## ASIA PACIFIC METROLOGY PROGRAM

## INTERCOMPARISON OF SURFACE ROUGHNESS

## APMP.L-K8

2021 - 2023

## PROTOCOL version 5

Prepared by Andrew Baker National Measurement Institute Australia

Issued 16 August 2021

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### 1. <u>Introduction</u>

The broad objective of the Asia Pacific Metrology Program (APMP) is to improve the measurement capabilities in the Asia Pacific region by sharing facilities and experience in metrology. Comparison of calibrations by different laboratories on given artefacts adds confidence in the measurement of standards and leads to international acceptance of the measurements carried out by these laboratories. This intercomparison concerns the calibration of surface roughness standards listed in section 5. Selected parameters are to be calculated according to well established international standard documents. Measurement conditions for each standard are described in the appropriate section of this document. On completion of measurements by all participants, the results of the intercomparison will be circulated for comment and a final report presented to the APMP.

### 2. <u>Participation</u>

The project will be coordinated (piloted) by Mr Andrew Baker, NMIA and supported by Dr Jariya Buajarern, NIMT. The program coordinators will be responsible for:

- Selecting, preparing and declaring the value of the artefacts,
- Planning the program and organising the schedule,
- Maintaining a list of participant's information,
- Liaising with participants,
- Making initial, mid-cycle and final measurements,
- Collating and assessing results by accepted statistical methods,
- Preparing the draft report and distributing the draft report for comment,
- Reviewing comments and completing the final report and executive report.

Participants, listed in Table 1, are expected to be ready to conduct measurements at the scheduled period, listed in Table 2. A laboratory may withdraw **any one or all** of its results from the intercomparison at any stage prior to submission of their results to the coordinator. Once the coordinator receives the submission of results, it is considered the laboratory regards them as appropriate to contribute to the calculation of mean results. The coordinator will then proceed with the analysis which will statistically determine which results are included. Any removal of results at a later date will be considered in exceptional circumstances and is at the discretion of the coordinators.

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### 3. <u>Scheduling, Packaging and Customs</u>

The program is to commence in April 2021 with measurement at the pilot (coordinating) laboratory with the measurement schedule listed in Table 2. Each laboratory is expected to make all required measurements in a two (2) week period and allow a further two (2) week period for transferring the artefacts to the next listed laboratory. Those scheduled for December are allowed eight (8) weeks due to expected public holidays. The pilot laboratory will make repeat measurements in the middle and at the end of the schedule to assess artefact condition and stability. Only the first results from the pilot laboratory will be included in the analysis.

The standards are made of glass and steel. Due to the length of this intercomparison, great care must be taken to ensure all surfaces are maintained in uncontaminated and undamaged condition. Section 5 and Appendix B list strict handling and measurement instructions including cleaning instructions. The artefacts are transported inside a solid protective case as shown in Figures 1 and 2. Each standard is also its individual case inside a single wooden box as shown in Figure 3. Each participant is responsible for carefully re-packing and shipping the artefacts to the next participant and bearing the costs associated with forwarding the artefacts. **If possible, place a new or refreshed satchel of desiccant in the wooden box with the artefacts to minimise risk of rust for the steel artefacts.** 

A strong envelope is to be attached to the outside of the box (with a second copy inside the box) with of the following documents:

- 1. This protocol.
- 2. Customs declaration / Proforma invoice (Appendix A of this document).
- 3. *ATA Carnet covering participating countries within the scheduled period.* (Since a Carnet is valid for 12 months, when the shipment returns to Australia in mid-cycle, a second ATA Carnet will be established to cover the second half of the intercomparison.)
- 4. For the end of the intercomparison an additional document: *English/Russian translation letter* will be added to the list of documents for passage through Russia.

If any documents are lost or missing, please contact the coordinator immediately for replacement. When repacking the box, put a copy of the documents outside of the box as well as a copy inside to help with customs inspections.



Figure 1. Solid case.



Figure 2. A small wooden box containing artefacts inside the case.



Figure 3. Individual artefact cases.

