APMP.PR-S1.1 Bilateral Comparison of Irradiance Responsivity of UVA Detectors

FINAL REPORT

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(NMC-A*STAR Singapore)
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Contents

1 Introduction

The aim of this comparison is to assess the improvement of the standards and techniques used for calibration and measurement of UVA irradiance responsivity of photo-detectors in NMISA.

This comparison is a follow-up of comparison APMP.PR-S1 and was registered as a bilateral comparison (APMP PR-S1.1). The comparison was carried out following the technical protocol [10.1] of supplementary comparison (APMP PR-S1) in the Key Comparison Data Base (KCDB) of BIPM.

The principle, organization and method of the comparison as well as the preliminary measurements at the pilot laboratory (NMC-A*STAR Singapore) were described in the final report of APMP PR-S1 comparison [10.2].

2 Time schedule

The measurements of the transfer detectors followed the agreed schedule in Table 2.1.

Table 2.1 Time schedule for measurements

s/n	Calibration period	Calibration lab
1	May 2007 - Jun 2007	NMC-A*STAR (Singapore)
2	Jun 2007 - Nov 2007	NMISA (South Africa)
3	Dec 2007 - Jan 2008	NMC-A*STAR (Singapore)

3 Pilot lab's reference standards and transfer detectors for the comparison

Two reference standard silicon photodiodes (Model: Hamamatsu S2281, s/n: E458 & E460) were selected as reference standards of the comparison in the pilot lab. Each photodiode was directly calibrated against a silicon trap detector in the spectral range (400-900nm) and the calibration was extended to UV range (250-400nm) through a cavity pyroelectric detector. The trap detector itself was regularly calibrated against the cryogenic radiometer in the pilot lab.

The stability of the reference standards was monitored by the pilot lab. The change of narrow band irradiance responsivity, *s(365),* of reference standard silicon photodiode (s/n: E458) is -0.25% and the change of broad band irradiance responsivity *s(UVA)* of reference standard silicon photodiode (s/n: E460) is -0.65% during the period of the comparison.

Two transfer detectors (Manufacturer: International Light, s/n: 7108 and 7112) were used in the comparison. Each consists of a UV enhanced silicon photodiode (Part No: SED033), a UVA broadband filter (Part No: UVA) and a quartz wide eye diffuser (Part No: W). For the selection and preliminary measurements of these detectors, please refer to Section 3 in the final report of APMP PR-S1 comparison.[10.2]

4 Calibration and uncertainty budget of transfer detectors at pilot lab

The calibration uncertainties have been assessed for both of *s(365)* and *s(UVA)* according to the method recommended by International Organization for Standardization (ISO).[10.3] The major sources of uncertainty have been investigated theoretically and experimentally and the results are shown in table 4.1 and table 4.2.

Table 4.1 Uncertainty budget of irradiance responsivity calibration of transfer detector, *s(365)*, under narrow band UV (365 nm) radiation ($k = 1$)

	Source of uncertainty	Value of standard uncertainty (%)
1	Calibration of reference standard detector (s/n:E458)	0.90
2	Drift of reference standard detector	0.33
3	Photocurrent measurement	0.05
4	Reading repeatability of standard detector	0.05
5	Reading repeatability of transfer detector	0.03
6	Alignment and positioning of standard detector	0.45
7	Alignment and positioning of transfer detector	0.22
8	Uncertainty due to uniformity of UV beam and area difference between standard & transfer detectors	1.20
	Combined relative standard uncertainty (%)	**1.6**

Table 4.2 Uncertainty budget of irradiance responsivity calibration of transfer detector, *s(UVA)*, under broad band UV radiation ($k = 1$)

	Source of uncertainty	Value of standard uncertainty (%)
1	Calibration of working standard detector (s/n: UVA-01)	2.30
2	Drift of standard detector	0.27
3	Photocurrent measurement	0.25
4	Reading repeatability of standard detector	0.03
5	Reading repeatability of transfer detector	0.03
6	Alignment and positioning of standard detector	0.45
7	Alignment and positioning of transfer detector	0.22
8	Uncertainty due to uniformity of UV beam and area difference between std & transfer detectors	0.04
	Combined relative standard uncertainty (%)	**2.4**

5 Calculation of the relative differences for NMISA's results

We denote s_{NMISA} the irradiance responsivity of a transfer detector calibrated at NMISA and s_{PILOT} the irradiance responsivity of the same detector calibrated at pilot lab. The relative difference Δ in the calibrations originating from NMISA and pilot lab is calculated as

$$\Delta = (s_{NMISA} - s_{PILOT}) / s_{PILOT} \tag{5.1}$$

As two transfer detectors have been calibrated against the pilot lab's reference, which is assumed to be stable, measurements provided by NMISA can be compared to each other via this common reference.

