Final Report

14.03.2016

COOMET.PR-S3

Supplementary Comparison Refractive Index (COOMET project 438/RU/08)

Gennady Vishnyakov, Andreas Fricke, Nataliya M. Parkhomenko, Yasuaki Hori and Marco Pisani

Pilot lab: VNIIOFI (Russian Federation) Laboratory of Optical Constants Measurements Mail address: 119361 Moscow, Russia Ozernaya str., 46 E-mail: vish@vniiofi.ru Phone: +7 495 781 45 76 Fax: +7 495 437 31 47

Contents

1	Int	roduction
2	Or	ganization
	2.1	Participants' details
	2.2	Comparison organization
3	De	scription of transfer standards
4	Me	easurement results
5	An	alysis
	5.1	Calculation of the comparison reference value and its uncertainty
	5.2	Calculation the difference from CRV 17
6	Comp	parison Results
A	PPEN	DIX A. Measurement procedures and facilities
	A.1	All-Russian Research Institute for Optical and Physical Measurements (VNIIOFI,
	Russ	ia)19
	A.2	Physikalisch-Technische Bundesanstalt (PTB, Germany)
	A.3	Ukrmetrteststandart (Ukraine)
	A.4	National Institute of Advanced Industrial Science and Technology (AIST, Japan)
	A.5.	Istituto Nazionale di Ricerca Metrologica (INRiM, Italy)

1 Introduction

The Mutual Recognition Arrangement (MRA) was signed in 1999 with the objectives of establishing the degree of equivalence of national measurement standards maintained by National Metrology Institutes (NMIs), and providing for the mutual recognition of calibration and measurement certificates issued by NMIs. The objectives are achieved by a set of international comparisons of measurements known as key and supplementary comparisons. Supplementary comparisons are usually carried out by the Regional Metrology Organizations (RMOs) to meet specific needs not covered by key comparisons.

The COOMET.PR-S3 Supplementary Comparison was carried out to ensure the correctness and comparability of refractive index of solid transmitting materials in visible spectral range measured by the Participants of the comparison, within the uncertainties claimed for their measuring facility.

COOMET.PR-S3 was conducted within the ROM "Euro-Asian Cooperation of National Metrological Institutions" known as COOMET, and has the RMO project number of 438/RU/08.

The Comparison was piloted by the All-Russian Research Institute for Optical and Physical Measurements (VNIIOFI). Five NMIs from free RMOs (COOMET, EURAMET and APMP) participated in the comparison.

2 Organization

NMI. address NMI acronym **Contact person Contact details** (RMO) All-Russian Research Institute for Optical and Tel: +7 (495) 781-45-76 Pilot Physical Measurements. Gennady +7 (495) 437-33-77 VNIIOFI Ozernaya str. 46, Vishnyakov Fax: +7 (495) 437-31-47 119361 Moscow, Russia E-mail: vish@vniiofi.ru (COOMET) Physikalisch-Technische Participant 1 Bundesanstalt. Tel.: +49 531 592 4213. Bundesallee 100, D-PTB Andreas Fricke E-mail: <u>Andreas.Fricke@ptb.de</u> 38116 Braunschweig, Germany (EURAMET)

2.1 Participants' details

nt 3 Participant 2	Research and Production Centre for Standardization, Metrology, Certification and Consumers Rights Protection. Ukraine (COOMET) National Institute of	Ukrmetrteststandart	Andriy B. Glebov Nataliya M. Parkhomenko	Tel.: +38 (044) 526-36-98 Fax: +38 (044) 526-36-98 E-mail: <u>optic@ukrcsm.kiev.ua</u> <u>natapar@mail.ru</u> Tel: +81-29-861-4211	
Participa	Science and Technology. Japan (AMPM)	AIST	Yasuaki Hori	Fax: +81-29-861-4080 E-mail: <u>y-hori@aist.go.jp</u>	
Participant 4	Istituto Nazio nale di Ricerca Metrologica. Italy (EURAMET)	INRiM	<u>Marco Pisani,</u> Milena Astrua	Tel +39 011 3919 961 Fax +39 011 3919 959 E-mail: <u>m.pisani@inrim.it</u>	

2.2 Comparison organization

The comparison covered the values of the absolute refractive index at the specified wavelengths determined by the Participants for one set of three transfer standards. Full description of the transfer standards is given in Section 3.

The measurements of the artefacts were carried out in the following sequence:

$Pilot \rightarrow Participant \ 1 \rightarrow Participant \ 2 \rightarrow Participant \ 3 \rightarrow Participant \ 4 \rightarrow Pilot$

The Pilot measured the artefacts for the first time and sent the set to the Participant 1. The Participant 1 carried out measurements of the refractive index for each transfer standard and sent the set to the next Participant and then sent the measurement results and uncertainty budget to the Pilot by e-mail and etc. Finally the comparison standards return to the Pilot.

3 Description of transfer standards

The transfer standard was a set of three prismatic samples made of different types of glass. Prisms have identification numbers: 01, 02 and 03. Prisms differed from each other by nominal values of refractive index and temperature coefficient of the refractive index. Parameters of the prisms are listed in the Table 3.1.

The dimensions of the prisms are: the edge length is varied from 75 to 98 mm; the height of the prisms 01 and 03 is 48 mm; the height of the prisms 02 is 37 mm.

Prism	Glass type, (Schott)	Nominal Refractive index, n _d	Temperature coefficient of refractive index Δn _{abs} /ΔT [10 ⁻⁶ /K]			Dispersion <i>n</i> _F - <i>n</i> _C	Apex angles (approximate)
			1060 nm	e	g		
No 01	N-BAF 10	1.670	2.4	3.5	4.5	0.014222	60°; 60°; 60°
No 02	N-BK 7	1.517	1.1	1.6	2.1	0.008054	55°; 65°; 60°
No 03	SF 1	1.717	3.6	6.4	9.8	0.024307	53°; 67°; 60°

Table 3.1. Parameters of the transfer standard prisms used as the comparison artifacts

The apexes of each prism have identification: 1, 2 and 3.

The following apexes and surfaces were used for refractive index measurements:

Prism No 01	Apex 2	Surfaces 1-2 and 2-3
Prism No 02	Apex 1	Surfaces 1-2 and 3-1
Prism No 03	Apex 1	Surfaces 1-2 and 3-1

4 Measurement results

In the current section the results received from all the participants are presented.

Three participants, VNIIOFI (Pilot), PTB and Ukrmetrteststandart, submitted values of relative refractive index reduced to the standard atmospheric conditions (101325 Pa; 20 °C; 50% of relative humidity, 0.03% of CO₂ volume content) for wavelengths of 480 nm, 509 nm, 546 nm, 589.3 nm and 644 nm.

Three participants, VNIIOFI (Pilot), INRiM and AIST submitted values of absolute refractive index reduced to standard conditions for the wavelength of 632.8 nm. Pilot did a recalculation of these values to refractive index relative to "normal air". For this purpose, the submitted values of absolute refractive index were divided on the value of refractive index of "normal air" at the wavelength of 632.8 nm, which equals to 1.000271 and calculated using the Edlén's formula [1].

For analysis of results we used values of combined standard uncertainty (instead the expanded uncertainty) provided by the participants with uncertainty budgets.

Each of the Tables 4.1 - 4.6 shows refractive index measurement results provided by all participants for a certain wavelength.

In the tables n_i is a measured value of refractive index, $u(n_i)$ - its associated combined standard uncertainty, $u_{adj}^s(n_i)$ - adjusted uncertainty after correction by Mandel-Paule method, N_{CRV} - the comparison reference value (CRV), Δ_i - the difference from CRV, $U(\Delta_i)$ - the expanded uncertainty of the Δ_i with k=2. More explanations are given below in section 5.

After every table a graph of $\Delta_i \times 10^6$ for all transfer standards is presented (Fig.4.1 – 4.6).

Tables are divided by three different color areas corresponding to three prisms.

In the tables the red marked data are identified as outliers. These data were excluded from calculations of the final Comparison Reference Value (CRV) because the deviation from the tentative CRV was 3 times larger than of expanded uncertainty of the deviation with k=2.

Prism	Participant	n _i	$u(n_i)$ ×10 ⁶	$u_{\rm adj}(n_i) \times 10^6$	N _{CRV}	$\Delta_i \times 10^6$	$U(\Delta_i) imes 10^6$
	VNIIOFI	1.681054	1.8	1.8		2	3
No 01	РТВ	1.681051	0.7	1.3		-1	1
N-BAF10	Ukrmetrtest	1.681046	3.5	3.5	1.681052	-6	7
	-standart						
	VNIIOFI	1.523109	1.8	1.8		1	3
No 02	РТВ	1.523108	0.5	1.2	1 522109	0	1
N-BK7	Ukrmetrtest -standart	1.523112	4.7	4.7	1.525108	4	9
	VNIIOFI	1.736029	1.8	1.8		3	3
No 03	РТВ	1.736024	0.7	1.3	1.736026	-2	1
SF 1	Ukrmetrtest -standart	1.736025	4.2	4.2		-1	8

Table 4.1. Refractive index measurement results at 480 nm



Figure 4.1. Difference from CRV (Δ_i) with expanded uncertainties $U(\Delta_i)$ at 480 nm.

