EURAMET Supplementary Comparison

EURAMET.L-S26.1

Measurement of groove depth standards in the range 5 µm up to 0.9 mm

Final report

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1 Document control

Version: Final report Issued on 28th March 2023.

2 Introduction

At its meeting in October 2015, the Consultative Committee for Length, CCL, decided upon a supplementary comparison on groove depth standards in the range 1 µm up to 1000 µm, named EURAMET.L-S26, with PTB as the pilot laboratory. The comparison was finished during 2020. This comparison is a follow-up comparison for S26, and the technical protocol has been adapted from that of S26. PTB serves as linking laboratory for S26 comparison.

The procedures outlined in this document cover the technical procedure to be followed during the measurements. A goal of the supplementary comparisons for topics in dimensional metrology is to demonstrate the equivalence of routine calibration services offered by NMIs to clients, as listed in Appendix C of the Mutual Recognition Agreement (MRA). To this end, participants in this comparison agree to use the same apparatus and methods as routinely applied to client artefacts.

The comparison was registered on 21st June 2021. Circulation started in July 2021 and ended in March 2022. The draft report was made available in April 2022.

3 Organization

3.1 Participants

Table 1. List of participant laboratories and their contacts.

Laboratory Code	Contact person, Laboratory	Phone, Fax, email
PTB	Jürgen Kirchhoff PTB Bundesallee 100, 38116 Braunschweig (Germany)	Tel. +49 531 592 5185 Fax +49 531 592 69 5111 e-mail: Juergen.Kirchhoff@ptb.de
VTT MIKES co-ordinator	Antti Lassila VTT MIKES Metrology VTT Technical Research Centre of Finland Ltd Tekniikantie 1, FI-02150 Espoo (Finland)	Tel. +358 40 514 8658 e-mail: antti.lassila@vtt.fi
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CEM	Laura Carcedo, Milagros Ozaita Micro and Nanometrology Standards Length and Precision Engineering Area CENTRO ESPAÑOL DE METROLOGÍA Alfar, 2, 28760- Tres Cantos (Madrid) (Spain)	Tel. +34 918 074 795 Fax +34 918 074 807 e-mail: lcarcedo@cem.es mmozaita@cem.es

3.2 Schedule

The timetable for measurements is shown in Table 2.

Table 2. Schedule of the comparison.

No	Laboratory	Country	Date of measurement	Results received
1	UME	Turkey	July 2021	19 August 2021
2	PTB	Germany	September 2021	28 September 2021
3	CEM	Spain	November 2021	31 March 2022
4	VTT MIKES	Finland	March 2022	18 March 2022

4 Artefacts

4.1 Description of artefacts

The package contains a PTB depth setting standard. The depth setting standards contain V-shaped grooves of type A1, approximately according to the standard ISO 5436-1:2000. The material is nickel coated copper (OFHC-Cu).

The coefficient of thermal expansion of copper given in Table 3 below was obtained from the manufacturer and should be used as such.

Table 3. List of artefacts.

Identification	Depths	Expansion coefficient	Manufacturer
EN 25_7	1/5/20/50/200/600/900 µm	$16.6 \pm 0.5 \times 10^{-6}$ K^{-1}	PTB

5 Measuring instructions

5.1 Handling and inspection of artefacts

The following instructions were delivered to participants regarding the handling of the artefacts:

> The depth setting standards should only be handled by authorized persons and stored in such a way as to prevent damage and contamination. Before the measurement is started, the standards must be inspected for damage or contamination using an optical microscope. Any scratches, dirty spots, or other damage must be documented with a photograph or drawing using the form provided in appendix B.
>
> No participant shall try to re-finish the measuring faces, and cleaning is only allowed using clean air or nitrogen; any other methods require permission from the pilot laboratory.
>
> Measurements may only be performed using equipment normally used to offer the relevant CMC service. The depth setting standards may not be given to any party other than the participants in the comparison.
>
> The depth setting standard should be examined before despatch and any change in condition during the measurement at each laboratory should be communicated to the pilot laboratory. Ensure that the content of the package is complete before shipment. Always use the original packaging.

