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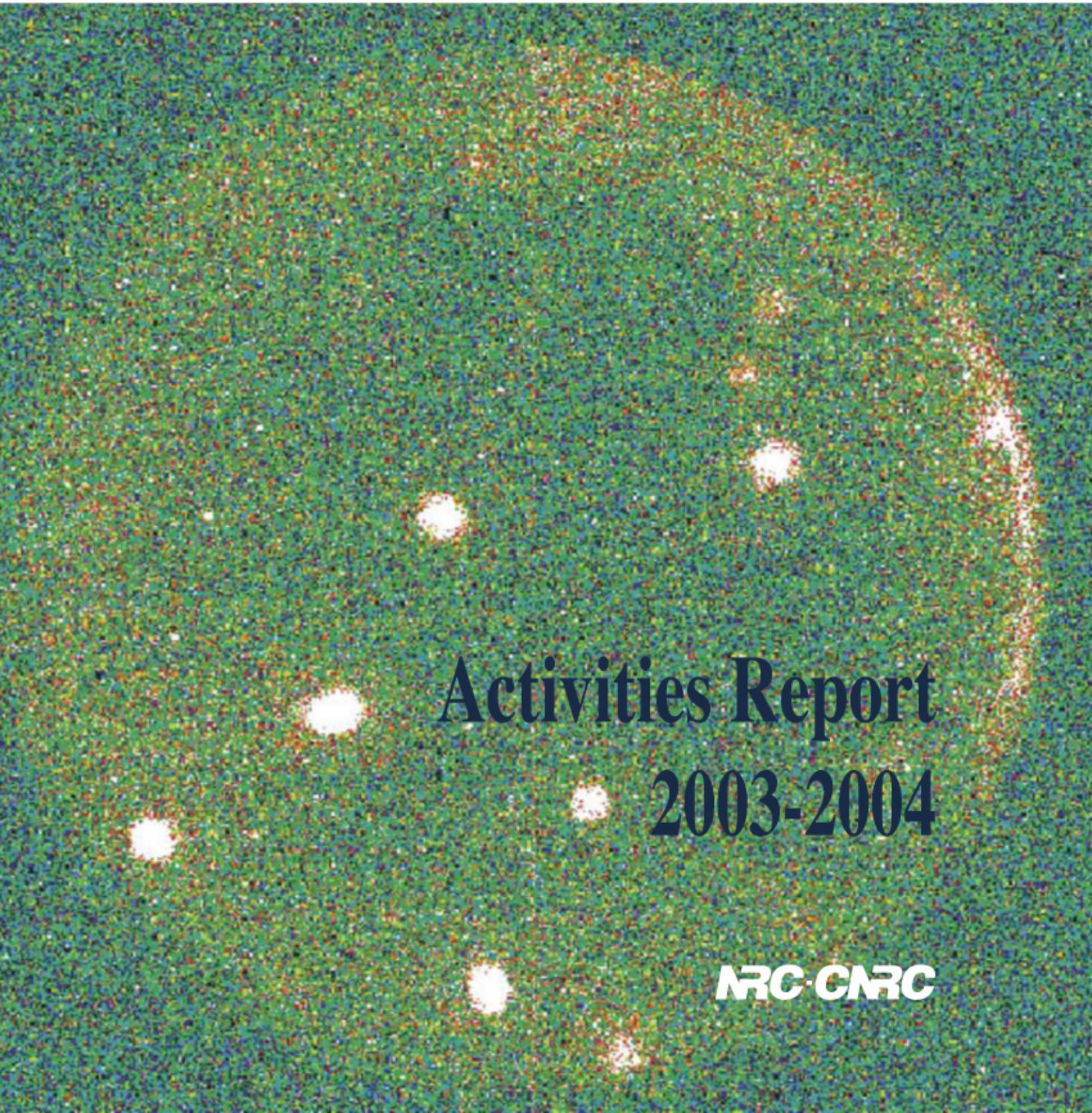
NRC-Institute for National Measurement Standards



Activities Report 2003-2004

NRC·CNRC

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NRC-Institute for National Measurement Standards

Institute Mission and Role

As the national metrology institute, INMS strengthens Canada's innovation and competitiveness, supports international trade, and advances social well-being by providing the technical and infrastructural foundation for the national measurement system.

The Institute's physical metrology programs develop, maintain, improve, and disseminate standards for the base quantities of mass, length, time, electricity, temperature and luminous intensity as well as a number of derived measurement standards. The chemical metrology program develops and maintains world-class capabilities in selected areas of organic and inorganic trace analysis, and provides certified reference materials.

Traceability of all measurement in Canada to these measurement standards provides the basis for fair trade, development in science and technology, the achievement of product quality, and demonstration of conformance to international quality standards.

INMS also supports the development of competencies and technology in selected areas of optical science, technology, and measurements, as well as in selected areas of photonics technology related to high-performance computers.

NRC's Uncertainties

(95% confidence level)

absorbed dose	0.010
acoustical pressure	5×10^{-5} db
frequency	1×10^{-13}
length	5×10^{-11}
luminous intensity	0.01
mass	4×10^{-8}
resistance	4×10^{-8}
temperature	0.2 mK
voltage	4×10^{-10}

Director General's Foreword

I am pleased to introduce this report on the activities of the NRC Institute for National Measurement Standards for the period from April 2003 to March 2004. Despite a number of challenges, the high calibre of the staff the Institute has resulted in a sustained level of excellence and recognition that is clearly evident from the pages that follow.

In addition to activities in traditional "core" areas of metrology, the INMS Strategic Plan approved by NRC Council in June 2002 calls for initiatives in four new metrology areas related to biotechnology, natural health products, electrical power measurement and nanotechnology. The most successful "new metrology" initiative to date has been the natural health products (NHPs) project of the Chemical Metrology Group. New equipment valued at 1.65M$ was commissioned and a strong team formed that allowed INMS to make a very significant technical contribution to the world's first certified reference materials for ephedra. The team has now initiated production of reference materials related to ginseng.

Synergy and collaboration between INMS and NRC's Institute of Microstructural Sciences remains strong. A partnered project focusing on absolute optical frequency measurement in the optical telecommunications spectrum has achieved significant advances in the development of a mode-locked Cr^{4+}:YAG femtosecond laser system. In the Photometry and Radiometry area, joint research has shown that far-infrared polarized regular reflectance at oblique incidence is a powerful method to investigate the phonons and other phenomena in thin films. This latter work was the subject of thirteen of the 108 papers published by INMS in 2003 in refereed journals and conference proceedings.

As a signatory to the CIPM Mutual Recognition Arrangement, INMS is committed to the establishment of a quality system that complies with ISO/IEC 17025, the international quality standard for calibration and testing laboratories. During the reporting period, two INMS groups underwent the external audits that will lead to formal accreditation; three more groups are scheduled for 2004/2005, with the remainder in 2005/2006. At the same time as many INMS staff were heavily engaged in the preparation of quality systems documentation, several of them also participated as technical assessors at NMIs in Brazil, France, Japan, Korea, Mexico and South Africa.

INMS continues its strong commitment to client services, metrology outreach and training. Three hundred and twenty-seven clients received 962 calibration reports, two hundred and ninety customers purchased 1490 units of Certified Reference Materials, and forty-two Glow Discharge Mass Spectrometry clients had 2098 samples analyzed. Highly-praised courses in mass metrology, dimensional metrology, coordinate metrology, and ionizing radiation standards were presented during the year.

The diversity of our activities not only demonstrates the expertise of INMS metrologists but their dedication to the dissemination of knowledge and technology to industry and individuals.

James McLaren
Director General (acting)

DIRECTOR GENERAL

Dr. James W. McLaren (acting)

DIRECTORS

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Photometry & Radiometry
Photonic Systems

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Frequency & Time
Mechanical Metrology

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HR Systems Coordinator	Betty Rodriguez	993-3918

INMS and International Cooperation

Reducing Technical Barriers to Trade

With international trade agreements now demanding demonstrated equivalence between the measurement standards and accreditation systems of buyer and seller nations, metrology has become vital to the regulation of trade and in the resolution of trade disputes. This has led to the creation of metrology-related cooperations supporting regional and international trade agreements, including:

- Asia Pacific Laboratory Accreditation Cooperation (APLAC), supporting APEC
- Asia Pacific Metrology Programme (APMP), supporting APEC
- Inter-American Metrology System (SIM), supporting FTAA
- International Laboratory Accreditation Cooperation (ILAC), supporting WTO
- North American Cooperation in Metrology (NORAMET), supporting NAFTA
- North American Calibration Committee (NACC), supporting NAFTA

INMS is a member of SIM, NORAMET and NACC and an associate member of APMP. It also plays an essential role in the Standards Council of Canada (SCC)'s membership in APLAC and ILAC. INMS is active in these organizations and in approximately 150 international committees under the auspices of global bodies such as the Comité International des Poids et Mesures (CIPM). A key element in these activities is the international comparison of measurement standards and calibration services.

International Comparisons

The reliability of the international measurement system depends on continued effort by each National Metrology Institute (NMI) to base its measurements and measurement uncertainties on universally accepted units, usually those of the SI, and to compare its measurements with those of other NMIs to establish their mutual equivalence. INMS, as Canada's NMI, participates regularly in measurement comparisons with other NMIs. In many cases these are multilateral comparisons coordinated by the Bureau International des Poids et Mesures (BIPM) and other bodies such as SIM or NORAMET, but bilateral comparisons are also undertaken.

The purpose of these comparisons is to ensure that realizations of the SI units by participating NMIs are equivalent within known uncertainties, thus creating a uniform global metrology system. The participation of INMS enables increased competitiveness of Canadian industry within the global market.

In order to extend and document the practice of comparisons and declarations of equivalence, NMIs throughout the world have signed a Mutual Recognition Arrangement (MRA) under the auspices of the CIPM. The MRA provides for formal mutual recognition of national measurement standards and calibration capabilities including measurement capabilities related to certified reference materials. It is expected to become the basis for wider agreements relating to trade and commerce signed by the competent authorities in each country or region. In October 1999, the Director General of INMS signed the MRA alongside the directors of NMIs in 37 other countries. Forty-nine NMIs and two international organizations are now signatories.

Under the MRA, INMS scientists participate in an increasing number of measurement comparisons with other NMIs. Each inter-NMI comparison requires several years to plan, implement, document and establish equivalence. INMS participated in the planning or implementation of some 37 such comparisons in pursuit of the MRA aims during 2003-2004. In addition, INMS was involved in the planning or implementation of some eight comparisons under the auspices of SIM. INMS is also involved in the extensive

and on-going international review process that takes place following the completion of the comparisons to determine which calibration and measurement capabilities can be recognized for inclusion in Appendix C of the BIPM key comparison database (KCDB) and the parallel SIM database, known as the ICDB. The review process looks at the results of the comparisons, at quality systems, and at other appropriate evidence. Review of quality systems involves assessment by peers from other NMIs: INMS scientists are in high demand around the world, visiting Australia, Japan, South-Korea, South Africa, USA, Brazil, France, Mexico, and New Zealand in the reporting period.

In addition to these activities, INMS provides the NORAMET representative on the SIM Technical Committee and participates in all relevant consultative committees of the CIPM. This participation has enabled us to protect and enhance Canada's interests by direct involvement at the highest level. This benefit affects virtually all of Canadian industry involved in the export of products to countries with which agreements have been reached, as well as customers of products imported from these countries.

Accreditation Systems

Canada is also signatory to two important international MRAs related to laboratory accreditation, those of APLAC and ILAC. As a result of these arrangements, all signatory countries now recognize calibration certificates issued by CLAS-certified, SCC-accredited calibration laboratories. Likewise, CLAS and SCC recognize laboratories accredited by the other MRA countries. Canadian participation in these international and regional arrangements provides the infrastructure needed for Canadian industries to have their goods and services accepted in a client country without the need for re-calibration.

Traceability

The concept of traceability is fundamental to a unified system of metrology. Traceability is defined as the property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties. In most cases, the ultimate reference for a measurement result is the definition of the appropriate unit in the International System of Units (SI). However, the stated reference is usually an NMI, such as NRC or NIST, or, more precisely, a national standard maintained by an NMI. This is an appropriate and practical way of stating traceability and reflects the usual chain of measurement comparisons (calibrations) that exist in a country.

Thus, "traceable to NRC" or "traceable to NIST" are phrases that are often used in regulatory requirements, in contracts, or even in advertising. These phrases indicate that a measurement is related to a particular primary realization of an SI definition. However, the purpose of the SI is to provide a practical system of units that can be adopted by all countries, be reliable and consistent over the long term, and lead to measurements that are comparable throughout the world. Thus, 'traceability to an NMI' is really a shorthand notation for traceability to the SI through an NMI. Although many NMIs have independent realizations of the SI units, the intent is always the same–to realize the SI definition (or, in some cases, an agreed representation such as the Josephson volt). The uncertainty with which the definition is realized is a component of the uncertainty of an NMI calibration and, thus, measurements in different NMIs should all agree with each other within their quoted uncertainties. With the increasing globalization of trade, this concept is becoming more and more important and is superseding the idea that each country's primary standards should be thought of as separate independent sources of traceability. Thus, INMS documents and calibration reports now refer to 'traceability to the SI through NRC' rather than 'traceability to NRC'.

INMS Quality System

As a signatory to the CIPM Mutual Recognition Arrangement, INMS is committed to the establishment of a quality system that complies with ISO/IEC 17025, the international quality standard for calibration and testing laboratories. The MRA provided a transition period, with a 31 December 2003 deadline, for NMI compliance with the requirement that all CMCs published in the BIPM database must be supported by a quality system.

Under the rules of the MRA, a quality system must be reviewed by the appropriate Regional Metrology Organization. In the case of INMS, this is SIM, which in October 2002 set up a Task Force to review quality system implementation. The Task Force reports its findings to the SIM Council and the SIM Council reports this information to the Joint Committee of the Regional Metrology Organizations and the BIPM (JCRB). The Task Force held its first meeting in July 2003 and defined SIM procedures to approve NMI implementations of quality systems: at the February 2004 meeting, several SIM NMIs presented their quality system for approval.

In May 2003, INMS approved an action plan for the accreditation to an ISO/IEC 17025 quality system supporting all calibration and measurement capabilitites (CMCs). The plan involves three implementation phases leading to full compliance by the last quarter of 2005.

In 2003-2004, the INMS Photometry and Radiometry Group and the Acoustical Standards Program implemented their parts of the system: written documentation was accepted and internal audits were completed. The Standards Council of Canada commenced the assessment process for the accreditation of these two groups: Mary Ryan from the National Accreditation Testing Authorities (NATA) in Australia was the team leader, with technical experts from France, USA, Germany and Australia.

Other quality system activities included continued training in ISO/IEC 17025 internal audit techniques. In addition, numerous improvements have been made, based on the internal audits,on-site accredition assessments and the work of the Quality System Steering Committee. The following procedures were developed:

- INMS-105: *Maintenance and Updating of the Controlled Electronic Version of the INMS Quality System* (approved)
- INMS-114: *Requirements for the Protection of the Integrity and Confidentiality of Calibration Reports and Associated Data in Electronic Transmissions*. (approved)
- INMS-117: *Review of Requests, Tenders and Contracts (*draft)
- INMS-118: *Collection and Maintenance of Technical Records* (draft).

In July/August 2003, the Institute organized and hosted a workshop on the implementation of quality systems in NMIs as part of a joint SIM-APMP project to write test methods in generic terms to assist in the implementation of quality systems especially in developing NMIs. The project will identify the technical methods to be written, specify suitable traceability pathways and uncertainty estimations, and identify the appropriate method-validation procedures.

INMS and Services to Canadian Industry

The rich resource of INMS facilities and expertise are available to industry through calibration services and certified reference materials, consultancy, collaborative research, and the transfer of technology. In addition, the institute organizes metrology courses and workshops, delivers presentations at conferences, and steers professional technical associations.

Calibration Services

Maintaining high measurement accuracy requires regular calibration of industrial measuring devices to physical reference standards such as gauge blocks and standard resistors. There are two avenues to this end: reference standards can be calibrated by NRC specialists against Canada's primary standards maintained at INMS, or reference standards can be calibrated at one of thirty-two CLAS (Calibration Laboratory Assessment Service) certified and SCC (Standards Council of Canada) accredited laboratories across Canada. During 2003-2004, 962 calibration reports were issued to 327 calibration clients.

Certified Reference Materials

Total sales of certified reference materials (CRMs) declined in the reporting year, possibly as a consequence of increased global competition from other producers, or a weaker economy: 1490 units were distributed, about 12% less than in the previous year. Sales of biological tissue CRMs for trace elements rose by 3% to 471 units and the four sediment CRMs totaled 224 bottles, about the same as last year. Almost 23,000 units of reference materials have been distributed since 1981 with our six natural water CRMs accounting for over half of this total.

Glow Discharge Mass Spectrometry

The glow discharge mass spectrometry (GD-MS) facility, unique in Canada, provided elemental analysis of approximately 2100 samples of high purity conducting and semiconducting materials (e.g., gallium, cadmium, tellurium, gallium arsenide, cadmium selenide, cadmium zinc telluride) for about 42 clients, mostly in the electronics industry. The Standards Council of Canada has fully accredited the laboratory to ISO/IEC 17025.

Calibration Laboratory Assessment Service

National calibration laboratory accreditation systems provide infrastructural sources of calibration services with verified measurement capabilities and traceability to national and international measurement standards. Canada's calibration laboratory accreditation program is managed and facilitated as a partnership between the SCC Program for the Accreditation of Laboratories-Canada (PALCAN) and the NRC Calibration Laboratory Assessment Service (CLAS).

CLAS assesses the quality systems and the measurement capabilities of calibration laboratories seeking SCC accreditation. Through a combination of proficiently testing by traceability and by an exchange of measurement artifacts, CLAS determines whether the applicant laboratory is capable of making the measurements for which it is seeking accreditation. Once satisfactory implementation of the quality system has been confirmed and the measurement capability has been verified with the assistance of INMS measurement experts, the laboratory becomes eligible for a CLAS certification and for SCC accreditation to ISO/IEC 17025.

The implementation of CLAS at INMS was an important step in the establishment of a network of Canadian Calibration Laboratories that ensures an easier access to calibration

services with certified traceability to the International System of Units (SI). This traceability provides the basis for fair trade, increased global market acceptance of test reports and products, enhanced product quality, and demonstration of conformance to such international quality standards as ISO 9000. In 2003-2004, six new Canadian calibration laboratories were certified by CLAS and accredited by SCC/PALCAN. In addition, there are ten laboratories at various stages of the assessment process. Currently, there are 32 CLAS-certified/SCC-accredited laboratories.

CLAS operates under a Quality System that has been in place since 1999. Continuous management reviews take place and result in improvements to the system such as the implementation in 2003 of a management information system database to control the activities of the program. In April 2003, SCC audited the CLAS program: continued compliance of the CLAS program to ISO Guide 58 was confirmed.

AceTRONIC Industrial Controls, Inc.
Mississauga, ON

Alcan International Ltd. - Kingston Research & Development Centre
Kingston, ON

CCR – MKS Process Products Group
Nepean, ON

Environment Canada (NWRI)
Burlington, ON

Fisher Scientific Company, Montreal Region
St-Laurent, QC

Fisher Scientific Company, Toronto Region
Markham, ON

Fisher Scientific Company, Edmonton Region
Edmonton, AB

Fisher Scientific Metrology
Nepean, ON

Fluke Electronics Canada Inc.
Mississauga, ON

Gavtt Precision Balances Limited
Mississauga, ON

Industrial Technology Centre
Winnipeg, MN

Industry Canada Certification and Engineering Bureau
Ottawa, ON

Instruments Canada Company Ltd.
Mississauga, ON

Institut de recherche Robert-Sauvé en santé et en sécurité du travail (IRSST)
Montréal, QC

IREQ Institut de recherche d'Hydro-Québec
Varennes, QC

ISOLAB Inc.
Windsor, ON

Labcor Technical Sales Inc.
Anjou, QC

Measurement International
Prescott, ON

Miller Instruments Ltd.
Burnaby, BC

Mitutoyo Canada Inc. Calibration Laboratory
Mississauga, ON

Primo Instrument Inc.
Montréal, QC

Pylon Atlantic - A Division of Pylon Electronics Inc.
Dartmouth, NS

Pylon Electronics Inc.
Mississauga, ON

Pylon Electronics Inc.
Ottawa, ON

Rohde & Schwarz Canada Inc.
Kanata, ON

Staveley Services Canada Inc. (CONAM-Quantum Inspection and Testing)
Burlington, ON

Staveley Services Montreal (CONAM-Quantum Inspection and Testing)
Dollard-Des-Ormeaux, QC

Stephens Analytical Inc.
St-Laurent, QC

Stephens Analytical Inc. (U.S.)
Newtown Square, PA

Technisol Inc.
Québec, QC

Ulrich Metrology Inc.
Lachine, QC

VACS Ltd.
Brampton, ON

For more information on the SCC Accredited/CLAS Certified Calibration Laboratories please visit our web site at http://inms-ienm.nrc-cnrc.gc.ca/clas/directory_e.html.

INMS and the Transfer of Technology

As Canada's National Metrology Institute, the core role of INMS is in innovation support. However, in the course of its fundamental activities, new techniques and technologies are often developed that have commercial applications.

Thirty-five license agreements were active at the end of the reporting period and six new patents were filed or issued.

Existing Licenses	*Licensee*
ELECTROMAGNETIC AND TEMPERATURE STANDARDS SECTION	
Replicator Circuit	Measurements International Ltd.
Digital to Analog Converter	Measurements International Ltd.
	Guildline
Electronically Aided Current	Guildline
	Pacific Magnetics
High Voltage Capacitance Bridge	Measurements International Ltd.
Time Division Multiplier Wattmeter	Measurements International Ltd.
AC/DC Transformer	Guildline
Transimpedance Circuit	Measurements International Ltd.
Automated Resistance Bridge	Measurements International Ltd.
AC Source	Measurements International Ltd.
Power Comparator	Measurements International Ltd.
One Ohm Standard	Measurements International Ltd.
Quantum Hall Resistor System	Measurements International Ltd.
CHEMICAL AND MECHANICAL STANDARDS SECTION	
Acoustical Isolation Cap for Sound Level Meters	ACO Pacific Ltd.
FAIMS Technology (13 patents)	Ionalytics Corp.
RADIATION STANDARDS AND OPTICS SECTION	
Beam Version x.x.	NOMOS Corp.
Beam 00 (or Beam NRC)	Varian Medical Systems Int'l.
	ADAC Lab.
	Elekta Oncology Sys.
Source Code for Monte Carlo Electron Beam Dose Calculations	Nucletron Operations BV
Source Code for Monte Carlo Photon Beam Dose Calculations	Nucletron Operations BV
	Varian Medical Systems Int'l.
Infrared Spectrophotometer Detector	Mono Instrument Services Ltd.
Silicon Coated Mylar Beam Splitter	YueWang Tech Trading
Laser Prism	Cemar Electronic
	StockerYale Inc.
Laser Beam Cross Lens	StockerYale Inc.
Single Element Laser Beam D Shape Projectors	StockerYale Inc.

Patent application and filing / 2003-2004	*Inventor*
Compact hybrid integrated polarization insensitive optical dynamic channel gain equalizer	G. Xiao, Z. Zhang, Z. Lu, J. Liu, C. Grover
Light Induced Generation of Volatile/Alkylated Metals and Semi-Metal	X. Guo, R. Sturgeon, Z. Mester
Multilabeling Biomolecules with Dendritic Labelling Reagents with Multiple Ruthenium Bipyridine Moieties for Bionanlysis	M. Zhou, C. Roovers, C. Grover
Novel Technology for Full-Field Optical Coherence Tomography and Its Application to Multiple-Layer Information Decoding	S. Chang, X. Liu, C. Grover
Spectral Coding by Fluorescent Semiconductor Nanocrystals for Document Identification and Security Applications	S. Chang, M. Zhou, C. Grover
High Performance Optical Tracking System Based on a Quartic Phase Plate	X. Cai, X. Liu, C. Grover
Object Identification Using Quantum Dots Fluorescence Allocated on Fraunhofer Solar Spectral Lines	S. Chang, M. Zhou, C. Grover
Apparatus and Method for Atmospheric Pressure 3-Dimensional Ion Trapping *	R. Guevremont

TANDEM FAIMS/Ion-Trapping Apparatus and Method*	D. Bennett, R. Purves, R. Guevremont
FAIMS Apparatus and Method with Laser-Based Ionization Source*	D. Bennett, R. Purves, R. Guevremont
Highly Accurate Multistage Clamp-on Current Transformer*	E. So, D. Bennett
Acoustical Isolation Cap for Sound Level Meters*	G.S.K. Wong, N. Lewis
Atmospheric Pressure Ion Storage and Focusing Device*	R. Guevremont, R. Purves

* patents issued

INMS and the Dissemination of Knowledge

Information dissemination ensures that the results of our work are available to our scientific colleagues, and to the technical and business communities likely to benefit most.

Publications and Presentations

The excellence of INMS research is the core of its value to its partners. During 2003, 108 publications were published in refereed journals and conference proceedings. In addition, in the reporting period, 718 technical reports were written. The excellence of our staff is further evident in invitations to present at conferences and in the adjunct professorships that are held. A summation of these accomplishments is provided in each of the functional Group reports contained in this annual activities review.

Courses and Technical Associations

The Institute for National Measurement Standards holds tutorial courses on the measurement of basic physical quantities. These courses are concerned not only with the various primary and derived physical standards but also include discussion of fundamental principles and related measurement techniques with emphasis on high accuracy.

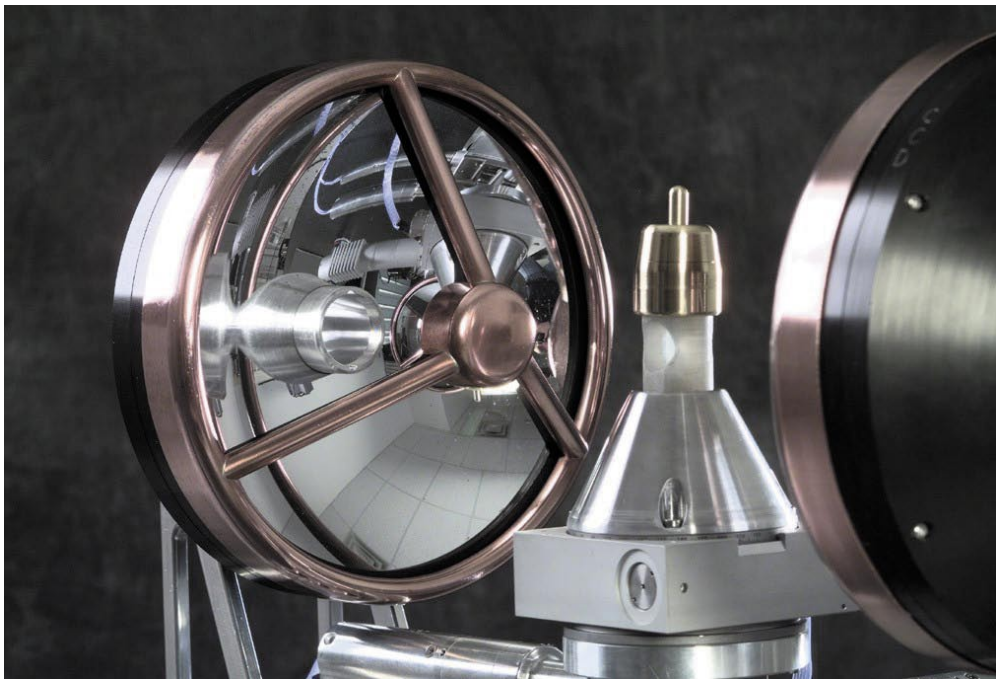
During 2003-2004 three courses were provided at our facilities in Ottawa:

✧ Mass Metrology Course	January 2004
✧ Dimensional Metrology Course	November 2003
✧ Coordinate Measuring Machine Workshop	November 2003
✧ Radiation Transport Calculations using EGSnrc Monte Carlo System	November 2003
✧ National Conference of Standards Laboratories International	October 2003

One hundred and thirty-nine persons from six countries participated in the INMS courses—eight-one from industry, ten from universities, and forty-eight from government.

In addition to INMS organized and delivered courses, Institute staff are key players in the Association for Coordinate Metrology Canada. This specialized users club draws participants mainly from the automotive, aerospace, and tool and die sectors. Coordinate measuring machines (CMM) are an integral part of the inspection process in virtually every production requiring dimensional control and CMM calibration is an integral factor in the Big Three auto makers' QS-9000 supplier quality program. CMM users convene once a year to share technical measurement issues with experts from INMS and counterpart institutes in other countries, Canadian universities and colleges, and industry.

Electrical Power Measurements



The Electrical Power Measurements Group has developed a system to detect emission of light from points of electric stress enhancement, i.e. electroluminescence (EL). The onset of EL marks the onset of electrical aging in dielectrics subjected to high voltages and thus EL measurements could be used to screen materials for high voltage applications. The figure shows special parabolic mirrors used to enhance the detection of a very faint light of electroluminescence.

The Electrical Power Measurements Group maintains standards, provides calibration services, develops instrumentation and testing procedures, and investigates new technology in the area of electrical power measurements. The Group focuses on meeting present and future requirements of the Canadian electrical power industry for precise measurements and in particular develops new state-of-the-art instrumentation and measurement techniques including:

- special revenue meters for electric utilities
- calibration facilities for equipment such as instrument transformers, active/reactive power and energy meters under sinusoidal/nonsinusoidal waveform conditions, high voltage capacitors, high current resistors and shunts (ac and dc), and high voltage loss measurement systems for transformers and inductive reactors
- diagnostic techniques and instrumentation to assess the operating conditions of high power apparatus in service.

The Group also provides basic knowledge relating to national and international standards for electrical power measurements and diagnostic and maintenance techniques for high power apparatus.

Electricity Metering

D. Angelo, R.J. Arseneau, B. Djokic, M. Frigault, E. So, J. Zelle

This project involves the research and development of unique and more accurate measurement techniques and instrumentation to precisely measure electrical quantities of interest to the electrical power industry (e.g. voltage, current, and active/reactive power and energy). The techniques include automated systems for the calibration of the corresponding meters under sinusoidal and non-sinusoidal waveform conditions and instrumentation for the calibration of systems/ instrumentation used in power quality related tests.

We used the three-phase, non-sinusoidal calibration system for calibration and testing of equipment of external clients including tests at 25 and 50 Hz. A study of the possible implications of applying the Institute of Electrical and Electronics Engineers (IEEE) Standard 1459-2000 on metering charges was conducted with the system and the results presented at the 2003 IEEE Power Engineering Society Summer Meeting in Toronto. In collaboration with BC Hydro, a harmonic producing load was investigated. The measured waveforms were reproduced with the calibration system to determine the most appropriate metering equipment for the site. These activities contribute to accurate and traceable measurements in the electricity trade sector in Canada.

NRC Traveling Standard

R. Arseneau, M. Frigault, J. Zelle

We have completed the automation of the on-site test procedure and data analysis, as well as a new, high accuracy transfer device for the Traveling Standard Program. The new instrument is based on two sampling voltmeters, custom built voltage and current input circuits, and a computer. The equipment was used for on-site tests at Canadian electrical utilities metering laboratories in Manitoba and Quebec. In April 2003, IEEE Transactions on Instrumentation and Measurements published our paper describing this new transfer device.

The sampling system standard will be applied to other on-site measurements particularly testing related to losses in power transformers. The goal is to achieve accurate measurements in a shorter time, in order to minimize the interruptions of the production schedules of clients in the utility and power transformer sectors.

Measurement and Correction of the No-load Losses of Power Transformers

R. Arseneau, M. Frigault, E. Hanique (SMIT Transformers), E. So

No-load losses of power transformers represent a significant operating expense for utilities. Correct measurements of these losses are therefore extremely important for transformer manufacturers as they are often the determining factor in the utility's selection of supplier. This project was conducted in cooperation with an NRC client in the Netherlands, a transformer supplier to the North American market. A digital sampling system was used as the reference value to identify the most appropriate measurement strategy and equipment for the measurement of the no-load losses. *IEEE Transactions on Instrumentation and Measurements* published our paper describing this work.

Further tests will be conducted in 2004 to determine the influence of the connections of the transformer on the determination of losses. A paper will be presented at the CPEM 2004 Conference in London, UK, in June 2004. It is believed that this project will eventually result in more accurate measurements during the no-load tests.

High Direct Voltage Measurements

T. McComb

Canada and the United States have several high voltage dc transmission links for which the uncertainty of power measurements is becoming an issue. INMS must improve the uncertainty of its high voltage dc measurements to meet the growing client demand for higher accuracy dc calibrations. The high voltage direct current (HVDC) measuring systems presently in use at INMS are based on shielded high voltage resistors mounted on a spiral, each connected in series to ground through a low voltage resistor (Park dividers). The high voltage resistors have continuous insulation and hence there are no intermediate connection points to monitor the behavior of parts of the resistor. Self-heating is the main contribution to the uncertainty of these dividers. Previously, the magnitude of this uncertainty has been estimated from the general characteristics of the constituent resistors. Over the past year, we have developed a test to estimate this contribution to the uncertainty by applying each high voltage for a set time and then taking measurements of the ratio as the divider cools. The method provides corrections that are sufficiently accurate to improve the overall uncertainty of the high voltage measurement.

We have designed a new 10 kV rated voltage divider and built it from precision resistors: the construction is open to allow connection at any point along the resistor chain. Each 1 kV section will be evaluated for linearity and stability using a calibrated 1 kV source that can be set to intermediate values. In addition, the upper section will be evaluated while the other sections are subjected to 1 kV to estimate the cumulative effects of heating from lower sections. This divider will be used to extend the base calibration of the higher voltage units to 10 kV. There are two options under consideration for future improvements: the construction of further stackable units of the 10 kV divider to give a better voltage divider, and/or creating access points in the existing dividers to allow their calibration as 10 kV sections.

Making comparison measurements with other National Metrology Institutes (NMIs) is an important part of verifying each NMI's capability. INMS has started comparison measurements with NIST (USA); it is expected these will be completed this year.

Calibration System for Optical Instrument Transformers With Digital Output

B. Djokic, E. So

This project involves the development of a new calibration system for non-conventional, optical instrument transformers with digital output. Optical instrument transformers are becoming attractive alternatives to conventional instrument transformers, as their costs, especially for installation, are lower. They are expected

to capture a considerable share of future high voltage measurement installations worldwide.

The prototype of a new high accuracy calibration system based on the International Electrotechnical Commission (IEC) standards 61850, 60044-8 and 60044-7 has been built and its performance evaluated. However, future progress will depend on the availability of relevant technical information from various manufacturers, which still appears to be in many instances proprietary. The results of this work will be presented at the CPEM 2004 Conference in London, UK, in June 2004.

Optically Powered Current Measurement System for High Voltage Applications

B. Djokic, E. So

This project aims to simplify calibrations using non-conventional, high current sensors operating at high voltage that have the potential to be widely implemented by electric utilities.

The development of an optically powered current calibration and measurement system for high voltage applications has reached a major milestone. Electronic components such as analog-to-digital converters and voltage references suitable for micro-power applications were selected and tested. Circuitry for transmission of optical power, including laser photovoltaic power converters, and low voltage drop regulators were tested. Circuitry for data transmission was developed and tested. A fully functional prototype equipped with a microcomputer control system will be developed next.

Digital Sampling System for Low Power Factor Measurements for High Voltage Capacitive/Inductive Reactors

B. Djokic, E. So

The objective of this project is to improve and enhance calibration capabilities for in-house and on-site power measurements on high voltage reactors.

We have completed the development of a digital sampling system for high voltage and high current/low power-factor measurements. A new digital signal processing-based functionality for phase angle measurements under distorted waveform conditions was added to the system. It allows the system to operate as a high voltage and high current magnitude and phase angle digital sampling bridge. The operation of the system was automated to make the calibrations more convenient and time efficient as compared with the conventional current-comparator based bridges. The estimated uncertainty under real high voltage conditions is less than 50 parts per million.

The new system was used to calibrate the voltage transformer from the Korean Research Institute for Standards and Science (KRISS) that will be used as a transfer standard for Korean utilities. A field trial is planned to test the new system's suitability in an industrial environment. The results of this work will be presented at the CPEM 2004 Conference in London, UK, in June 2004.

