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1	Verification of Knoop indenters with a Vickers-addressed optical
2	system
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#### 25 Abstract

ISO 4545-2 and 4545-3 of Knoop hardness tests require the geometrical verification of the indenters. IN-RiM hardness laboratory, in cooperation with Galileo-LTF®, has developed the Gal-Indent optical measuring system for the verification of Vickers indenters. This system can measure the vertex angles between two op-posite faces, the quadrilateral base angles and the pyramid axis tilt angle. Using these measured quantities as inputs of a suitable geometrical model, the angles between the opposite edges at the vertex of Knoop indenters, nominally 172.5° and 130°, and the angle between the pyramid and indenter holder axes can be verified with an expanded uncertainty of 0.05°. Comparison of experimental measurements performed on three different Knoop indenters, previously verified by an accredited laboratory, shows compatible results. The proposed geometrical model could be easily implemented by laboratories that adopt similar measuring systems ad-dressed for the verification of Vickers indenters without any modification of the experimental apparatus. Keywords: Hardness, Knoop indenter, Geometrical model, Gal-indent optical system. 

#### 50 1. Introduction

51 Knoop indenter is a pyramidal diamond with a robic quadrilateral base that produces an elongated indent. 52 The angles between the opposite edges at the vertex of the diamond pyramid of the indenter,  $\alpha$  and  $\beta$ , are 53 172.5° and 130°, respectively, and the ratio between long and short diagonals is approximately 7.11 to 1 (Fig. 54 1). This entails that the angles of the rhombic base,  $\varphi_i$  and  $\tau_i$  (*i*=1,2), are approximately 164° and 16°, respec-55 tively, and that the angles between the two opposite faces of the vertex  $\theta$  are approximately 129.57°.

56 These characteristics make Knoop hardness ideal for testing surface defects, brittle materials and small 57 specimens, including thin metal films [1]. Moreover, due to the sensitivity of Knoop hardness to the indenter 58 orientation, it is useful to evaluate the anisotropy of materials [2]. The influence of indenter characteristics on 59 hardness measurements is largely reported in literature in particular for Vickers and Rockwell hardness. By 60 performing the analysis of variances on a large set of Vickers and Rockwell tests, it was found that the geom-61 etry of the indenters was statistically significant in most of the cases [3] producing a relevant uncertainty 62 contribution [4]. In Rockwell hardness, besides tip radius and cone angle, this can also be due to roughness, 63 indenter deformations under load [5] or to the soldering of the diamond cone into the holder [6]. In addition, 64 it was found that increasing the cone angle and the tip radius of the indenter entails an increase in Rockwell 65 hardness value [7]. In Vickers hardness, on the contrary, it was found that an indenter with a larger angle, 66 although within the limits allowed by the ISO standard, entails a wider indentation, thus a decrease in hardness 67 value [8], which might exceed permissible values [9]. However for Knoop hardness, few studies on the influ-68 ence of the indenter geometry are found. One showed that small geometric imperfections of the indeters have 69 a negligible influence on the contact area but a noticeable influence on the force-depth response [10]. Others 70 showed that the influence of indenter geometry on hardness may not be negligible when testing enamels [11] 71 and cobalt-based alloys [12]. Furthermore, in depth-sensing indentation, it was shown that the geometry of 72 Knoop indenter affects the evaluation Young's modulus of the indented material [13], while, in nanoindenta-73 tion, non-geometrically perfect Knoop indenters provide accurate results even at the very low loads at which 74 a nanoindenter operates [14].

75 ISO 4545-2 and 4545-3 [15,16] specify the requirements of the indenters with different tolerances. The 76 second is more restrictive since it refers to the calibration of reference blocks. The tolerance for the angle of

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172.5° is  $\pm 0.1^{\circ}$  in both documents, whereas for the angle of 130° the tolerance is  $\pm 1^{\circ}$  and  $\pm 0.1^{\circ}$ , respectively. Furthermore, the angle  $\delta$  between the axis of the diamond pyramid and the axis of the indenter holder (normal to the seating surface), named tilt angle, shall not exceed 0.5° and 0.3°, respectively. The four faces of the diamond pyramid shall also be polished and free from surface defects and the indenter constant  $c=\tan(\beta/2)/(2\tan(\alpha/2))$  shall be within 1,0% of the ideal value 0.07028, i.e. 0.06958  $\leq c \leq 0.07098$ . In addition, the device used for the verification shall have a maximum expanded uncertainty of 0.07°.

83 At present, verification of Knoop hardness is performed by few manufacturers and laboratories that own 84 specific instrumentation addressed for the scope. Conversely, verification of squared-based Vickers indenters, 85 based on the measurement of the angles between the opposite faces of the vertex, the squareness of the quad-86 rilateral base angles and the angle between the axis of the diamond pyramid and the axis of the indenter holder, 87 can be performed by a larger number of calibration laboratories and industries with dedicated systems. Given 88 the similar geometry of the two indenters, in this paper it is investigated the possibility to use Vickers-ad-89 dressed systems for the verification of Knoop indenters by implementing a simple geometrical model, in order 90 to extend the measurement capability of these laboratories without changing the experimental apparatus or 91 developing new ones.

92 Verification of Vickers indenters is usually performed with optical measuring systems using scanning in-93 terferometry [17], microscopes [18] or scanning confocal probes [19]. INRiM hardness laboratory, in cooper-94 ation with Galileo-LTF® [20,21], has developed a specific optical measuring system (commercialized by the 95 Galileo-LTF<sup>®</sup> as Gal-Indent) for the verification of the geometry of Vickers indenters [22]. This system is 96 able to directly measure the main geometrical parameters of Vickers indenters required by the standard, i.e. 97 the two vertex angles between two opposite faces, the four angles of the square base, and the angle between 98 the axis of the diamond pyramid and the axis of the indenter holder, with an expanded uncertainty of  $0.05^{\circ}$ . 99 By measuring these quantities for Knoop indenters and with a suitable geometrical model, the possibility to 100 evaluate their geometrical parameters required by the relevant Standards is investigated. This paper deals with 101 a brief description of the Gal-Indent optical system (Section 2), the geometrical model (Section 3) and a com-102 parison of experimental results among three different Knoop indenters with values obtained by a German ac-103 credited laboratory to validate the proposed method (Section 4).





Fig. 1. Schematic draw (left) [23] and picture (right) of a Knoop indenter.

#### 107

2.

The Gal-Indent optical system

108 In INRiM hardness laboratory a specific measuring system, commercialized by Galileo-LTF® as Gal-In-109 dent optical system (Fig. 2), was developed and is currently used for the verification of Vickers indenters. The 110 system is also adopted by different National Metrological Institutes (NMIs) and calibration laboratories around 111 the world. It is able to measure the vertex angle of the indenter between two opposite faces, and the quadrilat-112 eral base angles by means of two angular encoders [24]. The optical system is based on Mirau interferometry. 113 A green laser beam with a wavelength of 546 nm, emitted by a light source, is divided in two beams: the first 114 reaches the observer through the eyepiece and the second strikes the surface of the indenter and is reflected 115 back creating an interference pattern. Through a mechanical system, the indenter is simultaneously rotated 116 around the indenter-holder axis and around the axis parallel to the plane of the microscope lens passing through 117 the diamond pyramid vertex until the number of interference fringes is minimized, thus obtaining a lateral 118 indenter face parallel to the microscope lens. These two rotations are measured by means of two angular en-119 coders. The first rotation, around the indenter-holder axis, represents the measurement of quadrilateral base 120 angles. The second rotation around the axis parallel to the plane of the lens represents the measurement of the 121 supplementary angles of each lateral face from which the angles between two opposite faces and the angle 122 between the axis of the diamond pyramid and the axis of the indenter holder are obtained [25], as required for the verification of Vickers indenters. Using these measurements as input of a suitable geometrical model, presented in the following Section, the possibility to evaluate the geometrical parameters of Knoop indenters is investigated.

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127

128

Fig. 2. The Galileo-LTF® Gal-Indent optical system.

#### 129 **3.** The geometrical model

#### 130 *3.1 Evaluation of the tilt angle*

131 The geometry of an ideal Knoop indenter, i.e. an indenter with four generic faces (A, B, C, D), with angles 132 between the opposite edges at the vertex of the diamond pyramid  $\alpha$  and  $\beta$  equal to 172.5° and 130°, respec-133 tively, with angles  $\theta$  between two opposite faces equal to 129.57°, and with a tilt angle  $\delta$  equal to 0°, is sche-134 matically depicted in Fig. 3. xvz and x'y'z' coordinate systems correspond, respectively, to the diagonals of the 135 Knoop indenter rhombic base, and to the optical reference system that is perpendicular to the perimeter of two 136 opposite faces. Therefore, the angle between x- and y-axis is nominally 90°, whereas the angle  $\sigma_{AB}$  between x'-137 and y'-axis is nominally 164°. For each *j*-th face (*j*=A, B, C, D), the intersection between an optical reference 138 axis (x'- or y'-axis) and the base perimeter is identified by point  $H_i$ , whereas the intersection with x- and y-axis 139 are identified by points  $S_i$  and  $P_i$ , respectively (thus  $S_A \equiv S_D$ ,  $S_B \equiv S_C$ ,  $P_A \equiv P_B$ ,  $P_C \equiv P_D$ ). The pyramid vertex V is 140 arbitrally placed on z=1. A cross-section of an ideal Knoop indenter along x'z' optical system plane is also 141 shown in Fig. 4. The quadrilateral base angles  $\varphi_i$  and  $\tau_i$  (i=1,2), nominally 164° and 16°, respectively, and the 142 supplementary angles of each *j*-th lateral face (A, B, C, D) along x' and y'-axis,  $\omega_j$ , nominally 143 (180°-129.57°)/2~25.22°, are measured by means of the optical system previously described.







Fig. 5. 3-D schematic representation of a real Knoop indenter rhombic-based pyramid.

From quadrilateral base angles measurements  $\varphi_i$  and  $\tau_i$  (*i*=1,2), in order to take into account possible asymmetries of the rhombic-base, the mean angle  $\rho_j = \widehat{H_j OS_j}$  between *xy* and *x'y'* reference systems (e.g., in Fig. 5 for face A between *x'*- and *x*-axis), for each *j*-th indenter face, can be evaluated according to

159

$$\rho_j = \frac{\left(90 - \frac{\varphi_j}{2}\right) + \frac{\tau_j}{2}}{2}$$
(1)

160 where  $\varphi_{A}=\varphi_{D}=\varphi_{1}$ ,  $\varphi_{B}=\varphi_{C}=\varphi_{2}$ ,  $\tau_{A}=\tau_{B}=\tau_{1}$ ,  $\tau_{C}=\tau_{D}=\tau_{2}$ .

161 In this way, considering the mean value  $\rho = \sum_{j=1}^{4} \rho_j / 4$  among the four faces, the angle  $\sigma_{AB}$  between x'- and 162 y'-axis can be obtained according to Eq. (2).

$$\sigma_{AB} = 180 - 2\rho \tag{2}$$

From the measurement of the supplementary angles  $\omega_j$  of each *j*-th lateral face, the two vertex angles  $\theta_{x'}$ and  $\theta_{y'}$  and the pyramid tilt angles  $\delta_{x'}$  and  $\delta_{y'}$ , along *x'*- and *y'*- axis, can be calculated according to Eqs. (3) and (4), respectively. In Fig. 6, the cross-section of a real Knoop indenter through x'z' plane with vertex angle  $\theta_{x'}$ and pyramid tilt angle  $\delta_{x'}$  is depicted.

167

$$\theta_{x'} = 180 - (\omega_{\rm A} + \omega_{\rm C}); \qquad \theta_{y'} = 180 - (\omega_{\rm B} + \omega_{\rm D})$$
(3)

168

$$\delta_{\chi\prime} = \frac{\omega_{\rm A} - \omega_{\rm C}}{2}; \quad \delta_{\gamma\prime} = \frac{\omega_{\rm B} - \omega_{\rm D}}{2} \tag{4}$$





**Fig. 6.** Cross-section of a real Knoop indenter along x'z' optical reference system plane.

171

By projecting the pyramid tilted axis vector **v** along non-orthogonal x'z' and y'z' planes, according to Fig. 7 and Fig. 8, Eq. (5) is derived. Successively, implementing the equations of non-orthogonal systems (Fig. 9) and using Eq. (5), Eqs. (6)-(8) and Eqs. (9)-(10) can be derived.





178

Fig. 7. Projection of Knoop indenter axis  $\mathbf{v}$  along x'- and y'- axis.



**Fig. 8.** Upper view of a real Knoop indenter with axis vector  $\mathbf{v}$  and its projections along x'- and y'- axis. 



$$\sin\delta\cos\gamma = \frac{\cos\delta}{\cos\delta_{x'}}\sin\delta_{x'} + \frac{\cos\delta}{\cos\delta_{y'}}\sin\delta_{y'}\cos\sigma_{AB}$$
(7)

$$\tan \delta \cos \gamma = \tan \delta_{x'} + \tan \delta_{y'} \cos \sigma_{AB} \tag{8}$$

$$\|\mathbf{v}\|\sin\delta\sin\gamma = \|\mathbf{v}_{\mathbf{y}'\mathbf{z}'}\|\sin\delta_{\mathbf{y}'}\sin\sigma_{AB} = \|\mathbf{v}\|\frac{\cos\delta}{\cos\delta_{\mathbf{y}'}}\sin\delta_{\mathbf{y}'}\sin\sigma_{AB}$$
(9)

$$\tan\delta\sin\gamma = \tan\delta_{\gamma'}\sin\sigma_{AB} \tag{10}$$

187 By performing the squared sum of Eqs. (8) and (10), the total tilt angle  $\delta$  can be obtained (see Eqs. (11)-(13)): 188

$$\tan^2 \delta \cos^2 \gamma + \tan^2 \delta \sin^2 \gamma = \tan^2 \delta_{x'} + \tan^2 \delta_{y'} \cos^2 \sigma_{AB} + 2 \tan \delta_{x'} \tan \delta_{y'} \cos \sigma_{AB} + \tan^2 \delta_{y'} \sin^2 \sigma_{AB}$$
(11)

$$\tan^2 \delta = \tan^2 \delta_{\chi'} + \tan^2 \delta_{y'} + 2 \tan \delta_{\chi'} \tan \delta_{y'} \cos \sigma_{AB}$$
(12)

$$\delta = \arctan \sqrt{\tan^2 \delta_{x'} + \tan^2 \delta_{y'} + 2 \tan \delta_{x'} \tan \delta_{y'} \cos(\sigma_{AB})}$$
(13)

and from the ratio between Eqs. (10) and (8), the angle  $\gamma$  can be derived (see Eqs. (14)-(15)):

$$\tan \gamma = \frac{\tan \delta_{y'} \sin \sigma_{AB}}{\tan \delta_{x'} + \tan \delta_{y'} \cos \sigma_{AB}}$$
(14)

$$\gamma = \arctan\left(\frac{\tan \delta_{y'} \sin \sigma_{AB}}{\tan \delta_{x'} + \tan \delta_{y'} \cos \sigma_{AB}}\right)$$
(15)

190

### 191 *3.2 Evaluation of the angles between the opposite edges at the vertex*

192 From the scheme of Fig. 5 and reminding that the pyramid vertex V is placed on z=1, the vector of the tilted 193 pyramid axis **v** referred to the *xyz* reference system can be written according to Eq. (16), where  $\rho$  is the mean 194 angle between *x'*- and *x*-axis (see Eq. (2)).

195

$$\mathbf{v} = [\tan(\delta)\cos(\gamma + \rho), \tan(\delta)\sin(\gamma + \rho), 1]$$
(16)

197 Considering the triangle  $OH_jV$  in Fig. 5 and Fig. 6 for each *j*-th indenter face (A, B, C, D), given that 198  $\widehat{OH_jV} = \omega_i$ , and implementing the law of sines, Eq. (17) is obtained:

$$\overline{OH}_{j} = \|\mathbf{v}\| \frac{\sin\frac{\theta_{j}}{2}}{\sin\omega_{j}}$$
(17)

199 where  $\theta_A = \theta_C = \theta_{x'}$  and  $\theta_B = \theta_D = \theta_{y'}$ .

200 In this way, Eqs. (18) and (19) can be derived. The sign of the vector components for the four faces follows

201 the position on the xyz reference system as in Fig. 5.

$$\mathbf{OS}_{j} = \left(\frac{\overline{\mathrm{OH}}_{j}}{\cos\rho_{j}}, 0, 0\right) \tag{18}$$

$$\mathbf{OP}_{j} = \left(0, \frac{\overline{\mathrm{OH}}_{j}}{\sin \rho_{j}}, 0\right)$$
(19)

202 Considering the triangle OVS<sub>j</sub>, it is obtained that

$$\widehat{VOS_j} = \arccos\left(\frac{\mathbf{OS}_j \cdot \mathbf{v}}{\|\mathbf{v}\| \|\mathbf{OS}_j\|}\right)$$
(20)

203 Again, by applying the law of sines to triangle OVS<sub>j</sub>, it is obtained that

$$\frac{\|\mathbf{v}\|}{\sin \widehat{OS_j V}} = \frac{\|\mathbf{OS}_j\|}{\sin \widehat{OVS_j}} = \frac{\|\mathbf{VS}_j\|}{\sin \widehat{VOS_j}}$$
(21)

Given that  $\widehat{OS_jV} = 180 - \widehat{OVS_j} - \widehat{VOS_j}$  and with some trigonometric calculations, Eq. (22) is obtained. 205

$$\sin \widehat{OS_jV} = \sin(180 - \widehat{OVS_j} - \widehat{VOS_j}) = \sin \widehat{OVS_j} \cos \widehat{VOS_j} + \sin \widehat{VOS_j} \cos \widehat{OVS_j}$$
(22)

In this way, combining Eq. (21) and Eq.(22), Eqs. (23)-(25) are obtained:

$$\frac{\|\mathbf{v}\|}{\|\mathbf{OS}_{j}\|} = \frac{\sin \overline{OS_{j}V}}{\sin \overline{OVS_{j}}}$$
(23)

$$\frac{\|\mathbf{v}\|}{\|\mathbf{OS}_{j}\|} = \frac{\sin \overline{OVS_{j}} \cos \overline{VOS_{j}} + \sin \overline{VOS_{j}} \cos \overline{OVS_{j}}}{\sin \overline{OVS_{j}}}$$
(24)

$$\frac{\|\mathbf{v}\|}{\|\mathbf{OS}_{j}\|} = \cos \overline{VOS_{j}} + \frac{\sin \overline{VOS_{j}}}{\tan \overline{OVS_{j}}}$$
(25)

208 and from Eq. (25), Eq. (26) is also obtained:

$$\widehat{OVS_j} = \arctan\left(\frac{\|\mathbf{OS}_j\|\sin\widehat{VOS_j}}{\|\mathbf{v}\| - \|\mathbf{OS}_j\|\cos\widehat{VOS_j}}\right)$$
(26)

209

210 By applying the same calculations from Eq. (20) onward to triangle VOP<sub>j</sub>, it is found that,

211

$$\widehat{OVP_j} = \arctan\left(\frac{\|\mathbf{OP}_j\|\sin\overline{VOP_j}}{\|\mathbf{v}\| - \|\mathbf{OP}_j\|\cos\overline{VOP_j}}\right)$$
(27)

212

Therefore, considering a single j-th indenter face, the angles between two opposite edges can be found according to:

215

$$\alpha_j = 2 \ \widehat{OVP_j} \tag{28}$$

$$\beta_j = 2 \ \overline{OVS_j} \tag{29}$$

Averaging the results obtained for each *j*-th indenter face, the angles between two opposite edges, nominally  $172.5^{\circ}$  (Eq. 30) and  $130^{\circ}$  (Eq. 31), are finally obtained:

$$\alpha = \frac{\sum_{j=1}^{4} \alpha_j}{4} \tag{30}$$

$$\beta = \frac{\sum_{j=1}^{4} \beta_j}{4} \tag{31}$$

#### 219 4. Comparison of experimental measurements

220 In order to validate the geometrical model, experimental measurements were performed on three different 221 Knoop indenters previously verified by a German DKD accredited laboratory having comparable measurement 222 uncertainties. Calibration certificates data with expanded uncertainties at a confidence level of 95% are re-223 ported in Table 1. Verification of the Knoop indenters' geometrical parameters was performed with the Gali-224 leo-LTF® Gal-Indent optical system at INRiM. Experimental results with expanded uncertainties at a confi-225 dence level of 95% (CMCs declared in the CIPM-MRA database) are reported in Table 2. In this way, by 226 applying the geometrical model of Section 3, the complete set of values required for the verification of the 227 Knoop indenters are obtained and summarized in Table 3. Expanded uncertainties (at a confidence level of 228 95%, k=2), evaluated according to GUM [26] by propagating the experimental uncertainties, are in the order 229 of  $0.05^{\circ}$ , thus below the maximum expanded uncertainty of  $0.07^{\circ}$  required by the Standard. By way of exam-230 ple, the detailed uncertainty budget for the angle between the opposite edges  $\beta$  of Knoop indenter 1 is shown 231 in Table 4.

232

#### 233 Table 1

#### 234

	Knoop indenter 1	Knoop indenter 2	Knoop indenter 3
ID number	3522	3528	3521
Angle between the op- posite edges α /°	172.53±0.03	$172.50 \pm 0.03$	$172.50 \pm 0.03$
Angle between the opposite edges $\beta / ^{\circ}$	$130.13\pm0.07$	$129.83\pm0.07$	$130.02\pm0.07$
Tilt angle $\delta$ /°	$<\!0.42 \pm 0.07$	$<\!0.42 \pm 0.07$	${<}0.42\pm0.07$
Numerical factor c / -	$0.07018 \pm 0.00030$	$0.07001 \pm 0.00030$	$0.07031 \pm 0.00030$

Calibration certificate values of the three tested Knoop indenters.

235

## 237 Table 2

	Knoop indenter 1	Knoop indenter 2	Knoop indenter 3
$\omega_{ m A}$ /°	$25.146\pm0.05$	$25.297\pm0.05$	$25.197\pm0.05$
$\omega_{ m B}$ /°	25.151 ± 0.05	$25.275\pm0.05$	$25.184\pm0.05$
$\omega_{\rm C}/^{\circ}$	$25.151 \pm 0.05$	$25.310\pm0.05$	$25.193 \pm 0.05$
$\omega_{ m D}$ /°	$25.152 \pm 0.05$	$25.278\pm0.05$	$25.174\pm0.05$
<i>φ</i> 1 /°	$165.03 \pm 0.06$	$163.86\pm0.06$	$163.48\pm0.06$
$ au_1$ /°	$16.07\pm0.06$	$16.05\pm0.06$	$15.43\pm0.06$
$\varphi_2/^\circ$	$163.01 \pm 0.06$	$164.23 \pm 0.06$	$164.56\pm0.06$
$ au_2$ /°	$15.90\pm0.06$	$15.86\pm0.06$	$16.53\pm0.06$

238 Experimental measurements on the three tested Knoop indenters.

# 239

## 240 **Table 3**

241 Geometrical parameters of the three tested Knoop indenters evaluated with the geometrical model.

	Knoop indenter 1	Knoop indenter 2	Knoop indenter 3
ID number	3522	3528	3521
Angle between the op- posite edges α /°	$172.53\pm0.05$	$172.50 \pm 0.05$	$172.52\pm0.05$
Angle between the opposite edges $\beta \mid^{\circ}$	$130.13\pm0.05$	$129.85 \pm 0.05$	$130.06\pm0.05$
Tilt angle $\delta$ /°	$0.09\pm0.05$	$0.03\pm0.05$	$0.06\pm0.05$
Numerical factor c / -	$0.07019 \pm 0.00048$	$0.07007 \pm 0.00047$	$0.07019 \pm 0.00048$

#### **Table 4**

Variable $x_k$				_	2(	Danla
Symbol	Value	Note	$\mathcal{U}^{2}(X_{k})$	$C_k$	$u_k^2(a_x)$	Kank
ω <sub>A</sub>	25.146	CMC	6,4E-04	-5,1E-01	1,7E-04	1
$\omega_{\mathrm{B}}$	25.151	CMC	6,4E-04	-5,1E-01	1,7E-04	2
ωc	25.151	CMC	6,4E-04	-5,1E-01	1,7E-04	3
ωd	25.152	CMC	6,4E-04	-5,1E-01	1,7E-04	4
$\varphi_1$	165.03	CMC	9,1E-04	-1,3E-02	1,5E-07	5
$ au_1$	16.07	CMC	9,1E-04	+1,3E-02	1,7E-07	6
$\varphi_2$	163.01	CMC	9,1E-04	-1,3E-02	1,5E-07	7
$ au_2$	15.90	CMC	9,1E-04	+1,3E-02	1,7E-07	8
β	130.13			Variance, $u^2(\beta)$	6,6E-04	
				St. unc. $u(\beta)$	2,6E-02	

