VSL Holland Metrology

EURAMET.PR - Key Comparison K2.a

Spectral Responsivity, (A/W), 900 nm to 1600 nm

Technical protocol

Contents

1. Introduction

1.1 Under the Mutual Recognition Arrangement (MRA) [1] the metrological equivalence of national measurement standards will be determined by a set of key comparisons chosen and organized by the Consultative Committees of the CIPM working closely with the Regional Metrology Organizations (RMOs).

1.2 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, one of which is spectral responsivity. A working group in charge of planning these key comparisons decided to split this exercise into three distinct comparisons: K2.a: IR (900 nm – 1600 nm), K2.b: VS (300 nm – 1000 nm) and K2.c: UV (200 nm – 400 nm).

1.3 This document treats the technical protocol for the extension of the CCPR key comparison CCPR.PR-K2.a on spectral responsivity in the wavelength range from 900 nm to 1600 nm.

1.4 This technical protocol has been drawn up by the pilot laboratory of this key comparison VSL (NL). The proposed transfer detectors used will be Ge photodiodes instead of InGaAs used in CCPR.PR-K2.a. In this comparison VSL (NL) and NPL (UK) will act as link laboratories to KC CCPR.PR-K2a.

1.5 The procedures outlined in this document cover the main technical procedure to be followed during measurement of the transfer standards. The procedure, which follows the guidelines established by the BIPM [2], is based on current best practise in the use of standard detectors and takes account of the experience gained from the previous spectral responsivity comparison CCPR.PR-K2.a [3].

2. Organization

2.1. Participants

2.1.1 All EURAMET members were invited to participate in this comparison. The list of participants which was submitted to the CCPR for approval was drafted by the pilot and is given in section 2.2.

2.1.2 All participants must be able to demonstrate traceability to an independent realization 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.

1 MRA, Mutual Recognition Arrangement, BIPM, 1999.

2 T.J. Quinn, “Guidelines for key comparisons carried out by Consultative Committees”, Appendix F to the MRA, BIPM, Paris

3 Steven W Brown, Thomas C Larason and Yoshi Ohno (2010). Final report on the key comparison CCPR-K2.a-2003: Spectral responsivity in the range of 900 nm to 1600 nm, *Metrologia,* **47-**02002, doi: 10.1088/0026-1394/47/1A/02002

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

Institute	Contact Person	Address
UME :	Kamuran Turkoglu	TUBITAK Ulusal Metroloji Enstitusu PO Box.54 Gebze 41470 Kocaeli-Turkey Phone: +90 262 6795000 Ext:3353 Fax: +90 262 6795001 E-Mail: akt@ume.tubitak.gov.tr
GUM:	Ms Dorota Sobótko	Główny Urząd Miar/Central Office of Measures Radiation and Influence Quantities Department Photometry and Radiometry Laboratory Ul. Elektoralna 2 P.O. Box P-10 PL-00-950 Warsaw, Poland Phone: +48 22 581 94 58 Fax: +48 22 581 93 88 email: radiation@gum.gov.pl
CMI:	Dr Marek Šmíd	Czech Metrology Institute Laboratory of Fundamental Metrology V Botanice 4 CZ-150 72 Praha 5 Czech Republic Phone: +420 602 751 168 Fax: +420 257 328 077 E-mail: msmid@cmi.cz
SP :	Anne Andersson	SP Technical Research Institute of Sweden Measurement Technology Brinellgatan 4 PO Box 857 SE 50115 BORÅS Sweden Phone: + 46 105165403 Fax: + 46 105165620 E-mail:anne.andersson@sp.se
JV :	Mr Jarle Gran	Justervesenet Norwegian Metrology Service Fetveien 99 NO-2007 Kjelle Norway Phone: +47 64 84 84 84 Fax: +47 64 84 84 85 E-mail: jag@justervesenet.no
VSL :	Dr. Elena A. Revtova	VSL Holland Metrology Thijsseweg 11 2629 JA Delft The Netherlands Phone: + 31 152 691 500

