EUROMET.PR-K6.1
Bilateral Key Comparison
(EUROMET project 766)

Spectral Regular Transmittance

Technical Protocol

V1.1 2008-03-04

1. Introduction

1.1 The metrological equivalence of national measurement standards will be determined by a set of key comparisons chosen and organised by the Consultative Committees of the CIPM working closely with the Regional Metrology Organisations (RMOs).

1.2 In order to link to the CIPM key comparisons the laboratories which are not participating in the key comparison organised by the Consultative Committee of the CIPM it has been decided to organise the same comparisons within the Regional Metrology Organisations.

1.3 At its meeting in March 1997, the Consultative Committee for Photometry and radiometry, CCPR, identified several key comparisons in the field of optical radiation metrology. In particular, it decided that a key comparison of spectral regular transmittance shall be carried out, with the BNM-INM (France) acting as pilot laboratory.

1.4 The EUROMET project 538 " Comparison of spectral regular transmittance" in charge of doing the link between the European laboratories and the CCPR key comparison has been started in April 1999 with the BNM-INM (France) acting as pilot laboratory. It was decided to use exactly the same technical protocol as for the CCPR key comparison. The Bureau of Measures and Precious Metals (ZMDM) was at that time unable to join this comparison. In order to established the metrological equivalence of this laboratory, a bilateral key comparison was organised in the EUROMET project 766 with also the BNM-INM acting as pilot laboratory and the exactly the same technical protocol as for CCPR and EUROMET key comparison was used.

1.5 This technical protocol has been drawn up by a small working group comprising the Institut National de Metrologie, France (BNM-INM), the Helsinki University of Technology, Finland (HUT), the Measurement Standard Laboratory, New Zealand (MSL), the National Institute of Standard and Technology, USA (NIST), the National Physical Laboratory, UK (NPL), and the Physikalisch-Technische Bundesanstalt, Germany (PTB).

1.6 The procedures outlined in this document cover the technical procedure to be followed during measurement of the transfer standard filters. The procedure, which followed the guidelines established by the BIPM[1], is based on current best practise in the use of

[1] T.J. Quinn, « Guidelines for key comparison carried out by Consultative Committees », BIPM, Paris, 1 March 1999

standard filters and takes account of the experience gained from the previous comparisons organised in this field[2].

2. Organisation

2.1 Participants

2.1.1 The information related to the two participants are given in the table below.

2.1.2 The participants must be able to demonstrate independent traceability to the realisation of the quantity, or make clear the route of traceability to the quantity via another named laboratory.

2.1.3 By their declared intention to participate in this key comparison, the laboratories accept the general instructions and the technical protocols written down in this document and commit themselves to follow the procedures strictly.

2.1.4 Once the protocol and list of participants has been agreed, no change to the protocol or list of participants may be made without prior agreement of all participants.

2.2 Participants' details

BNM-INM	Mr Jean Bastie BNM-INM/CNAM 292 rue Saint Martin 75003 Paris France	Phone : 33 1 40 27 20 25 Fax : 33 1 42 71 37 36 E-mail : bastie@cnam.fr
ZMDM	Mr. Predrag Vukadin Bureau of Measures and Precious Metals Mike Alasa 14 11000 Beograd Serbia	Phone : + 381-11-32 82 736 Fax : + 381-11-21 81 668 E-mail : vukadin@szmdm.sv.gov.yu

2.3 Form of comparison

2.3.1 The comparison will principally be carried out through the calibration group of transfer standard filters.

2.3.2 A full description of the transfer standard filters is given in section 3 of this protocol.

2.3.3 The artefacts (filters) will initially be calibrated by the pilot laboratory. They will then distributed to the participant who will perform the calibration. They will be returned to the pilot laboratory to carry out a repeat calibration to check the stability.

