THEMATIC NETWORK FOR ULTRAVIOLET MEASUREMENTS

Working Group 2: Improvement of measurement and calibration methods for spectrally resolved UV measurements

Improvement of measurement and calibration methods for spectrally resolved UV measurements

J. Metzdorf, W. Möller, and S. Galal Yousef
PTB, Germany

Final Report
November 2000

CONTENTS

ACKNOWLEDGEMENTS

Members of Working Group 2

J.Metzdorf (Chairman)

I. Abu-Kassem
K. Andersson
S. Anevski
A. Bais
Y. Barnes
M. Blumthaler
B. Carlsson
A. Corrons
K. Dehne
R. Distl
M. Durak
N. Fox
S. Galal Yousef
X. Gan
J. Gran
C. Gross
W. Halbritter
G. Hammond
T. Hanken
N. Harrison
W. Hartree
S. Hartwell
B. Heits
T. Hunt
E. Ikonen
D. Irish
K. Jokela
W. Jordan
W. Josefsson
P. Kärhä
R. Köhler
H. König
T. Koskela
T. Kübarsepp
L. Liedquist
D. Lindner
A. Los
M.Misslbeck
W. Möller
K. Möstl
R. Morley
J. Müller
T. Nägele
A.Page
J.C. Petersen
J. Pietzrzykowski
H. Rabus
H. Reinen
H. Sandmann
G. Sauter
C. Schrama
O. Schrems
H. Seidlitz
P. S. Shaw
M. Smid
P. Sperfeld
A. Sperling
M. Steinmetz
T. Svenoe
R.Tax
S. Thiel
P. Toivanen
H. Tüg
G. Ulm
P. von Nandelstadh
A. Webb
L. Werner
S. Winter
O. Wolf
L. Yliantilla
H. Yoon

Special acknowledgement for contributions

N. Fox
C. Groß
W. Halbritter
N.J. Harrison
E. Ikonen
W. Jordan
F. Manoocheri
S. Nevas
P. Kärhä
T. Koskela
A. Krins
T. Kübarsepp
A.J. Page
H. Rabus
H. K. Seidlitz
M. Steinmetz
W. Steudtner
S. Winter

1 Introduction

Spectrally resolved UV measurement are carried out

(i) to calibrate detectors, spectroradiometers, lamps (sources), reflection standards and filters as well as

(ii) to characterise and test detectors, radiators, optical instruments, components, materials and surfaces.

In the classification of measurement and calibration methods including QA/QC requirements, it may be useful to distinguish:

- characterisation and testing based on relative measurements (e.g. measurement of the angular response of a diffuser to determine cosine error);
- absolute calibration requiring traceability to SI units and national standards and (recognition of the) equivalence of the standards for the respective units and scales (e. g. calibration of a lamp as a standard of spectral irradiance).

It was found to be more challenging to improve the (absolute) calibration methods which are combined with more pressing problems.

The objectives of Working Group 2 (WG2) have been defined during the first meeting based also on the replies to the first questionnaire:

- To determine the most important user requirements for spectrally resolved calibrations of standards and characterisations of instruments and components in the field of non-coherent UV radiometry and spectrometry.
- To stimulate and promote the improvement and development of measurement techniques and exchange of experiences based on the respective needs and problems as well as on the state-of-the-art methods, devices and instruments identified before.
- To prepare and publish examples of the expression of the uncertainty of measurement in UV radiometric calibrations based on the documents *EAL-R2* and *EAL-R2-S1* and in compliance with the recommendation of the *ISO Guide to the Expression of Uncertainty in Measurement*. The goal is to demonstrate the method of evaluating the uncertainty of measurement in spectroradiometry, to support the reduction of the too large uncertainties in UV radiometry and to facilitate the evaluation of realistic reported uncertainties.

It was evident from the very beginning that it is not the objective of WG2 to produce a special document (e.g. draft of a written standard) but to report on results obtained and projects carried out by WG2 members individually or in co-operation.

Although UV dosimetry (irradiance of more than about 10 $mWcm^{-2}$; see also EUROMET project 437; EC project SMT4-CT98-2242 "Improving the accuracy of UV radiation measurement," WP3: Improving industrial measurements) and UV laser radiometry (see however: [M. Rahe, K. Mann; "High Power UV Lasers and UV Measurement Techniques for Excimer Laser (157 nm - 351 nm) – State of the Art and Future Developments," *UVNews* **3**, 6 (July 1999)] is of increasing importance, these two fields were excluded because only very few working group members were involved.

