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INTRODUCCIÓN

Es imprescindible para el desarrollo de una economía asegurar la uniformidad de las medidas y vincular el conocimiento científico y el progreso tecnológico con aplicaciones que aseguren la consistencia de las mediciones. Contribuye a ello, el conocimiento de las innovaciones que se hacen en estos campos, y su divulgación.

Por eso es importante el INFOSIM como medio de difusión para la comunidad del Sistema Interamericano de Metrología.

En este número se reproducen aportaciones sobre el ensayo de aptitud para validar el método de calibración de pipetas como un medio para demostrar la capacidad técnica de los analistas de laboratorios de ensayo y para unificar sus criterios en torno a los métodos de calibración, así como sobre las técnicas para la calibración dimensional de balanzas de presión usadas en la nueva determinación de la constante de Boltzmann. En el campo de la nanotecnología, tenemos tres contribuciones: uno referente a patrones para la caracterización de las propiedades físico-químicas y biológicas de los nanomateriales, otra referente a la medición de dureza en el Brasil en la era de la nanotecnología y por último, la nanoescala en el NRC. El establecimiento de la escala de tiempo del SIM, es otra interesante contribución que aparece en esta edición.
Además de la columna NOTISIM en la que damos a conocer los acontecimientos relevantes de nuestra comunidad, en este número hemos iniciado la sección, CRONOSIM, que nos llevará por el tiempo y el espacio a recordar, para los que lo vivimos y a conocer para los que no, el orígen de nuestro SIM, con el testimonio de aquellos que fueron protagonistas de este acontecimiento.

Humberto Brandi,
Presidente del SIM

INTRODUCTION

It is essential for the development of the economies to ensure the uniformity of the measurements and to link the scientific knowledge and the technological progress with applications aimed to assure the consistency of the measurements. The knowledge of innovations which are realized in this fields and its dissemination contribute to it.

Therefore the INFOSIM plays an important role as a means of diffusion toward the community of the Inter-American Metrology System (SIM).

This edition reproduces contributions about the proficiency test to validate the calibration method of pipettes as a medium to demonstrate the technical capability of the test laboratory analysts and to unify its criteria within calibration methods, as well as about the techniques for the dimensional calibration of pressure balances to be used in the new determination of the Boltzmann constant. In the nanotechnology field we count with three contributions: one on the standards for the characterization of the physical-chemical and biological properties of the nanomaterials, the other about the measurement of hardness in Brazil in the era of nanotechnology and finally the nanoscale in the NRC. The establishment of the SIM time scale is another interesting contribution which appears in this edition.
In addition to the section NOTSIM in which the relevant events of our community are presented, this edition also started the section CRONOSIM, to guides us throughout time and space to remind, to those who experimented it, and to present to those who did not, the origin of the SIM, through the testimony of its protagonists.

Humberto Brandi,
President of the SIM

METROLOGÍA, INNOVACIÓN Y COMPETITIVIDAD

Oscar Harasic
Organización de los Estados Americanos

A través de la historia no ha habido desarrollo tecnológico que se haya logrado sin una base de mediciones confiable. A su vez, la metrología, que es la ciencia de las mediciones, cuenta con instrumentos cada vez más sofisticados que son producto de constantes innovaciones tecnológicas, generalmente provenientes de los países con alto grado de desarrollo.

La medición de longitud ilustra claramente el proceso de innovación a partir de tecnologías disponibles y que, con el paso del tiempo, han permitido mediciones más exactas. A partir de su definición, el metro ha pasado de un patrón físico (barra de platino-iridio), a la longitud de onda de una radiación emitida por el isótopo 86 del krypton y recientemente a la medición basada en la velocidad de la luz utilizando un láser estabilizado. El láser fue el resultado de un proceso de investigación y desarrollo sin aplicaciones previstas cuando fue inventado. Sin embargo, hoy en día, tiene múltiples aplicaciones no solo en la metrología sino en varias ramas de la medicina, la astronomía y la exploración espacial, solo por mencionar algunos ejemplos.

En efecto, la innovación es la aplicación de una idea hasta su explotación efectiva en beneficio directo para la sociedad. Su relevancia ha sido destacada en la reciente Segunda Reunión de Ministros y Altas Autoridades en Ciencia y Tecnología promovida por la OEA, llevada a cabo en octubre de 2008 en la Cd. de México, cuyo tema central fue "Ciencia, tecnología, ingeniería e innovación para la prosperidad", y la cual produjo una nota de atención a las autoridades para esforzarse en incrementar la inversión pública y privada en ciencia, tecnología, ingeniería e innovación. Para lograrlo, evidentemente debe considerarse que la medición constituye uno de los pilares indispensables.

Throughout history, no technological development has been achieved without the support of a reliable measurement basis. Besides, Metrology, the science of measurements, uses more sophisticated instruments every time obtained from technological innovations generally originated in economies with high levels of development.

The measurement of length clearly illustrates the innovation process starting from available technologies that, throughout the time, have allowed more accurate measurements. From its definition, the meter has been realized, first, by a physical standard in the form of a platinum-iridium bar, then by the wavelength of a radiation emitted by the isotope 86 of krypton, and nowadays the definition is based on the speed of light by using a stabilized laser.

The laser became the result of a research and development process without any foreseen applications at the time of its invention. However, today it has multiple applications in the astronomy and in space exploring, as examples, besides those in metrology.

As a matter of fact, innovation means to pursue an idea until its effective exploitation to directly benefit the society. Its relevance has been highlighted in the Second Meeting of Ministers and High Authorities on Science and Technology held in Mexico in October 2008, promoted by the OAS, where the "Science, technology, engineering and innovation for prosperity" was its central theme, and produced a call to the authorities for "endeavoring to increase public and private investment in science, technology, engineering and innovation". In order to achieve it, measurements should be considered as one of the indispensable pillars.

Mientras que en el pasado, los recursos naturales y materias primas eran los elementos principales del comercio y la riqueza, hoy en día la mayor parte del comercio mundial está compuesta por productos manufacturados y dentro de ellos predominan aquellos con alto contenido tecnológico. Los países con mayor desarrollo tecnológico han atraído empresas que han sabido innovar sus procesos y productos, y en consecuencia han logrado una mayor participación en el comercio mundial, lo cual ha redundado en una mejor calidad de vida para sus habitantes. Estos países cuentan con una excelente infraestructura nacional de calidad resultado de varias décadas de fomento y apoyo a sus sistemas de mediciones. Dicha infraestructura debe apoyarse en cinco componentes principales: metrología, normalización, pruebas y ensayos, acreditación y certificación, siendo la metrología la base para que funcione dicha infraestructura. Por ello, una infraestructura nacional de calidad es fundamental para impulsar la innovación, la productividad y la comptetitividad internacional.

La búsqueda del mejoramiento de calidad se inicia con mediciones para llegar a la certificación de productos y procesos y puede tomar la forma de un sello de calidad que garantiza las especificaciones declaradas por el productor o los requerimientos del consumidor. La certificación de la calidad junto con el precio de los productos y servicios, y las formas en que éstos son proporcionados garantiza la competitividad en los mercados nacionales e internacionales. Por medio de la competitividad las empresas obtienen acceso a nuevos mercados y mantienen sus mercados ya establecidos. Esta expansión ayuda a crear nuevos empleos y mayores ingresos económicos, que a su vez contribuyen a elevar la calidad de vida, ayudan a erradicar la pobreza y a un mayor desarrollo tecnológico, económico y social. Los sistemas de medición en las empresas son esenciales para apoyar procesos de innovación que ayuden a elevar su productividad y competitividad.

While the natural resources and raw materials were the main elements for trade and wealth in the past, nowadays most of the worlds commerce deals with manufactured products, with predominance of those of high technological content. The highly developed countries host organizations that have been able to innovate their processes and products, and so have reached a major share in the world trade, impacting the quality of life of their people. These countries count on an excellent national structure for quality as a result of several decades of fostering and support to their measurement systems. Such an infrastructure should be based on five main components: metrology, standardization, testing, accreditation and certification, being metrology the basis for this structure to operate. Therefore, a national quality infrastructure is fundamental to promote innovation, productivity and international competitiveness.

The search for improving quality starts with measurements to reach products and processes certification, by a quality seal to guarantee the stated specifications by the supplier or the customer requirements as one of the ways. Quality certification together with the prices of products and services, and the way these are supplied are warrants of the competitiveness in the national as well as international markets. By means of the competitiveness, the organizations obtain access to new markets and keep their already established ones. This expansion helps to create new jobs and greater income, that contribute to raise the quality standards, to eradicate poverty and to a better technological, economical and social development. Measurement systems in the organizations are essential elements to support innovation processes to help the raise in productivity and competitiveness.

ENSAYO DE APTITUD REALIZADO EN LATU PARA VALIDAR EL MÉTODO DE CALIBRACIÓN Y USO DE PIPETAS DE VIDRIO Y PIPETAS AUTOMÁTICAS

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RESUMEN

Se realizó en 2008 un ensayo de aptitud con la participación de varios analistas (nuevos en su mayoría) de los laboratorios de ensayo del LATU. Para la misma se utilizaron dos pipetas, una de vidrio de 1 ml, clase A y una pipeta automática de volumen regulable entre 0 y 1 ml, la cual se calibró para el valor correspondiente a 1 ml, Se pidió que cada analista repitiera entre 6 y 10 veces cada pipeta, con el fin de evaluar la repetibilidad en la medición. Se evaluó la media de cada analista para cada pipeta y se estimó la incertidumbre expandida del valor medio obtenido por cada analista. Se presentan los gráficos con las medias y las incertidumbres obtenidas para cada tipo de pipeta. Se realiza un estudio comparativo de los mismos. Como conclusión se evalúa la reproducibilidad de cada tipo de instrumento, en base a lo cual se realizan recomendaciones para su uso y calibración.

1. INTRODUCCIÓN

El LATU es una institución certificada por la norma ISO 9001 [1] y aproximadamente 250 ensayos químicos y físicos se encuentran acreditados por la norma ISO 17025 [2]. Está implementado por lo tanto un Sistema de Gestión que asegura la calidad de los resultados emitidos. En el punto 7.6 de la ISO 9001, así como en el punto 5.6 de la ISO 17025 se establece la necesidad de utilizar equipamiento calibrado durante la realización de los ensayos.

Como estamos hablando de una institución grande, que incluye diferentes Departamentos analíticos especializados, la cantidad de material volumétrico que se utiliza en los mismos es muy grande. En el pasado todo el material volumétrico era calibrado en al área de volumen correspondiente al Departamento de Metrología. Para mejorar la gestión de los recursos y la calidad de los resultados analíticos se implementó la política de calibrar el material volumétrico por parte del personal de cada uno de los departamentos que hace uso del mismo. Esto tiene la ventaja de asegurar que el material se calibre en las mismas condiciones y con la misma metodología con que se usa, disminuyendo de esta forma los errores sistemáticos en el uso y favoreciendo el entrenamiento del personal que utiliza el material volumétrico, haciéndolo participar del proceso de calibración.
Para evaluar la competencia del personal, la calibración del equipamiento y el aseguramiento de calidad en los distintos Departamentos, se han implementado Ensayos de Aptitud en calibración de material volumétrico periódicos en el que participan los nuevos analistas (de forma de validar la forma de uso del material volumétrico) y analistas más experimentados (para evaluar si sus resultados siguen estando en tolerancia).

Debemos tener en consideración que los problemas detectados en la calibración del material volumétrico, reflejan problemas en el uso del mismo, o sea en todos los procesos que involucren material volumétrico.

Frente a la gran cantidad de métodos químicos existentes que involucran, a su vez, un número creciente de matrices y analitos, nos enfrentamos, desde el punto de vista metrológico, ante la falta de oferta de interlaboratorios y/o de materiales de referencia para cada método y en cada analito y matriz. Es en estos casos que se debe recurrir a la evaluación de los métodos químicos, asegurando metrológicamente cada subproceso involucrado (por ejemplo: uso de material volumétrico, extracciones, separación cromatográfica, etc.). Teniendo esto en cuenta es importante tener el subproceso de uso de material volumétrico en control.

En cada Ensayo de Aptitud, se selecciona un par de ítems de material volumétrico que son calibrados por técnicos del Departamento de Metrología para asignarles el valor de referencia. El método utilizado está plasmado en un procedimiento interno de calibración basado en las normas ISO 4787 [3] e ISO 648 [4] por ejemplo en el caso de pipetas. Luego los

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mismos se hacen circular según un plan especificado entre los analistas de los distintos Departamentos analíticos del LATU para que procedan a su calibración utilizando el mismo procedimiento. Luego de realizada la misma, los resultados de calibración acompañados de sus respectivas incertidumbres son informados al organizador, quien procesa los datos, evalúa los mismos y confecciona un reporte. Luego se realiza una reunión de devolución con los participantes donde se discuten los resultados, se asignan causas probables a las tendencias y desviaciones y se evalúan posibles acciones correctivas y preventivas a implementar.

En el caso del ensayo de aptitud objeto del presente estudio se evaluó la competencia de distintos analistas en el uso y calibración de una pipeta aforada de vidrio de 1 ml (la cual fue seleccionada de forma de que los errores de manipulación se vieran amplificados por su forma de construcción y descarga) y una pipeta automática de volumen variable en la cual se seleccionó un volumen de 1 ml.

Los resultados obtenidos sirvieron no solo para evaluar la calidad de las mediciones de volumen en los Departamentos, sino también para comparar el comportamiento en el uso de estos dos tipos de pipetas y las fuentes de incertidumbre asociadas a cada una y su cuantificación.

2. PARTICIPANTES

Participaron 10 analistas de distintos departamentos del LATU, 8 de ellos pertenecientes al área química y los 2 restantes al área de volumen del Departamento de Metrología.

El valor de referencia fue calculado como el promedio ponderado de los valores obtenidos por los técnicos del área de volumen del Departamento de Metrología en el caso de la pipeta aforada y por la media aritmética de los participantes en el caso de la pipeta automática. Las mediciones de volumen son trazables al Sistema Internacional, ya que se tiene un sistema de calidad basado en la ISO 17025, y las capacidades de masa y temperatura están declaradas en el apéndice C del acuerdo de reconocimiento mutuo del CIPM. Además se ha participado en Intercomparaciones en volumen en el marco del SIM en forma exitosa.

3. RESULTADOS

3.1. Resultados Pipeta de Vidrio

Tabla 1. Resultados obtenidos para la pipeta aforada.

Analista	1	2	3	4	5	6	7	8	9	10
Resultado Laboratorio/ cm^3	0,9612	0,9655	0,9696	0,9983	0,9677	0,9925	0,9995	0,9708	0,9800	0,9926
Incertidumbre Analista/cm^3	0,0077	0,0018	0,0044	0,0054	0,0073	0,0079	0,0029	0,0012	0,021	0,0084
Valor de referencia/cm^3	0,9653	0,9653	0,9653	0,9653	0,9653	0,9653	0,9653	0,9653	0,9653	0,9653
Incertidumbre Valor Referencia/cm^3	0,0150	0,0150	0,0150	0,0150	0,0150	0,0150	0,0150	0,0150	0,0150	0,0150
Error /cm^3	-0,0041	0,0002	0,0044	0,0330	0,0024	0,0272	0,0342	0,0055	0,0147	0,0273
Error relativo	-0,0041	0,0002	0,0044	0,0330	0,0024	0,0272	0,0342	0,0055	0,0147	0,0273
Error normalizado	-0,24	0,01	0,28	2,07	0,14	1,61	2,24	0,37	0,57	1,59

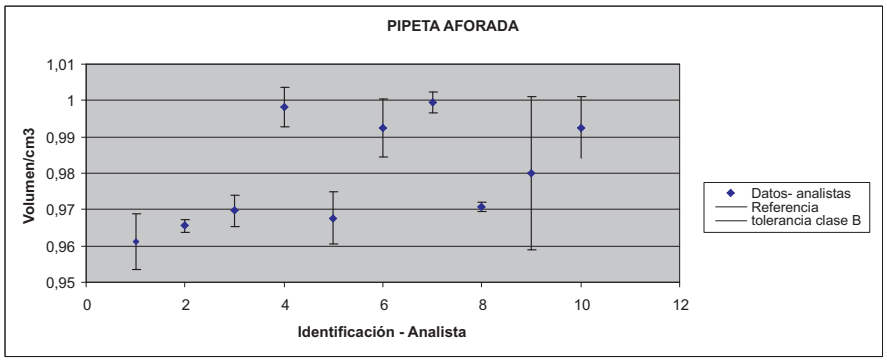

Fig.1. Gráfico de resultados con su incertidumbre- pipeta aforada.

3.2. Resultados de Pipeta Automática

Tabla 2. Resultados obtenidos para la pipeta automática

Laboratorio	1	2	3	4	5	6	7	8	9	10
Resultado Laboratorio/ cm^3	1,0035	1,0056	0,9933	1,0004	1	0,9993	0,9986	1,0028	0,9995	1,0053
Incertidumbre Laboratorio/cm^3	0,0008	0,0008	0,0049	0,0044	0,0002	0,0007	0,0015	0,0008	0,0013	0,0032
Valor de referencia/cm^3	1,00083	1,00083	1,00083	1,00083	1,00083	1,00083	1,00083	1,00083	1,00083	1,00083
Incertidumbre Valor Referencia/cm^3	0,005	0,005	0,005	0,005	0,005	0,005	0,005	0,005	0,005	0,005
Error /cm^3	0,0027	0,0048	-0,0075	-0,0004	-0,0008	-0,0015	-0,0022	0,0020	-0,0013	0,0045
Error relativo	0,0027	0,0048	-0,0075	-0,0004	-0,0008	-0,0015	-0,0022	0,0020	-0,0013	0,0045
Error normalizado	0,53	0,94	-1,08	-0,06	-0,17	-0,30	-0,43	0,39	-0,26	0,75

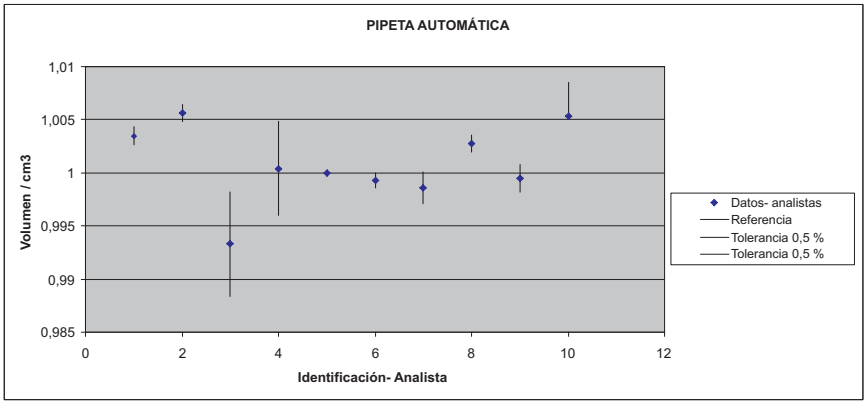

Fig. 2. Gráfico de resultados con su incertidumbre- pipeta automática.

3.3. Resultados de Repetibilidad de las dos Pipetas

Tabla 3. Desvíos estándar de los valores obtenidos para cada pipeta.

ANALISTA	1	2	3	4	5	6	7	8	9	10
PIPETA DE VIDRIO/ml	0,0108	0,0024	0,0130	0,0024	0,0065	0,0110	0,0027	0,0019	0,0182	0,0030
PIPETA AUTOMÁTICA/ml	0,0007	0,0009	0,0059	0,0050	0,0027	0,0009	0,0019	0,0009	0,0017	0,0017

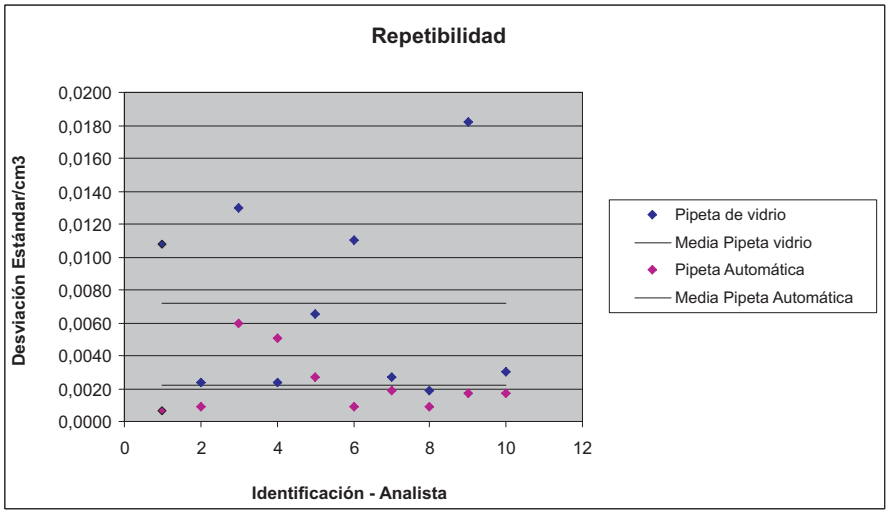

Fig. 3. Gráfico de desvíos estándar

NOTAS:

A) $$E_n \quad \frac{E}{\sqrt{U^2_{LATU} \quad U^2_{LAB}}}$$

E_n - Error normalizado

E - Error = Valor informado por el analista – Valor de referencia

U_{LATU} - Incertidumbre en el valor de referencia En el caso de la pipeta aforada se tomó igual a la tolerancia de la misma y en caso de la automática a un 0,5 % de su volumen.

