CSIR-National Physical Laboratory, INDIA

FINAL REPORT ON SUPPLEMENTARY COMPARISON ON STEP HEIGHT STANDARDS (APMP.L-S7)

Dr. Rina Sharma (CSIR-NPLI), Dr. Girija Moona (CSIR-NPLI), Mr. Anusorn Tonmueanwai (NIMT), Dr. Jariya Buajarern (NIMT), Dr. Wang Shihua (NMC, A*STAR), Dr. Michael Matus (BEV), Dr. Takuma Doi (NMIJ/AIST), Dr. Kentaro Sugawara (NMIJ/AIST)

Contents

1. Introduction 3
2. Organization 3
   2.1 Participants and contact details 3
   2.2 Schedule 4
3. Description of artefacts 4
4. Measurement Instructions 5
   4.1 Equipment used for measurements 5
   4.2 Recommended measurement procedure 5
5. Results and standard uncertainties reported by participating laboratories 6
6. Analysis 7
7. Discussion of results 11
8. List of references 12

1. Introduction

This is a supplementary comparison dealing with only one parameter (step height) from the APMP.L-K8 comparison, but with different measuring range. This intercomparison concerned the measurement of step height standards by non-contact methods. Standards were circulated for step height measurement to each participating laboratory. The standards circulated include:
Four step height standards with one step (height/depth) on each of them and specified central area of the step is to be measured by non-contact method.
Measurement instructions for each standard were described in the technical protocol. Participants were expected to provide details of their measurement methods, environmental conditions and uncertainty evaluation. Measurement and evaluation of uncertainty were expected to be in line with ISO 5436 and JCGM 100:2008 [1, 2]. On completion of measurements, results of the international intercomparison are being circulated in the form of a draft report (Draft A) for comments from all the participating laboratories and a final report shall be submitted to APMP TCL chair. CSIR-NPLI agreed to act as the pilot laboratory and coordinated this intercomparison with 5 other NMI's.

2. Organization

2.1 Participants and contact details

List of participating NMIs and their contact details are given in table 1.

Table 1: List of participants and their contact details

Country	Laboratory / Address	Contact person, email and phone
India	CSIR-National Physical Laboratory India (CSIR-NPLI) Dr. K. S. Krishnan Marg, New Delhi 110012, India	**Dr. Rina Sharma** Tel: 91 11 4560 9275, Fax: +91 11 45609310 Email: rina@mail.nplindia.org Alternate Person : **Dr. Girija Moona** Tel : 91 11 47091669 Email : moonag@nplindia.org
Thailand	National Institute of Metrology, Thailand (NIMT) 3/4-5 Moo 3, Klong 5, Klong Luang, Pathumthani, 12120 Thailand	**Mr. Anusorn Tonmueanwai** Tel: +66 2577 5100 ext 1202, Fax: +66 2577 5088 Email: anusorn@nimt.or.th Alternate Person: **Dr. Jariya Buajarern** Tel: +66 2577 5100 ext 1216, Fax: +66 2577 5088 Email : jariya@nimt.or.th
Singapore	National Metrology Centre (NMC), Agency for Science, Technology and Research (A*STAR) 1 Science Park Drive, Singapore 118221	**Dr. Wang Shihua** Tel: (65) 6279 1941, Fax: (65) 6279 1993; Email: wang_shihua@nmc.a-star.edu.sg
Austria	Bundesamt fuer Eich- und Vermessungswesen (BEV) Arltgasse 35, A-1160 Wien, Austria	**Dr. Michael Matus** Tel +43 1 21110 6540, Fax +43 1 21110 996000 Email: michael.matus@bev.gv.at
Japan	National Metrology Institute of Japan (NMIJ/AIST) Tsukuba Central 3, 1-1-1, Umezono, Tsukuba, Ibaraki 305-8563, Japan	**Dr. Takuma Doi** Tel: +81-298-61-4360 Email: amb.doi@aist.go.jp Alternate Person : **Dr. Kentaro Sugawara** Tel: +81-298-61-4088 Email: sugawara.k@aist.go.jp
Egypt ***(* NIS could not participate due to carnet validity)***	National Institute for Standards (NIS) Tersa Street, El Haram, Giza P.O. Box: 136 Giza, code 12211 Giza – Egypt	**Prof. Dr. Mohamed A. Amer** Tel: +202 37401340, Fax: +202 37408111 Email: amer_nis@yahoo.com **Alternate person: Ezzat Oraby** Tel.: +020/1289732377 Email: ezzatoraby@hotmail.com

2.2 Schedule

The program started in December 2014 with measurement at the coordinating (pilot) laboratory. The measurement schedules are given in Table 2 and 3. Each laboratory made all required measurements in the specified period (with some grace period) and transferred the artefacts to the next listed laboratory.