A Hybrid Electronically Coupled Current-Comparator

B. Djokic, E. So

INMS has developed a versatile, electronically coupled current-comparator usable in a very diverse range of measurement applications. It features a significantly simpler and less expensive design than conventional current-comparators and yet retains the high accuracy associated with current-comparator technology. A prototype of the device, comprising a simplified current-comparator, high resolution multiplying digital-to-analog converters and control circuitry, has been built and tested for effective resolution, linearity, frequency response and overall accuracy. It has a resolution of 1 part in 10^6 and an estimated uncertainty on the order of 10^{-5}. The results of this work will be presented at the CPEM 2004 Conference in London, UK, in June 2004.

Diagnostic Measurement Techniques and Instrumentation

M. Abou-Dakka, S. Bamji, D. Bennett, A. Bulinski, Y. Chen, L. Cisse, D. McIntyre, E. So, D. St-Jean

This project involves research and development of various diagnostic measurement techniques and instrumentation to assess the operating conditions of different types of high voltage apparatus in service, and to predict their remaining lifetime.

Electroluminescence Measurements

Electroluminescence (EL), the emission of light in dielectrics subjected to high electric stress, has been successfully employed to detect the very early stages of degradation of insulation used in high voltage devices. The EL inception voltage is the threshold voltage at which the polymer starts to degrade.

INMS and Laboratoire de Genie Electrique de Toulouse (LGET) of Centre national de la recherche scientifique (CNRS) in France are the two leading research centres for EL studies in polymeric insulation. A joint Canada-France research proposal, “Determination of threshold stress of electrical aging for selected solid dielectric materials used by the electrical and electronic industries,” was approved for funding under the NRC International Relations Research Program. The total cost of the two-year project between NRC and CNRS is $1.35M. The collaborative project, which started in April 2003, investigates the electrical properties of polymeric materials and complements the pioneering R&D in EL pursued at each laboratory. The two centres will also use the thermal step method and the pulsed electro-acoustic technique for space charge measurements in thin insulating films used by the electronics industry.

The project will have a two-fold impact. It will provide: (1) valuable engineering data to assist designers in the development of optimal and reliable insulation systems and (2), a novel material evaluation tool, which would reduce the cost of determining the performance of new materials under various stresses including the new generation of nano-dielectrics.

Mutual benefits derived from the joint project include:

- enhancement of the present knowledge of aging processes in solid dielectrics
- training of new graduates with state-of-the-art techniques in cutting-edge research
- publications in archival journals
- patents and possible licensing of new technology to the Canadian and French industries
- establishing industrial partnerships for both centers.

At NRC and CNRS the work has focused on thin films of low density polyethylene (LDPE) subjected to uniform electric fields of up to 30 kV/mm at various frequencies of ac voltage. The EL pulses were detected in a test cell equipped with a specialized optical system, photomultiplier tube and a charge-coupled device (CCD) camera. By compensating the capacitive current the harmonics of the insulation loss current were also measured.

The tests revealed that the maximum number of EL pulses never occurs at the peaks of the ac voltage but prior to them. It is attributed to the space charge effect that causes the local field in the dielectric to be ahead of the applied field. Also, it was found that the number of EL pulses emitted during the negative half-cycle is always about two times greater than during the positive half-cycle because at all frequencies of the ac voltage the injection efficiency of electrons into LDPE is greater than that of holes.

The figure below shows a typical picture obtained by the CCD camera of LDPE film subjected to an electric field of 29 kV/mm. The intensity of light emitted across the sample is not uniform but has several bright spots, which could be attributed to the non-uniformity of the specimen thickness or defects in the polymer. The intensity of the bright spots is ten times higher than that of the surrounding area.

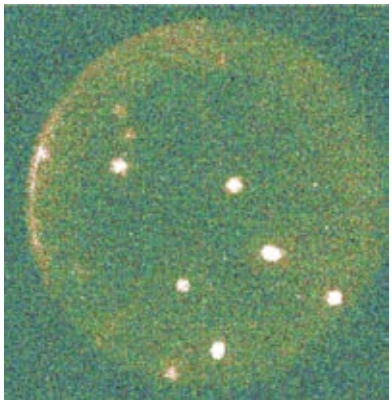


Figure 1. Spatial distribution of EL in LDPE under 29 kV/mm ac field detected by CCD.

Long-term space charge measurements revealed that the amount of charge in the polymer increases with the time of aging, and that the space charge moves from the electrodes into the bulk of the material, creating regions of higher internal electric field concentrations.

The results of this work will be presented at the 2004 IEEE International Conference on Solid Dielectrics in Toulouse, France, in July 2004.

Polarization Current Measurements

Many research labs have focused on the development of reliable diagnostic tests for polymeric cable insulation, and have proposed a multitude of techniques. Some of the techniques have already been tested and used in the field, while others are still in the developmental stage. Electric utilities widely appreciate the practical significance of diagnostic tests for the electrical power industry, as their use could reduce unscheduled outages, improve maintenance planning and increase electric systems reliability.

A general premise in developing a diagnostic technique is that physico-chemical changes caused by electrical aging must result in a measurable change in the dielectric properties of the insulating material. The technique tested at NRC is based on a link between the polarization current characteristics easily obtained at relatively low dc fields and the material ac failure characteristics.

Polarization currents were obtained at a low dc field for two different brands of cross-linked polyethylene (XLPE) and ethylene propylene rubber (EPR) used as high voltage insulation of power cables. The materials were subjected to an ac electric field in the presence of an ionic solution, which induced the growth of water and ionic impurities containing structures detrimental to the material's electrical performance. The parameters of polarization currents were correlated with the time to breakdown characteristics.

Two parameters of the polarization current, the RC time constant and the area under the polarization current curve, show a consistent increase with insulation aging. However, the actual correlation between the two polarization current parameters and the insulation failure probability is material dependent. In EPR, measurable changes in the polarization current characteristics happen only just before the insulation failure. Thus, the polarization current measurements would not be an effective diagnostic tool for this insulation. Although the diagnostic use of this technique for XLPE insulation appears feasible, more tests on full size cables are required to confirm the results obtained on laboratory samples. Such tests are being planned with Hydro Ottawa for late summer 2004.

*Space Charge Measurements in Solid Dielectric*s

The correlation between space charge accumulation and dielectric failure has been investigated in four polyethylene-based materials subjected to a dc field of 50 kV/mm. Two of the materials contained tree-retardant additives and all four were aged for extended periods up to 26,000 hours. The specimens were removed periodically from the external field and subjected to space charge density measurements under no-voltage conditions using the thermal step technique developed at NRC. Not a single parameter that would consistently correlate with times to insulation failure could be derived from the space charge measurements. However, the space charge measurements provided valuable insight into a possible link between the dynamics of space charge development and the time of dc aging. In particular, a consistent surge in the amount of accumulated space charge shortly before insulation breakdown was observed in practically all samples. Also, a consistent relationship

between time to breakdown and the position of the internal space charge density peaks in the sample was observed. Samples with space charge density peaks farther apart consistently survived longer than those with peaks closer together. These phenomena, if confirmed, could be used to screen new materials for dc voltage applications. The effect of the voltage polarity reversal after a long single polarity voltage application was found to be significantly less detrimental than claimed in literature. Typical aging tests carried out by industry do not exceed several thousand hours and would not reveal the effects that require a longer time to develop.

Underground dc transmission of electric power offers significant advantages over ac transmission especially over large distances and has been explored by electric utilities around the world, most notably in Sweden and Australia. Long distance power transmission is prevalent in many parts of Canada. This project develops space charge measurements and the test procedures to determine the space charge effects in dielectric materials.

International Comparisons

R. Arseneau, M. Frigault, E. So, J. Zelle

In 2000, INMS conducted a comparison of the non-sinusoidal calibration systems with SP Swedish National Testing and Research Institute, the Swedish NMI. The comparison relied on a stable source, developed at NRC, as the transfer standard. Under the sponsorship of the Working Group on Key Comparisons of the CIPM Consultative Committee on Electricity and Magnetism, this project is being expanded to include four other National Measurement Institutes. INMS has been chosen as the pilot laboratory responsible for the transfer standard and data analysis. In 2003, tests were done at INMS and at PTB in Germany. The next tests are planned for NPL (UK) and NIST (USA). Final results are expected for the CPEM 2006 Conference.

Client Services

D. Angelo, R.J. Arseneau, D.A. Bennett, A.T. Bulinski, M. Frigault, T. McComb, E. So, D. St-Jean, G.Williscroft, N. Wise, J. Zelle

This project is devoted to providing calibration and test services to the electrical power industry. Calibrations include various types of measurement equipment such as bridges, instrument transformers, active/reactive power and energy meters under sinusoidal and non-sinusoidal waveform conditions, electrostatic voltmeters, high voltage capacitors, high current ac/dc resistors and shunts. Also included are measurements of various electrical quantities such as electromagnetic fields, large power transformers and inductive reactor losses, and the calibration of industrial measuring systems (ac, dc, and impulse) both in-house and in-situ. The Group also performs dielectric tests and partial discharge measurements on high voltage insulating materials and high voltage system components.

Committees and Offices

International

Comité International des Poids et Mesures

Comité Consultatif d'Électricité et Magnétisme (CCEM)
E. So, Member

Conference on Precision Electromagnetic Measurements (CPEM)
E. So, Chair, CPEM Executive Committee
T.R. McComb, Secretary, CPEM Executive Committee

International Electrotechnical Commission (IEC)

IEC Technical Committee 42: High Voltage Test Techniques,
T.R. McComb, ex officio member as chair of CSC TC42

TC68 - Magnetic Alloys and Steel
E. So, Member

Conference Internationale des Grands Réseaux Electriques (CIGRE)

WG 12.16 - Instrument Transformers
A.T. Bulinski, Member

WGD1.33 - High Voltage Measuring Techniques
T.R. McComb, Member

Institute of Electrical and Electronics Engineers, Inc. (IEEE)

Power Engineering Society (PES)

PES Power Systems Instrumentation and Measurements Committee (PES PSIM)
E. So, Past Chair and Liaison, Transformers Committee and Instrumentation and Measurements Society
R. Arseneau, Secretary
T.R. McComb, Member

PES/PSIM Digital Techniques in Electrical Measurements Subcommittee
T.R. McComb, Member
E. So, Member and Liaison, Transformers Committee

PES/PSIM High Voltage Test Techniques Subcommittee
T.R. McComb, Member
E. So, Member

PES/PSIM Electricity Metering Subcommittee
E. So, Chair
R. Arseneau, Member
T.R. McComb, Member

PES/PSIM WG Non-Sinusoidal Situations
R. Arseneau, Member
T.R. McComb, Member

PES/PSIM WG Instrumentation for Non-Sinusoidal Situations
R. Arseneau, Chair

PES/PSIM WG Diagnostic Field Testing of Power Apparatus
E. So, Member and Liaison, Transformers Committee

PES/PSIM WG High Voltage Low Power Factor Power Measurements
E. So, Chair

PES/PSIM WG Optical Instrument Transformers
E. So, Member

PES/PSIM Awards Committee
T.R. McComb, Coordinator

PES Transformers Committee - Instrument Transformers Subcommittee
E. So, Member

PES Transformers Committee - Performance Characteristics Subcommittee
E. So, Member

PES Transformers Committee - WG Loss Tolerance and Measurements
E. So, Member

Instrumentation and Measurements Society

TC10 Digital Waveform Recorders
T.R. McComb, Corresponding Member

Dielectrics and Electrical Insulation Society (DEIS)

Administrative Committee
S.S. Bamji, President

Meetings Committee
A.T. Bulinski, Chair

Education Committee
A.T. Bulinski, Member

Statistics Committee
A.T. Bulinski, Member

Conference on Electrical Insulation and Dielectric Phenomena
S.S. Bamji, Member, Executive Committee
A.T. Bulinski, Member, Executive Committee

IEEE International Conference on Solid Dielectrics
A.T. Bulinski, Member, International Advisory Committee (IAC)

IEEE International Symposium on Electrical Insulation
A.T. Bulinski, Member, IAC

IEEE International Symposium on Electrets
A.T. Bulinski, Member, IAC

IEEE International Conference on Liquid Dielectrics
A.T. Bulinski, Member, IAC

IEEE International Conference on Properties and Applications of Dielectric Materials
A.T. Bulinski, Member, IAC

International Conference on Electrical Insulating Materials (Japan)
A.T. Bulinski, Chair, IAC

International Symposium on Discharges and Electrical Insulation in Vacuum
A.T. Bulinski, Member, IAC

2003 Symposium on High Voltage Engineering, Delft, The Netherlands

A.T. Bulinski, Member, Organizing Committee

2004 International Symposium on Gaseous Dielectrics, Athens, Greece

A.T. Bulinski, Member, International Organizing Committee

National

Canadian Standards Association (CSA)

Technical Committee on Performance of Electric Motors
E. So, Member
R. Arseneau, Alternate

Technical Committee on Instrument Transformers
R. Arseneau, Member
E. So, Member

Standards Council of Canada (SCC)

CSC TC 42: Technical Advisory Group on IEC TC 42: High Voltage Test Techniques
T.R. McComb, Chair

Institute for Electrical and Electronics Engineers, Inc. (IEEE) - Ottawa Section

IEEE Ottawa Section Conferences, Inc.
R. Arseneau, Secretary and Treasurer

Sudbury Neutrino Observatory (SNO)

Advisory Committee on High Voltage Connectors
A.T. Bulinski, Member

Editorships

IEEE Electrical Insulation Magazine
A.T. Bulinski, Contributing Editor

Wiley Encyclopedia of Electrical and Electronics Engineering
A.T. Bulinski, Member of International Editorial Board

Materials Science – an Interdisciplinary Journal of Physics, Chemistry and Technology of Materials
A.T. Bulinski, Member of Advisory Editorial Board

Instrumentation and Measurements Society
T.R. McComb, Member of Editorial Review Committee
E. So, Member of Editorial Review Committee

Published Papers and Presentations

This publication list is for the calendar year 2003. A complete list of publications, from 1999, is available on the INMS web site at http://inms-ienm.nrc-cnrc.gc.ca/research_and_development/electrical_power_publications_e.html.

Articles in Refereed Journals

Arseneau, R., M. Sutherland and **J. Zelle.** "A New Transfer Device for the NRC Travelling Standard Program", IEEE Trans. Instrumentation and Measurements, **52.2,** 411-414 (April 2003).

Cisse, L., S.S. Bamji and **A.T. Bulinski.** "Electric field calculations for needle-plane geometry and space charge in polyethylene", IEEE Trans. Dielect. Elect. Insul., **10.1,** 176-180 (February 2003).

R.C. Hughes, H.A. Lightfoot and **McComb, T.R.** "Assuring the Quality of Impulse Voltage Calibration Results issued by a Calibration Laboratory. IEEE Trans. Power Delivery, **18.3,** 701-704 (July 2003).

Y. Li, J. Rungis and **McComb, T.R.** "Comparative Impulse Voltage Measurements at the National Measurement Laboratory (NML), CSIRO, Australia, and the Institute for National Measurement Standards (INMS), NRC, Canada", IEEE Trans. Instrumentation and Measurement, **52.2,** 404-407 (April 2003).

So, E., R. Arseneau and E. Hanique. "No-Load Loss Measurements of Power Transformers under Distorted Supply Voltage Waveform Conditions", IEEE Trans. Instrumentation and Measurements, **52.2,** 429-432 (April 2003).

So, E., R. Arseneau, D. Bennet, T.L. Nelson and B.C. Waltrip. "NRC-NIST Intercomparison of Calibration Systems for Current Transducers with Voltage Output at Power Frequencies", IEEE Trans. Instrumentation and Measurements, **52.2,** 424-428 (April 2003).

Conference Proceedings

Abou Dakka, M., S.S. Bamji and **A.T. Bulinski.** "DC Polarization Characteristics of XLPE and EPR Insulation Subjected to Water Treeing", Proc. 2003 CEIDP, Albuquerque, New Mexico, USA., 181-184 (October 2003).

Arseneau, R. "Application of IEEE Standard 1459-2000 for Revenue Meters". IEEE PES Summer Conference, Toronto, Ontario, Canada (July 2003).

K. Tohyama, **Bamji, S.S.** and **A.T. Bulinski.** "Simultaneous Measurement of Electroluminescence and Dissipation Current in Cable Insulation", Proc. 2003 IEEE Internatinal Conference on Properties and Applications of Dielectric Materials (ICPADM 2003), Nagoya, Japan, Paper S16-4 (June 2003).

So, E. "Harmonic Measurements: Current and Voltage Transducers", IEEE PES Summer Conference, Toronto, Ontario, Canada (July 2003).

Invited Oral Presentations

Abou Dakka, M., S.S. Bamji and **A.T. Bulinski.** "Polarization/Depolarization Current Measurements – a Diagnostic Tool for High Voltage Cable Insulation Subjected to Water Treeing", Hydro Quebec Research Institute (IREQ), Varennes, Quebec (January 2003),

So, E. "INMS/EPMG, On-site Calibrations of Instrument Transformers and High Voltage Power Measurements Systems", Alstom Power, Richmond, VA, USA (March 2003).

So, E. "INMS/EPMG, On-site Calibrations of Instrument Transformers and High Voltage Power Measurements Systems", Trench Electric Ltd, Toronto, Ontario (September 2003).

So, E. "Overview of NRC/INMS/EPMG", NxtPhase, Vancouver, British Columbia (February 2003).

So, E. "The Need for a Measurement Assurance Program for Electric Power/Energy and its Economic Impact", APMP General Assembly, TCEM, Singapore (December 2003).

So, E. "Traceability of High Voltage Power Measurements", Hyosung Power Transformers Manufacturer", ChangWon, South Korea (November 2003).

So, E. "Traceabilty of High Voltage Power Measurements", General Electric, Peterborough, Ontario, September (2003).

Technical Reports

Bulinski, A.T. "Diagnostic Tests on Specimens of the Hydro Ottawa Cable that Failed in Service in September 2002", Report prepared for Ottawa Hydro, Ottawa, Ontario (May 2003).

Honours and Awards

Dr. Soli Bamji. Re-elected President of the IEEE Dielectrics and Electrical Insulation Society.

Patents

E. So and **B. Djokic.** "A Computer Controlled Four Terminal AC Resistance Bridge", US Patent 10/096,859, application October 2003. "A Current Comparator-based Four Terminal Resistance Bridge for Power Frequencies", Canada Patent 2,376,732.

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Nicholas Wise	990-3620
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Ladji Cisse	990-3619
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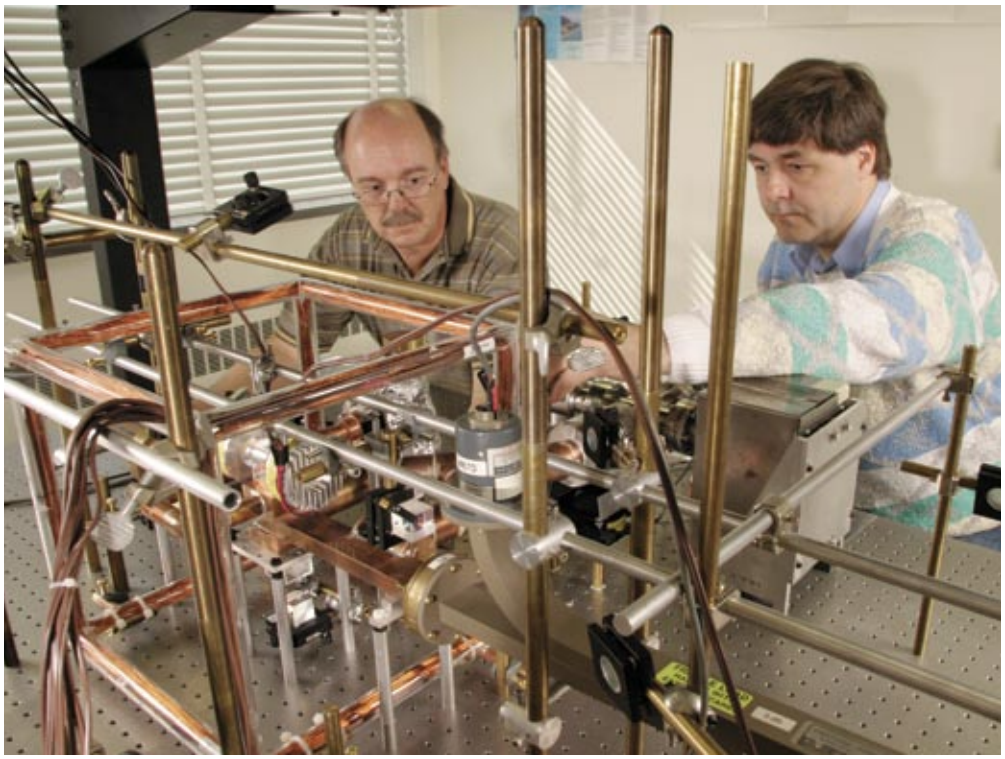
William Moore	
Yang Sup Song	990-5826



Using the laser cooling of atoms, researchers are developing a new type of microwave power standard. Scientists have built an experimental apparatus in which rubidium atoms are laser-cooled in an optical trap, then allowed to fall through an aperture in a waveguide. The microwave radiation in the waveguide induces an oscillation in the atoms, which are probed as they emerge from the lower face of the guide. The power of the incident microwave radiation can be determined by measurement of the induced oscillation frequency. This new technique takes advantage of modern laser-cooling techniques to provide a more accurate microwave power standard.

The Electrical Standards Group maintains primary standards, performs calibrations, provides technical consultation, and maintains and upgrades its calibration facilities.

The program includes the electrical standards of voltage, resistance, capacitance, inductance, dc voltage ratio, low frequency ac voltage ratio, ac/dc difference, and ac resistance; and voltage, impedance, attenuation, power and noise at RF and microwave frequencies up to 110 GHz.

Values of these standards are disseminated through a comprehensive calibration service, and technical support is provided for the Institute's Calibration Laboratory Assessment Service (CLAS) activities. A client list of over 120 contacts is maintained. These activities impact on most technical, manufacturing, transportation, military and utility sectors.

Low Frequency

P. Filipski, M. Boecker, K. Kochav, B. Wood, M. Côté

This project covers the field of low frequency (below 1 MHz) standards of capacitance, inductance, ac/dc transfer, ac voltage and ac voltage ratio. Also included are dc voltage ratio standards.

Capacitor and inductor calibrations are automated in the most utilized ranges: capacitors from 1 pF to 1 μF at 1 kHz and inductors from 1mH to 10 H at 1 kHz. The process of inter-comparing the primary standards continues.

The primary standard of capacitance is a bank of 10 pF capacitors that are infrequently compared with calculable capacitors in other national measurement institutes (NMIs), and indirectly with the dc QHR. Work continues on developing a method of relating the NRC unit of capacitance to the ohm (Ω). In the future this will eliminate a dependence on the international community in the determination of the Canadian unit of capacitance, and the impedance scale in general. We are also actively considering developing an INMS Thompson-Lampard calculable capacitor.

A major provincial power outage in Ontario, Canada in the summer of 2003 led to loss of thermal control in the capacitance laboratory, and a consequent step change in the value of the primary standard. Fortunately check standards have allowed us to accurately determine the magnitude of the change. The laboratory is to be fitted in the next year with an independent air-conditioning system to prevent recurrence of this problem.

A multifrequency comparison of 10 pF and 100 pF capacitors with the BIPM failed when one of the capacitors was damaged and the other subjected to the environmental problems discussed above. Additional Andeen-Hagerling capacitors have been purchased to improve the redundancy and independence of the standard capacitors.

A new ac voltage comparison system based on Thompson's method of comparing ac coaxial voltages was investigated.

Work continues in the area of ac/dc transfer where we develop and maintain primary standards of ac/dc difference of voltage and current, perform calibrations of ac/dc transfer standards, ac shunts and voltmeters at the highest accuracy levels, and provide technical consultation.

Our calibration capabilities for ac/dc difference cover the voltage range from 2 mV to 1000 V in the frequency range 10 Hz to 1 MHz and up to 30 V in the frequency range 1 MHz to 100 MHz. At present, ac current/shunt calibration capabilities are offered at the 5 mA to 20 A level, in the frequency range 10 Hz to 20 kHz (30 kHz at lower currents).

In the last year we have concentrated on the improvement of the ac shunt calibration capabilities, in step with clients requirements to extend our frequency range up to

100 kHz. Three series of high frequency coaxial shunts, ranging in current from 10 mA to 10 A have been built and the selected working standards are in the last stages of characterization. We will be able to offer these extended capabilities to our clients in the incoming calibration period. This work will continue in the upcoming year to extend the current range up to 100 A at 100 kHz.

Work on improving RF voltage ac/dc transfer capabilities is continuing. We are planning to upgrade and modernize calibration facilities, to increase the RF voltage test range up to 50 V.

Josephson Effect and Quantised Hall Resistance

B. Wood, D. Inglis, B. Young, K. Kochav, M. Côté

This project involves the research, development and maintenance of standards of voltage based on the Josephson effect, and of resistance based on the quantised Hall resistance (QHR). It includes development work on cryogenic current comparators, standard resistors, resistance bridges and Josephson array applications.

The Josephson effect is a quantum-mechanical phenomenon occurring when two superconductors are weakly joined to form a Josephson junction. When such a junction is cooled to about 4 K and irradiated with high frequency electromagnetic radiation it functions as an excellent frequency to voltage transducer. Given an input frequency, f, the device generates a voltage, V, which is linearly and precisely related to f by the ratio 2e/h, where e is the magnitude of the charge of an electron and h is Planck's constant. Series arrays of several thousand Josephson junctions have been fabricated to form a microwave transmission line. Such arrays are utilized in NRC's voltage standard. This structure can generate potentials up to 10 V and is accurate to parts in 10^9 or better. It is routinely operated to determine the value of various standard cells and Zener references. A direct comparison of voltages produced by the NRC and BIPM Josephson array systems showed agreement to within 2 x 10^{-10}.

The 10 V chip failed and was replaced with a chip which was much more sensitive to rf interference. Improvements to the filtering, shielding and bias electronics were implemented to combat this problem. A multiple frequency microwave synthesizer provided by PTB (Germany) was used to calibrate the JAVS frequency counters at some hundreds of frequencies in the band of interest (around 75 GHz). Results show differences between our two frequency counters, and that one of them is accurate to within 2 x10^{-11}.

In the International System of Units, the ohm is derived from the volt and the ampere. In practice, quantised Hall resistors (QHR) have been used in national standards laboratories to represent resistance since 1990. These resistors are semiconductor devices which, when cooled to around 1 K and subjected to a magnetic field of several tesla, yield values of resistance which are essentially invariant, and which are believed to be multiples of fundamental constants. By international agreement, the first multiple is taken to be equivalent to 25 812.807 Ω.

A cryogenic current comparator (CCC) bridge is used to transfer values from the QHR to 100 Ω, 1 Ω, and 10 kΩ wire-wound resistors, which in turn are used to transfer values to the working standards used for calibrations of client resistors. The CCC has a repeatability of 5 parts in 10^9 for the QHR to 100 Ω transfer.

In the fall of 2003, Measurements International Ltd. (MIL) delivered and installed the first commercial "Quantohm" primary resistance standard at the National Physical Laboratory in India. The apparatus is a portable, relatively economical QHR primary standard designed for use in national metrological laboratories, and also in industrial laboratories. The Quantohm was designed by the Electrical Standards Group and developed in collaboration with MIL, who build and market the standard under an NRC-INMS license. A QHR device developed at NRC is cooled in liquid ^{4}He to 1.2 K in fields up to 8 T. The refrigerator and magnet have been designed to fit into a helium transport dewar. A commercial current-comparator bridge is used to attain accuracies better than 2×10^{-8} when transferring values from the QHR to a 1000 Ω wire resistor. The installation and testing of the NPL India system was completed in 2 days. Subsequently, Measurements International has received several enquiries about their system.

Since 2002, we have been involved in an intensive collaborative effort with METAS and PTB, the NMIs of Switzerland and Germany, to better understand some of the problems related to the use of QHR devices at frequencies to 10 kHz. Through a series of inter-laboratory visits we have compared cryogenic systems, measurement bridges and techniques, and QHR devices from different sources. Outcomes include: an improved understanding of ratio and quadrature bridges, and especially of the role of active and passive current equalizers in these bridges; a determination that any frequency dependence of the QHR is limited to less than 0.02 ppm at these frequencies; the discovery that some of the recently manufactured Gibbings-type resistors exhibit a linear frequency dependence which is not understood; and the finding that the 'curved plateaux' exhibited uniquely by PTB QHR devices are indeed device related and not an artifact of the mounting of the devices or of the measurement system used. Work continues on this latter problem. In the meantime, a series of three papers outlining the progress of this collaboration will be presented at CPEM 2004 in London.

Also in 2002, at the request of the international community, we commenced a project to produce a batch of several hundred QHR devices for use in NMIs. We have purchased two GaAs/AlGaAs wafers from the NRC Institute for Microstructural Sciences and have tested the properties of a dozen devices from these wafers. The heterostructures are good, yielding devices that operate at magnetic fields just below 10 tesla. Work continues to optimize the contacts for these devices, and it is hoped that this work will be completed and the first of the devices distributed to NMIs in the late fall of 2004.

RF and Microwave Measurements

A. Michaud,
D. Paulusse,
R.F. Clark

This project includes work on RF and microwave standards of voltage, attenuation, impedance, power and noise in the range of 0.1 MHz to 110 GHz, and includes routine customer calibrations, special calibrations, and research.

Most of the power measurement calibrations are done on an automated system that is continuously maintained and upgraded. During the last year, the range of this system has been extended: a new transfer standard has been developed in order to directly measure the Q-band waveguide thermistor mounts (WR-22, 33-50 GHz). We also characterized a set of non symmetrical adaptors and attenuators including those compatible with the 2.4 mm connectors. We can now do power sensor calibrations to 50 GHz (2.4 mm) on a routine basis.

Last year we also developed some waveguide transfer standards for the P and R bands (WR-62, 12-18 GHz and WR-28, 26-40 GHz respectively). Those standards are now commonly used with the automatic system, resulting in faster customer calibrations with improved turnround time. The measurements of power sensors are done by direct comparison with working standards, which are referred to the calorimeter at high accuracy.

Accurate calibration of transfer standards of attenuation is available on a fixed frequency basis for coaxial devices over the frequency range 1 MHz to 18 GHz and for waveguide devices from 2.6 GHz to 36 GHz.

For reasonably accurate measurement of 'S' parameters obtained at high speed, vector network analyzers are used to give coaxial frequency coverage from 5 Hz to 50 GHz and waveguide coverage up to 110 GHz.

We have continued our development of a new type of microwave power standard, based on the interaction of laser-cooled atoms with a microwave field. A laser-cooled atomic sample (rubidium vapour in this case) is allowed to fall through the microwave field in a waveguide. On emerging from the lower face of the guide the sample is probed with a second laser to determine the microwave power encountered in traversing the guide. The demonstration of this principle was presented at CPEM 2002 in Ottawa and was well received. The addition of a new vacuum chamber with an enclosed section of waveguide has allowed us to evaluate the accuracy of the method. Calculations are in agreement with the latest experimental results to within 1.3%, with an uncertainty of 5%. These results will be published in a paper that has been accepted for CPEM 2004.

We have been actively involved with a total of six rf/microwave international comparisons during the reporting year. In the microwave field, as with ac/dc measurements, this type of activity is an increasing part of the workload.

Richard Clark, guest worker with the Electrical Standards Group, served as an external assessor in the accreditation to ISO 17025 of the RF Metrology Laboratory of CSIR, South Africa, by SANAS, the South African accreditation body.

Client Services

Client services in 2003-2004 included:

- calibration services in the fields of dc and low frequency voltage, resistance, capacitance and inductance; and at RF and micro-wave frequencies attenuation, voltage, impedance and power. In addition we have calibrated several current comparator bridges at the highest accuracy for Canadian manufacturers
- provision of technical information to a broad range of Canadian and American interests relating to such topics as availability of calibration services, traceability of measurements, international document standards, and Canadian requirements in the area of metrology
- provision of technical expertise and on-site reviews of client calibration facilities to the NRC Calibration Laboratory Assessment Service (CLAS) in the assessment of two commercial calibration laboratories seeking SCC accreditation or continued SCC accreditation under ISO 17025.

International Comparisons

CIPM MRA comparisons for which reports have been authored:

- CCEM-EM K6c: RF AC/DC Transfer Difference
- CCEM-EM K8: DC Voltage Ratio
- CCEM-EM K9: HV AC/DC Transfer Difference

CIPM MRA comparisons in progress:

• CCEM-EM K7: AC Voltage Ratio	Measurements completed.
• CCEM-EM K10: QHR via 100 Ω resistors	Measurements completed.
• CCEM-EM K11: AC/DC Low Voltage	Measurements completed.
• CCEM-EM K12: AC/DC Current Transfer	In preparation.
• CCEM-RF K10.CL: Power at 26 GHz	Draft report issued.
• CCEM-RF K19.CL: Attenuation, 60 MHz and 5 GHz, type “N” step attenuator	Measurements completed.
• CCEM-RF S1.CL: Power 50 GHz	Measurements completed.

Other comparisons:

• EUROMET 555: s-parameters for 2.4 mm connectors	Draft report issued.
• EUROMET 633: Power for N-type and 3.5 mm connectors, 50 MHz to 18GHz	Draft report issued.
• APLAC: 1MΩ and 10 MΩ	In progress, ES reference laboratory, CLAS coordinating.
• NACC: 1 Ω	In progress, ES reference laboratory, CLAS coordinating.
• ILC 2002: 10 V comparison, by traveling Zeners	Measurements completed, report awaiting publication.

Committees and Offices

International

CODATA

Task Group on Fundamental Constants
B.M. Wood, Member

National Institute of Science and Technology (NIST)

Review Panel for Electronics and Electrical Engineering
B.M. Wood, Member

Comité International des Poids et Mesures

Consultative Committee on Electricity and Magnetism
A.D. Inglis, Member

CCEM Working Group on Low Frequency Quantities
A.D. Inglis, Member

CCEM Working Group on Radio Frequency Quantities
A.D. Inglis, Rapporteur

CCEM Working Group on ACQHR
A.D. Inglis, Member
B.M. Wood, Member

Institute of Electrical and Electronics Engineers (IEEE)

Instrumentation and Measurement Technology Conference IMTC'05
P. Filipski, Co-Chairman of Organizing Committee

Power Engineering Society (PES)

PES/PSIM WG Practical Definitions of Powers in Nonsinusoidal Situations
P. Filipski, Member

Editorships

IEEE Transactions on Instrumentation and Measurements
P. Filipski, Editorial Board

Published Papers and Presentations

This publication list is for the calendar year 2003. A complete list of publications, from 1999, is available on the INMS website at: http://inms-ienm.nrc-cnrc.gc.ca/research_and_development/electrical_standards_publications_e.html.

Articles in Refereed Journals

Filipski, P.S. and **M. Boecker.** "Experience with High-Output-Resistance MJTC AC-DC Transfer Standards,""*IEEE Trans. Instrum. Meas.*, **52**, 1314-1319 (August 2003).

Inglis, A.D., B.M. Wood, M. Côté, R.B. Young and **M. Early**, "Direct Determination Of Capacitance Standards Using A Quadrature Bridge And A Pair Of QHR Resistors", *IEEE Trans. Instrum. Meas.*, **52**, 559-562 (April 2003).

Inglis, A.D., B.M. Wood, M. Côté, R.B. Young and **M. D. Early.** "Plateau Flatness In The ACQHR: Do Gates Really Help?", *IEEE Trans. Instrum. Meas.*, **52**, 569-573 (April 2003).

G. Marullo-Reedtz, R. Cerri, I. Blanc, O. Gunnarsson, J. Williams, F. Raso, K. Kim, R.B. Frenkel, Z. Xiuzeng, A. S. Katkov, R. Dziuba, M. Parker, **Wood, B. M.,** L.A. Christian, E. Tarnow, S.K. Mahajan, A. Singh and Y. Sakamoto, "Comparison CCEM-K8 of DC Voltage Ratio: Results", *IEEE Trans. Instrum. Meas.*, **52**, 419-423 (April 2003).

Articles in Conference Proceedings

C. Hamilton, S. Kupferman, M. Salazar, D. Deaver and **Wood, B.** "Josephson Volt Interlaboratory Comparison At 10 V DC", NCLSI Conference Proceedings 2003 (August 2003).

Invited Oral Presentations

Wood, B. "Electrical Metrology I", BIPM Metrology Summer School, Sèvres, France (July 2003).

