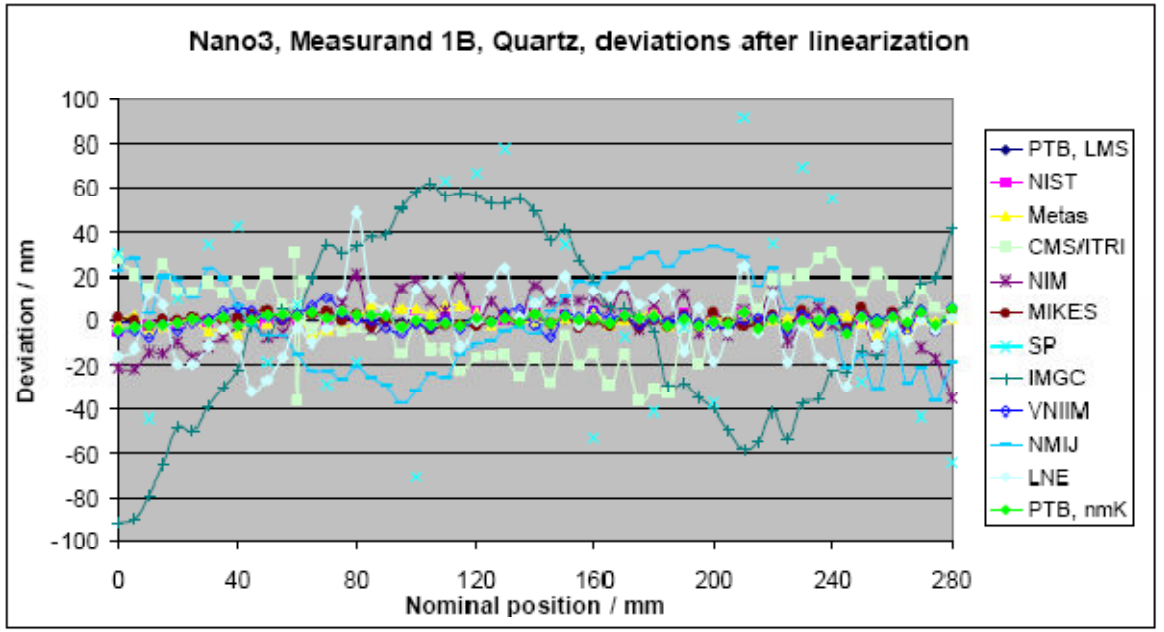

Fig. 26: Results on quartz scale, measurand 1B: deviations from reference data after linearization.

- Artificial gratings used in nanotechnology: description and measurement of dimensional quality parameters
- Refers to latest international comparisons of national metrology institutes:
  - **CCL-S3 (Nano3)**, CCL-S1 (Nano4 1D gratings), CCL-S5 (Nano5 2D grids)

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NRC-INMS Initiatives

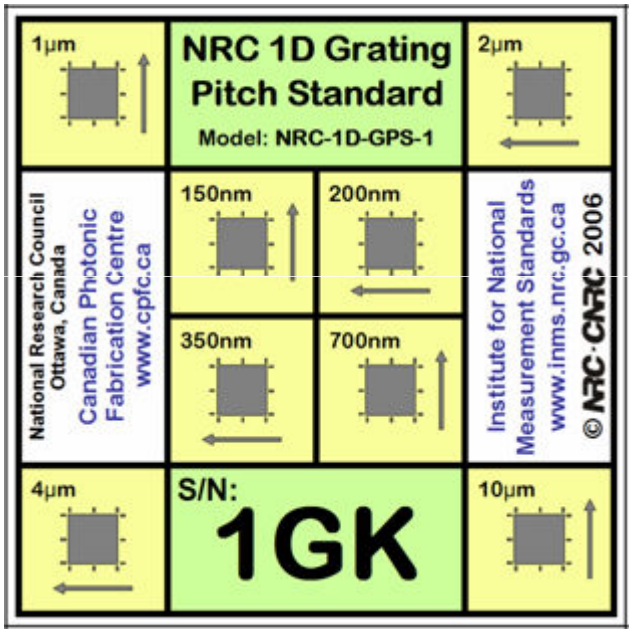
- Chairman of Metrology Study Group + participate in Strategy Group of ISO/TC229 JWG2
- Canadian Advisory Committee Mirror Chairmanship of JWG2
- Negotiated Category A Liaison between ISO/TC229 & International Bureau of Weights and Measures (BIPM)
- Pre-treatment protocols for single-walled carbon nanotubes
  - Canada-US-UK collaboration
- Liaison with Canadian delegation to OECD

