Protocol for the comparison on spectral responsivity EUROMET PR-K2.b

DOCUMENT APPROVED BY THE COMPARISON'S WORKING GROUP

EUROMET PR Key Comparison K-2.b

Spectral Responsivity 300 nm to 1000 nm

Technical protocol

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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 EUROMET has approved the realisation of this comparison on Spectral responsivity within the 300 nm to 1000 nm range under the title EUROMET PR K2.b, as project # 587. The object of this comparison is to link participants to the the reference value of this quantity derived from comparison CCPR-K2.b.

1.3 This document treats the technical protocol for the key comparison EUROMET PR-K2.b and it has been drawn up by the working group of the comparison, comprising the IFA-CSIC (Spain), the BNM-INM/CNAM (France) and the NMi (Netherland). The three of them have participated in the comparison CCPR-K2.b

1.4 IFA (CSIC) has been recognised as the pilot laboratory for this comparison.

1.5 The procedures outlined in this document cover the technical procedure to be followed during measurement of the transfer standards. The procedure, which follows the guidelines established by the BIPM, is based on current best practise in the use of standard detectors and takes account of the experience gained from the previous BIPM spectral responsivity comparisons of 1993 and 2000, and that of the working group.

2. Organization

2.1. Participants

2.1.1 All EUROMET members were invited to participate in this comparison. The list of participants which was submitted to the EUROMET PR CP meeting for approval, was drafted by EUROMET PR rapporteur.

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

1 MRA, Mutual Recognition Arrangement, BIPM, 1999.

2.2. Participants' details

Laboratory	Contact Person	Contact details
Pilot		
IFA-CSIC	Dr. Joaquin Campos Acosta	Dpto. Metrologia Instituto de Fisica Aplicada CSIC C/. Serrano, 144 28006 Madrid Spain phone: +34 91 561-8806 fax: +34 91 411-7651 E-mail: jcampos@fresno.csic.es
Group 1		
METAS	Dr. Peter Blattner	Bundesamt für Metrologie und Akkreditierung Lindenweg 50 CH-3084 Wabern Switzerland Telephone: 41 31 32 33 340 Fax: 41 31 32 33 210 E-mail: Peter.Blattner@metas.admin.ch
Norwegian Metrology and Accreditation Service (JV)	Mr. Jarle Gran	Justervessenet Fetvn. 99 N-2007 KJELLER NORWAY Telephone: 47 64 84 84 84 Fax: 47 64 84 84 85 E-mail: Jarle.Gran@justervesenet.no
GUM	Dr. Jerzy Pietrzykowski	Central Office of Measures Optical and Ionising Radiation Laboratory Str. Elektoralna 2 00-950 Warszawa POLAND Telephone: (48 22) 620 02 41 E-mail: radiation@gum.gov.pl
INM- Romania	Mr. Mihai Simionescu	National Institute of Metrology Optical Quantities Laboratory Sos. Vitan Barzesti nr. 11 Cod postal: 75669 Bucharest, Sector 4 ROMANIA Telephone: (004021) 334 50 60 Fax: (004021) 334 53 45 e-mail: optica_ro@yahoo.com
Group 2		
NMi	Mr. Daniel Bos	NMi Van Swinden Laboratorium B.V. Schoemakerstraat 97 P.O. Box 654 NL-2600 AR Delft THE NETHERLANDS Telephone: 31 15 269 15 86 Fax: 31 15 261 29 71 E-mail: DBos@NMi.nl

Laboratory	Contact Person	Contact details
Optics Laboratory of UME	Dr. Farhad Samedov	TUBITAK-UME P. O. Box 54, 41470 Gebze-Kocaeli TURKEY Telephone: 90 262 679 5000, ext. 3300, 3352 Fax: 90 262 679 5001 E-mail: farhad.samedov@ume.tubitak.gov.tr
Czech Metrology Institute (CMI)	Dr. Marek Smid	Czech Metrology Institute Laboratory of Fundamental Metrology V botanice 4 CZ-150 72 Praha 5 CZECH REPUBLIC Telephone: +420 257 288 328 Fax: 420 2 57 32 80 77 E-mail: msmid@cmi.cz
BNM-INM/CNAM	Mr. Jean Bastie	BNM-INM/CNAM 292 Rue Saint Martin 75003 Paris FRANCE Telephone: 33 (0) 1 40 27 20 25 Fax: 33 (0) 1 42 71 37 36 E-mail: bastie@cnam.fr
Swedish National Testing and Research Institute (SP)	Mrs. Anne Anderson Fäldt	Swedish National Testing and Research Institute (SP) P.O. Box 857 SE-501 15 Borås SWEDEN anne.andersson-faldt@sp.se

2.3. Form of comparison

2.3.1 The comparison will principally be carried out through the calibration of a group of transfer detectors, single silicon photodiodes and trap detectors. This type of detectors has shown excellent short and long-term stability. The same type of detectors has been used in an earlier comparison of spectral responsivity in similar conditions as for this comparison.