Due to the small number of participants and significantly short time of circulation, the stability of the artefact was not checked after circulation. However, some longitudinal scratches were reported by partners.

5.2 Traceability

The following instructions were delivered to participants regarding the traceability of the measurements:

Depth measurements should be traceable to the latest realization of the metre as set out in the current "*Mise en Pratique*". Temperature measurements should be done using the International Temperature Scale of 1990 (ITS-90).

5.3 Measurands

The depth setting standard was measured based on the standard procedure that the laboratory regularly uses for this calibration service for its customers, but consistent where possible with the instructions given in 5.3.1.

Table 4. Laboratories measure following grooves.

No	Laboratory	Grooves to measure
1	VTT MIKES	5 µm, 20 µm, 50 µm, 200 µm, 600 µm, 900 µm
2	UME	200 µm, 600 µm, 900 µm
3	PTB	5 µm, 20 µm, 50 µm, 200 µm, 600 µm, 900 µm
4	CEM	600 µm, 900 µm

The PTB 900 µm depth setting standard EN25_7 (see Figure 1) contains seven grooves and two alignment grooves with a depth of 450 µm (s. Fig. 2). For instruments with a z-measurement range of only 1 mm, the alignment grooves allow alignment of the zero value in the z-direction by probing in one of the two alignment grooves.
Three parallel profiles ("a, b, c" s. Figure 3) in the middle of the standard separated by a distance of 1 mm shall be measured. This procedure is to be repeated twice. The measurand is the groove depth d, which must be determined for each groove profile according to ISO 5436-1 [3] (see Figure 2) but with a fixed groove width of 300 µm. The two profile sections in the upper surface between the marks 1 and 2 ("A") and between the marks 5 and 6 ("B") should have a distance from the upper groove edges of 0.1 mm (see Figure 2). The C profile section should have a length of 0.1 mm.
The arithmetic mean of all groove depths is the measurand to be reported (s. Appendix C).

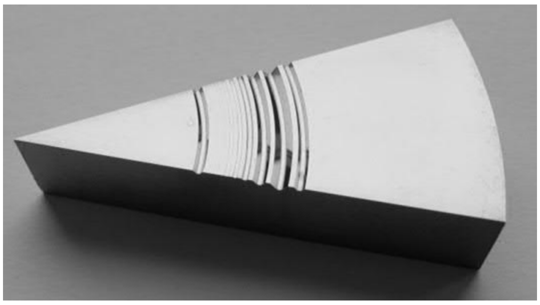

Figure 1. 900 µm depth setting standard.

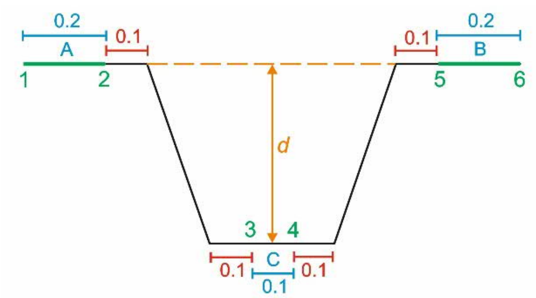

Figure 2. Evaluation of groove depth *d* according to ISO 5436-1 (dimensions in mm)