NRC-INMS Program of Activities

- Primary Metrology = Direct traceability to the SI
- Development of metrological instruments and methods
- Client calibration services and reference materials; intrinsic standards
- Rigorous validation of measurements impacts ability to interpret results
  - international round-robin comparisons with other national metrology institutes

NRC-INMS R&D Projects

- Reference standard artefacts for length calibration
- Metrological Atomic Force Microscope for traceable custom shape/size
- Quantum-based Voltage standards
- Optical Characterization of Nanomaterials
- Dielectric measurements of nanocomposites
- FTIR Spectroscopy & thickness measurement
- Molecular imaging: Detection of nanoparticles in biological systems



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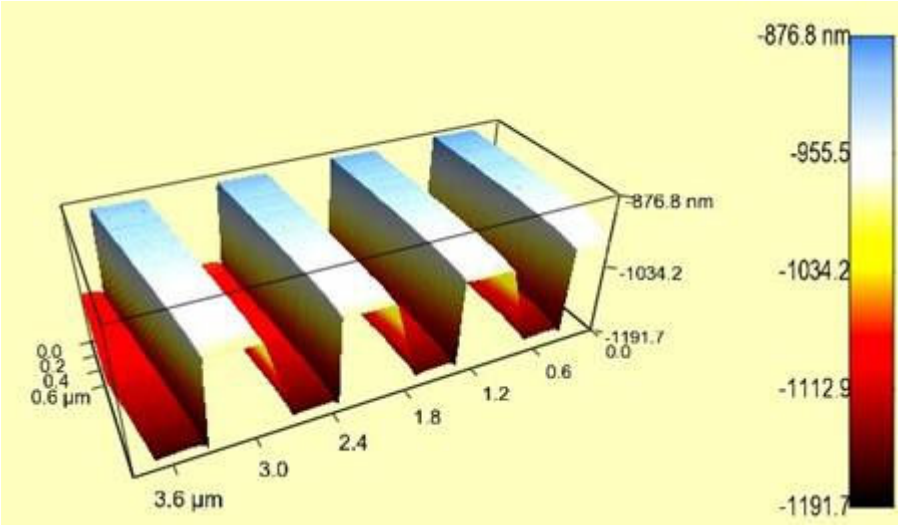
1-Dimensional Grating Pitch

- Measurements traceable to the definition of the metre through wavelengths of light by:
  - Optical diffraction technique
  - Interferometer coupled to microscope stage translation

- Important characteristics
  - Spatial uniformity of grating pitch
  - Flatness of substrate
  - Accurate angle measurement