Wood, B. "Electrical Metrology II", BIPM Metrology Summer School, Sèvres, France (July 2003).

Other Oral Presentations

Inglis, A.D. "NRC QHR devices for NMIs", Euromet Experts Meeting QHE and JAVS, Bratislava, June 2003.

Inglis, A.D. "AC-QHE: Report of the co-operation NRC-METAS-PTB", Euromet Experts Meeting QHE and JAVS, Bratislava, June 2003.

Staff Members

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Chantal Miller	
Michael Ouellette	993-9619
Dave Paulusse	993-5772
Brent Young	993-5771
Barry Wood	990-9225

Guest Workers

Richard Clark	998-6925
Murray Early	

The Thermometry Group's Radiation Thermometer Calibration Facility has already attracted both scientific and commercial interest, even though some of the sources of blackbody radiation remain a work-in-progress. The facility was financed over a period of years within the Group's annual financial allocation due to the perceived need of Canadian industry for such a capability. A presentation describing its capabilities was well received by an international gathering of thermometry experts and attracted a level of interest certain to lead to future collaborative activity in the increasingly important area of non-contact thermometry. Blackbody sources covering the temperature range from –40 °C to 2500 °C give clients access to world-class calibration while INMS scientists and technicians continue to evolve the science. Thermo-Kinetics, a Canadian manufacturer and distributor of temperature measuring sensors and instrumentation, has used the calibration service as an alternative to sending their instrumentation to the United Kingdom for recertification. This made-in-Canada solution offers considerable convenience and savings in turn-around time for Thermo-Kinetics and other Canadian clients. In fact, interest in the facility's capabilities has also come from the United States. MedicalCV Inc., a Minnesota-based manufacturer of heart valves, has had instrumentation vital to their high-temperature manufacturing operations verified by the facility, and cited rapid turnaround time as critical to their operations. To date, no concerted effort to publicize the facility has taken place, yet the client base continues to grow mainly by word-of-mouth. The future is bright with non-contact thermometry growing rapidly in diverse application areas ranging from food storage and road maintenance to metallurgical heat treatment and high temperature manufacturing. Canadians will not only have access to the facility, but–just as importantly–will receive expert advice directly from the scientists and technicians who represent Canada internationally while working to extend the limits of metrology.

The objectives of the Thermometry Group are to conduct research and development, maintain primary standards, provide technical consultation, perform calibrations, and ensure that calibration facilities are adequate to meet the needs of Canadian science and industry for the measurement of temperature.

The program's mandate is to implement the International Temperature Scale of 1990 (ITS-90) over its defined temperature range of 0.65 K to above 2000 °C. Areas of study include: refinements and improvements to the ITS-90; characterization of specific fixed point temperature references; characterization and calibration of various temperature indicators, including platinum resistance thermometers (PRTs) and thermocouples; the study of secondary temperature references such as eutectic fixed points and vapour pressure furnaces; and the study of various heat flow problems.

The temperature standards are disseminated through a comprehensive calibration service to more than 300 clients and by support for Calibration Laboratory Assessment Service (CLAS) activities. The program activities impact many technical, manufacturing, transportation, and utility sectors, and frequently include interactions with other National Metrology Institutes (NMIs) as well as the BIPM.

Group members actively participate in the activities of the Consultative Committee for Thermometry (CCT), the international body charged with defining and disseminating the ITS, as members of various working groups. As participants in the various Key Comparisons organized by the CCT, they examine the compatibility of the calibrations carried out by the world's leading NMIs.

The Group's responsibility to maintain primary and secondary temperature standards sets the direction of its research programs. The activities include realization of the primary fixed points, studies of effects that limit the accuracies of these realizations and of the standard instruments, and research towards improvement in the scale itself (e.g. accurate measurements of thermodynamic temperatures, development of better fixed points, and/or improved methods of interpolation).

Fixed Points

J. Ancsin, K.D. Hill, C.K. Ma, A.G Steele, D.J. Woods

Above 5 K, the ITS-90 assigns temperatures to sixteen fixed points. The temperatures chosen are the best estimates of their thermodynamic temperatures. The project seeks to characterize specific fixed point temperature references in order to improve their performance and better estimate the associated uncertainties. The work includes the study of eutectic fixed points in addition to pure materials.

J. Ancsin investigated the effect of various impurities on the freezing point of aluminum, a fixed point of the ITS-90 at 660.323 °C. He doped aluminum samples with an assortment of impurities and determined the shifts of their equilibrium melting curves under static adiabatic experimental conditions. The shift of run-off temperatures (i.e., when the sample temperature breaks off from the melting plateau and rapidly begins to rise) of originally 99.9999% pure Al samples caused by impurities was determined to be as follows (in millikelvin per part per million

(ppm) by weight): Ag, -0.12; Zn, -0.16; Cu, -0.29; Fe, -0.35; In, -0.21; quartz, -0.33; Si, -0.66; Ti, +3.30; Mn, -0.063; Cd, -0.10; Sb, -0.19; Ca ?; Ni, -0.43.

J. Ancsin also investigated the influence of various gases on the freezing point of aluminum. The experiments revealed certain shortcomings of the current definition of the Al point. He monitored the pressure in sealed-type Al cells made of quartz, and found the pressure steadily decreased after sealing. He concluded that Al fixed points using sealed quartz cells are in a state of drift. In addition, the initial Al point temperatures yielded by open cells are higher than the subsequently realized ones. Five different gases were used to apply the initial 1 atm pressure over the Al samples (air, N_2, Ar, He, CO_2). Each of them appeared to slightly contaminate the Al samples, lowering the initial value of the Al point by 1 or 2 mK. However, sealed quartz triple point cells remained stable at their initial value indefinitely. Thus, the triple point of Al is a more reliable fixed point than the defining Al freezing point itself.

K.D. Hill, in a presentation at the 6th International Conference on Advanced Mathematical and Computational Tools in Metrology in Turin, Italy, discussed the potential for isotopic effects on fixed point temperatures. The workshop concluded that isotopic effects are clearly important for the triple points of hydrogen and water. In general, however, the influence on other fixed points remains uncertain due to a lack of experimental data.

K.D. Hill and A.G. Steele were among the authors of two reports to the CCT addressing the influence of deuterium on the triple point temperature of hydrogen (13.8033 K). The present definition of the fixed point is ambiguous since variations in the deuterium content lead to variations in the triple point temperature of up to 0.5 mK. A redefinition of the fixed point would associate the temperature with a specific deuterium concentration. Corrections would be applied to accommodate samples of differing concentrations (with respect to that of the reference).

C.K. Ma reported findings relevant to the precise realization of the freezing point of copper. He showed that it is necessary to account for the temperature drop across the wall of the cavity of the copper-freezing-point blackbody. This temperature drop (~1 mK) is difficult to measure and is usually estimated by theoretical calculations. In the first experiment, he constructed a number of cylindrical cavities, each having a circular base of non-uniform thickness. During freezing or melting, the temperature distribution of the base should reflect the varying temperature drop due to the varying thickness. Results showed that the effect of the non-uniform surface condition dominated that of the varying thickness, making the latter undetectable. In the second experiment, he constructed four atypical copper-freezing-point blackbodies which are practically identical except that the wall thicknesses are uniform but different from one another. The melting points of these blackbodies were compared with the melting point of a reference blackbody. The results showed an increase in the temperature drop with increasing wall thickness. Ma applied the results to a more typical copper-freezing-point blackbody and estimated a 1.1 ± 0.6 mK ($1\ \sigma$) temperature drop at the centre of the base. A comparable figure

for a similar cavity, based on the upper and lower limits calculated by detailed balancing of radiative and conductive heat flows in opposing limiting conditions, is 1.2 ± 0.5 mK.

A.G. Steele presented the results of recent measurements on equilibrium deuterium triple point cells. Two new-generation e-D2 cells, constructed from "vacuum arc remelt" (VAR) stainless steel and filled at IMGC using Gd_2O_3 as the spin conversion catalyst were measured using thermometers that were included in CCT-K2: 213865 (NPL) and 1872174 (NRC). Good agreement (within 0.15 mK) between the temperature scales was obtained. Data showing the spin equilibration time constant for one VAR cell, collected over a one-month period, were included. The new cells, compared against the well-known EUROMET-1 cell, were found to be more than 1.5 mK lower in temperature, probably due to impurity contamination.

A.G. Steele contributed to an article presenting an overview of an international star intercomparison of low-temperature fixed points. Between 1997 and 2002, 52 sealed triple-point cells (STPCs) of the thirteen laboratories represented by the authors were investigated at PTB, Germany. The STPCs are used to realise the triple points of hydrogen, neon, oxygen, and argon, respectively, as defining fixed points of the International Temperature Scale of 1990, ITS-90. The melting curves of all STPCs were measured on the same experimental equipment, adhering strictly to a single measurement program. This protocol enables separation of the effects influencing the melting curves and direct comparison of the thermal behaviour of the STPCs, which are quite different with respect to design, age, gas source, and filling technology. The paper emphasized the typical properties of the four fixed-point substances, and the spread of the STPC parameters. It also discussed connections between the star intercomparison and completed and on-going international activities, including the International Committee of Weights and Measures (CIPM) Key Comparisons.

Thermometers

J. Ancsin, K.D. Hill, L. Reesink, C.K. Ma, A.G Steele, D.J. Woods

Between the fixed points, temperatures on the ITS-90 are obtained by interpolation using standard instruments and assigned formulae. These standard instruments are the helium gas thermometer (3 K to 24.5561 K), the platinum resistance thermometer (13.8033 K to 961.78 °C), and the optical pyrometer (above 961.78 °C). This project concerns refinement and improvement of the ITS-90; characterization of various temperature indicators, including PRTs and thermocouples; and the study of various heat flow problems.

The ITS-90

K.D. Hill and A.G. Steele reported estimates of the non-uniqueness of the ITS-90 based on comparisons of capsule-style standard platinum resistance thermometers at more than eighty temperatures between 13.8033 K and 273.16 K. Using the measurements reported, as well as those from other studies, they conclude that the non-uniqueness takes on its largest value (± 0.4 mK) in the range from 83.8058 K

to 234.3156 K. This result may have been anticipated because of the fact that the argon and mercury fixed points are separated in temperature by more than 150 K, with no intermediate calibration points. Discussions concerning the details of the temperature scale that will eventually replace the ITS-90 must consider this fact if the non-uniqueness is to be minimized. At the present time, however, there are no candidate fixed points within this temperature range realizable to the required level of accuracy for inclusion into a revised International Temperature Scale.

Platinum Resistance Thermometers

J. Ancsin reported a study in which he soaked PRTs at different temperatures, and observed the resulting changes caused by oxidation and the subsequent dissociation of the formed oxide. Soaking below 400 °C initiated oxidation in some PRTs; their resistance then steadily increased with rising temperatures up to about 525 °C. When soaking near 525 °C, the resistance of the PRT remained stable. This suggests the thermal energy "kT" equals the energy binding oxygen to platinum, at the existing oxygen pressure. At temperatures higher than 525 °C, the formed oxide dissociated. The higher the temperature, the higher the thermal energy, and thus the higher the rate of dissociation. One hour of soaking near the Al point dissociated oxide that took hundreds of hours to accumulate. Three out of the seven PRTs tested would not oxidize despite soaking for several months. Further soaking, however, did initiate oxidation in two of the three PRTs.

Thermocouples

K. D. Hill and Y.-G. Kim (KRISS, Korea) compared the equivalence of thermocouple calibrations carried out in their respective facilities. Type S and Pt/Pd thermocouples, having a common welded junction, were calibrated at the metal fixed-points of Sn, Zn, Al, and Ag through five cycles at each NMI. The immersion properties in the fixed-point cells were also investigated. For the type S thermocouple, the differences were less than the equivalent of 0.15 °C at all fixed points. The equivalent temperature differences for the Pt/Pd thermocouple were within 0.04 °C from the Sn point to the Al point, but a difference of 0.32 °C was obtained at the Ag point. This difference may be attributed to the 12 μV value for the thermoelectric inhomogeneity of the Pt/Pd thermocouple revealed by the immersion profiles. The comparison confirmed that the fixed-point thermocouple calibrations carried out by the two NMIs are in good agreement, though the results appear limited by the less-than-ideal thermoelectric inhomogeneity of the thermocouples employed.

Fourier Transform Infrared Radiation Thermometry

L. Reesink and A.G. Steele presented results of experimental determinations of thermodynamic temperatures using Fourier transform spectroscopy of blackbody radiation sources. They compared these results to theoretical simulations of blackbody spectra in order to assess the feasibility and accuracy of the method. Much of the information on thermodynamic temperature contained in such spectral sets is redundant. Their presentation included a method (based on Gebbie's original

suggestion and recent work done at NRC) that exploits this redundancy in a straightforward way to obtain thermodynamic temperatures for each of the spectral sources. They briefly described the experimental setup, together with the analysis of the most recent experimental results and a comparison with the simulations.

Radiation Thermometry (Optical Pyrometry)

Temperatures above 962 °C on the ITS-90 are realized by means of an optical pyrometer calibrated at any one of the freezing points of silver, gold, or copper in accord with Planck's radiation law. The pyrometer is required to be effectively monochromatic and the reference source should be a blackbody simulator.

K.D. Hill and D.J. Woods reported the establishment of a blackbody-based calibration laboratory for radiation thermometers. Modern radiation thermometers frequently require a target larger than that presented by the filament of a tungsten-strip lamp. While we have used such lamps for many years to calibrate disappearing-filament optical pyrometers, it is apparent that our traditional calibration facilities require supplementation to meet the demands of today's instruments. To achieve this we have established a number of blackbody sources, some operating at fixed points of the ITS-90, and some which are variable in temperature. We have established blackbody sources at the fixed points of indium, tin, zinc, aluminum and silver. Three liquid baths (alcohol, water, oil), with side ports for blackbody cavities, cover the range from –50 °C to 300 °C. A cesium-filled heat-pipe blackbody operates from 300 °C to 660 °C; a sodium-filled heat-pipe blackbody from 660 °C to 1000 °C; and a directly heated graphite blackbody from 800 °C to 2500 °C. For the variable-temperature blackbody sources other than the directly-heated graphite furnace, we established traceability to ITS-90 by means of a Standard Platinum Resistance Thermometer (SPRT) below 660 °C, and a gold/platinum thermocouple from 660 °C to 1000 °C. A well-characterized radiation thermometer (LP3), calibrated at the freezing point of silver, provides traceability to the ITS-90 for the graphite blackbody.

Statistical Methods/Analysis of Key Comparisons

K.D. Hill, A.G. Steele

K.D. Hill and A.G. Steele (in collaboration with R.J. Douglas of the Frequency and Time Group) contributed to the analysis of Key Comparison data sets through a presentation at the Advanced Mathematical and Computational Tools in Metrology conference. Measurement comparison data sets are generally summarized using a simple statistical reference value calculated from the pool of the participants' results. This value can become the reference point against which the performance of the participating laboratories is judged. Consideration of the comparison data sets, particularly with regard to the consequences and implications of such data pooling, can allow informed decisions regarding the appropriateness of choosing a simple statistical reference value. The researchers examined recent key comparison results drawn from the BIPM database to illustrate the nature of the problem, and presented a simple approach to creating pooled data distributions. They recommend the proper use of detailed analysis when arguing in favor of a Key Comparison Reference

Value (KCRV), or when deciding that a KCRV is not warranted for the particular data sets obtained experimentally.

International Comparisons

- CCT-K1 Realizations of the ITS-90 from 0.65 K to 24.5561 K, using rhodium-iron resistance thermometers
  The draft report was distributed in January 2004, and a paper detailing the results is planned for TEMPMEKO 2004. NPL (UK) is the pilot laboratory and the participants include NIST (USA), NMIJ (Japan) , NRC, NMi-VSL (Netherlands), PTB (Germany), and VNIIFTRI (Russia).

- CCT-K5 Comparison of local realizations of the ITS-90 between 962 °C and 1700 °C using vacuum tungsten strip lamps as transfer standards
  The report continues to progress towards conclusion, albeit rather slowly, under the direction of the pilot laboratory, NMi-VSL (Netherlands). A revised draft is anticipated by mid-2004.

- CCT-K7 Comparison of water triple point cells
  The measurements are complete, and the cells have been returned to the participating laboratories from the pilot laboratory, BIPM. The draft report is in progress and the project is expected to conclude within 2004.

- NRC-VNIIFTRI (Russia) Comparison of Capsule-type Standard Platinum Resistance Thermometers from 13.8 K to 273.16 K
  Based on the results for one of the two thermometers, the NRC and VNIIFTRI scales are consistent within the expanded combined uncertainties (k = 2). The same cannot be said for the results of the second thermometer, where the differences are within the expanded combined uncertainties for only two of the eight temperatures of the comparison. A comparison in the cryostat at 273.16 K indicates that the value for thermometer 476 is clearly discrepant, thereby suggesting that either the original calibration was in error, or that the thermometer changed since its calibration at VNIIFTRI. We conclude that the results for the second thermometer are unrepresentative of VNIIFTRI's capabilities. A draft report was submitted to the CCT WG7, though we anticipate some revisions before the results will be approved for entry in the BIPM database.

- NRC-KRISS (Korea) Comparison of Type S and Pt/Pd Thermocouples
  The equivalent temperature differences for the Pt/Pd thermocouple were within 0.04 °C from the Sn point to the Al point, but a difference of 0.32 °C was obtained at the Ag point. This difference may be attributed to the thermoelectric inhomogeneity of the Pt/Pd thermocouple as revealed by the immersion profiles. For the type S thermocouple, the differences were less than the equivalent of 0.15 °C at all fixed points. The comparison confirmed that the fixed-point thermocouple calibrations carried out by the two NMIs are in good agreement, though the results were limited by the less-than-ideal thermoelectric inhomogeneity of the thermocouples employed.

- NRC-NIST (USA) Comparison of local realizations of the ITS-90 between 962 °C and 1700 °C using vacuum tungsten strip lamps as transfer standards
  All the measurements have been completed and the analysis is in progress.

- NRC-PTB (Germany) Comparison of local realizations of the ITS-90 between 962 °C and 1700 °C using vacuum tungsten strip lamps as transfer standards
  The comparison is complete and the results will be presented at TEMPMEKO 2004. In addition, a detailed report will be submitted to the CCT Working Group (WG7) charged with overseeing Key Comparisons. The overall result is very satisfactory, with the agreement well within the combined uncertainties (k=1).

Client Services

J. Ancsin, D. Gee, K.D. Hill, D.J. Woods

The Group offers a complete range of temperature calibrations for resistance thermometers, liquid-in-glass thermometers, thermistors, thermocouples, and radiation thermometers. The Group also maintains a variety of fixed points, furnaces, circulating baths, standard lamps, and blackbodies necessary for calibrations. Within the past year, calibrations of 195 individual thermometers were carried out for 82 clients.

Committees and Offices

International

Comité International des Poids et Mesures

CCT Consultative Committee for Thermometry
K.D. Hill, Member
A.G. Steele, Rapporteur

CCT WG 1: Defining fixed points and interpolating instruments
K.D. Hill, Member

CCT WG 2: Secondary fixed points and techniques of approximation to the ITS-90
K.D. Hill, Chair

CCT WG 4: Thermodynamic temperature determinations and extension of the ITS-90 to lower temperatures
L. Reesink, Member

CCT WG 7: Key Comparisons
A.G. Steele, Chair

Inter-American Metrology System (SIM)

Technical Committee, Thermodynamic Group
K.D. Hill, Member

IMEKO

Technical Committee 12
A.G. Steele, Member

International Program Committee for TEMPMEKO 2004 (Dubrovnik, Croatia, June 2004)

J. Ancsin, Member
K.D. Hill, Member

Published Papers and Presentations

This publication list is for the calendar year 2003. A complete list of publications, from 1999, is available on the INMS web site at http://inms-ienm.nrc-cnrc.gc.ca/research_and_development/thermometry_publications_e.htm*l*.

Articles in Refereed Journals

Ancsin J. "Impurity dependence of the aluminium point", *Metrologia*, **40,** 36-41 (2003).

Ancsin J. "ITS-90: The instability of the Al point", *Metrologia*, **40,** 232-234 (2003).

Articles in Conference Proceedings

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INMS is collaborating with Health Canada, Agriculture and Agri-Food Canada, Canadian industry, academia, and the international research community to establish measurement standards in the Natural Health Products (NHP) field. The Institute has commissioned state-of-the-art analytical instrumentation to enable capability in this sector. With the support of the Canadian NHP research community, INMS will become Canada's lead laboratory engaged in the development of reference methodologies and the production of certified reference materials for the characterization of NHPs and functional foods with respect to their safety and quality.

The Chemical Metrology Group at NRC's Institute for National Measurement Standards is the de facto national chemical metrology laboratory for Canada. It is well networked internationally with the National Metrology Institutes (NMIs) of other countries and staff serve on many international standards setting committees.

The principal objectives of the Chemical Metrology Group are to:

- perform research and development in selected areas of organic and inorganic trace analysis, in collaboration with Canadian instrument manufacturers when appropriate
- participate in comparisons with the national laboratories of other countries and cooperate with international organizations to ensure uniformity of chemical analysis procedures
- maintain strong linkages with Canadian and foreign environmental analytical laboratories through production of environmental Certified Reference Materials (CRMs) and through technical support of laboratory proficiency testing and accreditation activities
- operate a national facility for the elemental analysis of high purity materials by glow discharge mass spectrometry.

The laboratory has an internationally established reputation for the quality of its work on the trace analysis of heavy metals and selected organochlorine pollutants. Research in this area has supported the Marine Analytical Chemistry Standards Program (MACSP) for the past 25 years, with the principal goal of developing robust and accurate methodologies for the analysis of trace elements, PCBs, pesticides, dioxins and furans in samples of environmental interest. The MACSP advances the development, certification and dissemination of CRMs to support such measurements. The Program has distributed approximately 23,000 units of CRMs worldwide.
The validity of many environmental analyses in Canada and elsewhere is

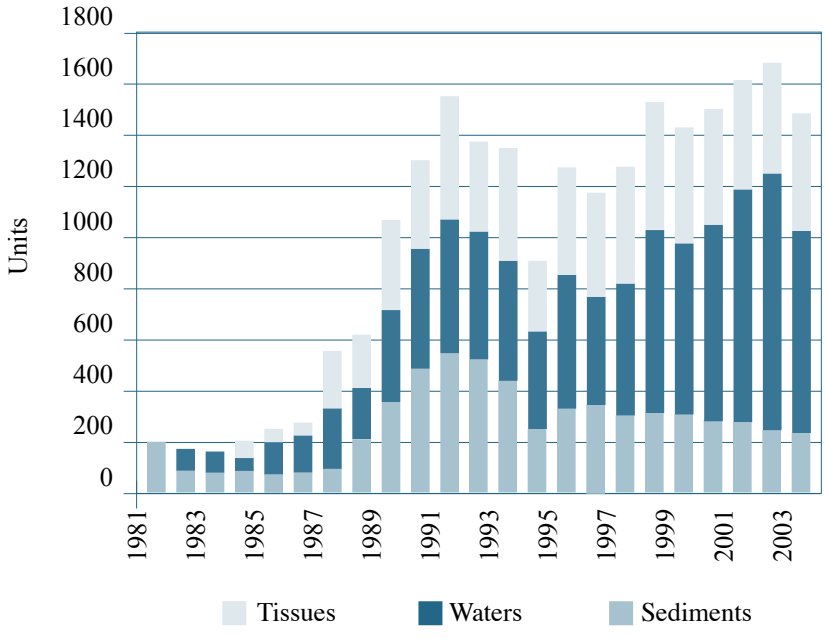
Certified Reference Materials

demonstrated by accurate results for one or more of the CRMs produced by INMS. The suite of CRMs currently available comprises four natural waters and four biological tissues for trace elements, three marine sediments for major and trace elements, three marine sediments for organotin speciation, one fish tissue for organochlorines, and CRMs for both total mercury in river water and nutrients in seawater.

Initiatives in the natural health products (NHP) arena promise to deliver validated methodologies and CRMs that will support Health Canada's new regulatory framework for the import, production, and sale of NHPs, enhancing consumer safety and confidence. This initiative is linked to similar activities in the U.S. under the auspices of the National Institutes of Health and the Food and Drug Administration.

Two of our newest sediment CRMs, HIPA-1 and SOPH-1, produced in support of speciation of butyltin in sediments, are the result of a joint exercise with LGC Limited, the national metrology institute for chemistry in the UK, marking the first such collaborative venture.

Total sales of CRMs declined in the reporting year, possibly as a consequence of increased global competition from other producers, or a weaker economy: 1490 units were distributed, about 12% less than in the previous year. Sales of biological tissue CRMs for trace elements rose by 3% to 471 units and the four sediment CRMs totaled 224 bottles, about the same as last year. Almost 23,000 units of reference materials have been distributed since 1981 with our six natural water CRMs accounting for over half of this total.



There is still a critical shortage of matrix reference materials, while even in the environmental arena clients are requesting more diversified products. Work is in progress on several new CRMs: an otolith reference material for trace elements, a suite of mercury and organomercury solutions for isotopic composition, and a selenized yeast for selenomethionine. Also, raw materials have been sourced and acquired for both American ginseng (Panax quinquefolius) root and a 5:1 spray dried extract of American ginseng root.

Methodologies and Instrumentation

These activities encompass many projects, the primary objectives of which are to extend our capabilities for organic and inorganic trace analysis and to support our CRM certification activities.

The validation of analytical methodologies and production of CRMs in the health field necessitate development of general instrumentation and techniques to address arising issues. This can only be accomplished through an active program of fundamental research. Continued development of "tunable" sources and the application of novel separation techniques, such as: capillary electrochromatography, atmospheric pressure matrix-assisted laser desorption ionization (MALDI), and solid phase microextraction with gas chromatography in tandem with plasma source detection systems, are necessary. Fundamental studies continue in the area of metal vapour generation techniques, as this discipline forms the basis for many current speciation schemes and enhances general capabilities for ultra-trace analysis. The applications base of high field asymmetric waveform ion mobility spectrometry (FAIMS) is being broadened as the details of the mechanisms and structural basis for separation of ions in FAIMS become apparent. In this way, fast separations with little or no reduction in complexity of the sample matrices will become possible.

Biotechnology

J. McLaren

Progress in biotechnology depends on reliable measurement of physical, chemical and biochemical parameters in biological and biomolecular systems. Biometrology encompasses the array of measurement science particular to biotechnology. The need for international biomolecular measurement standards is being felt in areas as diverse as trade, regulation and health.

Rapid progress in this area is particularly critical in light of the growing international concern surrounding genetically modified organisms (GMOs) and demands for mandatory labelling of transgenic seeds and foods. Possible restrictions on the importation of certain cereal grains and oil seeds are a major threat to Canadian exports of canola, wheat and potatoes. Any such technical barrier to trade will be based on the measurement of GMO content in commodity foods or food products. Therefore, accurate, equitable, and internationally accepted measurement standards for such determinations will be of great importance to the Canadian agricultural industry.

In 2002-2003, NRC-INMS received $250 K of Canadian Biotechnology Strategy (CBS) funding for a project titled *Quantitative Measurement of GMOs–Phase I: Needs Definition and Methods Development*. The project's overall objective is co-ordination of the development of internationally recognized and accepted measurement standards, and CRMs for quantitative DNA analysis of GMO content in foods.

The 'needs definition' part of the project was addressed primarily through a one-day interdepartmental GMO Needs Definition Workshop hosted by NRC-INMS in June 2002 and a one-day Symposium on GMO Analysis held in conjunction with the International Union of Pure and Applied Chemistry (IUPAC) 2003 Congress held in Ottawa in August 2003. The more recent event featured speakers from Australia, Canada, the EC, Japan, and the USA. Industry and government stakeholders were represented.

Two Canadian Food Inspection Agency (CFIA) labs–one in Ottawa, the other in Sidney, B.C.–with established facilities and expertise in real-time polymerase chain reaction-based quantitative DNA analysis, received $100 K of the CBS funds in 2003/2004 for continuation of the methods development work specifically related to the Quantitative Measurement of GMOs project. The BC laboratory participated in an international pilot study with NRC-IBS to test the degree of equivalence among NMIs and expert laboratories designated in quantitative DNA analysis.

In February 2004, Monsanto and NRC-INMS signed a Material Transfer Agreement allowing INMS to co-ordinate the development of the world's first genetically modified canola CRMs in collaboration with the CFIA and possibly other NMIs engaged in related activities. Preliminary studies at the CFIA Ottawa laboratory demonstrated the feasibility of producing such material from canola seeds.

A Steering Committee to guide the Canola CRM Project, with representation from INMS, federal departments, the Canadian Seed Growers Association and Monsanto was formed and met at INMS four times between November 2003 and April 2004 to develop a detailed project plan and discuss funding strategies.

Solid Phase Microextraction

Z. Mester, R. Sturgeon, P. Maxwell, L. Yang, P. Grinberg, V. Colombini, L. Abranko

Solid Phase Microextraction (SPME), in combination with atomic spectroscopy and organic mass spectrometry, is yet another area providing unique opportunities to explore trace element equilibria in many systems. In contrast to exhaustive extraction techniques, it does not perturb the natural equilibrium in the system it samples. This permits measurements of the distribution of molecular vs. dissociated or active vs. inactive forms of metal containing macromolecules. The "non-invasive" characteristics of SPME coupled with its ease of use and rapid sampling capabilities make it ideal for investigating volatile species. This is because SPME can achieve a clean, rapid separation of the analyte from a complex matrix, yielding enhanced sensitivity and detection power. SPME has been used in combination with analyte derivatization techniques to significantly advance our progress in speciation

studies, and to provide data for the certification of species content in several CRMs. Its applications base continues to be explored in combination with various instrumentations. Most recently, classical derivatization of aquo metal ions to produce volatile chelates, which can be sampled by SPME, has revealed a vast potential for ultra-trace analysis of transition metals using gas chromatography-mass spectrometry (GC-MS) techniques.

Speciation

Z. Mester, L. Yang, S. McSheehy, P. Maxwell, C. Scriver, M. Cui, C. Bancon-Montigny, B. Sadi, R. Sturgeon

Research has shown that the effect of trace elements in living systems, food and the environment depends on the chemical form in which the element enters the system and the final form in which it is present. The form, or species, governs its own biogeochemical behaviour. Obtaining such data is often difficult due to the extremely low concentrations of the analytes, the complexity of the matrices, and frequent instability of the species. Quality assurance of analytical procedures necessary for measuring such species can only be achieved using representative reference materials, certified for relevant species. The major concerns are the bioavailability of trace elements in food and improvement of species-specific waste management. Responding to this challenge, the CM Group is a consistent leader within the global community in the area of speciation, and the first reference material producer to provide CRMs certified for speciation content of selected elements (Hg, Sn and As). In the past year, the Group has co-ordinated both a pilot (P43) and a key (K18) international calibration exercise under the auspices of the CIPM Consultative Committee on Amount of Substance (CCQM). These exercises attempt to assess the capabilities of NMIs for the quantitation of di- and tributyltins in marine sediment. These activities have led directly to the certification of two new CRMs for tributyltin (SOPH-1 and HIPA-1) in sediment, which constitute a joint release from the NRC and LGC (UK). A project is underway focusing on the identification and quantitation of selenium containing amino acids such as seleno-methionine and seleno-cystine, the building blocks of proteinaceous seleno-macromolecules. This project's goal is to produce a CRM for selenized yeast dietary supplements. Institute Rosell-Lallemand, a major yeast manufacturer, is assisting us in this venture by providing support for a PDF as well as materials and expertise.

Natural Health Products

A. Windust, J. Lam, G. Gardner, P. McCooeye, C. Fraser, M. Le

The production and consumption of NHPs is booming on a global scale. Canadians are taking greater control over their health, utilizing alternative or traditional medicines, complementary therapies and natural health products. Herbs, herbal extracts, vitamins, minerals, essential fatty acids and probiotics are all familiar examples. They are routinely employed in traditional, Chinese and North American Aboriginal medicine and widely available to the public at large. In Canada and the United States it is estimated that about 50% of the population use NHPs. Thousands of different NHPs are available to the consumer and the North American market is worth billions of dollars annually. Canada is also an important producer of NHPs, with our largest market in the United States. However, due to the rapid expansion of

this market and the lack of regulated standards in the NHP industry, consumers are becoming increasingly wary of the quality, safety and efficacy of these products.

INMS has recently embarked on an ambitious new program to produce CRMs for NHPs. Methods are urgently needed to measure the levels of medicinally active components (assumed or documented biomarkers) in these products and to screen them for the presence of toxic metals and pesticides. The reference materials will function as analytical tools and facilitate the ability of laboratories to develop analytical methods, calibrate equipment, compare data, and trace results to nationally recognised standards.

The INMS Natural Health Products Initiative addresses the importance of these products to Canadians and the implementation of new Health Canada regulations governing their production and quality, thereby providing the nutraceutical industry with the analytical tools it requires, and enhance consumer confidence. The program has attracted over a million dollars in Major Initiative Capital funding from NRC to build analytical capacity for natural products, primarily through acquisition of extraction, separation and state-of-the-art mass spectrometry systems. Interest and support has come from external stakeholders in Canada, including the Pacific Agricultural Research Center (Agriculture Canada) in Sumerland, the Okanagan University College Institute of Technology in BC, the University of Ottawa, the Saskatchewan Nutraceutical Network, the BC Functional Food and Nutraceutical Network, and organizations in the US such as the Food and Drug Administration (Center for Food Safety and Applied Nutrition), the National Institutes of Health (Office of Dietary Supplements), the National Institute for Standards and Technology (NIST), the Department of Agriculture, as well as the Association of Official Analytical Chemists (AOAC) International.

To ensure this work is harmonized with efforts in other countries, INMS works in cooperation with international associations: it participates, for example, on the AOAC International Task Group on Dietary Supplements, as well as the NSF International Joint Committee on Dietary Supplements. The US NIH has provided funding to support these activities in 2003/04.

Collaborations with NIST have advanced the certification of the first suite of herbal product Standard Reference Materials (SRMs) for the ephedra alkaloids, amphetamine-like compounds with potentially powerful stimulant effects on the nervous system and heart. In various formulations they are often used for weight loss, increased energy and enhanced athletic performance. We supplied data to NIST based on a variety of extraction protocols and detection systems as well as from application of FAIMS (see High-Field Asymmetric waveform Ion Mobility Spectrometry, page 8). This unique capability in the Chemical Metrology Group permitted quantitation of the alkaloids in extracts without an intervening chromatographic separation step. These new SRMs will be released in 2004. The Chemical Metrology group has identified American ginseng (Panax quinquefolius) and Asian ginseng (Panax ginseng) as its first suite of new CRMs. Also, a dried root powder and 5:1 extract have now been sourced for American ginseng. Canada is the world's largest producer of American ginseng with centres of production in

British Columbia and Ontario. American ginseng is also Canada's most important medicinal plant, estimated to be worth over one hundred million dollars a year.

Canadian Chemical, Radiological, and Nuclear Research and Technology Initiative

P. Grinberg, S. Willie, R. Sturgeon

Following a radiological/nuclear terrorist attack, an analytical system that can screen samples and provide results in minutes rather than days or weeks is essential to rapidly assess and mitigate health, economic and environmental damage. Rapid radio-analytical techniques apply broadly to the following key issues:

- surveillance: to monitor specific radionuclides currently so difficult to measure that it is rarely done
- immediate reaction and consequence management: to quickly assess and identify contaminated first responders so that they can be treated rapidly to reduce the uptake of harmful radionuclides into their bodies
- long-term management: to assess contaminated foods, water supplies and other commercial products
- criminal investigation: to identify the origin of the nuclear material to support attribution
- public confidence: to reassure individual members of the public, and test that they are not contaminated or at risk.

In each of these areas, rapid radionuclide detection and identification are needed to assess the risk so that an action plan can be developed to manage the situation. Except at high concentrations, the actinides (e.g. Pu-239) and key beta emitting radionuclides (e.g., Sr-90) cannot be identified and quantified with existing radiometric detection systems. Even in dedicated radiochemistry laboratories, many of these key radionuclide analyses are very slow and labour intensive.