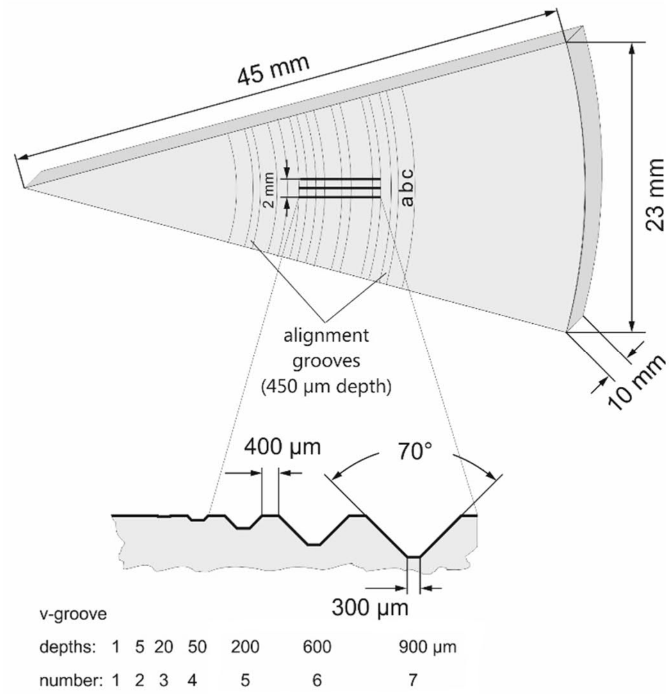

Figure 3. Depiction of the three profiles (a, b, c) to be measured on the 900 µm PTB standard, and location of the grooves on the standard

5.4 Measurement uncertainty

The uncertainty of measurement has been estimated according to the ISO *Guide to the Expression of Uncertainty in Measurement*. The participating laboratories were encouraged to use all known influence parameters for the method applied by them. The groove depth *d* of the standards is expressed as a function of the input quantities x_i

$$d = f(x_i) \tag{1}$$

The combined standard uncertainty $u_c(d)$ is the square sum of the standard uncertainties of the input quantities $u(x_i)$, each weighted by a sensitivity coefficient c_i

$$u_c^2(d) = \sum_i c_i^2 u^2(x_i) \text{ with } \qquad c_i = \frac{\partial d}{\partial x_i} \tag{2}$$

The participants were requested to report their measurement uncertainty budget in a table (s. appendix C) structured as follows:

Quantity X_i	Estimate x_i	Uncertainty $u(x_i)$	Probability distribution	Sensitivity coefficient c_i	Uncertainty contribution $u_i(d)$	Degrees of freedom ν_i

For the type of probability distribution please use: N = normal; R = rectangular; T = triangular; U = U-shaped.

5.5 Reference conditions

Measurement results were to be reported for the reference temperature of 20°C. For corrections, the linear thermal expansion coefficient provided in this document (Table 3) was to be used.

6 Results

6.1 Results and standard uncertainties as reported by participants

The results with associated uncertainties are given in Tables 5, 6, 7, 8, 9, and 10. For 5 µm, 20 µm, and 50 µm, where there were only two results a bilateral analysis was performed—i.e., calculation of the difference between the results of PTB and MIKES and the corresponding combined uncertainty of difference. For longer groove depths giving more than two results, the weighted mean and corresponding uncertainty was calculated. In both cases also *En* values were calculated.

Table 5. Results and analysis for 5 µm groove depth.

5 µm	x_i /µm	$u(x_i)$ /µm	$U(x_i)$ /µm	E_n	x_{i1}-x_{i2} /µm	$U(x_{i1}$-$x_{i2})$ /µm
PTB	5.005	0.012	0.023	0.42	0.011	0.026
VTT MIKES	4.994	0.006	0.013	-0.42	-0.011	0.026
Bilateral						

Table 6. Results and analysis for 20 µm groove depth.

20 µm	x_i /µm	$u(x_i)$ /µm	$U(x_i)$ /µm	E_n	x_{i1}-x_{i2} /µm	$U(x_{i1}$-$x_{i2})$ /µm
PTB	20.014	0.012	0.023	0.34	0.009	0.026
VTT MIKES	20.005	0.006	0.013	-0.34	-0.009	0.026
Bilateral						

Table 7. Results and analysis for 50 µm groove depth.

50 µm	x_i /µm	$u(x_i)$ /µm	$U(x_i)$ /µm	E_n	x_{i1}-x_{i2} /µm	$U(x_{i1}$-$x_{i2})$ /µm
PTB	49.965	0.012	0.024	-0.74	-0.020	0.027
VTT MIKES	49.985	0.006	0.013	0.74	0.020	0.027
Bilateral						

Table 8. Results and analysis for 200 µm groove depth.

