Physikalisch-Technische Bundesanstalt
Braunschweig und Berlin

EURAMET Supplementary Comparison

EURAMET.L-S26

Measurement of groove depth standards in the range 1 µm up to 1 mm

EURAMET project 1407

Technical protocol

U. Brand (PTB)

Braunschweig, May 2016

Contents

1 Document control

Version Draft A.1 Issued on 31 May 2016.

2 Introduction

The metrological equivalence of national measurement standards and of calibration certificates issued by national metrology institutes is established by a set of key and supplementary comparisons chosen and organized by the Consultative Committees of the CIPM or by the regional metrology organizations in collaboration with the Consultative Committees.

The EURAMET Technical Committee for Length, TC-L, decided upon a supplementary comparison on groove depth standards in the range 1 µm up to 1 mm, named EURAMET.L-S-xx, with PTB as the pilot laboratory. The comparison will be registered in July 2016, and artefact circulation is planned to start in September 2016.

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

By their declared intention to participate in this supplementary comparison, laboratories accept the general instructions and to strictly follow the technical protocol of this document. Due to the large number of participants, it is very important that participating NMIs perform their measurements during assigned dates. Participants should keep in mind that the allocated time period is not only for measurements, but transportation and customs clearance as well. 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.

3 Organization

3.1 Participants

Table 1. List of participant laboratories and their contacts.

Laboratory Code	**Contact person, Laboratory**	**Phone, Fax, email**
PTB	Uwe Brand PTB Bundesallee 100, 38116 Braunschweig (Germany)	Tel. +49 531 592 5111 Fax +49 531 592 69 5111 e-mail: Uwe.Brand@ptb.de
BEV	Michael Matus Bundesamt für Eich- und Vermessungswesen (BEV) Arltgasse 35, 1160 Wien (Austria)	Tel. +43 1 21 110 6540 Fax +43 1 21 110 6000 e-mail: michael.matus@bev.gv.at
CEM	Laura Carcedo Surface Quality Laboratory. Length Area CENTRO ESPAÑOL DE METROLOGÍA Alfar, 2 - Tres Cantos 28760 Madrid Spain Milagros Ozaita	Tel. +34 918 074 716 Fax +34 918 074 807 e-mail: lcarcedo@cem.minetur.es mmozaita@cem.minetur.es

GUM	Zbigniew Ramotowski Central Office of Measures/Glówny Urzad Miar (GUM) ul. Elektoralna 2, P.O. Box 10, 00 950 Warszawa (Poland) Lukasz Slusarski GUM Laboratorium Pomiarów Przemysłowych ul. Elektoralna 2, 00-139 Warszawa (POLAND)	Tel. +48 22 581 9543 Fax +48 22 620 8378 e-mail: z.ramotowski@gum.gov.pl Tel. +48 22 581 93 18 e-mail: l.slusarski@gum.gov.pl
INRIM	Massimo Zucco Istituto Nazionale di Ricerca Metrologica (INRIM) Strada delle Cacce 91, 10135 Torino (Italy) Gian Bartolo Picotto	Tel. +39 011 39 19 968 Fax +39 011 39 19 959 e-mail m.zucco@inrim.it Tel. +39 011 39 19 969 e-mail: g.picotto@inrim.it
MIKES	Antti Lassila MIKES Metrology VTT Technical Research Centre of Finland Ltd Tekniikantie 1, FI-02150 Espoo (Finland)	Tel. +358 40 514 8658 Fax +358 20 722 7001 e-mail: antti.lassila@vtt.fi
NMISA	Oelof Kruger NMISA Private Bag X34, Lynnwood Ridge, Pretoria 0040 (South Africa)	Tel. +27 12 841 4340 Fax +27 12 841 2131 e-mail: oakruger@nmisa.org
SP	Sten Bergstrand SP Technical Research Institute of Sweden (SP) P.O. Box 857, 50115 Borås (Sweden)	Tel. +46 10 516 57 73 Fax +46 10 516 56 20 e-mail: sten.bergstrand@sp.se
UME	Okhan Ganioglu Murat Aksulu Muharrem Aşar UME - Ulusal Metroloji Enstitüsü Barış Mah. Dr. Zeki Acar Cad. No:1 41470 Gebze, Kocaeli (Turkey)	Tel. +90 262 679 50 00 ext. 5300 Fax +90 262 679 50 01 okhan.ganioglu@tubitak.gov.tr muharrem.asar@tubitak.gov.tr murat.aksulu@tubitak.gov.tr
VNIIMS	Vladimir Kosteev VNIIMS Russian Research Institute for Metrological Service 46, Ozernaya st. , Moscow 119361 (Russia)	Tel. +7 495 781 4506 Fax + e-mail: vkosteev@vniims.ru