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 batch of detectors consisting of two single element photodiodes and two three-elements reflection trap detectors. Detectors are handed over by IFA-CSIC for the purpose of this comparison.

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 the pilot laboratory.

2.3.4 All detectors have been supplied by the BIPM. They will remain BIPM property throughout and after completion of the comparison.

2.3.5 The comparison will take the form of a star comparison, carried out in two phases. The calibrated detectors will be distributed to the first group of participants without the pilot's calibration results. The participants will then calibrate the detectors at the wavelengths required. They will then return the packages to the pilot laboratory to carry out a new calibration to monitor drift. The batches will then be sent to the second group of participants, following the same procedure. No preliminary result can be given by the pilot before all the reports from both groups have been received at the pilot's laboratory.

2.3.6 All results are to be communicated directly to 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 given below gives an overview how the comparison is planned.

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

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 participant results from the report may occur if the results are not available in time to prepare the draft report

2.4. Timetable

Activity	Start Date
Invitation to Participate sent including principle technical details	December 2001
Meeting to start comparison, agree measurement conditions and form groups	April 2003
Full protocol approved by WG	November 2003
Full protocol sent to participants	December 2003
Detectors sent to first group of participants	July 2003
Participants of first group send packages back to the IFA-CSIC	September 2003
Pilot return measurements and checks finished (Phase 1)	October-November 2003
Detectors sent to second group of participants	November 2003[1]
Participants of second group send packages back to the IFA-CSIC	January 2004
Pilot return measurements and checks finished (Phase 2)	February-May 2004
Draft A ready	September 2004

2.5. Handling of artefacts

2.5.1 The standard 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 rooms environment thus preventing any condensation etc. The condition of the detectors and associated packaging should be noted and communicated to the pilot laboratory. Please use the fax form in chapter 8.4.

[1] Because of the flooding they suffered, detectors will be sent to the Czech National Laboratory at the beginning of March 2004.

2.5.2 The detectors should only be handled by authorised persons and stored in such a way as to prevent damage.

2.5.3 No cleaning of any detector windows should be attempted, except using dry air (see section 3). If a transfer standard 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.

2.5.4 During operation of the detectors, if there is any unusual occurrence, e.g. change of sensitivity etc the pilot laboratory should be notified immediately before proceeding.

2.5.5 Please inform the pilot laboratory via fax or e-mail when the measurement on the detectors are completed to arrange a suitable date for dispatch.

2.5.6 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. A copy of the provisional results should be included in the package.

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 unauthorised persons.

2.6.2 Packaging for the artefacts has been 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 is preferred.

2.6.4 The artefacts will be accompanied by a suitable customs carnet (where appropriate) or documentation identifying the items uniquely. The packaging will be lockable e.g. by clasp, but is easy to open with minimum delay to allow customs inspections to take place.

2.6.5 Transportation is at 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. Artefacts

3.1.1 The measurement artefacts will be two single element photodiodes type Hamamatsu 1337-1010BQ (quartz window) and two three element reflection trap detectors containing Hamamatsu 1337-1010N (windowless) photodiodes.

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 pressurised neutral gas bottles should be used with precautions to avoid any liquid projection, or strong refrigeration of the photodiode surface leading to condensation of contaminants.

3.1.3 The detector housings are shown in Figure 1 together with the size. Each detector is fitted with a BNC connector.

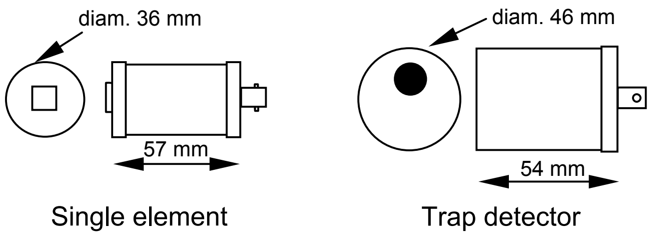

Figure 1. Dimensions of the transfer detectors

4. Measurement instructions

4.1. Traceability

4.1.1 Temperature measurements 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 and Volt.

4.2. *Measurand*

4.2.1 The measurand is the spectral responsivity of a detector, i.e. its responsivity as a function of the wavelength of the quasi-monochromatic radiation centred onto 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.

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 BIPM using the form given in section 8.2.

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 300 to 1000 nm. Measurements should be made at intervals of 20 nm starting from 300 nm up to 400 nm and at steps of 50 nm from there on until 1000 nm.

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. The exact bandwidth used for each spectral point must be reported, together with the centre wavelength (or effective wavelength).

4.3.6 Parameters used at the pilot laboratory:
- square spot 3 mm x 4 mm imaged on the detector (single element detectors) or on the third inner photodiode for trap detectors (see section 9).
- f/#: about f/8
- room temperature : 23 ºC (recorded)

Participants are not obliged to use the same parameters but must state which parameters were used.

4.3.7 No other measurements are to be attempted by 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 280 nm nor above 1100 nm. They must never be placed under vacuum. If used with a laser source (trap detectors only) the optical power should be lower than 300 µW for a 2 mm diameter beam ($1/e^2$ diameter). 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.

4.3.8 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 with the transfer detectors.

5.2 As soon as possible the final results should be communicated to the pilot laboratory and at the latest within six weeks.

5.3 The 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).

5.4 The report on the calibrations must include a description of the participants measurement facility. It would be useful for a schematic diagram of the facility to be included.

5.5 The measurement report forms in section 8 of this document will be sent by e-mail (Word95 -V7.0 document) to all participating laboratories. It would be appreciated if the report forms (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 case of any differences, the paper forms are considered to be the definitive version.

5.6 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 EUROMET Guidelines will be followed.

6. Reporting of uncertainties of measurements

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.

6.1 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.

6.2 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.

6.3 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**	
Other …					
Combined standard uncertainty	**x**	**x**	**x**	**x**	**x**

Table 1. 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). A cross indicates that the component described is usually relevant to calibration procedures based on the primary reference given on top of the column.

7. Determination of the reference value of the comparison and the link.

A key comparison reference value (KCRV) will be determined as recommended by the CCPR Key Comparison Working Group for the comparison CCPR-K2.b and approved by the CCPR on its meeting held on April 2003. This KCRV will be obtained as the weighted mean according to the expressions:

$w_i = \dfrac{1/u_i^2}{\sum_1^n 1/u_i^2}$ for the weights and $x_w = \sum_1^n w_i x_i$ for KCRV. Where x_i is the laboratory average value. This KCRF value will be used for checking the goodness and dispersion of the comparison. However, since the object of the comparison is to link laboratories to the comparison CCPR-K2.b, a link coefficient will be calculated for every participant. The calculation of this coefficient will have to be approved by the Working Group on Key Comparisons of CCPR. It will be proposed to that Working Group to use the procedure found in "Delahaye F. and Witt T. J., 2002 Metrologia **39** (Tech. Suppl.) 01005", which has been approved already by CCEM. As soon as an agreement is got, it will be notified to the participating laboratories.

8. Questionnaires and forms

The attached measurement summary should be completed for each detector.

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

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

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

Std Deviation 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

Uncertainty The total 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	Bandwidth / nm	Relative Std Dev.	Num of measurements	Temperature / °C	Uncertainty / %
300						
320						
340						
360						
380						
400						
450						
500						
550						
600						
650						
700						
750						
800						
850						
900						
950						
1000						

Laboratory: ……………………………………………………………………………………………

Date: …………………………………………… Signature: ………………………………..

8.2 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: ………………………………..

8.3 Description of the measurement facility and primary reference

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.

Type of primary standard..
....
....
....

Laboratory transfer standards used if any: ....
....
....
....

Monochromator used::..
....

Primary reference or traceability route of primary reference and breakdown of uncertainty: ....
....
....
....
....
....
....
....
....
....

Description of calibration laboratory conditions: e.g. temperature, humidity etc ....
....
....

Laboratory:
…………………………………………………………………………………………

Date: …………………………………………… Signature:

8.4 Receipt confirmation

FAX

To: **Dr Joaquín Campos**
IFA (CSIC)
Serrano, 144
28006 MADRID
Fax: 34 91 411 7651
e-mail: jcampos@ifa.cetef.csic.es

From: (*Participating Laboratory*)

We confirm having received the standards of the EUROMET Key Comparison of Spectral Responsivity
on ....(date).

After visual inspection

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

9. Alignment procedure for transfer detectors

(also sent together with the transfer detectors)

9.1 Alignment procedure for the reflection trap.

At the IFA, the alignment is made using the zero order of the monochromator, as indicated below (see figure 2):

a) remove the whole plastic dust cap
b) insert the alignment device C in the input hole
c) translate the trap along the x and y axis to align the central hole in the beam
d) rotate the trap about the x and y axis so that the weak residual reflected beam is collinear with the input beam.
e) repeat steps 'b' to 'd' to check both the position and the orientation.

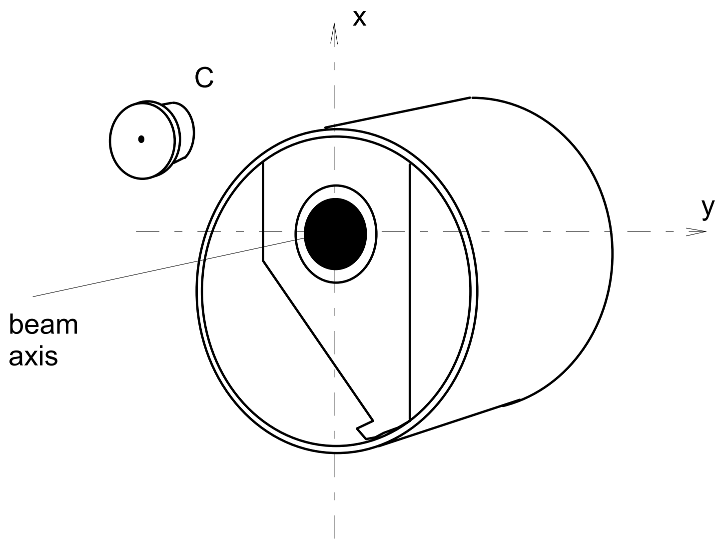

Figure 2 . Alignment procedure for the reflection trap. C: alignment device (brass)

Alternatively, the trap can be oriented perpendicularly to the optical axis using a small piece of mirror placed on the front face of the photodiode assembly (the outer cylindrical housing should not be used as a reference).

9.2 Beam divergence and trap detectors.

The maximum spot size and beam divergence can be deduced for each individual experimental arrangement from the following pictures. Figure 3 shows a view of the unfolded optical path inside the trap, the right hand-side of the picture representing the beam reflected from the third photodiode. Figure 4 shows the superposition of the three photodiodes as seen from the entrance hole. Remember that vignetting can have spectral effects[1].

1 K.D. Stock and R. Heine, Influence of vignetting errors on relative spectral responsivity of trap detectors, *Metrologia*, 1998, **35**, 447-450.

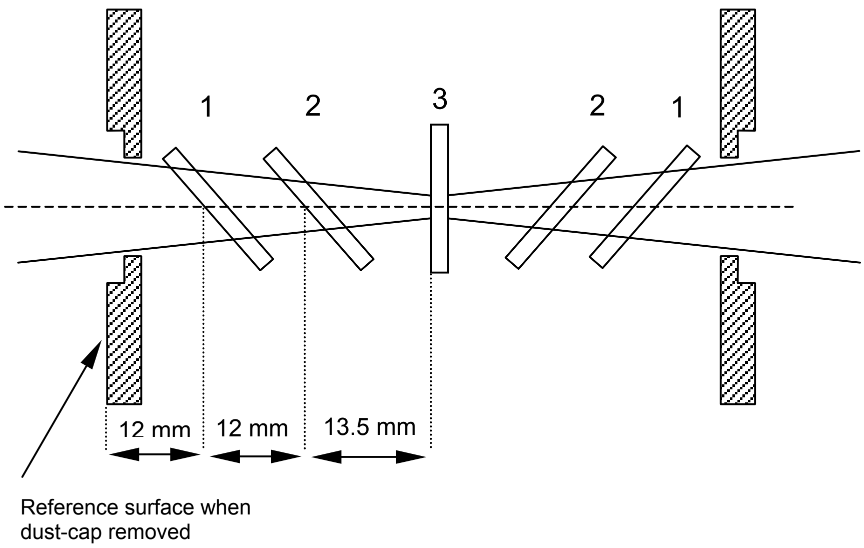

Figure 3 . Optical path (unfolded) inside the trap

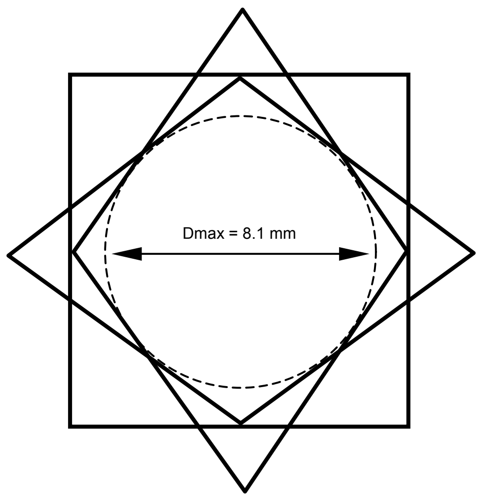

Figure 4. Superposition of the three photodiodes as seen from the entrance hole.

9.3 Alignment of single element detectors:

The optical beam is centred onto the sensitive surface, the sensitive surface being perpendicular to the beam axis.