Over a period of four years, the Chemical Metrology Group, with Health Canada, Trent University and MDS Sciex, received funding from the Canadian Chemical, Radiological and Nuclear Research and Technology Initiative (CRTI) under a Memorandum of Understanding to collaborate on a *New Technologies for the Rapid Assessment of Radioactive Contamination* project designed to address the above key issues. This collaboration seeks a rapid analytical protocol to provide radionuclide measurements in minutes rather than hours or days. This will be accomplished by: eliminating manual sample preparation, separating the radionuclides of interest from other interferences (including isobars) on-line, using gaseous volatilization modifiers, chromatographic and dynamic reaction cell techniques, and replacing conventional counting with mass spectrometric (ICP-MS) detection of the radionuclides. Based on its extensive past experience with plasma source mass spectrometry and electrothermal atomization, INMS has elected to focus on ICP-MS sample introduction via electrothermal vaporization (ETV), coordinating with the efforts of its partners to deliver a workable system which will satisfy the above objectives. Several unique ETV devices have been constructed and various volatilization modifiers tested for detection of Cs, U and Th.

Vapour Generation

R. Sturgeon, Z. Mester, J. Lam, X.-M. Guo, T. Matousek, M. Kan, S. Xu

The Group has a long history of significant contributions to the development of vapour generation as an ultra-trace analytical technique. It has pioneered in-situ concentration approaches with the graphite furnace for atomic absorption detection, and expanded the suite of elements amenable to vapour generation with tetrahydroborate (III) reagents to include several transition and noble metals. More recently, the scope of vapour generation appears ready to undergo yet another remarkable revolution. Use of low molecular weight acids added to sample solutions permits the high yield reductive synthesis of volatile compounds of a number of metallic species under exposure to UV radiation at mild fluxes. Numerous potential applications are open for exploitation, including environmental remediation, organometallic synthesis, purification of metals and enhanced sample introduction efficiency for detection by atomic spectrometry. We are currently studying the products and chemistry relating to this vapour generation approach.

Determination of Organic Contaminants

A. Windust, G. Gardner, C. Fraser, P. Maxwell, M. McCooeye

The organic analytical section of the Chemical Metrology Group has expanded significantly in the past four years. We have built a state-of-the-art small molecule mass spectrometry facility equipped to handle both aqueous and volatile samples. Methodology for improving the speed and efficiency of the extraction and clean-up of sediment and tissue samples prior to determination of organic contaminants by GC and GC/MS is under continuous development. We are evaluating sample cleanup with florisil and alumina columns by studying the impact of column preparation and treatment on separation of PCBs and pesticides. Methods for removal of interferences, particularly sulphur, using activated copper are continuously being refined. Solid phase extraction and accelerated solvent extraction techniques have been shown to be time-saving alternatives to Soxhlet extraction and were evaluated for preparation of samples for PCB and pesticide determinations. The Chemical Metrology Group continues to participate in high level comparison exercises under the auspices of the CCQM, such as the recent CCQM-P31b (PCBs in iso-octane) and CCQM-P31c (chlorinated pesticides in iso-octane) exercises.

High-Field Asymmetric Waveform Ion Mobility Spectrometry

Z. Mester, M. McCooeye

The Group continues to work in formal collaboration with Ionalytics providing much needed mass spectrometry TOF instrumentation and laboratory space to pursue their future studies for expanding the application of FAIMS. We used the technique with liquid chromatography (LC) electrospray sample introduction to provide analytical data characterizing a number of ephedra alkaloids in a suite of candidate NIST SRMs. Because this approach does not require classical separation of these analytes by conventional chromatography, it may be considered an independent measurement technique and was thus an invaluable aid to the NIST certification process. Several of our LC-MS machines are now equipped with custom FAIMS interfaces.

Glow Discharge Mass Spectrometry

B.A.J. Methven, A.P. Mykytiuk, K. Henderson

The glow discharge mass spectrometry (GD-MS) facility, unique in Canada, provided elemental analysis of approximately 2100 samples of high purity conducting and semiconducting materials (e.g., gallium, cadmium, tellurium, gallium arsenide, cadmium selenide, cadmium zinc telluride) for a group of about 42 clients, mostly in the electronics industry. The Standards Council of Canada has fully accredited the laboratory to ISO/IEC 17025. This accreditation provides the needed compliance options for many clients. Elemental analysis of high-purity materials by glow discharge mass spectrometry continues to be a high priority service offered to Canadian and foreign industries. In order to deliver more accurate and precise data to clients, the facility requires more investment in research to develop alternative means and methodologies to directly address analyte concentration levels in solids at the sub ng/g level.

International Comparisons

A growing demand on Group resources is participation in international comparison programs. Both nationally and internationally, it is becoming increasingly important to achieve and demonstrate measurement traceability. International comparisons of primary standards serve as the foundation for traceability claims and capabilities underlying all Memoranda of Understanding and Mutual Recognition Arrangements. The Group took part in the following comparisons in 2003-2004:

- NOAA National Status and Trends Program
  Participated in the sixteenth comparison for trace metals in marine sediments and biological tissues with Canadian and American laboratories that historically take part in the National Status and Trends Program of the U.S. National Oceanic and Atmospheric Administration.
- SIM.8.10P: Trace Elements in Drinking Water
  Coordinated a SIM pilot comparison exercise targeting the determination of trace elements in drinking water.
- CCQM-K18: TBT in sediment
  Coordinated a key calibration exercise under the auspices of the CIPM Consultative Committee on Amount of Substance (CCQM) aimed at assessing the capabilities of NMIs for the quantitation of tributyltin in marine sediment.
- CCQM-P43: DBT in sediment
  Coordinated a pilot calibration exercise under the auspices of the CIPM Consultative Committee on Amount of Substance (CCQM) aimed at assessing the capabilities of NMIs for the quantitation of dibutyltin in marine sediment.
- CCQM-P39: As, Hg, Pb, Se and Methylmercury in Tuna Fish
  CCQM-P31b: PCBs in iso-octane
  CCQM-P31c: Chlorinated pesticides in iso-octane
  Participated in pilot comparisons.

- NIST SRMs 3240-3244Cd and Pb comparison
  Provided analytical data to aid the certification of a suite of four ephedra-based SRMs in support of the nutraceutical industry and to strengthen our existing collaboration with the Analytical Chemistry Division of NIST (USA).

The Group also participated in the annual international Proficiency Testing Program (Quasimeme AQ1 Nutrients in Seawater, Round 32 Exercise 544 and Round 34 Exercise 581) assessing capabilities for the determination of nutrients in seawater.

Committees and Offices

International

AOAC International

Ninth International Symposium on Biological and Environmental Reference Materials (BERM) Conference, Berlin, Germany
S.N. Willie, Scientific Committee

International Task Group on Dietary Supplements
R.E. Sturgeon, Member

NSF International

Joint Committee on Dietary Supplements
R.E. Sturgeon, Member

Comité international des poids et measures (CIPM)

Consultative Committee on Amount of Substance (CCQM)
Inorganic Working Group
R.E. Sturgeon, Member

European Virtual Institute for Speciation Analysis

R.E. Sturgeon, Advisory Board member

National

Standards Council of Canada (SCC)

ISO/TC 147/SC2: WG Metals by ICP-MS
J.W.H. Lam, Member

ISO TC-147: Canadian Advisory Committee, Water Quality
S.N. Willie, Member

Spectroscopy Society of Canada, Ottawa Valley Section

J.W.H. Lam, Treasurer

Chemical Institute of Canada

Analytical Chemistry Division
S.N. Willie, Chairperson

Adjunct Professorships

R.E. Sturgeon, Adjunct Research Professor, Dept. of Chemistry, Carleton, University, Ottawa

Editorships

Spectrochimica Acta Reviews
R.E. Sturgeon, Editor

Journal of Analytical Atomic Spectrometry
R.E. Sturgeon, Advisory Board Member

Atomic Spectrometry Updates
R.E. Sturgeon, Advisory Board Member

Encyclopedia of Analytical Science
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Published Papers and Presentations

This publication list is for the calendar year 2003. A complete list of publications, from 1999, is available on the INMS web site at http://inms-ienm.nrc-cnrc.gc.ca/research_and_development/chemical_metrology_publications_e.html.

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Mester, Z. "UV light mediated alkylation of trace metals", International Conference on Analytical Sciences and Spectroscopy, Ottawa (June 2003).

Mester, Z. "Speciation in nutritional supplements", IR Annual Scientific Exchange 2003, Quebec City (September 2003).

Mester, Z. "Volatile Metal Derivatives", 39th IUPAC Congress and 86th Conference of The Canadian Society for Chemistry, Ottawa (August 2003).

Mester, Z., R.E. Sturgeon, X.-M. Guo and **G.J. Gardner.** "Novel vapor generation approaches for trace element analysis: UV light mediated alkylation of selenium and nickel", FACSS 2003, 30th Annual Meeting, Ft. Lauderdale, Florida (October 2003).

Willie, S.N. "NRC's CRM Program: Past, Present and Future", International Conference on Analytical Sciences and Spectroscopy, Ottawa (June 2003).

Sturgeon, R.E. "Improved Sample Introduction Systems for Atomic Spectrometry", Pittcon 2003, Orlando, Florida (March 2003).

Sturgeon, R.E. "Natural Health Products: a Metrology Laboratory Response to Regulation, Compliance and Enforcement", Challenges and Opportunities in NHP Anlaysis, BC Functional Food and Nutraceutical Network, Vancouver (June 2003).

Sturgeon, R.E. "Vapor Generation Sample Introduction for Atomic Spectrometry", CSI XXXIII, Presymposium on Sample Introduction Strategies (September 2003).

Sturgeon, R.E. "Speciation, Reference Materials and Methodologies - A Synergistic Experience", 5th International Symposium on Speciation of Elements in Biological, Environmental and Toxicological Sciences, Almunecar, Spain (September 2003).

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Technical Reports

Willie, S.N. Sixteenth Intercomparison for Trace Metals in Marine Sediments and Biological Tissues. NRC Document No. 42782 (2003).

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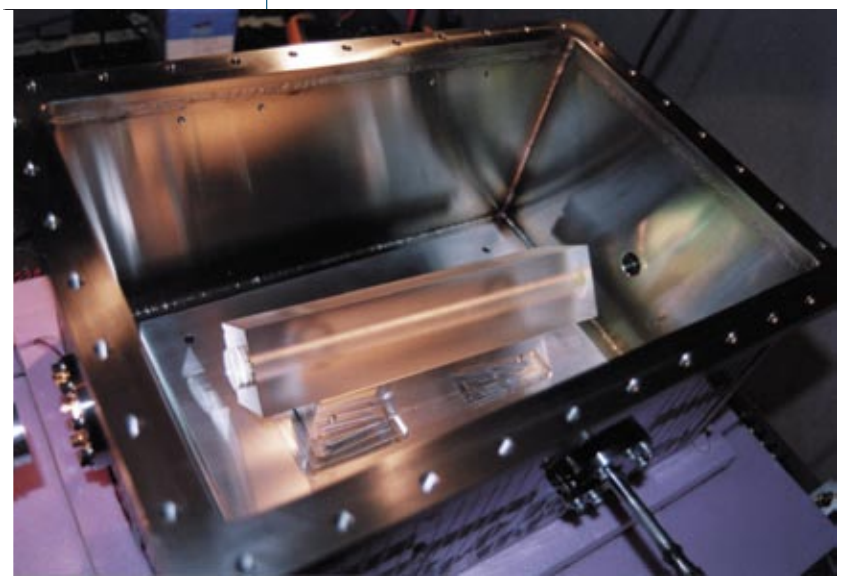
An improved vacuum system is being tested for the Cesium Fountain Atomic Clock. Entirely built with non magnetic materials, the system will include two newly developed electro-static shutters, a longer detection zone and a uniform C-field. With this enhancement, the Fountain Clock will be the Time Standards Program's most accurate clock and NRC's best realization of the SI second. As well, this long-term time standard will provide a better reference for optical frequency measurements and a greater contribution to the international time scale maintained by the BIPM.



With the completion of the new ultra-stable probe laser, linewidths below 100 Hz at 445 THz (674 nm) have been observed for the dipole-forbidden 5s $^2S_{1/2}$-4d $^2D_{5/2}$ transition in the single trapped and laser-cooled $^{88}Sr^+$ ion. As a result, it has been possible for a computer, which counts the quantum jumps in the fluorescent cooling radiation, to lock the frequency of the ultra-stable laser to the centre of the S-D transition with an error of only a few hertz. We have used the femtosecond optical frequency comb to measure the absolute frequency of the probe laser, and hence, that of the line centre of the S-D transition. These measurements have determined a value of 444 779 044 095 477 Hz, with a standard uncertainty of 7 Hz ($2x10^{-14}$). The accuracy has advanced to the level where it is currently limited by the uncertainty in the NRC realization of the SI second.

Time Standards Program

The Time Standards Program is concerned with generation of the SI units of time (the second) and frequency (the Hertz) for Canada in accordance with their internationally accepted definitions in terms of a microwave transition in cesium atoms. The Frequency and Time Standards Group maintains primary cesium clocks and other facilities to provide internationally coordinated continuous time scales and frequency standards. Official time is disseminated for legal, commercial and other purposes. We calibrate clocks and frequency standards, both in our laboratories and remotely by signal or artefact exchanges, for use in navigation, telecommunications and other areas of science and engineering.

Time Dissemination

S. Cundy, J.-S. Boulanger, R.J. Douglas, R. Pelletier, B. Hoger, L. Proulx

Canada's frequency and time standards are continually compared against those of other national metrology institutes (NMIs) through the BIPM, using GPS-based common-view techniques. Over the past thirty years, NRC has been able to provide input to the international time scale construction of the BIPM, by developing its own atomic frequency standards based on cesium beam tube technology.

A compatible link is established between various types of frequency standards, based essentially on the exchange of frequency or timing signals. A local time scale has to be maintained in order to characterize the frequency relationship between devices. Hydrogen masers are the tools of choice for periods ranging from 1 second to several days. They are instrumental in the validation of cesium microwave frequency standards and optical frequency standards as well as in calibrations in client laboratories. To realize a proper short-term time scale, a new, state-of-the-art hydrogen maser was purchased and commissioned in FY 2003-2004.

The new maser will also maximize the benefits of a collaboration with Natural Resources Canada (NRCan) and the US Naval Observatory. The Canadian Active Control System (CACS) project, led by NRCan, aims to provide Canadian users real time corrections for GPS signals in order to improve GPS positioning accuracy. NRCan has installed over $180 K worth of equipment at NRC to take advantage of our stable time scale.

The project will strengthen the time and frequency references provided to external clients and to projects within the Frequency and Time Group. It will link the NRC Frequency and Time program with the Natural Resources Canada CACS Program to give Canadian users nearly direct access to international time scales and the SI definition of the second.

The definition of the unit of length is directly derived from the SI second and is the basic building block for dimensional measurements and telecommunication.

Cooled Cesium Fountain Clock

L. Marmet, E. Guillot, C. Gigault, B. Hoger, J.-S. Boulanger, R. Pelletier

The Cooled Cesium Fountain Clock Project is developing a clock with a higher accuracy than any of the existing clocks maintained by the group. This new clock will operate with laser cooled atoms in a magneto-optical trap. The atoms will be launched vertically through a microwave cavity to increase the Ramsey interrogation time up to 640ms, thus providing a 30-fold improvement in the linewidth of the cesium resonance compared to our cesium beam clocks. This will proportionally improve the resolution and the accuracy of the Group's primary standards and benefit clients of Canada's time services. Additionally, the higher accuracy will benefit the fundamental physical measurements made in the Optical Frequency Standards Program. Lastly, these advancements will bring greater international recognition to Canada by increasing it's contribution to the International Atomic Time.

The progress on NRC's first cesium-fountain atomic clock continues with tests, new designs and the construction of several of the components of the clock. Earlier tests made with the prototype clock produced a time signal with a stability comparable to that of the cesium beam clocks. These tests were crucial for the design of the first fountain clock primary standard. The optimum launch height was found to be between 30 cm and 50 cm above the microwave cavity. A new type of electro-static shutter was designed and is being built to reduce light shifts. Improvements to the detection system are being made with a high-stability laser interferometer necessary for the high performance of the device. Additionaly, the four detection zones are separated by sufficient space to eliminate cross talk and increase detection contrast.

A collaboration with the Optics group resulted in the production of eight large windows, also used in the detection area, with anti-reflection coatings. A new synthesizer was acquired for the stable generation of the 9.192 GHz microwave field and interfaced to the system's control computer. The C-field was improved by making the magnetic field more uniform near the end of the shields resulting in a shorter clock that will have a stronger detected signal. The entire vacuum chamber was redesigned and built from non-magnetic materials (titanium, copper and aluminium). The entire optical system was rebuilt inside a temperature stabilized box to improve the long term stability of the laser source.

Optical Frequency Standards Program

The Optical Frequency Standards Program is concerned with accurately measuring the frequency of electromagnetic radiation in the optical region of the spectrum, and developing frequency-stable optical sources. Optical frequency standards are important for a number of applications, for example: dimensional metrology, atomic and molecular spectroscopy, and precise time keeping.

The program maintains the SI metre for Canada through the vacuum wavelength of an ensemble of iodine-stabilized helium-neon (I_2/HeNe) lasers at 633 nm or 474 THz. The program offers accurate vacuum wavelength/frequency

calibrations, traceable to the SI metre, for client lasers over much of the visible and near infrared spectral region. Client lasers at 633 nm can be calibrated directly through heterodyne beat measurements with the ensemble lasers. The program also maintains an optical frequency comb and can provide calibrations at other wavelengths from 550 nm to 1150 nm, traceable to the SI metre, with the accuracy normally limited by the reproducibility of the client laser. Facilities are also available for the calibration of vacuum wavelength/frequency in the telecommunication region near 1500 nm and at 9-11 μm. Calibrations at other wavelengths may be possible through special arrangements.

The program performs research in a number of areas. It has developed frequency standards based on saturated absorption in molecules, and maintains them in the optical telecommunication region of 1500 nm and in the infrared at 9-11 μm. The program also developed and maintains a classical frequency chain for measuring optical frequencies in the 30 THz (10 μm) region and femtosecond optical frequency combs at 1500 nm and 550-1150 nm. The development of an optical frequency standard at 674 nm (445 THz), based on a narrow (0.4 Hz) transition in a single trapped and laser-cooled $^{88}Sr^{+}$ ion, has been one of the laboratory's long-term projects. Since this single ion can be isolated from environmental perturbations, it makes an ideal frequency standard with a potential reproducibility several orders of magnitude better than that of the best cesium and hydrogen-maser microwave standards. Optical standards have already shown superior short-term stability, and can be used to evaluate microwave standards, or even replace them for certain applications.

Strontium Ion Frequency Standard

A.A. Madej, P. Dubé, J. Bernard, L. Marmet

Trapped and laser-cooled single ion standards promise unsurpassed frequency accuracy. Certain ion systems are predicted to reach levels of 10^{-17} to 10^{-18} relative accuracy once the small systematic shifts of a well-chosen narrow transition are understood and controlled. In our experiments, a trapped and laser-cooled ion is held in a shielded, high-vacuum environment and confined to a sub-micron length scale much smaller than the probe wavelength (Lamb-Dicke regime). Such a single, cooled, and isolated particle provides a nearly ideal system for a frequency standard.

We have been developing the $^{88}Sr^{+}$ ion frequency standard for a number of years. We probe the frequency of its narrow, 0.4 Hz wide, 5s $^{2}S_{1/2}$-4d $^{2}D_{5/2}$ transition at 445 THz (674 nm) – the so-called *clock transition* – with an ultra-narrow and ultra-stable probe laser source. The *absolute* frequency of the probe laser is then measured relative to the ensemble of NRC cesium atomic clocks by an octave-spanning femtosecond optical frequency comb system. This system acts as a direct link between the atomic clocks' rf frequency (provided by a hydrogen maser), and the optical frequency of the probe. The aim is to produce a practical and reliable frequency/time standard in the visible region of the spectrum, with a performance superior to that of cesium atomic clocks.

We have made significant progress during the past year in the measurement of the absolute frequency of the $^{88}Sr+$ ion frequency standard, for two main reasons. First, the reliability and relative ease of making absolute frequency measurements with the optical frequency comb (compared to the older frequency synthesis chain) has allowed us to make measurements on a regular basis, and to carry out studies of the ion trap frequency reproducibility under changes in the operating parameters. Second, a new probe laser system, with improved linewidth (50 Hz–120 Hz FWHM) and stability, has provided more accurate and reproducible determinations of the ion linecentre than ever before in our laboratory. We have measured the ion frequency by probing a number of different Zeeman component pairs of the 5s $^2S_{1/2}$-4d $^2D_{5/2}$ transition under a large number of magnetic field orientations to study the effect of the quantization axis direction. These measurements revealed the presence of a quadrupole shift that was determined by combining the measurements of several pairs of Zeeman components. The best present value for the ion frequency linecentre is 444 779 044 095 477 Hz, with a standard uncertainty of 7 Hz or 2 parts in 10^{14}. We expect the remaining systematic shifts will be small (< 1 Hz), but we require more studies to evaluate them properly. The uncertainty of the NRC rf reference signal for the absolute frequency measurement is 1 part in 10^{14}.

We performed an extensive series of calculations examining the limiting systematic shifts of the single ion standard. The shifts examined include those due to the second-order Doppler effect (relativistic time dilation), stray fields, blackbody radiation, and other contributions. We incorporated these calculations into the 2002 absolute frequency measurements of the Sr^+ single ion with the NRC frequency comb. A paper was prepared and has been accepted for publication. Precise experimental evaluation of the sources of uncertainty requires more studies of the ion trap's sensitivity to changes in the operating parameters. We are constructing a new trap that will provide better control over the ion environment (such as trimming electrodes to center the ion in three dimensions), and cooling in three dimensions. Furthermore, two ion traps will make it possible to study the stability and reproducibility of the ion clock frequency without the limitation imposed by the instability of the cesium clocks, currently at the 10^{14} level. A better absolute frequency determination of the ion frequency will be possible in the near future, once the NRC cesium fountain becomes operational.

We plan to improve the probe laser linewidth further, to make even more accurate frequency determinations of the ion linecentre in shorter averaging times. This will become especially important when directly comparing the two trapped ion systems, and when making studies of the systematic shifts of the ion clock frequency.

The Femtosecond Optical Frequency Comb

J.E. Bernard, P. Dubé, A.A. Madej

Whereas, only four years ago, years of effort were required to build a frequency chain to measure just one optical frequency, the femtosecond optical frequency comb (OFC) has made it possible to measure almost any optical frequency in a single day. The NRC OFC was completed approximately two years ago and is now used routinely in calibrations of optical frequencies.

The OFC is based on a mode-locked Ti:sapphire laser which produces a train of 30-50 fs pulses at a repetition rate of approximately 700 MHz. The spectrum of these pulses is broadened to more than an optical octave through self-phase modulation in a special microstructured optical fibre. On close inspection, the spectrum (Fourier transform of the temporal behavior) consists of hundreds of thousands of distinct optical frequencies arranged in a regular comb with a spacing equal to the laser's repetition frequency. The repetition frequency and the common offset of the comb elements from perfect harmonics of the repetition frequency, are phase locked to stable rf signals provided by a hydrogen maser, which is referenced to the NRC ensemble of cesium time standards. In this way, the OFC transfers the accuracy of the hydrogen maser to each of the comb elements, any one of which can be used in a heterodyne beat measurement of an unknown laser source's optical frequency.

In the past year, we have performed measurements at 260 THz (1153 nm), 390 THz (770 nm from a frequency doubled source at 1540 nm), 445 THz (674 nm), 474 THz (633 nm), and 552 THz (543 nm). The ultimate accuracy of the OFC is limited by the accuracy with which the hydrogen maser produces a known frequency (approximately 1 part in 10^{14}), and is typically several orders of magnitude better than the reproducibility and stability of the laser sources undergoing measurement. However, this is not the case in measurements of our 445-THz ultra-stable laser, which acts as the probe in our single ion experiment. There, we found that the comb shows a 1-s instability of 200 Hz or 5×10^{-13}, approximately twice the 1-s instability of the hydrogen maser. If the sample time is increased to 10 s, the sample standard deviation drops to only 25-30 Hz. Our current measurements of the ion's transition frequency have a statistical uncertainty of 5 Hz or 1×10^{-14}, and appear to be limited by the accuracy of the SI second as realized through the NRC clock ensemble.

Laser Calibrations

J.E. Bernard, A.A. Madej

The Optical Frequency Standards Program calibrates lasers used by the INMS Dimensional Metrology Program. Lasers at 633 nm are calibrated through the ensemble of I_2/HeNe lasers, while lasers at 543 nm, 612 nm, and 1153 nm have been calibrated by means of a tuneable dye laser system that is locked to known transitions in iodine. The argon-ion pump laser used in the latter system is failing, and as a result, we decided to investigate whether our OFC could be used for these calibrations. We were uncertain whether the span of the OFC was sufficient to calibrate all the dimensional metrology lasers, whose frequencies span over an octave. Test calibrations were carried out at 543 nm and 1153 nm. It was found that, with proper adjustment, the comb provided the necessary power for successful calibrations at both these wavelengths, although at 1153 nm, it was difficult to obtain the strong signals required for electronic counting. In these situations, a modified method of recording the data, which provides sufficient accuracy for the calibration of the lasers, can be used. These experiments, along with successful calibrations at several other wavelengths, show that the OFC can accurately measure optical frequency at wavelengths anywhere in the range of 550 nm to 1150 nm.

Optical Frequency Standards and Measurement Techniques in the 1.5 micron Region of the Spectrum

A. Czajkowski
, S. Chepurov,
A.A. Madej,
J.E. Bernard, P. Dubé

This project focuses on developing optical frequency standards and methods of absolute optical frequency measurement in the optical telecommunications spectrum. Some current applications include: the traceable calibration of wavemeters and optical spectrum analysers, the measurement of tuneable lasers, and the allocation of optical frequency channels. This work began in 1999 as part of a joint program supported by the Canadian Institute for Photonic Innovations (CIPI). One of the Program's main thrusts has been to develop a precision diode laser system at 1.5 μm (194 THz) that utilizes stabilization to saturated absorption resonances in acetylene. The performance of this system is now equivalent to that used for the practical realization of absolute wavelength/frequency in the visible spectrum, as obtained by the 633-nm iodine-stabilized HeNe laser, and is a significant achievement in bringing such precision and accuracy to the 1.5 μm region. We published studies of the frequency-stabilized laser system's sensitivity to the operating parameters of cell pressure, power, and modulation excursion, together with studies of the linewidth, stability, and reproducibility. The results showed that moderate control of the operating parameters resulted in a system reproducibility at the kHz level.

In addition, we have obtained the absolute frequencies of a number of reference transitions.The radiation provided by the laser standard was amplified via an optical fiber amplifier and frequency, doubled in waveguide-based periodically-poled lithium niobate. This produced 10 to 30 μW at 770 nm, which was sufficient to be measured with the optical frequency comb system. Absolute frequency measure-ments of the laser frequency standard were obtained for six different ro-vibrational reference lines in acetylene ($^{13}C_2H_2$) at the sub-kilohertz level ($< 5x10^{-12}$). The P(16) line adopted by the Comité International des Poids et Mesures (CIPM) in 2001 as a frequency/ wavelength reference in the 1.5 μm optical telecom region of the spectrum was among the transitions studied. A paper describing these results has been published. Work also continued on a novel ultra-stable reference cavity constructed of ultra-low expansion glass. The device's purpose is to investigate the accuracies obtained by such a macroscopic reference standard, by providing a comb of calibrated resonances spaced by the cavity free spectral range. In principle, the system could be simple to fabricate and see wide usage. Studies have shown that although the device demonstrates very good reproducibility of the cavity-free spectral range across the spectral region of interest, the dimensional stability of the device is limited by temperature fluctuations of the cavity spacer. We have implemented a temperature stabilization system to examine the optimal performance of the cavity, which should yield kilohertz level accuracy for the spacing of successive cavity modes.

In collaboration with Dr. A.J. Alcock at NRC's Institute for Microstructural Sciences, the project has achieved significant advances in the development of a mode-locked Cr^{4+}:YAG femtosecond laser system. We have tried to see if the laser can be used to generate a frequency comb for the mid-infrared region of the spectrum (1-2 μm). Experiments in which we injected the optical pulses into a photonic crystal fiber, specially developed for the infrared, resulted in a frequency comb spectrum spanning 1.3 μm to over 2.0 μm. The mode-locked laser system has been transferred

to INMS, where work on it has focussed on the accurate control of the spacing of the frequency comb elements through control of the laser repetition rate. Successful phase locking of the repetition rate to a hydrogen maser-referenced synthesizer signal was achieved, thus providing atomic clock accuracy to the spacing of the individual frequency modes of the comb. Work is currently directed at applying the IR comb to precision measurement of the optical frequency intervals for the entire series of acetylene reference lines provided by our 1.5 μm frequency standard. Combined with our absolute frequency measurements for six of these lines, these interval measurements will allow any of the over 80 lines, spanning 1510 to 1550 nm, to serve as an accurate standard wavelength.

Mid-infrared CO_2 - OsO_4 Laser Standard

K.J. Siemsen, J.E. Bernard, P. Dubé, A.A. Madej

The CO_2 - OsO_4 laser frequency standard was originally built and used successfully to bridge the frequency gap between an iodine-stabilized HeNe laser (I_2/HeNe) and the NRC strontium ion frequency standard. Subsequent direct frequency measurements of both the strontium ion frequency standard and the HeNe laser frequency, with NRC's optical frequency comb, bypassed the use of the CO_2 laser altogether. Although the original application of this laser has disappeared, it is still important to have a stand-alone standard in the 10-mm (30 THz) region with an accuracy better than that of the I_2/HeNe system. A laser stabilization scheme using two external cavities has achieved the desired accuracy. We use the first cavity to pre-stabilize the CO_2 laser frequency by the well-known Pound-Drever technique, while the second cavity, which contains the OsO_4 gas, is locked to the laser frequency. Long-term frequency stability is achieved by locking the laser frequency to the saturated absorption dip Q(15) of OsO_4. The Q(15) frequency was measured with the NRC infrared frequency chain and found to be 28 832 016 698 560±150 Hz. The results were published in early 2004.

We are currently investigating the effects of small changes in the operating parameters on the frequency of the CO_2/OsO_4 laser system. Since the frequency changes are very small, we have used the infrared frequency chain for these investigations in the past. However, the infrared chain is a very complex device, unsuitable for an extended series of measurements. We have therefore developed a low frequency phase-sensitive or lock-in technique, which allows us to detect small changes in the frequency of our standard laser when it is heterodyned against a laser with much worse stability and accuracy. A computer program slowly controls some laser parameter in a periodic manner (e.g., the laser power), and reads the heterodyne beat frequency as recorded by a counter. The program can post-process and filter the counter readings to pick the modulated signal out of the noise. Experiments have shown the technique to be effective.

Our isotopic CO_2 lasers are capable of operating from approximately 9 to 11 μm. In the past year we have used this tuneability to measure the transmission of some commercial hollow core fibres for industrial use in the difficult 8.9 μm to 9.2 μm wavelength region of the rest-strahlen band of quartz. The results are in good agreement with theory and show that the optimal transmission wavelength is below 8.9 μm. The results have been published.

Committees and Offices

International

Institute of Electrical and Electronics Engineers (IEEE)

Lasers and Electro-Optics Society (Ottawa Chapter)
J.E. Bernard, Treasurer

2003 Lasers and Electro-Optics Society (LEOS) Summer Topical Meeting on Photonic Time/ Frequency Measurement and Control (PTFMC), July 2003, Vancouver, B.C.
A.A. Madej, Conference Program Committee

2005 Lasers and Electro-Optics Society (LEOS) Summer Topical Meeting on "Optical Frequency & Time Measurement and Generation" (OFTMAG)
A.A. Madej, Conference Co-Chair

Comité international des poids et mesures (CIPM)

Comité Consultatif des Longueurs (CCL)
Mise-en-Pratique Working Group
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Comité Consultatif des Longueurs and Comité Consultatif de Temps et des Frequences (CCL/CCTF)
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National

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Published Papers and Presentations

This publication list is for the calendar year 2003. A complete list of publications, from 1999, is available on the INMS website at: http://inms-ienm.nrc-cnrc.gc.ca/research_and_development/frequency_time_publications_e.html and http://inms-ienm.nrc-cnrc.gc.ca/research_and_development/optical_frequency_publications_e.html.

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Articles in Conference Proceedings

Bernard, J.E., A.A. Madej, P. Dubé, L. Marmet, A. Czajkowski and R.S. Windeler. "Optical Frequency Comb Measurements at 633 nm, 674 nm, and 1556 nm", Proceedings of the 2003 IEEE International Frequency Control Symposium/17th European Frequency and Time Forum, Tampa, Florida. IEEE Press, Piscataway NJ. IEEE Catalog No. 03CH37409C, 162-167 (2003).

Madej, A.A., J.E. Bernard, A. Czajkowski, P. Dubé, L. Marmet, K.J. Siemsen and R.S. Windeler. "Accurate absolute frequency measurements across the optical spectrum using a single ion", Proceedings of the SPIE Vol. 5137 *Advanced Lasers and Systems*, Eds. Guenter Huber, Ivan A. Scherbakov, Vladislav Ya. Panchenko, (SPIE press, Bellingham WA), 373-380 (2003).

Madej, A.A., J.E. Bernard, P. Dubé, A. Czajkowski and **L.Marmet.** "Femtosecond Frequency Comb Measurements across the Optical Spectrum", 2003 Digest of the LEOS Summer Topical Meetings, (IEEE Press, Piscataway NJ, USA) IEEE Catalog No. 03TH8701, 71-72, (2003).

Oral Presentations

Bernard, J.E., A.A. Madej, P. Dubé, L. Marmet, A. Czajkowski and R.S. Windeler. "Optical Frequency Comb Measurements at 633 nm, 674 nm, and 1556 nm", 2003 IEEE International Frequency Control Symposium/17th European Frequency and Time Forum, Tampa, Florida (May 2003).

Madej, A.A., **J.E. Bernard, P. Dubé** and **L. Marmet.** "Precision Measurements on Laser Cooled Single Ions", 1st Canadian Cold Atom Workshop, Banff, Alberta (February 2004).

Madej, A.A., J.E. Bernard, P. Dubé, A. Czajkowski and **L. Marmet.** "Femtosecond Frequency Comb Measurements across the Optical Spectrum", Photonics Time/Frequency Measurement and Control Meeting, IEEE 2003 LEOS Summer Topical Meeting, Vancouver, BC (July 2003).

Madej, A.A., J.E. Bernard, P. Dubé and **L. Marmet.** "Toward the Optical Atomic Clock: Hertz Level measurements of the Strontium Ion Frequency Standard", 2003 Canadian Association of Physicists Annual Congress, Charlottetown, PEI (June 2003).

Marmet, L. and **A.A. Madej.** "NRC's Cooled-Cesium Time Standard" (presented by A.A. Madej), 1st Canadian Cold Atom Workshop, Banff, Alberta (February 2004).

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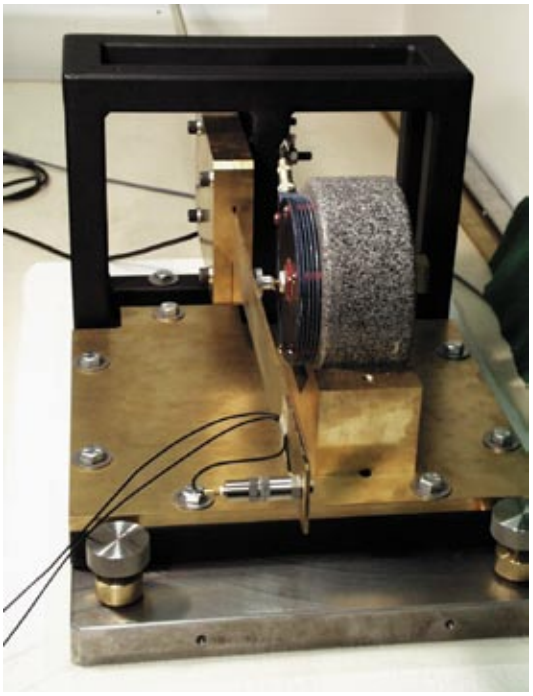
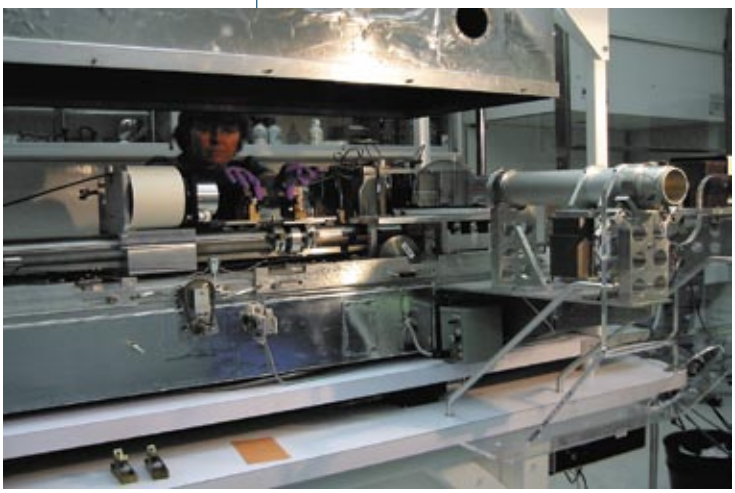
Mechanical Metrology

The Mechanical Metrology Group is comprised of three distinct programs. A report of the 2003-2004 activities of each program is offered within this chapter.