U_{LAB} - Incertidumbre en el valor informado por el analista.

Un error normalizado superior a 1 implica que el error en la medición es significativo.

Es una medida del número de incertidumbres (valores que surgen de combinar la incertidumbre en el valor de referencia con la informada por cada analista) que estamos alejados del valor de referencia.

Para ver la significancia real en un posible resultado, por ejemplo de una solución valorada, si se sigue el mismo procedimiento que el utilizado en la calibración se ve en la fila de “errores relativos”. Por ejemplo el valor final de una solución valorada preparada por cada analista participante tendrá un error relativo mayor o igual que el que aparece en esta fila de la tabla.

4. DISCUSIÓN

En el caso de la pipeta aforada, 4 de los 10 analistas presentan errores normalizados mayores que 1.

En el caso de la pipeta automática todos los errores normalizados son inferiores a 1.

Inferimos que la pipeta automática tiene mejor reproducibilidad

Podemos ver además que en el caso de las pipetas automáticas los valores de repetibilidad son mejores

5. CONCLUSIONES

A los efectos de analizar los resultados obtenidos y tomar las acciones correctivas necesarias, en caso de resultados no conformes para el uso propuesto, es importante tener en cuenta que algunas de las posibles causas de errores sistemáticos pueden ser:

En el caso de la pipeta aforada:
- Errores en el enrase
- Diferencias en la forma de vaciado de la misma respecto a la especificada en el procedimiento, sobre todo en la descarga final, ya que se debe dejar el tiempo de escurrido especificado y no realizar ningún movimiento brusco con la misma que provoque la descarga de las últimas gotas que no caen por gravedad

Con estos errores considerados en la mayoría de los casos se obtendrían valores superiores a los que se obtienen si se sigue el procedimiento en todos sus términos

En el caso de la pipeta automática:
- Diferencias en la forma y la fuerza utilizadas para presionar el mecanismo de vaciado de la misma

En ambas pipetas:
- Errores en las mediciones de temperatura
- Errores originados por la calidad del agua destilada utilizada.

Se observa que en el caso de la pipeta aforada, hubo problemas de manipulación por parte de algunos analistas, que hacen que la reproducibilidad de los resultados no sea buena. Se eligió una pipeta de 1 ml clase B, de formato tal que los posibles errores de manipulación se vieran amplificados, de forma de evidenciar y corregir los mismos. Esta evaluación se realizó en la reunión de discusión de resultados, donde se discutieron las posibles causas de los errores constatados, planteándose todas las posibilidades evaluadas. Se detectó que los valores que están por encima, en su mayor parte se deben a que se realizó algún movimiento brusco de la pipeta, el cual provocó la salida de la última porción de agua de su interior que en otras condiciones no se hubiera dado. Pueden haber existido problemas también en la visualización del menisco. Se resolvió entonces proceder a un entrenamiento exhaustivo del personal para unificar los criterios de uso, sobre todo en el caso de bajos volúmenes, ya que en los ensayos de aptitud anteriores, con volúmenes mayores, los resultados han sido buenos. Como en este caso se usó una pipeta que amplificaba los problemas, convendría repetir el estudio una vez finalizado el entrenamiento además con una pipeta de las mejores de plaza y comparar el desempeño de ambas con cada analista.

En el caso de la pipeta automática, los valores obtenidos tienen una mayor reproducibilidad, lo que evidencia que el método de descarga es el mismo en todas las ocasiones y no parece depender apreciablemente de la fuerza y la forma en que cada analista realiza la descarga.

Teniendo esto en cuenta y observando los valores de repetibilidad en la Fig. 3 se evidencia que la repetibilidad y reproducibilidad de las mediciones de volumen en el caso de la pipeta automática son mejores que en el caso de la pipeta aforada de vidrio.

Este ejercicio nos hace llegar a las siguientes conclusiones:

- Cuando se usa material volumétrico aforado, sobre todo cuando sus volúmenes son pequeños, debe procederse a una apropiada selección del mismo
- Debe mantenerse un entrenamiento continuo del personal en el uso adecuado del material volumétrico
- Es recomendable, en el caso de querer minimizar las incertidumbres, que la persona que usa el material volumétrico sea la misma que lo calibra. Vemos en ambos tipos de pipetas que los valores de repetibilidad son mucho mejores que los de reproducibilidad, lo que nos hace concluir que si es la misma persona que usa y calibra, se obtendrán menores incertidumbres en los resultados
- El uso de pipetas automáticas disminuye la influencia del analista en los volúmenes entregados, siempre y cuando estas sean de buena calidad.

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[3] ISO 4787 Laboratory glassware volumetric glassware Methods for use and testing of capacity (1984) International Organization for Standardization, Geneva, Switzerland.

[4] ISO 648 Laboratory glassware One-mark pipettes (1977) International Organization for Standardization, Geneva, Switzerland.

DIMENSIONAL CALIBRATION TECHNIQUES FOR PRESSURE BALANCES TO BE USED IN THE NEW DETERMINATION OF THE BOLTZMANN CONSTANT

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Abstract

A measurement procedure for the dimensional calibration of piston-cylinder type primary pressure standards is described. The piston-cylinder assemblies are intended to be used in a project for the re-determination of the Boltzmann constant. The measurement procedure includes optical and mechanical contacting, form and diameter measurement. A numerical post-processing procedure is applied to generate precise three-dimensional data sets of the piston-cylinder surfaces required for the effective area determination.

Key words: pressure metrology, Boltzmann constant, dimensional metrology

1. INTRODUCTION

Primary gas pressure standards up to 2 MPa are frequently realized by pressure balances with piston-cylinder assemblies as major measuring components. The nominal effective area of such piston-cylinder assemblies varies typically between 5 cm^2 and 20 cm^2. Their corresponding diameters range from 25 mm to 50 mm.

The effective area of pressure balances is usually calculated by using Dadson's theory [1]. The calculation is based upon dimensional input data.

This paper describes the needed measurement procedures and the principle of the data analysis with emphasis on the project for the re-determination of the Boltzmann constant.

2. THE RE-DETERMINATION OF THE BOLTZMANN CONSTANT

PTB and other NMIs have started a project towards a re-determination of the Boltzmann constant k_B. The Boltzmann constant is the proportional factor between thermal and mechanical energy. Therefore, the project may lead to a new definition of the Kelvin [2]. The chosen experimental method of the Boltzmann project is the dielectric constant gas thermometry (DCGT). The uncertainty of that method strongly depends on the uncertainty of the absolute pressure to be measured in the range up to 7 MPa (requirement: 1 part per million (ppm) [3]. Consequently the pressure measurement uncertainty for the Boltzmann constant project is very demanding, resulting also in high demands for the dimensional calibration utilised for the calculation of the effective areas. The target for the standard uncertainty of the radial values of the 3D data net of the piston-cylinder calibration, including form and size, is approximately 25 nm.

2.1 Piston-cylinder assemblies for the Boltzmann project

To achieve the goal, special prototype piston-cylinder assemblies of 20 cm^2 and 2 cm^2 nominal effective area have been designed and manufactured by DH Instruments (USA). The assemblies are made from tungsten-carbide. The effective area of the three 2 cm^2 piston-cylinders will be linked to that of the three 20 cm^2 assemblies and, as they are operated in the same pressure balances, the 2 cm^2 assemblies will cover the pressure range up to 7.5 MPa. The pressure distortion coefficients of the piston-cylinder assemblies of both sizes will be determined from the elastic properties of tungsten carbide, which the assemblies are made of, and their dimensional properties applying FEM (Finite Element Method) [4]. The consistency of the dimensionally based effective areas of the six piston-cylinder assemblies will be verified by cross-float experiments.

Fig 1. Piston (left) and cylinder (right) of a pressure standard piston-cylinder assembly. This model was custom-made for the Boltzmann constant project. The artefacts are mounted on clamping disks.

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3. MEASUREMENT EQUIPMENT

3.1 Coordinate measurement machines

In principle, the 3D calibration of piston-cylinder pressure standards is a typical measurement task for 3D coordinate measuring machines (CMM). However, the measurement uncertainty of CMMs generally is larger than approx. 0.5 µm, in many cases larger than *1* µm. These values are more than a decade too high. Some new developments in the field of micro-CMMs, as, e.g., the Zeiss F25 already touch the interesting measurement uncertainty class of U = 100 nm and below, but probe system geometries severely constrain the axially reachable surface to some few mm [5]. Specialised form measurement and 1D length measurement instruments can achieve expanded uncertainties below 10 nm. Therefore, PTB has calibrated piston-cylinder assemblies with these instruments for more than two decades.

3.2 Form measurement instruments

For roundness measurements with the lowest achievable measurement uncertainty, a modified RTH Talyrond 73 operated in multi-step error separation mode is utilised. With that instrument, an uncertainty of U = 6 nm can be achieved [6].

For combined roundness, straightness and parallelism measurement a modified MarForm MFU8 (called MFU8PTB) is used [7].

Fig. 2 Reference roundness measuring instrument Talyrond 73. The image shows the set-up for running the multi-step error separation procedure.

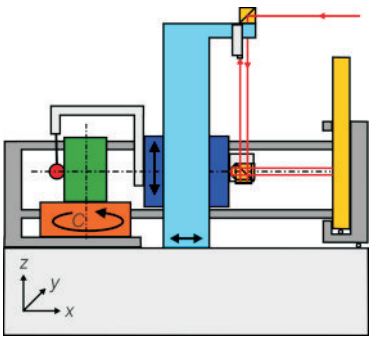

Fig. 3 Functional schematic of PTB's modified MFU8 cylinder form measuring instrument with an additional plane-mirror interferometer.

3.3 Diameter measurement instruments

The MFU8 can also be used for diameter measurements in the U > 50 nm uncertainty range, because PTB's version was extended by a plane-mirror interferometer.

Diameter measurements with the lowest achievable uncertainty can be performed with PTB's reference length comparator KOMF [8]. That instrument is capable of achieving uncertainties of the order of U = 25 nm for cylindrical parts.

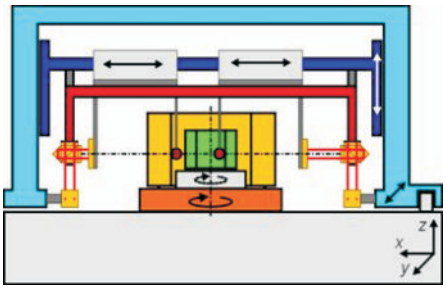

Fig.4 Functional schematic of PTB's reference Abbe comparator "KOMF". It is equipped with dual probe and dual interferometer.

3.4 Multi-purpose measurement machine MFU110WP

For the dimensional measurement tasks of the Boltzmann constant project, PTB has extended its capabilities by a new measurement instrument for form and size, the MFU110WP [9]. That machine is equipped with a high speed rotary table and interchangeable feeler systems. Among these are systems for both size and form measurements, and in addition, special purpose probe systems, as, e.g., the probe system 1320D, which is optimized for diameter measurements. The machine control eliminates most guide error influences on measured profiles by internally subtracting reference data which are gained by capacitive scanning of an internal metrology frame during positioning.

Most cylinder form measurement machines can only measure planar, i.e. roundness or axial straightness profiles of the cylinders [10]. The MFU110WP additionally is able to scan the cylinder helically by moving its rotary table and Z-axis in parallel [11]. That measurement mode is advantageous for the numerical calculation of the effective area of the pressure balances, because it provides the most complete information on the piston and cylinder bore topography. However, the needed measurement time, drift effects, and wear of the contacting element limit the usefulness of such a scanning mode when operated by mechanical contacting.

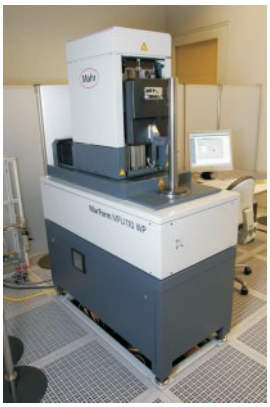

Fig. 5 Form and size measuring instrument MFU110WP at PTB's clean room facility.

To overcome this limit, the MFU110WP features an additional optical feeler system based on a heterodyne white light interferometer, which can acquire data with high speed. This system makes it possible to scan a full cylinder surface with high data density within less than a minute [11]. Of course, such high speeds should be avoided when the stability and noise level of the measurement signal has to be very low as it is in the case of application in the Boltzmann project. But even under these demands, the speed gain of the optical measurement is significant and helps to avoid drift influences which become dominant with longer measurement durations.

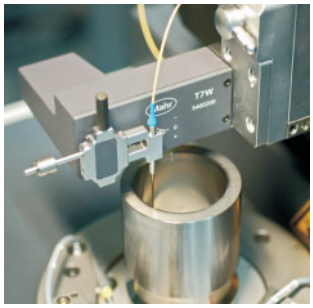

Fig. 6 Optical probe "WhitePoint" measuring the inner surface of a pressure gauge cylinder. The swivel axis is positioned to the 0° position, such that the 90° beam of the probe is enabled.

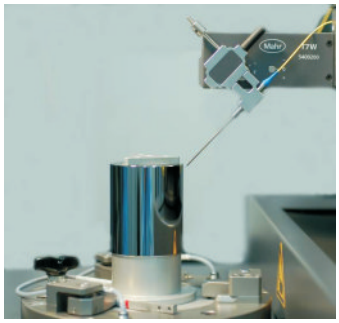

Fig. 7 WhitePoint probe measuring the outer surface of a pressure gauge piston. The swivel axis is positioned to the 45° position to enable the 45° beam of the probe system. This measurement geometry allows the scanning of the artefact with less geometrical constraints than at the 0° swivel axis position and is more stable than the 90° position.

3.5 Measurement set-up and artefact clamping

The precision of the dimensional calibration strongly depends on the reproducibility of the measurement positions. This is especially important when more than one measurement instrument is used for the full calibration procedure. Therefore, special artefact clamping tools based on kinematical mounts were manufactured. They enable an easy and reliable positioning of the artefact to be measured.

The system is based on clamping disks with kinematic mounts (fig 8-9). These ensure a fast and reliable positioning. The kinematic mounts consist of precision rollers in the 120° positions of a circle which are glued to the clamping disks and their counterparts at the rotary tables of the MFU110WP and KOMF which are composed of a pair of balls. A notch-nose pair breaks the 120° symmetry and thus ensures a correct polar positioning.

The MFU110WP rotary table is equipped with a quick clamping mechanism which allows a clamping disk exchange within seconds (see fig 10). In fig. 11, a mounted clamping disk with a piston and a second clamping disk with a cylinder are shown. The second clamping disk was put near to the rotary table to achieve thermal equilibrium between the artefact and the measurement machine.

The temperatures of artefacts during measurements can be measured by Pt100 resistance thermometers that are read out through sliding contacts in the rotary table. These thermometers are not calibrated. Therefore, they are compared to calibrated Pt100 thermometers that can be applied to the artefact during the standstill of the rotary table.

A major uncertainty source in earlier measurement procedures for piston-cylinder assemblies was the restricted reproducibility of the Z-position of the measurement, especially when moving the artefact from one measurement machine to another. The Z-component of the artefact coordinate system is of greater importance for the application, because piston-cylinder assemblies have to be paired for pressure measurement. The effective area has to be calculated for the paired system. Therefore, reference spheres were mounted to the clamping disks coaxially with the artefacts (fig. 12). They can be contacted by both optical and tactile probe systems of different measurement machines and thus may be used for identifying a certain Z-position.

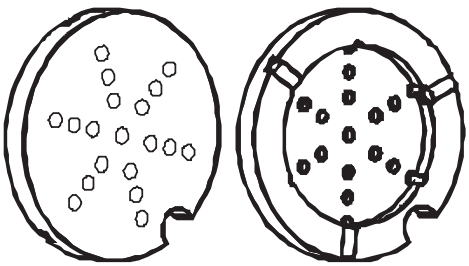

Fig. 8 Schematic drawing of a MFU110WP clamping disk for piston-cylinder assemblies. Left: upper face; Right: lower face

Fig. 9 Lower face of a clamping disk. The three rolls for the kinematic mount are visible at the 120° positions. The notch fits into a nose of the rotary table and fixes the rotational orientation.

Fig. 10 MFU110WP rotary table with double pairs as kinematic bearing counterpart and quick-clamping mechanism for the clamping disks. The cables are connected to Pt100 resistance thermometers.

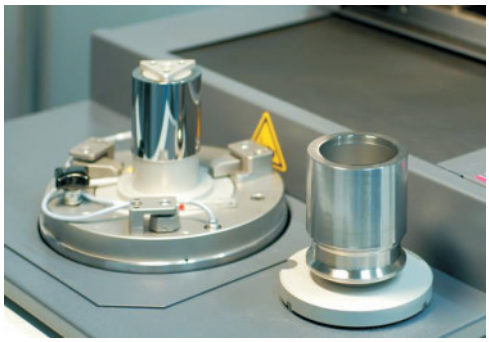

Fig. 11 In the background: Piston with clamping disk mounted to the rotary table of the MFU110WP. In the front: Cylinder mounted to clamping disk in waiting position. In this position, the next artefact to be measured can be left until thermal equilibrium with the machine is achieved.

4. CALIBRATION PROCEDURE PERFORMANCE TESTS

PTB has data records for certain piston-cylinder assemblies at its disposal which go back up to 20 years. During that time some assemblies were measured repeatedly by different machines and measurement procedures, including different error separation techniques. These artefacts are very well-known and consequently were selected to compare the results of the MFU110WP with those of the other machines. In addition, first measurement repeatability checks at the new artefacts were accomplished.

One of the piston-cylinder assemblies (abbr. “PCU”) with long measurement history is a 5 cm^2 unit identified by serial number 6222. It was used to study consistency between results of the new MFU110WP machine and those of other dimensional measurement instruments.

Another assembly measured with the MFU110WP was a 20 cm^2 unit identified by serial number 1162, which is one of the six piston-cylinder assemblies to be used in the experiments for the re-determination of the Boltzmann constant. This unit was very new and was dimensionally studied at PTB for the first time.

Both piston-cylinder assemblies are made of a tungsten carbide material, each have a different design and were manufactured by DH-Budenberg, France (unit 6222) and by DH Instruments, USA (PCU 1162).

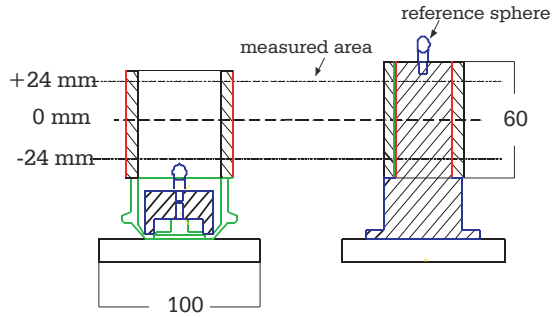

Fig. 12 Schematic drawing of the measured area of the piston and the cylinder. The coordinate system is common to both artefacts. The positions of the Z-coordinate reference spheres are indicated. These spheres are utilised to transfer the axial origin of the coordinate system between different instruments.

4.1 Form measurements

Form measurements of known assemblies were repeatedly performed with the MFU110WP with both tactile and optical probing. The resulting data were analyzed with respect to noise, stability, reproducibility, and comparability to the historic data. It was found that optical contacting leads to the most stable results, at least under the high quality environmental conditions in which the machine is operated. Therefore, only the optical data are used for further discussion.

For comparability to historic data, all straightness profiles were filtered with a Gaussian low-pass filter with a 0.8 mm cut-off length. All roundness profiles were filtered with a low-pass, with a cut-off wave-number of 150 UPR (the filters were implemented as described in

DIN EN ISO 11562 [12]). The form profiles of common piston-cylinder assemblies generally don't carry significant harmonic content for cut-off lengths lower than 2.5 mm or wave-numbers greater than 50 UPR. Therefore, a stronger filtering could be applied without information loss. This might be an option for future data evaluations. The measurement uncertainty of form profiles is tightly connected to the filtering: Stronger filtering means lower uncertainty [13]. Here is a limited source, therefore, for uncertainty improvement.

In fig.13, the straightness reference calibrations of four generatrices of piston PCU 6222 are shown. All straightness deviations amount to approx. 0.1 µm in conjunction with a parallelism deviation less than 0.2 µm. This means that this piston-cylinder assembly is one of the best quality cylindrical artefacts which has ever been measured at PTB. For that artefact, PTB has a measurement history available of nearly 20 years. The data of fig.13 was gained by applying the reversal method [14]. That method is notably time-consuming and took some weeks to be completed. The calibration dated from 2006.