3.2 Schedule

The participating laboratories were asked to specify a preferred timetable slot for their own measurements of the depth setting standards – the timetable given in table 2 has been drawn up taking these preferences into account. Each laboratory has six weeks that include customs clearance, calibration and transportation to the following participant. With its confirmation to participate, each laboratory is obliged to perform the measurements in the allocated period and to allow enough time in advance for transportation so that the following participant receives them in time. If a laboratory has technical problems to perform the measurements or customs clearance takes too long, the laboratory has to contact the pilot laboratory as soon as possible and, according to whatever it decides, it might eventually be obliged to send the standards directly to the next participant before completing the measurements or even without doing any measurements.

Table 2. Schedule of the comparison.

No	Laboratory	Country	Starting date of measurement
1	PTB	Germany	1 September 2016
2	VNIIMS	Russia	1 October 2016
3	NMISA	South Africa	1 December 2016
4	UME	Turkey	1 February 2017
5	PTB	Germany	1 April 2017
6	SP	Sweden	15 April 2017
7	MIKES	Finland	1 June 2017
8	BEV	Austria	15 July 2017
9	INRIM	Italy	1 September 2017
10	GUM	Poland	15 October 2017
11	CEM	Spain	1 December 2017
12	PTB	Germany	1 February 2018

3.3 Reception, transportation, insurance, costs

A plastic case containing four plastic cases with three SiMetrics depth setting standards and one PTB depth setting standard, respectively, is used for the transportation of the artefacts (Figure 1). Upon reception of the package, each laboratory has to check that the content is complete and that there is no apparent damage of the box or any of the standards. The reception has to be confirmed immediately to the pilot with a copy to the former participant (sender), using the form of Appendix A.

The organization costs will be covered by the pilot laboratory, which include the standards themselves, the cases and packaging, and the shipping costs to the next laboratory. The pilot laboratory has no insurance for any loss or damage of the standards during the circulation.

Fig. 1 – The standards to be measured, three SiMetrics and one PTB depth setting standard in a plastic case

Once the measurements have been completed, the package shall be sent to the following participant.

Each participating laboratory shall cover the costs of shipping and transport insurance against loss or damage. The package should be shipped with a reliable parcel service of its choice. Once the measurements have been completed, please inform the pilot laboratory and the following participant when the package leaves your installations indicating all pertinent information. If, at any point during circulation, the package is damaged, it shall be repaired by the laboratory before shipping it again. In the case that a laboratory or its shipping agent damages one or more artefacts, they may be required by the pilot to replace the artefacts at their own cost (or from the insurance).

The package is not accompanied by an ATA carnet. But a low value of 100 € can be used for customs purposes. Paying the customs taxes is in this case cheaper and easier to handle than the ATA carnet.

If a delay occurs, the pilot laboratory will inform the participants and if necessary revise the time schedule, or skip one participant and put them at the end of the circulation.

4 Artefacts

4.1 Description of artefacts

The package contains three SiMetrics and PTB depth setting standard. The depth setting standards contain v-shaped grooves of type A1 according to the standard ISO 5436-1:2000.

The coefficients of thermal expansion given in the following table are obtained by the manufacturers and should be used as such.

Table 3. List of artefacts.

Identification	Depth	Expansion coefficient /10^{-6} K^{-1}	Manufacturer
EN 19_7	600 µm, 200 µm, 900 µm	16.6 ± 0.5 [1]	PTB
SN 497	5 µm	2.56 ± 0.5 [2]	SiMetrics
SN 499	20 µm	2.56 ± 0.5 [2]	SiMetrics
SN 502	50 µm	2.56 ± 0.5 [2]	SiMetrics

5 Measuring instructions

5.1 Handling and inspection 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 have to be inspected for damages or contamination using an optical microscope. Any scratches, dirty spots or other damages have to be documented by a photograph or drawing using the form provided in appendix B.

Laboratories should attempt to measure all depth setting standards. No participant shall try to re-finish measuring faces and also cleaning is only allowed by clean air or nitrogen and any other methods are only allowed by previously contacting the pilot.

