Questionnaire on activities in radiometry and photometry

Reply from: National Research Council of Canada (NRC)

Delegate: Joanne Zwinkels

- 1. Summarize the progress in your laboratory in realizing top-level standards of:
 - (a) broad-band radiometric quantities
 - (b) spectral radiometric quantities

New Realization of NRC Spectral Irradiance Scale: NRC is upgrading to a source and detector based spectral irradiance scale ranging from 250 nm to 2500 nm. This scale realization incorporates a BB3500M high temperature blackbody as the primary standard source and wide-band filter radiometers that are traceable to the NRC cryogenic radiometer. Preliminary spectral irradiance measurements have been taken in the visible wavelength range. An extended InGaAs photodiode that will cover the longer wavelength range up to 2500 nm is presently being installed.

New Absolute Radiometry Facility: NRC is developing a new facility for the primary realization of optical radiant power. This facility incorporates a new primary instrument – a new cryogenic radiometer as well as a laser driven broad-band light source and double monochromator system for photodetector calibration. A customized motion platform, optical window and vacuum bellows system was designed to allow for the calibration of two transfer standard detectors using the cryogenic radiometer in one measurement cycle. The establishment of this facility is in the final stages, where the degree of equivalence between the new and existing optical power scales remains to be determined.

- (c) photometric quantities
- 2. What other work has taken place in your laboratory in scientific or technological areas relevant to the CCPR?

Pilot of CCPR-K3.2014: NRC is the pilot laboratory for the 2nd round of CCPR-K3 (luminous intensity) comparison. The measurements for the comparison have been completed. The measurement artifacts were prepared and measured at each NMI before shipment to the pilot, then measured by the pilot laboratory, and re-measured by the NMI when the lamps were returned to the NMI. Sixty-six lamps from the 12 participating laboratories were measured at the pilot laboratory. Two of the Pre-Draft-A Processes, Verification of reported results and Review of uncertainty budgets, have been completed. The third process, Review of relative data, is in progress.

LED Metrology facility: NRC is continuing the development of a measurement facility for solid state lighting (SSL). We have established a total spectral flux lamp standard from our total luminous flux lamps that has been used to prepare working standards of total spectral flux. Several SSL modules have been purchased and preliminary measurements have begun for the purpose of testing their suitability as luminous and spectral intensity standards. We have begun the control programming to enable the measurement of angular output characteristics of SSL modules using a small five-axis robot. Initial measurements have shown the feasibility of this project for the measurement of small modules.

Routine Spectral Responsivity Facility Upgrade: As part of our effort for continuous improvement of the radiometric measurement chain, an upgrade of the NRC Routine Spectral Responsivity Facility is being carried out to include electronics, computer hardware and software. The computer and software upgrades and validation have been completed. This upgrade has consolidated a number of instrument operation and data acquisition programs into one versatile piece of code that will be easier to maintain.

Single-Photon Detector Efficiency Capability: NRC is working towards the establishment of a single-photon detector metrology apparatus. The development of this capability is currently in the planning stage, with experiment design and preliminary measurements to be taken in 2017.

NRC Fluorescence Capabilities – Improved Traceability for Different Illumination and Viewing Measurement Geometries: The development, characterization and validation of a versatile reference goniospectrofluorimeter for traceable fluorescence measurements using different illumination and viewing geometries specified in practical colorimetry, has been completed for both a bidirectional (45:0) and a sphere (8:d) geometry. This work has been written up in a two part series of papers. The first paper reviews the background to this work and provides details of the basic design of the new instrument and its characterization for measurements using a bidirectional geometry (45a :0), including a representative uncertainty budget for the total radiance factor calibration of a fluorescent paper standard. For this type of fluorescent material it has been demonstrated that the relative expanded (k=2) uncertainty in the visible range is 0.8%, which compares very well with other state-of-the-art reference instruments for fluorescence measurements in this referee bidirectional geometry. The part 1 paper has been recently accepted for publication in Metrologia. The part 2 paper is close to completion with an ongoing analysis of the Type A uncertainty components for a sphere geometry (8 :d).

Upgrade of NRC Reference Spectrofluorimeter (45a :0 geometry): A significant upgrade of the >15 year old NRC Reference Spectrofluorimeter has been carried out. This involved a major upgrade of the electronics and the positioning components. The incorporation of automated positioning of the polarizer for the s- and p-polarized measurement runs has significantly reduced the need for operator intervention and improved the calibration volume throughput. The upgrade has also included the development of more user-friendly and versatile software for instrument operation and data analysis.

Status of CCPR K6. NRC participated in CCPR-K6:2010 comparison of regular spectral transmittance. The draft B report for this comparison has been submitted by the pilot lab (MSL) to CCPR WG-KC for review and approval with a deadline of 12 September 2016.