It was generally accepted and confirmed that the whole area covered by the general subject of WG2 is extremely large. Thus, based on the replies to the questionnaires, the discussions at three WG2 meetings and the contributions made by individual members representing industrial calibration laboratories, testing institutes, National Metrological Institutes (NMIs) and solar UV monitoring organisations, it was decided to concentrate on the identified two main areas (1. Calibration problems; 2. Uncertainty evaluation) and specific subjects within these areas, especially concerning measuring methods and evaluation of results. The following two subjects were recognised to be important as well but to be postponed to future activities and exchange of information (Continuation of the UV network and WG2 activities ≥ 2001):

(i) Measurement of the transmittance of UV filters considering collection of knowledge and experience.

(ii) Interpolation of spectral data and associated additional uncertainty also making use of physical models for the interpolation of the relative spectral distribution (e.g., relative spectral irradiance of a blackbody source with adjustable distribution or ratio temperature).

In view of our subject and program "Improvement of measurement and calibration methods for spectrally resolved UV measurements," the many hardware problems due to inadequate performance and/or lack of standards and instruments/components increasing the uncertainty and affecting the use of calibration methods have not been treated in detail but are only summarised in the following table. In this matrix, critical parameters and features of the respective devices are specified.

At present stability and reproducibility of UV standard lamps are the most pressing and challenging problems in UV radiometry. As new developments and improvements in UV detectors are the main subjects of the EC project SMT4-CT98-2242, it is also referred to the report of this project as far as detectors are concerned. Test of different materials suitable to be used as UV reflection standards and development of improved methods for the calibration of diffuse reflection standards in the UV is well underway at several NMIs; however, positive results will take some time.

The development and improvement of more stable UV filters is becoming more and more important. Nevertheless, only two filter types may be mentioned here:

(i) Filter materials to be used as stable broadband/cut-off UV filters in solar radiation simulators (see Appendix 1);

(ii) UV interference filters to be used in filter radiometers (FWHM < 2 nm) have been developed at the Fraunhofer Institute IOF in Jena, Germany, where new techniques for manufacturing stable interference systems by plasma ion-assisted deposition have been demonstrated to provide long-term stable UV-B narrow-band filters with excellent optical parameters. Contact: Norbert Kaiser, Fraunhofer IOF, e-mail: Kaiser@iof.fhg.de (see also: [H. Uhlig, N. Kaiser; "Improved narrowband filters for the UV-B region," *Appl. Opt.* (submitted)]).

Table 1. Problems due to inadequate performance and/or lack of standards and instruments/components increasing the uncertainties and affecting the use of calibration methods.

	sources	detectors	filters refl. stand.	spectrora- diometers
stability (ageing)	x[a)]	x	x	x
reproducibility	x			x
temp. dependence		x	x	x
linearity		x		x
spatial uniformity		x	x	
wavelength dependence	x	x[b)]	x[b)]	x
signal level (<250nm)	x			x
robustness (transport)	x			x
wavelength alignment				x
cosine approximation		x	x	x
dynamic range		x		x
contamination[c)]		x	x	x
application of tuneable UV lasers [d)]		x	x	x
(costs)	x			x

a) short- and long-term stability, detector monitoring or stabilisation, prediction of stability?
b) out-of-band blocking, fluorescence, spectral distribution with pronounced structures
c) sometimes combined with fluorescence
d) for laser-based calibration and characterisation

2 Calibration Problems

Five specific subjects were selected in this area.

2.1 Equivalent and consistent radiometric scales

It has been identified that (i) differences between the UV spectral irradiance scales realised and disseminated by different NMIs and (ii) ageing and inadequate reproducibility of the respective standard lamps are the most critical problems in UV radiometry. This experience is based on several internal comparisons of standard lamps calibrated by different NMIs and by the "Results of an intercomparison of UV source measurement scales" carried out in 1993/94 (see: Working document CCPR/97-1 submitted to the 14th meeting of the CCPR in 1997; see also: [J. H. Walker, R. D. Saunders, J. K. Jackson, K. D. Mielenz, "Results of a CCPR Intercomparison of Spectral Irradiance Measurements by National Laboratories," *J. Res. Natl. Inst. Stand. Technol.* **96**, 647 (1991)].