6 Transfer uncertainty and correction

During the comparison, the uncertainty reported by the participating laboratory should be combined with the uncertainty associated with the comparison (transfer uncertainty) that can contribute to the variability of the results (e.g. the drift of irradiance responsivity of transfer detectors and the stability of the pilot lab scale during the period of the comparison).

The correction should be made for the different measurement conditions between pilot and participating labs (e.g. Temperature conditions and UV irradiance levels etc.).

6.1 Drift of *s(365)* and *s(UVA)* of transfer detectors before and after transportation

The drifts of *s(365)* and *s(UVA)* are determined by the value change of *s(365)* and *s(UVA)* before sending the detectors to a participating lab and after receiving them by the pilot lab. The drift are 0.14% for *s(365)* and -5.27% for *s(UVA)*, which should be included in the transfer uncertainty budget.

The drift is usually small but it is large for *s(UVA)* in this comparison. After investigation, we found this unusual large drift was caused by the mould spots developed on the surface of the UVA filter. We believe high humidity and vast temperature change during the transportation of the detector between the labs were responsible for the growth of the mould on the filter surface. The possible cause of this incident is the container of artefacts had been inspected during transportation as NMISA staff remembered they have packed the items in the same way as they received them.

As calibration results of participating lab were compared with average values measured by the pilot lab before sending out and after receiving the transfer detectors, the transfer uncertainty (assuming a rectangular distribution) due to drifts of *s(365)* and *s(UVA)*, u_{drift}, can be calculated out by using equation (6.1.1).

$$u_{drift} = \frac{|\Delta(drift)|}{2\sqrt{3}} \tag{6.1.1}$$

where $\Delta(drift)$ is the drift value.

6.2 Stability of the pilot lab's scale during the period of comparison

The stability of the pilot lab scale depends on the stability of the reference standards maintained in the pilot lab. The standard uncertainty (assuming a rectangular distribution) due to those changes, u_{stab}, can be calculated out by using equation (6.2.1).

$$u_{stab} = \frac{|\ \Delta(change)\ |}{2\sqrt{3}} \qquad (6.2.1)$$

where *Δ(change)* is the change (in percentage) of *s(365)* or *s(UVA)* of reference standard silicon photodiodes during the period of the comparison.

To take into account the change of *s(365)* and *s(UVA)* of reference standard, the *s(365)* and *s(UVA)* of transfer detector should be corrected by using equation (6.2.2).

$$s_{c1} = s_1 \times CF_1 \qquad (6.2.2)$$

where s_1 and s_{c1} are the irradiance responsivity values of transfer detector before and after correction for the change of reference standard. The correction factor (CF_1) is defined by using equation (6.2.3).

$$CF_1 = 1 + \frac{change}{2 \times 100} \qquad (6.2.3)$$

6.3 Different measurement conditions

As the calibrations are under different lab's conditions (e.g. lab temperature and UV irradiance), the correction should be done for the NMISA's results using equation (6.3.1).

$$s_{c2} = s_2 \times CF_2 \qquad (6.3.1)$$

where s_2 and s_{c2} are the irradiance responsivity values before and after correction for different measurement conditions. The correction factor (CF_2) is defined by equation (6.3.2).

$$CF_2 = 1 + \frac{difference}{2 \times 100} \qquad (6.3.2)$$

6.4 Transfer uncertainty

The transfer uncertainty, u_T, of NMISA is the combination of u_{drift} and u_{stab} and can be calculated by using equation (6.4.1).

$$u_T = \sqrt{u_{drift}^2 + u_{stab}^2} \qquad (6.4.1)$$

6.5 Correction associated with the comparison

Appendix 1 shows the flow chart for the calculation of relative difference of *s(365)* or *s(UVA)* of transfer detectors between pilot lab and participating lab.