Measurements may only be performed using equipment normally used to offer the relevant CMC service. In case of multiple CMC services in this area, only the service/equipment with the smallest uncertainty should be used, unless the pilot and other participants agree to allow additional instruments to be used; in which case, only the results of the instrument/service with the smallest uncertainty may contribute to the key comparison reference value (KCRV). No other measurements are to be attempted by the participants and the depth setting standards should not be used for any purpose other than described in this document. The depth setting standards may not be given to any party other than the participants in the comparison.

The depth setting standards 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.

5.2 Traceability

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

5.3 Measurands

The depth setting standards shall be measured based on the standard procedure that the laboratory regularly uses for this calibration service for its customers.

5.3.1 900 µm PTB depth setting standard EN19_7

The PTB 900 µm depth setting standard (see Figure 1) contains seven grooves and two alignment grooves with a depth of 450 µm (s. Fig. 3). For instruments with a z-measurement range of only 1 mm the alignment grooves allow to align the zero value in z-direction by probing in one of the two alignment grooves.

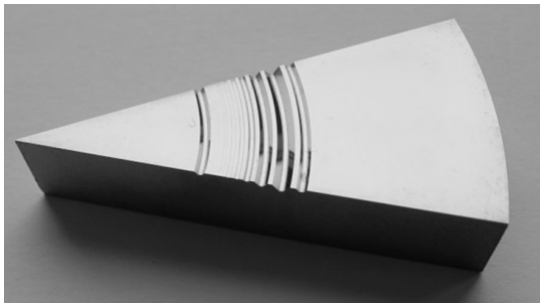


Fig. 2 Foto of a 900 µm PTB depth setting standard

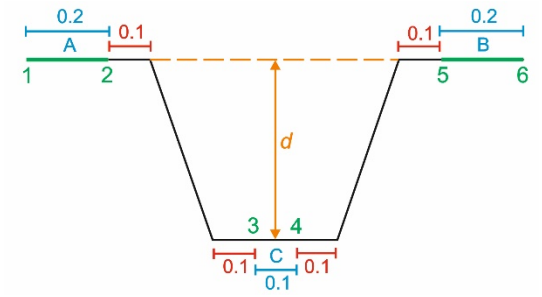


Fig. 3 Evaluation of the groove depth *d* according to ISO 5436-1 (dimensions in mm)

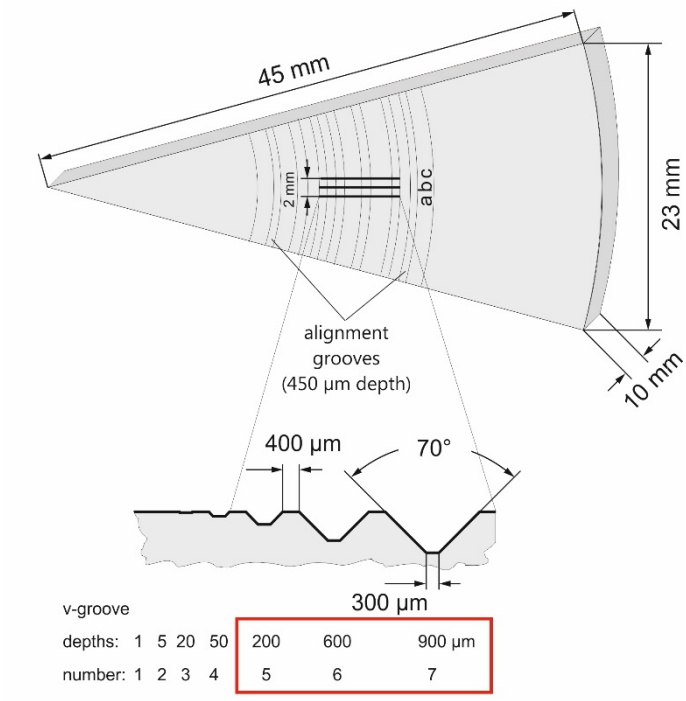


Fig. 4 Depiction of the three profiles (a, b, c) to be measured on the 900 µm PTB standard and the location of the grooves on the standard

Only the depths of the three grooves with the nominal depths of 200 µm, 600 µm and 900 µm have to be measured.
Three parallel profiles (“a, b, c” s. Fig. 4) in the middle of the standard separated by a distance of 1 mm shall be measured. This procedure has to be repeated two times in order to obtain nine profiles. The measurand is the groove depth *d* which has to be determined for each profile according to ISO 5436-1 [3] (s. Fig. 3) but with a fixed groove width of 300 µm. Thus for tilt correction the two profile sections 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. After tilt correction the groove depth *d* has to be determined as the mean value of the profile section “C” between the marks 3 and 4. This profile section should have a length of 0.1 mm.
The arithmetic mean of all nine groove depths is the measurand to be reported (s. Appendix C).
The ASCII data of the nine profiles measured for each groove have to be attached to the final report and the file names have to be listed in the report form in Appendix F.