Table 2: Planned Schedule

Sl. No.	Date	Country	Lab	Region	Carnet
1	2nd -3rd week of January 2015	India	CSIR-NPLI	APMP	-
2	1st week of February 2015	Thailand	NIMT* if NIMT is not able to take the measurements, this slot can be assigned to any other participant	APMP	Yes
3	4th week of February 2015	Singapore	NMC, A*STAR	APMP	Yes
4	3rd week of March 2015	Austria	BEV	EURAMET	Yes
5	4th week of April 2015	Egypt	NIS* NIS Egypt could not participate as carnet validity was expiring	APMP	No
6	3rd week of May 2015	Japan	NMIJ	APMP	Yes
7	3rd week of June 2015	India	CSIR-NPLI	APMP	-

Table 3: Actual Schedule

Sl. No.	Date of receipt and dispatch of artefacts (as per the carnet document)	Date of reporting measurement	Country	Lab	Region	Carnet
1	Dispatched on 18/02/2015	01/01/2015	India	CSIR-NPLI	APMP	-
2	Received on 17/04/2015 and dispatched on 19/05/2015	06/07/2015	Thailand	NIMT	APMP	Yes
3	Received on 26/05/2015 and dispatched on 05/07/2015	24/06/2015	Singapore	NMC, A*STAR	APMP	Yes
4	Received on 22/07/2015 and dispatched on 15/10/2015	25/08/2015	Austria	BEV	EURAMET	Yes
5	Received on 20/10/2015 and dispatched on 04/12/2015	24/12/2015	Japan	NMIJ/AIST	APMP	Yes
6	Received on 15/12/2015	09/02/2016	India	CSIR-NPLI	APMP	Yes

3. Description of artefacts

All the standard artefacts exhibit excellent accuracy and thermal stability. The standards circulated for measurement of Step height are described in Table 4.

Table 4: Nominal sizes of standards and other details

Model	Serial number	Nominal Step
SHS-80 QC	5904-97-06	8 nm
SHS-180 QC	5904-80-24	18 nm
SHS-880 QC	8897-37-16	88 nm
SHV	2727	10 µm

Figure 1. SHS

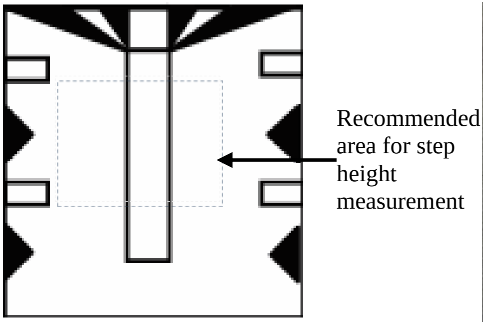

Figure 2. Central region of step

Figure 3. SHV 2727

The Thin Step Height Standards (SHS) consist of positive step, along with a pitch cluster diagnostic feature, etched at the center of a 25 mm × 25 mm × 3 mm quartz block, coated with chromium layer (90 nm thick, to ensure high reflectivity). The step is 100 micrometres wide and 750 micrometres long and is clearly marked with pointers, as shown in figure 1. The 400 micrometre × 400 micrometre area located at center of the step for (8 nm, 18 nm and 88 nm), as shown in figure 2 is to be measured. The SHS standards may have form deviations in sub-nanometres. The standard SHV 2727 is a chromium coated etched quartz block, mounted on a metal substrate, as shown in figure 3. The step is 1 mm wide and 5 mm long. The 3.0 mm × 2.5 mm area located at center of the step is to be measured. These step height standards are highly accurate and demonstrate excellent thermal stability.

4. Measuring instructions

After arrival of the travelling standard, they were checked for any physical damage, and allowed to stabilize in a temperature and possibly, humidity-controlled environment for at least 24 hours before commencing measurements.