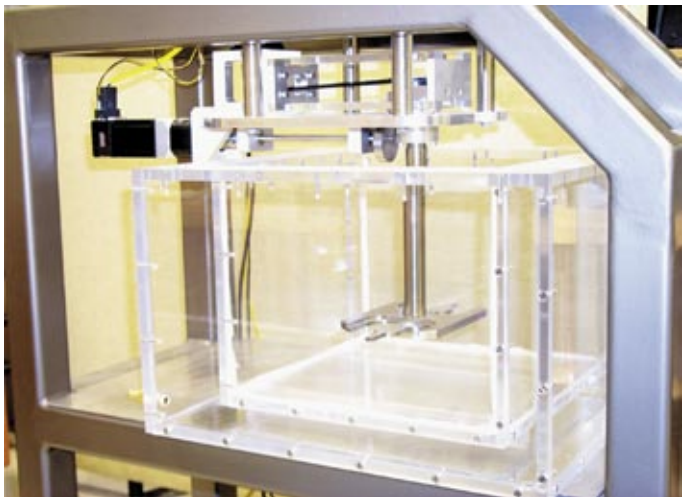
To satisfy the requirements of the calibration of accelerometers for low frequency applications such as seismic resource exploration and machine vibration monitor, Acoustical Standards has developed an economically attractive low frequency accelerometer calibration system by comparison. The principle of the system is based on a beam vibrating at its resonance frequency. With the system, NRC will be participating in the coming SIM comparison of low frequency calibration of accelerometers.





The SI metre is defined as the length of the path travelled by light in vacuum during a time interval of 1/299 792 458 of a second. NRC disseminates this definition by performing gauge block calibrations using the technique of optical interferometry. The Gauge Block Interferometer offers a measuring method to determine the number of wavelengths of light that span the length of the gauge block. These calibrated reference standard gauge blocks are then used to calibrate working standards in calibration and metrology laboratories.

New carousel for a volume comparator for two weights of stainless steel of up to 5 kg. The carousel is composed of a lower part, immersed in liquid, with a two positions handler, and of an upper part with step motors, belt and gears for exchanging the weights on a balance pan. The upward movement is based on a bent parallelogram moved by a cam.



Acoustical Standards

The Acoustical Standards program maintains the primary acoustical, ultrasound, and vibration standards for Canada. These standards provide the technical infrastructure for federal and provincial government guidelines on hearing conservation, the use of ultrasound for therapeutic and diagnostic applications, and machine noise and vibration emission. The program provides calibration services, technical consultation, and laboratory certification assessment. These activities help disseminate standards used by regulatory and international trade agencies, government departments, and the industrial and health sciences sectors.

High-power Ultrasound Standard

G.S.K. Wong, L. Wu

INMS maintains the Primary Ultrasound Standard for Canada. The Program provides traceability for Health Canada, and services for the calibration of the power output of medical ultrasound devices. The best expanded uncertainty (k=2) is 2.9%. We achieve this between 1 W and 5 W at frequencies of 1 MHz and 3.5 MHz. We provide a relatively inexpensive new service to calibrate the power output of medical ultrasound devices such as those used in diagnostics and physiotherapy in the range of 0.5 W to 5 W. The best measurement uncertainty (expanded uncertainty with k=2) is 4%.

Acceleration Standard

G.S.K. Wong, L. Wu, W. Ohm

We maintain the Canadian Primary Sinusoidal Acceleration Standard based on interferometric fringe counting techniques. Our services to industry include sinusoidal acceleration measurement from 10 Hz to 5000 Hz, with an expanded uncertainty ranging from 0.5% to 1.5% depending on frequency. We also offer high acceleration comparison shock measurement to 100 000 m/s^{2,} with a best expanded uncertainty of 3%.

We have developed a low frequency vibration generator, based on a cantilever beam, for calibration of accelerometers by comparison at low frequencies (from 2 Hz to 10 Hz), with an expanded uncertainty (k=2) of 2.6%. The system satisfied the requirements of ISO 16063-21: 2003.

International Comparisons

G.S.K. Wong, L. Wu, P. Hanes, W. Ohm

- CIPM Consultative Committee on Acoustics, Ultrasound and Vibration
  CCAUV.A-K3: Microphone Comparison
  The participants have completed the measurements, and the pilot laboratories (DPLA, Denmark /CENAM, Mexico) are preparing the Draft A report.

- Sistema Interamericano de Metrologia (SIM)
  SIM AUV.A-K1: Microphone comparison
  The measurements are complete. INMS, the pilot laboratory is preparing the Draft B report.

- SIM AUV.V-K1: Accelerometer comparison
  The measurements are complete, and the pilot laboratory (NIST, USA) is gathering data for the preparation of the Draft B report.

- SIM AUV.A-S1: Pistonphone comparison
  The measurements are complete, and the pilot laboratory (CENAM, Mexico) is preparing the Draft A report.

- SIM AUV.V-S2: Low frequency accelerometer comparison
  The pilot laboratory (CENAM, Mexico) is preparing the Technical Protocol in collaboration with other participants.

Client Services

G.S.K. Wong, L. Wu, P. Hanes

Based on the pressure reciprocity technique, staff calibrate primary microphones in an environmentally controlled chamber. The chamber enables the calibration to be repeatable to a few thousandths of a decibel. For the calibration of LS1P laboratory standard microphones, over a frequency range of 40 Hz to 10 kHz, the expanded uncertainty is between approximately 0.04 dB and 0.06 dB. For stringent calibrations, such as those for SIM and CCAUV comparisons, we used ISO standard air. We also provided microphone calibration by comparison, sound calibrators, and sound level meters.

Calibrations for our clients included sound level meters, microphones, accelerometers, sound calibrators, a medical ultrasound device for physiotherapy, and a belt-tension meter. The Program evaluated the variation of microphone sensitivity with atmospheric pressure for two models of laboratory standard microphones: Brüel and Kjær 4180 and G.R.A.S. 40AG. We developed empirical equations for sensitivity level pressure corrections for these microphones. With these equations, laboratories without an environmentally controlled chamber can apply corrections to their measurements, and obtain more accurate microphone calibration.

Equipment inspection was carried out for the Canadian Standards Association (CSA) Z107.53 Working Group's round robin tests on industrial sound identification.

The Program provided Calibration Laboratory Assessment Service (CLAS) reassessment of Institut de Recherche Robert-Sauvé en Santé & Sécurité du Travail (IRSST), Montreal, and gave advice on the APLAC AMP010 inter-laboratory comparison of sound level meter calibration.

ISO 17025 Quality System Implementation

The Acoustical Standards Program has completed external assessment in accordance with ISO/IEC 17025. For Vibration accreditation, the team leader was Mary Ryan, National Association of Testing Authorities (NATA), Australia, and the external expert, Dr. Hans Von Martens, PTB, Germany. For Acoustics and Ultrasound accreditation**,** the team leader and external expert were Elaine Dowd, SCC, and Dr. Bruce Meldrum, CSIRO, Australia, respectively. We expect to obtain accreditation from SCC by the end of 2004.

Patents

In an ongoing collaboration with ACO Pacific Inc., the Program designed and patented a sound isolation cap for sound level meters. The cap enables the user of sound level meters to discern whether electromagnetic interference is acting on their equipment.

G.S.K. Wong and N. Lewis. "Sound Insulating Cap for Sound Level Meters". Canadian Patent 2,199,060 10 February 2004.

Committees and Offices

International

AOAC International

Comité International des poids et mesures (CIPM)

Consultative Committee on Acoustics, Ultrasound and Vibration (CCAUV)
G.S.K. Wong, Member

Sistema Interamericano de Metrologia (SIM)

Metrology WG 9: Acoustics Ultrasound & Vibration
G.S.K. Wong, Member

Acoustics Ultrasound & Vibration (AUV)
G.S.K. Wong, Rapporteur

NORAMET

Acoustics Ultrasound & Vibration (AUV)
G.S.K. Wong, Technical Contact

Acoustical Society of America

Standards Committee in Acoustics S1
G.S.K. Wong, Vice-Chairman

Accredited Standards Committee on Acoustics, S1
G.S.K. Wong, Individual Expert

Accredited Standards Committee on Acoustics, S12
G.S.K. Wong, Individual Expert

Committee on Engineering Acoustics
G.S.K. Wong, Member

Committee on Noise
G.S.K. Wong, Member

Committee on Standards (ASACOS)
G.S.K. Wong, Member

Education Committee
G.S.K. Wong, Member

American National Standards Committees

ANSI S1- Advisory: Advisory Planning Committee
G.S.K. Wong, Chairman

ANSI S1-1: Standard Microphones and their Calibration
G.S.K. Wong, Member

ANSI S1-17: Sound Level Meters and Integrating and Sound Level Meters
G.S.K. Wong, Member
P. Hanes, Member

ANSI S1-25: Specification for Acoustical Calibrators
L. Wu, Member
P. Hanes, Member

ANSI S1-26: High Frequency Calibration of the Pressure Sensitivity of Microphones
G.S.K. Wong, Member

International Electrotechnical Commission

IEC/TC29: Electroacoustics

IEC/TC29/WG4: Sound Level Meters
G. S. K. Wong, Member

IEC/TC29/WG5: Calibration of Standard Condenser Microphones
G. S. K. Wong, Member

IEC/TC29/WG17: Sound Calibrators-Test Procedures
G.S.K. Wong, Member

IEC/TC29/WG18: Amendments of relevant IEC/TC29 standards with respect to developments on EMC
G.S.K. Wong, Member

IEC/TC29/MT 19: Revision of IEC 61260 (Filters)
G.S.K. Wong, Member

International Organization for Standardization

ISO/TC43/SC1/WG28: Sound Power Levels of Noise Sources
G.S.K. Wong, Member

ISO/TC43/SC1/WG25: Measurement of Sound Intensity
G.S.K. Wong, Member

ISO/TC108/SC3/WG6: Calibration of vibration and shock transducers
G.S.K. Wong, Member

Three international acoustical standards committees, (1) IEC/TC 29: Electroacoustics, (2) ISO/TC 43: Acoustics; SC 1: Noise; SC2: Building Acoustics; and (3) ISO/TC 108: Mechanical vibration & shock; and their subcommittees (SC 2, and SC 6) will meet in Toronto in 2005. The meetings are jointly organized by the NRC-INMS Acoustical Standards Program, the NRC-Institute for Research in Construction, Health Canada, and the SCC.

National

Canadian Standards Association

CSA Z107: Committee on Acoustics and Noise Control
G. S. K. Wong, Executive Member

CSA Z107.53: Quantities and Procedures for Description of Environmental Sound
G. S. K. Wong, Member

Subcommittee on Calibration, IEC/TC29 and related ANSI Activities
G. S. K. Wong, Chairman

Technical Committee B166: International System of Units (SI)
G. S. K. Wong, Chairman

Standards Council of Canada

Canadian Sub-Committee/IEC/TC29: Electroacoustics
G.S.K. Wong, Chairman
L. Wu, Member
P. Hanes, Member

Canadian Advisory Committee/ ISO/TC12: Quantities, Units and Symbols
G.S.K. Wong, Chairman

Canadian Advisory Committee/ISO/TC108/SC3: Use and Calibration of Vibration & Shock Measuring Instruments
G. S. K. Wong, Chairman

Canadian Advisory Committee/ISO/TC43/SC1: Acoustics
G.S.K. Wong, Member

Canadian Sub-Committee/IEC /TC87: Ultrasonics
G.S.K. Wong, Member

Editorships

Acoustical Society of America

Journal of the Acoustical Society of America (Acoustical Standards News)
G.S.K. Wong, Joint Associate Editor

Published Papers and Presentations

This publication list is for the calendar year 2003. A complete list of publications, from 1999, is available on the INMS web site at http://inms-ienm.nrc-cnrc.gc.ca/research_and_development/acoustical_standards_publications_e_.html.

Articles in Refereed Journals

Wong, G.S.K. and **L. Wu.** "Primary microphone calibration system stability,"*J. Acoust. Soc.Am.,* **114** (2), 577-579(2003).

Published Abstracts

Hanes, P., L. Wu, W. Ohm and **G. Wong.** "Calculation of uncertainty in calibration of microphones by the pressure reciprocity technique", *J. Acoust. Soc. Am.* ***113****(4), Pt. 2 (2003).*

Editorials

S. Blaeser and **Wong, G.S.K.** "Acoustical Standards News", *J. Acoust. Soc. Am.* **113**(1), 5-12, (January 2003).

S. Blaeser and **Wong, G.S.K.** "Acoustical Standards News", *J. Acoust. Soc. Am.* **113**(3), 1183-1186 (March 2003).

S. Blaeser and **Wong, G.S.K.** "Acoustical Standards News", *J. Acoust. Soc. Am.* **113**(5), 2375-2378 (May 2003).

S. Blaeser and **Wong, G.S.K.** "Acoustical Standards News", *J. Acoust. Soc. Am.* **114**(1), 7-13 (July 2003).

S. Blaeser and **Wong, G.S.K.** "Acoustical Standards News", *J. Acoust. Soc. Am.* **114**(3), 1191-1195 (September 2003).

S. Blaeser and **Wong, G.S.K.** "Acoustical Standards News", *J. Acoust. Soc. Am.* **114**(5), 2517-2523 (November 2003).

Books and Book Chapters

Wong, **G.S.K.,** "Air sound speed measurements and computation: A Historical Review", *Handbook of the Speed of Sound in Real Gases,* Vol. 3, Chapter 17, pp. 265-284, edited by Allan J. Zuckerwar (Elsevier Science (USA), (2002).

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Dimensional Metrology

The Dimensional Metrology Program provides calibration services for dimensional measurements that are traceable to the SI unit of length (the metre) in accordance with the internationally accepted definition. Dimensional parameters supported by the program include length, angle, flatness, roundness, diameter, surface roughness, and 3-D form.

Gauge Block and Length Bar Interferometry

J.E. Decker, D. Woods, L. Munro

We continue to implement phase stepping interferometry on the NRC-INMS gauge block interferometer (GBIF). For most of the year, J.E. Decker collaborated with PTB scientists in Braunschweig, Germany, developing the next-generation Kösters interferometer. This work, funded by the Alexander von Humboldt Foundation (Germany), emphasized the use of phase step interferometry and the integration of a refractometer vacuum tube; these activities improved the accuracy of corrections to the vacuum wavelengths due to the refractive index of air. Dr. Decker continued this work upon returning to INMS in August 2003. She returned to the phase stepping hardware, and began an upgrade with in-house software. She also started an analysis with the long-term goal of adding a refractometer to the GBIF. The installation of a refractometer in the NRC GBIF is a timely addition that will be particularly useful following the collaboration with the Optical Frequency Project. This latter collaboration resulted in the addition of a frequency-stabilized laser source at 1152 nm nominal wavelength to the GBIF. Long gauge block calibration data collected over the past two years have demonstrated a clear advantage of pairing the infrared with the optical wavelengths, however there are some small differences between gauge-length results obtained with optical and infrared wavelengths that warrant further investigation. Details, such as the correction for refractive index of air, are targeted at improving the successful application of near-infrared lasers to gauge block interferometry.

Angle Calibration Facility

J. Pekelsky, J.E. Decker, P. Nistico, L. Munro

An N-face optical polygon or N-step index table can divide the circle to generate large absolute angles of 360°/N. A small absolute angle θ, traceable to the SI metre, can be generated by a sine bar of calibrated length L, where one end is lifted a calibrated height h, such that sin θ = h/L. The NRC Angle Calibration Facility (ACF) is equipped with a high-resolution (0.001 sec) autocollimator, a high-accuracy 1440-step index table, and a laser-interferometer sine-bar, such that the ACF is able to self-calibrate its equipment, traceable to the SI metre and to absolute circle subdivision. Performance in calibrating optical polygons and angle gauge blocks to U = 0.15 sec was recently proven in the CCL K3 key comparison. We also use the ACF to calibrate client autocollimators and rotary or index tables. Additional jigs allow us to calibrate inclinometers and laser-interferometer angle optics in comparison with the ACF master autocollimator.

This year, we made new fixtures to better mount and align polygons and angle GBs onto the index-table stack for calibration by circle-closure techniques. We

developed a new 'weather station' (lab-environment sensor cluster) to correct the sine-bar laser wavelength for the effects of local air temperature, pressure, and humidity.

Aside from continuing to provide for client services, the ACF will begin a renewed R&D role in a planned nanometrology project: a grating diffractometer. Still in the design phase, this NRC-built instrument will calibrate grating pitch (a nano-sized line scale) by measuring the diffraction angles made by beams from a laser with calibrated wavelength. The ACF will be used to calibrate the commercial rotation stage's high-performance angle encoder (360° range measured to 0.01 sec resolution, accurate to U = 0.1 sec) of the commercial rotation stage used to orient the grating samples through the required diffraction angles.

Long-Bed Line-Standard Comparator

J. Pekelsky, P. Nistico, L. Munro

The NRC Long-Bed Line-Standard Comparator, installed on a 4.4 metre granite beam, will allow automated measurement of all kinds of line scale devices (microscope scales, encoders, precision rules, flexible tapes, surveyor leveling rods, etc.), up to 4 metres. We will conduct line sensing with a microscope/camera system using VIM techniques (see below). The line sensing will be flexible enough to view down to the finest lines of microscope scales (1 μm) and up to the coarsest lines of metal tapes (1 mm), and will even view other configurations of edges (such as sequences of squares or crosses). The target uncertainty for measuring the centre-position of best-quality lines is U = [25, 0.2 L] nm, where L is the measured length of line-scale intervals in millimetres.

In the last reporting period, a Dutch co-op student wrote a software driver for the laser interferometer system, in order to measure the position of the carriage as it scans along a line-scale artifact. In this reporting period, we made refinements to the carriage drive controller and air-bearing pads. We built special workpiece jigs, designed to mount and align two initial ranges of line scales: up to 100 mm, and up to 300 mm. These jigs have clear glass platforms to support and clamp the scales so they can be viewed in either transmitted or reflective illumination. The jigs allow each end of the scale to be leveled; they also, set the lateral position and make the 3 angular orientation alignments to the laser beam axis. An auxiliary fixture can be set along the instrument axis to allow quick confirmation that the microscope focus is properly set on the laser beam axis, ensuring zero Abbe offset. We also worked on a lab-environment sensor system, needed to correct the laser wavelength to account for air temperature, pressure, and relative humidity. Finally, we are developing a quadrant photosensor tool to perfectly align the laser beam (measurement scale) axis to the axis traced by the microscope focus as it travels the length of the bed on the carriage (to minimize the comparator-alignment cosine error).

We will next develop software to control the overall Long-Bed system. It will read a script of desired nominal measurement positions along a sample line scale. For each scripted position, the software will also run the motors that drive the carriage to within 1 μm of the nominal position, read the interferometer to measure the actual

carriage position, and use VIM techniques to automatically measure the remaining offset of the target line centre from the centre of the camera image. We combine this measurement with the interferometer reading to get the final distance from the reference origin. We will also investigate the fibre-optic 'cold' light source to illuminate the scale lines, as any spurious heating away from 20 °C causes the scale to change size.

Video Image Metrology (VIM)

J. Pekelsky, P. Nistico, L. Munro

We continue to develop a flexible system for non-contact image-based dimensional metrology. Our goal is to refine digital image processing (DIP) software to automatically find image target features such as simple lines, edges, or complex fiducial crosses, in order to extract specific location attributes in image space and relate them to dimensions on the actual object. The principle requires that object features be resolvable in the image (lines and blobs are visible); so VIM, for optical microscopy, is roughly limited to line widths greater than 2 x the wavelength of light (2 x 500 nm). However, images from any source can be used, meaning nano-sized structures imaged by scanning microscopes (AFM, STM, SEM) can benefit from our research. VIM is the core-competency toolbox for all nanoscale metrology.

Our current image capture system is based on a variable-magnification microscope with fibre-optic illuminators, a CCD camera, and a PC framegrabber. Image/object scaling can be tuned to the job, with a 600-pixel wide video image spanning as little as 0.2 mm on the object (1 pixel spans 300 nm). Sub-pixel line-centre 'pointing' performance depends on magnification and line quality, but the repeatability (1s) for best-quality lines (2 μm wide) is about 10 nm. The initial R&D was conducted in past years with the NRC-assembled microscope/camera system mounted on a 1-D mechanical comparator. While well suited for line scales, the comparator has only manual carriage translation. We are readying the motorized Long-Bed Comparator [see above] as the successor instrument for line-scale metrology.

This year we started R&D on more complex structures, using a commercial 3-D co-ordinate measuring machine (CMM) and its camera probe (Mitutoyo LEGEX CMM and QuickVision probe). This probe uses interchangeable lenses for different magnifications. Though this is less convenient than a zoom feature, the probe also offers useful integral LED illumination for ring and/or coaxial illumination of workpieces (limited to reflection mode only). One test object is a 26 x 26 'dot grid' consisting of a chrome-on-glass pattern of small circular disks (dots) in 3 sizes (1, 0.5 and 0.25 mm). We are interested in characterizing the performance of the CMM 'black-box' software/hardware system in performing VIM tasks. We have written programs that drive the CMM to centre each dot feature in the camera field, use the available DIP tools to extract the feature location in the image, and then report the overall measurement coordinates. By working at the centre of the image, image distortion effects are minimized, and the feature position can be measured with a repeatability of 50 nm (1s) or better. Software tools have been developed for finding disk centroids, but also for finding edge points around a disk perimeter, and reporting the centre coordinates and diameter of a fitted circle.

There is still much to be learned from the dot-grid, but work is planned for other objects, such as a knife-edge ring aperture – called a 'fusion standard''– that can be both optically viewed and mechanically probed. The fusion standard allows dual-probing CMMs (both image and mechanical) to correlate their two probe technologies.

Joint Project with CSIRO Australia - Piston-Cylinder Calibrator

J. Miles, J. Pekelsky, L. Munro, K. Doytchinov, P. Nistico

During 2002-2003, as a major step towards this goal, INMS developed a prototype instrument designed to measure the diameter of PCUs. The main measurement platform and component structure, with probe heads, motion stages, interferometers, and sample alignment and position, was designed by Lorne Munro in collaboration with John Miles (a visiting scientist from CSIRO) and Jim Pekelsky. The novel temperature-compensating all-stainless-steel structure required several thousands of man-hours to design, fabricate, and assemble. The instrument was completed and underwent preliminary metrology tests prior to shipment to CSIRO (Melbourne Branch) in August 2003. There, the instrument will undergo more detailed performance tests for the next stages of design, development, and testing. This joint project will likely span several years before the full metrology potential is realized. Our initial target is to measure diameters up to 50 nm to uncertainties the order of a few nanometers. We will eventually equip the device to measure roundness and straightness of cylindrical forms, also to uncertainties targeted at a few nanometres.

International Comparisons

- CCL-K2: Long Gauge Blocks
  NPL (UK) is piloting this comparison with one to three participants from each of the five regions. NRC is a member of the pilot team. Four gauges, ranging from 175 to 900 mm, were circulated from September 1999 to May 2001. The final report is undergoing final review.

- CCL-K3: Angle Standards
  CSIR (South Africa), piloting a comparison to link five regions, circulated a 12-face polygon and several angle gauges. NRC, a member of the pilot team, provided the polygon and angle gauges. The comparison is in the reporting stage.

- CCL-K6: 2-D CMM Artifacts
  The final report is in its final stages of revision.

- CCPR-S2: Aperture Area
  NIST (USA) is piloting with nine participants. Eight apertures were circulated (5 and 25 mm diameter, each in two materials, each in two edge profiles). This comparison is undertaken in support of the NRC Photometry & Radiometry Group.

- SIM 4.2: Gauge Blocks
  NRC piloted this comparison with participants from SIM and one from EUROMET. Twelve gauges, ranging from 2 to 100 mm, were circulated from June 1998 to June 2001. An Executive Summary was prepared, and the final report published in the Metrologia database for the CIPM-MRA.
- SIM 4.8: Surface Roughness & Step Heights
  The final report is undergoing final review.
- SIM 4.x: Long Gauge Blocks
  Planning for this comparison was initiated with NRC on the pilot team. Discussions regarding participation and piloting are ongoing.
- NIST (USA)-NRC bilateral comparison of 1-D CMM Artifact (620 mm step gauge)
  NIST was the pilot laboratory: the report preparation is underway.
- NML (Australia)-NRC bilateral comparison of Diameter/Roundness Standards (piston/cylinder pressure standard)
  A piston-cylinder pressure standard was sent to NML (Australia) for comparison testing. The report preparation is underway.
- CMI (Czech Republic)-NRC bilateral comparison of Diameter/Roundness Standards (piston/cylinder pressure standard)
  A piston-cylinder pressure standard was sent to CMI (Czech Republic) for comparison testing. The report preparation is underway.

Client Services

The CMM upgrade resulted in improved measurement accuracy and broadened the range of capability for custom dimensional calibrations. R&D is underway to further elucidate the capabilities and limitations of this state-of-the-art instrument.

A total of 33 calibration reports we issued for a wide variety of dimensional calibrations including:

- custom low-thermal-expansion (LTE) ceramic length bars and 54 gauge blocks calibrated by end-standard interferometry
- microscope stage micrometer calibrated using video image metrology
- roughness patch specimen
- glass hemispheres calibrated for roundness
- steel plug gauges and ring gauges calibrated for roundness and diameter
- CMMs using the NRC ball-plate
- step gauges, industrial die cavity and inserts, lens optical components, and radiometric apertures calibrated using the CMM
- plug gauges and ring gauges
- laser displacement system calibrated using the long-bed apparatus.

Committees and Offices

International

Comité international des poids et mesures (CIPM)

Consultative Committee for Length (CCL)
J.R. Pekelsky, Member

CCL WG on Dimensional Metrology (WGDM)
J.R. Pekelsky, Member

International Organization for Standardisation (ISO)

ISO/TC 213: Dimensional and Geometrical Product Specifications and Verification

ISO/TC 213/WG1: Roundness, Cylindricity, Straightness, Flatness
K.I. Doytchinov, Member

ISO/TC 213/WG2: Datums and Datum Systems
K.I. Doytchinov, Member

ISO/TC 213/WG5: Calibration Procedures for Surface Texture
K.I. Doytchinov, Member

ISO/TC 213/WG10: Coordinate Measuring Machines
K.I. Doytchinov, Member

ISO/TC 213/WG11: ISO3650 Gauge Blocks
J.E. Decker, Member

Interamerican Metrology System (SIM)

SIM Technical Committee
J.R. Pekelsky, Member

SIM Metrology Working Group for Length
J.R. Pekelsky, Member

SIM 4.x Long Gauge Block Calibration by Optical Interferometry
J.E. Decker, Member

NORAMET

NRC Technical contact for Length Metrology
J.R. Pekelsky

Optical Society of America (OSA)

J.E. Decker, Member

American Society of Mechanical Engineers (ASME)

Standards Committee B89.7: Measurement Uncertainty
J.E. Decker, Technical Consultant

National

Standards Council of Canada (SCC)

ISO/TC 213 Advisory Committee for WG1, WG2, WG5, WG10
K.I. Doytchinov, Member

ISO/TC 213 Advisory Committee for WG11
J.E. Decker, Member

Association for Coordinate Metrology Canada (ACMC)

Executive Committee
K.I. Doytchinov, Chairman

Editorships

SPIE Conference Proceedings "Recent Developments in Traceable Dimensional Measurements II", SPIE Vol. 5190, San Diego, California, USA, 4-6 August 2003, 477 pages. Chairs/Editors **J.E. Decker,** N. Brown.

Published Papers and Presentations

This publication list is for the calendar year 2003. A complete list of publications, from 1999, is available on the INMS web site at http://inms-ienm.nrc-cnrc.gc.ca/research_and_development/dimensional_metrology_publications_e.html.

Articles in Refereed Journals

Decker,**J.E.,** Miles, J.R., Madej, A.A., Siemsen, R.F., Siemsen, K.J., de Bonth, S., Bustraan, K., Temple, S., **Pekelsky**, **J.R.,** "Increasing the range of unambiguity in step-height measurement using multiple-wavelength interferometry – Application to absolute long gauge block measurement", Applied Optics,**42(28)**, 5670-5678 (2003).

Decker, **J.E.,** Alschuler, J., Castillo Candanedo, J., De la cruz Garcia, L., Prieto Esteban, E., Morales, R., Valente de Oliveira, J.C., Stone, J., Stoup, J., **Pekelsky**, **J.R.,** "Report on SIM.4.2 Regional Comparison Stage Two: Calibration of Gauge Blocks by Mechanical Comparison," Metrologia, **40,** Tech. Suppl., 04003, (2003).

Decker, J.E., "Erratum – Some important characteristics of gauge block artefacts for international comparison", Metrologia, **40,** 1 (2003).

Editorials

Decker J. Book Review: "Evaluating the Measurement Uncertainty: Fundamentals and Practical Guidance," *Metrologia*, **40(4),** 207 (2003).

Refereed Conference Proceedings

Decker, **J.E.,** Schoedel, R., Boensch, G., "Next-Generation Koesters Interferometer," Proc. of SPIE Conference Recent Developments in Traceable Dimensional Measurements, San Diego, CA, SPIE Vol. 5190, pp. 14-23, August (2003).

Oral Presentations

Decker, **J.E.,** Schoedel, R., Boensch, G., "Next-Generation Koesters Interferometer," PTB Colloquium Series, (May 2003).

Decker, **J.E.,** Schoedel, R., Boensch, G., "Next-Generation Koesters Interferometer for Calibration of Long Gauge Blocks," National Conference of Standards Laboratories International (NCSLI) Canadian Region Fall Meeting, (October 2003).

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John Miles (CSIRO, Australia)	
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Crhistian Baldo, UFSC, Brazil	(student)

Mass Standards

The Mass Standards Program establishes and improves traceability of mass and the derived units of pressure, density, force, and mass flow to the Canadian prototypes of the kilogram. These activities support trade and legal metrology, as well as other industrial and government activities. The program specifically supports the mandate of the NRC, under the Weights and Measures Act, with research, calibration, and active participation in the work of national and international standards organizations and regulatory bodies. The program also supports the activities of CLAS through calibrations, inspections, and documentation.

Mass

G.D. Chapman, G.R. Matthews, C. Jacques

Mass Standards activities directly address NRC's mandate under the Weights and Measures Act, and underpin Canadian legal mass metrology. This project involves the design, construction, and emplacement of equipment, programs, policies, and procedures for the improvement of our knowledge of the national prototypes of the kilogram and its dissemination to industry, government, and university laboratories.

The project's hardware tools are force comparators, which provide the data for mass comparisons. Computational tools, notably software developed in-house, permit accurate, traceable calibration of mass pieces, while taking account of important environmental influences. The most significant determinations are the estimates of variances and their propagation through the mass chain. The role of mass determinations in realizing the SI force unit is significant to the pressure project.

The maintenance and improvement of the Canadian mass scale continue to be a significant part of the work of the Program. This year we implemented a drift-trend linear fit to the values of the mass standards. As uncertainties increase over time with extrapolation, we use a tolerance analysis to decide if a weight needs to be re-calibrated.

Five Consultative Committee for Mass (CCM) Key Comparisons in the range of 100 mg to 10 kg were carried out this year. These comparisons take a large investment of resources, but are significant because they specifically test the abilities of the participating NMI laboratories over a large part of the normal mass range. The Program also piloted a magnetic susceptibility comparison.

As a result of the international commitment to ISO/IEC 17025 accreditation in National Metrology Institutes, senior researcher Geoerge Chapman frequently participates as assessor and peer reviewer. During the year he was a member of the assessment team for CSIR/NML (South Africa) and the peer review team for INMETRO (Brazil).

Density

C. Jacques

This project disseminates the derived SI unit of density (kg/m^3) to support industry and national and international trade. The project designs, constructs, and emplaces equipment, programs, and procedures for this dissemination.

The project draws heavily on the facilities of the mass project and is, in turn, indispensable to the accurate calibration of mass. The project requires force comparators, accurate mass standards traceable to the national prototypes of the kilogram, thermostatically controlled baths and enclosures, and fused silica transfer standards.

The density standard in use at NRC is still pure water, the density of which is determined from its temperature. We evaluated the isotopic content of NRC distilled tap water, and took it into account in the calculation of the density of pure water. We observed that the filtration process, which follows the distillation needed to obtain pure water, doesn't alter the isotopic content.

We performed hydrometer calibrations, and determined the densities of various solids and liquids. Precision pycnometers are calibrated traceable to primary base SI quantities. Air densities were routinely computed, and were used in aerostatic density measurements when necessary.

We continue to work on the conversion to the silicon density standard. Silicon is more stable and more predictable than pure water as a primary density standard. The density of a silicon artefact is also measurable from its dimensions and mass. This work is in collaboration with NRC's Optical Fabrication Laboratory where staff are fabricating the required artifacts by means of a 5 kg single crystal of Si, which has been stored in the mass lab vault for twenty years in anticipation of this opportunity. The project is also dependent on the expertise and active involvement of colleagues in the Dimensional Metrology Program and in the Mechanical Components laboratories. The fabrication of a carousel was completed this year and will be tested in the coming year. A silicon artifact of 100 g nominal mass has also been fabricated and its volume has been determined twice, with an intervening period of eighteen months. The difference in the two volume measurements amounted to 0.25 ppm, while the volume uncertainty is 0.54 ppm.

We participated in three CCM comparisons: 1) CCM.D-K1 on solid Silicon standards; 2) CCM.FF-K4 on 100 mL pycnometers; and 3) CCM.D-K2 on liquid standards.

Medium and High Pressure

A.K. Agarwal, B. Holbrook

This project maintains standards for pressure measurements up to 240 MPa. We recently acquired a deadweight balance capable of operating in either pneumatic or hydraulic mediums up to a pressure of about 100 MPa. This new equipment allows us establish the traceability chain between the primary mercury barometer and the high pressure deadweight balance.

Barometric Pressure

A.K. Agarwal,
B. Holbrook

This project establishes and improves traceability of the derived SI unit of pressure (the pascal). The project provides the primary pressure standard, embodied in a 1.5 m mercury manometer. Dead weight piston gauges provide traceable overlapping standards to higher pressures. In addition, a number of reference and working standards for the capacitive diaphragm type allow accurate propagation of the pascal in the range of 100 Pa to 7 Mpa. We have established a traceability chain between the primary manometer and other dead weight testers for pressure measurements up to 250 MPa. This will enable us to provide traceable high pressure measurement services at accuracies comparable to other NMIs.

Flow

A.K. Agarwal,
B. Holbrook,
C. Jacques

This project began as a collaboration with industry to establish a national facility for the measurement of mass flow in the gaseous state, and to enable the calibration of mass flow measurement and control devices for industry and OGDs. The CLAS certification of industrial laboratories in the mass flow area is one of the project's primary goals.

The Flow Laboratory maintains a piston-prover flow standard to measure gas flow rates up to 50 slm. We have also established its accuracy and uncertainty budget. In addition, the laboratory maintains a transfer standard, embodying laminar flow elements of various flow ranges up to 50 slm. To improve accuracy and reduce measurement uncertainty, the laboratory has begun developing and installing a gravimetric flow measuring system. This research and development project will continue for the next few years.

International Comparisons

- CCM.M-K1 Key Comparison in Mass, 1 kg
  After nine years, the results of this comparison have been published. This comparison illustrates that to attain a high level of accuracy in mass measurements it suffices to know the conditions of temperature, pressure, and humidity and that they are stable during the measurements.

- CCM.M-K2 Key Comparison in Mass, 10 kg, 500 g, 20 g, 2 g, and 100 mg
  NRC did extremely well, except for 10 kg where low-level equipment caused our results to be relatively poor. The situation has since been corrected to our satisfaction by the acquisition of a very good 10 kg mass comparator.