5.3.2 SiMetrics depth setting standards

The SiMetrics depth setting standards consist of a 50 mm x 50 mm glass plate on which the depth setting standard silicon chips are bonded (s. Fig. 5 and 6).

Three parallel profiles (“a, b, c” s. Fig. 6) separated by a distance of 0.5 mm shall be measured. This procedure has to be repeated two times in order to obtain nine profiles. The measurand is the groove depth *d* which has to be determined for each profile according to ISO 5436-1 [3] (s. Fig. 7). For tilt

correction the two profile sections 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. After tilt correction the groove depth *d* has to be determined as the mean value of the profile section between the marks 3 and 4. This profile section “C” has a width of one third of the groove width *w*. The following groove widths *w* should be used:

Table 4. List of groove widths for the evaluation of the groove depth

Depth setting standard	nominal groove depth µm	groove width *w* / mm	*length of “C” w/3* mm
SN 497	5	1.772	0.591
SN 499	20	1.749	0.583
SN 502	50	1.708	0.569

The arithmetic mean of the three groove depths for each section (“a, b, c”) is the measurand to be reported (s. Appendix D).
The ASCII data of the nine profiles measured for each groove have to be attached to the final report and the file names have to be listed in the report form in Appendix F.

Fig. 5 The SiMetrics depth setting standards consist of a glass plate with the bonded silicon chip on it and an engraved serial number

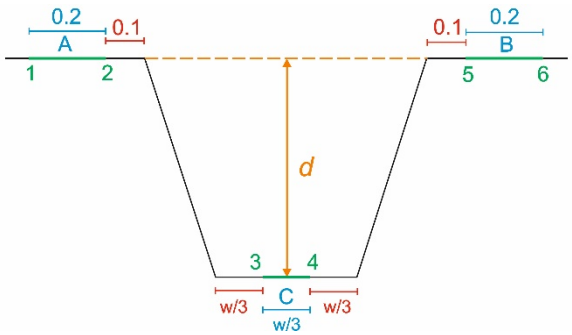


Fig. 7 Evaluation of the groove depth *d* for the SiMetrics grooves according to ISO 5436-1 (dimensions in mm)

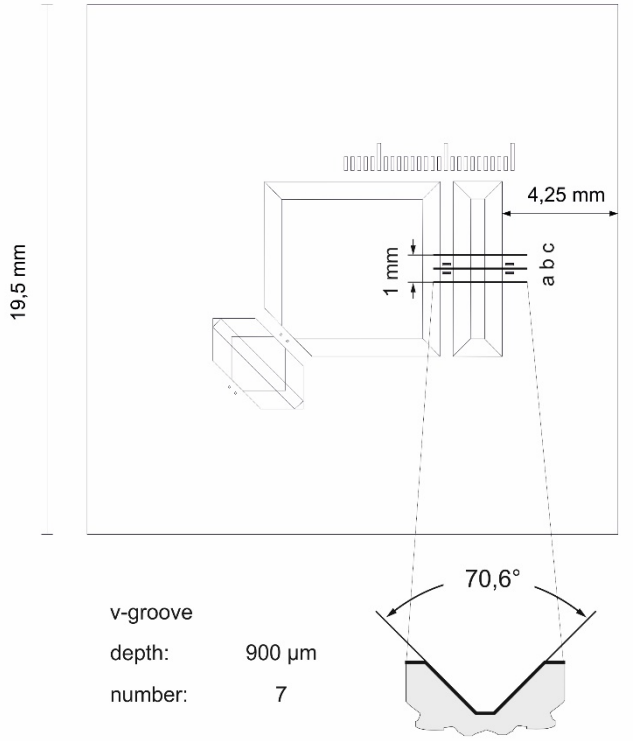


Fig. 6 Depiction of the SiMetrics depth setting standards and the location of three profiles (a, b, c) to be measured

5.4 Measurement uncertainty

The uncertainty of measurement shall be estimated according to the ISO *Guide to the Expression of Uncertainty in Measurement*. The participating laboratories are 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

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

The participants are requested to report their measurement uncertainty budget in a table (s. appendix C and D) whose format corresponds with the scheme below:

quantity X_i	estimate x_i	uncertainty $u(x_i)$	probability distribution	sensitivity coefficient c_i	uncertainty contribution $u_i(h)$	degrees of freedom v_i

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

5.5 Reference conditions

Measurement results should be reported for the reference temperature of 20 °C. For corrections the linear thermal expansion coefficient provided in this document (table 3) should be used.