7 Results from NMISA (South Africa)

7.1 Measurement conditions and methods

a) Measurement conditions

- Spectroradiometer type: Jobin Yvon H10D double grating 1200/mm + integrating sphere
- Standards used:
    - A spectral irradiance standard (FEP lamp F-61) (ORW-301.SR)
    - An absolute radiometer with aperture area of 1 cm^2 (ORP-002.RA)
    - A standard detector -SUV100 PtSi/n-Si Schottky photodiode with aperture diameter of 8 mm (ORS-227.UV)

- Irradiance level: 0.112 mW/cm^2 (365±5 nm), 0.67 – 0.68 mW/cm^2 (UVA)
- Ambient temperature: 25 ^{o}C

b) Method used to determine *s(365)* of transfer detector

s(365) of transfer detectors were calibrated by indirect comparison to that of an absolute radiometer (ORP-002.RA) through a standard detector-SUV100 PtSi/n-Si Schottky photodiode (ORS-227.UV) under radiation from the comparison UV source plus a UV(365nm) filter.

c) Method used to determine *s(UVA)* of transfer detector

s(UVA) of transfer detector was calibrated by direct comparison to that of an absolute radiometer (ORP-002.RA) under radiation from the comparison UV source.

7.2 Uncertainty budget

Table 7.2.1 and 7.2.2 are the uncertainty budgets of calibration of transfer detector under narrow band UV (365nm) and broad band UVA irradiance ($k = 1$).

Table 7.2.1 Uncertainty budget of *s(365)* of transfer detectors

	Source of uncertainty	Value of standard uncertainty (%)
1	NMISA's standard detector	1.38
2	Filter matching	1.00
3	Lamp stability	0.10
4	Alignment and positioning of transfer detectors and NMISA's standard detector	0.23
5	Uniformity of UV beam	0.58
6	Expected standard deviation of the mean	0.50
	Combined relative standard uncertainty(%) (k = 1)	**1.89**

Table 7.2.2 Uncertainty budget of *s(UVA)* of transfer detectors

	Source of uncertainty	Value of standard uncertainty (%)
1	NMISA's absolute radiometer	0.42
2	Lamp stability	0.25
3	Alignment and positioning of transfer detectors and NMISA's standard detectors	0.23
4	Uniformity of UV beam	1.15
5	Relative spectral irradiance	0.58
6	Expected standard deviation of the mean	1.00
	Combined relative standard uncertainty (%) (*k* = 1)	**1.72**

7.3 Correction and transfer uncertainty

a) Correction for different measurement conditions

- Corrections are applied for different temperature (25°C at NMIA against 23°C at pilot lab). The correction factors are 1.000608 for s(365) and 1.002718 for s(UVA).
- Irradiance levels measured in NMISA are 0.112 mW/cm^2 (365nm) and 0.675 mW/cm^2 (UVA). The UV irradiance levels are similar with those of pilot lab and no correction has been applied.

The corrections for the different measurement conditions according to equations (6.3.1) and (6.3.2) are summarised in the table 7.3.1.

Table 7.3.1 Correction for different measurement conditions with pilot lab

	$s(365)_{NMISA}$		$s(UVA)_{NMISA}$	
Correction factor	1.000608		1.002718	
Detector s/n	7108	7112	7108	7112
Before correction	0.007640	0.007765	0.005667	0.005785
After correction	0.007645	0.007770	0.005682	0.005801

b) Corrections for stability of pilot scale

The change of *s(365)* of reference standard silicon photodiode (s/n: E458) is -0.25% and the change of *s(UVA)* of reference standard silicon photodiode (s/n: E460) is -0.65% during the whole period of the comparison. The correction factors (CF_1) can be calculated by using equation (6.2.3) which are 0.99875 and 0.99675 for *s(365)* and *s(UVA)* of transfer detectors respectively.

The corrections for the stability of pilot scale according to (6.2.2) are summarised in the table 7.3.2.