Prism	Participant	n _i	$u(n_i)$ ×10 ⁶	$u_{\rm adj}(n_i) \times 10^6$	N _{CRV}	$\Delta_i \times 10^6$	$U(\Delta_i) \times 10^6$
	VNIIOFI	1.677415	<mark>1.8</mark>	-		<mark>-31</mark>	-
No 01	РТВ	1.677450	0.7	3.3	1 (4	2
N-BAF10	Ukrmetrtest	1.677439	3.5	4.6	1.677446	-7	7
	-standart						
	VNIIOFI	1.521110	1.8	-		<mark>-26</mark>	-
No 02	РТВ	1.521136	0.5	1.2	1 501126	0	1
N-BK7	Ukrmetrtest -standart	1.521139	4.7	4.7	1.521150	3	9
	VNIIOFI	1.729602	<mark>1.8</mark>	-		<mark>-72</mark>	-
No 03	РТВ	1.729674	0.7	1.3	1 720674	0	1
SF 1	Ukrmetrtest -standart	1.729672	4.2	4.2	1.729074	-2	9

Table 4.2. Refractive index measurement results at 509 nm





Prism	Participant	n _i	$u(n_i)$ ×10 ⁶	$u_{\rm adj}(n_i) \times 10^6$	N _{CRV}	$\Delta_i \times 10^6$	$U(\Delta_i) imes 10^6$
	VNIIOFI	1.673617	1.8	3.1		5	3
No 01	РТВ	1.673610	0.7	2.8	1 672612	-2	2
N-BAF10	Ukrmetrtest	1.673605	3.5	4.3	1.0/3012	-7	6
	-standart						
	VNIIOFI	1.518997	1.8	1.8		-1	3
No 02	РТВ	1.518999	0.5	1.2	1 510000	1	1
N-BK7	Ukrmetrtest -standart	1.518997	4.7	4.7	1.310990	-1	9
	VNIIOFI	1.723045	1.8	3.0		6	3
No 03	РТВ	1.723037	0.7	2.7	1 723030	-2	2
SF 1	Ukrmetrtest -standart	1.723033	4.3	4.9	1.723039	-6	8

Table 4.3. Refractive index measurement results at 546 nm



Figure 4.3. Difference from CRV (Δ_i) with expanded uncertainties $U(\Delta_i)$ at 546 nm.

Prism	Participant	n _i	$u(n_i)$ ×10 ⁶	$u_{\rm adj}(n_i) \times 10^6$	N _{CRV}	$\Delta_i \times 10^6$	$U(\Delta_i) \times 10^6$
	VNIIOFI	1.670090	1.8	1.8		-3	3
No 01	РТВ	1.670095	0.7	1.3	1 (50002	2	1
N-BAF10	Ukrmetrtest	1.670091	3.5	3.5	1.0/0093	-2	7
	-standart						
	VNIIOFI	1.517000	1.8	1.9		-4	3
No 02	РТВ	1.517005	0.5	1.3	1 517004	1	1
N-BK7	Ukrmetrtest -standart	1.517009	4.6	4.7	1.517004	5	9
	VNIIOFI	1.717084	1.8	1.8		-1	3
No 03	РТВ	1.717086	0.7	1.3	1 717095	1	1
SF 1	Ukrmetrtest -standart	1.717084	4.3	4.3	1./1/005	-1	8

Table 4.4. Refractive index measurement results at 589.3 nm



Figure 4.4. Difference from CRV (Δ_i) with expanded uncertainties $U(\Delta_i)$ at 589.3 nm.

Prism	Participant	n _i	$u(n_i)$ ×10 ⁶	$u_{\rm adj}(n_i) \times 10^6$	N _{CRV}	$\Delta_i \times 10^6$	$U(\Delta_i) \times 10^6$
	VNIIOFI	1.667260	1.8	1.8		1	3
No 01 N-BAF10	AIST	1.667258	1.3	1.3	1.667259	-1	2
	INRiM	1.667243	<mark>1.9</mark>	-		<mark>-16</mark>	-
	VNIIOFI	1.515364	1.8	2.6		3	3
No 02 N-BK7	AIST	1.515363	0.9	2.1	1.515361	2	2
	INRiM	1.515356	1.7	2.6		-5	3
	VNIIOFI	1.712384	1.8	1.8		2	3
No 03	AIST	1.712382	1.1	1.5	1 71222	0	2
SF 1	INRiM	1.712380	1.9	1.9	1.712302	-2	3

Table 4.5. Refractive index measurement results at 632.8 nm



Figure 4.5. Difference from CRV (Δ_i) with expanded uncertainties $U(\Delta_i)$ at 632.8 nm.

Prism	Participant	n _i	$u(n_i)$ ×10 ⁶	$u_{\rm adj}(n_i) \times 10^6$	N _{CRV}	$\Delta_i \times 10^6$	$U(\Delta_i) imes 10^6$
	VNIIOFI	1.666631	1.8	3.0		3	3
No 01	РТВ	1.666631	0.7	2.6	1 (((20)	2	2
N-BAF10	Ukrmetrtest	1.666620	3.5	4.3	1.666629	-9	6
	-standart						
	VNIIOFI	1.514995	1.8	1.8		0	3
No 02	РТВ	1.514995	0.5	1.2	1 514005	0	1
N-BK7	Ukrmetrtest -standart	1.514959	<mark>4.6</mark>	-	1.514995	<mark>-36</mark>	-
	VNIIOFI	1.711351	1.8	1.8		0	3
No 03	РТВ	1.711351	0.7	1.3	1 711351	0	1
SF 1	Ukrmetrtest -standart	<mark>1.711321</mark>	<mark>4.3</mark>	-	1./11551	-30	-

Table 4.6. Refractive index measurement results at 644 nm





5 Analysis

5.1 Calculation of the comparison reference value and its uncertainty

Preparation of this report was carried out according to the Section 4 of the "Guidelines for CCPR Key Comparison Report Preparation", CCPR Working Group on Key Comparisons, CCPR-G2 Rev.3, July 1, 2013.

We assume the total number of participants submitting results is *I*. Each laboratory *i* reports a measured value n_i and its associated combined standard uncertainty $u(n_i)$. The combined standard uncertainties are shown in the Table 5.1.

The comparison reference value (CRV) N_{CRV} for each wavelength and prism is calculated as weighted mean with cut-off uncertainty. The cut-off value $u_{cut-off}$ is calculated by

$$u_{\text{cut-off}} = \text{average}\{u(n_i)\} \text{ for } u(n_i) \le \text{median}\{u(n_i)\}; i=1, ..., I.$$
(1)

The calculated cut-off uncertainty values are shown in the Tables 5.1.

Prism	Wavelength, nm	480	509	546	589.3	644	632.8
	VNIIOFI	1.8	1.8	1.8	1.8	1.8	1.8
	РТВ	0.7	0.7	0.7	0.7	0.7	-
No 01	AIST	-	-	-	-	-	1.3
N-BAF10	INRiM	-	-	-	-	-	1.9
	Ukrmetrteststandart	3.5	3.5	3.5	3.5	3.5	-
	Cut-off uncertainty			1.3			1.6
	VNIIOFI	1.8	1.8	1.8	1.8	1.8	1.8
	РТВ	0.5	0.5	0.5	0.5	0.5	-
No 02	AIST	-	-	-	-	-	0.9
N-BK7	INRiM	-	-	-	-	-	1.7
	Ukrmetrteststandart	4.7	4.7	4.7	4.6	4.6	-
	Cut-off uncertainty			1.2		•	1.3
	VNIIOFI	1.8	1.8	1.8	1.8	1.8	1.8
	РТВ	0.7	0.7	0.7	0.7	0.7	-
No 03	AIST	-	-	-	-	-	1.1
SF 1	INRiM	-	-	-	-	-	1.9
	Ukrmetrteststandart	4.2	4.2	4.3	4.3	4.3	-
	Cut-off uncertainty			1.3		·	1.5

Table 5.1. The combined standard uncertainty $u(n_i) \times 10^6$ and cut-off uncertainty $u_{\text{cut-off}} \times 10^6$.

The reported uncertainty $u(n_i)$ of each NMI *i* is adjusted by the cut-off,

$$u_{\text{adj}}(n_i) = u(n_i) \text{ for } u(n_i) > u_{\text{cut-off}}$$

$$u_{\text{adj}}(n_i) = u_{\text{cut-off}} \text{ for } u(n_i) \le u_{\text{cut-off}}, i=1, \dots, I.$$
(2)

The Table 5.2 shows the adjusted by the cut-off uncertainties $u_{adj}(n_i) \times 10^6$.