Two bilateral intercomparisons have also been performed (1995 to 1998) between NIST and PTB (250 nm to 450 nm; relative difference -2 % to ± 0 %) and between NPL and PTB (200 nm to 360 nm; relative difference 4 % to 6 % below 320 nm decreasing to ± 1 % above 320 nm); results are presented by W. Moeller in the PTB's annual report 1998 (Jahresbericht'98, 2.4.5).

New CCPR key comparisons are currently underway and are in preparation, respectively, to establish a world-wide reference scale (reference values at discrete wavelengths) and to find out whether the uncertainties and the deviations between different scales could be reduced (see Appendix 2.1 Status report of UV radiometric CCPR key comparisons; see also the BIPM key comparison database http://www.bipm.fr/kcdb). The key comparison CCPR-K1.b "Spectral Irradiance 200 nm – 400 nm" is planned to be carried out in 2001/02 with the PTB acting as the pilot laboratory. However, before starting the measurements, more stable or at least reproducible transfer standard lamps have to be available (see below).

In addition, typical results illustrating the uncertainty of the irradiance scale at the user's site based on standard lamps calibrated at different NMIs and calibration laboratories are presented and discussed in a short report by T. Koskela (see Appendix 2.2).

The differences between spectral responsivity scales are much less even in the UV spectral range, which has clearly been verified recently by bilateral intercomparisons between different NMIs (see Appendix 2.3, contribution by H. Rabus).

In connection with the inadequate equivalence of the UV spectral irradiance scales, the question arises how to reduce the uncertainty of the respective scales and improve the stability of the standard lamps of UV spectral irradiance?

One task is to improve the traceability chain from cryogenic radiometer via the radiometric measurement of the blackbody temperature using (broadband) filter radiometers (see, e.g. [P. Sperfeld, J. Metzdorf, S. Galal Yousef, K. D. Stock, W. Moeller, "Improvement and extension of the blackbody-based spectral irradiance scale," *Metrologia* **35**, 267 (1998)]). Further work and reduction of uncertainty is currently underway at several NMIs, especially at the NIST, NPL, VNIIOFI and PTB.

Another task is to reduce the uncertainty of the spectral irradiance calibration of UV standard lamps. Thus, a UV-optimised spectroradiometer has been developed and is operated increasing optical throughput and signal-to-noise ratio (e.g. avoiding mirrors/reflections with the exception of the two gratings themselves) and reducing stray radiation using a solar-blind photomultiplier [W. Moeller, P. Sperfeld, B. Nawo, K. Hube, J. Metzdorf, "Realization of the spectral irradiance scale in the air UV using thermal radiators," *Metrologia* **35**, 261(1998)]. A new, upgraded UV spectroradiometer will be used at the PTB to carry out the respective key comparison CCPR-K1.b.

Finally, the stability of the UV standard lamps have to be improved. A special FEL type 1 kW Osram Sylvania tungsten halogen lamp has been selected and optimised as an improved standard of spectral irradiance above 250 nm [A. Sperling, S. Winter, K.-H. Raatz, J. Metzdorf, "Entwicklung von Normallampen für das UV-B-Mess-programm," PTB report PTB-Opt-52, 1996, 53 p.] including some best practice instructions; see also in Appendix 2.4, report of N. J. Harrison). The most critical disadvantage of this FEL type lamp is the sensitivity to shock and vibrations during transport. A further improvement is achievable by using detector-stabilised irradiance sources (see Appendix 2.4, report of N. J. Harrison and references therein).

The improvement of deuterium lamps to be used below 350 nm is much more difficult to achieve (see also Appendix 2.7, report by A. J. Page), where the ageing is strongly dependent on wavelength (extensively studied, e.g. by W. Möller). OMTec has developed and manufactured a special elaborate detector-stabilised deuterium lamp system used internally at the PTB, which is no longer commercially available. Therefore, special deuterium lamps with SiC monitor detectors are currently being tested whether they are suitable to be used as transfer and travelling standards of UV spectral irradiance, especially in the respective key comparison CCPR-K1.b.

2.2 Pulsed UV source spectroradiometry

Spectrally resolved measurements of pulsed UV sources require the application of array spectroradiometers. Although several WG2 members showed interest in this subject, only one contribution was submitted for the calibration of pulsed UV sources combined with the identification of industrial applications of pulsed UV radiation and types of UV sources used. (see Appendix 2.5, contribution by A.J. Page, Cathodeon Ltd.).