 Table 2. Measurement schedule

Country	Lab	Region	Scheduled Month	Cumulative Months	ATA (	Carnet
Australia	NMIA	APMP	2021 August	1	Yes	
Japan	NMIJ	APMP	2021 September	2	Yes	
South Korea	outh Korea KRISS		APMP 2021 October		Yes	
Chinese Taipei	CMS/ITRI	APMP	2021 November	4	No <sup>1</sup>	
Hong Kong	SCL	APMP	2021 December / 2022 January	5,6	Yes	First
Thailand	NIMT	APMP	2022 February	7	Yes	
Malaysia	NMIM	APMP	2022 March	8	Yes	
India	NPLI	APMP	2022 April	9	Yes	
Saudi Arabia	SASO-NMCC	GULFMET	2022 May	10	No <sup>2</sup>	
Australia (stability check) NMIA		APMP	2022 June	11	Yes	
China	NIM	APMP	2022 July	12	Yes	
Singapore	NMC, A*STAR	APMP	2022 August	13	Yes	
Indonesia	SNSU-BSN	APMP	2022 September	14	Yes	
Argentina	INTI	SIM	2022 October	15	Yes	
Mexico	CENAM	SIM	2022 November	16	Yes	Second
UK	NPL	EURAMET	2022 December / 2023 January	17,18	Yes	
Russia VNIIMS		COOMET	2023 February	19	Yes <sup>3</sup>	
New Zealand	MSL	APMP	2023 March	20	Yes	
Australia (stability check)	NMIA	APMP	2023 April	22	Yes	

<sup>1</sup> Chinese Taipei require a different type of Carnet which is not being used for this project.

<sup>2</sup> At time of protocol development, Saudi Arabia are in the process of approving the ATA Carnet system.

<sup>3</sup> Russia requires an accompanying letter with direct English to Russian translation.

#### 4. Equipment

Each laboratory has nominated the equipment listed in Table 3 for measurement of included artefacts.

Lab	Lab Instrument and software		nges	Traceability		
		(IIIII) XZ		X	7	
	Zeiss Jena Epival Interference Microscope with He-Ne laser Software: Excel	-	~0.005	-	NMIA He-Ne	
NMIA	Hommelwerke T8000 Software: Turbo Wave v7.2	200	1.6	NMIA He-Ne	NMIA Step H.	
NIMT	Kosaka Surfcorder ET4000AK Software: i-Star	100	32	NMIT	NIMT	
NIM	Metrological Interference Microscope Software: Areal Metrology v1.2	-	1	NIM	NIM	
	Profilometer Instrument Software: Roughness Metrology v1.0	120	2			
CMS / Kosaka Surfcorder ET-4100		200	0.05	CMS	CMS	
ITRI	Taylor Hobson PGI freeform Software: Talymap platinum 3D Astigmatism Pack	200	14	line scale	grooves	
SCL	Taylor Hobson Talysurf i-Series M112-4581 Software: Ultra v6.2.4.1	120	2	NPL	METAS	
NPLI	Mitutoyo Formtracer Extreme CS 5000 CNC Software: Formtracepak v6.0 CNC	200	12		NPLI (JCSS)	
NMH	Taylor Hobson Talysurf S2 Software: Homemade	120	1	NMIJ	NMIJ	
INIVIIJ	Panasonic UA3P Software: Homemade	100	10			
NMIM	KLA Tencor P7 Software: Apex analysis	156	~0.3		NIST	
	Taylor Hobson Form Talysurf 120i Software: Ultra	120	~2		NMIM	
MSL	Mitutoyo Surftest SV-3100H4 Software: Formtracepak	100	0.8		MSL	
NMC, A*STAR	Taylor Hobson Form Talysurf PGI2540 Software: Ultra	200	12.5	NPL	NPL	
KRISS	Taylor Hobson Form Talysurf 2	200	10	KRISS He-Ne	KRISS Step H.	
SNSU- BSN	Taylor Hobson Talysurf i120 Software: Ultra (data acquisition only)		1		METAS	
SASO- NMCC	Mahr profilometer Software: MarWin MarSurf XCR 20 7.00-12 SP3	120	0.5		РТВ	
NPL	NanoSurf IV stylus tracing instrument		0.02	NPL	NPL	
VNIIMS	Talysurf PGI420Software: Talyprofile ver. 7.4.9391 (based on Mountains)		4	VNIIMS	UKAS	
CENAM	Taylor Hobson Form Talysurf PGI2540     Software: Talymap Gold v6.5.4.2	200	12	CENAM	CENAM	
INTI	Stylus roughmeter Taylor Hobson Tsi 120 Software: Taylor Hobson µltra V 6.1.12.1	120	1	INTI	INTI/ PTB	

#### 5. **Artefacts and Measurement Instructions**

Artefacts are to be measured to the conditions described in Table 4. Any or all of the listed parameters for that artefact may be submitted. Parameters are calculated according to ISO 5436-1 [4] or ISO 4287 [2] with measurement conditions from ISO 4288 [3] tables 1 and 3 and ISO 3274 [1] table 1 and are to be determined on the filtered roughness profile with a Gaussian filter as defined in ISO 16610 [5].