Prism	Wavelength, nm	480	509	546	589.3	644	632.8
	VNIIOFI	1.8	-	1.8	1.8	1.8	1.8
	РТВ	1.3	1.3	1.3	1.3	1.3	-
No 01 N-BAF10	AIST	-	-	-	-	-	1.6
	INRiM	-	-	-	-	-	1.9
	Ukrmetrteststandart	3.5	3.5	3.5	3.5	3.5	-
	VNIIOFI	1.8	-	1.8	1.8	1.8	1.8
	РТВ	1.2	1.2	1.2	1.2	1.2	-
No 02 N-BK7	AIST	-	-	-	-	-	1.3
	INRiM	-	-	-	-	-	1.7
	Ukrmetrteststandart	4.7	4.7	4.7	4.6	4.6	-
	VNIIOFI	1.8	-	1.8	1.8	1.8	1.8
	РТВ	1.3	1.3	1.3	1.3	1.3	-
No 03 SF 1	AIST	-	-	-	-	-	1.5
	INRiM	-	-	-	-	-	1.9
	Ukrmetrteststandart	4.2	4.2	4.3	4.3	4.3	-

Table 5.2. Adjusted by the cut-off uncertainties $u_{adj}(n_i) \times 10^6$.

The normalised weight w_i for the result n_i is given by:

$$w_i = u_{\rm adj}^{-2}(n_i) / \sum_{i=1}^{I} u_{\rm adj}^{-2}(n_i)$$
(3)

Then the weighted mean N_{CRV} is calculated:

$$N_{\rm CRV} = \sum_{i=1}^{I} w_i \cdot n_i \tag{4}$$

The uncertainty of the weighted mean is calculated by:

$$u(N_{\rm CRV}) = \sqrt{\sum_{i=1}^{I} u^2(n_i) / u_{\rm adj}^4(n_i)} / \sum_{i=1}^{I} u_{\rm adj}^{-2}(n_i)$$
(5)

The Chi-square value χ^2_{obs} is calculated for consistency check. *i*=1 represents the pilot lab:

$$\chi_{\rm obs}^2 = \sum_{i=1}^{I} \frac{(n_i - N_{\rm CRV})^2}{u_{\rm adj}^2(n_i)} \,. \tag{6}$$

If $\chi^2_{obs} \le \chi^2_{0,05}(\nu)$, consistency is satisfied. The value $\chi^2_{0,05}(\nu)$ is determined from Table 5.3 for $\nu = I - 1$. For different prism&wavelength combinations we have two cases: I = 3 or I = 2. Therefore, for the first case $\nu = 2$, and for the second $\nu = 1$. Thus, $\chi^2_{0,05}(2) = 5.991$ and $\chi^2_{0,05}(1) = 3.841$ for total number of participants *I* equals to 3 and 2, respectively.

v	$\chi^{2}_{0.05}(v)$
1	3.841
2	5.991
3	7.815
4	9.488
5	11.07
6	12.592
7	14.067
8	15.507
9	16.919
10	18.307
11	19.675
12	21.026
13	22.362
14	23.685
15	24.996
16	26.296
17	27.587
18	28.869

Table 5.3. $\chi^{2}_{0,05}(v)$ values.

The original results of Chi-square test are shown in Table 5.4. Unfortunately for some cases $\chi^2_{obs} > \chi^2_{0,05}(\nu)$, i.e. the consistency failed. For these cases the χ^2_{obs} values are highlighted in red in Table 5.4.

For the failed cases the Mandel-Paule method was applied, i.e. the s^2 term was added to the $u_{adj}(n_i)$:

$$u_{\rm adj}^{s}(n_{i}) = \sqrt{u_{\rm adj}^{2}(n_{i}) + s^{2}}$$
 (7)

The value of *s* was determined by iterative process so that $\chi^2_{obs} \approx \chi^2_{0,05}(\nu)$. The values of this additional "interlaboratory variance" $s \times 10^3$ are shown in Table 5.5. New adjusted uncertainty values $u_{adj}(n_i) \times 10^6$ are presented in Table 5.6.

Finally, when the consistency is satisfied (after the Mandel-Paule method), we recalculated the normalised weights, weighted mean N_{CRV} and its uncertainty using (3) to (5).

Prism		480 nm	509 nm	546 nm	589.3 nm	644 nm	632.8 nm
No 01	$\chi^2_{ m obs}$	4.558	<mark>8.760</mark>	<mark>14.184</mark>	5.629	<mark>9.878</mark>	0.709
N- BAF10	$\chi^2_{0,05}(\nu)$	5.991	3.841	5.991	5.991	5.991	3.841
	Ι	3	2	3	3	3	2
	$\chi^2_{ m obs}$	0.842	0.400	0.968	<mark>6.823</mark>	0	<mark>13.566</mark>
No 02 N-BK7	$\chi^2_{0,05}(\nu)$	5.991	3.841	5.991	5.991	3.841	5.991
	Ι	3	2	3	3	2	3
	$\chi^2_{ m obs}$	5.226	0.200	<mark>15.557</mark>	0.926	0	2.290
No 03 SF 1	$\chi^{2}_{0,05}(v)$	5.991	3.841	5.991	5.991	3.841	5.991
	Ι	3	2	3	3	2	3

Table 5.4. Original Chi-square value χ	.2 obs•
---	------------

Table 5.5. Values of Mandel-Paule addition $s \times 10^3$.

Prism	Wavelength, nm	480	509	546	589.3	644	632.8
	VNIIOFI	-	-	2.45	-	2.2	-
	РТВ	-	3.0	2.45	-	2.2	-
No 01 N-BAF10	AIST	-	-	-	-	-	-
	INRiM	-	-	-	-	-	-
	Ukrmetrteststandart	-	3.0	2.45	-	-	-
	VNIIOFI	-	-		0.6	-	1.9
	РТВ	-	-	-	0.6	-	-
No 02 N-BK7	AIST	-	-	-	-	-	1.9
	INRiM	-	-	-	-	-	1.9
	Ukrmetrteststandart	-	-	-	0.6	-	-
	VNIIOFI	-	-	2.4	-	-	-
	РТВ	-	-	2.4	-	-	-
No 03 SF 1	AIST	-	-	-	-	-	-
	INRiM	-	-	-	-	-	-
	Ukrmetrteststandart	-	-	2.4	-	-	-

Prism	Wavelength, nm	480	509	546	589.3	644	632.8
	VNIIOFI	1.8	-	3.1	1.8	3.0	1.8
	РТВ	1.3	3.3	2.8	1.3	2.6	-
No 01 N-BAF10	AIST	-	-	-	-	-	1.3
	INRiM	-	-	-	-	-	-
	Ukrmetrteststandart	3.5	4.6	4.3	3.5	4.3	-
	VNIIOFI	1.8	-	1.8	1.9	1.8	2.6
	РТВ	1.2	1.2	1.2	1.3	1.2	-
No 02 N-BK7	AIST	-	-	-	-	-	2.1
	INRiM	-	-	-	-	-	2.6
	Ukrmetrteststandart	4.7	4.7	4.7	4.7	-	-
	VNIIOFI	1.8	-	3.0	1.8	1.8	1.8
	РТВ	1.3	1.3	2.7	1.3	1.3	-
No 03 SF 1	AIST	-	-	-	-	-	1.5
	INRiM	-	-	-	-	-	1.9
	Ukrmetrteststandart	4.2	4.2	4.9	4.3	-	-

Table 5.6. New values of adjuste	ed uncertainty $u_{adi}^{s}(n_{i}) \times 10^{6}$
----------------------------------	---

5.2 Calculation the difference from CRV

The difference from CRV, Δ_i , for a laboratory result n_i is calculated simply as

$$\Delta_i = n_i \cdot N_{\rm CRV} \,. \tag{8}$$

The expanded uncertainty of the difference from CRV with k=2 is calculated using

$$U(\Delta_i) = 2\sqrt{u^2(n_i) + u^2(N_{\rm CRV}) - 2\left[\frac{u^2(n_i)}{u_{\rm adj}^2(n_i)} / \sum_{i=1}^I u_{\rm adj}^{-2}(n_i)\right]}$$
(9)

for results which contributed to the weighted mean.

6 Comparison Results

The COOMET.PR-S3 comparison results are presented in Table 6.1.

Table 6.1. Comparison results

Duiana	Wave	VNI	IOFI	P	ТВ	A	IST	IN	RiM	Ukrn -sta	netrtest ndart
Prism	nm	$\lambda_i \times 10^6$	$egin{array}{l} m{U}(\Delta_i)\ imes m{10}^6 \end{array}$	$\lambda_i \times 10^6$	$U(\Delta_i) \times 10^6$	$\lambda_i \times 10^6$	$U(\Delta_i) \times 10^6$	$\lambda_i \times 10^6$	$U(\Delta_i) \times 10^6$	$\lambda_i \times 10^6$	$U(\Delta_i) imes 10^6$
	480	2	3	-1	1	-	-	-	-	-6	7
	509	-	-	4	2	-	-	-	-	-7	7
No 01	546	5	3	-2	2	-	-	-	-	-7	6
BAF10	589.3	-3	3	2	1	-	-	-	-	-2	7
	644	3	3	2	2	-	-	-	-	-9	6
	632.8	1	3	-	-	-1	2	-	-	-	-
	480	1	3	0	1	-	-	-	-	4	9
	509	-	-	1	9	-	-	-	-	3	9
No 02	546	-1	3	1	1	-	-	-	-	-1	9
N-BK7	589.3	-4	3	1	1	-	-	-	-	5	9
	644	0	3	0	1	-	-	-	-	-	-
	632.8	3	3	-	-	2	2	-5	3	-	-
	480	3	3	-2	1	-	-	-	-	-1	8
No 03 SF 1	509	-	-	0	1	-	-	-	-	-2	9
	546	6	3	-2	2	-	-	-	-	-6	8
	589.3	-1	3	1	1	-	-	-	-	-1	8
	644	0	3	0	1	-	-	-	-	-	-
	632.8	2	3	-	-	0	2	-2	3	-	-

APPENDIX A. Measurement procedures and facilities

Each Participant measured refractive index of the transfer standards using its own method of measurement and facility.