		Fax: + 31 152 612 971 E-mail: erevtova@vsl.nl
NPL:	I : Subrena Harris II : Malcolm White	National Physical Laboratory Hampton Road TEDDINGTON Middlesex TW11 0LW United Kingdom Phone: +44 20 8943 6387 E-mail: subrena.harris@npl.co.uk; malcolm.white@npl.co.uk
METAS	Jacques Morel	Federal Office of Metrology Lindenweg 50 3003 Bern-Wabern Switzerland Phone: +41 31 32 33 350 Email: jacques.morel@metas.ch

2.3. Form of comparison

2.3.1 The comparison will principally be carried out through the calibration of a group of single Germanium (Ge) photodiodes. This type of detectors has shown excellent short and long-term stability.

2.3.2 A description of the transfer standards for use in this comparison is given in section 3 of this protocol. Each participating laboratory will receive a set of three detectors consisting of single element Ge photodiode set (square 10x10 mm).

2.3.3 All detectors are suitably mounted in a housing with a standard BNC connector, and are pre-aged with soft UV radiation. They will all have been checked for stability and been calibrated at VSL. All the detectors will include a PT100 temperature resistor supported with a 4-pin LEMO connector cable.

2.3.4 All detectors will be supplied by VSL. They will remain a VSL property throughout and after completion of the comparison.

2.3.5 The comparison will take the form of a 3-group star comparison (Figure 1). At first, VSL will calibrate all three sets of Ge detectors. Then the calibrated detectors will be distributed to the first group of the participants without VSL calibration results. The participants will then calibrate the detectors at the wavelengths required. The detectors will then be returned to VSL to carry out a repeat calibration to monitor drift. The sets will then be sent to the second and third groups of the participants, following the same procedure as given above. The last step will be carried out by VSL for repeat calibration and analysis. No preliminary result can be given by VSL before all the reports from all the participants have been received at VSL.

2.3.6 All results are to be communicated directly to VSL (the pilot laboratory) as soon as possible and certainly within six weeks of the completion of the measurements by a laboratory.

2.3.7 The timetable shown below gives an overview how the comparison is planned.

2.3.8 Each laboratory has two months for calibration and two weeks for transportation. With its confirmation to participate, each laboratory has confirmed that it is capable to perform the measurements in the time allocated to it.

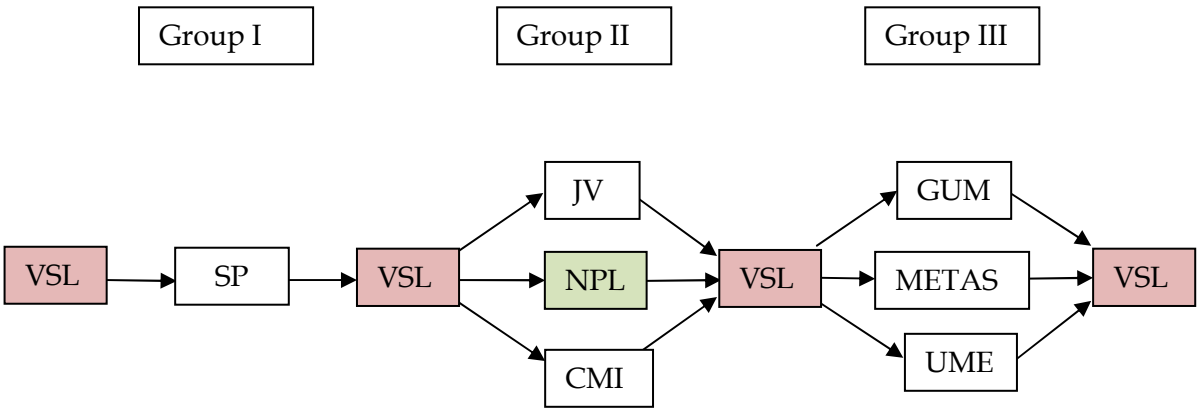


Figure 1. 3-group star comparison: VSL is the linking pilot laboratory; NPL is the linking participant laboratory.