6 Reporting of results

6.1 Results and standard uncertainties as reported by participants

As soon as possible after measurements have been completed, the results should be communicated to the pilot laboratory **within six weeks** at the latest.

The measurement report forms in appendix C and D of this document will be sent by e-mail (Word 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 pilot. In any case, the signed report must also be sent in paper form by mail or electronically as a scanned pdf document. In case of any differences, the signed forms are considered to be the definitive version.

Following receipt of all measurement reports from the participating laboratories, the pilot laboratory will analyse the results and prepare within one month a first draft A.1 report on the comparison. This will be circulated to the participants for comments, additions and corrections.

7 Analysis of results

7.1 Calculation of the KCRV

The distribution of the measurands is assumed to be normal for the comparison. The key comparison reference value (KCRV) is calculated on a standard-per-standard basis as the weighted mean of the participant results. The check for consistency of the comparison results with their associated uncertainties will be made based on Birge ratio [4], the degrees of equivalence for each laboratory and each depth setting standard with respect to the KCRV will be evaluated using E_n values [5], along the lines of the *WG-MRA-KC-report-template* [6]. To set up the $|E_n| \leq 1$ criterion, we will use the expanded uncertainty U with a coverage factor of $k = 2$. Measurements with E_n values larger than 1 will be omitted one by one for the calculation of the reference value. All other values contribute to the reference value. This means the evaluation starts with the whole data set and successive removal of those measurement data (E_n>1) with the largest E_n value. After each removal a new reference value and its uncertainty will be recalculated. This iteration stops when there is no data with E_n>1.

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] R. T. Birge, „Probable Values of the General Physical Constants“, *Rev. Mod. Phys.*, Bd. 1, Nr. 1, S. 1–73, 1929.

[5] W. Woeger, „Remarks on the En-Criterion used in measurement comparisons“, *PTB Mitteilungen*, Bd. 109, S. 24 – 27, 1999.

[6] „Guide to preparation of Key Comparison Reports in Dimensional Metrology - WG-MRA-KC report template“. [Online]. Verfügbar unter: http://www.bipm.org/wg/CCL/CCL-WG/Allowed/General_CCL-WG_docs/CCL-WG-MRA-GD-3-v1.5.doc. [Zugegriffen: 18-Dez-2015].

Appendix A – Reception of Standards

To:	Uwe Brand, PTB Bundesallee 100, 38116 Braunschweig (Germany) Fax: +49 531 592 69 5111 e-mail: uwe.brand@ptb.de		
From:	NMI: ……	Name: ……	
	Signature: ……	Date: ……	

We confirm having received the four depth setting standards for the EURAMET L.S-xx comparison on the date given above.

After a visual inspection:

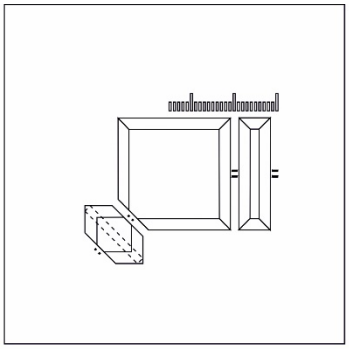
☐ There are no apparent damages; their precise state will be reported in the form provided in Annex B once inspected in the laboratory along with the measurement results.

☐ We have detected severe damages putting the measurement results at risk. Please indicate the damages, specifying every detail and, if possible, include photos. If it is necessary use additional sheets to report it.