Before starting measurements Participants inspected the transfer standards for damage. Any damage should be documented. No damages happened during the comparison.

The temperature of the prism during the measurements should be within the limits of 19.9°C to 20.1°C. Each Participant used its own method of temperature measurement.

The measuring light beam should be circular. Participants could use beam diameter suitable for they facility. The beam should meet the faces of the prisms in their centre during the measurement of the prism angle as well as the angle of deviation.

To prevent influence of inner reflexes in goniometric measurements it was recommended to use RI-matching material (for ex. oil) or to fix black paper on the backside of the prism.

All measurement conditions, including atmospheric pressure, temperature, humidity and content of CO₂, should be recorded and reported. The refractive indexes should be reported as measured at actual conditions, and recalculated for the following standard atmospheric conditions ("Feuchte Normalluft"): 101325 Pa; 20 °C; 50% relative humidity, CO₂ volume content of 0.03%.

After completion of measurements the prism surfaces should be cleaned by pure alcohol. The facility and method of each participant are described below.

A.1 All-Russian Research Institute for Optical and Physical Measurements (VNIIOFI, Russia)

Description of the measuring facility

VNIIOFI used original measurements method and equipment (Fig. A.1.1) developed at VNIIOFI and described in [2-4].



Fig. A.1.1. VNIIOFI facility for refractive index measurement: 1 – table; 2 – dynamic goniometric spectrometer DG-1L; 3 – mirror rotating stage; 4 – optical fiber; 5 – triangular glass prism; 6 – interferometric null-indicator; 7 – air temperature detector; 8 – mirrors; 9 – stepping motor rotates the stage; 10 – two-sided mirror; 11 – prism stepping rotating stage.

The refractive index is calculated by minimum-deviation method.

Measuring system is based on a dynamic goniometer DG-1L with a ring laser, which provides the necessary accuracy characteristics in angle measurement. All angle measurements are carrying out automatically without the presence of the operator in the measurement zone. Object stage of goniometer DG-1L is rotating continuously. We determinate angle of minimum deviation automatically. The essence of our method is that one first performs a series of automatic measurements of the deviation angle ε with various angular positions of the prism relative to the probing beam, i.e., for various angles of incidence φ_i , i = 0, 1, 2, ..., K, where K is the total number of measurements. Then the experimental $\varepsilon(\varphi_i)$ is fitted to a polynomial of second degree and ε_{min} is calculated from it.

The refractive index is calculated in two stages. One first measures the angle of minimum deviation ε_{\min} and from it calculates the refractive index of the substance relative to the surrounding environmental conditions, which is called the relative refractive index.

$$n(\lambda) = \frac{\sin\left(\frac{\alpha + \varepsilon_{\min}(\lambda)}{2}\right)}{\sin\left(\frac{\alpha}{2}\right)}$$
(A.1.1)

where α is the apex angle of the prism.

One measures the temperature of the prism and of the surrounding air as well as the air pressure and humidity. In the second stage, one uses well known Edlén's formula [1] to calculate the refractive index of the air N_{air} (λ , t, p, f), where t is the air temperature in °C; p the air pressure in kPa; f the partial pressure of water vapor in kPa; k_0 the wave number in vacuum, which is equal to $1/\lambda$ in mcm⁻¹; and λ is the wavelength of the light used.

Then one calculates the absolute refractive index of the measure and converts it to normal conditions of measurement in accordance with [5]

$$n_0(\lambda) = \frac{n(\lambda) \cdot N_{\text{air}}(\lambda, t, p, f) - \beta \cdot (t_{\text{sample}} - 20^\circ)}{N_{\text{air}}^0(\lambda)}, \qquad (A.1.2)$$

where β – temperature coefficient of the sample's material, t_{sample} – temperature of sample during measurements, N_{air}^0 - refractive index of air under "normal" conditions [1, 5].

The main element of measuring system (dynamical goniometer) is located within a special chamber of volume 18 m^3 . The walls, floor, and ceiling of the chamber are made of special thermal insulation panels faced by sheet metal to reduce the temperature gradients. The chamber has a thermally insulated door for access and a window for visual monitoring. It is equipped with systems for humidifying and cleaning the air, and also with the hygrometer and barometer.

The light source is provided either by a He–Ne and Ar-Kr lasers. The radiation enters to the climatic chamber via an optical fiber bundle.

Constant temperature within the chamber is maintained by a precision air conditioning system: a split system of inverter type. It provides temperatures of the air constant to $20,0 \pm 0,1^{\circ}$ C in the internal volume with active thermal stabilization. This system is disconnected during the measurements, which last several minutes.

To reduce the temperature gradients, the climatic chamber is located in a room also equipped with an air-conditioning system that maintains a temperature of $20 \pm 1^{\circ}$ C.

The measurements are completely automated. The operator remains outside the chamber during the measurements. All the heat-producing units in the monitoring and measuring equipment, power supply sources, sources of optical radiation, and the processing computer are placed outside the chamber.

In precision measurements, a holographic table from UIG-2M equipment provides reliable protection of the refractometer from the vibration.

The chamber is equipped with a multichannel digital thermometer for measuring the temperatures of the standard measure and the air within the chamber. The limit to the permissible error in the thermometer measurements is ± 2 mK. The temperature measurement

system includes three separate detectors, two of which are placed in the air near the measurement volume and one contact detector is placed on the standard measure. The readings from all three detectors pass in real time to the computer and are displayed on the screen.

The data-acquisition and processing system consists of a personal computer, interfaces for collecting the measurement data from the spectrometer and the thermometers, together with software for processing the angular measurements and calculating the refractive index.

Size of aperture of measuring beam: 10×18 mm.

Description of adjusting the comparison standard

Prism has been installed on the object stage of the dymanical goniometer so that the beam of light illuminated the central part of its working surfaces. Influence of pyramidal errors of the prism's working faces was minimized by adjustment screws of object stage of the goniometer. The adjustment process was controlled visually using the autocollimator.

Uncertainty budget

a. For wavelengths:

- Ar-Kr laser: 476.5 nm; 487.9 nm; 496.5 nm; 501.7 nm; 514.5 nm
- He-Ne laser: 632.8 nm

Uncertainty source	Туре	Standard	Sensitivity	Contribution in
X _i		measurement	coefficient	standard
		uncertainty	$c = \frac{\partial F}{\partial F}$	uncertainty
		$u(x_i)$	$C_i = \partial x_i$	$u_i(y_q) = \left \frac{\partial F}{\partial x_i} \right \cdot u(x_i)$
Repeatability of	A	1.0×10 ⁻⁶		
refractive index n_0				
measurements				
Angle measurements	В	9.7×10 ⁻⁷		
on the dynamic				
goniometer, radian				
Flatness of prism	В	6.8×10 ⁻⁷		
surfaces, radian				
Angle of minimum	В	4.8×10 ⁻⁷		
deviation ε_{\min}				

(method of				
determination),				
radian.				
Measurements of apex	В	1.2×10 ⁻⁶	$\partial n(\lambda)$	7.8×10^{-7}
angle of prism α ,			$\partial lpha$	
radian				
Determination of	В	1.2×10 ⁻⁶	$\partial n(\lambda)$	7.8×10 ⁻⁷
angle of minimum			$\partial arepsilon_{\min}$	
deviation ε_{\min} , radian				
Measurements of air	В	$\frac{0.04}{0.04} = 0.02$	$\partial N_{ m air}$	2.1×10 ⁻⁸
temperature <i>t</i> , °C		$\sqrt{3}$ - 0.02	∂t	
Measurements of air	В	$\frac{0.05}{=} = 0.03$	$\partial N_{ m air}$	9.7×10 ⁻⁹
pressure p , $\times 10^3$ Pa		$\sqrt{3}$	∂p	
Measurements of	В	$\frac{0.05}{5} = 0.03$	$\partial N_{ m air}$	7.7×10 ⁻⁸
vapor pressure f , × 10 ³		$\sqrt{3}$	∂f	
Ра				
Wave length λ , mcm	В	$\frac{2 \cdot 10^{-6}}{10^{-6}} = 1.2 \cdot 10^{-6}$	$\partial N_{ m air}$	1.4×10 ⁻⁸
		$\sqrt{3}$	$\partial \lambda$	
Measurements of the	В	0.0004	∂n_0	6.9×10 ⁻⁸
sample temperature		$-\frac{1}{\sqrt{3}} = 0.0002$	$\frac{1}{\partial t_{\text{sample}}}$	
$t_{\text{sample}}, ^{\circ}\text{C}$				
Refractive index n_0	В	1.1 · 10 ⁻⁶		
Total combined standa	1.5×10 ⁻⁶			
Expanded uncertainty	3×10 ⁻⁶			

<u>b. For wavelengths:</u> F' (Cd 480.0 nm), F (H 486.1 nm), e (Hg 546.1 nm), d (He 587.6 nm), C' (Cd 643.8 nm) and C (H 656.3 nm)

To determine the refractive index at the wavelengths F' (Cd 480.0 nm), F (H 486.1 nm), e (Hg 546.1 nm), d (He 587.6 nm), C' (Cd 643.8 nm) and C (H 656.3 nm) the following operations should be done:

1. Measure refractive indexes of a prism at each Ar-Kr laser wavelength (476.5 nm, 487.9 nm, 496.5 nm, 501.7 nm, 514.5 nm) and He-Ne laser wavelength (632.8 nm).