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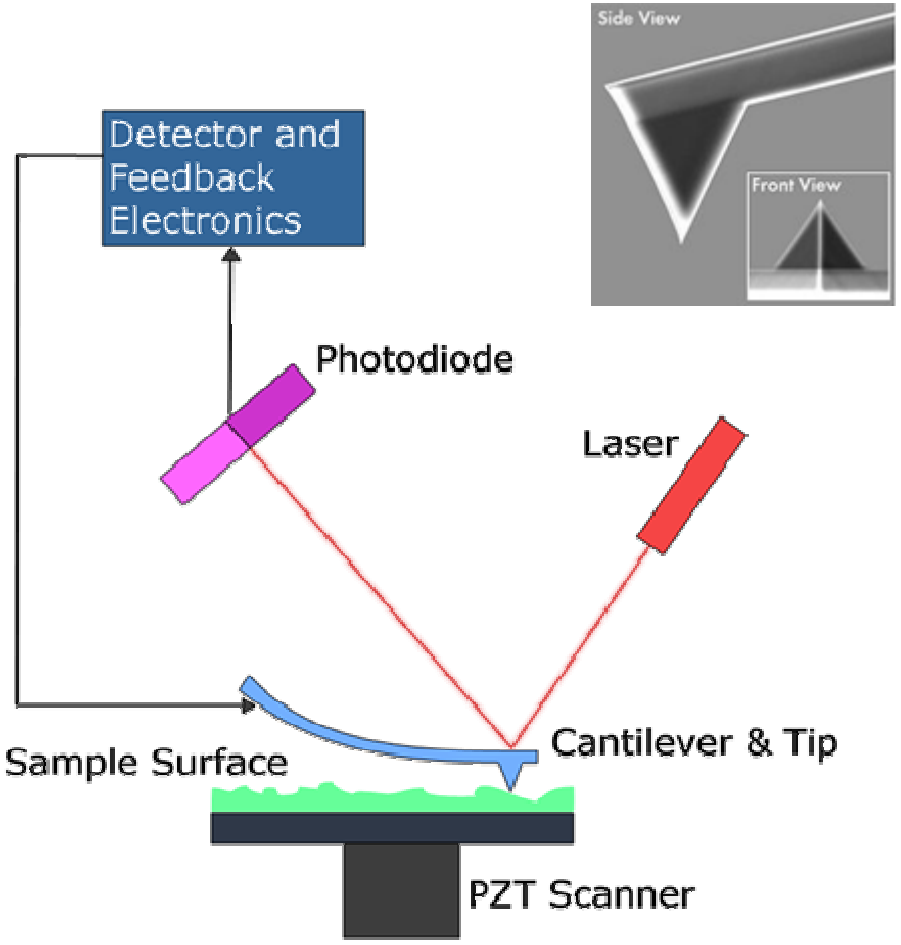
Atomic Force Microscope (AFM)

- "Feeling" the surface with a mechanical probe
  - Topography (mechanical contact)
  - Electrostatic force
  - Magnetic force
  - Measure additional quantities e.g. thermal microscopy
- Develop techniques & methods for accurate measurement with commercial AFMs
  - Reference materials: grating pitch, step height, line width



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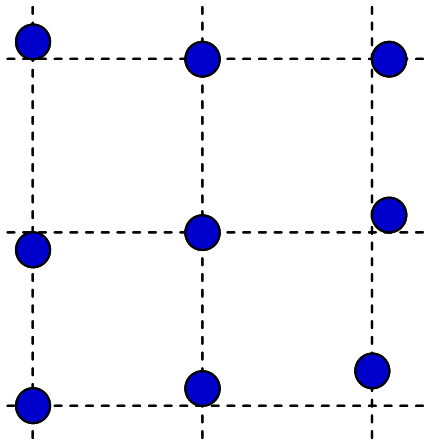
Metrological Atomic Force Microscope

- Measurement errors in measured points of a grid in relationship to a perfect ‘true’ grid (dashed lines)
- Advance technological developments in metrological SPM instrumentation and in so doing, provide highest accuracy and precision measurement capability to clients
- In-house development project:
  - motion control stage built on 2-D flexure stage
  - x,y,z co-ordinates read using homodyne interferometers
  - autocollimators sense sample orientation