Stylus measurements should be performed without a skid, a stylus tip radius of 2 µm, a traverse speed of  $\leq 0.2$  mm/s and a sample spacing of  $\leq 0.5$  µm. For non-contact instruments, approximate the conditions as appropriate. Possible variations that should be considered and noted in any report.

- Obvious scratches or flaws should be avoided in measurement and discarded from analysis.
- A profile length may include a lead-in or lead out which may be 1 cut-off or  $\frac{1}{2}$  cut-off at each end. Where possible, adjust the profile location to account for this.
- Some laboratories or instruments make mathematical adjustment to parameter values due to stylus radius which may be for a 2  $\mu$ m radius or for a theoretically zero tip size. Any corrections must be discussed with the pilot to ensure that submitted results are appropriate for comparison.
- $R_z$  is to be determined from ISO 4287-1 [1997]. If the instrument reports the old ten-point-height from ISO 4287 [1984], then parameter  $R_{y}$  from ISO 4287 [1984] should be determined.
- For  $RS_m$  ISO 4287 recommends a height discrimination of 10% of the  $R_z$  value to be used.

**IMPORTANT:** Handle only with gloves. Do not touch the measurement surfaces with bare hands. Only remove items from their protective cases at the location of measurement. If an item must be transferred across a room, return it to its case and only remove it at the required instrument work table.

In measurement, the contact tip MUST be raised OFF the surface at the end of each profile. Do NOT allow automatic return of the tip across the surface! Contact with the surface must be minimised.



#### Table 4. Artefacts.

 Table 4. Artefacts (continued).



#### 6. <u>Intercomparison Analysis (Coordinator)</u>

Analysis methods are consistent with the ISO *Guide to the Estimation of Uncertainty in Measurement* (GUM). When all measurements are completed, the reference value for each measured quantity is to be the weighted mean of the values  $x_i$  from each individual laboratory, as given by

Weighted mean: 
$$\overline{x}_{w} = \left[\sum_{i=1}^{N} x_{i} / u^{2}(x_{i})\right] / \left[\sum_{i=1}^{N} 1 / u^{2}(x_{i})\right]$$
(1)

To avoid bias, the first set of measurements only will be included from the pilot laboratory.

Assessment of each laboratory result will be made with the degree of equivalence ratio (E<sub>n</sub>):

$$E_n = \frac{x_i - \overline{x}}{2\sqrt{u^2(x_i) - u^2(\overline{x})}}$$
(2)

where  $u(x_i)$  is the standard uncertainty of the individual laboratory and u(x) is the standard uncertainty of the weighted mean.

Any result with an  $E_n > 1$  with respect to the reference value will be excluded and the reference value and  $E_n$  ratio recalculated. If more than one laboratory result is to be excluded this will be tested one result at a time.

The **Birge ratio** is a test of the consistency of a set of data and its uncertainties. If the measurements being compared come 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 in equations 3 and 4.

$$u_E^2(\bar{x}_w) = \left[\sum_{i=1}^N 1/u^2(x_i)\right]^{-1}$$
(3)

$$u_{I}^{2}(\bar{x}_{w}) = \left[\sum_{i=1}^{N} (x_{i} - \bar{x}_{w})^{2} / u^{2}(x_{i})\right] / \left[ (N-1) \sum_{i=1}^{N} 1 / u^{2}(x_{i}) \right]$$
(4)

For an infinite population size, the ratio  $R_B = u_E(\bar{x}_w)/u_I(\bar{x}_w)$  should approximate a value of 1. For a limited population size, the Birge criterion is given by:

$$R_B = \sqrt{1 + \sqrt{\frac{8}{N-1}}} \tag{5}$$

where N is the number of accepted measurements. The criterion is recalculated if a result is excluded.

#### 7. Draft and Final Report (Coordinator)

It is anticipated that the draft report will be sent to all participants within two months of the final measurements. Participants are expected to correct any mistakes in the draft with respect to their own submissions and review and comment on analysis and conclusions. After all discussions are complete, the final report will then be submitted to the APMP for publication following recommended international guidelines.