- CCM.M-K3 Key Comparison in Mass, 50 kg
  NRC participated in a pilot study with LNE (France) for the feasibility of the comparison. The results, which are not yet published, are looking very good for NRC.

- CCM.M-K4 Key Comparison in Mass, 1 kg
  Comparisons have to be done on a regular basis. This one repeats CCM.M-K1. The protocol is complete.

- CCM.M-K5 Key Comparison in Mass, 200 mg, 1 g, 50 g, 200 g, and 2 kg
  The measurements are completed.

- CCM.D-K1 Key Comparison in Density – density of silicon
  The standard is a silicon sphere. The preliminary results from Draft A show a very good performance from NRC, although pure water was used as the density standard.

- CCM.D-K2 Key Comparison in Density – liquid density standards
  The protocol is complete and measurements are to be done in 2004.

- CCM.FF-K4 Key Comparison in Liquid Volume, 100 mL
  The measurements of the liquid volume of 100 mL pycnometers are completed.

- CCM.P-K2 Key Comparison in Pressure, 10 kPa to 120 kPa absolute mode and CCM.P-K6 Key Comparison in Pressure, 10 kPa to 120 kPa gauge mode
  The comparisons were carried out between 1998 and 2002 and were piloted by NPL (UK). The participants were BNM/LNE (France), CSIRO (Australia), METAS (Switzerland), NIM (China), NIST (USA), NPL, PTB (Germany) and NRC. The results have been circulated in a Draft A report. The results indicate that all the participants agree with each other within their stated expanded uncertainty for both gauge and absolute mode measurements. Our results in particular are within one standard deviation of the reference value which was computed as the average value of all the participants' results.

- CCM.P-K7 Key Comparison in Pressure, 10 MPa to 100 MPa gauge mode
  This comparison was completed in April 2004 and was piloted by PTB (Germany). The results of the comparison have already been submitted in Draft A report. The participants were BNM/LNE, CENAM (Mexico), NIST, NMIJ (Japan), NPL (UK), NPL (India), PTB and NRC. The results indicate that all the participants agree with each other within their stated expanded uncertainty. We used our newly acquired oil dead-weight tester PG-7202 for this comparison. Our results in particular have been exceptionally good (within 5ppm of the reference value) in spite of large temperature fluctuations in the lab. Results of some other participants vary as much as 30 ppm from the reference value.

- CCM International comparison of magnetic susceptibility and residual magnetism of weights
  NRC is the pilot lab for this comparison. The measurements are nearly completed.

- SIM.7.3 Comparison in Mass, 1 mg to 1 kg
  This comparison is in progress.

- SIM.M.FF-S1 Supplementary comparison in Liquid Volume, 50 mL and 100 mL
  This comparison was piloted by NRC: the results were published in 2003.

- International comparison of hydrometers
  Participating Laboratories: NIST (US), NRC, IMGC (Italy), NIS (Egypt). NRC has made the measurements but the comparison has been halted and is unlikely to resume.

- International comparison of pipettes, 1 L and 10 mL
  Participating Laboratories: NIST (US), NRC, IMGC (Italy), (NIS Egypt). NRC has made the measurements but the comparison has been halted and is unlikely to resume.

Client Services

All projects in the mass standards program involve the routine calibration of artefacts and devices for external clients. A brief, non-exhaustive list of such items calibrated in the past year includes:

- densities of quality reference weights
- reference weights
- dead weight piston gauges and loading weights
- vacuum gauges
- a wide variety of barometric pressure gauges
- volumetric standards, including both reference and production samples
- hydrometers
- magnetic susceptibility of weights and metal samples.

Committees and Offices

International

International Standards Organization

ISO/TC24: Sieves, Sieving and Other Sizing Methods
G.D. Chapman, Member

ISO/SC2: Test sieving
G.D. Chapman, Member

ISO/SC4: Sizing by Methods Other Than Sieving
G.D. Chapman, Member

Comité international des poids et mesures (CIPM)

Consultative Committee for Mass and Related Quantities (CCM)
G.D. Chapman, Member

CCM WGM: Mass Working Group
G.D. Chapman, Member

CCM WGD: Density
C. Jacques, Member

CCM WGHP, WGMP, WGLP: High, Medium and Low Pressures
A.K. Agarwal, Member

CCM: Task Group on Standards Transport
G.D. Chapman, Chairman

SIM and NORAMET

MWG-7: Mass and related quantities
G.D. Chapman, Member
C. Jacques, Member
A.K. Agarwal, Member

MWG-10: Flow and Related Quantities
C. Jacques, Member
A.K. Agarwal, Member

National Association of Testing Authorities (NATA)

G.D. Chapman, Accredited Technical Assessor

South African National Accreditation Service (SANAS)

G.D. Chapman, Accredited Technical Assessor

National

Canadian General Standards Board

Committee on Standard Sieves
G.D. Chapman, Member

Canadian Standards Association International

CSA Technical Committee on the International System of Units (Canadian Metric Practice Guide)
G.D. Chapman, Member

Published Papers and Presentations

This publication list is for the calendar year 2003. A complete list of publications, from 1999, is available on the INMS web site at http://inms-ienm.nrc-cnrc.gc.ca/research_and_development/dimensional_mass_publications_e.html.

Articles in Refereed Journals

Jacques, C., S. Trujillo Juarez, J.M. Maldonado, and V. Bean. "NORAMET intercomparison of volume standards at 50 mL and 100 mL (SIM. M.FF-S1)", Metrologia **40 (1A)**, Technical Supplement, 07001 (2003).

Conference Proceedings

Agarwal, A.A., B. Holbrook. "Traceability Chain for Pressure Measurement from 10kPa –280 MPa at the INMS/NRC", Proceedings of the NCSLI Conference (2003).

Technical Reports

G.D. Chapman. "Least squares comparison of four weights on comparators with weight handlers", NRC Publication 42779 (2003).

G.D. Chapman. Mass Course, NRC Publication 46668 (2004).

Staff Members

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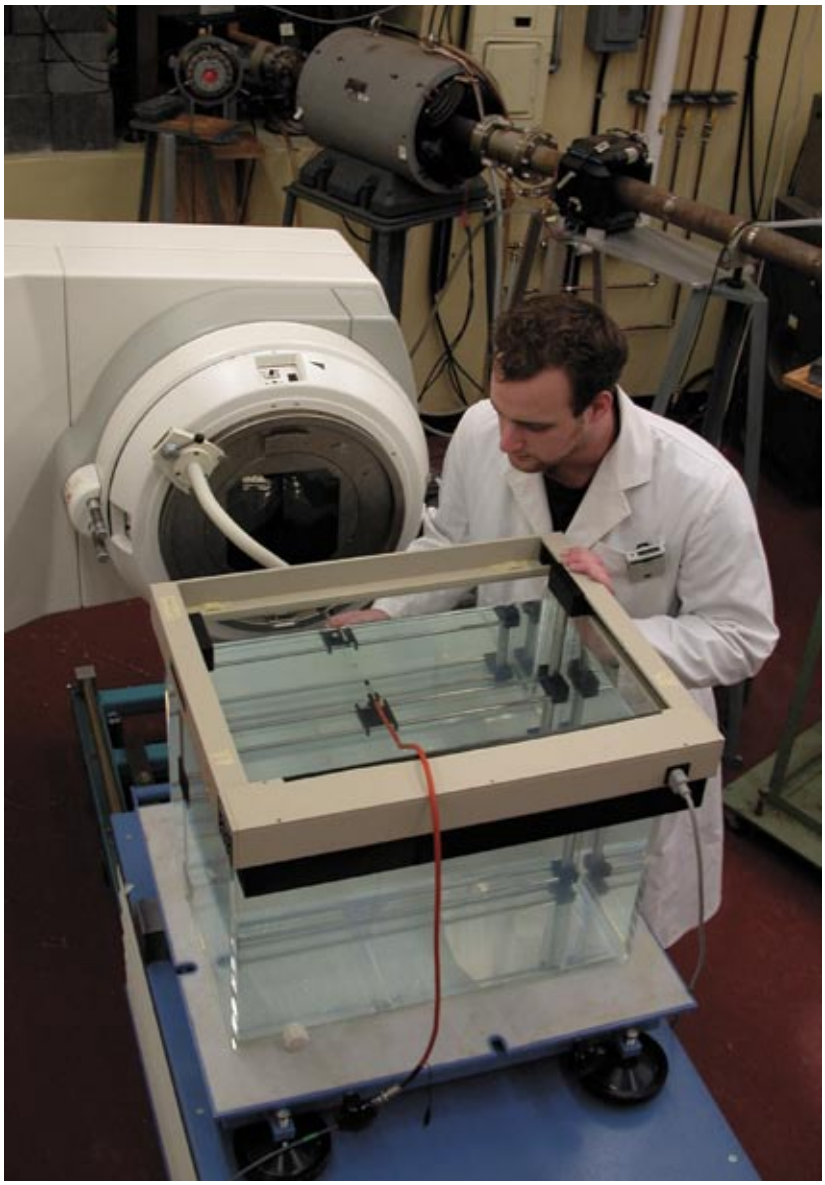

A 3-D scanner is used to measure the absorbed dose distribution in water prior to establishing the dose absolutely using water calorimetry. The detector, a small ionization chamber, is being aligned with the central axis of the radiation field by Michel Lalonde,a co-op student working with the IRS group. The high-energy x-ray beams are generated using an Elekta clinical accelerator, which produces radiation beams similar to those, used for radiation therapy.

The Ionizing Radiation Standards Group (IRS) disseminates, maintains, and develops standards for ionizing radiation. It also conducts research and development related to the measurement of ionizing radiation, and provides services to its client community, such as: calibrations, irradiations, consultations, and training. The client community includes organizations and institutions involved with cancer radiotherapy, radiation protection, industrial irradiation, and international standards.

Major activities include the development of standards and measurement techniques related to: exposure and air kerma in low-energy x-ray and ^{60}Co beams; absorbed dose to water in ^{60}Co beams, as well as photon and electron beams from linear accelerators; neutron fluence and neutron dose equivalent; and absorbed dose to tissue in a ß-ray field. The group is also well known for its work developing and exploiting Monte Carlo calculational techniques for the simulation of electron and photon transport in materials.

Facilities and Services

The IRS group has several facilities for generating ionizing radiation. Radioactive sources provide beams of γ-rays, neutrons, and ß-rays, while electrically powered units generate beams of x-rays and electrons.

^{60}Co units provide beams with variable field sizes and dose rates from close to background to 1 Gy/min. Applications for these irradiators include the calibration of ionization chambers for radiotherapy clinics, irradiation of TLD badges for radiation protection, and the investigation of different detectors such as alanine, film, and ionization chambers. In the spring of 2004, a new source was installed and commissioned in the high-intensity ^{60}Co unit.

Two x-ray tubes, operating with voltages from 10 to 300 keV, generate x-rays used for calibrations, quality assurance testing for radiation protection, and a variety of research projects.

Beta-ray standards are required to protect workers at nuclear power plants from radiation. The ß-ray laboratory has been re-established in recent years and a new irradiator is being commissioned. The irradiator contains three sources – ^{90}Sr, ^{85}Kr and ^{147}Pm – that cover the range of energies found around nuclear power facilities. ß-ray standards are also of interest to the emergency preparedness community, and funding from the CRTI has helped upgrade the laboratory equipment.

The group also has an accelerator laboratory that houses two linear accelerators. A Vickers research accelerator has operated since 1968 and offers a wide range of electron energies (3 to 40 MeV) and beam currents (from a few electrons per second to currents capable of delivering absorbed dose rates of several kGy/min). This wide operating range, combined with the well-characterized energy spectrum and stable output, makes it a unique research tool for radiation dosimetry. In 2002, an Elekta clinical linac was installed. This accelerator is similar to those used for cancer treatment in radiotherapy clinics across Canada, and allows dosimetric problems encountered in the clinic to be investigated directly at NRC.

The complex Monte Carlo calculations required in radiation dosimetry are carried out using a Linux cluster of PCs. This approach allows the system to be independent of any single processor, and supports sequential PC upgrades without introducing any significant downtime. We have developed a queuing system that allows users to prioritise computations by selecting the speed of the processors used for a given calculation; this system is now distributed with the BEAMnrc Monte Carlo Code. The computing network also provides support for courses in Monte Carlo techniques, which cover the two codes, EGSnrc and BEAMnrc, developed at NRC.

The group provides calibration services for devices used to measure x-rays, γ-rays, neutrons and ß-rays. Usually, the client sends a device to NRC for calibration, but in some cases IRS personnel will visit the client's facility and calibrate their radiation field.

Our radiation facilities also provide fee-for-service irradiations. In some cases customers come with devices to irradiate, and they participate in the measurements. In other cases, devices or materials are sent to NRC, irradiated, and returned to the user. A more detailed description of the calibration and irradiation services provided by the Group are listed on the INMS web site at http://inms-ienm.nrc-cnrc.gc.ca/calserv/ionizing_radiation_e.html.

Radiation Standards for ^{60}Co and ^{137}Cs Air Kerma

J. McCaffrey, B. Downton, H. Shen, S. Walker, D. Marchington, D. Rogers, F. Farahvash, C. Ross

This project seeks to disseminate, maintain, and develop standards for air kerma for ^{60}Co and ^{137}Cs γ-ray beams. Until recently, these standards were the basis for Canadian radiation therapy dosimetry. Although still used by some, most clinics now use IRS standards for absorbed dose.

Radiation protection is now one of the major areas for the application of γ-ray air-kerma standards, and IRS provides a quality assurance program for all personnel monitoring services in Canada. The nuclear power industry is one of the ^{60}Co quality assurance program's major clients.

Research activities have focused on the conversion from extrapolated values of the wall correction factor, k_{wall}, to Monte Carlo based calculations of this factor. Experimental work with a specially designed graphite spherical ion chamber supports these calculations. A re-examination of all the correction factors resulted in a change of 0.59% in the exposure and air-kerma primary standard at INMS in 2003.

Because the half-life of ^{60}Co is about 5 years, we must install a new source in our high intensity irradiator every 4 to 5 years. We purchased, installed, and successfully commissioned a new source in March 2004.

Radiation Standards for kV X-ray Air Kerma

J. McCaffrey, H. Shen, S. Walker, D. Marchington, F. Farahvash, P. Saull

This project seeks to disseminate, maintain, and develop standards for air-kerma for x-ray beams with energies from 10 to 300 kV. X-ray air-kerma standards are required for Canadian radiation therapy dosimetry for skin and breast treatments. We have made a significant effort to re-measure and verify most of the half-value layers used for specifying the large number of x-ray beams maintained by IRS. The x-ray laboratory is in the process of a major renovation, which will update and replace aging support structures and equipment.

As well as dosimetry, the x-ray facility provides standards for quality assurance testing at x-ray energies. All workers in Canada exposed to radiation have individual records of their cumulative radiation dose maintained at the National Dose Registry of Health Canada. This registry includes workers in dentistry, radiation therapy, chiropractic and veterinary medicine, as well as laboratory workers, industrial radiographers, etc. The smaller groups are monitored through services offered by Health Canada and other secondary QA labs, all of which are traceable to the radiation standards maintained at IRS.

Research activities using the x-ray standards included work by graduate student Steve Davis, under the supervision of Carl Ross, with funding provided by Ontario Power Generation. The work investigated the energy response of a new generation of thermoluminescent devices (TLDs), used for dose monitoring.

Radiation Standards for Absorbed Dose to Water in Photon and Electron Beams

M. McEwen, C. Ross, N. Klassen, D. Marchington, L. Heistek

Accurate measurement of the absorbed dose to water in radiation beams is a critical component of cancer radiotherapy. The IRS group has devoted considerable effort to the development of water calorimetry as a standard for the absorbed dose to water. This work led to the development of our ^{60}Co absorbed dose calibration service, which is used by most Canadian radiation therapy centres.

During the past year, we have used our water calorimeter to establish the absorbed dose in the x-ray fields provided by our new Elekta accelerator. An intercomparison is underway to compare our work with that of several European laboratories. We intend to offer Canadian users calibration services in high-energy x-ray beams, a service similar to those offered by several European standards laboratories.

Radiation-induced chemical reactions can affect the response of the calorimeter by several percent. In the case of pure water or certain well-defined aqueous systems, the radiation chemistry can be accurately modeled. However, unknown impurities can also affect the response, and it is therefore important to control the water purity. We constructed a vessel in which the only material contacting the water is glass. Initial measurements indicate that this system's response remains stable over long periods of time.

Tissue in a ß-Ray Field

P Saull, D. Marchington, S. Walker, D. Rogers, C. Ross,

This project seeks to disseminate, maintain, and develop standards for absorbed dose to tissue in a ß-ray field based on extrapolation chamber measurements.

Re-activated in 2002 using two old SrY-90 sources, the project has recently benefited from an infusion of CRTI funding, which facilitated the acquisition of a new ß-ray irradiator. The irradiator includes two new source types. These sources, Kr-85 and Pm-147, in addition to a SrY-90 source, extend the range of study to energies lower than previously available.

The ongoing commissioning of a new system for beta standards has involved the full integration of the extrapolation chamber hardware with that of the irradiator, and the development of new computer code running under Linux to control the entire irradiation and data acquisition process. We will next determine the correction factors required to establish these standards, using primarily measured data supplemented by the Monte Carlo results using the BEAMnrc code.

The new system will be put to the test in early 2005 in a EUROMET intercomparison titled "Supplementary comparison of absorbed dose rate in tissue

for beta radiation" (EUROMET project No. 739, BIPM KCDB: EUROMET. R(I)-S2), which involves the participation of 6 European countries, as well as Canada and the US. The results will be sent to the BIPM for inclusion in the Key Comparison Database.

The main application of ß-ray standards is in the area of radiation protection. With the new system, the IRS group will provide a quality assurance program for all providers of personnel dosimetry services in Canada, as mandated by the CNSC.

Neutron Fluence and Ambient Dose Equivalent Studies

P. Saull, L. Heistek

The measurement of the dose-equivalent due to neutrons is important for cancer clinics and the nuclear power industry. The IRS group offers a neutron survey meter calibration service based on the known emission rates of its Am-Be neutron sources, calibrated using the manganese bath technique. We apply corrections for air-scatter, room-scatter, and the finite size of the detector (geometry correction), in order to derive an expected fluence, which is subsequently converted to an ambient absorbed dose estimate using the known Am-Be source spectrum and ICRU-tabulated fluence-to-ambient-dose conversion coefficients. We employed the MCNP5 Monte Carlo code to study the accuracy of the geometry correction factor, the origin of the observed variation in detector directional response, and the effect of the room- and air-scattered neutrons on the fluence, particularly regarding their shift to lower energies.

The Am-Be neutron sources are also used to test the behaviour of personal dosimeters. The IRS group is developing a quality assurance program for all providers of personnel monitoring services in Canada. The goal is to achieve a 2-3% uncertainty (1 sigma) in the absorbed dose delivered to a personnel dosimeter over the range 0.01 to 1 mSv.

Computational Radiation Physics

I. Kawrakow, D. Rogers, L. Buckley, B. Walters, E. Mainegra-Hing, F. Farahvash, M. Proulx

The goal of this project is to develop, refine, and apply the Monte Carlo technique to problems related to ionizing radiation standards and radiation therapy. Codes developed at NRC include EGSnrc and BEAMnrc, both widely used within the medical physics community.

We used the EGSnrc code to re-examine the various correction factors needed to establish air kerma standards. This work showed that the insulator in the Canadian cavity chamber standard affects its response by about 0.5%, and we adjusted the Canadian air kerma standard accordingly. As well, we calculated wall correction factors for various plane-parallel ionization chambers using EGSnrc, and found better agreement with experimental data compared to earlier EGS4 simulations.

We implemented a correlated sampling (CS) algorithm into the CAVRZnrc (an EGSnrc user code). The CS algorithm improves the efficiency of in-phantom ion

chamber correction factor calculations. The new algorithm is currently being used to calculate correction factors for a variety of ionization chambers and beam qualities.

The electron impact ionization process was implemented in EGSnrc using cross sections, based on a new theory, that are in very good agreement with experimental data. This will permit a more accurate simulation of the spectra of kilo-voltage x-ray tubes. Also, we developed a new approach for the treatment of Compton scattering, in collaboration with J. Williamson and C. Costescu of the Washington University Medical School in St. Louis, MO. The theory, based on a modification of the impulse approximation, improves the agreement with experimental data for Compton scattering with the K-shell of high-Z materials.

The EGSnrc run-time environment was completely re-worked to permit the use of the package on all major operating systems (Linux/Unix/Windows/Mac OSX). We expect this work to increase the use of EGSnrc and therefore the Group's impact on the medical physics community.

We implemented a new variance reduction technique called DBS in the BEAM package. DBS dramatically increases the efficiency of photon beam treatment head simulations with BEAM (up to a factor of 8 compared to the most efficient simulation technique previously available), thus permitting more detailed studies of the output of medical linear accelerators. The DBS technique, with a series of additional improvements, was implemented in the VMC++ package and named DRS. VMC++ with DRS permits extremely fast photon beam treatment head simulations, so that the simulation time in the treatment head is short compared to the simulation time of particle transport through a patient geometry. This development will be potentially very useful for the implementation of photon Monte Carlo in commercial treatment planning systems.

We successfully completed the implementation of VMC++ into the treatment planning software of MDS Nordion (now Nucletron) for electron beam dose calculations. The Ottawa General Hospital has become the first site worldwide to routinely use Monte Carlo dose calculations for electron beam radiation treatment planning.

We published a new theoretical derivation of the influence of Monte Carlo statistical uncertainties on treatment plan evaluation. The theory demonstrates that statistical uncertainties lead to a systematic error in a cost function that should be taken into account when using Monte Carlo calculated dose distributions for treatment plan optimization.

Experimental Radiation Physics

M. McEwen, C. Ross, N. Klassen, M. Kosaki, D. Marchington, G. Zeng

This project encompasses several short-term sub-projects that address problems of interest to medical physics or radiation physics. The projects often involve graduate students, post-doctoral fellows, or other external collaborators. The work, which frequently involves the linear accelerators, attempts to exploit their unique capabilities.

In 2002, a project was started to investigate alanine dosimetry primarily for radiotherapy applications, but also for radiation processing. The principal objectives were to:

- obtain the lowest uncertainty possible to determine what factors affect the measurement of the alanine response; and
- determine the energy dependence of alanine in high-energy photon and electron beams. Alanine is usually calibrated in a ^{60}Co field; it is then assumed that the same calibration factor can be used in all high-energy beams, be they photon or electron.

A measurement protocol was developed that yields an uncertainty of about 0.3% for doses down to 10 Gy, as low as any achieved by other laboratories. Measurements were made in ^{60}Co and beams from the NRC Elekta linear accelerator (6-25 MV x-rays, 8-22 MeV electrons), and the alanine energy response was determined by comparison with the dose derived using a calibrated ionization chamber (traceable to the primary standard water calorimeter). We found a small change in response, for both photon and electron beams, in going from ^{60}Co to linac energies – 1% for photon beams and approximately 2.5% for electron beams. Monte Carlo studies with EGSnrc suggest this energy dependence is due to the energy dependence of the alanine stopping power.

Standards for Low Energy Electron Beams

M. McEwen and collaborators at NPL and Riso National Laboratory

The use of low energy electron beams (< 250 keV) for radiation processing has increased in recent years. These beams are well suited for surface treatments, such as sterilizing packaging materials or curing inks. The significant challenge facing dosimetry at these low energies is the very short electron range– typically less than 100 μm in unit density material. At present there are no dose standards for such low electron energies, and the current practice is to calibrate film dosimeters at 10 MeV and assume that the calibration is energy independent. The aim of this project, in collaboration with Riso National Laboratory in Denmark, and the National Physical Laboratory in the UK, is to develop a primary standard calorimeter system to calibrate film dosimeters at energies around 100 keV.

We developed a system based on a totally absorbing graphite calorimeter. The calorimeter measures the total energy in the beam, and the dose is derived using film measurements and Monte Carlo calculations. Initial testing showed that heat transferred from the air above the calorimeter had a significant effect on the temperature rise (and therefore dose) measured. A simple thermal model based on a proprietary finite element software package gave good agreement (at the 5% level) with experimental estimations of the thermal effect. Initial results for the calibration of two types of dosimeter film used for this type of application show no significant energy dependence from 10 MeV to 100 keV within the measurement uncertainty (currently ~ 10-15%). We estimate that with refinement of the model it should be possible to obtain a film calibration at 100 keV with an overall uncertainty of 8% (1 Σ).

Committees and Offices

International

Institute of Physicists and Engineers in Medicine

Working Party on electron dosimetry
M.R. McEwen

Comite international des poids et mesures

Comite Consultatif des Rayonnements Ionisants: Section I, Rayons X et gamma, electrons
D.W.O. Rogers

American Association of Physicists in Medicine

ADCL subcommittee
M.R. McEwen

TG-71 Electron beam dosimetry
D.W.O. Rogers

Journal Business Management Committee
D.W.O. Rogers

Research Committee
D.W.O. Rogers

Editorial Board of Microscopy Research and Techniques
J.P. McCaffrey

IAEA (International Atomic Energy Agency)

Dosimetry Symposium Advisory Committee
M.R. McEwen

National

Canadian Nuclear Safety Commission

Working group on external dosimetry (personal radiation monitoring)
P.R.B. Saull

Radiological/Nuclear Lab Cluster of CRTI

C.K. Ross, NRC representative
P.R.B. Saull, NRC representative

Carleton University

Medical Physics Organized Research Unit
M.R. McEwen, Member of Executive

Standards Council of Canada

ISOTC85/SC2: Radiation Protection
C.K. Ross

IEC SC62C: Equipment for Radiotherapy, Nuclear Medicine and Radiation Dosimetry
C.K. Ross

Adjunct Professorships

D.W.O. Rogers, Carleton University, Department of Physics

Editorships

Medical Physics Journal
D.W.O. Rogers, Associate Editor

Published Papers and Presentations

This publication list is for the calendar year 2003. A complete list of publications, from 1999, is available on the INMS web site at http://inms-ienm.nrc-cnrc.gc.ca/research_and_development/ionizing_radiation_std_publications_e.html.

Articles in Refereed Journals

Buckley L.A., I. Kawrakow and **D.W.O. Rogers.** "An EGSnrc investigation of cavity theory for ion chambers measuring air kerma", Med. Phys. **30,** 1211-1218 (2003).

Davis, S.D., C.K. Ross, P.N. Mobit, **L. Van der Zwan,** W.J. Chase and **K.R. Shortt.** "The response of LiF TLDs to photon beams in the energy range from 30 kV to ^{60}Co γ-rays", Radiat. Prot. Dosim. **106,** 33-44 (2003).

R. Doucet, M. Olivares, F. DeBlois, E. B. Podgorsak, **Kawrakow, I.** and J. Seuntjens. "Comparison of measured and Monte Carlo calculated dose distributions in inhomogeneous phantoms in clinical electron beams", Phys. Med. Biol **48,** 2339-2354 (2003).

F. Sánchez-Doblado, P. Andreo, R. Capote, A. Leal, M. Perucha, R. Arráns, L. Núnez, **Mainegra-Hing, E.**, J.I. Lagares and E. Carrasco. "Ionization chamber dosimetry of small photon fields: a Monte carlo study on stopping power ratios for radiosurgery and IMRT beams", Phys. Med. Biol. **48,** 2081-2099 (2003).

Mainegra-Hing, E., I. Kawrakow and **D.W.O. Rogers.** "Calculations for plane-parallel ion chambers in 60Co beams using the EGSnrc Monte Carlo code", Med. Phys. **30,** 179-189 (2003).

McEwen, M.R. and A.R. DuSautoy. "Characterization of the water-equivalent material WTe for use in electron beam dosimetry", Phys. Med. Biol. **48,** 1885-1893 (2003).

IPEM Working Party: D.I. Thwaites, A.R. DuSautoy, T. Jordan, **McEwen, M.R.**, A. Nisbet, A.E. Nahum and W.G. Pitchford. "The IPEM code of practice for electron dosimetry for radiotherapy beams of initial energy from 4 to 25 MeV based on an absorbed dose to water calibration", Phys. Med. Biol **48,** 2929-2970 (2003).

P.H.G. Sharpe, **McEwen, M.R.,** J.P. Sephto and D.R. Shipley. "Use of a range scaling method to determine alanine/water stopping power ratios", Rad. Phys. Chem. **68,** 1017-1022 (2003).

N.I. Kalach and **Rogers, D.W.O.** "When is an accelerator photon beam clinic-like for reference dosimetry purposes", Med. Phys. **30,** 1546-1555 (2003).

Rogers, D.W.O. and **I. Kawrakow.** "Monte Carlo calculated correction factors for primary standards of air-kerma", Med. Phys. **30,** 521-543 (2003).

Saull, P.R.B. and the ZEUS High Energy Physics Collaboration. "Measurement of Deeply Virtual Compton Scattering at HERA", Physics Letters B, **573,** 46-62 (2003).

Refereed Conference Proceedings

S.R. Sheng, S.P. McAlister, **McCaffrey, J.P.** and S. Kovacic. "Ionizing Radiation Effects in SiGe Heterojunction Bipolar Transistors", Eleventh Canadian Semiconductor Technology Conference, Ottawa (Aug. 2003).

S.R. Sheng, S.P. McAlister, **McCaffrey, J.P.** and S. Kovacic. "Degradation and recovery of SiGe HBTs following radiation and hot-carrier stressing", International Semiconductor Device Research Symposium, Washington, DC. (Dec. 2003).

McEwen, M.R. and S. Duane. "A portable graphite calorimeter for measuring absorbed dose in the radiotherapy clinic", IAEA-CN-96/9P, Proceedings of an International Symposium on Standards and Codes of Practice in Medical Radiation Dosimetry, IAEA, Vienna 2002, 115-121 (2003).

K.N. Govinda Rajan, S. Vandanaa, M. Vijayam, J.B. Shigwan, **McEwen, M.R.,** and S. Duane. "Testing of N_K and $N_{D,w}$ based IAEA Codes of Practice for clinical photon beams", IAEA-CN-96/57P, Proceedings of an International Symposium on Standards and Codes of Practice in Medical Radiation Dosimetry, IAEA, Vienna 2002, 467-474 (2003).

McEwen, M.R., S. Duane and I.A. Stoker. "A comparison of graphite standard calorimeters in megavoltage photon and electron beams, IAEA-CN-96/4", in Proceedings of an International Symposium on Standards and Codes of Practice in Medical Radiation Dosimetry, IAEA, Vienna 2002, 67-73 (2003).

McEwen, M.R., S. Duane and R.A.S. Thomas. "Absorbed dose calibration factors for parallel-plate chambers in high energy photon beams, IAEA-CN-96/28", in Proceedings of an International Symposium on Standards and Codes of Practice in Medical Radiation Dosimetry, IAEA, Vienna 2002, IAEA, 35-341 (2003).

R. Thomas, S. Duane, **McEwen, M.R.** and K.E. Rosser. "The role of the National Physical Laboratory in monitoring and improving dosimetry in UK radiotherapy, IAEA-CN-96/80P", in Proceedings of an International Symposium on Standards and Codes of Practice in Medical Radiation Dosimetry, IAEA, Vienna 2002, 201-208 (2003).

Other Publications

J. Helt-Hansen, M. Millar, **McEwen, M.R.,** P.H.G. Sharpe, and S.C. Duane. "Development of a Primary Standard Calorimeter for Low Energy Electron Beams", Proceedings of a Workshop on Recent Advances in Absorbed Dose Standards, ARPANSA, Melbourne (2003), www.arpansa.gov.au/absdos/proc.htm.

McEwen, M.R. and **C. Ross.** "Performance of an "Off-The-Shelf" Clinical Linac for Radiation Standards Dosimetry", Proceedings of a Workshop on Recent Advances in Absorbed Dose Standards, ARPANSA, Melbourne (2003), www.arpansa.gov.au/absdos/proc.htm.

Invited Oral Presentations

Rogers, D.W.O. "2003 Monte Carlo Applications to Radiation Standards", World Congress on Medical Physics and Biomedical Engineering, Sydney (2003).

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Kawrakow, I. "Fast Treatment Head Simulations Using Directional Bremsstrahlung Splitting in VMC++", World Congress on Medical Physics and Biomedical Engineering, Sydney (2003).

Klassen, N.V. "The role of radiation chemistry in establishing water calorimetry as the world's standard for absorbed-dose-to-water", 23rd Miller Conference on Radiation Chemistry, Bialowieza, Poland (2003).

McEwen, M.R. "Development of a Primary Standard Calorimeter for Low Energy Electron Beams", Workshop on Recent Advances in Absorbed Dose Standards, Melbourne (2003).

McEwen, M.R. "Performance of an "Off-The-Shelf" Clinical Linac for Radiation Standards Dosimetry", Workshop on Recent Advances in Absorbed Dose Standards, Melbourne (2003).

McEwen, M.R. "Pre-Irradiation of Ionization Chambers Used In X-Ray And Gamma-ray Calibrations", World Congress on Medical Physics and Biomedical Engineering, Sydney (2003).

Rogers, D.W.O. "Changes in Co-60 Air-Kerma Standards: The Rationale for Change and the Impact On Clinical Practice", 45th Annual AAPM meeting, San Diego, CA (2003).

Zeng, G. "Experimental and Monte Carlo Investigations of the Energy Dependence of Alanine/EPR Dosimetry System for Clinical Electron and Photon Beams", 45th Annual AAPM meeting, San Diego, CA (2003).

Technical Reports

Davis, S.D. "High sensitivity lithium fluoride as a detector for environmental dosimetry", (MSc. thesis, McGill University), PIRS-0832 (2003).

Rogers, D.W.O. and **J. McCaffrey.** "The 2003 revision of the NRC standard for air-kerma in a ^{60}Co beam", NRC Report PIRS-876 (2003).

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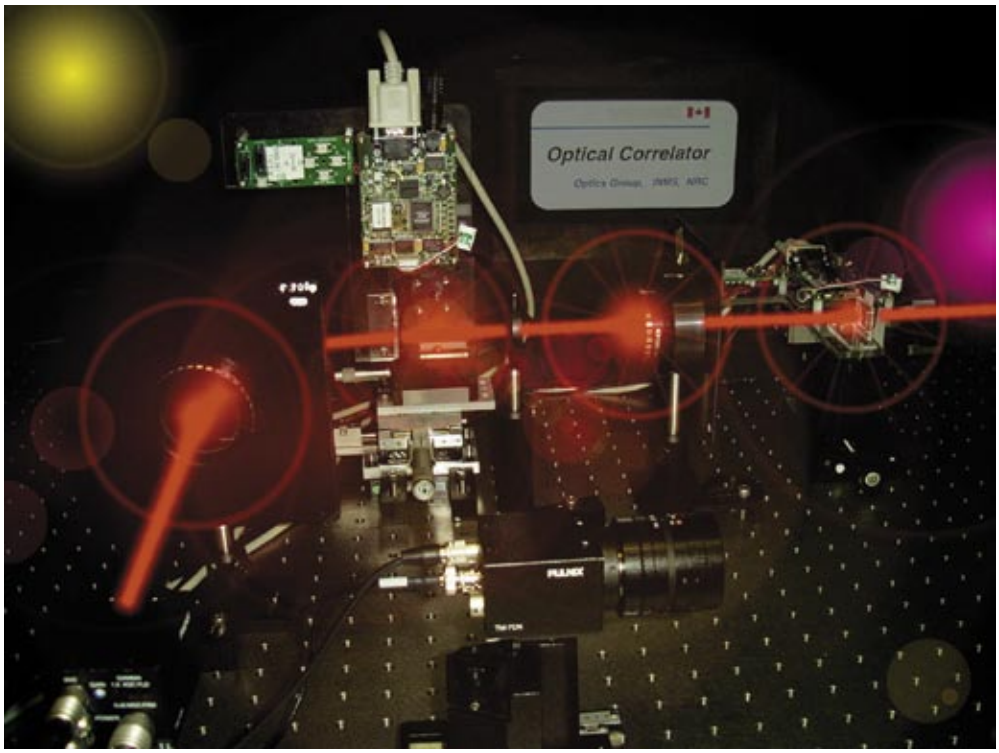

The hybrid optical correlator developed by Optics Group is a hybrid information-processing device consisting of Spatial Light Modulators (SLM), CCD cameras, a modified 4f optical correlator, a laser source and a computer. It can perform almost all the known linear and some non-linear operations including convolution and correlation. The processing speed is instant, i.e. the speed of light. Computer programmable SLMs make the input and filter real-time updatable. It is also able to perform both multiple-gray-level intensity and phase correlation operations.