2.3.9 If for some reasons, the measurement facility is not ready or customs clearance takes too much time in a country, the participating laboratory must contact the pilot laboratory immediately. Exclusion of a participants result from the report may occur if the results are not available in time to prepare the draft report

2.4 Timetable

Activity	Time required	Date due
Preparation		
Invitation to Participate sent including principle technical details (Protocol)		June 2010
Full protocol approved by TG		Dec 2010
Full protocol sent to participants		Dec 2010
Protocol sent to and approved by WG-KC	6 weeks	Mid Feb 2011
Measurements		
Detectors sent to the first group of participants	2 weeks for transport	End Feb 2011
Measurements by first group	**2 months**	**End Apr 2011**
Detectors sent back to VSL	2 weeks for transport	Mid May 2011
Measurements at VSL and detectors sent to second group	4 weeks + 2 weeks for transport	End Jun 2011
Measurements by second group	**2 months**	**End Aug 2011**
Detectors sent back to VSL	2 weeks for transport	Mid Sept 2011
Measurements at VSL and detectors sent to third group	4 weeks + 2 weeks for transport	End Oct 2011
Measurements by third group	**2 months + 2 weeks holiday**	**Mid Jan 2012**
Detectors sent back to VSL	2 weeks for transport	End Jan 2012
Measurements at VSL	4 weeks	End Feb 2012
Analysis and Reporting		
All measurement reports, results and uncertainty budgets due at VSL	Within 6 weeks of measurement completion	Group 1: Jun 2011 Group 2: Oct 2011 Group 3: Feb 2012

		Pilot: Mar 2012
Pilot sends measurement reports of all participants and uncertainty statements to all participants (pre-draft A)	2 months	End Apr 2012
Pilot sends relative data to each participant and reported values to each participant for checking (pre-draft A)	2 months	End Apr 2012
Participants provide comments on their own and others' reports and uncertainties	6 weeks	Mid Jun 2012
Participants respond to comments on uncertainty statements and pre-draft A discussion closed	4 weeks	Mid July 2012
Pilot distributes Draft A	2 months	Mid Sep 2012
Comments on Draft A due	2 months	Mid Nov 2012
Draft A-2 sent to participants for approval	6 weeks	Jan 2013
Draft B sent to Euramet-PR committee	1 month	Feb 2013
Draft B approved by CCPR WG-KC	6 weeks	Mid Mar 2013
Draft B approved by CCPR	6 weeks	End Apr 2013
Final Report published	6 weeks	Jun 2013

2.5 Handling of artefacts

2.5.1 The key comparison transfer detectors should be examined immediately upon receipt at the final destination. However, care should be taken to ensure that the detectors have sufficient time to acclimatise to the room's environment thus preventing any condensation etc.

2.5.2 The participants should inform the pilot laboratory on the receipt of the artefacts by sending an e-mail with the form 8.2 to the pilot laboratory. If any damaged found, the form 8.3 should be filled in and provided as soon as possible to the pilot laboratory as well.

2.5.3 Based on the date of detectors receipt the pilot laboratory will estimate the deadlines to return the detectors and to send the final results together with the technical report. The participants will receive an email with the deadlines in the following format: (1) deadline for measurements and provisional results which is within 8 weeks from the detectors receipt in format "dd.mm.yyyy"; (2) deadline for final results and technical report which is within 6 weeks from completion of measurements and shipment day of the detectors back to the pilot laboratory in format "dd.mm.yyyy". The detectors should only be handled by authorized persons and stored in such a way as to prevent damage.

2.5.4 No cleaning of any detector windows should be attempted, except using dry air (see section 3). If a transfer detector appears damaged a replacement will be available from the pilot laboratory. However, appropriate insurance should be taken out by participating laboratories to cover the cost of such a replacement if the damage occurred in transit.