The same generatrices were measured with the MFU110WP system in optical and tactile mode. These data took only some minutes per measurement cycle to be acquired. The corresponding optical data are shown in fig. 14. They are identical to the tactile data. In general, the profiles agree well with the reference. However, near the axial measurement position 10 mm in the MFU110WP profiles, there are apparent local straightness deviations which are not visible in the reference profiles. This is possibly a measurement artefact which stems from a non-perfect calibration of the MFU110WP metrology frame. This effect only slightly affects the straightness and parallelism parameters.

For the purpose of scanning at generatrices, the probe shaft may be positioned vertically, i.e. to the 0° position of the swivel axis (fig. 6). This is the mode of choice for inner cylinder surfaces. This geometry requires shaft lengths that allow complete penetration into the cylinder without touching the upper front face with the probe clamping or swivel axis housing or - when used for pistons - to travel completely down over the piston's generatrix. But there are limits for the shaft length which stem from rigidity and vibration insensitivity requirements.

The MFU110WP swivel axis can be positioned to any inclination angle. Therefore, for piston measurements the required shaft length may be chosen smaller. This is important, because the piston of most piston-cylinder assemblies is much longer than the cylinder.

For the optical probe system, the inclination angle has to match the angle of an optional second laser beam, if available (see fig. 7).

Fig. 13 Reference straightness profiles of the four generatrices 0°, 90°, 180° and 270° of the piston of assembly PCU 6222. The measurement was performed with the MFU8PTB in reversal mode.

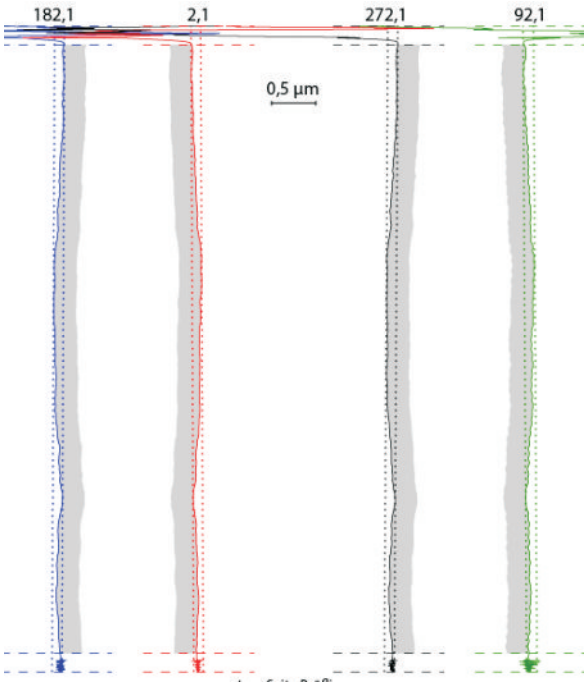

Fig. 14 Straightness profiles of piston PCU 6222 acquired with the optical probe of the MFU110WP.

Figs. 15 and 16 show roundness profiles at various axial measurement positions of the same artefact measured by the MFU8PTB and the MFU110WP in optical mode (short helical integration was applied [11]). The calculated roundness deviations are of the order of 45 nm. It is obvious that the roundness varies only slightly with the axial position. The MFU110WP profiles show much less “waviness” than the MFU8PTB profiles. The apparent waviness is mainly caused by the spindle error of the MFU8PTB. However, in this case the MFU8PTB data are not the reference calibration data. The reference calibration was performed by using a Talyrond 73. The profiles such achieved carry an even lower noise level and because the error separation technique utilised with the Talyrond additionally enables nearly perfect elimination of the spindle error. The resulting roundness deviations amount to 25 nm only (see fig.17). But because the measurement of straightness profiles is much more demanding than roundness measurements and thus is associated with larger measurement uncertainties the resulting uncertainty of the 3D data set of the piston-cylinder assemblies will not be limited by the roundness uncertainty. Furthermore, for the Talyrond the clamping disks cannot be used and the Z-position is uncertain to at least 0.5 mm, because it cannot be positioned CNC-controlled. It seems, therefore, more practical and targeted with respect to the overall measurement uncertainty to use the optical data of the MFU110WP for the calibration of piston-cylinder assemblies.

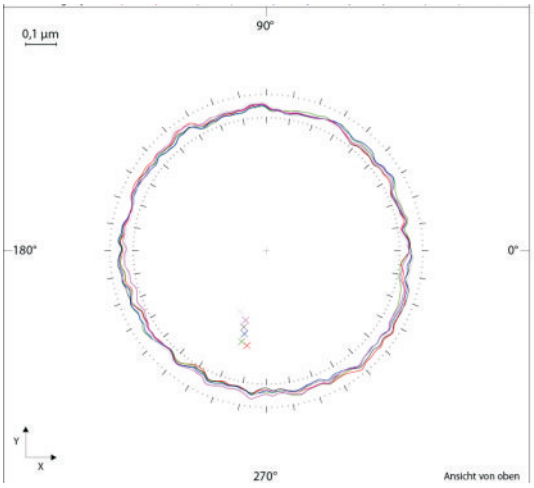

Fig. 15 Superposed roundness profiles measured with the MFU8PTB at different axial positions of the piston PCU 6222. The apparent roundness deviations amount to approx. 60 nm.

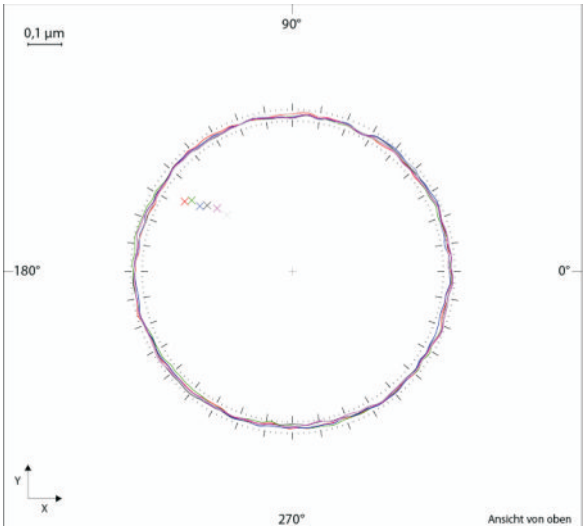

Fig. 16 Superposed roundness profiles measured with the optical probe of the WP110WP at the same axial positions as above. The apparent roundness deviations amount to approx. 45 nm.

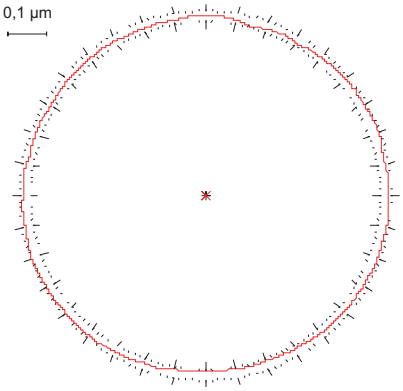

Fig. 17 Reference roundness profile measured with Talyrond 73 of one of the axial positions as measured in fig.16 . The roundness deviation amounts to 25 nm.

In fig.18, a 3D representation of the piston PCU 6222 surface is shown. These data were gained by applying the MFU110WP optical helical scan mode. The data were not filtered, because so far there is no widely accepted or even standardized multi-dimensional filter algorithm available. In principle, data sets like this should be superior to line scans with respect to piston-cylinder calibration, because they carry topographic information about the full cylinder surface. However, so far there is no algorithm to integrate the independently measured diameter information. Therefore, the directly measured full 3D data set can only be dealt with as additional information.

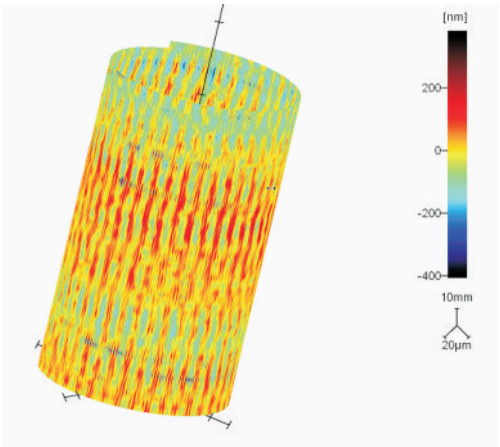

Fig. 18 Helical scan of the full surface of a pressure gauge piston performed with the optical probe system.

4.2 Diameter measurements

For the full calibration of the piston cylinder assemblies, diameter measurements are needed. Unfortunately, at the project start of the Boltzmann project the KOMF was temporarily not available due to technical problems. Therefore, diameter measurements were performed with the MFU110WP by using the diameter probe system 1320D. The KOMF will be used after re-adjustment to verify the MFU110WP data.

In figs. 19 and 20 , the diameter calibration history of the piston-cylinder assembly PCU 6222 (material: tungsten carbide, α = 5*10^{-6} K^{-1}) is compared to the diameter results of the MFU110WP with the probe 1320D. The data stems from a MFU8PTB measurement in 2006, a KOMF measurement in 2006, and a LAKO measurement in 1995. The LAKO was PTB's former diameter reference instrument. The common expanded measurement uncertainties U = 50 nm of the KOMF reference data are marked by error bars. That uncertainty assumption is quite large for the capabilities of the KOMF [15]. However, in 2006, the clamping disks were not yet available and such additional uncertainty contributions were introduced to account for the more difficult measurement position identification. It is assumed that future KOMF measurements at piston-cylinder assemblies can achieve uncertainties in the range of U = 20 nm to 25 nm. Nearly all nominal diameter results agree with the KOMF data. When a measurement uncertainty of U = 60 nm is assumed for the MFU8PTB, of U = 40 nm for the LAKO, and U = 50 nm for the MFU110WP-1320D, all data are compatible. These results also prove the outstanding long-term stability of the piston-cylinder assemblies, which always get in mechanical contact during the pairing process.

Thus the diameter measurement capabilities of the MFU110WP are at least sufficient for first quality checks of the Boltzmann project assemblies and to monitor possible changes during the lifetime of the project. Because the MFU110WP can measure diameters about one decade faster than the reference machine KOMF, this result helps to save time or allows more repeat measurements.

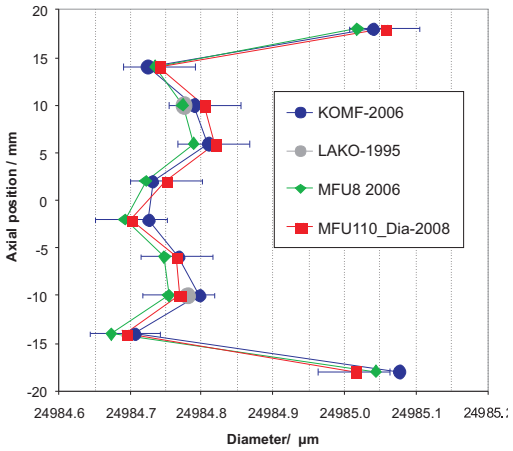

Fig. 19 Cylinder PCU 6222 diameter measurement comparison. The data stems from four different instruments. The instruments and calibration years are specified. The MFU110 data was measured with the diameter probe 1320D. All results agree with the uncertainty U = 50 nm of the reference calibration (KOMF).

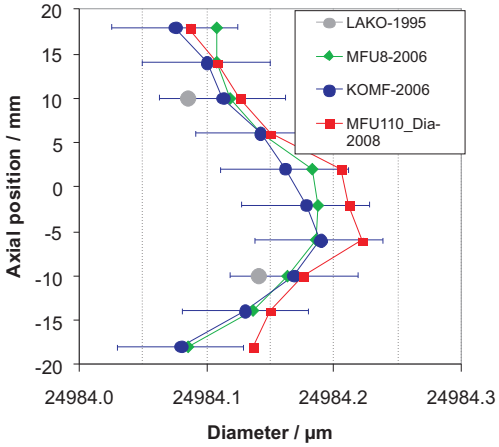

Fig. 20 Piston PCU 6222 diameter measurement comparison. Nearly all nominal results are within the uncertainty U = 50 nm of the reference calibration (KOMF).

5. DATA EVALUATION

To generate 3D data sets describing the topography of the piston and the cylinder bore, straightness deviations (S) and roundness deviations (R) are linked to diameters (D).

The choice of the linking procedure is important because, apart from the uncertainty of S, R and D measurements, the discrepancies between the linked data is a contribution to the uncertainty of the 3D data.

So far [16], only two pairs of diameters, measured in two reference levels and in two orthogonal directions, have been used for a 3D data generation which was performed successively: First, two roundness traces, measured in the reference levels, were linked to the two diameters pairs; then the generatrix traces were positioned in the space to meet the defined two roundness traces; finally, the remaining roundness traces were adjusted to the generatrix traces. To

improve the consistency of 3-dimensional data, a new approach based on the least-squares method has recently been developed [3] which allows the S, R and D data to be linked with each other with only minimum discrepancies between them. When processing the dimensional data, it is possible to weight them, depending on their measurement uncertainties.

After the 3D data sets have been generated, they are transformed to a new coordinate system, in which the z-axis coincides with the axis of the LS cylinder of the 3D data found by the method described in [17]. This step is important to achieve a coaxial positioning of the 3D data sets for the piston and the cylinder bore.

The effective area of the piston-cylinder assemblies is calculated by the Dadson theory [1] which, for gas operated piston-cylinder assemblies, assumes a viscous flow in the piston-cylinder gap, with the gas obeying the ideal gas flow, and leads to the expressions given below:

$$A_0 \quad r_p\ 0\ r_c\ 0 \quad \frac{r_p(0)}{p_1 \quad p_2}^{\ l}_{\ 0} p_z \frac{\mathrm{d}r_p}{\mathrm{d}z} \frac{\mathrm{d}r_c}{\mathrm{d}z} \mathrm{d}z \qquad (1)$$

$$p_z \quad p_1^2 \quad p_2^2 \quad p_1^2 \ {}^{z}_{0} \frac{\mathrm{d}x}{r_c \quad r_p}^3 \Big/ {}^{l}_{0} \frac{\mathrm{d}x}{r_c \quad r_p}^3 \ {}^{0.5} \qquad (2)$$

Here r_p and r_c are the piston and cylinder radii, p_1 and p_2 are the measurement and ambient pressures, p_z is the pressure distribution in the piston-cylinder gap, and l is the length of this gap. Equations (1) and (2) furnish the effective area for one particular pair of the piston and cylinder generatrix line. They are applied for all possible combinations of the piston's and cylinder bore's generatrices, and, finally, the average of all calculated effective areas is taken.

6. RESULTS AND DISCUSSION

First measurements were performed at piston-cylinder assembly PCU 6222 with the MFU110WP included, along with a 3D scan, determination of straightness and roundness, but not diameters. For this reason, to create 3D data sets, the straightness and roundness deviations data were linked to the diameters previously determined with the MFU8 machine.

6.1. 3D link results

The 3D evaluation results of the tactile and optical measurements with the MFU110WP are shown in comparison with the older data obtained with the MFU8 in fig.21 . The graph illustrates the gap geometry of the virtually paired piston-cylinder assembly. The gap has an extent of approximately 200 nm. The new results, both obtained by tactile and optical measurements, are very close to the old data with the exception of the small measurement artefact at the straightness profiles already mentioned in section 4.1.

Also, for assembly PCU 1162 we observed quite good agreement between the tactile and optical data, as well as between the common points of the diameter, straightness, and roundness measurements. The straightness profiles reveal some stronger waviness than in the case of assembly PCU 6222 which evidently reflect real properties of both assemblies (see fig. 22). The gap of this unit is much narrower than that of PCU 6222. It amounts to less than 50 nm in the centre.

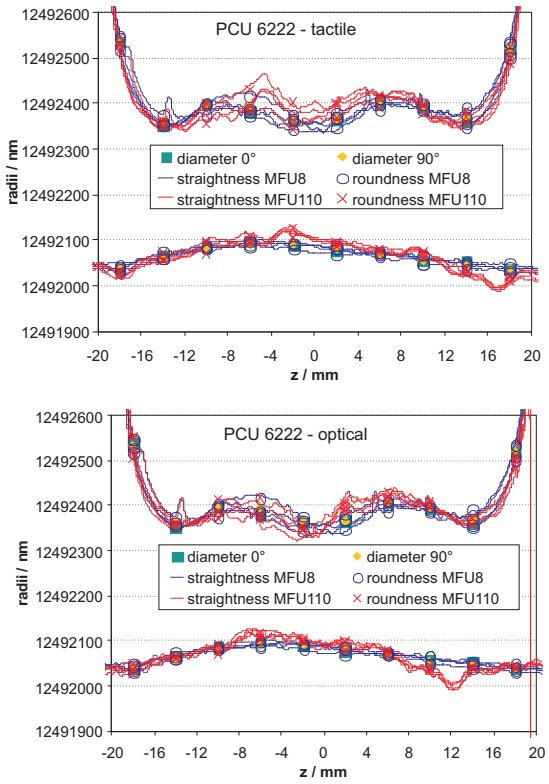

Fig. 21 Gap profile between piston and cylinder of the (virtually) paired assembly PCU 6222 measured with MFU110WP tactile a) and optical b) probe compared with the data obtained with MFU8. The vertical scale divisions amount to 100 nm.

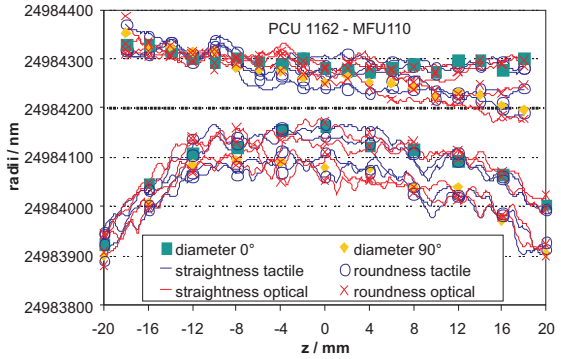

Fig. 22 Gap profile between piston and cylinder of assembly PCU 1162 measured with MFU110WP tactile and optical probe.

6.2. Uncertainty of 3D data

The uncertainty of the 3D radial values generated by a link of diameter, straightness, and roundness data is a combination of their uncertainties with the final discrepancies of the radii of the generatrices and roundness traces as well as the diameters at the common points. At this stage it is not possible to make a solid claim for the uncertainty of the diameter, straightness and roundness measurement performed with MFU110WP. However, as it was discussed in [18] an estimation of the radii uncertainties for generatrix and

circle traces, $u(r_S)$ and $u(r_R)$, can be done using the expressions

$$u\,r_S \quad \left\{ u\,D/2^{\,2} \quad {}^{2}\,r_{D\,S} \quad {}^{2}\,r_{R\,S} \right\}^{0.5} \qquad (3)$$

$$u\,r_R \quad u\,D/2^{\,2} \quad {}^{2}\,r_{D\,R} \quad {}^{2}\,r_{R\,S}^{\;0.5} \qquad (4)$$

where $u(D)$ is the uncertainty of the diameters, and $(r_{D\text{-}S})$, $(r_{D\text{-}R})$ and $(r_{R\text{-}S})$ are the differences between the diameter and the straightness, the diameter and the roundness, and the roundness and the straightness data, respectively.

The uncertainty estimation results are presented in Table 1. Compared with the measurements performed on unit PCU 6222 with MFU8 and Talyrond 73, measurements with the new MFU110WP furnish discrepancies which at the moment are higher by a factor of two to three. This results in the higher uncertainties of the radial values and of the effective areas. The measurements with the tactile and optical probes have approximately the same performance.

Table 1 Summary of the dimensional measurements and evaluation for piston-cylinder assemblies PCU 6222 and PCU 1162: artefact measured – cylinder (c) and piston (p); property (X) – diameter (D), roundness (R) and straightness (S); instrument applied for dimensional measurement; standard measurement uncertainty of property X (u_x); differences between half diameters, R and S radii (Y); standard deviations of these differences [(r_y)]; standard uncertainties calculated for R and S radii [$u(r_x)$]; their contributions to the effective area uncertainties [$u_{r,R}(A_0)/A_0$] and [$u_{r,S}(A_0)/A_0$]; the effective areas (A_0) and their combined standard uncertainties determined from the dimensional data [$u(A_0)/A_0$].