#### 8. <u>References</u>

- [1] ISO 3274 Surface Texture Instruments for the assessment of surface texture Profile method, International Organization for Standardization, Geneva, Switzerland, 1996.
- [2] ISO 4287 Geometrical Product Specifications (GPS) Surface texture: Profile method Terms, definitions and surface texture parameters, International Organization for Standardization, Geneva, Switzerland, 1997.
- [3] ISO 4288 Geometrical Product Specifications (GPS) Surface texture: Profile method Rules and procedures for the assessment of surface texture, International Organization for Standardization, Geneva, Switzerland, 1996.
- [4] ISO 5436-1 Geometrical Product Specifications (GPS) Surface texture: Profile method; Measurement standards Part 1: Material measures, International Organization for Standardization, Geneva, Switzerland, 2000.
- [5] ISO 16610 Geometrical Product Specifications (GPS) Surface texture: Profile method Metrological characteristics of phase correct filters, International Organization for Standardization, Geneva, Switzerland, 1996.
- [6] JCGM 100:2008 Evaluation of measurement data Guide to the expression of uncertainty in measurement.
- [7] M.G Cox, "The Evaluation of Key Comparison Data", Metrologia, 2002, 39, 589-595.
- [8] A.Baker, et.al., APMP.L-K8 International Comparison of Surface Roughness Final report January 2013 Measurements July 2008 to June 2010, Metrologia 50, January 2013

#### Appendix A: CUSTOMS DECLARATION

### TO WHOM IT MAY CONCERN

#### **APMP Regional Comparison**

The Asia Pacific Metrology Program (APMP) is an organisation representing the National Measurement / Standards Laboratories of a large number of countries/territories in the Asia-Pacific region. Its broad objective is to improve the measurement capabilities in the Asia-Pacific region by sharing facilities and experience in metrology. One very successful method used by the APMP is the comparison of calibrations performed by different laboratories on given artefacts. Successful completion of these intercomparisons adds confidence to the laboratories in the carrying out of measurements and leads to international acceptance of the measurements.

As part of a major intercomparison program, the APMP is conducting an intercomparison on surface roughness involving the highest level measurement laboratories in two rounds involving the following countries in order:

Round 1	Australia, Japan, South Korea, Chinese Taipei, Hong Kong, Thailand, Malaysia, India,
	Saudi Arabia (then return to Australia)
Round 2	Australia, China, Singapore, Indonesia, Argentina, Mexico, United Kingdom, Russia
	New Zealand (then return to Australia)

This program is coordinated by the National Measurement Institute of Australia.

Item	Item or	nly	With Individu	Approx.	
	Dimensions	Weight	Dimensions	Weight	Value
Taylor Hobson glass artefact s/n 18073	$75 \times 62 \times 12$	0.139 kg	$110\times92\times28$	0.265 kg	AU\$3000
Mahr glass artefact s/n 6820101	46 × 11	0.038 kg	$82 \times 60 \times 20$	0.055 kg	AU\$2000
Rubert steel artefact s/n P129	$63 \times 45 \times 3$	0.039 kg	$82 \times 60 \times 20$	0.054 kg	AU\$450
Rubert steel artefact s/n P316	$64 \times 50 \times 3$	0.042 kg	$82 \times 60 \times 20$	0.058 kg	AU\$550

The following 4 artefacts are circulated among the participants for calibration:

The purchase/manufacturing cost of the artefacts is approximately **AUS\$6,000**; however they have no commercial value (not for sale). They are meant solely for the calibration of national standards and **will be exported immediately after the calibration is complete.** Refer to the schedule on page 6 of the protocol.

The shipping case has dimensions of  $(440 \times 380 \times 200)$  mm and weighs approximately 4.0 kg when containing all artefacts, internal protective cases, collective box and documents.

We request that the artefacts are not handled or removed from the container/package. Please do not touch the artefacts with bare hands as this may contaminate the surfaces. If a Customs inspection is required then please contact the relevant contact for the region on pages 3 and 4 of this document so that he/she can be present and unpack the items in your presence.

#### Comparison Coordinator

Mr Andrew Baker National Measurement Institute 1/153 Bertie St, Port Melbourne, 3207 Australia Phone: +61 3 9644 4902 Email: andrew.baker@measurement.gov.au

### Appendix B: <u>Checklist of Instructions</u>

Participants are expected to perform the following actions in approximate order:

Act	ions	Done
Pri	or to receiving shipment	
1.	Have all resources and instrumentation ready for the scheduled month of measurement	
	and commit to carrying out measurements according to the schedule (Table 2).	
2.	Ensure funds are available for onward transport of the artefacts	
On	receipt of artefacts	
3.	Inspect the box on arrival for any damage. Take digital photographs of any damage.	
4.	Unpack the standards from the box.	
5.	Confirm the documents are in order (Carnet, customs and protocol).	
6.	Treat the artefacts as if they are fragile. At all times, handle only with gloves, do not	
	touch the surfaces with bare hands. Only remove items from their protective cases at the	
	location of measurement. If an item must be transferred across a room, return it to its	
	case and only remove it at the required instrument work table.	
7.	Examine the standards under magnification and glancing light and note any damage	
	including surface defects and scratches on the form in appendix C. If beneficial, take	
	digital photographs of any damage or scratches.	
8.	Remove any contamination by gentle blowing of clean air or nitrogen. If any debris	
	remains, try a clean soft-bristle brush. Finally, only if necessary, use a soft lint-free	
	tissue and residue free solvent such as isopar-E, ethanol or heptane.	
9.	Inform the pilot at <u>andrew.baker@measurement.gov.au</u> that the standards have been	
	received and any damage to the standards or the box.	
Me	asurement	
10.	Check the condition of a stylus prior to measurement of EVERY standard. If any	
	damage is apparent, do not use that stylus.	
11.	Measure the standards as directed by this document. Any stylus contact must be as	
	minimal as possible. Lift the stylus off the surface at the end of a profile measurement.	
	Do NOT return the stylus to its initial position in contact with the surface!	ļ
12.	If applicable, measure the stylus radius, cone angle and force for each stylus to be used.	
	Measure the system noise on the same range by measuring an optical flat or similar very	
10	smooth surface.	
13.	Reassess each standard after measurements are completed for any changes to surface	
	condition on the form Appendix C. Note any new damage or scratches. Inform the pilot	
C	of any change in condition to any of the standards.	L
	mpletion of measurements	
14.	If satisfied that measurements are acceptable, repack the standards with anti-rust paper	
	over the metal standards and a new satchel of desiccant in the box, one set of documents	
	the delivery address on the case to the part participant.	
15	Lies a suitable transport agent for forwarding the artificate to the part participant and	
13.	organise smooth movement through customs using appropriate documents	
16	Inform the pilot AND the next scheduled laboratory that the measurements have been	
10.	completed and the artefacts are in transit.	
17.	Prepare one or more typical calibration reports with appropriate uncertainty of	
- · ·	measurement calculations for each measured parameter. Send the summary of results	
	(Appendix D), uncertainty spreadsheets and calibration reports to the pilot within 6	
	weeks of receipt of the artefacts.	
18.	Agree not to discuss any measurement results prior to distribution of the draft report	
	(after completion of all measurements).	

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## Appendix C: Artefact Condition: BEFORE and AFTER measurements

[Print page to fill details. Take pictures as needed. When complete scan and email to pilot.]

Country/Laboratory:	Date of condition assessment:
---------------------	-------------------------------

Assessment before or after measurement: BEFORE / AFTER (circle)

Note damage to the shipping case or artefact box:





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## Appendix C: Artefact Condition BEFORE and AFTER measurements

Serial number P316												
Cleaning methods used:												
Comments:												
2												
		E B31										
	Measurement surface only											
Serial number	per P129											
Cleaning me	ethods used:											
Comments:												
	Measurement surface only P129											
	1 127											

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#### Appendix D: Results for Laboratory: \_\_\_\_\_

The table is provided in an Excel spreadsheet in the shown format with additional space for listing software, traceability and additional comments. The table assumes stylus measurement, however, if interferometric methods are used, please list details where considered influential. Please list all results in micrometers ( $\mu$ m) and ensure results are clear and comments are in English.

1. Country, Laboratory																
Artefact	Domona	Date of	Result	σ	uc	1.	U		Instrument	tip rad.	v	$l_n$	$\lambda_{c}$	Filter	force	
s/n	Palalli.	meas.	μm	μm	± µm	K	± µm	11	msuument	μm	mm/s	mm	mm	$\lambda_c/\lambda_s$	gf	
18073	d (A)												-	-	-	
	d (B)												-	-	-	
	R <sub>a</sub>															
	R <sub>z</sub>															
	R <sub>t</sub>															
	RS <sub>m</sub>															
6820101	d												-	-	-	
P316	R <sub>a</sub>															
	R <sub>q</sub>							-								
	Rz															
	R <sub>t</sub>															
P129	R <sub>a</sub>															
	Rz															
	R <sub>t</sub>															
	RS <sub>m</sub>															

Symbols  $\sigma$ : standard deviation, uc: standard uncertainty, k: coverage factor, U: expanded uncertainty, n: number of profiles tip rad: tip radius, v: speed of traverse, ln: profile evaluation length,  $\lambda c$ : cut-off length,  $\lambda c/\lambda s$ : filter ratio, gf: gram force.

## **End of Protocol.**