4.1 Equipment used for measurements

Table 5: Instruments used for measurement

Country	Lab	Equipment	Traceability
India	CSIR-NPLI	Wyko NT 9800, 3-Dimensional non-contact optical profiler	CSIR-NPLI
Thailand	NIMT	Interference microscope and SIOS NMM-1 with laser focus sensor	NIMT
Singapore	NMC, A*STAR	Zygo New View Non-contact profilometer interferometer	NMC, A*STAR
Austria	BEV	SIOS NMM-1 with laser focus sensor	BEV
Japan	NMIJ/AIST	PSI interferometric microscope based on Nikon’s Mirau Objective	NMIJ/AIST

4.2 Recommended measurement procedure

The laboratories were required to make an attempt to measure step heights as per ISO 5436 scheme as shown in figure 4. If this could not be done, the closest possible option from their software was to be used and this variation was supposed to be specified in the report summary.

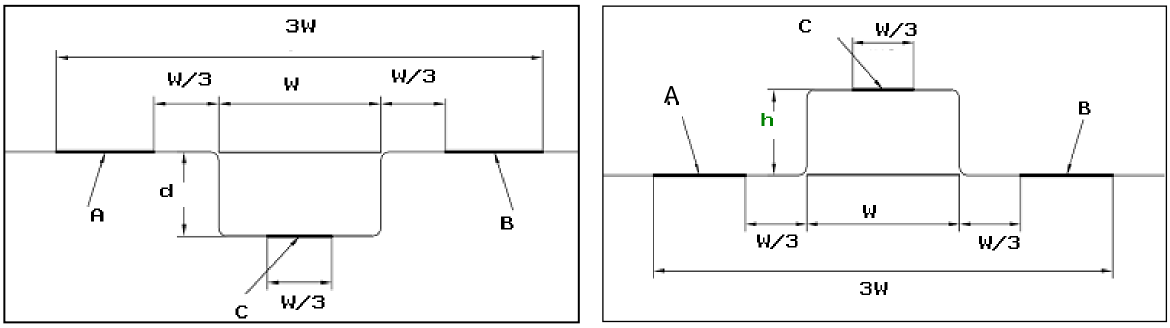

Figure 4: Step-height measurement for a negative step (left) and for a positive step (right)

Participants were to report the average height of the step (shown in figure 4, as ‘d’ for a negative step and as ‘h’ for a positive step) over a central area of 400 µm × 400 µm between the markers (for 8 nm, 18 nm and 88 nm step height standards), for the 10 µm step height standard the central area was 2.5 mm × 3 mm, resulting from at least 5 measurements within that area.
Measurement was to be made with the equipment and environment conditions that might achieve the smallest uncertainty, however the choice of instrument was at the discretion of the laboratory.
The standard ambient conditions for measurement were: -

Temperature: Reference temperature for the measurements: 20°C
Relative humidity: 50 % ± 10 %
It was requested to consider that the standards must be protected as much as possible.

5. Results and standard uncertainties reported by participating laboratories

Table 6 gives the values reported by various participants.
Note: NMIJ/AIST could not conduct measurements for 10 µm step height standard because it is out of their calibration service scope.

Table 6: Raw measurement results for nominal step height standard

	Step Size	**CSIR-NPLI** (Results reported are before dispatch)	**NIMT**	**NMC, A*STAR**	**BEV**	**NMIJ/AIST**
Measured Value	8 nm	8.95 nm	8.56 nm	8.6 nm	8.57 nm	8.8 nm
	18 nm	19.61 nm	18.67 nm	18.7 nm	18.81 nm	18.89 nm
	88 nm	91.63 nm	85.59 nm	86.5 nm	86.45 nm	86.48 nm
	10 µm	9.96 µm	9.972 4 µm	9.97 µm	9.961 4 µm	Not reported (Out of calibration service scope)
Reported Uncertainty (*k*=1)	8 nm	0.46 nm	0.1 nm	1.5 nm	0.07 nm	0.23 nm
	18 nm	0.46 nm	0.11 nm	1.5 nm	0.10 nm	0.23 nm
	88 nm	3.48 nm	0.21 nm	2.5 nm	0.07 nm	0.28 nm
	10 µm	0.015 µm	0.001 1 µm	0.04 µm	0.002 9 µm	Not reported (Out of calibration service scope)

6. Analysis

After completion of all the measurements, a supplementary comparison reference value (SCRV) and E_n ratio was calculated.