2. Approximate of the measured data using the Sellmeier dispersion formula:

$$n^{2}(\lambda) = 1 + \frac{B_{1}\lambda^{2}}{(\lambda^{2} - C_{1})} + \frac{B_{2}\lambda^{2}}{(\lambda^{2} - C_{2})} + \frac{B_{3}\lambda^{2}}{(\lambda^{2} - C_{3})}, \qquad (A.1.3)$$

where coefficients B1, B2, B3, C1, C2, C3 should be chosen in such a way that the sum of the squared deviations of the measured values of the curve is minimal.

3. Recalculates the value of the refractive index at wavelengths F' (Cd 480.0 nm), F (H 486.1 nm), e (Hg 546.1 nm), d (He 587.6 nm), C' (Cd 643.8 nm) и C (H 656.3 nm) using formula (A.1.3).

Expanded uncertainty (k = 2): 3.5×10^{-6} .

A.2 Physikalisch-Technische Bundesanstalt (PTB, Germany)

Description of the measuring facility

The measurement of the refractive index of glasses is performed by the determination of angles with a goniometer.



Fig.A.2.1. PTB measuring facility.

The angles between the two faces of solid prisms are measured with an autocollimator (AC). In this procedure, the prism face is illuminated with a parallel light beam of a defined diameter. The part of the measuring beam which is reflected back into itself is detected (autocollimation). The mechanical design of the goniometer allows the autocollimator (AC) to be turned around the calibration object in the plane vertically to the rotation axis. In this way, a second measuring face can be targeted. The AC is mounted to an air-bearing rotating stage together with a high-resolution divided circle. With the aid of 10 stationary readout units, the angular position of the AC is determined. From the difference of two divided circle readouts, the

angle between the two prisms faces results as the prism angle.

Refractive index measurements with lowest uncertainty are performed by measuring the angular effect of refraction, i.e. the deviation angle of a prism in accordance with the method of minimum of deviation. For such measurements, an angle measuring instrument with suitable measuring setups is required, and in addition measuring instruments to determine the atmospheric data and the temperature of the surrounding air [6-16].

The refractive index and the dispersion curves of optically transparent materials in the form of prisms are determined by measuring the prism angle and the deviation angle. The prism angle is determined with the autocollimation method (in the figure below: the red beam). For the measurement of the deviation angle, a parallel monochromatic beam of light of a specific wavelength and with a specific diameter is required. The calibration object (prism) is brought into the "minimum of deviation" position in the optical path. After the detector has been positioned to the deviated beam, the angles are read out. Then, the prism is "turned" so that the incidence and the exit plane are exchanged and, from that position, it is brought again into the "minimum of deviation" position. After the detector has been positioned again to the deviated beam, the angle is read out. Now, the beam is deviated towards the other side, symmetrically to the previous position. The deviation angle results from the mean value of these two positions. Then, the refractive index of the prism can be calculated from the angles, the wavelength and the measured ambient parameters.

Dispersion curves can be measured in the same way by varying the wavelength of the light used.

The measurement process begins with the measurement of the refractive angle. The prism is positioned on the prism table in such a way that -when the prism table is rotated the light beam of the AC meets the same surface elements in the centre of the prism surfaces. In addition, the two prism surfaces must be adjusted vertically to the axis of the prism table, i.e. the pyramidal error must be sufficiently small. To eliminate the pyramidal error, the height of the prism table is adjusted at three points. This adjustment is carried out visually with the AC. After the prism has been adjusted in the way described above, the angle measurement can be started. The automatic measuring cycle is performed in accordance with the following scheme:

1 The prism table is positioned to a starting position.

2The AC table is placed on a position shortly before the reflex of the first prism face.

3 The subsequent positioning of the AC table is controlled by the AC itself. The position is reached when the first reflex of the measuring face is detected. Now the AC is in autocollimation position to the optical axis of the AC. This angle value is read out on the divided circle and stored.

4 Then, the reflex from the second measuring face of the prism is searched with the AC and positioned, too. This value is stored as well. Thus, the prism angle is determined.

To eliminate the divided circle errors, the prism table can be adjusted to specific angle steps, and the measuring process described can be repeated. If the prism table is rotated in this way with a sufficiently number of steps (>12) by -all in all -360°, the mean value of all single measurements (all-around-measurement), is free from eccentricity and residual errors of the divided circle. This is an essential method for determining the refractive index with highest precision. If possible, the prism angle is determined in this way prior to each refractive index measurement. For reasons of construction, only an angle of 270° can be covered when the light source is installed.

After the prism angle is known, the lateral position of the prism is changed in such a way that the incoming beam of the light source exits the equivalent surface elements at the exit side of the prism in the minimum of deviation position. The displacement of the prism on the table is motor-controlled and is determined experimentally during the adjustment procedure which is performed prior to the measurements. The slit of the light source is illuminated with the light of a spectral lamp via a condenser. In front of the slit, suitable interference filters are placed on a motor-controlled filter disk for selection of the wavelengths used for the measurement.

To measure the deviation angle as exactly as possible, the angle must be determined in both possible prism positions. This is the "backwards" and the "forwards" deviation, for which the prism table has to be turned into the corresponding symmetric position. By averaging the two deviation angles, a certain part of the divided circle error is eliminated. Determination of the zero point (straight passage light source -AC) can be omitted as the positions "back" and "front" are symmetrically around this point.

The measurement of the refractive index is determined with respect to the ambient air in the measuring beam path.

The refractive index of transparent solids can be determined for specific discrete wavelengths of spectral lamps in the range from 400 nm -700 nm in a temperature range from 18 °C-22 C. The following wavelengths (nm) are available: Cd 644, Na 589, Hg 546, Cd 509, Cd 480, Hg 436, Hg 404.

The measuring room is completely separated from the operating panel, which is located in a separate room. After the calibration object has been adjusted, the operator leaves the measuring room. Then it takes a wait time between 6 to 16 hours until the temperature in the measuring room has been established. The entire measurement is performed automatically in the closed measuring room. It is not permitted to enter the room during this time.

The temperature conditions in the measuring room are controlled and monitored by a separate, computer-controlled temperature control situated in a side room. All temperatures prevailing at the measuring time are monitored and recorded by the measuring program.

The measurands are the angle between optical faces and the refractive index of prisms for specific discrete wavelengths of spectral lamps in the range from 400 to 700 nm in a temperature range from 18 to 22C. The following wavelengths (nm) are available: Cd 644, Na 589, Hg 546, Cd 509, Cd 480, Hg 436, Hg 404.

The measurement of the refractive index of glasses, liquids and other optically refracting substances is carried out by determining the prism angle and the deviation angle of prisms with the aid of a goniometer. The refractive index n_{rel} (relative to air) in the position of minimum of deviation is determined as follows:

$$n_{\rm rel} = \frac{\sin\left(\frac{\varphi + \delta_m}{2}\right)}{\sin\left(\frac{\varphi}{2}\right)} \tag{A.2.1}$$

with n_{rel} being the relative refractive index, measured relative to the so-called ambient "normal air", prism angle (apex angle), and δ_m the deviation angle of a light beam after having passed through the prism in the position of minimum of deviation.

The refractive index $n_{rel unkorr}$, measured against the ambient air, is converted into the refractive index n_{rel} by means of the refractive index n_{Luft} of the ambient air and the refractive index $n_{Normalluft}$ of "normal air" (20°C, 101325 Pa, 50% HR and 300 ppm CO₂) with the aid of the following formula:

$$n_{\rm rel}(\lambda) = n_{\rm rel\ unkorr}(\lambda) \cdot n_{\rm Luft}(\lambda) / n_{\rm Normalluft}(\lambda)$$
(A.2.2)

The refractive index of the ambient air is determined from the atmospheric air pressure, the air temperature and the humidity, and is calculated with the Edlén formula [1].

In addition, the temperature of the prism is adjusted to the desired temperature (accessible range: 18°C to 22°C) with an uncertainty of 0.01 K. The conversion of the refractive index from the current temperature into the desired temperature of 20.000°C is performed with tabular values for the temperature coefficient of the refractive index.