The Optics Group at NRC's Institute for National Measurement Standards conducts research and development in focused areas of optical science and technology, establishing competencies that enhance the competitiveness of Canadian industry. Research activities focus on applications in manufacturing technologies, measurement standards, information, and telecommunications technologies.

Services are offered in the areas of custom optical design, fabrication and testing of optical components and systems, calibration of aerial survey cameras and sensitometers, and scientific glassblowing.

Optical System Design - Advanced Imaging Systems

C.P. Grover, Y. Boiko, X. Cai, S. Chang, C. Flueraru, E. Murdock

The Group continues to develop the optical system design expertise necessary for providing support to industry in an effort to give our clients a competitive edge over their competition. Our work is heavily influenced by external demands. We continue to adjust our on-going major projects in anticipation of future requirements. This work not only keeps us abreast of the latest technology, but allows us to develop new techniques. The Group also provides optical system design to other NRC institutes and clients in the private sector.

Performance enhanced imaging systems

We have designed an optical imaging system with high tolerance to focus error, and explored the potential applications of the system for object tracking. The wavefront coding technique can enhance the performance of imaging systems. With a specially designed phase plate in the pupil plane of an imaging system, the energy re-distribution in the vicinity of the focal plane can be fully controlled, thereby extending its depth of focus to a designated value.

We have studied different types of phase pupil functions for increasing focal depth. We have found that we can apply a simple quartic aspheric plate with optimum parameters efficiently and flexibly to an existing imaging system used for small object tracking. The system built with the phase plate can more than double the focal depth of a conventional imaging system while retaining the same resolution. We have also developed the techniques for fabricating and testing the aspherical phase plates used in this application.

Design of zoom null lenses for testing optical surfaces

This study explores the design of new zoom null lenses for testing spherical and aspherical surfaces. The study sought an efficient, versatile, and compact testing system for a variety of optical surfaces. Currently, the testing of aspherical surfaces relies on a null lens designed specifically for the test surface. This reliance is very inconvenient for batch testing surfaces with only slight parameter changes. The design of a zoom null lens capable of batch testing lenses will improve and simplify the system while providing greater optimization of lens design. The design of zoom null lenses is also helpful in setting up a compact system for testing large surfaces with large radii of curvature. We have studied a variety of zoom lenses for this purpose, and configured a compact test system in a small space for a variety of large surfaces. Based on the results of a computer simulation, the anticipated performance of this system is satisfactory. The system is currently being fabricated, and will soon undergo further verification.

Optical Metrology

C.P. Grover, Y. Boiko, X. Cai, S. Chang, C. Flueraru, E. Murdock

Photonics metrology - Optical fibre and fibre-optic components measurements

Optical fibre telecommunication networks have required regulation through the specification standards of international standardization organizations, such as the International Telecommunications Union (ITU), the International Electrotechnical Commission (IEC), and regional metrology networks. The complete characterization of fiber- and waveguide components is increasingly important in wavelength division multiplexing (WDM), and dense wavelength division multiplexing (DWDM) based fiber-optic telecommunication systems, because of their requirements for high speed and channel capacity.

This project focuses on measurement standards for the polarization mode dispersion characterization within optical fiber and fiber-optic components. A computer controlled optical setup with a rotating quarter wave plate has been developed for high-accuracy retardance measurements, and the determination of Stokes vector components. We have implemented the Stokes parameter evaluation technique as the reference method for polarization mode dispersion measurement. Our current focus is to implement the State of Polarization and the Fixed Analyzer methods as the alternatives for polarization mode dispersion measurements. We provide wavelength dependent polarization mode dispersion and polarization dependent loss measurements. We also undertake custom measurements of other parameters upon request.

Phase amplification interferometry for nanometrology

Phase difference amplification is a useful technique for nanometrology. It is achieved by carrying out interference between n^{th} and $-n^{th}$ diffracted orders produced by nonlinearly recorded interferograms of the test object. We developed a near-real-time ultra-sensitive interferometer based on the Liquid Crystal Spatial Light Modulator. We used a computer to carry out phase difference amplification for interference fringes obtained by white light interferometry. These techniques will ultimately result in the improvement of quality assurance procedures for the optical manufacturing industry.

Using a sandwich-beam divider, we developed a variable shearing Michelson interferometer useful for lens aberration measurements with large apertures. This interferometer was also applied to the differential interference contrast method, which may be useful for nanometrology in bio-imaging.

Optical phase differentiation technique and its applications

We developed the phase differentiation technique for the indirect visualization of the phase shift of light passing through an object, whereby the derivative of the phase shift is converted into optical intensity. The phase shift can be calculated by integrating the measured intensity using a charge-coupled device (CCD) camera. We are investigating several novel techniques to include absorbent transparent

objects in the area of application. For the visualization of phase objects using a differentiation filter, the phase variation is converted to intensity variations by differentiation, and the image is then generated by integration. This technique has been applied to optical testing, material diagnostics, and microscopic analysis of living cells.

Electro-Optical and Nonlinear Optical Materials and Devices

C.P. Grover, Y. Boiko, X. Cai, S. Chang, C. Flueraru, E. Murdock

Electro-optical and nonlinear optical devices based on organic materials

This project is focused on technological processes and measurement standards needed in the design and fabrication of organic all-optical and electro-optical devices. Market analysis for integrated optics predicts a strong demand for the development of optical polymers as basic materials for integrated optical circuits. Different reviews have extensively presented and discussed the advantages of optical polymers. In our research, we have used two classes of optical polymers: guest-host and side-chain systems. We have developed methods to measure the electro-optical coefficients and second order susceptibility of thin polymeric layers and waveguide. A procedure for second-order susceptibility measurement relative to the z-cut quartz has been reported. This experimental set-up can be used for in-plane mapping of nonlinear optical properties of thin films.

We have developed new design structures based on multi-layer waveguide, with a focus on improved efficiency. A simple technological approach for improving the nonlinear optical interaction within inverted waveguide through maximization of the overlap integral has been reported. We continue to evaluate new materials and different technological processes.

Organic – inorganic materials for holographic storage and photonic waveguide application

Organic-inorganic materials have been established in photonics because of their ability to combine the advantageous features of both organic and inorganic components. This combination produces new and better performing materials. In the organic-inorganic system, one part (normally the host material) accommodates its counterpart (a guest material) with the desired functionality. Examples of functionality include nonlinear optical response and holographic recording properties. The basic advantageous properties of organic matrixes are: the ability to process at room temperature, an adjustable refractive index, and optical quality suitable for optical waveguiding. On the other hand, the major advantages of inorganic matrixes are: thermal stability, thermal conductivity, low shrinkage, and low thermal expansion of the resulting materials. The current project aims to develop novel organic-inorganic materials for photonic applications, particularly holographic recording in single and two-photon regimes, as well as fabrication of optical waveguide devices. We have demonstrated the fabrication of electro-optical films made of reactive mesogen, and the verification of second harmonic generation properties.

Information Processing and Computer Vision for Pattern Recognition Applications

C.P. Grover, Y. Boiko, X. Cai, S. Chang, C. Flueraru, E. Murdock

The Group has been working on Optical Coherence Tomography (OCT) for three years. This is a relatively new imaging technology that provides morphological and structural information of 3D samples. OCT's uniqueness is a consequence of its unsurpassed imaging resolution of 10-30 μm, compared to clinical resolutions of 1mm for MRI and 200 μm for ultrasound. We developed a compact full-field OCT system by incorporating a modified Michelson interferometer and a CCD camera. We are employing simple and practical methods to remove the interference fringes in the extracted cross-sectional images, and enhance the dynamical imaging range. Specially designed algorithms were introduced to remove the inter-layer-modulation effect. This system was successfully used to extract text from a multiple-layer info-chip, and images from an onion epithelium.

Secure information retrieval technologies are critical for status identification, particularly in battlefields, where friend/enemy discrimination is vital. The materials or devices used in these technologies should be as simple as possible, but also hard to find and difficult to counterfeit. Moreover, if the coding information is totally position-invariant (i.e., neither sequence nor pixel based), it will greatly simplify the retrieval system. To challenge these barriers, we have developed methods for status identification using quantum-dots (QDs) based info-inks, which contain fluorescent substances with well-defined emission spectra. The information is encoded by using luminescent semiconductor nanocrystals (or QDs), mixed with a transparent solution called info-ink. When an exciting light beam shines on the info-ink, its emitting spectral features (i.e., wavelength and intensity) reveal the encoded information. The info-ink could be applied on any kind of surface, e.g., document covers, helmet tops, or even fingernails. The retrieval device consists of an exciting light source, a mini-spectrometer, and a data processing unit. However, the brightness found in battlefields and other sunlit areas can overwhelm most reflected fluorescent signals. To overcome this shortcoming, the quantum dots are engineered to produce fluorescence at wavelengths corresponding to the absorption lines of the solar spectrum, more commonly known as Fraunhofer lines.

In the wake of the September 11 terrorist attack, the development of new biometric technologies has become a high priority. Of all the various personal identification technologies, face recognition has the highest social acceptability: eventually, 3D face recognition will replace the commonly used 2D technologies. To develop new 3D face recognition technology, we have developed the phase Fourier transform (PFT) of a 3D facial image, which can map a 3D face into a nebula-like signature. After the PFT, the 3D face becomes invariant to shift and rotation (in- or out-of-plane) with an uncertainty in the range of ±10°. The image is also scale invariant to a certain degree (scaled down to 70%).

Calibration Services

C.P. Grover, X, Cai, E. Murdock

The Optics Group calibrates optical instruments, such as aerial survey cameras and sensitometers, for use in private industry, government departments, and universities.

Calibration of aerial survey cameras

The calibration of aerial survey cameras is a unique service provided by the Optics Group for Canada's mapping industry. Using a photographic technique to ensure correspondence to actual use, we calibrate aerial survey cameras by measuring their focal length, distortion, positioning of fiducial marks, and alignment errors, with an uncertainty of a few micrometres. The optical image quality is determined via Optical Transfer Function (OTF) measurements. Twenty cameras were calibrated during the year.

Calibration and verification of sensitometers

The Optics Group provides a calibration and verification service for sensitometers, and maintains the necessary standards. Calibration services are available for density step tablets and other similar photographic recordings. Industries and government organizations engaged in aerial photographic work use sensitometers based on an NRC design. The Group also advises on the performance of commercially available sensitometers, and provides film density measurement, including microdensitometric scans on moderately sized samples up to approximately 10cm. Five sensitometers were calibrated during the year.

Fabrication and Testing of Precision Optical Components and Thin Films

C.P. Grover, S. Boisclair, W. Boland, M. Burill, G. Boyd

The Optics Group provides a unique optical fabrication service, which involves thin films coatings in prototype optical components and systems that are not readily available from the private commercial sector. The Group's expertise in thin films includes fabrication of optical and non-optical coatings in single and multi-layer stacks, using metals and dielectric materials.

We signed a collaborative agreement with Lightmachinery Inc, which increased our revenue throughout 2003-2004. We also completed other notable projects in the reporting year, for the following clients: Linnenbruegger, Luxonics, University of Waterloo, Lockheed Martin, Exfo, Claire Lasers Corp., Coherent-AMT and NRC institutes (INMS, SIMS, IMS, ICPET, IIT, IAR).

The Group's handling of a wide range of optical components brought about the development of several special in-house fabrication techniques, including the use of aspheric phase plates in optical systems for object tracking applications. These techniques are then sometimes passed on to industry as part of the Group's consultation service in optical fabrication. The fabrication, testing, and thin films services provided to internal NRC clients would cost approximately $600,000 if purchased externally.

Due to the large number of ongoing projects, we list some examples in this short list:

- broadband AR-coatings on Ge optimized for 8-12 mm
- replacement windows to meet original manufacturer specifications for the IAR-Pratt & Whitney Canada test facility
- a process for depositing durable scatter-free thin films for deep UV spectral regions on MGF2 substrate
- quartz etalon plates and Ge windows
- technology for generating and polishing aspheric surfaces with nanometric precisions (INMS)
- a replacement filter for the INMS Glossmeter
- silicon kilogram standards for INMS.

Scientific Glassblowing Services

P. L'Abbé, M.Vandenhoff

The Optics Group provides a complete custom scientific glassblowing service to NRC Institutes and external clients. These projects involve consultation on the design, custom fabrication, modification, and repair of scientific apparatus, in-situ or in our laboratory. In addition, we also offer a reference and referral service for the acquisition of equipment, supplies, and custom specialized training in glassblowing technology.

INMS, the Institute for Chemical Process and Environmental Technology, and the Steacie Institute for Molecular Sciences were our principal clients. Other NRC institutes, namely IBS, IAR, ARC, IIT, IRC, and ASPM, also received our services. Our external clients for glassblowing were Laurentian University, Natural Resources Canada, and Catherine Richards (Artist in Residence). We provided an equivalent of $150,000 worth of services to the Institutes, free of charge.

Notable projects completed during this review period were: an apparatus for the analysis of single platinum crystal (ICPET), an electrochemical cell (INMS), and an encapsulation of a brain artifact (Catherine Richards).

Published Papers and Presentations

This publication list is for the calendar year 2003. A complete list of publications, from 1999, is available at http://inms-ienm.nrc-cnrc.gc.ca/research_and_development/optics_publications_e.html.

Articles in Refereed Journals

Chang, S., M. Rioux and **C.P. Grover.** "Range face recognition based on the Phase Fourier Transform", Optics Communications, **222,** 143-153 (2003).

Flueraru, C. and **C.P. Grover.** "Overlap Integral Analysis for Second Harmonic Generation within Inverted Waveguide using Mode Dispersion Phase Match", IEEE Photonics Technology Letters, **15,** 697-699 (2003).

Flueraru, C. and **C.P. Grover.** "Relative measurements of second order susceptibility with reflective second harmonic generation method", Applied Optics - Lasers, Photonics and Environmental Optics, **42.33,** 6666-6671 (2003).

Furuhashi, H., K. Matsuda and **C.P. Grover.** "Visualization of phase objects by use of a differentiation filter", Applied Optics, **42.2,** 218-226 (2003).

Liu, X., X. Cai, S. Chang and **C.P. Grover.** "Optical system having a large focal depth for distant object tracking", Optics Express **11.24,** 3242-3247 (2003).

Refereed Conference Proceedings

Atieh, A., C. Flueraru and **C.P. Grover.** "Extremely flat ultra-wide band supercontinuum for WDM/ TDM applications", Proceedings of SPIE, **5260**, Applications of Photonic Technology 6, Ed. Roger A. Lessard, George A. Lampropoulos, 55- 61 (2003).

Chang, S. and **C. P. Grover.** "Hybrid optical correlator used as an intelligent instrument". Proceedings of the 6th International Symposium on Measurement Technology and Intellegent Instrument (ISMTII 2003). Paper No 217(2003).

Chang, S. and **C. P. Grover.** "Invariant features: extraction, visualization and applications", SPIE Proc. on the 7th World Multiconference on Systemics, Cybernetics and Informatics, **4**, 324-329 (2003).

Chang, S., M. Zhou and C. P. Grover. "Invisible information coding technology for security ID applications", Proceedings of Symposium on the Role of Academy in the War on Terrorism, 1-2 (2003).

Liu, X., S. Chang, X. Cai and **C. P. Grover**," Full-field optical coherence tomography using a spatial filter mask", Proceedings of SPIE Annual Meeting 2003, 5174-25 (2003).

Invited Oral Presentations

Flueraru, C. "Cascaded Second-Order Nonlinearity in Organic Waveguide", Brockhouse Institute for Material Research, McMaster University, (September 2003).

Conference Poster Presentations

Flueraru, C., C.P. Grover and **S. Schrader.** "Analysis of Cascaded Second-Order Nonlinearities in Inverted and Periodically Poled Organic Waveguides", Eleventh Canadian Semiconductor Technology Conference, Ottawa (August 2003).

Liu, J., F. Zhang, C. Flueraru, X. Liu, S. Chang and **C.P. Grover.** "Simultaneously Writing of Gratings and Waveguides in Fused Silica by Fentosecond Lasers", Eleventh Canadian Semiconductor Technology Conference, Ottawa (August 2003).

Liu, J., F. Zhang, C. Flueraru, X. Liu, S. Chang and **C.P. Grover.** "Waveguide Shaping and Writing in Fused Silica using Femtosecond Laser Technology", Eleventh Canadian Semiconductor Technology Conference, Ottawa (August 2003).

Technical Reports

Chang, S. and **S. Rao**, "Software Design of Spectral Data Acquisition Using USB2000 Fiber Optic Spectrometer", (October 2003).

Rao, S. and **S. Chang.** "Spectral Data Acquisition Using USB2000 Fiber Optic Spectrometer" (July 2003).

Patents

S. Chang, X. Liu and **C. P. Grover.** "Novel technology for full-field optical coherence tomography and its application to multiple layers information decoding." US patent application filed 24 June 2003.

S. Chang, M. Zhou and **C. P. Grover.** "Spectral coding by fluorescent semiconductor nanocrystals for document identification and security applications." US patent application filed 24 June 2003.

X. Liu, X. Cai and **C. P. Grover.** "Optical tracking system." US provisional patent filed 31 October 2003.

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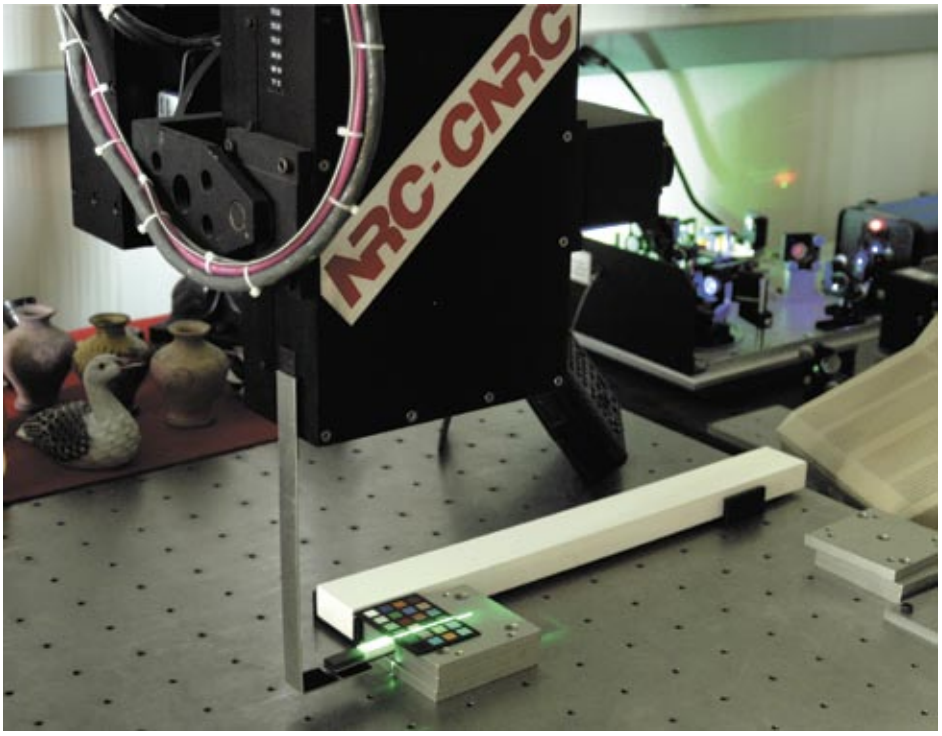
Photometry and Radiometry

The NRC 3D laser-scanning camera was developed to capture the shape and colour of 3D objects, such as paintings, sculptures and archeological artifacts, which can then be computer-rendered with high realism.

In partnership with the NRC Institute for Information Technology, the Photometry and Radiometry Group has enhanced the colour performance of the camera turning it into a multispectral system with more laser wavelengths and improved methods for calculating colours.



The Photometry and Radiometry Group provides internationally recognized primary standards for optical radiation measurements. These standards support the metrological needs of Canadian industry, and help reduce technical barriers to international trade. The Group maintains and improves standards for the measurement of light, ultraviolet and infrared radiation in the wavelength range 200 nm to 50,000 nm, and carries out targeted research and development in areas of current and anticipated Canadian technological growth. Photometric and radiometric standards are disseminated through a comprehensive calibration service, timely response to technical inquiries and requests for services, and collaborative R&D arrangements.

The Group's research program includes the photometric and radiometric standards of luminous intensity (the candela), luminous flux, spectral irradiance, spectral radiance, spectral responsivity, spectral transmittance and reflectance (regular and diffuse), specular gloss, and industrial colorimetry.

Our cooperative research with colleagues and clients, participation in international standards organizations, and comparisons of national standards with the other National Metrology Institutes (NMIs) insures international recognition.

Absolute Radiometry

L.P. Boivin, R. Gerson

NRC acquired a cryogenic radiometer in 1994 and it is now our primary radiometric standard. The main objectives of this project are: to develop and maintain a cryogenic radiometer facility and the associated transfer radiometers for primary spectral responsivity measurements; to realize cryogenic radiometer based spectral responsivity scales, and calibrate the working standards needed to disseminate those scales; to participate in international comparisons of spectral responsivity scales; and to assist in the realization of a luminous intensity scale and a spectral irradiance scale traceable to the cryogenic radiometer.

We have used the fully automated cryogenic radiometer facility to realize spectral responsivity scales in the UV (200 nm-400 nm), in the VIS-IR (400 nm–1000 nm) and in the near-IR (1000 nm–1600 nm). In order to do this, three types of transfer radiometers were developed, characterized, and calibrated for the different spectral ranges: single detector radiometers incorporating PtSi or Si diodes; trap radiometers incorporating Si diodes; and sphere radiometers incorporating InGaAs detectors. NRC is participating in Consultative Committee on Photometry and Radiometry (CCPR) international comparisons where cryogenic radiometer-based transfer standards were used to calibrate comparison artifacts. The results of the 1998-99 CCPR-S3 comparison show that, on average, NRC agrees with the world mean by about 0.04%, and with NIST by about 0.01%. NRC is currently participating in the three-phase CCPR Key Comparison K2 on spectral responsivity measurements.

Three transfer radiometers incorporating PtSi detectors were assembled and characterized for spatial uniformity. We measured the relative spectral responsivity of these radiometers, in the range of 200 nm-300 nm, using monochromator apparatus and spectrally flat thermopiles as reference detectors. By direct calibration with the cryogenic radiometer apparatus, we determined the absolute responsivity of the PtSi radiometers in the range of 250 nm-400 nm. The relative and absolute calibrations were spliced at 250 nm to obtain an absolute calibration from 200 nm to 400 nm. This constitutes a PtSi based UV responsivity scale for the range of 200 nm-400 nm. These transfer standards were used to calibrate working standard PtSi radiometers, which will be used for routine calibrations in the range of 200 nm-400 nm.

Detector Characterization/Calibration Services

L.P. Boivin, R. Gerson

This project's main objectives are to establish and maintain facilities for the calibration and characterization of optical radiation detectors, and to provide associated calibration and consultation services. We have developed automated facilities to carry out the following measurements: routine spectral responsivity measurements (250 nm-3000 nm); spatial uniformity of detectors in narrow spectral regions in the spectral range of 250 nm-2300 nm; angular variation of responsivity of detectors in narrow spectral regions in the spectral range of 250 nm-2300 nm; and absolute and relative detector linearity measurements in narrow spectral regions in the spectral range of 250 nm-2300 nm. These facilities serve both internal needs

for detector characterization, and provide calibration and measurement services to clients. We have also set up a UV-meter calibration facility for the calibration of UVA and UVC type UV meters.

The spectral responsivity facility has operated for many years, and all calibrations are now traceable to NRC's cryogenic radiometer. The facility uses three types of working standards: silicon photodiodes for the spectral range of 250 nm-1100 nm; germanium photodiodes for the spectral range of 700 nm-1800 nm; and liquid-nitrogen cooled indium antimonide detectors for the spectral range of 1200 nm-3000 nm. This calibration service (PAR-110) is part of the INMS Quality System; all documentation is now complete, including sub-procedures for the calibration of electronic and optical apparatus used for these measurements. External assessment of this calibration service has also been carried out.

Although uniformity measurements, angular variation of responsivity measurements, linearity measurements, and UV meter calibrations are not part of the quality system, we have developed calibration report templates that briefly describe the procedures used and give the results of measurements in tabular and graphical form. The linearity apparatus is undergoing a major modification in which the multiple double-aperture wheel has been replaced by a single large area double aperture used in conjunction with neutral density filters. This modification will allow measurements at a higher flux level. At the same time, we are replacing the data acquisition with a newer version that will allow data acquisition and analysis to be migrated to a Win2000 platform. This work is still underway.

We had to modify the working standard re-calibration schedule, following the external assessment of the spectral responsivity measurement procedure PAR-110. All working standards will now be intercompared and interchanged annually to detect any changes in the standards. We modified the PAR-110B reference document to incorporate this new working standard validation procedure. The procedure was carried out on all silicon and germanium working standards early in 2004. This indicated very little change in the working standards.

We recalibrated the indium antimonide (InSb) working standards this year, because their original calibration was five years old and not traceable to the cryogenic radiometer. The re-calibration was a two step procedure, involving: an absolute calibration using sphere radiometer transfer radiometers and Germanium working standards, in the spectral range 1100 nm-1600 nm; and a relative calibration using thermopiles in the spectral range 1500 nm-3000 nm. The relative calibration was then normalized and spliced to the absolute calibration at 1500nm to obtain the total spectral calibration.

We calibrated the Si working standards, using PtSi transfer standards in the range of 200 nm-400 nm. These measurements showed good agreement above 250 nm, but in the range of 200 nm-250 nm, revealed discrepancies much larger than anticipated. This is more than likely due to significant aging in the spectral range of the Hamamatsu S1337 silicon diodes used as working standards. As a consequence, we have discontinued calibrations below 250 nm using Si working standards. New

PtSi working standards were prepared and calibrated in the 200 nm-400 nm range, using the PtSi transfer standards. These PtSi working standards will be used for UV only calibrations in the 200 nm-400 nm range. We will initiate this new service in the near future.

Plans for a new facility for routine spectral responsivity measurements are under way; the current facility is over 20 years old and cannot be upgraded without major hardware replacement. The new facility will use a high-accuracy double monochromator, and will be used for both routine calibrations and transfer calibrations – that is, calibrations of working standards using transfer radiometers, and Key Comparison measurements. We have already selected, ordered, and received the double monochromator and a data acquisition system. We have also started software development and testing of the wavelength drives of the monochromator.

Spectroradiometry/Photometry/Calibration Services

A.A. Gaertner, F. Gauthier

This project develops and maintains, by means of primary lamp standards calibrated using absolute radiometers, the photometric measurement standards of luminous intensity, luminous flux, illuminance and luminance, and the spectroradiometric measurement standards of spectral irradiance and spectral radiance. It also provides associated calibration and consultation services.

We calibrate illuminance meters using our working standards for luminous intensity, which provide a range of illuminances from approximately 50 lux to 500 lux depending on the distance between the lamp standard and the illuminance meter. In response to client requests, we are evaluating the possibility of extending the illuminance range to 1000 lux by operating the lamp standards at a closer distance from the illuminance meter. We have measured the inverse-square-law behaviour of these lamps, and it has been determined that their irradiance does follow the inverse-square-law up to 1000 lux. Further work will determine whether the spatial uniformity of the irradiance produced at these short distances from the lamp is high enough to maintain the accuracies required for the calibrations.

The electrical operating parameters of many standard incandescent sources are determined by the requirement that, at the given electrical operating conditions, the spectral output of the lamp can be characterized by a certain correlated colour temperature. We have purchased and calibrated a compact silicon-diode-array spectroradiometer that can be used to perform the required spectral measurements rapidly and accurately. This instrument operates in a spectral radiance measurement mode, enabling us to perform many of the luminance calibrations required by our clients.

Our reference spectroradiometer performs our highest accuracy measurements of spectral irradiance. We offer calibration services for the wavelength range from 300 nm to 1600 nm. We are presently extending our measurement capabilities into the IR, up to 2500 nm, and into the UV, down to 200 nm. A temperature-controlled

InGaAs detector unit that operates near room temperature has been constructed and installed to provide reliable operation in the measurements from 1100 nm to 1700 nm. The InSb detector that we purchased is not sensitive enough to allow us to extend our calibration services to 2500 nm. The extension of our spectral irradiance measurements in the UV down to 200 nm required considerable modifications to our spectroradiometer. Because of the low output of lamps in this wavelength range, we will do the extension using 1000 Watt FEL lamps (to 250 nm) and deuterium arc lamps (to 200 nm). We have installed and are testing a solar-blind photomultiplier tube (PMT) for use in the 200 nm to 350 nm range. To increase the measurement signal, we have been testing a new optical configuration on the input to our monochromator that uses a flat diffuser instead of our usual integrating sphere. We are also evaluating the operation of the spectroradiometer in this wavelength range without the prism-predispersor. Our initial measurements indicate that more care will be necessary to remove unwanted stray light both internal and external to the spectroradiometer.

The laboratory is currently participating in four CCPR international comparisons: the Key Comparisons of luminous responsivity, spectral irradiance (250 nm to 2500 nm) using incandescent lamps, UV spectral irradiance (200 nm to 350 nm) using deuterium lamps, and a special comparison of spectral radiance using strip filament lamps. Our participation in these comparisons is vital to establish our measurement and calibration capabilities in relation to other international NMIs.

Many of our clients are requesting that our calibration services be ISO-17025 certified. We have been developing the procedures for four important calibration services: luminous intensity, total luminous flux, illuminance, and spectral irradiance. During this past year we have updated our Quality System procedures, and our laboratory has undergone an external peer review and audit. In response to the results of this audit, we will be revising our documentation. We are also planning to extend the scope of our certified measurements to include the measurements described above: higher illuminance levels, correlated colour temperature, and an extended spectral range for spectral irradiance.

In April 2004, we will participate in the 2nd APMP-SIM Joint-Workshop on the Implementation of Quality Systems in National Metrology Institutes. The workshop's objective is to develop test methods in generic terms to assist implementation of quality systems in developing NMIs.

Spectrophotometry/Gloss/Calibration Services

J. Zwinkels, M. Noël, J. Cox, É. Côté

Spectrophotometry involves the measurement of the spectral transmittance, reflectance, and scattering properties of materials in the ultraviolet, visible, and infrared wavelength regions. This information is needed for quality process control of visual appearance and optical performance, which are important to the optical component and colour-intensive industries. It is also needed for material identification and analysis, which are important to the chemical and pharmaceutical industries. Specular gloss is an important appearance attribute of many opaque

reflecting materials, including paints, papers, plastics and textiles. This project's objectives are to develop, maintain, and improve instrumentation, procedures, and standards for the accurate measurement of spectral transmittance and reflectance factors from 200-3000 nm; to realize and maintain primary specular gloss scales for standard geometries of industrial importance; to participate in international comparisons of these measurements to ensure consistency with scales maintained by other countries; and to provide associated calibration and consultation services.

To meet these objectives, we developed several reference instruments for realizing primary scales of transmittance, reflectance, and gloss. In 2003-2004, the incorporation of a new sphere detector for infrared measurements improved the performance of the reference spectrophotometer. This sphere detector incorporates two thermoelectrically-cooled PbS detectors in a 70 mm diameter Spectralon sphere. We tested this sphere design and found that it gives significantly reduced measurement errors. These errors are caused by beam displacement, which occurs with imperfect (thick or wedged) samples.

The NRC diffuse reflectance factor scale is based on the NRC Absolute Reflectometer, one of only three ISO-authorized reference reflectometers in the world. In 2003-2004, we participated in the CCPR Key Comparison of spectral diffuse reflectance factors. This comparison involved three samples, each made of Spectralon and a matte white ceramic tile, measured over the spectral range of 350 nm to 850 nm.

NRC also carried out measurements for a multi-laboratory comparison of the wavelength assignment of holmium oxide solution and Nelson wavelength standards. The pilot lab (NIST Analytical Chemistry Division) has finished compiling and analyzing the data from the 13 participating laboratories. The results support the use of holmium oxide solution as an intrinsic UV/visible wavelength standard with uncertainties of 0.2 nm for bandwidths below 2.0 nm.

Fluorescence Calibration Services

J. Zwinkels, F. Gauthier

This project's objectives are to develop and maintain state-of-the art spectrofluorimetric calibration facilities for high-accuracy colour evaluation of fluorescent materials; to investigate the fluorescent properties of materials used as transfer standards and other applications; to participate in comparisons as required; to ensure international acceptance of these measurements; and to provide associated calibration services.

The current fluorescence measurement facilities include a two-monochromator reference spectrofluorimeter for absolute radiometric calibrations of fluorescent materials (i.e. reflected and total spectral radiance factors) over a wavelength range of 250 nm to 1050 nm. This instrument has a measurement geometry of 45° annular illumination, and 0° viewing (45/0) in accordance with the American Society for Testing and Materials (ASTM) and the International Commission on Illumination (CIE) colorimetric standards. The uncertainty of these calibrations is typically better

than 1%, at an approximately 95% confidence level. In 2003-2004, this facility was used extensively for calibration of a variety of fluorescent papers, plastics, textiles, and retroreflective materials. Currently, NRC provides traceability for optical measurements of fluorescent paper standards for all paper industry's ISO-authorized laboratories, worldwide. In 2003-2004, we conducted significant work in realizing an absolute 45/0 diffuse reflectance factor scale based upon the NRC Reference Spectrofluorimeter. For this purpose, two large area silicon detector assemblies were designed, constructed, and calibrated for absolute spectral responsivity. Preliminary measurements demonstrate the feasibility of this approach.

Development of Goniospectrophotometric Facility

J. Zwinkels, M. Noël, Y. Li , J. Liu

This project aims to develop a versatile reference instrument with the following measurement capabilities: 1) specular gloss at 20, 60, and 85 degree geometries in accordance with ASTM D523 and ISO 2813 specifications (to replace the old NRC Glossmeter); (2) specular gloss at 75 degrees geometry in accordance with TAPPI T480 and ASTM D1223 specifications (for new application to paper samples); and (3) abridged spectral multi-angle measurements of gonioapparent materials, such as metallic and pearlescent paints, whose colour appearance changes depending on the angle of illumination and/or view. These gonioapparent materials are being increasingly used in applications such as currency and identity cards (as anti-counterfeiting deterrents) and automotive finishes (to enhance contours).

We have designed and built a versatile instrument to meet all three of these requirements. This fully-automated instrument incorporates a sphere source with color-correcting filter, chromatic aberration corrected optics, selectable angles of illumination and viewing, collection apertures and bandpass and polarizer filter options, and either a photometer or sphere detector for gloss and gonioreflectance measurements, respectively. The instrument is easily converted from collimated to converging beam geometry with the insertion of some auxiliary optics. This is the first reported reference glossmeter that meets these two very different gloss specifications. We have fully characterized the instrument for sources of error, including: beam uniformity, polarization, geometric effects, source and detector spectral conformance, and reproducibility. We completed an intercomparison between the new Goniospectrophotometer and the old Glossmeter by using a series of 12 NRC gloss standards. The agreement was well within the target uncertainty of 0.3 gloss units for 20, 60 and 85° geometries. Preliminary results for 75° gloss measurements indicate that we can also meet target uncertainty of 0.3 gloss units. We expect that the instrument can be commissioned for gloss calibration services in September 2004. Two papers and one poster will be presented on the results of this research at the joint ISCC-CORM Conference in May 2004.

Colorimetry – Visual Aspects

R. Baribeau, A.R. Robertson, J. Cox, C. Collin

Colorimetry is a sophisticated and rather complex interdisciplinary science. To describe colour perceptions accurately, one needs accurate measurements of the

physical properties of the stimulus, and a fundamental understanding of the human factors involved in producing the perception of the stimulus. Within this context, our work aims to improve standards for industrial colorimetry by developing and applying basic knowledge of the physical and psychophysical properties of colour stimuli. Accurate calibration of stimuli and discrimination of small colour differences are our current areas of research.

The project has developed a computer-controlled cathode-ray-tube (CRT) visual colorimeter for carrying out colour discrimination experiments. It relies on feedback from a tele-spectroradiometer to accurately set the colours of pairs of colour stimuli that differ in one of their colour attributes. Human observers can then judge the colour pair, allowing determination of visual thresholds. The ultimate goal of this research is to improve existing colour difference formulae to accurately predict decisions about the acceptability of colour for setting industrial colour tolerances. This work has important ramifications in reducing the production costs in a wide variety of color reproduction technologies.