2.5.5 During operation of the detectors, if there is any unusual occurrence (e.g. change of responsivity) the pilot laboratory should be notified immediately before proceeding.

2.5.6 The participants should inform the pilot laboratory via e-mail when the measurement on the detectors are completed (which should be before the deadline (1) defined by the pilot laboratory; refer to item 2.5.3) to arrange a suitable date for dispatch.

2.5.7 After the measurements, the detectors should be repackaged in their original transit cases. Ensure that the content of the package is complete before shipment. Always use the original packaging.

2.6 Transport of artefacts

2.6.1 It is of utmost importance that the artefacts be transported in a manner in which they will not be lost, damaged or handled by un-authorized persons.

2.6.2 Packaging for the artefacts will be made which should be suitably robust to protect the artefacts from being deformed or damaged during transit.

2.6.3 The artefacts are sufficiently robust to be sent by courier. The packages should be marked as 'Fragile'. If the possibility arises to hand-carry the packages this should be done.

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

2.6.5 Transportation and cost is at each laboratory's responsibility. Each participating laboratory covers the costs for its own measurements, transportation to the pilot laboratory and any customs charges as well as for any damages that may have occurred within its transportation. The overall costs for the organisation of the comparison and transportation to the participants are covered by the pilot laboratory.

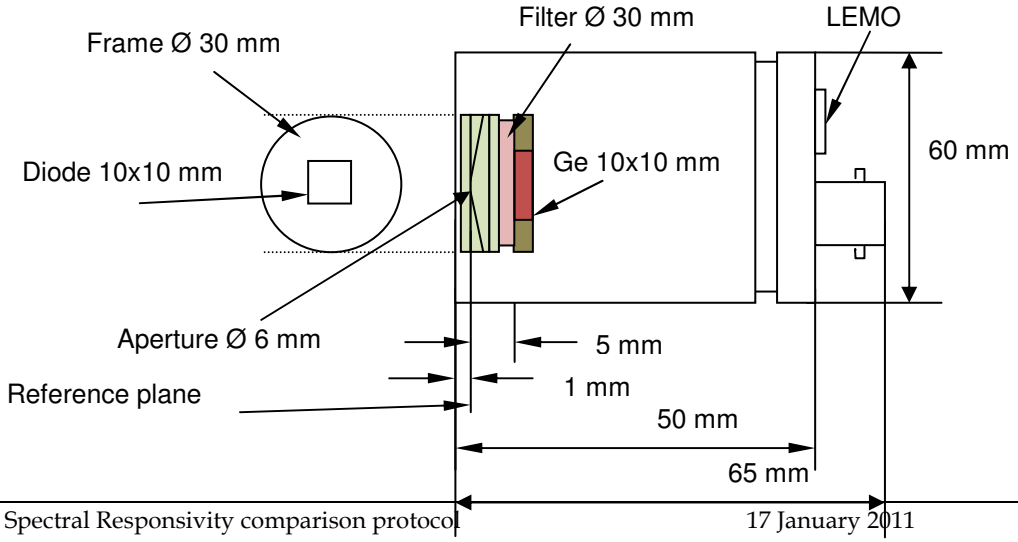
3. Description of the standards

3.1. Artefacts

3.1.1 The measurement artefacts will be a single element photodiode of type GM10HS (square 10x10 mm). The photodiode is separated from the working aperture (6mm) by a Schott RG FGL715 glass filter. The filter is used as a transmission window between 750 nm and 2000 nm, and to prevent fatigue effects in the Ge photodiode [refer to Metrologia, 2000, **37**, 567-570].

3.1.2 The detectors are mechanically robust but sensitive to dust and pollution. When not used they must always be stored in the anti-static bags they have been sent in. Dust free clean air can be used to remove any apparent dust particle by gently blowing onto the detector. Commercial pressurized neutral gas bottles should be used with precautions to avoid any liquid projection, or strong refrigeration of the photodiode surface leading to condensation.