200 µm	x_i /µm	$u(x_i)$ /µm	$U(x_i)$ /µm	E_n	x_i-x_w /µm	$U(x_i$-$x_w)$ /µm
PTB	199.936	0.015	0.030	0.18	0.005	0.028
VTT MIKES	199.929	0.006	0.013	-0.39	-0.002	0.005
UME	199.962	0.032	0.064	0.49	0.031	0.063
Reference value, x_w	199.931					
Uncert. of ref. value, $u(x_w)$	0.006					

Table 9. Results and analysis for 600 µm groove depth.

600 µm	x_i /µm	$u(x_i)$ /µm	$U(x_i)$ /µm	E_n	x_i-x_w /µm	$U(x_i$-$x_w)$ /µm
PTB	599.931	0.022	0.044	-0.45	-0.019	0.042
VTT MIKES	599.951	0.006	0.013	0.21	0.001	0.004
UME	600.030	0.052	0.104	0.77	0.080	0.104
CEM	599.937	0.032	0.064	-0.21	-0.013	0.063
Reference value, x_w	599.950					
Uncert. of ref. value, $u(x_w)$	0.006					

Table 10. Results and analysis for 900 µm groove depth.

900 µm	x_i /µm	$u(x_i)$ /µm	$U(x_i)$ /µm	E_n	x_i-x_w /µm	$U(x_i$-$x_w)$ /µm
PTB	899.943	0.028	0.055	-0.14	-0.008	0.054
VTT MIKES	899.951	0.006	0.013	0.13	0.000	0.003
UME	899.980	0.059	0.118	0.25	0.030	0.117
CEM	899.931	0.045	0.090	-0.22	-0.020	0.089
Reference value, x_w	899.951					
Uncert. of ref. value, $u(x_w)$	0.006					

7 Conclusion and discussion

According to the analysis, all reported results with associated uncertainties have a smaller |En| value than 1. Thus, these comparison results support the claimed uncertainties of the tested services.

After the comparison, several scratch marks were found on the nickel coated copper artefact EN25_7. This indicates that the contact pressure used for the measurements was too high for some participants. This could be caused by too high a probing force or too small a probing tip radius. Thus, partners should note that the actual probing force and actual tip radius should be measured during the measurements to ensure that the recommended values (2 µm tip radius and 0.7 mN probing force [4]) are not exceeded. The recently published German standard DIN 32567-3 "Production equipment for microsystems – Determination of the influence of materials on the optical and tactile dimensional metrology – Part 3: Derivation of correction values for tactile measuring devices" describes the methods for doing both.

7.1 *En* values with published CMCs

In the Table 11. is comparison of the results against published CMC values. Based on this analysis the results of the comparison support the CMCs of the NMIs participated.

Table 11. Comparison of the result against published CMCs.

Laboratory	Measurand	Uncertainty in comparison	*En*	CMC	CMC id	*En* w. CMC
PTB				*Q*[22 nm, 36E-06 *d*] Range: [0.01 to 5] mm	PTB/5.11/1	
	5 µm	0.023 µm	0.42	no CMC for this range	-	
	20 µm	0.023 µm	0.34	0.022 µm	PTB/5.11/1	0.41
	50 µm	0.024 µm	-0.74	0.022 µm	PTB/5.11/1	-0.91
	200 µm	0.030 µm	0.18	0.023 µm	PTB/5.11/1	0.22
	600 µm	0.044 µm	-0.45	0.031 µm	PTB/5.11/1	-0.62
	900 µm	0.055 µm	-0.14	0.039 µm	PTB/5.11/1	-0.20
VTT MIKES				no CMC for this service	-	
	5 µm	0.013 µm	-0.42	to be submitted	-	
	20 µm	0.013 µm	-0.34	to be submitted	-	
	50 µm	0.013 µm	0.74	to be submitted	-	
	200 µm	0.013 µm	-0.39	to be submitted	-	
	600 µm	0.013 µm	0.21	to be submitted	-	
	900 µm	0.013 µm	0.13	to be submitted	-	
UME				*Q*[33 nm, 13E-03 d] Range: [0.01 to 50] µm no CMC for this range	UME/34	
	200 µm	0.064 µm	0.49	to be submitted	-	
	600 µm	0.104 µm	0.77	to be submitted	-	
	900 µm	0.118 µm	0.25	to be submitted	-	
CEM				no CMC for this range		
	600 µm	0.064 µm	-0.21	to be submitted	-	
	900 µm	0.090 µm	-0.22	to be submitted	-	