2.3 Total UV spectral radiant power measurement

The calibration of UV standard lamps of total (i.e. 4π) spectral radiant power in the spectral range between 200 nm and 400 nm is becoming more important for industrial calibration and testing laboratories. In principle, both integrating sphere and goniospectroradiometric methods may be applied for these calibrations. However, the strong wavelength dependence of most coatings of integrating spheres in the UV as well as ageing and fluorescence (and contamination) effects may induce large errors (requiring corrections) and uncertainties. Thus, only expensive gonioradiometric methods allow the determination of both the integral (4π) quantities and their angular distribution and can be recommended without restrictions. The present goniophotometer (diameter 5 m) in the Photometry Section of the PTB can be equipped with specific filter radiometers or with an array spectroradiometer to carry out special gonioradiometric measurements/calibrations in the UV. In future, the roboter-based goniophotometer/-spectroradiometer in the new building of the Optics Division currently under construction will be better suited for goniospectroradiometric calibrations in the UV.

In addition to the sophisticated calibration method at an NMI, Appendix 2.6 illustrates the procedure and calibration chain how to calibrate standards of total spectral radiant power in an industrial laboratory in order to meet the following requirements:

- preparation and dissemination of reference material (lamps) to different measurement labs (QC and R&D) ensuring traceability and comparability;
- testing of prototype lamps during development processes (milestones);
- testing of typical lamps (mean of production lines) for the determination of typical radiation quantities provided as customer information.

The total UV spectral radiant power is the commonly used and generally accepted measurand for an overall lamp characterisation, where, in addition, biological risk assessment, energy budgets and efficiency calculations may be required.

2.4 Calibration of outdoor UV spectroradiometers

In the outdoor radiometric calibration of UV spectroradiometers for field measurements (e.g. for solar UV-B monitoring), portable calibrators or monitor radiators and, in general, the transfer from horizontal to vertical optical axis is required. The typical indoor calibration against FEL type standard lamps of spectral irradiance is restricted to be used with horizontal optical axis (see also Section 2.1).

A portable monitor radiator consisting of a medium-power tungsten-halogen lamp (e.g. 200 W Osram lamp HLX64382) in a closed housing with fan cooling by air has been developed and tested. Ageing within less than $\pm 10^{-4}$ h^{-1} of the seasoned lamp has been observed by current-stabilised operation over more than 1500 h operation time. Optional detector-stabilised operation (developed by OMTec) reduces the seasoning time and especially the total ageing to less than 0,5 % over 2000 h.

This portable radiator can be used to (re-)calibrate spectroradiometers under indoor or outdoor conditions with horizontal or vertical axis. The option with detector stabilisation (using a temperature-stabilised SiC photodiode) can also be applied to the calibration transfer from horizontal to vertical optical axis (wavelength-dependent change of spectral irradiance within 0,3 % in the UV that can further be minimised combined with a change of the electrical power of the lamp of opposite sign), because the responsivity of the SiC photodiode is independent of its spatial orientation.

A second procedure for the calibration transfer from horizontal to vertical optical axis including again a monitoring of the stability of the spectroradiometer is based on the application of a flexible optical fibre between spectroradiometer entrance (slit) and diffuser where the latter is additionally equipped with blue temperature-controlled GaN monitor LEDs in the (modified) diffuser head (drift/ageing typically better than $3 \cdot 10^{-5}$ h, independent of orientation). These optoelectronic semiconductor devices are again not sensitive to their spatial orientation. All the above results will be published as a PTB report

[T. Wittchen, K. Liegmann, P. Sperfeld, J. Metzdorf, *Development of calibration and testing procedures for UV spectroradiometers*, PTB-Opt-xx, 2001].

Finally, 1 kW DXW type tungsten-halogen lamps that can be operated with both horizontal and vertical optical axis can routinely be calibrated against FEL type standard lamps (only horizontal optical axis) including a horizontal/vertical transfer. In this case, a 45° plane mirror is rotated around the (horizontal) optical axis of the reflected beam (directed to the diffuser entrance of the spectroradiometer) switching between horizontal (FEL type lamp) and vertical (DXW type lamp) incident beam.