We carried out an experiment measuring hue discrimination thresholds of human observers. Special software was developed for the accurate display of colour pairs on a high resolution CRT using serial feedback from a spectroradiometer. We determined discrimination thresholds between a test and a target colour by repeatedly showing an observer a circle composed of four separate quadrants, one of which has a different colour from the other three. Three quadrants are of the test colour while one is of the target colour, or vice versa. Observers are asked to select the quadrant that differs from the others. Eighteen hue threshold values around the hue circle, at constant L* and C*, were measured for three observers. We found hue thresholds varied around the hue circle, and exhibited an abrupt change in the blue to purple region ($240^{o} \leq h^*_{ab} \leq 300^{o}$). This change is not fully accounted for by any CIELAB-based colour difference formula, including the most recent CIEDE2000 formula. We are preparing a paper on these findings.

We initiated a second project to study the influence of spatial effects, such as texture, on our ability to discriminate colour. In the past year, Dr. Charles Collins (NSERC Postdoctoral Fellow) developed a visual experiment using the CRT-based colorimeter, and he has tested observers' abilities to discriminate hues in the presence of lightness sinusoidal modulation across the visual fields. One colour centre has been tested, with three observers, for spatial frequencies ranging from 0 to 16 cycles per degree. Results suggest there could be some aliasing-like phenomena involved, i.e. our ability to discriminate the hues seems enhanced at some frequencies and degraded at others; discrimination thresholds oscillate with the spatial frequency, as if there were a frequency wrap-around at the Nyquist sampling limit.

Colorimetry - Characterization of Colour Imaging Devices

R. Baribeau, J. Cox, É. Côté

The Colorimetry Program at INMS collaborates, partners, and consults with the public and private sectors to develop new and improved facilities and standardized

procedures for the accurate measurement and specification of colour and light. These requirements are important in a wide range of applications and industries that make use of digital colour imaging systems such as displays, cameras, and scanners.

At the core of our measurement facilities are two spectroradiometers, traceable to the International System of Units (SI) through INMS-maintained photometric and radiometric scales. These instruments have been characterized in such a way that we can associate a detailed uncertainty budget with any measurement of luminance and chromaticity from light stimulus of arbitrary spectral composition. A tristimulus-colorimeter and a ccd-based imaging colorimetric camera complement our facility. These instruments can be used in conjunction with special accessories, such as our new 1,6m integration sphere, to characterize colour devices in custom configurations.

We have constructed an integrating sphere, 1,6 m in diameter, with an internal tungsten halogen lamp, to test displays under ambient lighting conditions. The white and black of the display are measured remotely with our tele-spectroradiometer, allowing the calculation of spectral reflectance factors. The contrast ratio can then be predicted for any level and any spectral composition of the ambient illumination. Using the charge-coupled-device (CCD) colorimetric camera, we can also measure the entire display and map the contrast-ratio. This facility can assist a variety of clients, including flat panel display manufacturers.

A prototype standard source called the Gamut Assessment Standard, currently being developed by NIST, was also measured in our facility for inter-comparison with measurements obtained at NIST and at NPL (U.K.). The prototype comprises an integrating sphere and a set of optical filters designed to simulate photometric and colorimetric conditions common in electronic displays.

Colorimetry – Development of a Multispectral 3D Camera

R. Baribeau, M. Rioux (IIT)

This project is in partnership with the NRC Institute for Information Technology (IIT) and aims to improve the colour performance of their 3D laser-scanning camera. The camera is used to digitize real objects, particularly artwork, in colour and 3D. These images are useful for studying and documenting the physical state of the object. In virtual reality applications, they serve to generate realistic renderings of the object as seen from interactively selected user view points.

The 3D laser formerly relied on three lasers, one red, one green, and one blue, for the capture of reflectance factors from surface elements at three wavelengths. Colour inaccuracy arose from this sparse sampling of the full spectral reflectance over the visible range. This technology is currently being turned into a multispectral system with more laser wavelengths and improved methods for calculating colours.

The optimal laser wavelengths used for the scanning were investigated theoretically and in the lab. Optimal wavelengths were first established theoretically based on the criteria of minimal CIEDE2000 colour difference over the set of reflectance

curves from the full Optical Society of America Uniform Color Scales (OSA-UCS) catalog. We considered PCA-based and spline-based spectral estimation methods, and derived sets of three, four, and five optimal sampling wavelengths for each method. These sets provided a basis for the selection of HeCd, ArKr, HeNe, and DPSS commercial laser lines, for which we predicted the colorimetric performance. This was then tested in the lab, where colour rendition charts were scanned with the camera at seven wavelengths; afterwards the charts were computer-rendered on a CRT display. Both the theoretical prediction and the experimental observation indicate that four well-chosen wavelengths are adequate for proper rendition of the charts.

Infrared Regular Spectrophotometry

N.L. Rowell, R. Gerson

This project aims to provide and improve standards for regular transmittance and reflectance factors in the mid-infrared (2.5 to 20 μm) wavelength range. We currently have one procedure in the final stages of the ISO accreditation process. This procedure is for the regular transmittance factors of a standard glass filter artifact in the low wavelength part of the mid-infrared spectrum. Presently, the calibration of client artifacts is possible; alternatively, we can supply calibrated standards. Accurate measurements of infrared transmittance and reflectance are important to many industries, including the chemical, pharmaceutical, plastics, and thin film sectors, which use infrared methods for quality control.

The current year's work concentrated on satisfying the requirements for ISO accreditation, and on participating in the internal and external audits related to this Quality System implementation.

Infrared Raman Scattering

N.L. Rowell, D.J. Lockwood (IMS), L.-L. Tay (IMS)

We are using Raman scattering for the characterization of materials. Raman scattering is extremely powerful, because it can be used to probe the vibrational and electronic properties in many types of materials. By using infrared laser excitation it is possible to avoid resonant effects such as fluorescence in organic molecules or carrier absorption in semiconductors, which hide the Raman signal. We have therefore developed an infrared Raman capability for the characterization of materials. Our first experiments have been on semiconductors, such as InP and GaAs, which have bandgaps lower than the 1.06 μm exciting laser. We were able to deduce effective doping levels in these materials with the method.

Recently, we have used 1.32 μm excitation to observe phonons in silicon in a 90^{o} scattering geometry, which was not possible with the shorter wavelength excitation used by previous authors. We were able, with this method, to obtain further fundamental information regarding the lattice vibrations in this important material.

Infrared Photoluminescence

N.L. Rowell and collaborators D.C. Houghton (Aixtron Inc.), S. Sheng (IMS & Dalhousie University), J.-M. Baribeau (IMS), D.J. Lockwood (IMS), S. McAlister (IMS), I. Berbezier (CNRS Marseille), A. Ronda (CNRS Marseille), D. Webb (ATMI Epitaxial Inc.), M. Ward (ATMI Epitaxial Inc.)

In this activity, we collaborate with various partners in applying infrared photoluminescence to semiconducting materials of technological importance.

Photoluminescence (PL) provides valuable information about the electronic properties of epitaxial, thin-film materials, notably on the morphology, impurities, point and extended defects, strain, and crystalline integrity. The present apparatus consists of a high performance Fourier transform PL arrangement, with the cryostats, detectors, and spectrometer, to measure PL in the near infrared from 800 nm to 4000 nm.

In the past year we have conducted a number of PL studies of the alloy semiconducting material, SiGe, now widely used in the semiconductor industry for high-speed applications. Our work resulted in two publications, two contributed conference presentations, two invited lectures, and a book chapter.

Far-Infrared Reflectance Spectroscopy of Semiconductors

N.L. Rowell and collaborators D.J. Lockwood (IMS), G. Yu (IMS), L.-L. Tay (IMS)

This project is in collaboration with IMS, and uses far infrared reflectance methods at wavelengths longer than 20 μm to study the phonon properties of semiconducting microstructures.

The measurement of lattice vibrational properties (phonons) in solid materials provides valuable and fundamental information about the materials notably in thin film form. For example, interface properties in multilayer structures, of great importance to device performance, can be investigated through vibrational spectroscopy. Such structures are ubiquitous in modern technology, e.g., in semiconducting diode lasers. The method we have developed is oblique incidence, polarized far infrared (15-500 μm) regular reflectance spectroscopy. The method has several distinct advantages over other techniques, not the least of which is its ability to allow unambiguous calculations of thin film properties from measurements. Another advantage is that, for thin films of polar materials such as GaAs, the material's phonons are observed directly as sharp changes in reflectance; this method also enables the evaluation of particularly thin films (1 nm thick). This work was presented at several conferences, and in four journal articles.

Infrared Diffuse Properties

N.L. Rowell

This project provides industry with the measurement of diffuse reflectance and transmittance factors in the mid-infrared (2-16 μm) spectral ra nge. Such measurements enable the calculation of a key thermal material property, emissivity, at ambient temperature.

Presently, the apparatus consists of a Fourier transform infrared spectrometer connected to a center-mount, diffuse gold interior integrating sphere, with a liquid

nitrogen-cooled InSb photodiode, and a liquid helium-cooled Si:Ga photoconductor, as detectors. Spectra are taken with samples at room temperature in vacuum, which has been greatly improved by recent modifications to the vacuum system. Also, we incorporated the new Si:Ga detector into the system with custom-designed relay optics and detector cryostat. The apparatus performed very well for a number of client samples and for the re-calibration of various in-house artifacts

Measuring Temperature with IR Spectroscopy

N.L. Rowell and A.G. Steele (Thermometry Group), A.R. Leesink (Thermometry Group)

This project in collaboration with the Thermometry Group investigates improved radiation thermometry using a multi-spectral approach. This project aims to use redundancy in infrared spectral measurements to deduce the temperature of blackbody cavities in the 500-1000 ºC range. Since the method relies only on Planck's law and certain reasonable assumptions about non-ideal blackbodies, as well as good radiometric precision, it will have the advantage of providing absolute thermodynamic temperature.

Presently, we have installed the apparatus, which consists of two heatpipe variable-temperature cavities and a benchtop Fourier transform spectrometer. Our experiments and calculations in the past year focused on measurement accuracy and blackbody non-ideality. Two conference papers were presented on this work.

Infrared Attenuated Total Reflectance

N.L. Rowell, L.-L. Tay (IMS) D.J. Lockwood (IMS) S. Morin (York University) R. Boukherroub (IEMN-IRI, Villeneuve d'Ascq, France)

This project is motivated by the need to have monolayer sensitivity to organic layers functionalized onto planar surfaces, such as those of silicon wafers. Functionalized materials have a wide range of applications, including molecular based electronics, sensors, Micro Electro-Mechanical Systems (MEMS), and biologically active surfaces. For Si, the power of functionalization occurs through the selective attachment of molecules covalently onto Si surfaces. This enables the introduction of chemical species with different functional group onto Si surfaces, which greatly alters in a controllable fashion the surface properties of the Si.

Using the information on vibrational bands obtained from infrared transmittance spectroscopy is one method of characterizing organic compounds. Unfortunately, the thinner the layer, the smaller the absorption effect; this means that very thin layers cannot be observed with conventional sampling. However, the use of attenuated total reflectance (ATR) surface enhancement and monolayer detection has been achieved with special multibounce ATR cells. Unfortunately, this approach suffers from the expense of the ATR cells, the large area sampled, few suitable substrate types, and a restricted spectral range. We recently achieved a single-reflection ATR measurement method in apparatus of our own design and construction in which we observed the vibrational modes of monolayer thick organic (e.g., undecylenic acid) films functionalized on Si(111). Very importantly, this method is scalable to smaller areas (i.e., microscopy). It employs optical incidence beyond the critical angle in a high index hemisphere in intimate contact

with the coated substrate; it also achieves a large surface enhancement, allowing the clear identification, through molecular vibrations, of the molecular type, orientation, and surface coverage. With this method we are able to detect monolayers on standard single-side polished Si wafers. It should also be possible to adapt the hemisphere technique, and to allow the deposition onto a more biologically favorable surface such as glass.

INMS Quality System

The external assessment of the Photometry and Radiometry Group calibration and measurement services, for the purposes of accreditation to ISO/IEC 17025, was carried out on October 6-8, 2003. The scope of accreditation (range and uncertainties) of the submitted measurement capabilities for luminous intensity, spectral regular transmittance, spectral irradiance, infrared regular transmittance, spectral responsivity, luminous flux, and illuminance, were largely accepted as submitted to the BIPM Appendix C database. The Group has prepared and implemented a plan of action to address the findings of this external assessment. We expect to receive formal accreditation by December 2004.

International Comparisons

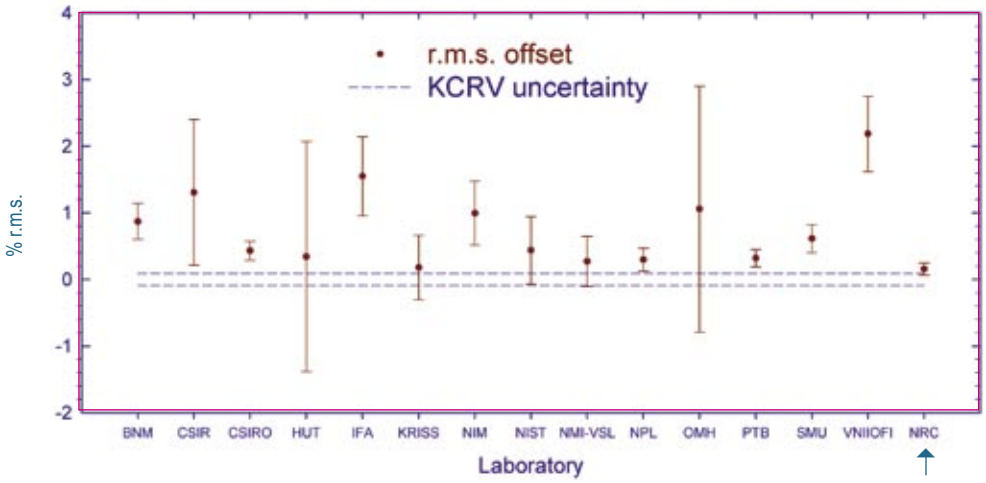
- CCPR K1a: spectral irradiance of lamps from 250 nm to 2500 nm
  We have performed the NRC measurements for this comparison. The pilot laboratory, NPL, is evaluating the results and preparing the draft report.

- CCPR K1b: spectral irradiance in the range 200 nm to 400 nm
  We have completed the first set of measurements for the UV spectral irradiance comparison, and the deuterium lamps are presently being measured by the pilot laboratory, PTB, before being returned to NRC for the final set of measurements.

- CCPR K2a: spectral responsivity of InGaAs detectors in the range 900 nm to 1600 nm
  Early in 2003, the Draft A report was sent to participants by the pilot laboratory, NIST. The comparison artifacts were InGaAs detectors calibrated in the range of 900 nm to 1600 nm. NRC results are the best of all participants; we have the smallest offset from the Key Comparison Reference Value (KCRV), and the smallest uncertainty. (See results on page 14.)

- CCPR K2b: spectral responsibity of Si diode based single detectors and traps in the range of 300 nm to 1000 nm
  The pilot laboratory is the BIPM. All measurements are now complete and this comparison is at the Draft B stage.

RESULTS OF THE CCPR K2a COMPARISION



- CCPR K2c: spectral responsivity of silicon and PtSi photodiodes (single and trap detectors) in the range of 200 nm to 400 nm
  The pilot lab, PTB, has distributed and finalized the protocol for this comparison. NRC is participating in Group C of this comparison and recent work with the cryogenic radiometer has consisted of developing, characterizing, and calibrating Si and PtSi transfer radiometers to realize a new UV spectral responsivity scale, and to prepare for this comparison.

- CCPR K36: luminous responsivity
  The final report for the luminous responsivity comparison was published in January 2004, and indicates that the NRC results differ from the key comparison reference value by only 0.03%, with a one standard deviation uncertainty of 0.50%.

- CCPR K5: spectral diffuse reflectance
  We have completed the NRC measurements for this comparison and we are working on the final report to be sent to the pilot laboratory (NIST).

- CCPR K6: regular spectral transmittance
  The measurements for this comparison have been completed and the pilot lab, BNM (France), is preparing the Draft A report.

- CCPR S1: supplementary comparison of spectral radiance using strip-filament lamps
  Measurements for this comparison are complete and we are working on the final report to be sent to the pilot laboratory,VNIIOFI (Russia).

Client Services

In 2003-2004, we provided a comprehensive range of calibration services for various photometric, radiometric, and colorimetric quantities, and issued a total of 76 calibration reports.

Committees and Offices

International

Commission International de l'Eclairage (CIE)

Division 2: Physical Measurement of Light and Radiation
J.C. Zwinkels, Canadian Member

Division 8: Image Technology
R. Baribeau, Canadian Member

TC 1-38 Compatibility of Tabular Data for Computational Purposes
A.R. Robertson

TC 1-44 Practical Daylight Sources for Colorimetry
J.C. Zwinkels

TC 1-45 Revision of CIE Publication No. 51 to Include D50 Simulators
J.C. Zwinkels

TC 1-47 Hue and Lightness Dependant Correction to Industrial Colour Difference Equation
A.R. Robertson

TC 1-48 Revision of CIE Document 15.2, Colorimetry
A.R. Robertson

TC 1-53 Standard Method of Assessing the Quality of Daylight Simulators
J.C. Zwinkels

TC 1-55 Uniform Colour Space for Industrial Colour Difference Evaluation
A.R. Robertson

TC 1-56 Improved Colour Matching Functions
A.R. Robertson

TC 1-57 Standards in Colorimetry
A.R. Robertson, Chairman
J.C. Zwinkels

TC 1-59 Standard Photometric 10° Observer
A. R. Robertson

TC 2-04 Stable Secondary Standard Sources
A.A. Gaertner

TC 2-25 Calibration Methods and Photoluminescent Standards for Total Radiance Factor Measurements
J.C. Zwinkels (Chairman)

TC 2-28 Methods of Characterizing Spectrophotometers
A.R. Robertson
J.C. Zwinkels

TC 2-35 CIE Standard for V(l) and V'(l)
A.R. Robertson

TC 2-39 Geometric Tolerances for Colorimetry
J.C. Zwinkels

TC 2-42 The Colorimetry of Visual Displays
R. Baribeau

Uncertainties in Photometry
A.A. Gaertner

TC 2-47 Characterization and Calibration Methods of UV Radiometers
L.P. Boivin

TC 2-48 Spectral Responsivity Measurement of Detectors, Radiometers and Photometers
L.P. Boivin

TC 2-57 Revision of CIE S014-2 (Colorimetry Part 2: CIE Standard Illuminants to include D50)
A.R. Robertson (Chairman)

International Colour Association

2005 Congress, Scientific Committee
A.R. Robertson

International Committee of Weights and Measures

Comité consultatif de Photométrie et Radiométrie
J.C. Zwinkels
L.P. Boivin

International Key Comparison of Spectral Irradiance (K1a)
A.A. Gaertner

International Key Comparison of UV Spectral Irradiance (K1b)
A.A. Gaertner

International Supplementary Comparison #1 (Spectral Radiance)
A.A. Gaertner

Working Group on Key Comparison #2 (Spectral Responsivity)
L.P. Boivin

Working Group on Key Comparison #5 (Spectral Diffuse Reflectance)
J.C. Zwinkels

International Key Comparison #6 (Spectral Regular Transmittance)
J.C. Zwinkels

International Organization for Standardization

TC 6/WG 3 Optical Properties of Paper, Board and Pulp
J.C. Zwinkels, Liaison

Munsell Color Science Laboratory

A.R. Robertson, Advisory Board Member

National

Canadian National Committee of the Commission Internationale de l'Éclairage

A.A. Gaertner, Secretary
J.C. Zwinkels, Division 2 Vice-President
R. Baribeau, Division 8 Member
A.R. Robertson, ex officio

Canadian Pulp and Paper Association

Subcommittee on Optical Properties of Paper
J.C. Zwinkels

Editorships

Color Research and Application
A.R. Robertson, Associate Editor

Metrologia
L.P. Boivin, Guest Editor

Published Papers and Presentations

This publication list is for the calendar year 2003. A complete list of publications, from 1999, is available on the INMS web site at http://inms-ienm.nrc-cnrc.gc.ca/research_and_development/photometry_radiometry_publications_e.html.

Articles in Refereed Publications

J. Taylor, J.-A. Beraldin, G. Godin, L. Cournoyer, **Baribeau, R.** , F. Blais, M. Rioux and J. Dorney. "NRC 3D imaging technology for museum and heritage applications", J. Visual. Comput. Animat., **14,** 121-138 (2003).

S. Sheng, **Rowell**, **N. L.** and S.P. McAlister. "Photoluminescence in tensile-strained Si type-II quantum wells on bulk single-crystal SiGe substrates", Appl. Phys. Lett., **83,** 857-859 (2003).

S.R. Sheng, **Rowell, N.L.** and S.P. McAlister. "Strong near-infrared photoluminescence and absorption from $Si/Si_{1-x}Ge_x$ type-II multiple quantum wells on bulk crystal SiGe substrates", *A*ppl. Phys. Lett., **83,** 2790-2792 (2003).

M. Nadal, **Zwinkels, J.C.** and **M. Noël.** "Specular Gloss Scales Comparison between The National Institute of Standards and Technology and the National Research Council of Canadas", Journal of Coatings Technology, **75,** 45-51 (2003).

Refereed Conference Proceedings

Reesink A.L., N.L. Rowell and **A.G. Steele.** "Using Fourier-Transform Blackbody Spectra To Determine Thermodynamic Temperature In The 600 °C To 1000 °C Range", Temperature: Its Measurement and Control in Science and Industry, Volume 7, Ed. Dean C. Ripple, AIP Conference Proceedings **684,** 19-24 (2003).

Rowell, **N.L.,** D.J. Lockwood, P.J. Poole and G. Yu. "Far infrared spectroscopy of $In_{0.53}Ga_{0.47}As$ quantum wells on InP(100)", Proceedings of the Materials Research Society, **744,** 583-588 (2003).

C.J. Chunnilal, F.J. Clarke and **Rowell, N.** "NPL-NRC Comparison of mid-infrared regular transmittance", Proceedings of the 4th Oxford Conference on Spectrometry, Davidson, North Carolina, June 2002, S.P.I.E., **4826,** 12 - 20 (2003).

Zwinkels, J.C. and **M. Noël.** "Methods for improving accuracy of optical coating analysis from spectrophotometric measurements", Proceedings of the 46th Annual Technical Conference of the Society of Vacuum Coaters, San Francisco, 2003, ISSN 0737-5921, 339-344 (2003).

Zwinkels, J.C. and **F. Gauthier.** "Investigation of photoluminescent effect in opal glasses used as diffuse reflectance standards", Proceedings of 4th Oxford Conference on Spectroscopy, S.P.I.E., **4826,** 70-78 (2003).

Invited Oral Presentations

Rowell, **N.** "What photoluminescence tells us about SiGe heterostructures", INRS-EMT Varennes, QC (February 2004).

Conference Poster Presentations

Rowell, **N.** "Dielectric Properties of InGaAs Epilayers on InP (100)", Proceedings of Materials Research Society – Fall Meeting, Boston (2003).

Rowell, **N.** "Dielectric Properties of GaN Epilayers", Canadian Semiconductor Technology Conference, Ottawa (2003).

Rowell, **N.** "Phonons in GaN Epilayers on Sapphire", Proceedings of Materials Research Society – Fall Meeting, Boston (2003).

Rowell, **N.** "Phonons in $InSb_{1-x}As_x$ alloys", Canadian Semiconductor Technology Conference, Ottawa (2003).

Rowell, **N.** "Phonons in superlattices of InGaAs lattice matched to InP", Canadian Semiconductor Technology Conference, Ottawa (2003).

Rowell, N. "Resolving dopant layers in SiGe/Si heterostructures", ICSi2 Conference, Santa Fe, New Mexico (2003).

Technical Reports

W. Springsteen, J. Sefl, J. Ladson, H.S. Fairman, M. Brill, and **J. Zwinkels**, "Inter-Society Color Council Technical Report 2003-1, Guide to Material Standards and their Use in Color Measurement, 2nd Revision 2003".

"Report on an Intercomparison of Measurements of the Luminous Flux of High-Pressure Sodium Lamps", (CIE Publication No. 153:2003) prepared by CIE Technical Committee TC 2-22 (**A. Gaertner,** member of TC 2-22).

J. Taylor, J.A. Beraldin, G. Godin, L. Cournoyer, **R. Baribeau,** F. Blais, M. Rioux, and J. Domey. "NRC 3D imaging technology for museum and heritage applications", J. Visual. Comput. Animat., **14,** 121-138 (2003).

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Charles Collin (NSERC Postdoctoral Fellow, University of Ottawa)	998-5526
Jian Liu, (NSERC Postdoctoral Fellow)	990-2136

Scientists have developed the novel tunable triple-wavelength erbium-doped fiber ring laser for measuring polarization mode dispersion of the optical fibers and wavelength conversion application based on the broad-band orthogonal-pump four-wave mixing in a semiconductor optical amplifier.

Photonics, the synthesis of optics and electronics for signal and data transmission and manipulation is an area of pivotal importance in information processing and is a key enabling technology for the information highway and multimedia applications. Photonics has achieved spectacular success in data storage and long-haul communications, and is rapidly becoming a competitive technology for the interconnection of electronic processors and peripheral equipment. Research focuses on photonic technology for processing and all-optical networking. Rapidly developing technologies, such as wavelength division multiplexing for transmission and reconfiguration of optical networks, open new possibilities for large bandwidth high performance networking, multiplexed sensor networks, and microwave photonic and signal processing applications.

Design of integrated optical components

Integrated optical components are generally believed to be the next generation of components for optical network system applications. In the past year, we established the design capabilities for a variety of waveguide based optical components, including arrayed waveguide gratings, variable optical components, and splitters. We also designed novel, high-performance, low-cost variable optical attenuators (VOAs) based on polymer material platforms and polymer/silica hybrid waveguide platforms. We developed prototypes for closed loop VOAs and high-performance controllers to be used along with the VOAs.

Component and subsystem development for optical network performance monitoring and enhancement

With the widespread deployment of optical communication networks, monitoring network performance and predicting its reliability have become important concerns. New technologies providing this function, as well as the ability to correct optical signal transmission errors, would significantly lower network operation and maintenance costs. To address these needs, the Photonic Systems Group continues to develop components and subsystems for optical network performance monitoring and enhancement; these include: wavelength blockers, dynamic optical channel equalizers, optical performance monitors, polarization mode dispersion monitors, and all-optical 3R regeneration (reamplification, reshaping, and retiming). More specifically, we have successfully developed new technology for measuring the polarization-mode dispersion (PMD) of optical fibers..

Active photonic devices and subsystems

This project's research activities are the design, fabrication, measurement, and assessment of a range of advanced devices and subsystems in telecommunication. Our goal has been to explore and develop new system concepts, and drive the applications of new active photonic devices and subsystems for next generation networks.

To achieve these goals, we have focused on the following research activities:

- next-generation erbium-doped fiber amplifiers;
- multi-wavelength laser source and fiber optical parametric amplifiers;
- high-power C- and L-band fiber amplifiers and lasers; and
- high-speed printed circuit board (PCB) hardware and firmware design.

All-optical integrated photonic component fabrication

The group has been developing all-optical photonic components and subsystems based on femtosecond laser direct writing and nanomovement automation

techniques. This fabrication technology gives the group a competitive edge in process complexity, cost, and prototyping time. During this review period, we successfully developed the following components and technologies:

- optical waveguide 1X2 and 2X2 couplers;
- optical waveguide 1X2 and 1X4 power splitters;
- balanced and unbalanced waveguide Mach-Zehnder interferometers;
- waveguide shaping technology for reducing bending loss; and
- fabrication of micro/nano- structures within glasses.

Testing and analysis of photonic components and systems

Testing and analysis are essential functions within the operation of the Photonic Systems Group, because they play a vital role in developing, qualifying, and improving photonic components and systems. The Group has continued to develop its photonic component testing facility and expertise necessary for providing support to its researchers and Canadian industry. The testing capability covers a wide range of photonic components and technical parameters. In addition, we have provided consultation and custom specialized training in photonic testing technology to clients.

Packaging of photonic components and development of optical materials

Packaging is the key to making a functional photonic device an affordable, quality product. The current project focus is on the packaging of waveguide-based photonic components covering packaging design, processes, and materials. In the last year, we:

- developed a packaging solution to reduce the manufacturing of arrayed waveguide grating (AWG) components by 30 to 40 %, and a cost-effective technology for eliminating the fiber stress in the packaged waveguide-based photonic components;
- established modeling capability for simulating the stress and heat transfer in the unpackaged waveguide devices and the packaged photonic components. We also improved the design of VOAs and AWGs by simulating stress and heat transfer in the devices.
- developed a silica/polymer hybrid VOA with extremely low power consumption and low dynamic polarization-dependent loss (PDL) based on the stress and heat analysis of conventional VOAs. We also developed a polymer with a super high thermo-optic coefficient, and used it as the cladding for the VOA;
- developed new technology for reducing the birefringence of polymers, and developed the new criterions for applying polymers in manufacturing AWGs and VOAs; and
- developed polymers for claddings used in VOAs.

Reliability testing and qualification of optical components and subsystems

Optical components and subsystems must pass rigorous industrial standards in order to be accepted by the system companies. We have established the capabilities in optical high power testing, damp heat testing, high temperature, and low temperature testing.

Materials for photonic sensing

In collaboration with the Optics Group, we developed semiconductor nanocrystal quantum dots-based optical coding and encryption technologies.

Biophotonics – photonic sensing technology

We established the validity of multilabeling biomolecules at a single site for signal amplification in electrochemiluminescence (ECL)-based biosensing technology. Our current focus is to expand it, such that it becomes a universal platform technology for various bioanalytical applications. Our target is to develop a prototype hand-held ECL detection device by combing the multilabeling technique with nanofabricated ECL chips.

Fiber optic sensor technology

In comparison with other sensing technologies, fiber optic sensors offer many advantages, such as passive designs immune to electromagnetic interference, high sensitivity and environment ruggedness. We have developed a novel wavelength interrogation technique and sensors for the measurement of temperature, voltage, and refractive index.

Client Services

- integrated optical component design
- photonic component testing and characterization
- photonic component packaging

Published Papers and Presentations

This publication list is for the calendar year 2003. A complete list of publications, from 1999, is available at http://inms-ienm.nrc-cnrc.gc.ca/research_and_development/photonic_systems_publications_e.html.

Articles in Refereed Journals

Chang, S., M. Zhou and **C.P. Grover.** "Information coding and retrieving using fluorescent semiconductor nanocrystals for object identification", Opt. Express, **12,** 143-148 (2003).

Liu, J., Z. Zhang, G.Z. Xiao and **C.P. Grover.** "Quantitative criterion for quantum interference within spontaneous emission modification of a driven ladder atom", Journal of Optics B: quantum and semiclassical optics, **5,** S633-S638 (2003).

Lu, Z.G. and **C.P. Grover.** "A high-power-conversion-efficiency L-band erbium-doped fiber amplifier", Acta Optica Sinica, **23,** 463-464 (2003).

Lu, Z.G. and **C.P. Grover.** "A power-controllable semiconductor fiber ring laser and its applications", Acta Optica Sinica,"**23,** 383-384 (2003).

Lu, Z.G., P. Lin and **C.P. Grover.** "A dynamically gain-flattened erbium-doped fiber amplifier", Microwave and Optical Technology Letters, **39,** 8-11 (2003).

Zhang, Z., V. Nawaby and M. Day. "CO2-Delayed Crystallization of Isotactic Polypropylene: a Kinetics Study", J. Polym. Sci. Polym. Physics Ed. **41,** 1518-1525 (2003).

Zhang, Z. and Y.P. Handa. "Polymer mica: layered polymer with nanometer sized interlayer gaps", Journal of Materials Science Letter, **22,** 135-137 (2003).

Zhang, Z., G.Z. Xiao, J. Liu and **C.P. Grover.** "Coupling fibers to planar waveguide with high-temperature epoxy", Fiber and Integrated Optics, **22,** 357-371 (2003).

Zhang, Z., G.Z. Xiao, J. Liu and **C.P. Grover.** "Encapsulating Planar Waveguide-Based Photonic Components with Polymer Coatings", Fiber and Integrated Optics, **22,** 343-355 (2003).

Zhang, Z., G.Z. Xiao, J. Liu and **C.P. Grover.** "Polymers in waveguide packaging", Acta Optica Sinica, **23,** 177-178 (2003).

Zhou, M., J. Roovers, G.P. Robertson and **C.P. Grover.** "Multilabeling biomolecules at a single site: 1. synthesis and characterization of a dendritic label for electrochemiluminescence assays", Anal. Chem. **75,** 6708-6717 (2003).

Zhou, M., J. Heinze, K. Borgwarth and **C.P. Grover.** "Direct voltammetric evidence for a reducing agent generated from the electrochemical oxidation of tripropylamine for electrochemiluminescence of ruthenium tris(bipyridine) complexes", Chemphyschem., **4,** 1241-1243 (2003).

S. Brownstein and **Zhou, M.** "Ring current and steric effects on ^{1}H chemical shifts of Ru co-ordinated 2,2'-bipyridyl", Magnetic Resonance in Chemistry,"**41,** 1041-1044 (2003).

Oral Presentations

Chang, S., M. Zhou, and **C.P. Grover.** "Invisible information coding technology for security ID applications", Oral presentation by S. Chang, Pre-proceedings of Symposium on the Role of Academy in the War on Terrorism, SCI, pages 1-2, Orlando, USA (July 2003).

Zhang, Z., G.Z. Xiao, J. Liu and **C.P. Grover.** "Packaging of photonic components I: Waveguide coupling", 2003 International Conference on Application of Photonic Technology, Montreal, Canada (May 2003).

Zhang, Z., G.Z. Xiao, J. Liu and **C.P. Grover.** "Packaging of photonic components II: Waveguide attachment", 2003 International Conference on Application of Photonic Technology, Montreal, Canada (May 2003).

Zhang, Z., G.Z. Xiao, J. Liu and **C.P. Grover.** "Packaging of photonic components III: Waveguide encapsulation", 2003 International Conference on Application of Photonic Technology, Montreal, Canada (May 2003).

Zhang, Z., G.Z. Xiao, J. Liu and **C.P. Grover.** "Packaging of photonic components IV: Profile cutting of waveguides", 2003 International Conference on Application of Photonic Technology, Canada (May 2003).

Zhang, Z., G.Z. Xiao, J. Liu and **C.P. Grover.** "Packaging of waveguide-based photonic components", ORAP Workshop, Ottawa, Canada (October 2003).

Zhang, Z., G.Z. Xiao, J. Liu and **C.P. Grover.** "Polymers in waveguide packaging", OECC 2003, Shanghai, China (October 2003).

Zhou, M. and **C.P. Grover.** "Electrochemilumine-scence-based bioanalytical assays for medical diagnostics and homeland security", Invited talk at Biolight 2003 – Canada's First Biophotonics Primer & Workshop, Gatineau, Quebec (November 2003).

Zhou, M., G.P. Robertson, **J. Roovers** and **C.P. Grover.** "Bioconjugatable supramolecular assmbly with multiple ruthenium(II) tris(bipyridine) units for boanalytical applications", 39th IUPAC Congress, Ottawa (August 2003).

Conference Poster Presentations

Zhou, M., J. Roovers and **C.P. Grover.** "Multilabeling biomolecules at a single site: a prototype label for electrochemiluminescence assay", Second Annual Chemical Biophysics Symposium, Toronto, Canada (April 2003).

Technical Reports

Lavigne, A. "Design of a High-Power Erbium/ Ytterbium Co-Doped Fiber Amplifier", Internship Report, École nationale supérieure de physique de Strasbourg. Supervisors: **Z.G. Lu** and **C.P. Grover** (2003).

Lu, Z.G. and **C.P. Grover.** "Business Plan of LightLink Technology", NRC Business Case Competition, Ottawa, Canada (2003).

Patents

S. Chang, M. Zhou and **C.P. Grover.** "Spectral coding by fluorescent semiconductor nanocrystals for document identification and security applications". Provision US Patent filed in 2003.

S. Chang, M. Zhou and **C.P. Grover.** "Object identification using quantum dots fluorescence allocated on Frauhofer solar spectral lines". Provisional US Patent filed in 2003.

M. Zhou, J. Roovers and **C.P. Grover.** "Dendritic polynuclear metal complexes as electrochemiluminescent labels for bioanalytic assays", Provisional Canada Patent filed in 2003.

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