The detector housing is shown in **Figure 2**. Each detector is fitted with a BNC connector for the photodiode signal (current) and a 4-pin LEMO connector for the 4-wire PT100 resistance thermometer.



Fi**gure 2**. Dimensions of the Ge transfer detector

4. Measurement instructions

4.1. Traceability

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

4.1.2 Electrical measurements should be independently traceable to the latest realisation of the Ampere, Ohm and Volt.

4.2. Measurand

4.2.1 The measurand is the spectral responsivity (A/W) of a detector, i.e. its responsivity as a function of the wavelength, measured at the center of its sensitive area.. The measurements should be performed in suitable laboratory accommodation maintained at a temperature as close as possible to 23 °C. The exact temperature of the laboratory during the time of the measurements must be reported. The pilot will correct the responsivity values to 23 °C based on the temperature dependence of each photodetector. The temperature dependence will be determined by the pilot.

4.2.2 Each independent measurement should consist of the detector being realigned in the measurement facility. It should be noted that each independent measurement may consist of more than one set of measurements, the exact number should be that normally used by the participating laboratory to obtain the appropriate accuracy as limited by the noise characteristics 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 is required to be included.

4.3. *Measurement instructions*

4.3.1. Before aligning the detectors they should be inspected for damage or contamination. Any damage should be documented and communicated to the VSL using the form given in section 8.3.

4.3.2. The operational conditions and alignment procedure for each detector should be followed according to the detail described in the notes supplied with each detector package. A summary is presented in section 9 of this document.

4.3.3. After alignment and before starting measurements, sufficient time should be allowed to let the detectors reach thermal equilibrium.

4.3.4. The spectral responsivity of the transfer standards should then be measured over the spectral region 900 to 1600 nm. Measurements should be made at intervals of 50 nm. For each measurement the internal PT-100 resistance of the detector should be measured as well. Measurement of the resistance is based on the 4-wire method. A LEMO cable for each photodetector will be provided by the pilot.No calibration of the PT-100 needed as relative measurements are of value.

4.3.5. The bandwidth used to measure the spectral responsivity should be less than 10 nm (Full Width at Half Maximum) and ideally less than 5 nm. If the bandwidth or center

wavelength shifts a lot, it is participant's responsibility to correct results (to zero bandwidth) or evaluate them into uncertainty budget.

4.3.6. Parameters used at the pilot laboratory:

- Round spot of 4 mm diameter imaged on the single Germanium detector
- f/#: around f/8
- room temperature : 23 °C (recorded).

Participants **are not obliged to use the same parameters** but **must report the values of these parameters used in their facility**.

No other measurements are to be attempted by neither the participants nor any modification to the operating conditions during the course of this comparison. In particular, the detectors must never be intentionally exposed to radiation below 400 nm nor above 1700 nm. They must never be placed under vacuum. If used with a laser source the optical power should be lower than 300 µW for a 2 mm diameter beam ($1/e^2$ diameter). To take into account the non-uniformity of detector responsivity due to beam size difference an uncertainty defined by the pilot laboratory will be distributed to the participants. The transfer standards used in this comparison should not be used for any purpose other than described in this document nor given to any party other than the participants in the comparison.

4.3.7. 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 as quickly as possible 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. Reporting of results

5.1. On completion of the measurements by the participating laboratory the **provisional results** of these measurements should be sent to the pilot laboratory together with the transfer detectors **before the deadline (1)** defined by the pilot laboratory (refer to item 2.5.3). The measurement results given in form 8.1 should be completed in the format MS Office Excel and sent to the pilot laboratory by e-mail. Any deviation from deadline (1) will lead to disqualification of the participant and exclusion from the comparison.