243 Uncerative budget for the angle between the opposite edges  $\beta$  of Knoop indenter 1.

244

An analysis based on the estimation of the normalized error  $(E_n)$  has been performed in order to assess the compatibility of the experimental measurements performed at INRiM with respect to calibration certificate values of the accredited laboratory, considered as reference.  $E_n$  is defined as the ratio of the difference between the measured value (*x*) and the reference value (*y*) compared to the root sum square of associated expanded uncertainties ( $U_x$  and  $U_y$ ) at a confidence level of 95 % (k = 2). According to ISO/IEC 17043:2010 [27], it is evaluated as follows:

$$E_n = \frac{|x-y|}{\sqrt{U_x^2 + U_y^2}}$$
(33)

251

252 Data can be considered compatible when  $E_n < 1$ . This is an indicator of accuracy/inaccuracy as compared 253 to an assigned reference value with respect to the associated uncertainties.

Combining data in Table 1 and Table 3, it is found that  $E_n$  is less than 1 for all geometrical parameters as shown in Table 5. For tilt angle  $\delta$ , since the calibration certificates report only that the values fall below the limit imposed by the standard, it is not possible to provide the exact normalized error. However, also experimental results show values below the standard limits. Given such evidence, the proposed method provides measurements compatible with the accredited laboratory.

259

#### **Table 5**

	Knoop indenter 1	Knoop indenter 2	Knoop indenter 3
Angle between the op- posite edges α	0.03	0.06	0.46
Angle between the opposite edges $\beta$	0.04	0.19	0.33
Numerical factor c	0.02	0.11	0.21

262 Normalized errors  $E_n$  evaluated for the three tested Knoop indenters.

#### 263

#### 264 5. Conclusions

265 ISO 4545-2 and 4545-3 of Knoop hardness tests require the geometrical verification of the indenter. At 266 present, verification of Knoop hardness is performed by few manufacturers and laboratories that use specific 267 instrumentation for the purpose. Since the verification of Vickers indenters can be performed by a larger num-268 ber of calibration laboratories and industries with dedicated systems and given the similar geometry of the two 269 indenters, the possibility to use Vickers-addressed systems for the verification of Knoop indenters, in order to 270 extend the measurement capability of these laboratories, is investigated. These systems are usually based on 271 optical measurements using microscopes, scanning interferometry or confocal probes. INRiM hardness labor-272 atory, in particular, uses a specific optical measuring system, based on Mirau interferometry, developed in 273 cooperation with Galileo-LTF® and commercialized by Galileo-LTF® as Gal-Indent. It is able to measure the 274 two vertex angles of the indenter between two opposite faces, the quadrilateral base angles and the angle be-275 tween the axis of the diamond pyramid and the axis of the indenter holder. This paper deals with the possibility 276 to use such quantities as inputs of a suitable geometrical model in order to verify the geometry of Knoop 277 indenters, i.e. to evaluate the angles between the opposite edges at the vertex, nominally  $172.5^{\circ}$  and  $130^{\circ}$ , and 278 the angle between the axis of the diamond pyramid and the axis of the indenter holder, nominally  $0^{\circ}$ . The 279 proposed geometrical model is described in Section 3. Experimental measurements, together with the associ-280 ated expanded uncertainties, were performed on three different Knoop indenters, previously measured by a 281 German DKD accredited laboratory, to verify the reliability of the model. Results of this work allow to high-282 light the following points:

- 283 Using Vickers-addressed measured quantities as input of the proposed geometrical model allows 284 to verify the geometry of Knoop indenters as requested by the relevant Standard. 285 Comparison of measurement data with reference values shows compatible results in terms of nor-• 286 malized error, thus validating the proposed procedure. 287 Expanded uncertainties are in the order of 0.05°, thus below the maximum expanded uncertainty • 288 of  $0.07^{\circ}$  required by the Standard. 289 The advange of this geometrical model is that it can be easily implemented, even on common 290 spreadsheets, and exploited by laboratories that adopt similar measuring systems addressed for the
- 291 verification of Vickers indenters without any modification of the experimental apparatus.

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