[2] K.L. Eckerle, J. Bastie, J. Zwinkels, V. Sapritsky, A. Ulyanov, « Comparison of regular transmittance scales of four national standardizing laboratories », Color research and application, volume 18, number 1, February 1993.
J.F. Verrill, « Intercomparison of spectrophotometric measurements of regular transmittance », Report contract N°. MAT1-CT940021, November 1996.
Etc…….

2.3.4 BNM-INM will act as the pilot laboratory. All results are to be communicated directly to the pilot laboratory as soon as possible and certainly within 6 weeks of completion of the measurements by a laboratory.

2.3.5 The laboratory has 3 months for calibration and transportation.

2.3.6 If for some reasons, the measurement facility is not ready or customs clearance takes too much time, the participant laboratory must contact the pilot laboratory immediately.

2.3.7 Timetable

Activity	**Date**
start of the EUROMET project 766	April 2003
Participants filters received	October 2003
Filters returned to BNM	January 2004
End of measurements	June 2004
Draft A comparison report	after EUROMET-PR-K6
Draft B report submitted to EUROMET	after EUROMET-PR-K6

2.4 Handling the artefacts

2.4.1 The standard filters should be examined immediately upon receipt at final destination. The condition of the filters and associated packaging should be noted and communicated to the pilot laboratory. Please use the fax form or e-mail form in appendix A4 and A5.

2.4.2 The standard filters should only be handles by authorised persons and stored in a such way as to prevent damage.

2.4.3 No cleaning of any filter should be normally done. Dust could be removed with a very soft brush or with a stream of dry nitrogen or dry CO_2. In case of accidental pollution cleaning will be made with alcohol and special optical paper. Cleaning must be indicated in the measurement report and documented using the appropriate form in appendix A.2. If a filter appears damaged a replacement will be available from the pilot laboratory.

2.4.4 After the measurements, the filters should be repackaged in their original transit cases or any other appropriate case for transportation. Ensure that the content of the package is complete before shipment. A copy of the provisional results should be included in the package.

2.5 Transport of artefacts

2.5.1 It is of outmost importance that the artefacts be transported in a manner in which they will not be lost, damaged or handled by unauthorised persons.

2.5.2 Artefact should be marked as “fragile”.

2.5.3 The artefacts should be accompanied by a suitable customs carnet (where appropriate) or documentation identifying the items uniquely.

2.5.4 Transportation is each laboratory's responsibility and cost. Each participating laboratory covers the costs for its own measurements, transportation and any customs charges as well as for any damages that may have occurred within its country. The overall costs for the organisation of the comparison are covered by the pilot laboratory. The pilot laboratory has no insurance for any loss or damage of the standards during transportation.

3. Description of the standards

3.1 The filter set to check the photometric scale is constituted by 5 neutral coloured glass filter plates 50 mm x 50 mm with nominal transmittance, at the wavelength of 546 nm, of approximately 92%, 56%, 10%, 1% and 0.1%. The filters are contained in a wood box specially design for transportation.

3.2 Each filter is identified by a reference engraved in a corner outside the area used for measurement. This reference has two parts. One is a letter indicating the type of glass (see table below) the other is the serial number of the filter.

3.3 The main characteristics of the filters are summarised in the following table :

Nominal transmittance %	Type of glass	Nominal thickness mm	Reference
92	BK 7	4.0	A
56	NG 11	1.5	B
10	NG 5	3.9	C
1	NG 4	3.9	D
0.1	NG 3	3.1	E

3.4 The manufacturing tolerances are for :

Size : 50 x50 (+0/-0.3) mm
Flatness : better than 5 μm over a central diameter of 30 mm
Parallelism : better than 0.02 mm except for the filter of transmittance 92% (<0.1 mm)

4. Measurement instructions

4.1 Traceability

Temperature measurements should be made using the International Temperature Scale of 1990 (ITS-90)

4.2 Measurand

4.2.1 The measurand is the spectral transmittance of the filters. The measurement should be performed in suitable laboratory accommodation maintained at a temperature of 23±2°C. The exact temperature of the laboratory during the time of the measurements should be reported.

