Consultative Committee for Photometry and Radiometry (CCPR)

24th Meeting (19 - 20 September 2019)

Questionnaire on activities in radiometry and photometry

Inputs from: National Research Council Canada (NRC)

Delegate:

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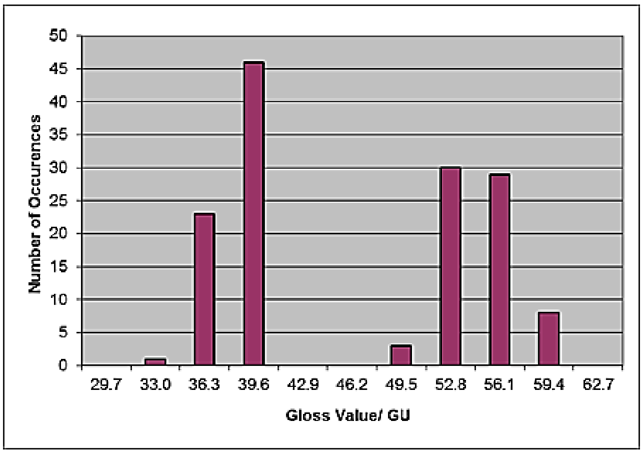
1. Summarize the progress in your laboratory in realizing top-level standards of:

   (a) broad-band radiometric quantities:

Gloss Study of the Impact of Converging and Collimated Beam Geometries
Very large inter-instrument differences have been reported in recent gloss comparison studies that greatly exceed both gloss manufacturer and test method specifications for repeatability and reproducibility. To understand the impact of differences in instrument beam geometries, we carried out a gloss comparison using the NRC reference goniospectrophotometer configured in both a converging and collimated beam geometry for measuring specular gloss of paper samples at 75° and 20° geometries. For the paper samples that were studied, it was found that for a given specular angle and beam geometry, the measurement reproducibility was very good, ranging from 0.6 -2.4 GU. However, for both 75° and 20° geometries, for a change in the beam condition, the measurement reproducibility significantly deteriorated. In the case of both the medium and high gloss coated paper samples measured at 75° geometry, the differences in the measured gloss values for the two different methods (6.3 –7.8 GU) were a factor of ~4 to 8 times larger than the measurement reproducibility for a given beam condition (0.7 -2.4 GU). In the case of the medium gloss paper sample (RG01) measured at 20° geometry, the NRC results showed a very large gloss difference of 23.4 GU in going from the converging (TAPPI) to the collimated (DIN) beam method compared with an excellent measurement reproducibility of 0.6-1.2 GU for a given beam condition. It is interesting to note that the NRC mean result for sample RG01 using these two methods (47.1 GU) is in very good agreement with the grand mean result of 44.9 GU that was reported for the CTS 20° TAPPI round-robin study using this glossy paper sample (14 participating labs, see Figure below). These results have been published in the Journal of Physics Conf. Series: **972**, 012025, 1-5 (2018).



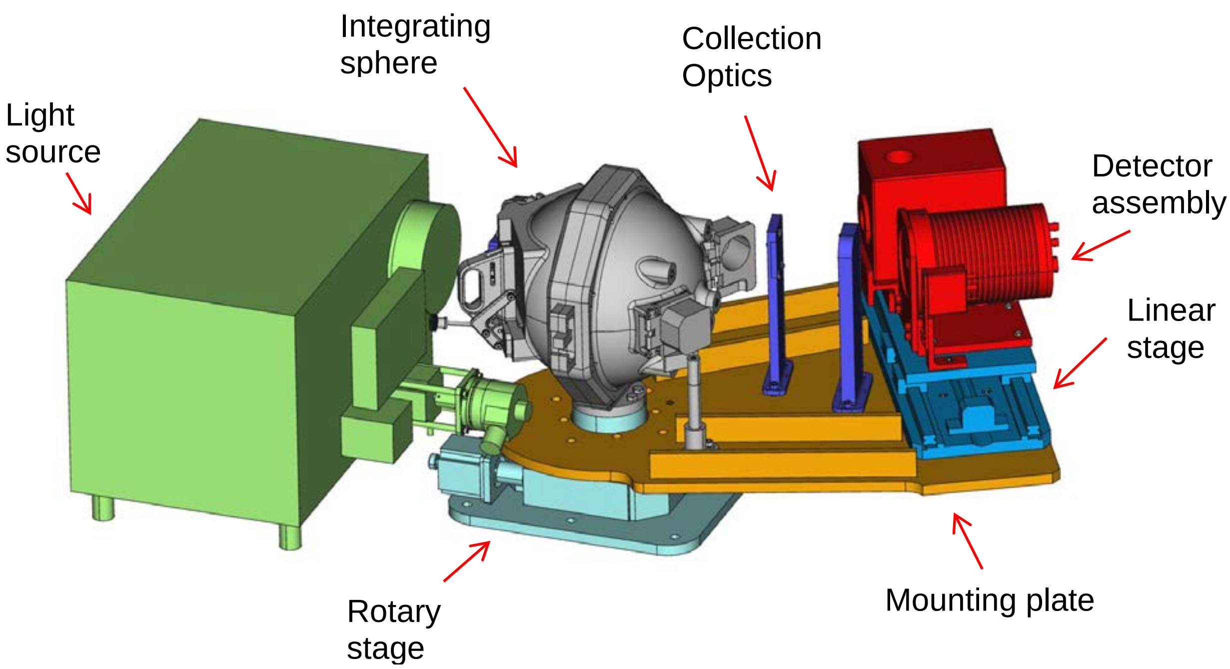
(b) Spectral radiometric quantities :