Table 7.3.2 Correction for the stability of pilot scale

	$s(365)_{PILOT}$		$s(UVA)_{PILOT}$	
Correction factor	0.99875		0.99675	
Detector s/n	7108	7112	7108	7112
Before correction	0.00748	0.00752	0.00581	0.00584
After correction	0.00747	0.00751	0.00579	0.00582

c) Transfer uncertainty

Drifts of *s(365)* and *s(UVA)* of transfer detectors before and after transportation are 0.14% and -5.27% respectively. The standard uncertainties due to drifts of *s(365)* and *s(UVA)* of transfer detectors, u_{drift}, are 0.04% and 1.52% according to equation (6.1.1).

The standard uncertainties due to stability of the comparison scale, u_{stab}, are 0.07% (365nm) and 0.19% (UVA) according to equation (6.2.1).

The transfer uncertainties, u_T, are 0.08% (365nm) and 1.53% (UVA) according to equation (6.4.1) which are summarized in Table 7.3.3

Table 7.3.3 Transfer uncertainty of NMISA's results

	365nm	*UVA*
u_{drift} (%)	0.04	1.52
u_{stab} (%)	0.07	0.19
u_T (%)	0.08	1.53

7.4 Comparison with the pilot lab reference value

The corrected relative difference Δ in the comparison was calculated for each detector, according to the equations (5.1) and results are shown in table 7.4.1.

Table 7.4.1 Relative difference in the comparison $(s_{NMISA} - s_{PILOT})/s_{PILOT}$

Detector (s/n)	*Δ (%),365nm*	*Δ (%),UVA*
7108	2.34	-1.87
7112	3.46	-0.33
Average	2.90	-1.10

The combined relative standard uncertainty of the comparison *u(Δ)* is calculated according to equation (7.4.1) and summarized in the table 7.4.2.

$$u^2(\Delta) = u_{rep}^2(\Delta) + u_T^2(\Delta) \tag{7.4.1}$$

$u_{rep}(\Delta)$: Total relative uncertainty of s(365) or s(UVA) reported by the participant.

$u_T(\Delta)$: The transfer uncertainty due to drift of transfer detectors and the stability of the pilot scale.

Table 7.4.2 Combined relative standard uncertainty of NMISA results ($k = 1$)

	365nm	*UVA*
$u_{rep}(\Delta)$,%	1.89	1.72
$u_T(\Delta)$,%	0.08	1.53
$u(\Delta)$,%	**1.89**	**2.30**

The results from NMISA have been compared with the pilot lab's calibrations using the standard uncertainty (k = 1), are shown in Table 7.4.3.

Table 7.4.3 Relative difference (Δ) from pilot lab's reference value and combined uncertainty, $u(\Delta)$

i	*NMI*	*365nm*		*UVA*	
		Δ(%)	$u(\Delta)$ (%, k = 1)	Δ(%)	$u(\Delta)$ (%, k = 1)
0	Pilot	0	1.60	0	2.40
1	NMISA	2.90	1.89	-1.10	2.30

7.5 Comparison with APMP.PR-S1 comparison reference value

The unilateral degree of equivalence of NMISA is given by

$$D_i = \Delta_{NMISA} + \Delta_{PILOT} \qquad (7.5.1)$$

$$U_i = k\sqrt{u^2(\Delta_{NMISA}) + u^2(\Delta_{PILOT})} \qquad (k = 2) \qquad (7.5.2)$$

Δ_{NMISA} is the relative difference of NMISA's results against the pilot lab's results and Δ_{PILOT} is the relative difference of pilot lab's results against CRV of APMP PR-S1 comparison. $u(\Delta_{NMISA})$ and $u(\Delta_{PILOT})$ are combined uncertainty for Δ_{NMISA} and Δ_{PILOT} respectively.

D_i is the relative difference of NMISA's results against CRV of APMP PR-S1 and U_i is the expanded uncertainty of D_i.

The results of unilateral degree of equivalence of NMISA are calculated according to equations (7.5.1) and (7.5.2) and listed in table 7.5 and shown in Fig. 7.5.1 and 7.5.2.