8 References

[1] T. A. Hahn, Thermal Expansion of Copper from 20 to 800 K—Standard Reference Material 736, *J. Appl. Phys.*, Bd. 41, Nr. 13, S. 5096, 1970.
[2] T. Middelmann, A. Walkov, G. Bartl, und R. Schödel, Thermal expansion coefficient of single-crystal silicon from 7 K to 293 K, *Phys. Rev. B*, Bd. 92, Nr. 17, Nov. 2015.
[3] DIN EN ISO 5436-1 Geometrical product specifications - Surface texture: Measurement standards - Part 1: Material measures.
[4] ISO 3274:1996(en), Geometrical Product Specifications (GPS) — Surface texture: Profile method — Nominal characteristics of contact (stylus) instruments. https://www.iso.org/obp/ui/#iso:std:iso:3274:ed-2:v1:en.
[5] JCGM 100:2008, Evaluation of measurement data — Guide to the expression of uncertainty in measurement, https://www.bipm.org/documents/20126/2071204/JCGM_100_2008_E.pdf

Appendix A. Measurement methods of participants:

PTB

Make and type of instrument(s) (state probing force, tip radius and scanning speed and describe how you measured these):

Groove depths were measured with a TENCOR P17 stylus instrument.

Three times five parallel measuring sections were measured on the depth setting standard. The measuring sections have a distance of 50 µm and are aligned to the centre marking of the standard.

- stylus tip radius: 2 µm, 45°
- static measuring force: 20 µN
- traverse speed: 20 µm/s
- sampling spacing: 0.4 µm

Traceability path:

The traceability of the groove measurements is carried out via comparative measurements on a traceably measured reference standard of the same depth before and after the measurement of the standard.

The grooves of the reference standard up to 450 µm were measured with the traceable stylus instrument HRTS, the deeper grooves with the PTB gauge block comparator INKO 5. Both instruments trace back to the wavelength of light.

Description of measuring technique (including any corrections such as temperature, etc):

Each groove was measured three times with five parallel measuring sections. The measuring sections have a distance of 50 µm and are aligned to the centre marking of the standard.

Before and after measuring the grooves of the depth setting standard, the grooves of a reference standard were measured. All determined depths of these grooves were converted to groove depths at 20°C using the recorded temperatures and the temperature expansion coefficient. A specific correction factor for the individual grooves was determined from the reference measurement and was used to correct the groove depths of the depth setting standard to be measured.

Range of depth setting standard temperature during measurements & description of temperature measurement method:

The temperature was 21.9 ±0.3°C during the measurements. The temperature is recorded every second by two calibrated air temperature gauges and averaged every minute. Including warm-up time, the measurement time takes approx. 18h 30 min.

Relevant 95% CMC uncertainty claim for the service(s) related to this comparison topic (if existing)

PTB/5.11/1: Q[22 nm, 36E-06 *d*]

Appendix A. Measurement methods of the participants:

VTT MIKES

Make and type of instrument(s) (state probing force, tip radius and scanning speed and describe how you measured these)

The VTT MIKES instrument for groove depth measurement was a modified gauge block interferometer. The imaging arm of the VTT MIKES phase stepping gauge block interferometer [1] was modified to have larger magnification at CCD, see Figure A1. For each groove, the bottom of the V-groove and surrounding reference areas were masked as defined in ISO 5436 to be used for analysis of groove depth. Next, phase stepping was performed with red and green He-Ne wavelengths, and similar analysis as for gauge blocks was performed. An example of the interferogram is shown in Figure A2.