Method of weighted means of the values x_i (value from each individual lab) was applied to assign reference value. The associated uncertainty was calculated using methods consistent with JCGM 100:2008.

The weighted mean is given by:-

$$\bar{x}_w = \frac{\sum_{i=1}^{N} \frac{x_i}{u^2(x_i)}}{\sum_{i=1}^{i=N} \frac{1}{u^2(x_i)}} \quad (1)$$

Here x_i is the reported value and $u(x_i)$ is reported standard uncertainty for each participating laboratory.

As all NMIs were expected to have their own traceability, no correlation was expected. In case of no correlation the standard uncertainty of the weighted mean is given by:

$$u(\bar{x}_w) = \sqrt{\frac{1}{\sum_{i=1}^{i=N} \left(\frac{1}{u(x_i)} \right)^2}} \quad (2)$$

Assessment of each laboratory result was made with the degree of equivalence ratio (E_n):

$$E_n = \frac{x_i - \bar{x}_w}{2 \cdot u(x_i - \bar{x}_w)} = \frac{x_i - \bar{x}_w}{2 \cdot \sqrt{u^2(x_i) - u^2(\bar{x}_w)}} \quad (3)$$

Where, $u(xi)$ is the standard uncertainty reported by individual laboratory and $u(\bar{x}_w)$ is standard uncertainty of the weighted mean as given in equation (2). Since it was expected that most of the measurement values from the NMIs will be used for SCRV calculation, the sign inside the square root of the denominator of equation (3) has been taken as minus ("-").

Note: Any result with an $|E_n| > 1$ with respect to the reference value was to be excluded and the reference value and E_n ratio recalculated. If more than one laboratory result was to be excluded this was to be tested one result at a time. In the E_n formula shown in equation (3) the minus sign ("-") was used in the denominator for values contributing to the reference value but a plus sign ("+") for values not contributing to the reference value.

The Birge ratio is a test of the consistency of a set of data and its uncertainties. If the measurement being compared comes from the same population, then the propagated (internal) uncertainty $u_I(\bar{x}_w)$ should agree with that calculated from the standard deviation of the weighted mean (external) uncertainty $u_E(\bar{x}_w)$ as given by-

$$R_B = \frac{u_E}{u_I} \quad (4)$$

$$u_I(\bar{x}_w) = \sqrt{\frac{1}{\sum_{i=1}^{i=N} \left(\frac{1}{u(x_i)} \right)^2}} \quad (5)$$

$$u_E(\bar{x}_w) = \sqrt{\frac{\sum_{i=1}^{N} \frac{(x_i - \bar{x}_w)^2}{u^2(x_i)}}{(N-1)\sum_{i=1}^{N} \frac{1}{u^2(x_i)}}} \quad (6)$$

For an infinite population size, the ratio value should be approximately 1. For a limited population size, the Birge criterion is given by-

$$R'_B = \sqrt{1 + \sqrt{\frac{8}{N-1}}} \quad (7)$$

The consistency condition is satisfied when $R'_B > R_B$
The first calculation of E_n values including all participants for step height standards are as in table 7.