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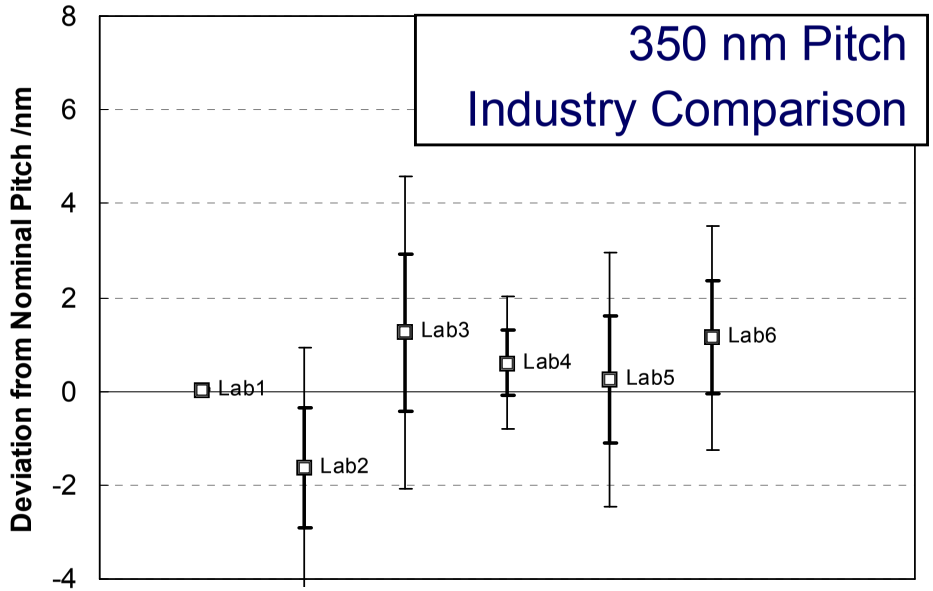
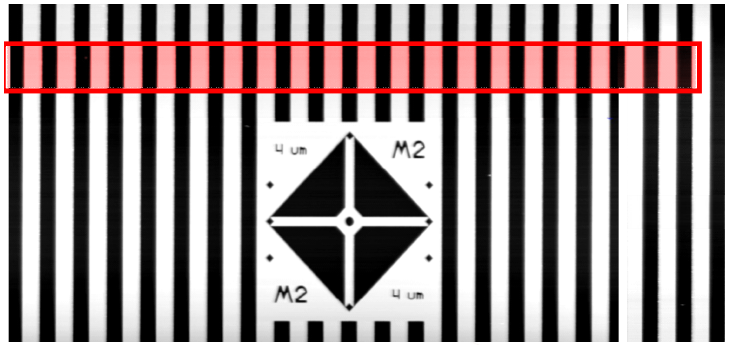
International Comparisons

- Two Comparisons:
  - National Metrology Institutes: Canada, Taiwan, Germany, Switzerland
  - Industry Labs: Canada + Taiwan
- *Reproducibility* & *global comparability* achieved through traceability to the SI