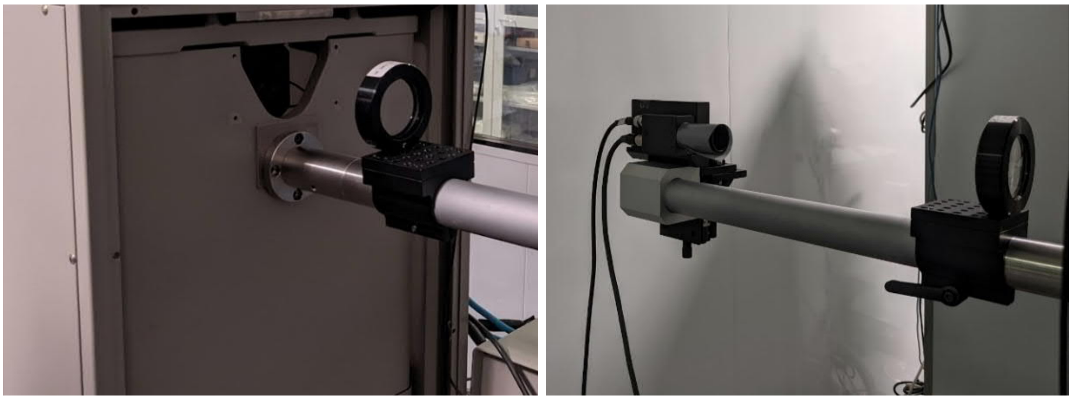

Figure A1. Modification to imaging arm of the VTT MIKES phase stepping gauge block interferometer.

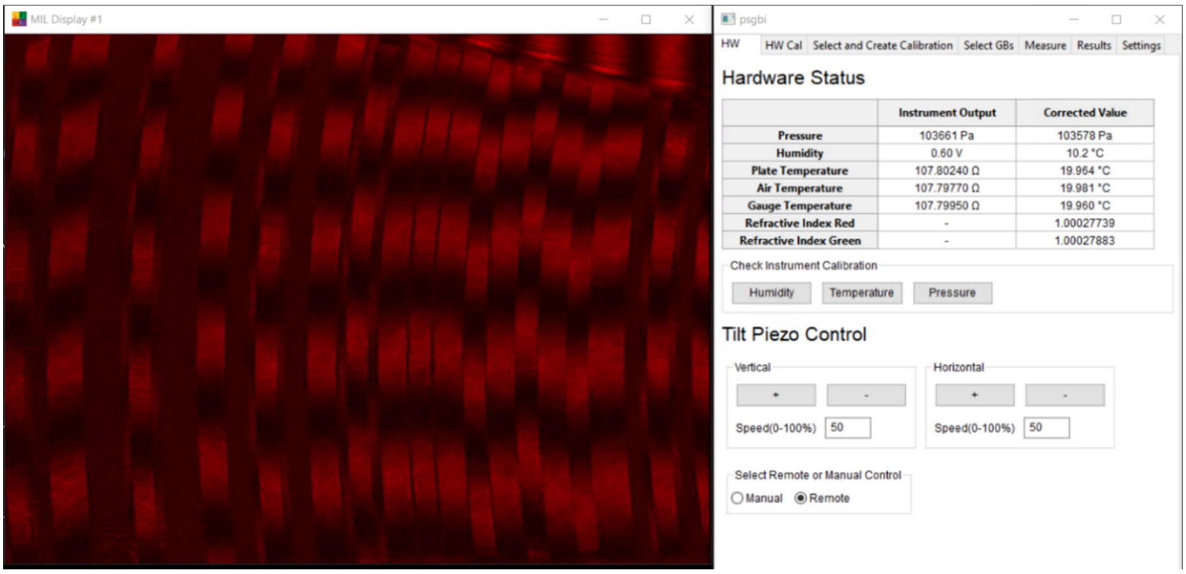

Figure A2. Example image of the interferogram from V-grooves of the transfer standard.