Table 7: Weighted mean calculation for SCRV and calculation of E_n value

		Laboratory				
	Step Size	**CSIR-NPLI**	**NIMT**	**NMC, A*STAR**	**BEV**	**NMIJ/AIST**
Measured Value	8 nm	8.95 nm	8.56 nm	8.6 nm	8.57 nm	8.8 nm
	18 nm	19.61 nm	18.67 nm	18.7 nm	18.81 nm	18.89 nm
	88 nm	91.63 nm	85.59 nm	86.5 nm	86.45 nm	86.48 nm
	10 µm	9.96 µm	9.972 4 µm	9.97 µm	9.961 4 µm	Not reported (Out of calibration service scope)
Uncertainty $u(x_i)$	8 nm	0.46 nm	0.1 nm	1.5 nm	0.07 nm	0.23 nm
	18 nm	0.46 nm	0.11 nm	1.5 nm	0.10 nm	0.23 nm
	88 nm	3.48 nm	0.21 nm	2.5 nm	0.07 nm	0.28 nm
	10 µm	0.015 µm	0.001 1 µm	0.04 µm	0.002 9µm	Not reported (Out of calibration service scope)
1/ ($u(x_i)$)^2	8 nm	4.73 nm^{-2}	100.00 nm^{-2}	0.44 nm^{-2}	204.08 nm^{-2}	18.90 nm^{-2}
	18 nm	4.73 nm^{-2}	82.64 nm^{-2}	0.44 nm^{-2}	100.00 nm^{-2}	18.90 nm^{-2}
	88 nm	0.08 nm^{-2}	22.68 nm^{-2}	0.16 nm^{-2}	204.08 nm^{-2}	12.76 nm^{-2}
	10 µm	4444.444 4 $µm^{-2}$	826446.281 0 $µm^{-2}$	625.00 $µm^{-2}$	118906.064 2 $µm^{-2}$	N/A (Out of calibration service scope)
$\bar{x}_w$	8 nm	8.59 nm				
	18 nm	18.78 nm				
	88 nm	86.37 nm				
	10 µm	9.971 0 µm				
$u(\bar{x}_w)$	8 nm	0.06 nm				
	18 nm	0.07 nm				
	88 nm	0.06 nm				
	10 µm	0.001 0 µm				
$x_i - \bar{x}_w$	8 nm	0.36 nm	-0.03 nm	0.01 nm	-0.02 nm	0.21 nm
	18 nm	0.83 nm	-0.11 nm	-0.08 nm	0.03 nm	0.11 nm
	88 nm	5.26 nm	-0.78 nm	0.13 nm	0.08 nm	0.11 nm
	10 µm	-0.011 0 µm	0.001 4 µm	-0.001 0 µm	-0.009 6 µm	N/A
E_n	8 nm	0.40	-0.15	0.01	-0.18	0.48
	18 nm	0.91	-0.64	-0.03	0.21	0.25
	88 nm	0.76	-1.96	0.03	1.44	0.20
	10 µm	-0.37	1.81	-0.01	-1.76	N/A

Deviation from weighted mean with uncertainty and E_n values for various step height standard artefacts are shown in figures 5, 6, 7, 8, 9, 10, 11 and 12.