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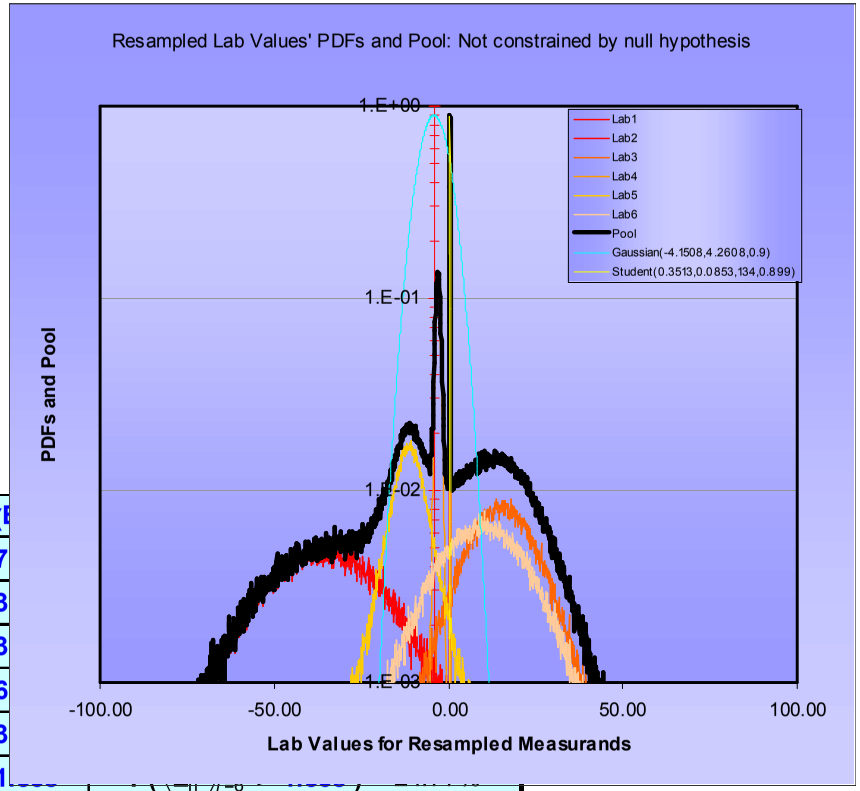
Rigorous Statistical Methods for Analysis of Comparison Results

NRC Monte Carlo Toolkit
http://inms-ienm.nrc-cnrc.gc.ca/qde/montecarlo/



	Lab1	Lab2	Lab3	Lab4	Lab5	Lab6	⟨E	
Lab1	2.687	1.96	-1.44	4.83	2.53	-0.70	7	
Lab2	-1.96	1.920	-2.41	-1.77	-1.29	-2.00	3	
Lab3	1.44	2.41	1.824	1.77	2.35	0.32	3	
Lab4	-4.83	1.77	-1.77	2.594	1.77	-0.95	6	
Lab5	-2.53	1.29	-2.35	-1.77	1.947	-1.49	3	
Lab6	0.70	2.00	-0.32	0.95	1.49	1.240	1	
	Experimental pair-difference $E_{n\,ij} = (x_i - x_j) / (u_i^2 + u_j^2)^{1/2}$						4.381	$P(\chi^2_{APD} > 4.381) = 5.78\%$
	RMS $E_{n\,j} = [\,(\sum_{i=1}^{N} (E_{n\,ij})^2 / (N-1)\,]^{1/2}$						564.261	$P(\chi^2_{SM} > 564.261) = 33.60\%$
							7.212	$P(\chi^2_{WM} > 7.212) = 3.24\%$
							81.916	$P(\chi^2_{MED} > 81.916) = 31.84\%$

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NRC-INMS R&D Collaborations

- NRC Cross Institute Metrology for Nanotechnology (INMS-IMS-NINT-SIMS)
  - Interfacial Force Microscope
  - Nanoimprint Lithography applied to artefact standards
  - Intrinsic length standards
  - Soft materials
- NRC-NSERC-BDC Quantum Candela
  - Metrological photon counting to support calibrations (single-photon detectors)
- Nanodielectrics & Diagnostics Electrical + Electronics Industries (INMS-IMI)
- Reference Materials for Single-walled Carbon Nanotubes (INMS-SIMS-IRC)