4.2.2 The filter transmittance has to be measured independently several times. The exact number of measurements should be that normally used by the participating laboratory to obtain the appropriate accuracy of their specific measurement facility. The exact number of measurements used should be stated in the measurement report but only the mean or final declared value of the set required to be included.

4.3 Measurement instructions

4.3.1 Before measurement each filter should be inspected for damage or contamination. Any damage or cleaning should be documented using the appropriate form in appendix A5.

4.3.2 The spectral transmittance measurement of the filters should be performed at the following wavelength : 380, 400, 500, 600, 700, 800, 900 and 1000 nm.

4.3.3 The beam geometry must be a parallel beam where possible. For instruments that do not use a parallel beam the departure from parallel should be stated. The beam size probably will be different for the different instruments. 20mm diameter or 20mm square might be considered an ideal compromise between the conflicting requirements of flux and uniformity.

4.3.4 The angle of incidence on the filter should be normal or near normal and should be stated in the report.

4.3.5 The bandwidth used for the measurement should be stated in the report. 1 nm might be considered the norm for this wavelength range. However, there is no need for an agreed value of bandwidth because of the spectral neutrality of the filters.

4.3.6 Any information obtained relating to the use or any results obtained by a participant during the course of the comparison shall be sent only to the pilot laboratory who will be responsible for co-ordinating how the information should be disseminated to other participants. No communication whatsoever regarding any details of the comparison other than the general conditions described in this protocol shall occur between any of the participants or any party external to the comparison without the written consent of the pilot laboratory. The pilot laboratory will in turn seek permission of all the participants. This is to ensure that no bias from whatever accidental means can occur.

5. Measurement uncertainty

5.1 Measurement uncertainty shall be estimated according to the ISO Guide to the expression of uncertainty in measurement. In order to achieve optimum comparability, a list containing the principal influence parameters for calibration of spectral transmittance is given below. An example table which should be completed by participants is included as appendix A3. The participating laboratories are encouraged to follow this breakdown as closely as possible, and adapt it to their instruments and procedures. Other additional parameters may be felt appropriate to include dependent on specific measurement facilities and these should be added with an appropriate explanation and or reference. As well as the value associated with the uncertainty, participants should give an indication as to the basis of their estimate. All values should be given for a coverage factor of k=1.

5.2 Type A

5.2.1 Repeatability of measurements.
The repeatability of measurements can be determined in calculating the standard deviation of a set of measurements without realignment or repositioning of the filter. It characterises mainly the noise and the stability of the experimental set-up.

5.2.2 Reproducibility of measurements.
The reproducibility of measurements can be determined in calculating the standard deviation of a set of measurement with realignment and repositioning of the filter between each individual measurement. It characterise the whole process of the measurement. It is this value which has to be taken into account for the uncertainty evaluated according the type A method.

5.3 Type B

5.3.1 Main uncertainty components.
The 3 main components of uncertainty usually determined by type B method are :

- The non linearity of the detector over the dynamic range of the detector used for the measurements
- The uncertainty of the wavelength setting of the monochromator
- The stray light.

The uncertainties related to these effects have to be clearly stated in the uncertainty budget provided with the results of the comparison.

5.3.2 Other uncertainty components.
The other uncertainty components which can be put in the uncertainty budget if necessary are :

- The beam displacement effect and the defocusing effect due to introduction of the filter in the beam.
- The inter-reflection between the filter and the various optical and mechanical components of the experimental set-up.
- The obliquity effect due to the residual non parallelism of the beam, a non parallel beam or the imperfect alignment of the filter.
- The effect of the polarisation of the light
- The drift of the detector and/or of the sources during the measurements.
- Any other uncertainty components specific to the apparatus used for the measurements as explained in § 5.1.

6. Reporting of results

6.1 On completion of the measurements by the participating laboratory the provisional results of these measurements should be sent to the pilot laboratory with the filters.