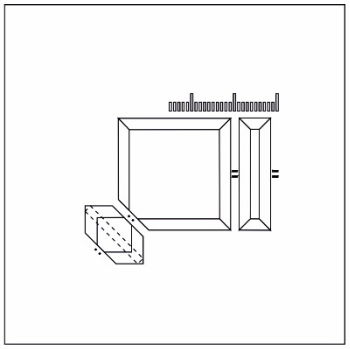
Appendix B – Conditions of Measuring Faces

To:	Uwe Brand, PTB Bundesallee 100, 38116 Braunschweig (Germany) Fax: +49 531 592 69 5111 e-mail: uwe.brand@ptb.de	
From:	NMI: ……	Name: ……
	Signature: ……	Date: ……

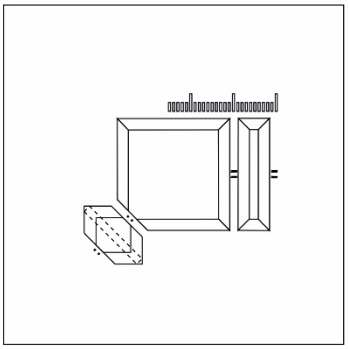
After detailed inspection of the measuring faces of the depth setting standards these are the results. Please mark significant surface faults (scratches, indentations, contamination, etc.).



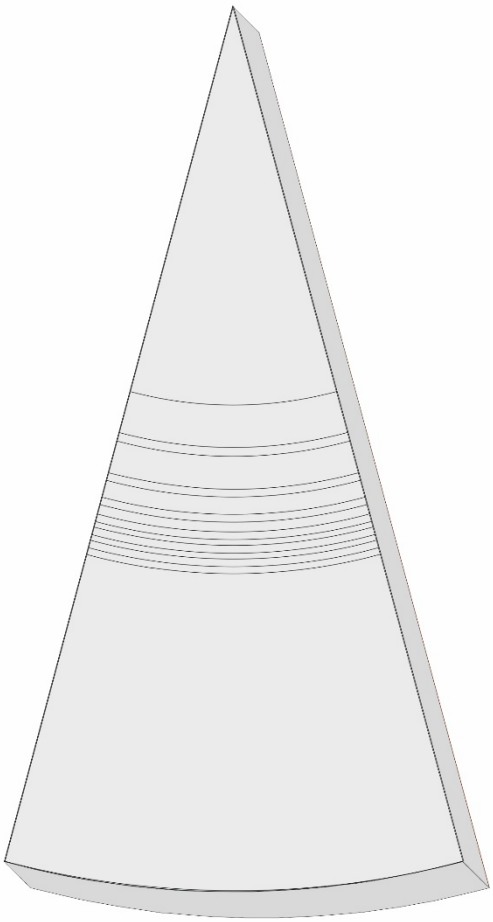
SN 497



SN 499



SN 502



EN19_7

Appendix C – Results Report Form

To:	Uwe Brand, PTB Bundesallee 100, 38116 Braunschweig (Germany) Fax: +49 531 592 69 5111 e-mail: uwe.brand@ptb.de
From:	NMI: …… Name: …… Signature: …… Date: ……

Depth setting standard	nominal groove depth µm	groove depth d / µm	standard deviation σ_{n-1}/ µm	standard uncertainty $u(d)$ / µm	ν_{eff}
EN19_7	200				
	600				
	900				

Standard uncertainty budget

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.

Appendix D – Results Report Form

To:	Uwe Brand, PTB Bundesallee 100, 38116 Braunschweig (Germany) Fax: +49 531 592 69 5111 e-mail: uwe.brand@ptb.de
From:	NMI: …… Name: …… Signature: …… Date: ……

Depth setting standard	nominal groove depth µm	groove depth d / µm	standard deviation σ_{n-1}/ µm	standard uncertainty $u(d)$ / µm	ν_{eff}
SN 497	5				
SN 499	20				
SN 502	50				

Standard uncertainty budget

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.

Appendix E – Description of the measurement instrument

To:	Uwe Brand, PTB Bundesallee 100, 38116 Braunschweig (Germany) Fax: +49 531 592 69 5111 e-mail: uwe.brand@ptb.de
From:	NMI: …… Name: …… Signature: …… Date: ……

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

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Traceability path: .....

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

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

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

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(use additional pages as needed)

Appendix F – ASCII Measurement Data

To:	Uwe Brand, PTB Bundesallee 100, 38116 Braunschweig (Germany) Fax: +49 531 592 69 5111 e-mail: uwe.brand@ptb.de
From:	NMI: …… Name: …… Signature: …… Date: ……

Depth setting standard	nominal groove depth µm	profile measured	ASCII file names		
			first meas.	second meas.	third meas.
SN 497	5	a			
	5	b			
	5	c			
SN 499	20	a			
	20	b			
	20	c			
SN 502	50	a			
	50	b			
	50	c			
EN19_7	200	a			
	200	b			
	200	c			
EN19_7	600	a			
	600	b			
	600	c			
EN19_7	900	a			
	900	b			
	900	c			