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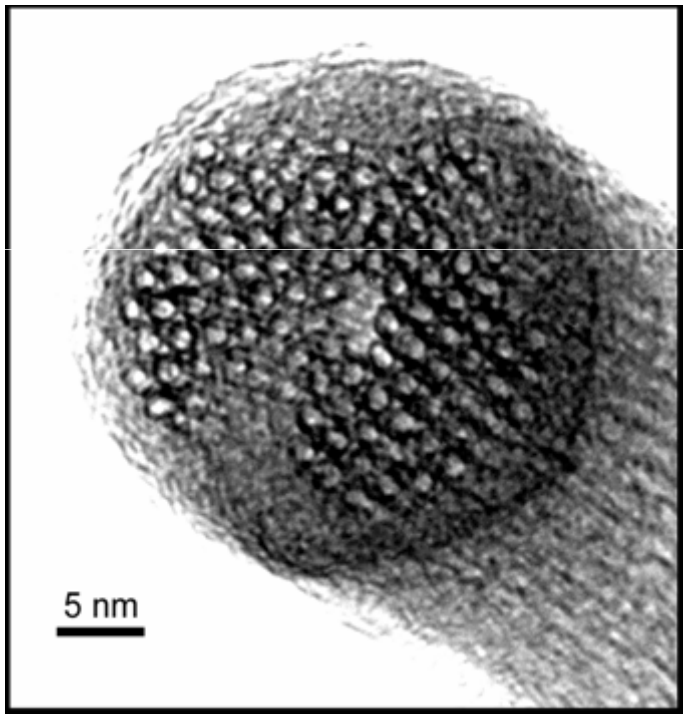
International Collaborations

- Canada-USA Reference Materials for Single-Walled Carbon Nanotubes
  - Sample preparation protocols for reproducible results
- Canada-USA-UK Sample Preparation Protocols for Single-Walled Carbon Nanotubes
  - Multiple methods
  - Precursor to international comparison for validation + documentary standards
- Molecular Imaging



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Upcoming Meetings

- **Tri-National Workshop Standards & Nanotechnology**
  - NRC (Ottawa) 2010: Reference Materials
    Chairs: Sturgeon & Simard
  - North American Partnership Platform (NAPP)
- **International conference on the science and application of nanotubes** (NT10) – Satellite Meeting on Reference Materials

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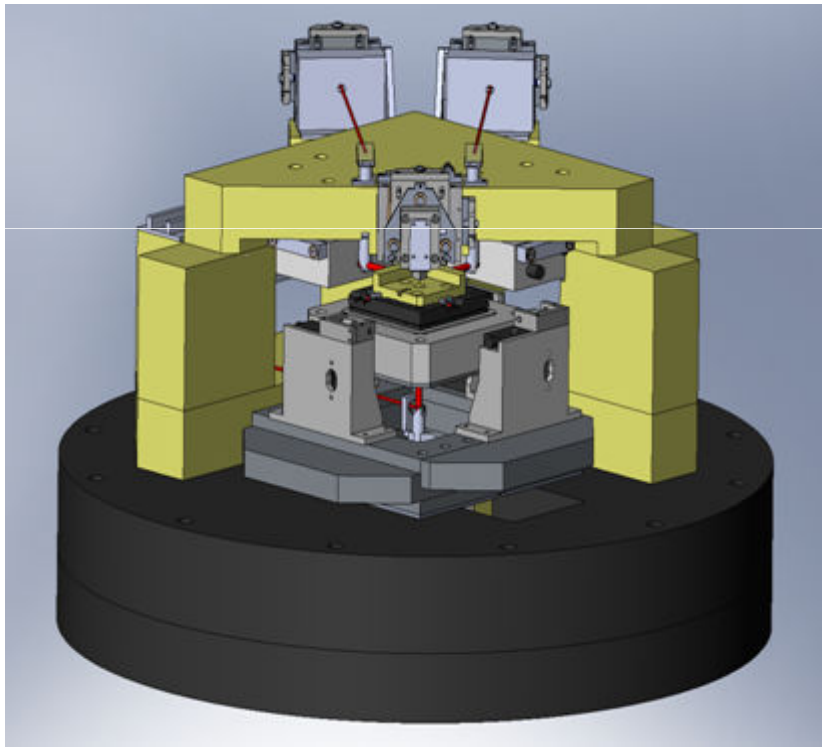
Standards & Metrology Support Nanotech Development

- *Nanoscience* can only evolve into *nanotechnology* once the measurement problems and metrology are under control
  - *Traceability to the SI*
  - *Reference materials*
- Economic impacts from product development and commercialization demand *documentary standards*
- Workplace safety, environment and health are key drivers



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