2.5 Work instruction, best practice instruction for the calibration and operation of radiometric UV standards

Two examples (i) for the operation of deuterium lamps as standard lamps for UV spectroradiometry including the improvement of reproducibility (switch-on repeatability) and (ii) for the calibration of standard lamps of spectral irradiance considering also QC/QA requirements, operation and handling of a UV standard lamp (see also Appendix 3.1 concerning uncertainty analysis) are presented in Appendices 2.7 and 2.8. Several WG2 members had expressed the opinion that such examples of instructions are very useful for the practical work of the technical personnel of calibration laboratories. The report on deuterium lamps should also be very useful in connection with the detector stabilisation of a deuterium lamp system.

3 Uncertainty Evaluation

Prerequisite for the desirable reduction of the uncertainty of spectrally resolved UV measurements and calibrations is the realistic and consistent evaluation of the uncertainty of measurement, as completely as possible. It is required, moreover, that the uncertainty budget is in compliance with the respective *ISO guide to the Expression of Uncertainty in Measurement* (GUM) and in accordance with EAL Publication *EAL-R2*. Only after consistently identifying the most critical uncertainty contributions and sources, investigations and measures to reduce the (standard) uncertainty associated with the measurement result are useful and promising.

Appendix 3.1 and the reference therein give an example for the evaluation of the uncertainty of measurement for the (indoor) calibration of a lamp against a standard of spectral irradiance. It is important and not trivial to take into account the uncertainties associated with all relevant corrections in addition to those associated with the quantities themselves involved in the calibration equation (in the mathematical model).

Similarly, the uncertainty budget of detector-based spectral irradiance measurements is described and discussed in Appendix 3.2

The evaluation of an uncertainty budget for the calibration of detectors based on the technical protocol of the respective key comparison CCPR-K2.c “Spectral responsivity 100 nm – 400 nm” is presented in Appendix 3.3, where calibrations against different standards have been distinguished.

Monitoring of the solar UV radiation is a very challenging and difficult task requiring extensive uncertainty analysis of the respective UV spectroradiometers and their (re-)calibration. Many of these QC/QA requirements are covered by WMO activities (see Appendix 3.4).

It is well known that it may happen very easily to forget or underestimate an uncertainty contribution in the measurement of a radiometric quantity or in the calibration of a radiometric standard. In addition, it may be helpful to present the following list of

Principal and common sources of uncertainty contributions to be always considered for an interlaboratory comparison

a) by participating lab.:
   - realisation of SI unit (primary standard) or reference standard/value with assigned uncertainty
   - maintenance of unit (drift of reference standard(s) at participating laboratories)
   - transfer/dissemination to travelling/transfer standard(s)
   - standard deviation of the mean of the group of transfer standards

b) by pilot lab.:

- measurement/dissemination at the pilot lab. during intercomparison (repeatability or reproducibility and maintenance depending on type of comparison, i.e. “star-like” or “round robin”)

c) for travelling standards/artefacts:

- change and drift due to transportation and instability during intercomparison (differences between first and final calibration).

4 Conclusions

In the improvement of measurement and calibration methods for spectrally resolved UV measurements, the main goal was how to reduce, as well as how to completely and consistently evaluate, the respective uncertainties of measurement. Especially, the achievable uncertainties of spectral irradiance calibrations combined with inadequate stability of UV standard lamps are not at all satisfying. The large differences between standards of UV spectral irradiance calibrated by different NMIs cause big problems both for industrial laboratories and for institutes involved in solar UV monitoring. However, the users of these calibrated standard lamps have to wait for the results of the respective key comparisons presently carried out above 250 nm with NPL acting as pilot laboratory. The range between 200 nm and 400 nm will be covered by a future key comparison with the PTB acting as pilot laboratory. However, improvements of a calibration chain from black-body source to different working standards of spectral irradiance are underway in order to reduce uncertainties and improve (speed up) calibration procedures.

The uncertainty due to the interpolation of spectral data is of importance in filter radiometry and if weighting functions and action spectra are applied. Thus, work is underway also in the fields of photometry and solar cell calibration to analyse the uncertainties of weighted spectra based on measurements at a limited number of discrete wavelengths. Moreover, a CCPR working group has been established in this field (contact: R. Köhler, BIPM).

Finally, it was unanimously decided during the last WG2 meeting in September 2000 to continue WG2 work after 2000 as a platform where to exchange information and agree upon co-operation and mutual assistance.