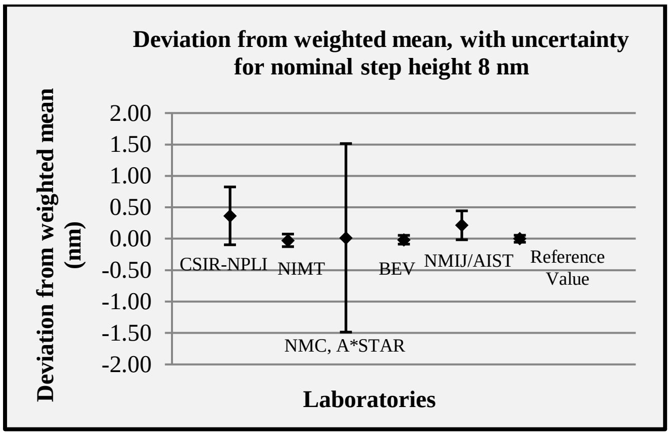

Figure 5: Deviation from weighted mean for the 8 nm step height standard

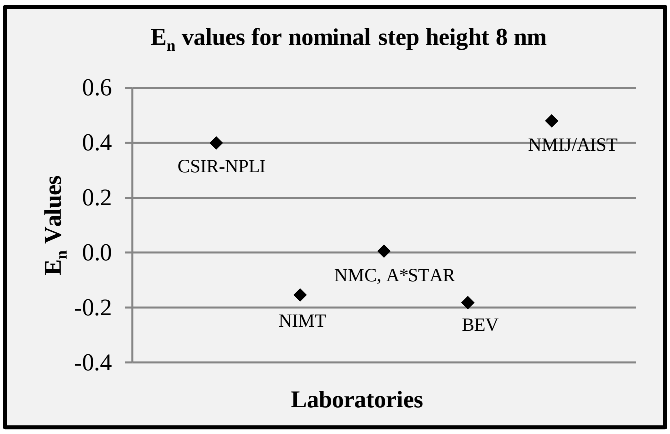

Figure 6: E_n values for the 8 nm step height standard

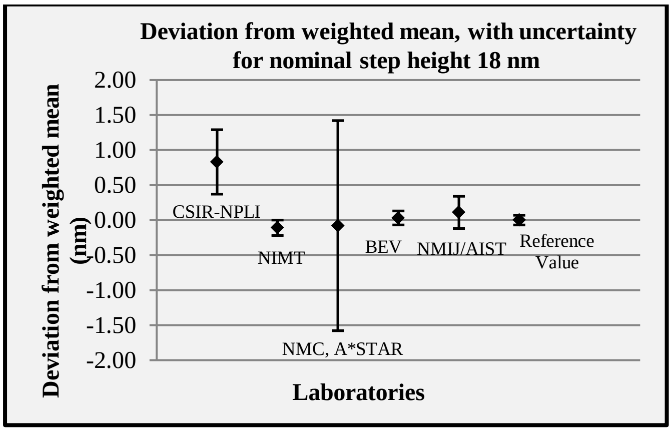

Figure 7: Deviation from weighted mean for the 18 nm step height standard

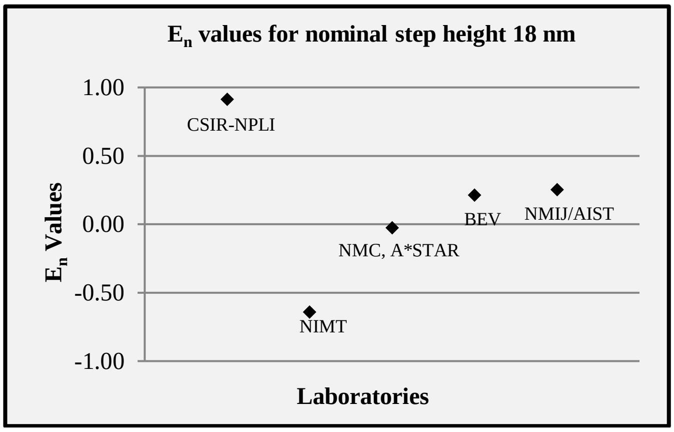

Figure 8: E_n values for the 18 nm step height standard

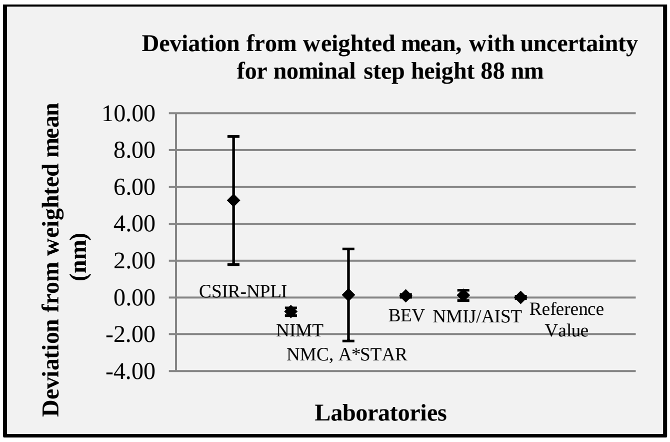

Figure 9: Deviation from weighted mean for the 88 nm step height standard before excluding NIMT for reference value calculation

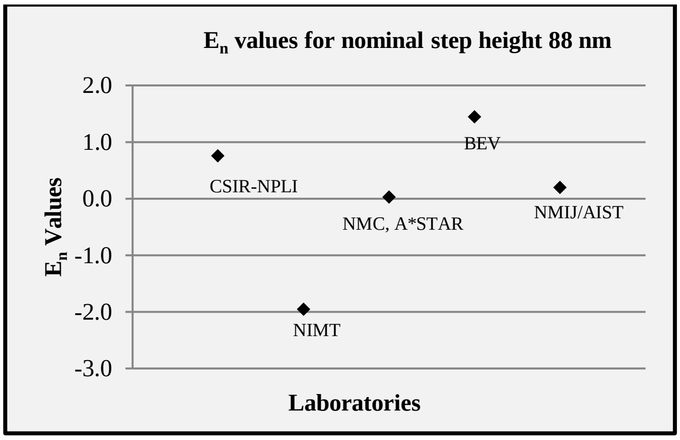

Figure 10: E_n values for the 88 nm step height standard before excluding NIMT for reference value calculation