Unit	Artefact	X	Instrument	u_x in nm	Y	(r_Y) in nm	$u(r_X)$ in nm	$u_{r,R}(A_0)/A_0$, $u_{r,S}(A_0)/A_0$, in 10^{-6}	A_0 in cm^2	$u(A_0)/A_0$ in 10^{-6}
6222	Cylinder	D	MFU8	25	D-R	4				
		R	Talyrond 73	5	D-S	4	14	2.3		
		S	MFU8	40	R-S	5	14		4.9026390	2.5
	Piston	D	MFU8	25	D-R	5				
		R	Talyrond 73	5	D-S	3	14	2.2		
		S	MFU8	40	R-S	5	14			
	Cylinder	D	MFU8	25	D-R	11				
		R	MFU110WP, tactile		D-S	15	19	3.2		
		S	MFU110WP, tactile		R-S	10	22		4.9026404	3.8
	Piston	D	MFU8	25	D-R	14				
		R	MFU110WP, tactile		D-S	19	20	3.7		
		S	MFU110WP, tactile		R-S	8	24			
	Cylinder	D	MFU8	25	D-R	14				
		R	MFU110WP, optical		D-S	21	22	3.1		
		S	MFU110WP, optical		R-S	10	26		4.9026407	3.7
	Piston	D	MFU8	25	D-R	10				
		R	MFU110WP, optical		D-S	14	17	3.6		
		S	MFU110WP, optical		R-S	6	19			
1162	Cylinder	D	MFU110WP, tactile	40	D-R	11				
		R	MFU110WP, tactile		D-S	13	25	2.0		
		S	MFU110WP, tactile		R-S	9	25		19.610121	2.4
	Piston	D	MFU110WP, tactile	40	D-R	13				
		R	MFU110WP, tactile		D-S	15	25	2.1		
		S	MFU110WP, tactile		R-S	8	26			
	Cylinder	D	MFU110WP, tactile	40	D-R	11				
		R	MFU110WP, optical		D-S	16	25	2.1		
		S	MFU110WP, optical		R-S	11	28		19.610118	2.5
	Piston	D	MFU110WP, tactile	40	D-R	14				
		R	MFU110WP, optical		D-S	15	26	2.2		
		S	MFU110WP, optical		**R-S**	**8**	**26**			

For the diameters of assembly PCU 1162 measured with MFU110WP, a standard uncertainty of 40 nm was estimated which will have to be verified in the future. The discrepancies and the combined uncertainties of the radial values are very similar to those for assembly PCU 6222.

6.3. Effective area

The effective areas calculated using equations (1) and (2) for different dimensional data sets and the associated combined standard uncertainties are presented in the two last columns of Table 1. The following uncertainty contributions were taken into account: uncertainty of the 3D data sets, standard deviation of the effective area values calculated for different combinations of the piston and cylinder generatrix lines, and the change of the effective area with pressure.

For assembly PCU 6222, the relative difference between the effective areas based on dimensional data measured with the MFU110WP tactile and optical probes is equal to only $6 \cdot 10^{-8}$. The relative difference between the older and the new effective areas is equal to $2.8 \cdot 10^{-7}$. Such a good agreement is not really surprising because the new shape deviations measured with MFU110WP were linked to the diameters measured with MFU8, the same diameters which were used for the calculation of the old effective area. However, the uncertainty of the effective area based on the MFU110WP data is larger than that of the old effective area because of larger discrepancies between the straightness and roundness data. The latter are evidently caused by the not yet optimal performance of the new instrument in straightness measurement mode, because the roundness measurements, as stated in section 4.1, appear to be very good.

For assembly PCU 1162, the relative difference between the effective areas based on dimensional data measured with the MFU110WP tactile and optical probes is equal to only $1.5 \cdot 10^{-7}$. In spite of the higher uncertainty of the diameter measurements carried out at unit PCU 1162 with MFU110WP than the uncertainty of MFU8 used for measuring diameters of unit PCU 6222, the relative uncertainty of the effective area of the first piston-cylinder assembly is smaller, which is explained by the fact that its nominal effective areas are four times as large as that of unit PCU 6222.

Even though these very first measurements with the new instrument MFU110WP have resulted in the effective area's relative standard uncertainty of $2.5 \cdot 10^{-6}$, the target uncertainty of $1 \cdot 10^{-6}$ appears to be achievable when the MFU110WP performance in straightness measurements is improved and, additionally, the diameters are measured using the reference length comparator KOMF.

6. CONCLUSION

A procedure for the dimensional calibration of piston-cylinder type pressure standards has been described. It was shown that the new instrument MFU110WP significantly improves the efficiency of the procedure. With respect to the quality of the measurement and the achievable uncertainty, the following may be concluded:

- The determined effective areas based on tactile and optical measurements with MFU110WP are equivalent. This justifies application of the optical technique for shape deviation measurements.
- Roundness measurements are sufficiently good.
- Straightness measurements require improvement (characterization/improvement of the internal reference frame, application of the reversal technique, …).
- Diameter measurements with the KOMF are necessary, they will also allow a conclusion about the diameter measurement capability of MFU110WP.
- After having reached the appropriate performance of MFU110WP in separate roundness and straightness measurements, the instrument's top helical scan capability can be used to get extended information about the topography of the piston and the cylinder bore.

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ENABLING STANDARDS FOR NANOMATERIAL CHARACTERIZATION

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ABSTRACT

A two-day international workshop was convened recently in order to scope out and address the urgent need for standards to accurately characterize the physico-chemical and biological properties of engineered nanomaterials. These standards are needed by industry and regulatory bodies in order to meet requirements for the production, application and lifecycle risk management of nanomaterial-based products ranging from cancer therapeutics to high-tech coatings and composites. The current deficiency in the availability of such standards, including both documentary and reference artifacts, is perceived as limiting the widespread adoption and implementation of nanoscale technologies. Herein is given a brief summary of that workshop, its findings and recommendations.

INTRODUCTION

Sufficient and accurate characterization of the physical and chemical properties of engineered nanomaterials (ENMs) and their interactions with biological systems are fundamental requirements for successful material or device design. Similarly, proper characterization of material properties is a critical component for understanding and managing the lifecycle risks posed by ENMs to the environment, health and safety (EHS). Formidable obstacles exist to obtaining adequate characterization. Foremost among these obstacles are those presented by the lack of relevant reference materials and standardized practices, protocols, and procedures for test specimen / sample preparation, measurement, and data analysis.

In order to accelerate research, development, risk identification, regulation, and widespread adoption of nanotechnology, there exists an urgent need to develop new protocols, practices, and reference materials (RMs), and to elevate these to the status of internationally accepted standards. With this in mind, a workshop was held October 8-9, 2008 on the campus of the National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland. The overarching goals of the workshop were to:

1. Stimulate interdisciplinary discourse regarding ENM characterization needs and issues
2. Promote cooperation among government agencies and international stakeholders
3. Accelerate development and validation of protocols, standards and RMs needed to underpin the safe and widespread use of ENMs

The workshop was organized around an agenda that included formal presentations, facilitated discussions, demonstration of a proposed new electronic medium for pre-standard development, and four parallel focused break-out sessions. As a starting point for discussions of measurement issues associated with ENM characterization, and to lay the foundation for future work needed to validate measurement protocols and provide precision statements for consensus standards, preliminary findings were summarized for three concurrently executed interlaboratory studies (ILSs) sponsored by the ASTM Interlaboratory Study Program and organized through E56, the committee on Nanotechnology. These studies involved roughly 35 participants and demonstrated both the benefits and the difficulties associated with the undertaking of such efforts.

KEY STAKEHOLDERS

Workshop participants, numbering just over 100, were drawn from government, academia and industry, and represented US and international interests including several national metrology institutes as well as international standards development organizations. Table 1 gives an organizational breakdown of the participation. Among the sponsors and contributors to this workshop were a number of major stakeholders from within the U.S. federal government, including the Food and Drug Administration (FDA), the Environmental Protection Agency (EPA), the National Cancer Institute (NCI), the National Institute for Occupational Safety and Health (NIOSH), the National Institute of Environmental Health Sciences (NIEHS), the Nanotechnology Characterization Laboratory (NCL) at NCI-Frederick, the National Nanotechnology Coordination Office (NNCO), and NIST. The Oregon Nanoscience and Microtechnologies Institute (ONAMI), a large academic-based consortium in the U.S., and ASTM International were non-governmental sponsors. The audience was highly interdisciplinary and included a number of leading scientists working in the nanobiotechnology arena, but also numbered among the audience were standards developers, regulators and policy makers. Thus many different perspectives were represented, providing for robust and lively discussion and meaningful consensus.

OVERARCHING ISSUES

Nanotechnology is enabling new therapies for fighting cancer, with ClinicalTrials.gov currently listing 59 clinical trials for nanoparticle-based cancer therapies. However, nanoparticles frequently cause false positive and false negative results in currently used in vitro assays and they present novel challenges for characterization. Piotr Grodzinski, Director of the NCI Alliance for Nanotechnology in Cancer, discussed the need for, and NCI's efforts to establish, a framework for the clinical translation of cancer nanotechnologies. A key component of this framework is standardization of analytical protocols for physical, in vitro and in vivo testing of ENM-based diagnostic, imaging and therapeutic platforms.

Potential exposure to free ENMs is likely greatest for those who manufacture and process these materials. Aleksandr Stefaniak of NIOSH stressed that current methods and protocols for risk assessment of ENMs in an occupational exposure scenario are not well established; biologically relevant metrics, suitable RMs, validated exposure assessment tools, and validated protocols are needed in order to make progress in addressing occupational exposure risk assessment. A few guides now exist that address worker health protection, including contributions from BSI British Standards, NIOSH and ASTM International. Standards for data management are also lacking; for example, a national exposure database for nanotechnology workers and/or medical registry would require a standardized framework for materials identification and data input.

Reference material development involves a balance of four factors, as described by Steve Hankin of the Institute of Occupational Medicine: 1) needs of the communities, 2) choice and relevance of physicochemical properties, 3) availability and suitability of materials, and 4) characterization ability. There is a need for the community to define questions that RMs might help solve; these are thematically stated by the UK Nanotechnology Research Coordination Group (NRCG) and US National Nanotechnology Initiative (NNI), but there is currently no consensus on specifics. REFNANO 2007, Organization for Economic Cooperation and Development (OECD) 2008 and NanoImpactNet 2009 have or will nominate and prioritize candidate materials (e.g., TiO_2, gold, silver, polystyrene, carbon nanotubes are common suggestions).

The field of nanotoxicology will require advancements in physical measurement capabilities, including the development of metrics for surface properties, capability to characterize and detect nanoparticles across a variety of biological and environmental media, characterization of agglomeration and aggregation, capability to differentiate the shape and aspect-ratios of particles in mixtures, and discrimination between engineered and background nanoparticles. These advancements will need to be supported by documentary standards that define their implementation across labs.

Participants stressed that *verifiable science* is needed for the assessment of ENM hazards, since there are significant potential implications for economic development and human health. It's critical to accurately assess ENM hazards using reproducible and validated standards and protocols. There are also unique issues for ENM hazard assessment that must be taken into consideration: e.g., small differences in nanoparticle size can dramatically influence results. Reproducibility in biological testing of ENMs is possible but challenging; it demands careful control of all parameters, standardized protocols, well-characterized materials, and careful laboratory practice. The International Alliance for NanoEHS Harmonization (IANH), chaired by Ken Dawson of University College Dublin (Ireland) will conduct round-robin studies to validate existing toxicity assessment strategies and thereby improve reproducibility and overall confidence in reported results.

An industry perspective on nanotechnology standards and needs was provided by representatives from Intel Corporation, Eli Lilly and Company, and Thermo Fisher Scientific. It was emphasized that knowledge is critical to enable effective life-cycle risk assessment and management, and this requires an understanding of the principles of nanoparticle-biological interactions and nanotoxicology. The need for well-characterized high purity ENMs and RMs for reliable toxicological and physical characterization tests was stressed, as was the need for biological testing protocols that deliver reproducible results and standardized biological media for assays. Without these tools in place, uncertainty with respect to potential hazards and liabilities limits commercial development and deployment of ENM products.

Key research needs include: a standardized testing strategy for assessing toxicity, determination of the best metrics for assessing particle toxicity, exposure monitoring methodologies, and risk assessment methodologies. In terms of dose metrics, mass is the most commonly used, but size, surface characteristics, etc. may be more biologically relevant. There is currently wide variability in reports of toxicology for the “same” materials, and a wide variability in the types of characterization and properties of the ENMs used in reported studies. Most importantly, it has not yet been established that current testing protocols correlate with acute toxicity and/or chronic effects.

Reference materials are also needed for instrument qualification and quality control in a manufacturing environment, where size characterization is of critical importance for drug development since nanoparticles are being engineered to perform pharmaceutical functions that depend on their size. Standards producers must be reputable, provide complete and clear supporting documentation, and use techniques/methods that are accepted by the industrial community. Both physical and documentary standards have finite lifetimes based on their applicability, and the lifetimes of physical standards are also limited by their shelf-life/stability. These issues may be particularly

relevant for the fast evolving field of nanotechnology, and for many ENMs that are unstable or reactive.

In the U.S., medical devices are classified and regulated according to the degree of risk to the public (Classes I-III, with III requiring the most stringent regulation). Use of FDA-recognized standards improves the quality of 510(k) applications and facilitates the regulatory process. Challenges remain for regulating nano-enabled medical devices, including development of standards for identification and assessment of ENMs in products and for biocompatibility and toxicity assessment. The FDA is specifically interested in ways of determining if a product contains nanoparticles, and if the inclusion of ENM changes the product classification. ISO technical committee 229 on Nanotechnologies is now working on establishing methods for determining nanoparticle release from ENMs (e.g. by abrasion and erosion).

The emerging role of academic institutions in nanotechnology standards development was discussed. According to Stacey Harper of ONAMI, academics have a different perspective on standards development since they can investigate libraries of ENMs that may not be of immediate interest to industry or suitable for certification as RMs. This perspective can be utilized, though, in an integrative approach in collaboration with industry and government partners to further the cause of standards development.

THE ROLE OF INTERLABORATORY STUDIES

ASTM International is a consensus standards development organization that operates on an individual participatory model. Their Interlaboratory Study Program was formed in 2005 with an initial investment of $4 million USD to provide support for organizing studies to develop precision and bias statements for ASTM standards. ASTM E56 committee on Nanotechnology has approved seven standards thus far; three of which have been associated with interlaboratory studies discussed at this workshop: E2490-08 Measurement of Particle Size Distribution of Nanomaterials in Suspension by Photon Correlation Spectroscopy, EE2526-08 Standard Test Method for Evaluation of Cytotoxicity of Nanoparticulate Materials in Porcine Kidney Cells and Human Hepatocarcinoma Cells, and E2524-08 Standard Test Method for Analysis of Hemolytic Properties of Nanoparticles.

The interlaboratory studies were conducted through the E56.02 subcommittee on Characterization: Physical, Chemical, and Toxicological Properties. The studies utilized three NIST gold nanoparticle RMs, and cationic and neutral dendrimeric nanoparticles. A total of 26 laboratories participated in ILS166, which tested widely used nanoparticle size measurement techniques including photon correlation spectroscopy (PCS; also known as dynamic light scattering, DLS), atomic force microscopy (AFM), transmission electronic microscopy (TEM) and scanning electron microscopy (SEM), and produced data necessary to develop a precision statement for E2490-08. The preliminary results for PCS were generally very consistent (see for example Table 2), but with some clear outliers. Not surprisingly, the smaller test materials (10 nm or less) exhibited the greatest variance. There were differences in the interpretation of micrographs between laboratories, especially with regard to identification of individual particles. Plans include a post-study analysis of the imaging data to assess the impact of different image analysis algorithms. The precision statement for E2490 was recently approved by committee ballot.

The two biological studies included nine participants in ILS201 (hemolysis) and six in ILS202 (cytotoxicity), and demonstrated the challenge of obtaining reproducible in vitro biological test results for ENMs across different laboratories.

The preliminary results for ILS201 showed that the protocol was successful in all participating laboratories, and that obtaining whole blood from commercial sources in a timely manner and in sufficient quality is achievable. Only one out of nine participating labs reported a problem with the quality of the whole blood, where the plasma free hemoglobin (PFH) value is out of specification. The overall assay performance, as judged by precision and accuracy of the standard curve and quality controls, was also successful. But determination of the hemolysis caused by nanomaterials was complicated by the fact that several participating laboratories did not follow the protocol as written. ILS202 was conducted for measurement of nanoparticle cytotoxicty to human hepatocarcinoma cells and porcine renal proximal tubular cells by two methods: MTT reduction and LDH enzyme leakage. Only the MTT data was presented at this meeting. Only one toxic nanoparticle (the cationic dendrimer) was included in the study, but only two labs saw enough toxic response to estimate an IC50 (50% inhibitory concentration) for this particle.

Valuable lessons were learned from these exercises: (1) a protocol training step should be included before the formal blinded validation is attempted, (2) RM nanoparticles with low-dose toxicity are needed to evaluate cytotoxicity assays, (3) complexity of biological protocols requires additional guides for users to follow, such as a schematic or video training, (4) true blinded study is not feasible for nanomaterials – knowledge of the sample material is an important component of sample preparation (e.g. adjusting sample pH to physiological range), and this is the step with which most labs participating in ILS201 had difficulties. and (5) access to well-characterized common test materials and RMs are critical for such studies. Plans to repeat these studies in the future are currently under consideration.

Additionally, a number of important issues were raised with respect to interlaboratory testing and protocol development. For instance, there are many sources of variability in cell-based assays, and these should be explored more quantitatively in order to address overall inconsistencies and to better define the limits of such assays. Interference caused by ENMs, especially in colorimetric-based assays, is a common problem that impacts the variability and accuracy of these tests.

Since the principal purpose of interlaboratory studies is to identify assays that are reproducible and to validate them in multiple laboratories with documentation of the results, it was concluded that an informal testing scenario with fewer participating laboratories may be needed prior to formal testing in order to screen for potential problems. Such preliminary round robins would help ensure success and efficiency in larger scale formal testing.

Finally, the principal contributing factor for variability in interlaboratory testing and protocol validation involving ENMs is most likely sample preparation (i.e., introduction of ENMs into the test medium). There is a general lack of validated dispersion protocols at the present time, and a poor understanding of the dispersion process and its impact on the biological interactions of ENMs. Controlling the effective concentration and dose is critical to obtaining accurate and reproducible assay results that are meaningful with respect to risk assessment.

INTERAGENCY AND INTERNATIONAL COOPERATION

The need for cooperation and coordination between U.S. federal agencies with a stake in nanotechnology standards was a unifying theme for this workshop. Clayton Teague, Director of the NNCO, pointed out that scientific standards development is a “tragedy of the commons” in that standards will benefit everyone, but it is in no one's rational best interest to invest in their development directly. Furthermore, there is a limited pool of qualified and willing participants to work on nanotechnology standards development worldwide, and this resource must be used efficiently for any progress to be sustained. It is equally important that nanotechnology standards are based on solid science and engineering. Standards not so founded can constrain innovation and entrench inferior technologies (e.g. cell phone networks in the U.S.), and this requires cooperation between stakeholders. NNCO is interested in supporting the formation of a community of interest to facilitate standards development at the pre-standard level and urges the SDOs to collaborate with each other and with individuals and agencies engaged in standards development.

Other current interagency efforts directed toward aligning collaborative resources to address nanotechnology health and standards issues were described. The NanoHealth and Safety Initiative at NIEHS is interested in understanding the interaction of engineered nanoscale materials with biological systems. The National Toxicology Program (NTP), which is part of NIEHS, is focused on studying nanoparticle toxicology through a formal nomination process that includes other agencies such as FDA. NIST and NCI have signed a memorandum of understanding to collaborate on measurements and standards related to cancer nanotechnologies. This collaboration became fully operational in 2006. The basic concept behind this collaboration was to leverage NIST's expertise in physical science to address issues related to the characterization of ENMs for cancer therapy and detection. The effort has focused on three areas: (1) measurement science and method development related to the physicochemical properties of ENMs, (2) critical data on nanoscale platforms submitted to NCL, and (3) development of RMs and standards. NIST and NCL have developed assay protocols for the physicochemical characterization of ENMs; these protocols will be available on the NCL website: http://ncl.cancer.gov.

The OECD sponsorship program for the testing of manufactured nanomaterials is an example of one mechanism that serves both interagency and international cooperation. In this program there must be agreement on which properties to measure and how to measure them, characterization implications of sharing test samples among sponsors, and consistent reporting of test results. The OECD program is designed to provide the information needs for decision makers regarding the environmental, health and safety assessment of ENMs, by coordinating many international efforts organized around 14 specific nanomaterials. It provides a framework for interaction and improved communication among researchers working on the environmental and health implications of ENMs.

The Asia-Pacific Economic Cooperation (APEC) nano project was initiated in 2005 and developed a roadmap for the characterization of ENMs, including thin films, latex spheres, nano silver, and carbon-based nanoparticles. The analysis of the preliminary interlaboratory comparison of nanoparticle size measurement took place in 2005; this interlaboratory study had ten participants and used TEM, AFM and dynamic light scattering (DLS) to measure particle size. In 2006, they revised the testing instructions and re-attempted the study, this time with 20 participants and using DLS, AFM, TEM and SEM. In 2007-2008, APEC concentrated on the measurement of thin film thickness by SE, SIMS, TEM, XF, XPS, and XRR.

COMMUNITY-DRIVEN SOLUTIONS

The benefits of using electronic and community-driven approaches for acceleration of pre-standard protocol development and management of interlaboratory studies was explored in some detail. Currently, there are sufficient electronic means for formal standards development, document creation and voting (through the SDOs); however the resolution of negative votes is tedious and rate limiting. There is also a need for greater transparency, and to accelerate the standards development process to comply with the urgent demand for standards in nanotechnology applications. Additionally, this process would benefit by broader inclusion of technical experts who are outside the SDO committee structure.

A collaborative website (Web 2.0) could be used to record comments, dissent, and resolution, to link to authoritative documents, and to organize and assist technical discussion and protocol validation. A prototype wiki was demonstrated by Raul Cachau of the Advanced Biomedical Computing Center at NCI Frederick. A wiki is

a software implementation of a social protocol; the important parameters are the policies and guidelines (how access is controlled, etc.). Points that need to be considered are access, traceability (IP tracking), data curatorship, ease of use, user requirements, data archiving, security (SSL), and notification (email, RSS). The prototype wiki is proposed to foster collaborative development and testing of protocols prior to entry into the formal standards development pathway, discussions of standards issues, identification of ENMs for validation of protocols including controls, and formation of a community of interest. SDOs such as ISO and ASTM could link to the wiki to complete the process of balloting and adopting international standards, once the protocols have been fully vetted and validated. The proposed wiki would also be used to support a community of interest for nanotechnology standards development and would be community-driven in content and focus. Public availability of the standards wiki is planned for the first half of 2009.

RECOMMENDATIONS AND CONCLUSIONS

A vital element in the development of an effective and validated protocol (or standard practice) is the conductance of an interlaboratory study, commonly referred to as round robin testing. These studies are necessary to provide estimates of measurement precision for a method, and to ensure that test procedures generate accurate and meaningful results while avoiding potential artifacts. One conclusion of the workshop was that consistent measurement results are difficult to achieve even under the best of conditions, and only the greatest care produces valid data. This was made particularly evident during the presentation of findings from three parallel ASTM E56.02 sponsored studies.

It was also clear from these studies that biological protocols are subject to much greater uncertainty relative to physicochemical measurements. Physical scientists participating in the workshop were surprised to hear that 20 % or greater variability in biological assay results is considered acceptable in most cases. Operator bias also seems to be a more significant factor for biological testing, probably due in part to the complexity and serial nature of many biological assays. Another important factor is the dependence of these tests on biological components that are inherently difficult to control and subject to adaptation and change over time.

The primary recommendation from the workshop participants was to foster a community guided effort for the exploration of laboratory best practices and the harmonization of preliminary stage development of standards for ENM characterization. The prototype wiki is proposed as the primary mechanism for community interaction in three areas:

- development of standard physico-chemical, in vitro and in vivo assay protocols
- collaboration in interlaboratory tests to determine the reproducibility and repeatability of assays
- development of study materials and RMs required for those protocols and for instrument calibration/qualification

Under the recommended plan, letters of intent would be solicited from U.S. and foreign agencies and institutions wishing to participate in the collaboration. Resources for operating the wiki would be solicited by the NNCO from the Nanoscale Science, Engineering and Technology (NSET) subcommittee based on those letters of intent. NSET is the interagency body responsible for coordination of the NNI. Because of NCI's efforts in developing the pilot wiki, it was requested that NCI initially host the wiki to allow rapid development of draft wiki pages, rules of governance, and solicitation of the letters of intent.

ACKNOWLEDGEMENTS

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Table 1. Organizational listing of workshop participants.

Governmental Organizations	Non-Governmental Organizations	Academic Institutions
U.S. Air Force Research Laboratory	Advanced Surface Microscopy Inc.	Cambridge University (UK)
U.S. Army Edgewood Chemical Biological Center	ASTM International	Central Michigan University
U.S. Department Of Homeland Security	Beckman Coulter Inc.	Emory University
U.S. Environmental Protection Agency	Bristol-Myers Squibb	Georgetown University
European Commission Joint Research Centre	The Dow Chemical Company	Industrial Technology Research Institute (Taiwan)
Federal Institute for Materials Research and Testing (Germany)	Dupont	North Carolina State University
U.S. Food and Drug Administration	Eli Lilly & Company	Oregon Nanoscience and Microtechnologies Institute
U.S. National Cancer Institute	Evonik Degussa Corporation	University College Dublin (Ireland)
U.S. National Institute for Occupational Safety and Health	FEI Corporation	University of California at Irvine
U.S. National Institute of Biomedical Imaging and Bioengineering	Horiba Instruments Inc.	University of Delhi (India)
U.S. National Institute of Environmental Health Sciences	Institute of Occupational Medicine (UK)	University of Massachusetts Amherst
National Institute of Advanced Industrial Science and Technology (Japan)	Intel Corporation	University of Michigan
U.S. National Institute of Standards and Technology	ISO TC229, TC24	University of Technology Dresden (Germany)
National Measurement Institute (Australia)	Luna Innovations	University of Texas Health Science Center at Houston
U.S. National Nanotechnology Coordination Office	Malvern Instruments Inc.	
National Physical Laboratory (UK)	Micromeritics Instrument Corporation	
National Research Council Canada	Microtrac, Inc.	
Oak Ridge National Laboratory	Midatech Biogune S.L. (Spain)	
	MVA Scientific Consultants	
	Pennsylvania Bio Nano Systems, LLC	
	SAIC Frederick Inc.	
	Thermo Fisher Scientific	
	Unidym Inc.	
	United States Pharmacopeia	

Table 2. Preliminary comparison of results for ILS166, showing mean values for Test Sample B (nominal 30 nm gold).

Measurement	Mean Size and Expanded Uncertainties (nm)			
	NIST Ref. Value	NIST *U**	Mean ILS166	ILS166 *U*[§]
AFM	24.9	1.1	25.1	2.4
SEM	26.9	0.1	28.4	8.0
TEM	27.6	2.1	27.3	4.6
PCS[¶]	28.6 (173º) 26.5 (90º)	0.9 3.6	29.4	7.0

[¶] NIST reference values[8] for PCS were obtained by a single operator on two instruments operating at two different scattering angles. PCS results for ILS166 represent multiple measurement configurations, instruments and operators.
* Expanded uncertainties, U, are calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO and NIST Guides[9]. The coverage factor, k, for 95 % expanded uncertainty intervals is based on a t multiplier with the appropriate associated degrees of freedom.
[§] Expanded uncertainties are calculated as $U = ks$, where s is the standard deviation of the mean. A coverage factor, k=2, is used to approximate a 95 % expanded uncertainty interval.

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HARDNESS MEASUREMENT IN BRAZIL IN THE NANOTECHNOLOGY ERA

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Abstract
The measurements of conventional indentation hardness (Brinell, Rockwell, Vickers and Knoop) are known and their methods were normalized more than 50 years ago. Their results are numbers that represent the size of the residual impressions on the sample surface after the indentation. Face to scientific and technological demands, noticed in the superficial engineering and more recently in nanotechnology field, different methods were developed for the characterization of hardness and other mechanical material properties in micro and nanometer levels. The method known as "Measurement of Hardness and Materials Parameters for Instrumented Indentation Test" is based on the use of diamond or hard metal indenters to indent the sample surface, with forces and indentation depths measured and presented simultaneously through a graphic. With this technique, is possible to characterize the plastic and elastic behavior of a material in nanometer regions (< 200nm), where it has been frequently the only one which it makes possible the research of nanostructural material, thin film, ceramic material, etc., independently from hardness. This article has the objective to show the state of the art of nanoindentation tests and the today activities of some Brazilian research groups in the characterization of the hardness values of the materials in micro and nanometer level.

Key words: Instrumented indentation, Microindentation, Nanoindentation.

1. HISTORY OF THE HARDNESS MEASUREMENT

Well before the quantitative and reproducible results of hardness be available, philosophers and scientists of the XVII century already discussed about this subject. In that time, the investigations contemplated speculations on the nature of its meaning only (Wilde et al., 2000). In fact, the first methods of hardness measurement studied, just as the scratch method, was convenient and simple, however they involved many variables to provide a meaning to the hardness scientific definition.

Hardness had indication as resistance to the indentation or permanent deformation in the beginning of the XVIII century. In 1722, Reaumur developed a scale for test in metals regards the scratch test with the use of an anvil cleaver as indentation tool in a bar, whose hardness increased from one side to the other. The great importance of this method was the fact that Reaumur was the first to investigate a measurement by indentation. In 1859, Calvert and Johnson showed the first results obtained by a hardness machine considering the force requested to produce an indentation depth of 3,5mm. This depth was measured through a scale equipped with a vernier. The force requested to indent the 3,5mm was called hardness (Wilde et al., 2000).

Based on the criterion for analysis of hardness established by Heinrich Hertz in 1881, where he postulated about the nature of the tension field due to the contact between two elastic bodies, the physical concept of hardness has having different meanings for the different professionals that use this property. The divergent concept depends on the experience of each one when studying the subject. In general, for a metallurgist, hardness means the resistance to the permanent plastic deformation. For a mechanical engineer, hardness is defined as the resistance to the indentation of a hard material in another. For a planner, it is considered a measure base for the knowledge of the mechanical resistance obtained in the thermal or mechanical treatment of a material and of its mechanical resistance to the wear. For a metal machining technician, it provides a measurement of the cutting resistance and, for the mineralogist, it means the measured of the scratch resistance that a material can do in another one (METALS HANDBOOK, 1995).

1.2. History of Conventional Hardness Method

There are several methods that can aid the understanding of hardness measurement, the three more used and mentioned method in the literature are:

- Indentation resistance (Brinell/Sweden, 1900; Rockwell/USA, 1920; Vickers/United Kingdom, 1925 and Knoop/USA, 1939).
- Shock resistance or energy absorption from dynamic application forces (Shore/USA, 1895).
- Scratch resistance (Mohs/Austria, 1822).

The indentation resistance is the one that represent the major applicability in the engineering research as well as in the process of the industry product.

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The technology of indentation hardness emerged as a simple and economic alternative related to the tensile test, as a way to determine for other manner the value of the mechanical resistance of the materials. The Brinell hardness method, created in 1900 for Johan August Brinell (Sweden), appears to attend that need (Dieter, 1988).

The company Wilson Co. (USA) introduced the Rockwell hardness method in 1920. This method objectified optimizer some limits of the Brinell method and, above all, to decrease the time of testing in order to obtain advantages of practical order, turning it, until today, the method more used by the industry (Low, 2001).

The Vickers method, introduced in 1925 by the Vickers Industry Ltd. (England), is strictly related to the Brinell method and it is generally used to measure high hardness when the Brinell method is not applicable.

2. PROPOSAL OF NEW METHOD

The proposal of a new method for hardness and other material property has to offer advances in relation with the other current hardness methods. In comparison to the conventional methods, the new one has to (Polzin, 1997):

- be cheaper, automatic and/or quicker.
- have smaller uncertainties.
- bring additional knowledge.
- be a unique method for the purpose.
- give values which can characterize special materials.
- have a standard or a standardization method established.

As described previously, the methods mostly used for metallic materials are Brinell, Rockwell and Vickers. For Brinell and Vickers, the relevant hardness value is determined after the total test force removal. For Rockwell hardness, the value is determined through the measurement of the indentation depth after the additional force test removal and can be done fully automatically.

For the hardness measuring of thin film, thin layer on compact materials or ceramic, the Rockwell method cannot be used because its high preliminary and total test forces would influence in the hardness results due to the crack formation induced during the indentation.

In the Brinell method, the metal ball indenter can cause differences in the hardness results because it can suffer deformation during the test.

In the Vickers method, all hardness scales are measurable, being a continuous measurement method and, the application of small test forces are estimable and available in standards, facilitating the measured of fine layers. The disadvantage of this method is that it's submit to the influence of the operator and this influence can affects the resolution power and analysis of the impression, increasing considerably the uncertainty measurements.

The uncertainty measurements of an impression with 5m residual diagonal given by a Vickers indenter, for example, can be in the order of 20% when used an optic microscope for the reading. This uncertainty can grow as the size of the indentation decreases and it can arrive as high as 100% for impressions with diagonals of 1m (Fischer-Cripps, 2002).

Better would be to have a method that could use a indenter like the Vickers one, in a wide scale of test force, in consonance with an automatic measurement just like the Rockwell method (Polzin, 1997).

2.2. Historical and Establishment of the Instrumented Indentation Test

In 1993 the German institute of standardisation, Deutsches Institut für Normung (DIN), presented to the technical subcommittee TC 164 SC3 of the International Organisation for Standardisation (ISO), the technical report TR 14577 in which proposed the establishment of a new hardness measurement method, the Universal Hardness (HU).

In 1994 the DIN decided after the local industrial demands that the set of standards sent to ISO by means of the technical report TR 14577 should be published immediately in Germany. Those standards had the following numbers and titles: DIN 50359: Universal hardness testing of metallic materials: “part 1 – Test method”, “part 2 - Verification of testing machines and” “part 3 - Calibration of reference blocks” (Machado, 2005).

This method was based on the use of diamond indenters like the Vickers (pyramid of square base) or Berkovich (pyramid of triangular base) to indent into a sample surface with the test forces and indentation depths measured and presented simultaneously by means of a curved Fxh in the screen of a computer, figure 1. With this new method, it was possible to characterise the plastic and elastic behaviour of a material with minimal operator influence.

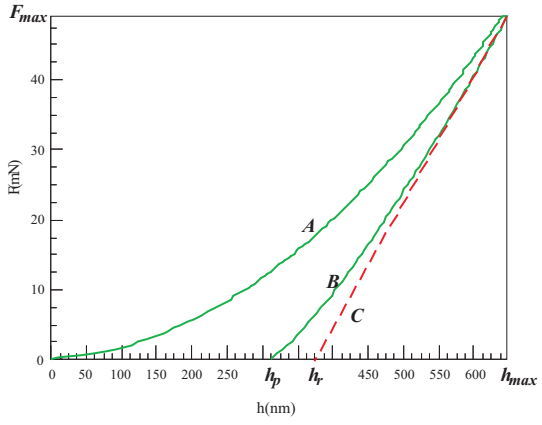


FIGURE 1 - Schematic graphic of the measurement cycle considering the force-indentation depth behaviour (Machado, 2005).

Where A is the application of the test force; B is the removal of the test force; C is the tangent to the curve B at F_{max}; F is the test force (mN); F_{max} is the maximum test force (mN); h is the indentation depth under applied test force (nm); h_{max} is the maximum indentation depth at F_{max} (nm); h_r is the point of intersection of the tangent to the curve B at F_{max} with the indentation depth-axis (nm) and h_p is the permanent indentation depth after removal of the test force (nm).

The figure 2 shows a cross section of one indentation where is possible to identify the depths monitored during the test.

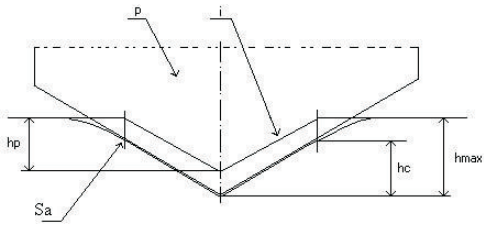


FIGURE 2 - Sketch of the cross section of one Vickers impression, considering the measurement cycle with the force-indentation depth behaviour (ISO 14577-1).

Where p is the indenter tip; i is the surface of residual plastic deformation in sample; S_a is the surface of sample at maximum indentation depth and maximum test force; h_{max} is the maximum indentation depth at F_{max} (mm); h_p is the permanent indentation depth after removal of the test force (mm) and h_c is the depth of the contact of the indenter with the sample at F_{max} (mm).

Comparing this method to the classic hardness measurement (Brinell, Rockwell and Vickers) where the size of the impression is measured only after the removal of the total or additional test force, it was noticed how much this new method was more powerful when one wanted to analyse material properties, mainly the localised measurements and small thickness of the materials, remarkably in the micro and nanometer ranges.

Only, in 1997 the technical subcommittee ISO/TC 164/SC 3 decided to work on this item and to develop an International Standard based on the document "ISO/TR 14577 Instrumented Indentation Test for Hardness and other Materials Parameters" with the following parts: "part 1 - Test method", part 2 - Verification and calibration of testing machines" and part 3 - Calibration of reference blocks. In 2001 it was decided to start a work in the "part 4 - Test method for coatings".

From the effective beginning of the establishment of this new method, called Instrumented Indentation Test (IIT) to the current days, the technical possibilities of the test force application, the measurements of indentation depth, the registration and storage of data and the graphic representations of the results, has being optimised to better understanding of the equipment, mainly in the meaning of the automated control of its functions, through a computer.

With the automation of the measure, this technique can provide the calculation of several important characteristics of the materials, among them (ISO 14577-1):

Martens Hardness (HM): defined as the test force F, divided by the contact surface area of the indenter $A_s(h)$ (up to 2001 it was called Universal Hardness), equation 2. It includes the plastic and elastic deformations of the material (N/mm^2).

$$HM \quad \frac{F}{A_s(h)} \tag{2}$$

Indentation Hardness (H_{IT}): defined as the maximum test force F_{max}, divided by the projected area of contact (cross section) between the indenter and the sample $A_p(h_c)$, equation 3. It includes the plastic deformation only (N/mm^2).

$$H_{IT} \quad \frac{F_{max}}{A_p(h_c)} \tag{3}$$

Indentation Modulus (E_{IT}): defined from the slope of the tangent of the force removal curve (N/mm^2). It includes characteristics of the indenter and of the sample. In the equation 4, v_a is the Poisson's ratio of the sample; E_r is the reduced modulus of the indentation contact; v_p is Poisson's ratio of the indenter and E_p is modulus of the indenter. In the equations 5 and 6, C_s is the compliance of the contact associated to the indentation in the sample, that is the inverse of the contact stiffness S.

$$E_{IT} \quad \frac{\left(1 \quad {}_a^{\,2}\right)}{\frac{1}{E_r} \quad \frac{1 \quad {}_p^{\,2}}{E_p}} \tag{4}$$

$$E_r \quad \frac{\sqrt{\quad}}{2 \quad C_s \quad \sqrt{A_p(h_c)}} \tag{5}$$

$$C_s \quad \frac{1}{S} \quad \frac{dh}{dF} \tag{6}$$

Thus, the IIT has been turned important technique in the development of nanostructural materials, thin films, ceramic materials, etc., for its speed and capacity to determine these mechanical properties independently from hardness. This method is frequently the only one which it makes possible the research of properties in tiny areas of materials (< 200 nm), (Göeken and Kempf, 2001).

2.3. Field of Application

The application fields of IIT are large and diversified, besides to provide information for the hardness and the elastic modulus, they also provide important information on other properties of the materials. Among them, some of general and current importance in the scientific research can be outstanding, such as (Hysitron Inc., Minneapolis, MN, USA):

- Characterization of nanoscale components.
- Assess of adhesion of bond pads.
- Quantification of wear resistance of wear-resistant coatings.
- Evaluation of interfacial toughness, mechanical properties of individual phases.
- Measurement of viscoelastic properties of polymers.
- Quantification of static and dynamic mechanics of MEMS and NEMS devices.
- Quantification of fracture toughness and interlayer adhesion.
- Wear resistance and multi-layer adhesion of optic cables.
- Characterization of compatibility of proposed bio-replacement materials.

3. AN OVERVIEW OF THE BRAZILIAN NANOINDENTATION RESEARCH GROUPS

In Brazil, the first research groups that showed the interest in the mechanical properties characterization in micro and nanometer levels where those linked to applied physics area of the universities, remarkably those from surfaces engineering area.

In 1996 was created in the Physics Department of Federal University of Paraná-UFPR, the Nanomechanical Properties Laboratory with the acquisition of the Nanoindenter II equipment (Nano Instruments Inc., USA).

Also in the middle of the nineties, the Surface Phenomenon Laboratory of the Mechanical Engineering Department from the São Paulo University USP-SP, acquired an instrumented indentation test, the Fischerscope H100V (Helmut-Fischer GmbH, Germany).

Later on, in 1998, the Group of Studies of Surfaces and Interfaces Properties of the Physics Faculty of Catholic University of Rio Grande do Sul PUC-RS, also acquired an equipment Fischer H100V.

In 2003, other two groups acquired equipment's of hardness using the instrumented indentation test technique. A Hysitron Triboindenter (Hysitron Inc., USES) was acquired by the Plasma and Applications Laboratory of Physics and Chemistry Department of the State University from São Paulo UNESP-Guaratinguetá, and a Dynamic Ultra-micro Hardness Shimadzu DUH-W201S (Shimadzu Co., Japan) and a Hysitron Triboscope were acquired by the Technological Metallurgy Section of CETEC-MG. In 2005 was the Laboratory of Tribological Coatings of the Metallurgy and Materials Department of UMFG that acquire a Shimadzu DUH-W201S, similar to the CETEC-MG machine.

Beyond of those research groups, the Tribology and Materials Laboratory of the Physical Sciences Department of the Federal University of Uberlândia-UFU, comes developing in the own laboratory instrumented indenter to determine properties in high temperatures environments (Franco et al., 2001).

It's important to point out that these research groups come maintaining an interactivity with the groups that investigate the processing of materials, e. g., there is a critical mass in the Country, although incipient, that is capable to caring out instrumented indentation tests with high quality, concentrating efforts to familiarize the necessary language for interpretation and discussion of the results (Pintaúde and Machado, 2003).

3.1. Brazilian demands

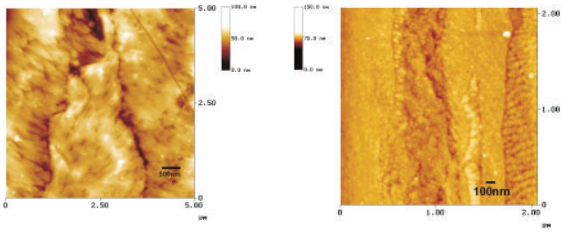
3.1.1. Nanostructural Materials

The Department of Metallurgy and Materials Engineering (DEMET) of the Federal University of Minas Gerais (UFMG) presents a high activity level in the field of mechanical conformation of metallic materials, where the need of determination of the final properties exists after processing that are quite heterogeneous, macro and microscopically. Local hardness measures in nanometer level are special for the evaluation of the deformation heterogeneity allowing studies on the consequences of this phenomenon. Nowadays is relevant the development of materials said nanostructured with very small grain sizes, in the nanometer range, what checks to the material a mechanical resistance much larger than the alloys with conventional microstructure.

The methods of structural processing more adequate for industrial applications are based on the Severe Plastic Deformation (SPD). Among the techniques of SPD, the most promising are the Accumulative Roll Bonding (ARB) and the Equal Angular Channel Pressing (ECAP)" (Costa et al., 2005).

From these methods, very high rates of plastic deformation can be obtained through the accumulative deformation after a convenient number of passes.

In the figures 3a) and 3b) examples of deformations are given by the process ARB in Interstitial Free (IF) steel. The understanding of the processes of structural refinement that take to the formation of nanometer grains size, requests detailed characterization of the nanostructure of the material, what can just be carried out with advanced microscopy techniques, like the Atomic Force Microscope (AFM), and the measurements of nanomechanical properties throughout IIT, that seams to be the only available technique in the study of this structural evolution (Costa et al., 2005).



a) AFM images, scan size = 5 mx5 m, Z scale = 100nm *b) AFM images, scan size = 2 mx2 m, Z scale = 150nm*

FIGURE 3 – IF steel: a) 2 passes ARB and b) 32 passes ARB. (Costa et al., 2005)

3.1.2. Trybological films

The DEMET/UFMG has also been investing in the research of surface engineering and, especially, in the study of trybological coatings, because the demand for measures in films with thickness more and more thin in areas mechanically and chemically aggressive has been increasing considerably.

The assessment of the substrate/coating performance depends on the measurement of mechanical properties of such deposited films as hardness, elastic modulus and toughness. The conventional hardness test in the micrometer range allows to determine some properties of the films but, due to the small thickness, lower application test forces are necessaries so that those properties are measure without or with the minimal of substrate influence and this is only possible from the application of the technique of micro and nanohardness by IIT (Batista, 2001).

3.1.3. Decorative films

Interference films deposition is an alternative for the formation of colored protective layers on the surface of stainless steel for decorative applications. The process involves the growth of a chromium oxide films by electrochemical alternating current method on the surface of stainless steel checking to the films different porosity depending on the duration of the current pulses.

Results of researches in progress in the Technological Foundation Center of Minas Gerais (Cetec/MG) for the durability assessment of those colored stainless steels, has been indicating that the morphology of the films can increase the mechanical resistance of those materials. The combination of IIT device in nanometer level with an Atomic Force Microscope (AFM) allows indenting the surface in very shallow depth and imaging the impression with the same tip. Besides, this combination enables to positioning the tip in very local area for nanoindentation test with a high level of accuracy.

The figure 4 shows the topographic images of the gold interference films deposited in an AISI 304 stainless steel sheet with low and high porosity before and after the nanoindentation test with an IIT-AFM Hysitron nanoindenter.

The mechanical properties extracted from the force-displacement curves with F_{max}=100μN, applied over a diamond indenter with triangular base (cube-corner), figure 4, are presented in table 1.

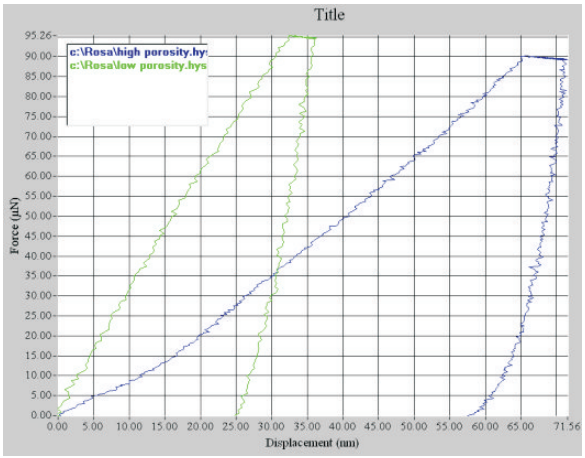
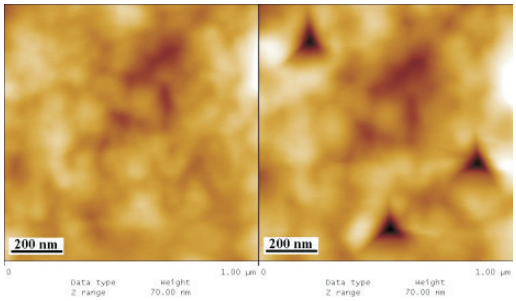


FIGURE 4 – AFM Images before and after the application of $F_{max} = 100\mu N$ in gold films with high [a.1) and a.2)] and low [b.1) and b.2)] porosity in an AISI 304 stainless steel.

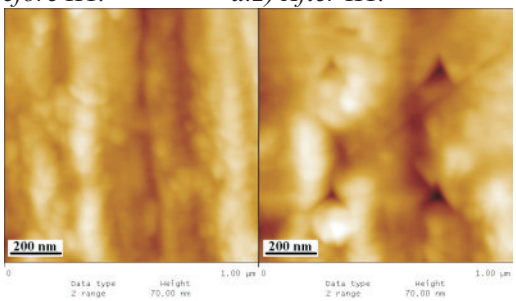
Table 1 - Mechanical properties of gold interference films electrodeposited on AISI 304 stainless steel.

Mechanical property	*High porosity film*	*Low porosity film*
H_{IT} (GPa)	2.4	6.9
E_{IT} (GPa)	75.2	101.5
h_{max} (nm)	71.6	36.2
$A_p(h_c)$ (nm^2)	37276.1	14233.0

In the figure 5 it is possible to observe that the residual indentations of the film with high porosity are larger than the indentation of the film with low porosity.



a.1) Before IIT. *a.2) After IIT.*



b.1) Before IIT. *b.2) After IIT.*

FIGURE 5 - Force-displacement curves for the high and low porosity in gold interference films on AISI 304 stainless steel.

The combination of Nanoindentation test with an Atomic Force Microscope is frequently the only method that makes possible the characterization of near-surface of nanomechanical properties of materials as small as 50nm. The possibility to have the indentation restricted to a small fraction of coating thickness might be suitable for isolating “coating-only” properties (Junqueira, et al., 2005).

3.2. Other Demands

3.2.1. Localized Measurements with Nanometer Resolution

The application of this new method can also be verified in the work done by Kempf et al. (1998). It was carried out studies in order to determine localized mechanical properties with nanometer resolution of metallic alloys to allow the best understanding of the materials with objectives to the improvement of theirs structural projects. It was used an nanoindenter equipment coupled to a AFM with forces varying of nanonewtons to micronewtons and they captured images with amplifications of 300.000 times to study the matrix and the precipitate ' of a CMSX-6 superalloy, figure 6 (Machado, 2003).

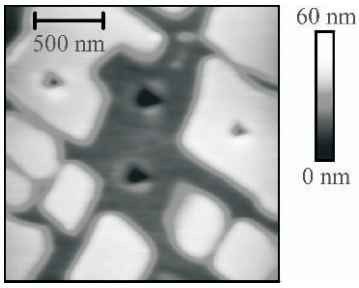
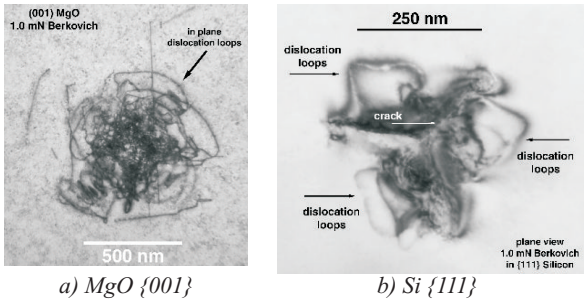


FIGURA 6 - Impressions in the CMSX-6 alloy, $F_{max} = 500mN$. The matrix corresponds to the dark area and the precipitate ' to the clear area.

3.2.2. Residual Dislocations Description and Elastic-plastic Responses

The nanoindentation behavior of various materials at low test forces is also currently used to investigate, with the immediate goal, the determination of the initial stages of material response to elastic-plastic contacts. Results relating to the indentation behavior of {111}Si and {001} MgO at force of 0,1mN to 5mN using a Berkovich indenter is being researched. Plane-section Transmission Electron Microscopy (TEM) observations are used to describe the residual dislocation structures produced in MgO at test forces down to 0,1mN and to describe the elastic-plastic response (i.e., dislocations, phase transformations and fracture) of silicon at test forces down to 0,25mN. The TEM results, figures 7a) e 7b), together with associated Atomic Force Microscopy (AFM) images of indentations and the indenter tip, are directly related to the initiation of plastic deformation in these materials and, more generally, to the elastic-plastic response seen in acquired load-displacement plots, figure 8 (Hockey, Machado and Guin, 2005).



a) MgO {001} *b) Si {111}*

FIGURE 7 - TEM images, $F_{max} = 1mN$.

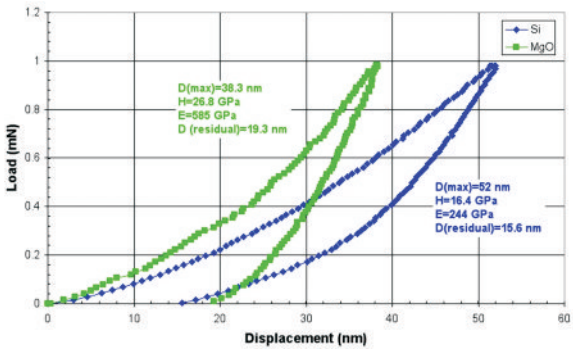


FIGURE 8 - Fxh curves in MgO {001}and in Si {111}, $F_{max} = 1mN$.

4. CONCLUSIONS

As it can be noticed, the main technological consequence in the difference of determination of the conventional hardness test with the hardness and other property for instrumented indentation test is that, since the hardness measurement doesn't depend more on the resolution of an equipment to analyse the produced plastic impression, the hardness and other mechanical properties can be measured in very small indentation depths under application of test forces also in very small scale. With the progress of technologies capable to produce superficial modifications of the materials in nanometer level (nanotechnology, nanoscience), the hardness and other mechanical properties, became dependent of the test force applied and of measurements in very tiny area of the material surface.

Because of that, the research centers and the universities in Brazil are beginning to make efforts in the development of projects in the field of the nanomechanical materials characterisation, because the nanotechnology and nanoscience areas are being shown as a strategic and important demand for the innovation in scientific and technological for the development of the nations, and the know-how of this of the micro and nanoindentation technique comes to support these needs.

Acknowledgments: The authors, 2 and 3, thank CNPq and FAPEMIG for financial support.

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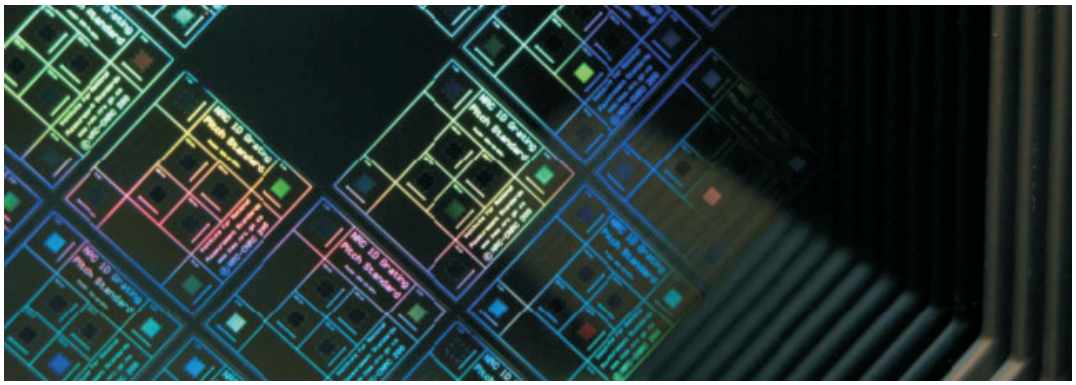
THE NANOSCALE: THE SMALLEST NEW BIG DEAL IN MEASUREMENT AT NRC

Co-written by National Research Council Canada scientists and writers



You'll never use this scale to measure anything at home but it could one day be the measure of the tiny technologies that transform our daily lives.

That's because a team of National Research Council of Canada scientists is developing a measuring tool with regularly spaced nanoscale lines or gratings much like those on a ruler. It could be what many engineers reach for when building nanotechnologies. These length standard artifacts will help guide Canada and the international community into a new age of minute measurement.

The nanometre (billionth-of-a-meter) is the frontier of industrial measurement. Scientists and engineers can already create and manipulate nanoscale devices in their labs, and many computer chips have nanometre-level detail. But there is a major missing ingredient in the nanotechnology revolution—a measurement tool. Scientists and engineers don't have an easily accessible internationally recognized nanoscale length standard.

It's a situation reminiscent of the one that led to Canadian Sir Sanford Fleming's leadership in the creation of International Standard Time. The rise of transcontinental railroads in the 19th century necessitated standardized time in order to make the trains commercially reliable and effective.

“We need to establish traceable and verifiable measurement standards and methods for nanotechnologies,” says Dr. Jennifer Decker, team leader for metrology for nanotechnology at the NRC Institute for National Measurement Standards (NRC-INMS). Dr. Decker is also a participant in the Canadian Advisory Committee to the International Standards Organization (ISO) Technical Committee on Nanotechnologies, in particular the Working Group meetings to determine nanoscale metrology standards and characterization.

“These standards will have a significant commercial impact since they'll set the rules that everyone will play by. It's important that Canada is at the table—the countries that participate dictate what the standards will be.”

“It's all about thinking one step ahead of the developing nanotechnology in order to facilitate commercialization,” says Dr. Mark McDermott, a principal researcher at the NRC National Institute for Nanotechnology (NRC-NINT) and an Associate Professor in the Department of Chemistry at the University of Alberta.

NRC Scientists Measure the (Small) Force

Imagine trying to glue pudding to Styrofoam. The inevitable result: a mess. It's what NRC scientists are working to avoid at the nanoscale. To do it, they're building one of the most sensitive small force detectors in the world.

Small forces are those that at the molecular level determine physical characteristics such as adhesion, hardness and elasticity. For large objects that we can see, we can readily measure these qualities. But at the nanometre (billionth-of-a-meter) level, it's a new world of material properties.

“When you start piecing together nanomaterials, their small force characteristics determine whether you're going to succeed or fail,” says Dr. Mark McDermott, a principal researcher at the NRC National Institute for Nanotechnology (NINT) and an Associate Professor in the Department of Chemistry at the University of Alberta. “In the emerging field of commercial nanotechnology, it's critical to be able to measure and characterize these small forces.”

Dr. McDermott is leading a collaborative NRC effort to create one of the world's most sensitive small force measurement devices. Called an Interfacial Force Microscope (IFM) it will function as an incredibly fine needle that pokes a nanomaterial and records how deep

the needle goes (nanoindentation). This will provide detailed measurements of a material's small force characteristics, and thus how it will behave in relation to other materials.

The research is a core part of an NRC cross-institute project, led in Canada by NRC-INMS, to develop quantifiable measurement standards for nanotechnology.

The IFM project is in collaboration with University of Western Ontario Professor Peter Norton. He's world-renowned for his work in using IFM in nanotribology to study the nanocharacteristics of interacting surfaces, such as gears, in motion. The NRC-NINT team includes Dr. David Munoz-Paniagua, a former Ph.D. student of Dr. Norton's, and one of the only people in the world with experience in IFM construction.

"At this level a small difference in length can make a big difference in function. So if materials need to be separated by two nanometres to work, and at 2.1 nanometres the device doesn't function, you need to be able to measure and control at that level."

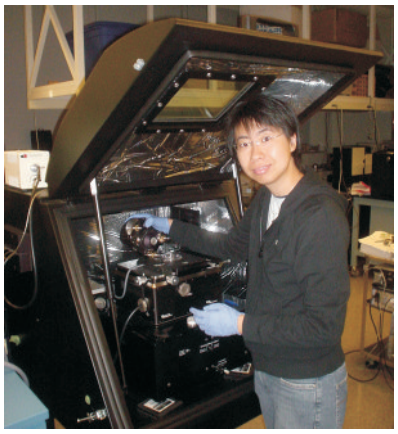
The Canadian nanoscale length standard is being developed through a NRC cross-institute initiative involving four NRC institutes and two universities. Together they have the unique mix of skills and abilities in nanoscale fabrication, instrument design, and metrology.

As envisioned, each measurement standard, (imagine a patch of regularly-spaced lines), will be approximately one square millimetre in size—eight of these grating patches will be fabricated on a silicon chip about one centimetre square. So this length standard chip contains eight gratings ranging in nominal pitch from 150 nm to 10 µm.

The prototype will be made at the recently established Canadian Photonics Fabrication Center (CPFC), part of the Ottawa-based NRC-Institute for Microstructural Sciences.
NRC-IMS scientists use electron beam lithography, which utilizes a precisely controlled beam of electrons to pattern nanophotonic and nanoelectronic devices. This expertise in creating nanoscale devices is being extended by the CPFC that is developing nanoimprint lithography to create a length standard prototype chip. Led by researcher Dr. Frank Shepherd, the group will first use electron beam lithography to make a template, or master, in quartz. This template will be then be used to replicate exact copies of these nanometer grating length standards.

One of the goals of the NRC Metrology for Nanotechnology Program is to determine the extent to which the gratings made by the nano-imprint lithography technique have the same line spacing. Metrologists in the dimensional metrology laboratory at NRC-INMS will do the determination.

In the metrology laboratories, an imaging diffractometer was developed to calibrate one-dimensional grating pitch standards: laser light is directed on the grating, the angle



of the reflected beam is measured, and the value of the average pitch is calculated. The NRC diffractometer calibrates the average line spacing with an uncertainty much less than the distance between atoms.

Another key tool is the metrological atomic force microscope currently being developed at NRC-INMS by Dr. Brian Eves. Most scanning probe microscopes rely upon calibrated artifacts such as the NRC length standard chip to ensure they correctly measure length. The metrological atomic force microscope will guarantee the correct measured length by using the definition of the metre, based upon the speed of light in a vacuum, as its standard. One of its tasks will be to calibrate line spacings smaller than currently possible with the imaging diffractometer.

Science is such that there will in all likelihood be another generation of nanoscale standards built perhaps on intrinsic standards such as the inter-atomic spacing of crystalline silicon. Dr. Marek Malac at NRC-NINT will be investigating the use of such standards using a transmission electron microscope.

For now, we need to answer the needs of scientists like Dr. Linda Johnston at the Steacie Institute for Molecular Sciences. Scanning probe microscopes and integrated optical-atomic force microscopes are among the most important tools for the development of nanotechnology. The best instruments enable researchers to image living cells and even the atomic structure of surfaces. For these microscopes to obtain accurate results it is necessary to calibrate them frequently using one-dimensional grating pitch standards. Dr. Shan Zou, NRC-SIMS, will also be working with NRC-INMS to develop artifacts for standard force and distance measurements on soft biological samples.

And with our eye on the economy, and in particular the support of trade and global manufacturing, the grating artifacts will be used to support international comparison experiments. If the pitch value of the artifacts fabricated by nanoimprint lithography can be shown to be essentially identical, then multiple artifacts can be sent simultaneously to National Measurement Institutes around the world, improving the metrological integrity of the comparison exercises—and giving scientists and engineers that all-important easily accessible internationally recognized nanoscale length standard.

ESTABLISHMENT OF THE SIM TIME SCALE

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ABSTRACT
The SIM time and frequency metrology working group has developed a comparison network for the Americas, with the goals of improving metrology in the SIM region and to allow as many countries as possible to participate in the network. As of May 2008, ten National Metrology Institutes (NMIs) were participating, and six additional NMIs are expected to join the network by the end of 2009. This paper describes how measurements from the SIM network will be used to generate a time scale named SIM-time to be used as the time reference for the SIM region.

1. INTRODUCTION

The *Sistema Interamericano de Metrología* (SIM) consists of the National Metrology Institutes (NMI's) located in the 34 states that are members of the Organization of American States (OAS). Currently, about one half of the countries that are members of the OAS operate and maintain a time and frequency metrology laboratory, and some of the remaining countries plan to establish a laboratory in the future. With the goal of developing an efficient comparison mechanism throughout the Americas for time and frequency metrology, the SIM time and frequency metrology working group has built a regional comparison network that uses the GPS common view technique. The SIM time and frequency comparison network is helping to improve the development of metrology in SIM region by promoting the participation of as many countries as possible, and allowing each country to see their comparison results in real time. The time and frequency (TF) comparison network of SIM began operations in 2005 with the participation of three national laboratories: National Research Council (NRC) of Canada, National Institute of Standards and Technology (NIST) of the United States of America and the Centro Nacional de Metrología (CENAM) of Mexico 1. As of May 2008, ten countries of the region were taking part in the comparison network, and 16 SIM NMIs are expected to participate by the end of 2009. A report with the most recent results of the SIM TF comparison network was presented in [2].

To make efficient use of the comparison results produced by the SIM TF network and to reinforce the continuing development of metrology in the SIM region, a SIM time scale, called SIM-time, will be generated. The SIM-time scale will provide a time reference for the SIM region that is as stable and accurate as possible. The comparison results of each of the NMIs time scales participating in the SIM network with respect to the SIM time scale will be available in real-time through the network itself.

This paper presents the algorithm that will be employed in the generation of the SIM-time scale, and also presents preliminary results obtained by the Centro Nacional de Metrología, CENAM, where the time scale algorithm is implemented on four Cs clocks and one active hydrogen maser. Much of the basic analysis presented in Section 2 is presented in detail elsewhere [3-5], but is summarized here for completeness.

2. ALGORITHM OF THE SIM-TIME SCALE

If a set of N atomic clocks is available, then an Atomic Time scale *TA* can be defined by the Eq. (1):

$$TA(t) \quad \frac{1}{N} \sum_{i\ 1}^{N} h_i(t) \ , \qquad (1)$$

where $h_i(t)$ is the reading of the clock *i* at the time *t*. It is important to notice that in Eq. (1) every participating clock has the same weight. If different weights are assigned to each clock, then the time scale *TA* is defined as:

$$TA(t) \quad \frac{1}{N} \sum_{i\ 1}^{N} {}_i h_i(t) \ , \qquad (2)$$

where ${}_i$ is the weight of the clock *i* and the condition of normalized weights is given by:

$$\sum_{i\ 1}^{N} {}_i \quad 1. \qquad (3)$$

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It is possible to define each weight $_i$ as constant in time. However, the best scenario is given when a dynamic weighting approach is implemented, that increases or decreases the weight given to a clock according to its frequency stability characteristics, which can change over time for different reasons. In this case the weights can be defined as inversely proportional to the frequency stability of the clock under consideration, where the frequency stability is measured in terms of the Allan deviation. In this scheme we can define the weights as:

$$_i(t) \quad \frac{1}{_i(\ )}\ , \qquad (4)$$

where $_i(\ )$ is the Allan deviation of the clock i for an integration time τ. The proportional constant is defined by the normalization condition on weights, Eq. (3). In this way, the *TA* time scale definition in terms of the dynamic weighting takes the following form:

$$TA(t) \quad \frac{\sum_{i\ 1}^{N} {}_i h_i(t)}{\sum_{i\ 1}^{N} \frac{1}{_i(\ )}} \quad \frac{\sum_{i\ 1}^{N} \frac{1}{_i(\ )} h_i(t)}{\sum_{i\ 1}^{N} \frac{1}{_i(\ )}}\ . \qquad (5)$$

Unfortunately, Eq. (5) cannot be implemented experimentally as written, because the readings from clocks $h_i(t)$ are not observables. Experimentally it is possible to know only the time difference between pairs of clocks.

We can rewrite Eq. (5) in terms of the time differences between participating clocks, by subtracting from each side of the equation the reading of clock *k* at the time *t* as follows:

$$\begin{aligned} &\sum_{k\ 1}^{N} {}_k x_k(t) \quad \sum_{k\ 1}^{N} {}_k \sum_{i\ 1}^{N} {}_i x_{ki}(t) \\ &\sum_{k,i\ 1}^{N} {}_k\ {}_i x_{ki}(t) \quad 0. \end{aligned} \qquad (6)$$

If we define $x_k(t)$ as the time difference between clock *k* and the time scale *TA* at the time *t*, and define x_{ki} as the difference between clock *k* and clock *i* and we take in consideration the normalization condition on the weights in Eq. (3), then Eq. (6) takes the form:

$$x_k(t) \quad \sum_{i\ 1}^{N} {}_i x_{ki}(t). \qquad (7)$$

This last equation defines the time scale *TA* in terms of the time differences between participating clocks. It is important to notice that the weighted average of $x_k(t)$ is zero, because $x_{kj}(t) = - x_{jk}(t)$; that is,

$$\begin{aligned} &\sum_{k\ 1}^{N} {}_k x_k(t) \quad \sum_{k\ 1}^{N} {}_k \sum_{i\ 1}^{N} {}_i x_{ki}(t) \\ &\sum_{k,i\ 1}^{N} {}_k\ {}_i x_{ki}(t) \quad 0. \end{aligned} \qquad (8)$$

Once the time differences $x_{ki}(t)$ are known, it is then possible to compute the time scale *TA* by using Eq. (7). However, it is important to notice that this computes the time scale using a post-processing scheme. In cases where it is necessary to generate a time scale *TA* in real time, we must introduce a scheme of prediction for the time differences among clocks and the time scale *TA*.

One of the simplest models to predict the time difference of a clock with respect to a reference (we assume the reference is more stable and accurate than the clock under consideration) is:

$$\hat{x}_k(t \quad) \quad x_k(t) \quad y_k(t) \quad \frac{D_k}{2} \quad \ldots \quad , \qquad (9)$$

where $\hat{x}_k(t \quad)$ is the prediction of the time difference of clock *k* respect to the reference for the future time t. $x_k(t)$ is the (known) time difference between the clock *k* and the reference, and $y_k(t)$ is the fractional frequency difference at the time *t*. Finally, D_k is a constant accounting for changes of $y_k(t)$ during the interval.

Eq. (9) can be seen as expansion of the x_k in terms of a Taylor series around the value $x_k(t)$ for a time interval of . Once the time difference $x_k(t)$ is known through Eq. (7), it is possible to predict the time scale TA for the time t using Eq. (9). Here it is important to note that the weighted average of the predictions $\hat{x}_k(t \quad)$ is not necessarily zero; that is,

$$\sum_{k\ 1}^{N} {}_k \hat{x}_k(t \quad) \quad 0. \qquad (10)$$

Once the (future) time $t + \tau$ is reached, it is possible to know the time differences between participating clocks, and then it is possible to compute the value of the time scale *TA* for that time t. Of course, the predicted value of the time scale *TA* computed at time *t* for the time t will not necessarily be equal to the computation of *TA* at time t. Under this scheme, the time scale prediction for t can be corrected by the time difference measurements by using:

$$x_k(t \quad) \quad \sum_{j\ 1}^{N} {}_j \hat{x}_j(t \quad) \quad x_{jk}(t \quad)\ . \qquad (11)$$

The prediction $\hat{y}_i(t \quad)$ of the fractional frequency deviation of clock i for time t is made according to:

$$\hat{y}_k(t \quad) \quad \frac{\hat{x}_k(t \quad) \quad x_k(t)}{}. \qquad (12)$$

Once the (future) time $t+\tau$ is reached, the correction for the frequency prediction can be made through the exponential filtering defined by

$$y_i(t \quad) \quad \frac{1}{1 \quad m_i}\, \hat{y}_i(t \quad) \quad m_i(\ )\, y_i(t)\ , \qquad (13)$$

where m_i is given by the Eq. (14) as:

$$m_i(\ ) \quad \frac{1}{2} \sqrt{\frac{1}{3} \quad \frac{4}{3} \frac{^{2}_{\min,i}}{^{2}}} \quad 1 \quad , \qquad (14)$$

and $_{\min,i}$ is the integration time at which the noise floor of the clock i is reached.

3. RESULTS

The algorithm presented in the previous section has been implemented at CENAM on four Cs clocks and one active hydrogen maser. The time scale TA is computed every hour with the dynamic weighting algorithm described in [4]. The system that measures the time differences between clocks, also referred to as the phase comparator, utilizes the dual mixer frequency technique and has a resolution of 20 ps. The phase comparator has 32 input channels, which allows it to compare that same number of clocks. It performs one time difference measurement every second for each of the 32 channels in use. One Cs clock (labeled as Cs I) is selected as the master clock, and all of the other clocks are compared to the master.

In this section we show the results obtained at CENAM during the implementation of the time scale algorithm. The results were obtained from six weeks of continuous time scale generation (from April 5 to May 16, 2008). The following results are considered as preliminary because it is necessary to measure the performance of the time scale algorithm for a longer period to obtain more confidence in the results.

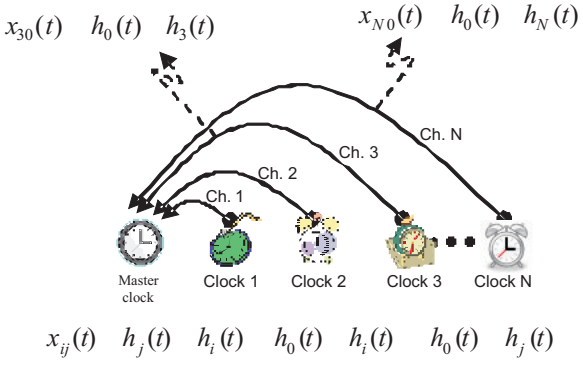

Fig. 1. Schematic of the time difference measurement system used at CENAM, also referred to as a phase comparator. The system performs one measurement every second.

Fig. 1 presents a schematic of the time difference measurement system (the phase comparator). The clocks are compared through their 5 MHz output signals. The phase comparator measures the time differences between clocks modulo one signal period (200 ns). To obtain the total phase difference it is necessary to post-process the phase comparator results.

To transform the time scale TA from a virtual time scale (with no physical signal defining the time scale) to a real time scale (with a physical signal defining the time scale) we use a micro phase stepper (MPS). The MPS steers the 5 MHz frequency signal of the active hydrogen maser so that it follows the virtual TA time scale.

The output of the MPS is used to measure the time difference between the time scale and the corrected frequency of the hydrogen maser. The MPS is controlled by an automatic servo loop so that the time difference between the TA and the MPS output remains constant (Fig. 2).

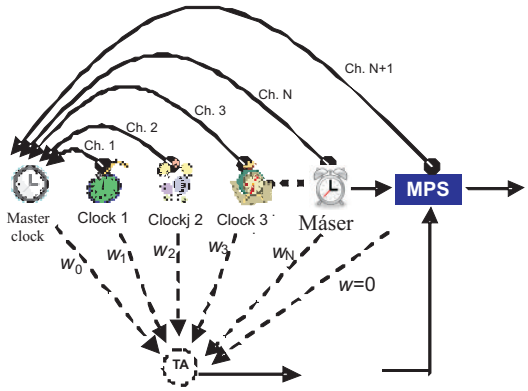

Fig. 2. Schematic showing how the output of the micro phase stepper is held in agreement with the time scale TA.

Fig. 3 shows the measurement results from the phase comparator during six weeks, from April 4th to May 16th, 2008. The vertical axis corresponds to the time difference of participating clocks with respect to Cs I, the master clock. Discontinuities observed at 0 ns and 200 ns are due to the fact that the phase comparator performs time difference measurements modulo one period of the 5 MHz signals (200 ns). To correct for these discontinuities, it is necessary to post-process the phase comparator measurements. It is also important to notice the frequency change of clock J on April 12th, 2008, marked in the figure within a circle.

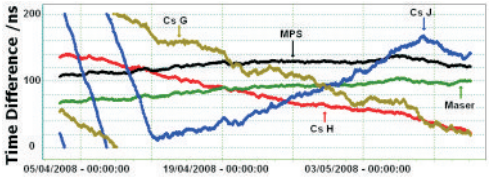

Fig. 3. Time difference measurement of participating clocks with respect to the master clock (Cs I). The black line corresponds to the MPS output.

Fig. 4 shows the time differences of the participating clocks with respect to the time scale TA. It is interesting to note how the frequency change of clock J on April 12th, 2008 affected the time scale *TA* performance.

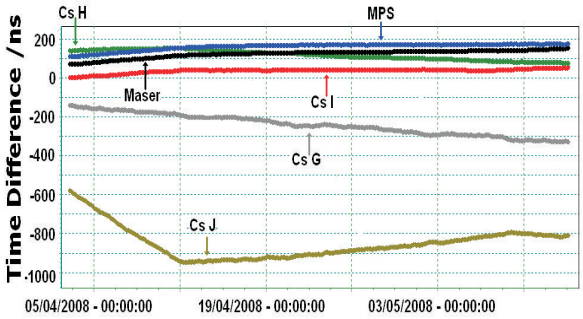

Fig. 4 Time difference of participating clocks with respect to the time scale TA.

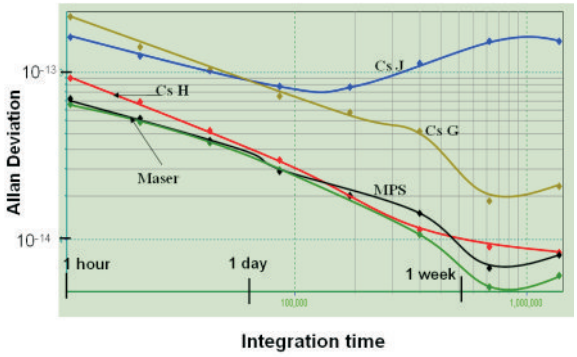

Fig. 5 Frequency stability of the time difference of participating clocks with respect to the master clock (Cs I). The black line corresponds to the frequency stability of the MPS output.

Figs. 5 and 6 show the frequency stability of the comparisons shown earlier in Figs. 2 and 3.

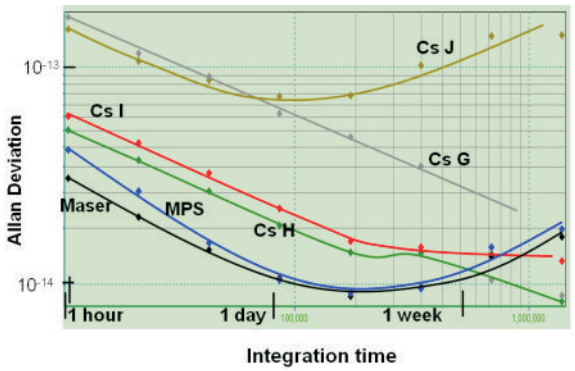

Fig. 6. Frequency stability of participating clocks with respect to the time scale TA.

4. DISCUSSION

Here we first present a discussion of the obtained results when the Cs clock is used as the master clock. Then, we will discuss results when the time scale TA is used as the master clock.

4.1. Measurement Results when Cs I is Used as the Master Clock (Fig. 5)

Due to the high frequency stability of the active hydrogen maser in the short term, the relative frequency stability between Cs I and the maser is a measure of the frequency stability of the master clock Cs I, which is about 8 1014 at 1 hour. If the Cs I clock is compared to another reference more stable than the maser, we will expect no significant change in this number, because it is already limited fundamentally by the Cs I clock itself. A similar argument can be applied to the results for the stability measurement of clock H, because clocks Cs I and Cs H have the same manufacturer's specifications for stability and perform similarly when they are measured in the laboratory. According to the manufacturer's specifications, Cs I and Cs H should have frequency stability around 7 10-14 for an average time of 1 hour, which is in close agreement with our results shown in Fig. 5.

Both Cs J and Cs G are less stable than Cs I and Cs H. This was expected, because both of them are low performance clocks. It is also interesting to note that the frequency correction on clock Cs J has an effect on the frequency stability results in the long term.

For integration time around one day or longer, note that the frequency stability of Cs H approaches the stability of the maser and MPS. It is actually because the frequency stability of the maser approaches the performance of a Cs clock. Finally, the frequency stabilities of clocks Cs J and Cs G are interesting for the purpose of evaluating the time scale performance. The frequency stability of these two clocks agrees with the manufacturer's specification for low performance Cs clocks. Because of their relatively large instabilities, if these two clocks are compared with a reference more stable than the Cs I clock, like the time scale TA, we will expect no significant changes in the frequency stability results.

4.2. Measurement Results when the Time Scale TA is Used as the Master Clock (Fig. 6)
As we discussed in the previous section, the frequency stability results for Cs J and Cs G are similar to the results when those clocks were compared with respect to Cs I.

The frequency stability for Cs I clock is slightly better than when it was compared with the maser. For integration time of 1 h the stability of Cs I when it was compared to the maser was about 8 10-14, as opposed to about 7 10-14 when compared to the time scale. This slight difference indicates that the stability numbers are limited by the (absolute) frequency stability of Cs I, and that the time scale is more stable than the maser itself. A similar discussion also applies to the Cs H clock.

The difference in stability between the maser and the MPS is due to the corrections of the maser frequency made by the MPS. The MPS corrects the maser frequency once per hour if necessary, causing a small perturbation on the maser frequency stability, as we can see in Fig. 6. These results suggest that corrections to the maser frequency output can be made less often than once per hour, perhaps once per day, and that the magnitude of the corrections can be made smaller. These possibilities will be carefully analyzed during the coming months.

The results of the TA time scale performance are satisfactory. The TA time scale appears to be more stable than the clocks participating on its generation. However, the stability of the time scale can probably be improved by optimizing the method used to apply frequency corrections to the maser output.

During the implementation stage of the SIM time scale with real data from the SIM TF comparison system will be necessary to take into account some important aspects, such as noise from the comparison system. To face that inconvenient we estimate the necessity to use of a previous filtering scheme on the measurement data before applying the algorithm here discussed. We estimate that about 30 Cs clocks and about 10 Hydrogen masers will contribute to the SIM time scale. Many of the individual clock signals are combined into a single time scale that is measured by the SIM network. For example, there are four Cs clocks and six Hydrogen Masers involved in the generation of UTC(NIST). Because the SIM network compares UTC(NIST) and not the individual clocks behind it, the weight of time scales like UTC(NIST) will be larger than the weight of signals currently supported by a single clock like UTC(ICE) in Costa Rica.

5. CONCLUSIONS

The Centro Nacional de Metrologia, CENAM, has developed and implemented an algorithm similar to the NIST AT1 algorithm 4 with the goal of providing a time scale for the SIM region, called SIM-time. The SIM-time scale will be generated using the algorithm discussed here; utilizing the time difference measurements provided by the SIM time and frequency network that is already in operation. The preliminary results obtained by implementing the SIM-time algorithm at CENAM on four Cs clocks and one active hydrogen maser are satisfactory. Special attention has been taken to implement the algorithm with the robustness required when generating a high performance time scale. Software is currently being added to the SIM time and frequency network that will feed the time scale algorithm with the necessary time difference data. We expect to generate the SIM-time scale by the end of 2008, and to make the results available in near real-time through the SIM time and frequency network.

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BREVE RESEÑA DE LOS ORÍGENES DEL SIM

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Centro Nacional de Metrología de Panamá

Durante la gestión del Sr. Roberto Monti dentro del Programa Regional de Desarrollo Científico y Tecnológico de la OEA, estando en marcha el Proyecto Especial "Sistema Regional de Metrología y Calibración", en 1974 se gesta la idea de constituir el Sistema Interamericano de Metrología-SIM.

En la Reunión de Coordinación Especial de Metrología y Seminario de la OEA, celebrado en Panamá y organizada por la Directora de la Comisión Panameña de Normas Industriales y Técnicas, COPANIT, Ing. Maricela Ferrer, del 25 al 30 de septiembre de 1977, se redacta la Resolución No. 1, que decía:

"Considerando: la resolución formulada por el CIECC de Mar del Plata (CIECC 174/72) referente a la implantación de un sistema regional de metrología y calibración, los acuerdos de las reuniones de Boulder (NBS, noviembre de 1974) y Buenos Aires (INTI, agosto de 1975); y considerando que las posibilidades que ya ha abierto el desarrollo aún en curso del Proyecto Especial originado en aquella resolución y vista la imperiosa necesidad de asegurar la coordinación de los esfuerzos realizados y por realizar; los participantes solicitan a la OEA y a los respectivos gobiernos, la consideración de la siguiente PROPUESTA: El establecimiento con carácter permanente del Sistema Interamericano de Metrología (SIM) de conformidad con las bases siguientes:

Promover la cooperación internacional entre los organismos competentes de los países participantes para contribuir al perfeccionamiento de las actividades en las áreas de la Metrología Legal, Industrial y Científica. A tal efecto, las acciones a realizar tenderán a lograr:

1. La definición del Sistema Nacional de Mediciones de cada país.
2. El establecimiento de la línea jerárquica de patrones en cada país y su enlace con los patrones internacionales.
3. La compatibilidad de los resultados de los procesos de medición correspondientes efectuados en los laboratorios del Sistema.
4. La formación de personal técnico y científico.
5. La obtención y distribución de los documentos técnicos y científicos.
6. La vinculación con la Conferencia General de Pesas y Medidas (CGPM), la Organización Internacional de Metrología Legal (OIML) y otros organismos nacionales e internacionales especializados en la materia.