5.2. As soon as possible and **before the deadline (2)** (refer to item 2.5.3) the **final results** should be communicated to the pilot laboratory. The form 8.1 should be completed, printed, signed, scanned (to PDF) and sent by email to the pilot laboratory in two formats MS Office Excel and PDF. Any deviation from the given deadline (2) will lead to disqualification of the participant and exclusion from the comparison.

5.3. A technical report template will be prepared by the pilot laboratory and send by e-mail at the beginning of the Key Comparison together with the first detectors. Participants should complete the **technical report** together with the **final results** before the deadline (2) (refer to item 2.5.3).

5.4. Following receipt of all measurement reports from the participating laboratories, the pilot laboratory will analyse the results and proceed on Pre-Draft A process and

prepare Draft A. This will be circulated to the participants for comments, additions and corrections. Subsequently, the procedure outlined in the BIPM Guidelines [2] will be followed.

5.5. The technical report on the calibration measurements must include a description of the participants' measurement facility. It would be useful for a schematic diagram of the facility to be included.

5.6. The technical report on the calibrations must contain a comprehensive uncertainty budget, comprising all the contributions to the total uncertainty. The uncertainty of measurements shall be estimated according to the ISO Guide to the Expression of Uncertainty in Measurements (see section 6).

6. Reporting of uncertainties of measurements

6.1. The following uncertainty contributions should be considered:

- Uncertainty associated with the primary reference used (for a given wavelength range covered by the primary reference) laser- or monochromator-based cryogenic radiometer, or room temperature ESR, or calculable quantum detector, or any other system.
- Uncertainty associated with the spectral interpolation and/or extrapolation if the primary reference does not cover each of the wavelengths used:
  - spectrally flat detector (departure from ideal behaviour, repeatability, etc.)
  - calculable models of the detectors to be calibrated.
- Uncertainty associated with the transfer from the primary reference: wavelength dependence, alignment, temperature, polarization state of the beam used, effects of beam divergence, non-uniformity, calibration of amplifiers, of voltmeters.
- 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 as standard uncertainties.

6.2. In order to achieve optimum comparability, a list containing the principal influence parameters for calibration of spectral responsivity standards is given below. Table 1 reviews some of the main sources of uncertainty contributing to the uncertainty to be associated with the calibration of the KC transfer detectors, for four main types of primary references given as examples. For various reasons, the KC transfer detectors are usually not calibrated directly against the primary reference. It is therefore assumed in these examples that they will be calibrated against NMI working standards directly traceable to the primary reference.

6.3. The uncertainty budget stated by the participating laboratory should include all the information and sources of uncertainty which are relevant to their type of primary reference and calibration procedures. In Table 1, a cross indicates that the component described is usually relevant to the primary reference given on top of the column.

6.4. It should be noted that since several parameters are wavelength dependent, the combined uncertainty must be calculated for each wavelength or for each wavelength range.

Source of uncertainty	Mono-chromator based CR	Laser based CR	Room temper. ESR	Calculable QD	Other
Primary reference					**?**
Window transmittance		**x**			
Scattered and diffracted light	**x**	**x**			
Cavity absorptance	**x**	**x**	**x**		
Electrical power measurements	**x**	**x**	**x**	**x**	
Non-equivalence electrical / optical power	**x**	**x**	**x**	**x**	
Other …					
Interpolation and / or extrapolation when the primary reference does not cover each of the wavelengths of interest					**?**
Spectrally flat detector (residual wavelength dependence, uniformity, stability, …)		**x**		**x**	
Mathematical models		**x**		**x**	
Other …					
Calibration of NMI reference detectors (internal transfer)					**?**
Uniformity	**x**	**x**	**x**	**x**	
Linearity	**x**	**x**	**x**	**x**	
Polarization dependence	**x**	**x**	**x**	**x**	
Vignetting effects	**x**	**x**	**x**	**x**	
Repeatability (stability of source, alignment, ..)	**x**	**x**	**x**	**x**	
Electrical calibrations (amplifiers, voltmeters, …)	**x**	**x**	**x**	**x**	
Temperature	**x**	**x**	**x**	**x**	
Wavelength calibration	**x**	**x**	**x**	**x**	
Bandwidth effects	**x**	**x**	**x**	**x**	
Stray light	**x**	**x**	**x**	**x**	
Other …					
Calibration of KC transfer detectors					**?**
Repeatability (stability of source, alignment, ..)	**x**	**x**	**x**	**x**	
Temperature	**x**	**x**	**x**	**x**	
Wavelength calibration	**x**	**x**	**x**	**x**	
Bandwidth effects	**x**	**x**	**x**	**x**	
Stray light	**x**	**x**	**x**	**x**	
Beam size	**x**	**x**	**x**	**x**	
Other …					
Combined standard uncertainty (k=1)	**x**	**x**	**x**	**x**	**x**
Expanded uncertainty (k=2).	**x**	**x**	**x**	**x**	**x**