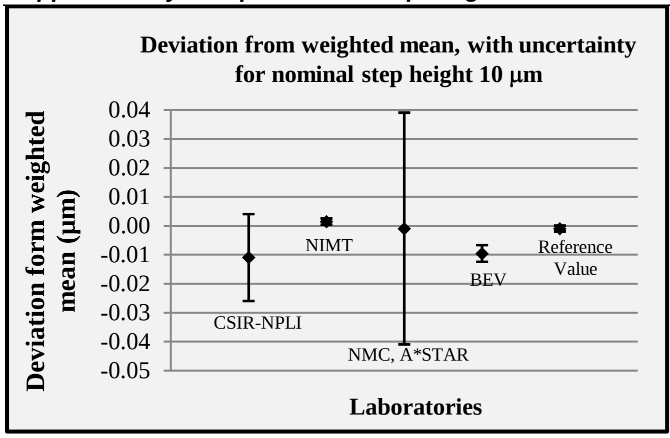

Figure 11: Deviation from weighted mean for the 10 µm step height standard before excluding NIMT for reference value calculation

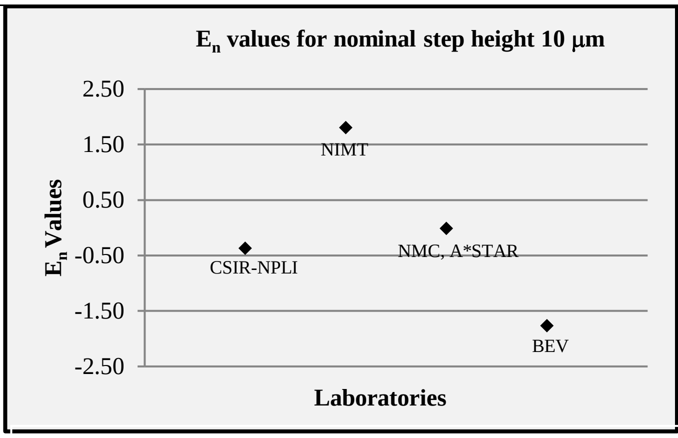

Figure 12: E_n values for the 10 µm step height standard before excluding NIMT for reference value calculation

The Birge ratio calculation has been done for the step height standard of 88 nm and 10 µm by considering values of x_i and $u(x_i)$ of each participating laboratory and $\bar{x}_w$, using equations (4), (5) and (6) as given in Table 8. On evaluating the Birge ratio using equation (7), for the 88 nm step height standard (N=5), the value comes out to be R'_B= 1.55, while for the 10 µm step height standard (N=4), R'_B= 1.62, which show no consistency at $k=2$ as $R'_B< R_B$.

Again $\bar{x}_w, R'_B$ and R_B value is calculated by considering measurement results of rest of the participants except one i.e., NIMT and the results are given in Table 9.

Table 8: Birge Ratio calculation

Step Size		R'_B	**Internal Uncertainty**	**External Uncertainty**	R_B
88 nm	***N*=5**	**1.55**	**0.065**	**0.135 2**	**2.09**
10 µm	***N*=4**	**1.62**	**0.001 0**	**0.002 1**	**2.09**

Table 9: Birge ratio after removing NIMT results of the 88 nm and the 10 µm for calculation of $\bar{x}_w$

Laboratory								
	Step Size	**CSIR-NPLI**	**NMC, A*STAR**	**BEV**	**NMIJ/AIST**			
Measured Value	88 nm	91.63 nm	86.5 nm	86.45 nm	86.48 nm	**N =4**	R'_B**=1.62**	
	10 µm	9.96 µm	9.97 µm	9.961 4 µm	Not reported	**N=3**	R'_B**=1.73**	
Uncertainty *u* (x_i)	88 nm	3.48 nm	2.5 nm	0.07 nm	0.28 nm			
	10 µm	0.015 µm	0.04 µm	0.002 9 µm	Not reported			
1/ ($u(x_i)$)^2	88 nm	0.08 nm^{-2}	0.16 nm^{-2}	204.08 nm^{-2}	12.76 nm^{-2}			
	10 µm	4444.444 4 $µm^{-2}$	625.00 $µm^{-2}$	118906.064 2 $µm^{-2}$	Not reported			
$\bar{x}_w$	88 nm	86.45 nm						
	10 µm	9.961 4 µm						
						Internal Uncertainty	**External Uncertainty**	**Birge Ratio** R_B
$x_i - \bar{x}_w$	88 nm	5.18 nm	0.05 nm	0.00 nm	0.03 nm	**0.067 8**	**0.058 4**	**0.86**
	10 µm	-0.001 4 µm	0.008 6 µm	0.00 µm	Not reported	**0.002 8**	**0.000 5**	**0.17**
E_n	88 nm	0.74	0.01	-0.11	0.05			
	10 µm	-0.05	0.11	0.01	Not reported			