The idea of building up a Sistema Interamericano de Metrología-SIM came up in 1974 during the administration of Mr. Roberto Monti within the OAS Regional Program for Scientific and Technology Development framework, when the special project on "Sistema Regional de Metrología y Calibración" was running.

During the OAS meeting about Special Coordination on Metrology and Seminar, held on Panama organized by the Director of the Panama Commission for Normas Industrial and Technical Standards, COPANIT, Eng. Maricela Ferrer, on September 25 to 30, 1977, a Resolution No. 1 was issued, to read:

Considering: the resolution issued by the CIECC in mar del Plata (CIECC 174/72) relative to the implementation of a regional system of metrology and calibration, the agreements of Boulder (NBS, November 1974) and Buenos Aires (INTI, August 1975); and considering the opportunities brought by the already started Special Project derived from such a resolution, and due to the imperilous need to ensure the coordination of the efforts done and to be done; the participants submit to the OAS and the conforming governments the following PROPOSAL: The establishment on a permanent basis of the Sistema Interamericano de Metrología (SIM), according to the following:

To promote the international cooperation among the involved organisms of the participant countries, aimed to contribute to the perfecting of the activities on Legal, Industrial and Scientific Metrology. In order to achieve that, actions will be taken directed to reach:

1. *The definition of National Measurement Systems in every country.*
2. *The establishment of a hierarchy of measurement standards in each country and its link to the international measurement standards.*
3. *The compatibility of the results of the measurement processes produced by the laboratories participating in the system.*
4. *The formation of technical and scientific personnel.*
5. *The acquiring and distribution of technical and scientific documents.*
6. *The link to the General Conference on Weights and Mesures (CGPM), the International Organization for Legal Metrology (OIML) and other relevant national and international organizations.*

Besides, voluntary participation as members of the SIM could be done through the corresponding national organizations for metrology.

Además serán Miembros del SIM los países que voluntariamente participen a través de sus órganos nacionales de metrología".

Finalmente el SIM se constituye formalmente en la Reunión celebrada en Buenos Aires, con patrocinio de la OEA durante la gestión de Marcelo Alonso, Director del Departamento de Asuntos Científicos y organizada por el INTI, del 4 al 6 de septiembre de 1979, con participación de representantes de las entidades Metrológicas de Colombia, Chile, México, Guatemala, Venezuela, Bolivia, Costa Rica, Brasil, Argentina, Paraguay, Perú, Uruguay y Panamá.

El Primer Comité Coordinador fue integrado por:

Presidente: Prof. Rafael Steinberg, por Argentina.

Secretario: Dr, Armenio Lobo Da Cunha Filho, por Brasil

Vocales: Dr. Héctor Nava Jaimes, por México
Ing. Esmeralda Hernández, por Panamá
Ing. Ramón de Colubi, por Venezuela.

Finally, the SIM is formally constituted in the meeting held on Buenos Aires, under the sponsorship of the OAS, during the administration of Marcelo Alonso, Director of the Department of Scientific Issues, organized by the INTI on September 4 to 6, 1979, with the attendance of representatives of the metrological organizations of Colombia, Chile, Mexico, Guatemala, Venezuela, Bolivia, Costa Rica, Brazil, Argentina, Paraguay, Peru, Uruguay and Panama.

The first Coordinating Committee was composed by:

President: Prof. Rafael Steinberg, Argentina.

Secretary: Dr, Armenio Lobo Da Cunha Filho, Brazil

Vocales: Dr. Héctor Nava Jaimes, Mexico
Eng. Esmeralda Hernández, Panamá
Eng. Ramón de Colubi, Venezuela.

La foto muestra, de izquierda a derecha, a: Ing. De Colubi, Dr. Lobo Da Cunha Filho, Prof. Steinberg, Dr. Nava Jaimes e Ing. Hernández.

En otra reunión de coordinación del SIM, celebrada en el INTI, Argentina, el representante del National Bureau of Standards, NBS, hoy National Institute of Standards and Technologies, NIST, Dr. Goldman, manifestó que por expresa decisión del gobierno de los EEUU de Norteamérica el NBS no iba a participar en el SIM como Miembro asociado, aunque sí dijo que en el NBS estaban anuentes a recibir becarios para su capacitación en los laboratorios de metrología, como así también enviar expertos a los laboratorios de los países miembros, con cargo a los fondos del SIM.

Ante esta situación de no participación de los EEUU de NA dentro del SIM, surgió la idea de transformar el SIM en el "Sistema Iberoamericano de Metrología", con inclusión de España y Portugal, en virtud que dentro de los objetivos establecidos en el SIM, podrían participar

In the picture, from left to right: Eng. De Colubi, Dr. Lobo Da Cunha Filho, Prof. Steinberg, Dr. Nava Jaimes and Eng. Hernández.

In other coordination meeting of SIM, held at INTI, Argentina, Dr. Goldman, as representative of the National Bureau of Standards, NBS, now National Institute of Standards and Technologies, NIST, stated that by a decision of the government of the USA the NBS was not going to become a SIM member, but that there was any intention to provide training in its metrology laboratories, as well as to provide experts to the laboratories of the members, under the funding of the SIM.

Given this latter situation, it came up the idea to transform the SIM into a "Sistema Iberoamericano de Metrología", to include Spain and Portugal, since voluntary membership was permitted within the agreed objectives for the SIM.

también otros países: "Además serán Miembros del SIM los países que voluntariamente participen a través de sus órganos nacionales de metrología". (sic)

El Dr. Manuel Cadarso, quien participó como observador español, fue un gran impulsor de esa idea.
En 1982 el Dr. Cadarso propuso crear un Centro de Metrología en España y en marzo de 1989 se inaugura el Centro Español de Metrología, CEM, con presencia de los Reyes de España Don Juan Carlos I y Doña Sofía. Cabe señalar que el Dr. Cadarso era compañero de armas del Rey, quien hizo suya la idea de crear un Centro de Metrología en España y apoyó firmemente la iniciativa.

La idea de un nuevo SIM, también fue bien vista por el PTB de Alemania, ya que entonces una estructura supra-regional le serviría para canalizar la importante ayuda en equipamiento, expertos y becas que ofrecía, y ofrece, Alemania a América Latina. Así, a través del CEM se hubiera podido coordinar la cooperación técnica alemana, dentro de una nueva estructura del SIM.

Esta nueva estructura supra-regional no se pudo concretar, ya que el Dr. Cadarso falleció prematuramente por una dolencia cardiovascular y también porque dentro del SIM, el representante del Ecuador no estaba de acuerdo con un sistema iberoamericano, ya que decía que eso cerraba las puertas a una participación futura de los EEUU de Norte América.

Después de varios años, cambió la postura oficial de EEUU de Norte América y a través del actual NIST se revitalizó el SIM, con la incorporación también de Canadá y otros países del Caribe, conformando la estructura actual del SIM.

Pero esto merece otra historia.

Dr, Manuel Cadarso, participant as observer for Spain, was a great promoter of this idea.

In 1982, Dr. Cadarso proposed to create a Metrology Center in Spain, and in March 1989 the Centro Español de Metrología, CEM, started operations being the King of Spain Don Juan Carlos I present and so was the Queen Doña Sofía. It is highlighted that Dr. Cadarso and the King were arm-comrades so it was not difficult for the King to become sympathetic with the idea and support the initiative.
The new idea about the SIM also was very well received in the PTB, Germany, since this regional supra-structure was going to facilitate the provision of resources as equipment, experts and scholarships into Latin America. Thus, the German technical cooperation could be forwarded through the CEM

This supra-structure could not be completed since Dr. Cadarso passed away by a cardiac disease and also because the representative from Ecuador had not agreed with an Iberoamerican system that, he said, was closing out the future participation of USA.

After some years, the USA position changed and the SIM got revitalized, with the inclusion of Canada and other Caribbean countries as well.

But that deserves another story.

NOTI-SIM

Noticias en el SIM | *SIM News*

3rd. Tri-national Workshop on Standards for Nanotechnologies
Llevado a cabo en las instalaciones del CENAM, Querétaro, México, el 12 de febrero de 2009, con la participación de 18 conferencistas de Brasil, Canadá, Estados Unidos de América y de México, por lo que de hecho fue un maratónico taller tetranacional en nanotecnología. Los materiales pueden descargarse de http://www.cenam.mx/nwsn/ .

Taller SIM - PTB Relación de los Institutos Nacionales de Metrología con sus usuarios.
Proyecto conjunto de PTB, INMETRO, CENAM, OEA y el SIM, el taller se llevó a cabo del 2 al 5 de marzo de 2009, en Querétaro, México, con el objetivo de compartir algunas herramientas de acercamiento de los INM con la industria, a partir de las experiencias del CENAM.

3rd. Tri-national Workshop on Standards for Nanotechnologies
Held on February 12, 2009, in CENAM facilities in Queretaro, Mexico, actually became a tetra-national workshop with the participation of 18 lecturers from Brazil, Canada, USA and Mexico, a one-day marathon on nanotechnology. Download materials from http://www.cenam.mx/nwsn/

SIM - PTB Workshop NMI - Metrology User Relation
As a joint project by PTB, INMETRO, CENAM, OAS and SIM, the workshop was held on March 2-5, 2009, in Querétaro, México, aimed to share tools for NMIs to approach industries, taking some of the CENAM experiences as a basis.

Reunión del QSWG
Con la hospitalidad del CENAMEP en Panama, Panama, el 29 de marzo de 2009 el QSWG elaboró su plan de actividades orientado a las actuales, y cambiantes necesidades de los institutos nacionales de metrología del SIM en materia de sistemas de gestión de calidad.

Reunión del QSTF
Celebrada en Panamá, Panamá, el 30 de marzo de 2009, y en la cual se revisaron sistemas de gestión de calidad (SGC) para nuevas capacidades de medición y calibración (CMC) e iniciaron las revisiones quinquenales de los SGC requeridas por el CIPM-MRA.

Reunión del Consejo del SIM
Celebrado el 2 de abril de 2009 en las instalaciones de la OEA en Washington, D.C., el Consejo del SIM tomó la decisión, entre otras, de llevar a cabo la Asamblea General del SIM y otras actividades del mismo del 25 al 30 de octubre de 2009 en Perú.

VIII SEMETRO
El 8th. International Seminar on Electrical Metrology tuvo lugar del 17 al 19 de Junio, 2009 en la ciudad de Joao Pessoa, Brasil, bajo los auspicios del INMETRO y otras instituciones, y en el cual se presentaron aproximadamente 120 trabajos por autores de 21 países.
Previo al VIII SEMETRO se realizaron cuatro cursos:

QSWG meeting
Hosted by CENAMEP in Panama, Panama, on March 29, where a plan of activities was elaborated aimed at supporting the present, and changing, needs of the SIM NMIs to strengthen the quality management systems supporting assessed CMCs.

QSTF meeting
Held in Panama, Panama, on March 30, 2009, reviews of quality management systems (QMS) for new CMCs were done and it was started the 5-year general reviews of the QMS as required by the CIPM-MRA.

SIM Council meeting
Held on April 2, 2009 in the OAS premises in Washington, D.C., among others, the SIM Council decided to use October 25-30 for the SIM General Assembly and related SIM events in Peru.

VIII SEMETRO
The 8th. International Seminar on Electrical Metrology held on June 17-19, 2009 in the city of Joao Pessoa, Brazil, organized by INMETRO and other institutions, where approximately 120 papers were presented by authors of 21 countries.
Prior to the VIII SEMETRO, four courses were delivered:

"DC resistance Measurements", impartido el NIST.

"AC-DC Thermal Transfer Standards and Calibrations" impartido por el NRC.

"Mediciones de Potencia y Energía" impartido por el UTE. "

"Mediciones de tensión eléctrica de alta exactitud" impartido por el CENAM.

Reunión del grupo de trabajo de electricidad y magnetismo del SIM
Se informó del término de la comparación SIM.EM-S2 en energía eléctrica para la cual el NIST fungió como piloto y cuyos resultados se publicaron en el número anterior del INFOSIM. Los resultados de la comparación sobre AC/DC están ya publicados en la KCDB del BIPM. Otras comparaciones en proceso incluyen capacitancia, multímetros, resistencia e inductancia. Un número importante de propuestas de comparación están siendo actualmente consideradas.

Comparación de masa en CAMET
Del 17 al 21 de noviembre de 2008, se realizó en LACOMET, Costa Rica, la reunión de inicio del estudio piloto para mediciones de masa de laboratorios nacionales de la región de CAMET. El CENAM participa caracterizando los patrones viajeros y aportando el valor de referencia para dicho ejercicio.

PRÓXIMAMENTE …

Third Tri-National Conference of the North American Coordinate Metrology Association and Sexta Reunión - Taller de la Asociación Mexicana de Metrología de Coordenadas
Promovida por Canadá, Estados Unidos de América y México, se celebrará en Querétaro, México, los días 17 y 18 de septiembre, 2009, con conferencistas de Brasil, Canadá, Estados Unidos de América, Francia, Alemania y México; así como exhibición de equipo.
Información adicional en www.cenam.mx.

Reunión del QSTF
A celebrarse en Lima, Perú, los días 27 y 28 de octubre de 2009.

SIM Metrology School
A celebrarse en la región de Rio de Janeiro, Brasil, tentativamente del 9 al 16 de diciembre de 2009.
Más información aparecerá pronto en http://www.sim-metrologia.org.br/

4th. Tri-national Workshop on Standards for Nanotechnologies,
Por realizarse los días 3 y 4 de febrero de 2010 en Ottawa, Canadá.
Mayores informes Jennifer.Decker@nrc-cnrc.gc.ca
Adicionalmente, se reunirá el grupo de trabajo que elabora la norma sobre rejillas artificiales del ISO TC 229 Nanotechnologies.

"DC resistance Measurements" by NIST.
"AC-DC Thermal Transfer Standards and Calibrations" by NRC.
"Mediciones de Potencia y Energía" by UTE.
"Mediciones de tensión eléctrica de alta exactitud" by CENAM.

***Electricity and Magnetism Working Group Meeting**,*
Among others, it was reported that the conclusions of the comparison SIM.EM-S2 on electrical energy piloted by NIST was reported in INFOSIM. The AC/DC piloted by CENAM was also completed and reported in the BIPM KCDB. Other comparisons going on include capacitance, multimeters, resistance and inductance. A number of proposals for other comparisons are being considered.

Mass comparison in CAMET
The starting meeting for the pilot study on mass measurements aimed to the members of CAMET took place on November 17-21, 2008 in LACOMET, Costa Rica. CENAM will measure the traveling standards and will provide the reference value for the study.

ONCOMING …

Third Tri-National Conference of the North American Coordinate Metrology Association and Sexta Reunión - Taller de la Asociación Mexicana de Metrología de Coordenadas.
This Conference is jointly promoted by Canada, Mexico and the United States to be held on September 17-18, 2009, in Queretaro, Mexico. Speakers will attend from Brazil, Canada, France, Germany, USA and Mexico. An equipment exhibit will also be part of the event.
Further information on www.cenam.mx.

QSTF meeting
To be held in Lima, Peru on October 27 and 28, 2009.

SIM Metrology School
To be tentatively held in the area of Rio de Janeiro, Brazil, on December 9-16, 2009. Further information coming soon on http://www.sim-metrologia.org.br/.

4th. Tri-national Workshop on Standards for Nanotechnologies,
To be held on February 3 and 4, 2010 in Ottawa, Canada.
Further information Jennifer.Decker@nrc-cnrc.gc.ca
This event will also provide an opportunity for the ISO TC 229 Nanotechnologies working group on the standard about artificial gratings to meet.

SOBRE LA POSIBLE REDEFINICIÓN DEL KILOGRAMO

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El kilogramo, unidad de masa del Sistema Internacional de Unidades (SI), es la única unidad cuya definición se mantiene sin cambio desde su adopción en 1901.

Un kilogramo esta definido como la masa igual a la del *prototipo internacional del kilogramo*, que es un cilindro de metal (Pt-Ir). Este cilindro de 39 mm de altura por 39 mm de diámetro, por ser un objeto, esta expuesto a daño físico y a la contaminación, lo cual es una amenaza para la conservación de la masa que define y por lo tanto, a la referencia de esta unidad de base del SI [1].

Fig. 1. Prototipo Internacional del kilogramo conservado en la Oficina Internacional de Pesas y Medidas ubicada en Sèvres Francia.

Debido a ello se han propuesto alternativas en donde la definición de la unidad de masa esté basada en alguna constante física que pueda reproducir el valor de esta magnitud con una incertidumbre aceptable para las necesidades de los usuarios finales, (p.e. en control de procesos o compra venta de productos, etc.).

Existe la posibilidad de que la nueva definición del kilogramo se adopte en la 24ª reunión de la Conferencia General de Pesas y Medidas en el 2011 [2], y para ello son dos experimentos que llevan la delantera en la tarea de redefinir el kilogramo, el primero de ellos es conocido como el experimento de la balanza de Watt y el otro es conocido como el proyecto de la constante de Avogadro [1].

El experimento de la balanza de Watt consiste en la comparación de la potencia mecánica con la potencia eléctrica, este proyecto se encuentra activo actualmente en el NPL-Reino Unido[1], en el NIST-Estado Unidos, METAS-Suiza, LNE-Francia y en el BIPM-Oficina Internacional.

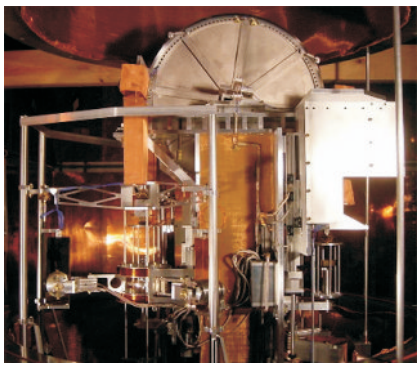

Fig. 2. Balanza de Watt del NIST- Estados Unidos.

[1] *La balanza de Watt del NPL-Reino Unido será transferida al NRC-Canadá en el transcurso del 2009.*

El proyecto de la constante de Avogadro consiste en definir el kilogramo como un número definido de átomos de algún material, p.e. carbono 12. Este es un proyecto multinacional que trabaja bajo la supervisión del Grupo de Trabajo de la Constante de Avogadro (WGAC) del Comité Consultivo de Masa (CCM).

La determinación experimental de la constante de Avogadro vía el silicio, esta basado en mediciones a esferas de monocristal de silicio de aproximadamente 1 kg.

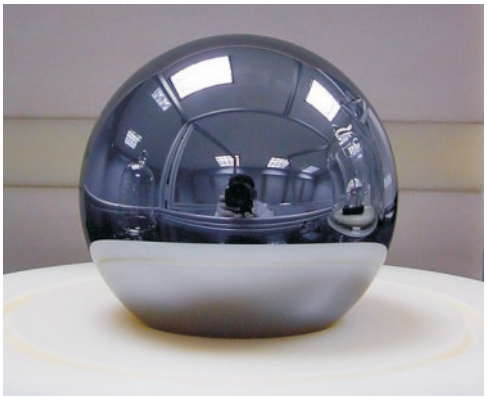

Fig. 3. Esfera de monocristal de silicio.

Actualmente existe una discrepancia del orden de una parte en un millón (1x10-6) [2] entre los resultados obtenidos en ambos experimentos, y debido a ello el CCM recomendó que previo a la redefinición de la unidad de masa, esta discrepancia debe ser resuelta y que la incertidumbre de la realización del kilogramo sea igual o menor a 2 x 10-8 [2].

Una más de las recomendaciones del CCM, es que la nueva definición del kilogramo debe mantener la referencia de la definición actual, lo cual implica que al nivel de los usuarios finales, Ud. o yo, cuando compremos un kilogramo de cualquier producto, siga siendo en esencia la misma cantidad de masa que hemos estado recibiendo. En cada país, los resultados de estas mediciones de masa seguirán siendo trazables al valor del patrón nacional de masa correspondiente, sólo que el valor de éste muy probablemente será determinado por la realización de alguno de estos dos experimentos: La balanza de Watt o el proyecto de la constante de Avogadro.

Fig. 4. Patrón Nacional de Masa de México, prototipo de platino iridio k21 conservado en el CENAM.

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[1] Mill I. M. et al. Redefinition of the kilogram: a decision whose time has come, Metrologia 42 (2005) 71-80

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Ligas de interés

http://www.nist.gov/public_affairs/releases/electrokilogram.htm

http://www.bipm.org/en/scientific/elec/watt_balance/

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