High Temperature Thermodynamic Fixed Points: NRC's contributed to the determination of the thermodynamic temperature of the inflection point of three high temperature fixed points as part of the Euramet *Implementing the new kelvin* project. These values have been published in *Phil. Trans. R. Soc. A*.

Raman Spectroscopy and Nanomaterial Characterization: Raman spectroscopy and nanomaterials optical properties activities continue to expand its capability. We are participating in the international measurement campaign (led by our colleagues in the Black Carbon Metrology group) on the study of Black Carbon emission from aviation sources. The results from the measurement campaign will be used to guide ICAO (International Civil Aviation Organization) in setting new guidelines for future aviation engines. We are also involved in a new initiative co-led by NPL and NIST in the review and development of practical Raman standards. We are currently participating in the pre-pilot interlaboratory comparison under CCQM- Surface Analysis working group. The first measurand being determined is the optical confocal volume in a microRaman spectrometer. We also continue to advance our material characterization capabilities. Specifically, we have studied the optical properties of plasmonic nanostructures, nanocarbon materials and quantum confined nanostructures.

- 3. What work in PR has been/will be terminated in your laboratory, if any, in the past /future few years? Please provide the name of the institution if it has been/will be substituted by a DI or accredited laboratory.
- 4. What are present, new or emerging needs of users of your services that are not being supported sufficiently by current CCPR activities or initiatives? In the light of this information please suggest desirable changes in the future working program of the CCPR.

Micro-spectrophotometry measurements covering the visible to the mid-IR are needed to better serve clients in the emerging nanotechnology, security, microelectronics, clean energy and biomedical industries. In particular, the emergence of the nanotechnology based optical sensors in all of the above mentioned sectors requires that the NRC photometry and radiometry laboratories extend its current measurement services to cover this area. It is desirable to have a way of comparing (and validating) these measurements with our standard spectrophotometric procedures.

5. What priorities do you suggest for new research and development programmes at NMIs in the area of Photometry and Radiometry?

- Spectral total flux: The emergence of novel light sources, particularly SSL, has resulted in increased demand for spectral total flux measurements of sources. These measurements require establishment of light sources whose spectral total flux output is known. We recommend research and development of light sources that are calibrated for spectral total flux that can be used with integrating spheres to calibrate our clients' light sources.
- **Spectral comparisons in the mid-IR:** Extension of the existing validation and intercomparison procedures for spectrophotometry in the mid-infrared would be of benefit to the energy, biotechnology, and nanotechnology fields. Parameters of interest are microspectrophotometric measurements of spectral regular transmittance and reflectance of nanomaterials in emerging applications, such as quantum dots.
- Extend spectral responsivity comparisons further in the NIR: With the completion of CCPR K2a, K2b and K2c comparisons, the spectral responsivity scales of NMIs will have been intercompared throughout the spectral range 200 nm to 1600 nm, using PtSi, Si, and InGaAs detectors. It would be useful for CCPR to organize an intercomparison in the spectral range 1500 nm to 2500 nm, using as artifacts either extended range InGaAs detectors, or liquid nitrogen cooled InSb detectors.
- Develop metrology capabilities for characterization of new nanostructures: There is an increasing need to evaluate the energy and efficiency of light emitted by semiconductor nanostructures such as quantum dots. New R&D programmes in nanophotonics wells. wires. and measurements will provide better understanding of the optical properties (spectrophotometry and/or Raman scattering) of these emerging nanomaterials. A high instrument sensitivity is required to quantify these parameters in single nanostructures. Ultimately, characterization of single photon emission will be required. Tools also need to be developed to perform this metrology on individual nanostructures. Comparison and validation of existing nanophotonics measurement techniques are well worth a broader scale of collaborations between NMIs. For example, emerging measurement techniques such as scanning probe enabled spectrophotometry measurements (tip enhanced Raman spectroscopy; tip enhanced Infrared absorption spectroscopy) and non-linear optics enabled super-resolution fluorescence microscopy are all techniques well worth developing.
- Develop new nanomaterial-based certified reference materials (CRMs): Research into new CRMs to serve the expanding industry sectors that adopt nanomaterials is another area that is worth pursuing. The CRMs would include metal nanoparticles (biomedical, catalysis, energy, security printing and sensors applications), nano-carbon materials and inorganic nanoparticles (quantum dots, metal oxides).
- 6. Are there any research projects where you might be looking for collaborators from other NMIs or are there studies that might be suitable for collaboration or coordination between NMIs?

We are interested in collaborating with other NMIs involved in the R&D areas of optical properties of novel nanomaterials and development of Raman spectroscopy standards.

- 7. Have you got any other information to place before the CCPR in advance of its next meeting?
- 8. Bibliography of radiometry and photometry papers of your laboratory since the last CCPR (September 2014)?

2016

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