Table 1. Expanded uncertainty (k=2). Main sources of uncertainty contributing to the expanded uncertainty to be associated with the calibration of the KC transfer detectors, for four main types of primary references (given as examples). A cross indicates that the component described is usually relevant to calibration procedures based on the primary reference given on top of the column. The last column “Other” is meant for the participants and should be filled in case of an extra uncertainty source present. Corresponding crosses should be added as well.

7. Determination of the reference value

To be completed following discussion by the CCPR and EURAMET Key comparison working group.

8. Questionnaires and forms

The attached form 8.1 of measurement results should be completed for each detector.

For clarity and consistency the following list describes what should be entered under the appropriate heading in the form:

Wavelength	The assigned centre wavelength (or effective wavelength) of the measured spectral responsivity.
Spectral Responsivity	The value of the spectral responsivity of the detectors as measured by the participating laboratory
STD	The standard deviation of the number of measurements made to obtain the assigned spectral responsivity.
Number of measurements	The number of independent measurements made to obtain the specified std deviation.
Temperature	The temperature of the transfer detectors during calibration (the resistance of PT100 / Ohm)
Uncertainty	The total combined standard uncertainty of the measurement of spectral responsivity including both Type A and B for a coverage factor of $k = 1$.

8.1 Measurement results

Type of Standard …… Reference Number …….

Wavelength / **nm**	Spectral Responsivity **A / W**	STD **A / W**	Num of measurements **/n**	Temperature / **Ohm**	**Combined standard** uncertainty (k=1) / **%**
900					
950					
1000					
1050					
1100					
1150					
1200					
1250					
1300					
1350					
1400					
1450					
1500					
1550					
1600					

Laboratory:
……

Date: …… Signature: ……..

8.2 Receipt confirmation

E-mail

e-mail: erevtova@vsl.nl

To: **Dr. Elena A. Revtova**
VSL Holland Metrology
Thijsseweg 11,
2629 JA Delft,
the Netherlands
Phone: +3115269 1640(direct)
Phone: +3115269 1500 (reception)
Fax: +312612971

From: (*Participating Laboratory*)

We confirm having received the standards of the EURAMET.PR - Key Comparison K-2.a of Spectral Responsivity on .....(date; format dd.mm.yyyy).

After visual inspection

- ☐ No damage has been noticed;
- ☐ The following damage must be reported:
  .....
  .....
  .....

8.3 Inspection of the transfer standards

Has the detector 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 detector or housing?…..Y / N

If Yes please give details (e.g. scratches, dust, etc)

……
….
……
….

Do you believe the transfer standard is functioning correctly ?…Y./ N.

If not please indicate your concerns……..……
……
……
……
……

Laboratory:
……

Date: …… Signature: ……..

9. Alignment procedure of a single Ge element detector

(also will be sent together with the transfer detectors)

The optical beam is aligned with the center of the 6 mm aperture of the Ge detector. The reference plane is the front face of the 6 mm aperture being perpendicular to the beam axis.