6.2 The Final results should be communicated to the pilot laboratory at the latest within six weeks.

6.3 In completing the description of the participants measurement facility, appendix A.2, it would be useful for a schematic diagram of the facility to be included.

6.4 The measurement report forms in appendix A.1, A.2, A.3 of this document will be sent by e-mail to all participating laboratories. It would be appreciated if the report form (in particular the results sheet) could be completed by computer and sent back electronically to the co-ordinator. **In any case, the signed report must also be sent in paper form by mail.** In of any differences, the paper forms are considered to be the definitive version.

6.5 Following receipt of all measurement reports from the participating laboratories, the pilot laboratory will analyse the results and prepare a first draft report on the comparison. This will be circulated to the participants for comments, additions and corrections. Subsequently, the procedure outlined in the BIPM and EUROMET Guidelines will be followed.

A.1 Measurement results

The attached measurement summary should be completed for each filter. For clarity and consistency the following list describes what should be entered under the appropriate heading in the table.

Wavelength The assigned centre wavelength of the measured spectral transmittance.

Spectral transmittance The value of the spectral transmittance of the filter as measured by the participating laboratory.

Bandwidth The spectral bandwidth of the instrument used for the measurement defined as the Full Width at Half the Maximum.

Standard Deviation The standard deviation of the number of measurements made to obtain the assigned transmittance of the filter.

Number of runs The number of independent measurements made to obtain the specified standard deviation.

Uncertainty The total uncertainty of the measurement of spectral transmittance including both Type A and B for a coverage factor of k=1.

Table of measurement results

Reference of he filter : **Ambient temperature :**

Wavelength nm	Spectral transmittance	Bandwidth nm	Standard deviation	Number of runs	Uncertainty
380					
400					
500					
600					
700					
800					
900					
1000					

Laboratory :

Date : **Signature :**

A.2 Description of the measurement facility.

This form should be used as a guide. It is anticipated that many of the questions will require more information than the space allocated, please use separate sheets of paper as appropriate.

Make and type of the spectrophotometer

……..……
……
……
……..
Method of characterisation of the spectrophotometer

……
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……
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……
……
……..
Description of measuring technique (please include a diagram if appropriate)

……
……
……
……
……..
Description of calibration laboratory conditions : e.g. temperature, humidity, cleaning of the filter if it has be done due to accidental pollution etc

……
……
……

Laboratory :

Date : **Signature :**

A.3 Uncertainty of measurement

Parameter	Type A	Type B	Uncertainty in spectral transmittance
Repeatability	U_1		
Non linearity		U_2	
Wavelength setting		U_3	
Beam displacement		U_4	
Inter-reflection		U_5	
Obliquity effect		U_6	
Polarisation		U_7	
Drift		U_8	
Others		U_9	
RMS total			

The table is a suggested layout for the presentation of uncertainties for the calibration of each filter. However, it should be noted that since the uncertainties are wavelength dependant this table can only present a range for the various parameters. The summary table associated with the results (appendix A.1.) will of course take account of the wavelength dependent parameters.
The RMS total refers to the usual expression i.e. square root of the sum of the squares of all the individual uncertainty terms.

Laboratory :

Date : **Signature :**

A.4 Receipt confirmation

To : Jean Bastie
BNM-INM / CNAM
292 rue Saint Martin
75003 Paris
France

Fax : + 33 1 42 71 37 36
E-mail : bastie@cnam.fr

From : *(participating laboratory)*

We confirmed having received the standards of the BIPM Key comparison “regular spectral transmittance”.

Date : **Signature :**

A.5 Inspection of the transfer standards

Has the filter transportation package been opened during transit ? e.g; Customs…..Y / N

If yes please give details……

Is there any damage to the transportation package ?…….Y / N

If yes please give details……..

Are there any visible signs of damage to the filters ?……Y / N

If yes please give details……..

Laboratory :

Date : **Signature :**