[1] Byman, V., & Lassila, A. (2015). MIKES' primary phase stepping gauge block interferometer. *Measurement Science and Technology*, *26*(8), [084009]. DOI 10.1088/0957-0233/26/8/084009

Traceability path:

The VTT MIKES gauge block interferometer has traceability to the SI-metre via calibrated vacuum wavelengths of stabilized green and red He-Ne lasers. Also, air pressure, humidity, material, and air temperature meters are calibrated against higher level Finnish national measurement standards.

Description of measuring technique (including any corrections such as temperature, etc):

The refractive index of air is determined using the updated Edlen formula[2]. The thermal expansion is corrected using the TEC provided.

Range of depth setting standard temperature during measurements & description of temperature measurement method:

The material temperature was 20 ±0.050°C during the measurements.

Relevant 95% CMC uncertainty claim for the service(s) related to this comparison topic (if existing)

No CMC claim with this instrument yet. To be submitted.

The uncertainty estimate of the V-groove measurements is given below.

	x_i	$u(x_i)$		Prob. dist	ν_i	$c_i = \delta l / \delta x_i$		$u_i(l)$ / nm	
l_{ab}	repeatability	8.4	nm	R	20	1		4.8	
$\delta\ l_{optics,L}$	error due to unideal optics and aligment, obliquity &	6.0	nm	N	30	1	L	6.0	L
$\delta\ l_{optics}$	error due to unideal optics and aligment, optics &	3	nm	N	30	1		3	
~~$d\ l_{optics}$~~	~~error due wringing variation~~	~~0.0~~	~~nm~~	~~N~~	~~30~~	~~1~~		~~0.0~~	
$I+\varphi$	length in fringes, uncertainty of fringe fraction	2.7	nm	N	30	1		2.7	
$\lambda_{or\ ,\ g}$	Vacuum wavelength of lasers	1.00E-08	rel	N	30	1E+09	L nm	10	L
n	Index of refraction of air, effect of equation	10	nm / m	N	30	1	L	10	L
t_{air}	Air temperature	6	mK	N	30	0.96	L nm/mK	6	L
dp	Dew point of air	0.2	K	N	30	30	L nm/K	6	L
p	Air pressure	7	Pa	N	30	2.70	L nm/Pa	19	L
x_{co2}	Air CO_2 concentration	30	ppm	N	30	0.15	L nm/ppm	5	L
α	Linear coeff. of thermal expansion	500	nm/m 1/K	R	30	0.100	L m K	29	L
Δt_s	Gauge temp. difference from 20°C	**20**	**mK**	**N**	**100**	**16.50**	**L nm/mK**	**330**	**L**
			degrees of freedom	103		standard unc. /nm	q[6.3;	332	L]
			95 %	k=2.0		expanded unc. /nm	Q[12.5;	659	L]

[2] Bönsch G and Potulski E (1998) Measurement of the refractive index of air and comparison with modified Edlen's formulae Metrologia 35 133–39. DOI 10.1088/0026-1394/35/2/8

Appendix A. Measurement methods of participants:

TÜBİTAK UME

Make and type of instrument(s) (state probing force, tip radius and scanning speed and describe how you measured these)

The stylus instrument is a MahrSurf XCR-20 (pick up is MFW-250 B, drive unit is GD 120, xy table is CT 300). The column of the instrument is not used. Instead, a drive unit is placed on a table with adjustable height to reduce vibration. Skidless measurements were always taken, and a short wavelength filter λ_s was not used at all in the comparison.
Stylus tip radius: 2 µm
Stylus tip angle: 60°
Sampling spacing: 0.1 µm
Scanning speed: 0.1 mm/s
Probing force: The static measuring force is given as 2.5 mN in the device manual. The force was attempted to be measured using a lever type force measurement device with 10 mN resolution. Since the measuring force is less than the resolution of the device, the analogue display value was estimated visually. The force is guaranteed not to exceed 4 mN.