Extension of NRC Fluorescence Calibration Capabilities to a Sphere Geometry
NRC has new calibration and measurement capabilities (CMCs) for fluorescence calibrations in a sphere geometry with near-normal illumination (8 degrees) and diffuse viewing (specular component included or excluded) that have been published in the BIPM key comparison database (KCDB) on 2 February 2018. These calibrations are performed on the NRC Reference Goniospectrofluorimeter equipped with a 300 mm diameter integrating sphere accessory which, to the best of our knowledge, is the only reference instrument world-wide providing traceable calibrations of fluorescent reflecting materials in a sphere geometry. The design, characterization and performance validation of this instrument has been published in a 2-part series of papers in *Metrologia* (**54** (2017),129-140; **53** (2016), 1215-30). The instrument uses a continuum xenon source and provides absolute calibrations in both a bidirectional (45:0) and sphere (8:d, 8:t) geometries of both the reflected and luminescent radiance factor components over the spectral range 300 nm to 850 nm with a 5 nm spectral bandpass at 5 nm or 10 nm intervals for CIE standard illuminant or client-specified illuminant conditions.

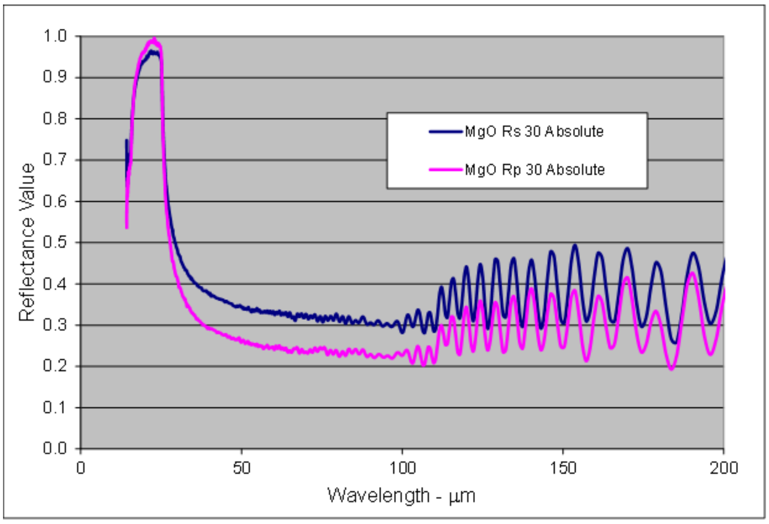
Reflectance Matrix Method for Improved Accuracies in Sphere-based Fluorescence Measurements and Derived Quantities (e.g. Absolute Quantum Yield)
It is well-known that integrating sphere-based fluorescence measurements are complicated by the interaction between sample and sphere, as this can modify the sample illumination and sphere throughput in a non-trivial way and lead to severe errors in quantities characterizing fluorescent materials such as absolute spectral quantum yield or colorimetric coordinates. Using the NRC Reference Goniospectrofluorimeter, we have recently completed a detailed study of this issue. A theory of integrating spheres with photoluminescent surfaces was developed and a reflectance matrix method for correcting for the effect of the sample on the illumination and sphere throughput was proposed and experimentally validated. It is hoped that this result will lead to improved accuracy in measurements of fluorescent materials and to a more complete understanding of this important source of uncertainty. More details can be found in *Optics Express* 27 423 2019.