After excluding NIMT and calculating the Birge ratio using equation (7), for the 88 nm step height standard (N=4), the value comes out to be R'_B= 1.62, while for the 10 µm step height standard (N=3), R'_B= 1.73, which show consistency at k=2 as $R'_B > R_B$.
E_n values are calculated again for all the participating laboratory using new reference values (calculated by excluding NIMT) as in Table 10 and Table 11.

Table 10: E_n value calculation for the 88 nm step height standard

			E_n calculation for the 88 nm step height standard NIMT included in calculation of $\bar{x}_w$				E_n calculation for the 88 nm step height standard NIMT excluded in calculation of $\bar{x}_w$			
LAB	x_i (nm)	u_i (nm)	SCRV ($\bar{x}_w$) (nm)	$u(\bar{x}_w)$ (nm)	Deviation from RV (nm)	E_n Value	SCRV-NIMT ($\bar{x}_w$) (nm)	$u(\bar{x}_w)$ (nm)	Deviation from RV (nm)	E_n Value
CSIR-NPLI	91.63	3.48	86.37	0.06	5.26	0.76	86.45	0.07	5.18	0.74
NIMT	85.59	0.21			-0.78	-1.96			-0.86	-1.96
NMC, A*STAR	86.5	2.5			0.13	0.03			0.05	0.01
BEV	86.45	0.07			0.08	1.44			0.00	-0.11
NMIJ/AIST	86.48	0.28			0.11	0.20			0.03	0.05

Table 11: E_n value calculation for the 10 µm step height standard

			E_n calculation for the 10 µm step height standard NIMT included in calculation of $\bar{x}_w$				E_n calculation for the 10 µm step height standard NIMT Excluded in calculation of $\bar{x}_w$			
LAB	x_i (µm)	u_i (µm)	SCRV ($\bar{x}_w$) (µm)	$u(\bar{x}_w)$ (µm)	Deviation from RV (µm)	E_n Value	SCRV-NIMT ($\bar{x}_w$) (µm)	$u(\bar{x}_w)$ (µm)	Deviation from RV (µm)	E_n Value
CSIR-NPLI	9.96	0.015	9.971 0	0.001	-0.011 0	-0.37	9.961 4	0.002 8	-0.001 4	-0.05
NIMT	9.972 4	0.001 1			0.001 4	1.81			0.011 0	1.81
NMC, A*STAR	9.97	0.04			0.001 0	-0.01			0.008 6	0.11
BEV	9.961 4	0.002 9			-0.009 6	-1.76			0.000 0	0.01

7. Discussions of result

Step height standard intercomparison (APMP.L-S7) was conducted for a wide range (8 nm, 18 nm, 88 nm and 10 µm) of step heights. Detailed results have been reported in sections 5 and 6. For the 8 nm and the 18 nm step height standards, all the participants reported measurement results along with associated uncertainties, with no outlier. However, for the 88 nm and the10 µm step height standards, NIMT had E_n values -1.96 and 1.81 and BEV had E_n values 1.44 and -1.76. NIMT's reported values for the 88 nm and the 10 µm step height standards were excluded for calculation of reference value, as NIMT had the largest absolute E_n value. This reference value (with exclusion of NIMT reported values for the 88 nm and the 10 µm step height standards) was considered for further calculation of E_n values and Birge Ratio analysis for establishment of consistency conditions as expressed in tables 9, 10 and 11.

8. List of references

[1] https://www.bipm.org/utils/common/pdf/final_reports/L/K8/APMP.L-K8.pdf
[2] Geometrical Product Specifications (GPS) – surface texture: Profile method; Measurement standards - Material measures, ISO 5436-1, (2000).
[3] https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf