

Bureau International des Poids et Mesures



CCPR Key Comparison K-2.b
Spectral Responsivity 300 nm to 1000 nm
Technical protocol

Contents

1. Introduction	2
2. Organization	2
2.1. Participants.....	2
2.2. Participants' details	3
2.3. Form of comparison.....	5
2.4. Timetable	5
2.5. Handling of artefacts.....	6
2.6. Transport of artefacts	6
3. Description of the standards.....	7
3.1. Artefacts.....	7
4. Measurement instructions.....	7
4.1. Traceability	7
4.2. Measurand	8
4.3. Measurement instructions.....	8
5. Reporting of results	9
6. Reporting of uncertainties of measurements.....	10
7. Determination of the reference value	11
8. Questionnaires and forms.....	12
9. Alignment procedure for transfer detectors.....	17

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 this key comparison consisting of the Bureau International des Poids et Mesures (BIPM, convener), the Measurement Standards Laboratory, New Zealand (MSL), the National Physical Laboratory, UK (NPL), the National Institute of Standards and Technology, USA (NIST), the National Research Council, Canada (NRC) and the Physikalisch-Technische Bundesanstalt, Germany (PTB), decided to split this exercise into three distinct comparisons: K2.a: IR (900 nm – 1600 nm), pilot NIST, K2.b (300 nm – 1000 nm), pilot BIPM and K2.c UV (200 nm – 400 nm), pilot PTB. The first part was to be carried out in 1999 with the second and third part following in 2000 and 2001, respectively. This document treats the technical protocol for the key comparison CCPR-K2.b.
- 1.3 This technical protocol has been drawn up by the small working group described above, comprising the BIPM (convener), the MSL, the NPL, the NIST, the NRC and the PTB.
- 1.4 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 [2], 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 comparison of 1993 [3,4] and that of the working group.

2. Organization

2.1. Participants

- 2.1.1 All CCPR 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.
- 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.

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 R. Köhler, R. Goebel, R. Pello, "Report on the international comparison of spectral responsivity of silicon detectors", rapport BIPM –94/9

4 R. Köhler, R. Goebel, R. Pello, "Results of an international comparison of spectral responsivity of silicon photodetectors", Metrologia, 1995/96, **32**, 463-468

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:	J-M. Coutin	292, rue Saint-Martin 75141 PARIS CEDEX 03 France Phone: + 33 1 40 27 27 04 Fax : + 33 1 42 71 37 36 email: coutin@cnam.fr
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CSIRO:	J. Gardner	PO Box 218 NSW 2070 Lindfield Australia Phone: +612 9413 7323 Fax: +612 9413 7474 email: jlg@tip.csiro.au
ETL:	I. Saito	1-1-4 Umezono Tsukuba-Shi 305-8568 Ibaraki Japan Phone: + 298 54 5557 Fax: + 298 54 5657 email: isaito@etl.go.jp
HUT:	E. Ikonen	P.O. Box 3000 FIN-02015 Espoo Finland Phone: + 358 9 451 2283 Fax : + 358 9 451 2222 email: erkki.ikonen@hut.fi
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IFA:	J. Acosta	Dpto. Metrologia Instituto de Fisica Aplicada CSIC C/. Serrano, 144 28006 Madrid Spain phone: +34 91 561-8806 fax: +34 91 411-7651 email: jcampos@fresno.csic.es

KRISS:	Young Boong Chung	Optics Laboratory Division of Quantum Metrology P.O. Box 102 Yousong Taejon 305-600 Republic of Korea phone: +82 42 868-5200 fax: +82 42 868-5022 optobit@kriss.re.kr
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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 [3,4].
- 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 element reflection trap detectors.
- 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 BIPM.
- 2.3.4 All detectors will be 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 BIPM calibration results. The participants will then calibrate the detectors at the wavelengths required. They will then return the packages to the BIPM to carry out a repeat 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 BIPM before all the reports from both groups have been received at the BIPM.
- 2.3.6 All results are to be communicated directly to the BIPM (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 participants 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:	26 Jan. 1999
Full protocol approved by WG	29 Nov. 1999
Full protocol sent to participants, to form Group 1 and Group 2	1 Dec. 1999
detectors sent to first group of participants	29 Feb. 2000

participants of first group send packages back to the BIPM	May. 2000
BIPM return measurements and checks finished (Phase 1)	Jul. 2000
detectors sent to second group of participants	31 Aug. 2000
participants of second group send packages back to the BIPM	Oct. 2000
BIPM return measurements and checks finished (Phase 2)	Dec. 2000
draft A ready	Jan. 2001
deadline for comments on draft A	Mar. 2001
draft B ready and submitted to CCPR	Apr. 2001

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.
- 2.5.2 The detectors should only be handled by authorized 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 un-authorized 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 should be done.
- 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 pressurized 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.

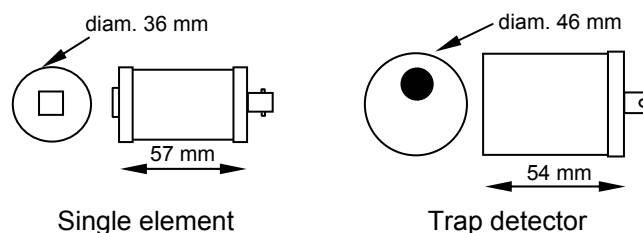


Figure 1. Dimensions of the transfer detectors

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 20.5 °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 2 mm x 2 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 : 20.5 °C (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 BIPM 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

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

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 Rainer Köhler
BIPM
Pav. de Breteuil
F-92312 Sèvres, Cedex
Fax: ++33 1 4534 2021
e-mail: rkohler@bipm.fr**

From: (Participating Laboratory)

We confirm having received the standards of the CIPM 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 BIPM, the alignment is made using a red He-Ne laser, so that the weak beam reflected from the trap can be observed. The QTH (or Xe) source is moved, and the laser beam enters the monochromator along the same optical axis. One has to make sure that the laser beam and the image of the output slits hit the same point on a target placed in image plane.

Then (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.

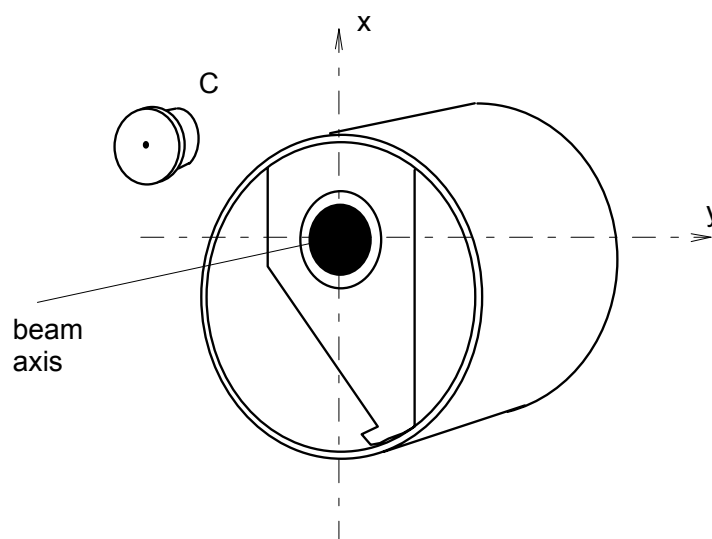


Figure 2 . Alignment procedure for the reflection trap. C: alignment device (brass)

In the absence of a laser beam, 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 effects¹.

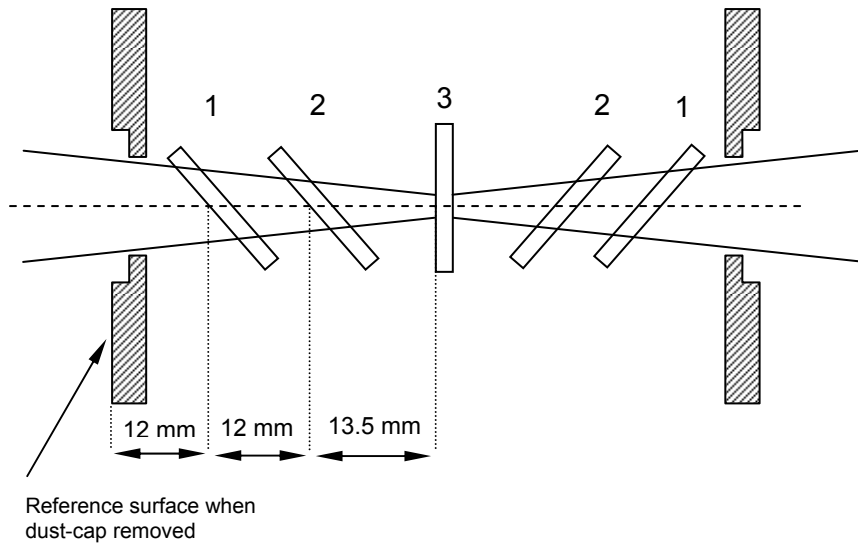


Figure 3 . Optical path (unfolded) inside the trap

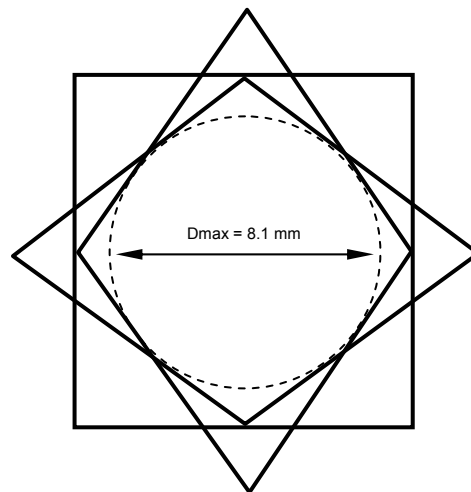


Figure 4. Superposition of the three photodiodes as seen from the entrance hole.

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.

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