Table 7.5 The unilateral degree of equivalence of NMISA

i	*NMI*	*365nm*		*UVA*	
		D_i(%)	U_i (%, k = 2)	D_i(%)	U_i (%, k = 2)
1	SPRING	-1.43	2.94	-0.72	4.50
2	NMISA	1.47	4.95	-1.82	6.65

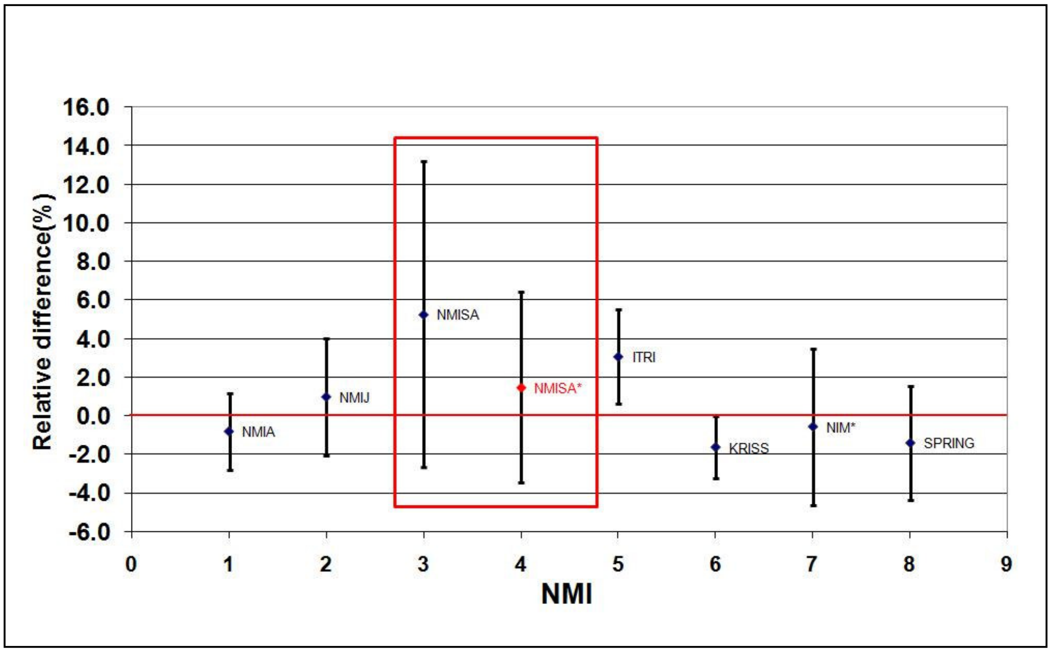


Fig. 7.5.1 Relative difference and combined uncertainty (k = 2) of *s(365)* against the APMP.PR-S1 comparison reference value of NMISA (see red rectangular box; NMISA: original result in APMP.PR-S1; NMISA*: new result in APMP.PR-S1.1).
*Note: SPRING has been transferred to NMC-A*STAR since 1 Jan 2008.*