NRC's New Absolute Diffuse Reflectance Scale

NRC is currently constructing a new monochromator-based absolute reflectometer. This new reference instrument is based on the integrating sphere Sharp-Little method to realize an absolute reflectance scale in a d:0 geometry across the UV-VIS-NIR spectral range. The new system will feature an extended spectral range (250 to 1900 nm), full automation, and improved spectral resolution compared with the existing NRC absolute reflectometer. A custom PTFE integrating sphere has been constructed in-house and the main components of the system were in place by June 2018. The system is currently operational and preliminary reflectance data has been collected in the visible spectral range. Present activities are focussed on validating the new instrument in comparison with NRC's existing absolute diffuse reflectance scale and on developing a detailed uncertainty budget that includes characterization of various integrating sphere asymmetry and non-ideality effects, such as the influence of finite port thickness, sphere coating inhomogeneity and baffle design. It is planned to use this new absolute reflectometer for NRC's participation in the upcoming 2nd round CCPR K5 comparison. More details can be found in : L.J. Sandilands, E. Côté, and J.C. Zwinkels, "Design and characterization of a new absolute diffuse reflectance reference instrument at the NRC." In *Reflection, Scattering, and Diffraction from Surfaces VI* (Vol. 10750, p. 107500G). SPIE 2018.



New NRC Calibration Services for Absolute Specular Reflectance in the IR Range
Complementing the long-established NRC absolute specular reflectance capabilities in the UV-visible-NIR region, we have extended these capabilities to the IR from 16 to 200 micrometers for selectable angles of incidence. These absolute measurements are performed using an IR reflectometer based upon the VW method to calibrate the absolute specular reflectance of an NRC aluminized reference mirror for the desired angle of incidence which is used, in turn, to calibrate client samples. This method has also been applied to our own metal oxide samples of Mgo, Mno and NiO. Representative results are shown below for the MgO sample measured for an angle of incidence of 30 degrees. The large peak in reflectance that is observed at the short wavelength side is due to the LO and TO optic phonons in MgO. An analysis of this range in collaboration with Professor Guolin Yu of the Shanghai Institute for Infrared Physics has provided more accurate values for the frequency and damping for the phonon modes and other dielectric material properties for MgO. The oscillations to longer wavelength are due to interference effects in the approximately 1 mm thick plane parallel sample.



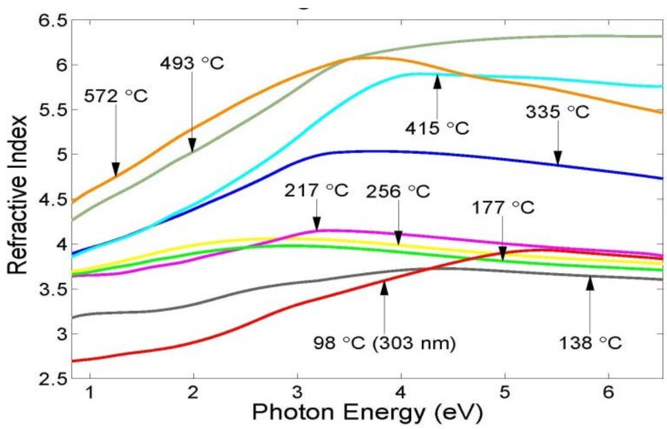
Absolute specular reflectance of MgO in the IR region (16 to 200 micrometres) for an angle of incidence of 30 degrees (s- and p-polarized components).

New Primary Realization of NRC Spectral Irradiance Scale:
NRC has upgraded 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 a wide-band filter radiometer that is traceable to the NRC cryogenic radiometer. This new scale was used in NRC's CCPR-K1a.2017 spectral irradiance key comparison measurements.

New Absolute Radiometry Facility
NRC has developed a new facility for the primary realization of optical radiant power. This facility incorporates a new cryogenic radiometer as well as a laser driven broad-band light source, tungsten-halogen light source and a double-subtractive monochromator system for photodetector calibration. This facility was utilized to calibrate NRC transfer-standard radiometers for NRC's CCPR-K2b.2016 and CCPR-K2a.2016 spectral responsivity key comparison measurements

Recent Advances in Nanomaterials Optical Properties

In collaboration with University researchers (UBC and University of Regina), NRC has carried out spectral regular reflectance and transmittance measurements of thin silicon films grown at temperatures ranging from 98 °C to 572 °C by ultra-high-vacuum evaporation on fused quartz substrates. These data have been analyzed to determine how the spectral dependence of the optical functions (refractive index and extinction coefficient) of these films is affected by the growth temperature. In particular, the extinction coefficient decreases as the growth temperature increases, whereas the refractive index generally decreases. These results are important for optimizing growth conditions for silicon film-based device applications requiring a high refractive index.