Uncertainty	Duugei			
Amount	Standard		Sensitivity	Uncertainty
	measurement uncertainty	Distribution	coefficient	contribution

Uncertainty budget

Prism angle φ (all-round measurement)	0.06 arcsec	Rectangle	$\frac{\sin\left(\frac{\delta_m}{2}\right)}{2\cdot\sin^2\left(\frac{\varphi}{2}\right)}$	2×10^{-7} (n = 1.5) 8×10^{-7} (n = 1.9)
Deviation angle m (not all-round measurement)	0.3 arcsec	Rectangle	$\frac{\cos\left(\frac{\delta_m + \varphi}{2}\right)}{2 \cdot \sin\left(\frac{\varphi}{2}\right)}$	$4 \times 10^{-7} (n = 1.5)$ $5 \times 10^{-7} (n = 1.9)$
Temperature solid	0.005 K	Rectangle	1	1×10 ⁻⁷
Refractive index air	3×10 ⁻⁷	Rectangle	1	3×10 ⁻⁷
Wavelength	1×10 ⁻³ nm	Rectangle	1	3×10 ⁻⁸
Minimum position prism	3 arcsec	Rectangle	1	1×10 ⁻⁸
Pyramidal error – angle	10 arcsec	Rectangle	1	1×10 ⁻⁷
Flatness of the prism surfaces	50 nm	Rectangle	1	4×10 ⁻⁷
Homogeneity of the prism material	50 nm	Rectangle	1	4×10 ⁻⁷

Standard uncertainty and expanded uncertainty for k=2

For the calibration certificate, the expanded measurement uncertainty, which is obtained from the standard measurement uncertainty by multiplication by the coverage factor k = 2, is calculated in accordance with GUM.

The expanded uncertainty for the determination of angles between surfaces amounts to 0.06 arcsec (if an "all-round measurement" is possible) and to 0.13 arcsec (for single measurements and if an "all-round measurement" is not possible).

For the refractive index of glasses in the range of n = 1.5, the expanded measurement uncertainty is determined to approx. 1.0×10^{-6} and in the range of n = 1.9 to approx. 1.4×10^{-6} .

A.3 Ukrmetrteststandart (Ukraine)

Description of the measuring facility:

Type: angle measurement system of GS-1L type is used for measuring the refracting angle of prism, and goniometer-spectrometer of GS-2 type is used for measuring the angle of minimum deflection of prism.

The method applied to measure the refractive index: the method of minimum deflection angle is used for measuring the refractive index [5]. We measured absolute refractive index 10 times for each prism for each wavelength. The temperature of the sample prism was from 19.9 °C to 20.1 °C during all measurements. The difference from 20.0 °C was compensated using the temperature coefficient of refractive index of each prism. Also, we monitored the environmental conditions (air temperature, pressure, humidity) during measurements, and used to obtain the refractive index of air by Edlen equation [1]. The temperature of the air was from 19.0 °C to 20.0 °C during all measurements.

The uncertainty claimed for the equipment: the uncertainty budgets which list each factor including the equipment are shown in Section 4 of Measurement protocol.

Diameter of aperture of measuring beam: 30 mm.

Description of how the comparison standard was adjusted:

The beam was met the faces of the prisms in their centre during the measurements of the prism angle and measurements of the angle of deviation.

Number of comparison standard	1		
Wavelength	589.29 nm		
Standard uncertainty	Standard	Standard	
Contribution due to:	Uncertainty	Uncertainty Type	
	Type A	В	
random errors in the measurement of refractive index	4.2×10 ⁻⁷		
measurement of the angle of the prism		1.5×10 ⁻⁶	
measurement of the angle of minimum deflection		3.1×10 ⁻⁶	
measurement of prism temperature		8.9×10 ⁻⁸	
measurement of air temperature		8.8×10 ⁻⁸	
measurement of humidity		2.9×10 ⁻⁸	
measurement of air pressure		5.2×10 ⁻⁷	
Combined uncertainty	4 2×10 ⁻⁷	3.5×10 ⁻⁶	
(Quadratic summation)			
Total combined standard uncertainty	3.5×10 ⁻⁶		
Expanded uncertainty (k=2)	7.0×10 ⁻⁶		
Level of confidence	95%		

Uncertainty budget

Number of comparison standard	1	
Wavelength	546.07 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty Type
	Type A	В
random errors in the measurement of refractive index	7.5×10 ⁻⁷	
measurement of the angle of the prism		1.5×10 ⁻⁶
measurement of the angle of minimum deflection		3.1×10 ⁻⁶
measurement of prism temperature		1.0×10 ⁻⁷
measurement of air temperature		8.5×10 ⁻⁸
measurement of humidity		2.6×10 ⁻⁸
measurement of air pressure		5.3×10 ⁻⁷
Combined uncertainty	7 5×10 ⁻⁷	3 5×10 ⁻⁶
(Quadratic summation)	7.3~10	5.5~10
Total combined standard uncertainty	3.5×10 ⁻⁶	
Expanded uncertainty (k=2)	7.1×10 ⁻⁶	
Level of confidence	95%	

Number of comparison standard	1	
Wavelength	643.85 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty Type
	Type A	В
random errors in the measurement of refractive index	6.3×10 ⁻⁷	
measurement of the angle of the prism		1.5×10^{-6}
measurement of the angle of minimum deflection		3.1×10 ⁻⁶
measurement of prism temperature		7.5×10 ⁻⁸
measurement of air temperature		8.7×10 ⁻⁸
measurement of humidity		2.8×10^{-8}
measurement of air pressure		5.2×10 ⁻⁷

Combined uncertainty	6 3×10 ⁻⁷	3 5×10 ⁻⁶
(Quadratic summation)	0.5 10	5.5*10
Total combined standard uncertainty	3.5×10 ⁻⁶	
Expanded uncertainty (k=2)	7.1×10 ⁻⁶	
Level of confidence	95%	
Number of comparison standard		1
Wavelength	508.58 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty Type
	Type A	В
random errors in the measurement of refractive index	5.3×10 ⁻⁷	
measurement of the angle of the prism		1.5×10^{-6}
measurement of the angle of minimum deflection		3.0×10 ⁻⁶
measurement of prism temperature		1.1×10 ⁻⁷
measurement of air temperature		9.1×10 ⁻⁸
measurement of humidity		2.9×10^{-8}
measurement of air pressure		5.2×10 ⁻⁷
Combined uncertainty	5.3×10 ⁻⁷	3.5×10 ⁻⁶
(Quadratic summation)	5.5~10	5.5~10
Total combined standard uncertainty	3.5×10 ⁻⁶	
Expanded uncertainty (k=2)	7.0×10 ⁻⁶	
Level of confidence	95%	

Number of comparison standard		1
Wavelength	479.99 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty Type
	Type A	В
random errors in the measurement of refractive index	7.2×10 ⁻⁷	
measurement of the angle of the prism		1.5×10^{-6}

measurement of the angle of minimum deflection		3.0×10 ⁻⁶
measurement of prism temperature		1.2×10 ⁻⁷
measurement of air temperature		7.9×10 ⁻⁸
measurement of humidity		2.1×10 ⁻⁸
measurement of air pressure		5.5×10 ⁻⁷
Combined uncertainty	7.2×10 ⁻⁷	3.4×10^{-6}
(Quadratic summation)		
Total combined standard uncertainty	3.5×10 ⁻⁶	
Expanded uncertainty (k=2)	7.0×10 ⁻⁶	
Level of confidence	95%	

Number of comparison standard	2	
Wavelength	589.29 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty
	Type A	Type B
random errors in the measurement of refractive index	7.9×10 ⁻⁷	
measurement of the angle of the prism		1.4×10 ⁻⁶
measurement of the angle of minimum deflection		4.3×10 ⁻⁶
measurement of prism temperature		4.0×10 ⁻⁸
measurement of air temperature		8.0×10 ⁻⁸
measurement of humidity		2.6×10 ⁻⁸
measurement of air pressure		4.7×10 ⁻⁷
Combined uncertainty	7 9×10 ⁻⁷	4 6×10 ⁻⁶
(Quadratic summation)	7.5~10	4.0/10
Total combined standard uncertainty	4.6×10 ⁻⁶	
Expanded uncertainty (k=2)	9,3×10 ⁻⁶	
Level of confidence	95%	

Number of comparison standard	2	
Wavelength	546.07 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty Type
	Type A	В
random errors in the measurement of refractive index	8.9×10 ⁻⁷	
measurement of the angle of the prism		1.4×10 ⁻⁶
measurement of the angle of minimum deflection		4.3`10 ⁻⁶
measurement of prism temperature		4.6×10 ⁻⁸
measurement of air temperature		8.8×10 ⁻⁸
measurement of humidity		3.1×10 ⁻⁸
measurement of air pressure		4.6×10 ⁻⁷
Combined uncertainty	8 9×10 ⁻⁷	4.6×10 ⁻⁶
(Quadratic summation)	0.7~10	1.0 10
Total combined standard uncertainty	4.7×10 ⁻⁶	
Expanded uncertainty (k=2)	9.3×10 ⁻⁶	
Level of confidence	95%	