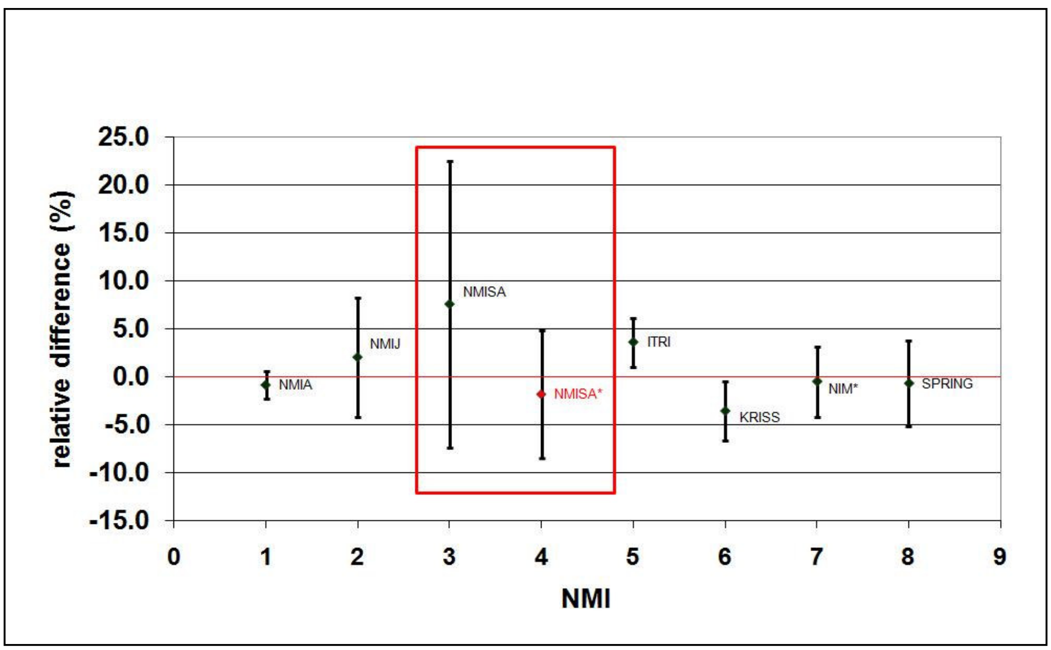


Fig. 7.5.2 Relative difference and combined uncertainty (k = 2) of *s(UVA)* against the APMP.PR-S1 comparison reference value of NMISA (see red rectangular box; NMISA: original result in APMP.PR-S1; NMISA*: new result in APMP.PR-S1.1).
*Note: SPRING has been transferred to NMC-A*STAR since 1 Jan 2008.*

8 Conclusion

The comparison results show the NMISA's results lie within ±2% against the comparison reference values of APMP PR-S1, which is greatly improved from the principal comparison.

9 Acknowledgements

The pilot lab wishes to thank LAG Monard from NMISA for his cooperation in the measurements and contribution of comments and feedback in the review of the report which enabled the comparison be completed smoothly.

10 References

10.1 SPRING Singapore, Technical protocol for APMP comparison on irradiance responsivity of UVA detectors, 26 Nov. 2002

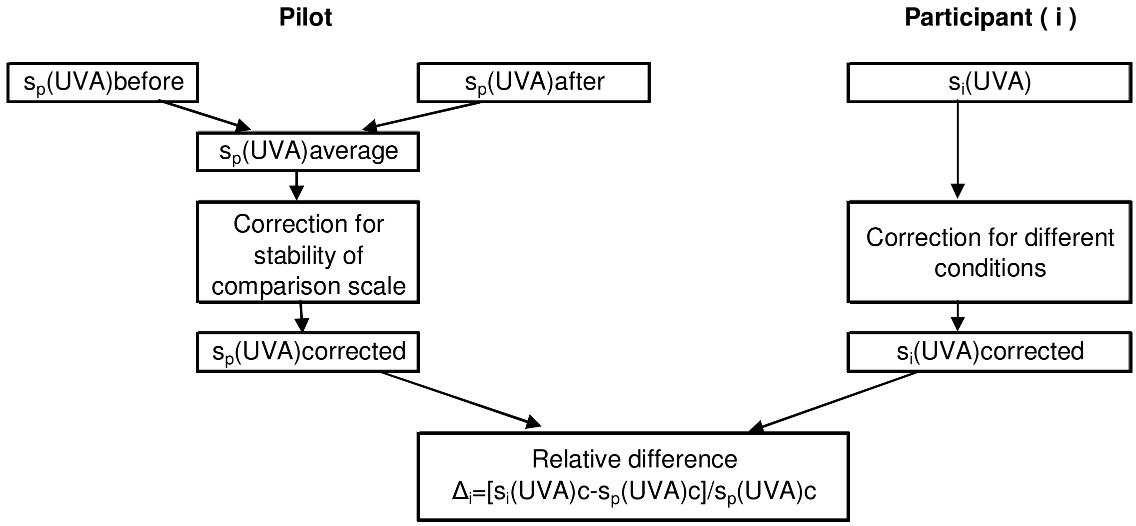
10.2 Final report of APMP PR-S1 comparison, Metrologia, 2007, 44, Tech. Suppl., 02001

10.3 Guide to the Expression of Uncertainty in Measurement, First edition 1995 (ISO, 1995).

10.3 Guidelines for CCPR Comparison Report Preparation, CCPR Key Comparison Working Group, Rev.1, March 2006

Appendix 1. Flowchart of data analysis for comparison results

(1) Flow chart of data analysis for comparison result of s(UVA) or s(365) of both transfer detectors



Note: Data analysis for s(365) is same as that of s(UVA)