Tip radius, tip angle, scanning speed, and sampling spacing are given in the device manual or as part of the software. We did not measure these specifications.

Traceability path:

A groove depth standard, model number 4090/03 (ISO 5436-1, Type A1), with eight grooves of depths varying from 1 µm to 900 µm was used as a reference depth standard for all measurements in the comparison. Grooves with nominal depths of 200 µm, 600 µm, and 900 µm were used to calculate calibration factors. The cross-sections of the grooves are trapezoid. The standard was calibrated at PTB, certificate number 50026 PTB 18 (dated 18th July 2018).

Description of measuring technique (including any corrections such as temperature, etc):

A profile method was performed to measure groove depths using a stylus instrument. Temperature correction was applied for all grooves.

Range of depth setting standard temperature during measurements & description of temperature measurement method:

A thermocouple was used to measure the temperature of the groove depth standards. The thermocouple was stitched to the standard. The max average temperature was 22.50°C and the minimum average temperature was 18.50°C during a single depth measurement. The temperature varied by max 0.17°C during a single depth measurement.

Relevant 95% CMC uncertainty claim for the service(s) related to this comparison topic (if existing)

UME/34: $U=Q[33\ \text{nm}, 13\text{E-}03\ d]$

Appendix A. Measurement methods of participants:

CEM

Make and type of instrument(s) (state probing force, tip radius and scanning speed and describe how you measured these)

The system used was a NanoMeasuring Machine with Laser Focus Sensor (SIOS); as this is an optical sensor, there are no probing force nor tip characteristics to report.

Measurements were made with sample spacing of 0.05 µm and 20 µm/s speed.

Analysis software to calculate *d*: Mountains Map 7.4

Traceability path:

Measured points on the standard surface to determine the depth of the grooves correspond to (x, y, z) coordinates of the NanoMeasuring Machine coordinate system. The position of the three coordinate axes was measured using three optical interferometers operating with stabilized lasers, whose traceability comes from the realization of the SI unit of length at CEM.

Description of measuring technique (including any corrections such as temperature, etc):

The EURAMET.L-S26.1 standard was measured using the NanoMeasuring Machine with a laser focus sensor manufactured by SIOS. Each point measured on the comparison standard surface was determined from the three coordinate axes of the system whose traceability relies on the realization of the SI unit at CEM.

All measurements were corrected with the expansion coefficients from the technical protocol and temperatures with CEM traceability. The mathematical model also includes corrections with zero value but non-zero uncertainties that come, apart from the measuring system, from the measurements on the standard (such as scattering, edge location, and software analysis) and from other effects such as groove depth gradient, out of flatness of the standard surfaces, and inequality of optical properties on top and bottom surfaces. A correction related to thermal inertia that could happen during measurements (and which is difficult to determine) has been added to the uncertainty.

Range of depth setting standard temperature during measurements & description of temperature measurement method:

Temperature was measured using a thermometer with Pt-100 temperature probes calibrated at CEM and installed in the chamber of the NanoMeasuring Machine. Temperature variations in the chamber during measurements have been taken into account for the corrections of the refractive index of air and the groove depths.

Relevant 95% CMC uncertainty claim for the service(s) related to this comparison topic (if existing)

CEM has not a CMC claim for 600 µm and 900 µm groove depths.

CEM standard uncertainty budget for 600 µm groove depths

Quantity X_i	Estimate x_i (µm)	Probability distribution	Sensitivity coefficient c_i	Uncertainty contribution $u_i(d)$ (µm)	Degrees of freedom v_i
System (traceability, thermal optics drift, resolution, noise)	0	R	1	0.009	∞
Measurements on the standard (scattering)	599.937	R	1	0.019	∞
Standard effects (gradient groove depth, inequality of optical surface proprieties, out of flatness surfaces)	0	R	1	0.006	∞
Measurement analysis (edge location, software)	0	R	1	0.001	∞
Rounding	0.0004			0.0004	
Inertial thermal expansion effect ± 1 °C	0.019			0.019	