Number of comparison standard	2	
Wavelength	643.85 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty Type
	Type A	В
random errors in the measurement of refractive index	6.2×10 ⁻⁷	
measurement of the angle of the prism		1.4×10^{-6}
measurement of the angle of minimum deflection		4.3×10 ⁻⁶
measurement of prism temperature		3.3×10 ⁻⁸
measurement of air temperature		8.1×10 ⁻⁸
measurement of humidity		2.7×10 ⁻⁸
measurement of air pressure		4.7×10 ⁻⁷

Combined uncertainty (Quadratic summation)	6.2×10 ⁻⁷	4.6×10 ⁻⁶
Total combined standard uncertainty	4.6×10 ⁻⁶	
Expanded uncertainty (k=2)	9.2×10 ⁻⁶	
Level of confidence	95%	

Number of comparison standard	2	
Wavelength	508.58 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty Type
	Type A	В
random errors in the measurement of refractive index	8.6×10 ⁻⁷	
measurement of the angle of the prism		1.4×10 ⁻⁶
measurement of the angle of minimum deflection		4.3×10 ⁻⁶
measurement of prism temperature		5.1×10 ⁻⁸
measurement of air temperature		8.3×10 ⁻⁸
measurement of humidity		2.7×10 ⁻⁸
measurement of air pressure		4.7×10 ⁻⁷
Combined uncertainty	8.6×10 ⁻⁷	4 6×10 ⁻⁶
(Quadratic summation)		
Total combined standard uncertainty	4.7×10 ⁻⁶	
Expanded uncertainty (k=2)	9.3×10 ⁻⁶	
Level of confidence	95%	

Number of comparison standard	2	
Wavelength	479.99 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty Type
	Type A	В

random errors in the measurement of refractive index	9.3×10 ⁻⁷	
measurement of the angle of the prism		1.4×10^{-6}
measurement of the angle of minimum deflection		4.3×10 ⁻⁶
measurement of prism temperature		5.5×10 ⁻⁸
measurement of air temperature		7.9×10 ⁻⁸
measurement of humidity		2.4×10 ⁻⁸
measurement of air pressure		4.8×10 ⁻⁷
Combined uncertainty	9 3×10 ⁻⁷	4.6×10 ⁻⁶
(Quadratic summation)		
Total combined standard uncertainty	4.7×10 ⁻⁶	
Expanded uncertainty (k=2)	9.3×10 ⁻⁶	
Level of confidence	95%	

Number of comparison standard	3	
Wavelength	589.29 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty Type
	Type A	В
random errors in the measurement of refractive index	1.1×10^{-6}	
measurement of the angle of the prism		9.0×10 ⁻⁷
measurement of the angle of minimum deflection		4.0×10 ⁻⁶
measurement of prism temperature		1.5×10 ⁻⁷
measurement of air temperature		8.8×10 ⁻⁸
measurement of humidity		2.7×10 ⁻⁸
measurement of air pressure		5.4×10 ⁻⁷
Combined uncertainty	1 1×10 ⁻⁶	4 2×10 ⁻⁶
(Quadratic summation)	1.1 10	1.2 10
Total combined standard uncertainty	4.3×10 ⁻⁶	
Expanded uncertainty (k=2)	8.6×10 ⁻⁶	
Level of confidence	95%	

Number of comparison standard	3	
Wavelength	546.07 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty Type
	Type A	В
random errors in the measurement of refractive index	9.9×10 ⁻⁷	
measurement of the angle of the prism		9.1×10 ⁻⁷
measurement of the angle of minimum deflection		4.0×10 ⁻⁶
measurement of prism temperature		1.8×10 ⁻⁷
measurement of air temperature		1.0×10 ⁻⁷
measurement of humidity		3.7×10 ⁻⁸
measurement of air pressure		5.1×10 ⁻⁷
Combined uncertainty	9 9×10 ⁻⁷	4 2×10 ⁻⁶
(Quadratic summation)		
Total combined standard uncertainty	4.3×10 ⁻⁶	
Expanded uncertainty (k=2)	8.5×10 ⁻⁶	
Level of confidence	95%	

Number of comparison standard	3	
Wavelength	643.85 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty Type
	Type A	В
random errors in the measurement of refractive index	8.8×10 ⁻⁷	
measurement of the angle of the prism		8.9×10 ⁻⁷
measurement of the angle of minimum deflection		4.1×10 ⁻⁶
measurement of prism temperature		9.5×10 ⁻⁸
measurement of air temperature		8.9×10 ⁻⁸
measurement of humidity		2.9×10 ⁻⁸

measurement of air pressure		5.3×10 ⁻⁷
Combined uncertainty (Quadratic summation)	8.8×10 ⁻⁷	4.2×10 ⁻⁶
Total combined standard uncertainty	4.3×10 ⁻⁶	
Expanded uncertainty (k=2)	8.6×10 ⁻⁶	
Level of confidence	95%	

Number of comparison standard	3	
Wavelength	508.58 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty Type
	Type A	В
random errors in the measurement of refractive index	7.0×10 ⁻⁷	
measurement of the angle of the prism		9.2×10 ⁻⁷
measurement of the angle of minimum deflection		4.0×10 ⁻⁶
measurement of prism temperature		2.2×10 ⁻⁷
measurement of air temperature		9.8×10 ⁻⁸
measurement of humidity		3.3×10 ⁻⁸
measurement of air pressure		5.3×10 ⁻⁷
Combined uncertainty	7.0×10 ⁻⁷	4 1×10 ⁻⁶
(Quadratic summation)	7.0710	T. 1/\10
Total combined standard uncertainty	4.2×10 ⁻⁶	
Expanded uncertainty (k=2)	8.4×10 ⁻⁶	
Level of confidence	95%	

Number of comparison standard	3	
Wavelength	479.99 nm	
Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty Type

	Type A	В
random errors in the measurement of refractive index	5.4×10 ⁻⁷	
measurement of the angle of the prism		9.2×10 ⁻⁷
measurement of the angle of minimum deflection		4.0×10 ⁻⁶
measurement of prism temperature		2.5×10^{-7}
measurement of air temperature		9.4×10 ⁻⁸
measurement of humidity		3.0×10 ⁻⁸
measurement of air pressure		5.4×10 ⁻⁷
Combined uncertainty	5 4×10 ⁻⁷	4 1×10 ⁻⁶
(Quadratic summation)		
Total combined standard uncertainty	4.2×10 ⁻⁶	
Expanded uncertainty (k=2)	8.3×10 ⁻⁶	
Level of confidence	95%	

A.4 National Institute of Advanced Industrial Science and Technology (AIST, Japan) Description of the measuring facility

We used measuring facility developed by AIST.

We used interferometric method developed by ourselves in this measurement. The absolute refractive index of sample prism (Ps) is obtained by the ratio between results of two interferometers in our instruments (Fig.A.4.1). The details of this method are described in Ref. [17, 18].

We measured absolute refractive index 60 times for each prism. The one-set of measurement (20 times continuous measurements) was executed within 7 hours. After every one-set, we reset and readjusted the prism. We report the averaged value of 60 measurements as the measurement results. Furthermore we estimated the uncertainty of repeatability and sample setting by analysis of variance using 60-measurement results for each prism. As the result of the analysis of variance, the uncertainty of sample setting was negligible and not listed in the uncertainty budget for all prisms (Section 3.1, 3.2 and 3.3).

The temperature of the sample prism (Ps) was from 19.9 °C to 20.1 °C during all measurements. Residual difference from 20.0 °C was compensated using the temperature coefficient of refractive index which were calculated by the equation described in "TIE-19:

Temperature Coefficient of the Refractive Index" downloaded from website of Schott (http://www.schott.com).

We monitored the environmental conditions (air temperature, pressure, humidity) during measurements, and used to obtain the refractive index of air by Ciddor's equation [19].



Fig. A.4.1 The schematic diagram of our method. Ps, sample prism; Pi, incident prism; ML, refractive index matching liquid; $\Delta \phi_{\rm l}$ (i=1,2),phase change of interferogram detected by interferometer 1 and 2. The respectively. absolute refractive index of Ps (n_s) is calculated by $n_s = n_a (\Delta \phi_1 / \Delta \phi_2)$, where n_a is refractive index of the air.

The uncertainty budgets which list each factor including the equipment are shown in tables below.

Diameter of aperture of measuring beam: 3 mm.

Description of how the comparison standard was adjusted

The sample prism (Ps) was adjusted so that the surface which faces to the Pi (surface β in Fig. A.4.1) was parallel with the translation axis of air slider. Using Fizeau type interferometer which use the reflection at surface β of Ps and surface β' of Pi, we confirmed that the gap between surface β and β' was constant during translation.