Spectral dependence of the refractive index of silicon thin-films as a function of growth temperatures ranging from 98 °C to 572 °C.

In collaboration with Sichuan University (China), NRC (Nelson Rowell) has been carrying out analysis of the UV optical absorption and photoluminescence data of their novel II-VI semiconductor clusters and quantum dots. In particular, the effect of an isomorphic change in particle shape, such as from spherical to cylindrical, has been investigated on the bandgap energy, i.e. where the absorption peaks. This collaboration has resulted in several recent joint publications (see bibliography of NRC photometry and radiometry papers below to Question 8)

Raman Spectroscopy of Black Carbon: NRC has carried out Raman spectroscopic analysis of black carbon (BC) and various nanocarbon analysis. The study was aimed to identify suitable surrogate BC materials in order to improve the measurement agreement for the BC mass concentration between various commercial optical instrumentations. Raman spectroscopy identified unique spectral feature from BC nanoparticles generated under fuel-rich and fuel lean conditions. Spectroscopic analysis was then used to guide the laboratory synthesis of BC nanoparticles in order to achieve suitable surrogate nanoparticles for optical instrument calibration. These optical instrumentation are used in the measurements of BC emissions from civil aviation aircraft gas turbine engines. This is a collaborative project with Transport Canada, Rolls-Royce and EMPA (Swiss Federal Laboratories for Materials Science and Technology).

(c) photometric quantities :

2. What other work has taken place in your laboratory in scientific or technological areas relevant to the CCPR?

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 a new monochromator, data acquisition electronics, and computer software. This new system is currently undergoing validation.

Few-Photon Metrology Capability: NRC is establishing a quantum metrology capability for optical radiometry. A free-space single-photon detector calibration system for silicon single-photon avalanche diode efficiency measurements has been established. In collaboration with NIST, a NIST-designed portable optical fibre-coupled superconducting nanowire single-photon detector (SNSPD) system was assembled and tested at NIST Boulder by both NRC and NIST researchers and has been transported back to NRC. This system will be used as standard fibre-coupled single-photon detectors as well as for other measurements. Presently, NRC-developed solid-state on-demand single-photon sources based on III-V semiconductor quantum dots are being implemented in metrology applications including investigating the possibility of using these sources as absolute standard single-photon sources and in the development and standardization of single-photon source performance metrics (i.e. source efficiency, single-photon purity, etc.) and measurement procedures.

Solid-state Lighting Standards Development: NRC is testing various solid-state modules to be used as working standards of total luminous flux and luminous intensity. Several SSL sources available on the market have been purchased with different optical and geometrical properties. Each SSL module has been through aging process in a controlled environment for long periods of time. Luminous output of each source has been analyzed as function of electrical current and ambient temperature. Spectrum of the SSL module is also recorded over the time to monitor the spectral variations of the source. SSL modules with smooth and predictable behavior over periods longer than 1000 hours are chosen as suitable candidates for working standards. Further seasoning of the lamps is in progress and gonio-measurements will be performed to test the geometrical properties of the modules.

Pilot of CCPR-K3.2014: NRC is the pilot laboratory for the 2nd round of the CCPR-K3 (luminous intensity) comparison. The measurements and initial data analysis for the comparison have been completed. The measurement artifacts (incandescent lamps) 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. The pre-Draft-A processes have been carried out to determine lamp stability, statistical outliers, and statistical consistency of the results. Of the 70 incandescent lamps that were received at the pilot, 62 will be used in the comparison. The first version of Draft A has been completed and is with all the participants for their review and comments.

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.

   None

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.
   - CCPR-K2.c was last carried out in 2004 – 2007 and CCPR should consider a repeat of this comparison.

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

- **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.
- **Develop new light sources for photometric standards**. With the rapid demise in the production of incandescent light sources, and the emergence of SSL sources, the development of light sources suitable for working standards and travelling artifact standards for photometric measurements, especially luminous intensity and total luminous flux, is now of high priority.
- **Spectral total flux:** The emergence of SSL sources 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.

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 research into single-photons sources and single-photon source performance metrics.
    - We are interested in collaborating with other NMIs involved in the R&D areas of optical properties of novel nanomaterials
    - We are interested in collaborating with other NMIs involved in validating new colour and appearance measurement scales, e.g. transmittance and reflectance haze, sparkle, etc.
    - We are interested in measurement comparisons with other NMIs involved in high-accuracy quantum efficiency measurements of both solid and liquid fluorescent materials

7. Have you got any other information to place before the CCPR in advance of its next meeting?

    N/A

8. Bibliography of radiometry and photometry papers of your laboratory since the last CCPR (September 2016)?

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