Uncertainty budget

1) Uncertainty budget of Prism No.1 at 633 nm, 20°C

Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty
	Type A	Type B
Alignment of interferometer 1		2.6×10^{-9}
Alignment of interferometer 2		2.1×10^{-8}
Alignment between prism and stage		$7.8 imes 10^{-7}$
Abbe's error (yaw)		4.9×10^{-8}
Abbe's error (pitch)		6.4×10^{-7}
Repeatability	5.8×10^{-7}	

Unwanted movement of Pi		2.6×10^{-7}
Temperature measurement of air		3.1×10^{-8}
Pressure measurement		$2.2 imes 10^{-8}$
Relative humidity measurement		$1.6 imes 10^{-8}$
Dispersion of refractive index		$5.6 imes 10^{-8}$
Temperature measurement of sample		$5.8 imes 10^{-8}$
Flatness of sample		5.9×10^{-7}
Temperature coefficient of refractive		$1.5 imes 10^{-9}$
index		
Combined uncertainty	$5.8 imes 10^{-7}$	$1.2 imes 10^{-6}$
(Quadratic summation)		
Total combined standard uncertainty	1.3×10^{-6}	
Expanded uncertainty (k=2)	2.7×10^{-6}	
Level of confidence	95%	

2) Uncertainty budget of Prism No.2 at 633 nm, 20°C

Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty
	Type A	Type B
Alignment of interferometer 1		6.9×10^{-9}
Alignment of interferometer 2		1.9×10^{-8}
Alignment between prism and stage		2.2×10^{-7}
Abbe's error (yaw)		$4.7 imes 10^{-8}$
Abbe's error (pitch)		6.1×10^{-7}
Repeatability	$4.5 imes 10^{-7}$	
Unwanted movement of Pi		2.4×10^{-7}
Temperature measurement of air		$2.8 imes 10^{-8}$
Pressure measurement		$2.0 imes 10^{-8}$
Relative humidity measurement		$1.5 imes 10^{-8}$
Dispersion of refractive index		3.3×10^{-8}
Temperature measurement of sample		$2.6 imes 10^{-8}$
Flatness of sample		1.8×10^{-7}
Temperature coefficient of refractive		6.5×10^{-10}
index		
Combined uncertainty	4.5×10^{-7}	7.2×10^{-7}

(Quadratic summation)		
Total combined standard uncertainty	8.5×10^{-7}	
Expanded uncertainty (k=2)	1.7×10^{-6}	
Level of confidence	95%	

3) Uncertainty budget of Prism No.3 at 633 nm, 20°C

Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty
	Type A	Type B
Alignment of interferometer 1		$6.4 imes 10^{-9}$
Alignment of interferometer 2		$2.2 imes 10^{-8}$
Alignment between prism and stage		$2.7 imes 10^{-7}$
Abbe's error (yaw)		$5.5 imes 10^{-8}$
Abbe's error (pitch)		7.1×10^{-7}
Repeatability	6.2×10^{-7}	
Unwanted movement of Pi		3.5×10^{-7}
Temperature measurement of air		3.2×10^{-8}
Pressure measurement		$2.3 imes 10^{-8}$
Relative humidity measurement		$1.6 imes 10^{-8}$
Dispersion of refractive index		9.1×10^{-8}
Temperature measurement of sample		1.0×10^{-7}
Flatness of sample		$2.2 imes 10^{-7}$
Temperature coefficient of refractive		$1.8 imes 10^{-9}$
index		
Combined uncertainty	6.2×10^{-7}	8.8×10^{-7}
(Quadratic summation)		
Total combined standard uncertainty	1.1×10^{-6}	
Expanded uncertainty (k=2)	2.2×10^{-6}	
Level of confidence	95%	

A.5. Istituto Nazionale di Ricerca Metrologica (INRiM, Italy).

Description of the measuring facility

Type of rotating prism is described in [20].

Minimum deflection angle method was applied to measure the refractive index.

The uncertainty for the equipment was evaluated for each measurement.

Diameter of aperture of measuring beam was around 10 mm.

During adjusting procedure the comparison standard was simply placed on the rotating platform so that the light spot hits the center of the prism face.

Measurement results

Number of	Absolute refractive index	Uncertainty of			
comparison	reduced to standard	measurement			
standard	conditions	(k = 2)			
Wave length $\lambda = 632.8$ nm (He-Ne red line)					
NBAF 10	1.6676949	3.72E-06			
NBK 7	1.5157664	3.42E-06			
SF 1	1.7128440	3.88E-06			

Uncertainty budget for NBAF 10 Prism

Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty
	Type A	Туре В
Apex angle measurement		4.34E-07
Minimum angle measurement		1.35E-06
Air refractive index measurement		8.07E-07
Partial combined uncertainty		1.64E-06
Corrected combined uncertainty for the	1.86E-06	
temperature interpolation effect		
Expanded uncertainty (k=2)	3.72E-06	
Level of confidence	95%	

Uncertainty budget for NBK 7 Prism

Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty
	Type A	Type B
Apex angle measurement		3.32E-07
Minimum angle measurement		1.38E-06
Air refractive index measurement		7.33E-07
Partial combined uncertainty		1.60E-06
Corrected combined uncertainty for the	1.71E-06	
temperature interpolation effect		
Expanded uncertainty (k=2)	3.42E-06	
Level of confidence	95%	

Uncertainty budget for SF 1 Prism

Standard uncertainty	Standard	Standard
Contribution due to:	Uncertainty	Uncertainty
Controlation due to.	Type A	Type B
Apex angle measurement	v	4.85E-07
Minimum angle measurement		1.53E-06
Air refractive index measurement		8.30E-07
Partial combined uncertainty		1.80E-06
Corrected combined uncertainty for the	1.94E-06	
temperature interpolation effect		
Expanded uncertainty (k=2)	3.88E-06	
Level of confidence	95%	

References

- 1. K.P.Birch, M.J. Downs, "An updated Edlen Equation for the refractive index of air", Metrologia 30 (1993) 155-162.
- 2. G. N. Vishnyakov and S. V. Kornysheva, "Provision of measurement uniformity in refractometry of solids, liquids and gases", Measurement Techniques 48 (2005) 1099 1102.
- 3. G. N. Vishnyakov, G. G. Levin, S. V. Kornysheva, "State standards. The State primary standard for the unit of refractive index", Measurement Techniques 47 (2004) 1039 1043.
- G. N. Vishnyakov, G. G. Levin, S. V. Kornysheva, G. N. Zyuzev, M. B. Lyudomirskii, P. A. Pavlov, and Y. V. Filatov, "Measuring the refractive index on a goniometer in the dynamic regime", J. Opt. Technol. 72 (2005) 929-933.
- 5. GOST 28869, "Optical materials. Methods of the refractive index measurements".
- 6. ISO 8402:1994-04, "Quality Management Terms".
- 7. ISO 17025, "General requirements for the competence of testing and calibration laboratories".
- BIPM/JCGM "Evaluation of measurement data Guide to the expression of uncertainty in measurement", JCGM 100 (2008), <u>http://www.bipm.org/en/publications/guides/gum.htm.</u>
- 9. VIM: International Vocabulary of Basic and General Terms in Metrology. DIN Deutsches Institut für Normung (editor), Beuth Verlag, Berlin, Köln 1994. ISO International Organization for Standardization, Geneva 1993.
- K.-J. Rosenbruch, H. Stenger, "Das neue Pr\u00e4zisions-Goniometer zur Brechzahlmessung in der PTB (The new precision goniometer for refractive index measurement at PTB)", Messtechnik Informationen, Firma Heidenhain, Traunreut, 8(1980)13-9.
- 11. D. Tentori, J. R. Lerma, "Refractometry by minimum deviation: accuracy analysis" Optical Engineering 29 (1990) 160 -168.
- 12. L. W. Tilton, "Prism size and orientation in minimum-deviation refractometry, Journal of Research of the National Bureau of Standards" 6 (1930) 59 -76.
- 13. L. W. Tilton, "Permissible curvature of prism surfaces and inaccuracy of collimation in precise minimum-deviation refractometry" Journal of Research of the National Bureau of Standards 11 (1933) 25 -57.
- 14. L. W. Tilton, "Standard conditions for precise prism refractometry, Journal of Research of the National Bureau of Standards" 14 (1935) 393 -418.

- 15. L. W. Tilton, "Prism refractometry and certain goniometrical requirements, Journal of Research of the National Bureau of Standards" 2 (1929) 909 -930.
- 16. A. J. Werner, "Methods in high-precision refractometry of optical glasses" Appl. Optics 7 (1968) 837 -844.
- 17. Y. Hori, A. Hirai, K. Minoshima and H. Matsumoto, "High-accuracy Interferometer with a prism pair for measurement of the absolute refractive index of glass" Appl. Opt. 48 (2009) 2045-2050.
- 18. Y. Hori, A. Hirai, K. Minoshima, "Prism-pair Interferometry by homodyne interferometers with a common light source for high-accuracy measurement of the absolute refractive index of glasses" Appl. Opt. 50 (2011) 1190-6.
- 19. P. E. Ciddor, "Refractive index of air: new equations for the visible and near infrared," *Appl. Opt.* 35 (1996) 1566–1573.
- 20. M. Pisani and M. Astrua, "Prism Refractive Index Measurement at INRiM" Meas. Sci. Technol. 20 (2009) 095305.