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COOMET Supplementary Comparison

COOMET.PR-S3

Refractive Index

(COOMET Project 438/RU/08)

Technical Protocol

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1. Introduction

Participants agreed to conduct a Supplementary Comparison on the absolute refractive index measurements.

The aim of this comparison is to ensure the correctness and comparability of refractive index measurements carried out by the Participants of the comparison, within the uncertainties claimed for their measuring facility.

The present Technical Protocol has been approved by the COOMET TC 1.7 “Photometry and Radiometry”.

2. Organization

2.1 Participants

VNIIOFI (Russia) is acting as a pilot laboratory in the comparison among the participants.

Participants must be able to demonstrate traceability to an independent realization of the quantity, or make clear the route of traceability to the quantity via another named laboratory.

Participants will accept the CCPR documents regarding to key and supplementary comparisons and the Technical Protocol written down in this document and commit themselves to follow the procedures strictly.

Once the Protocol has been agreed, no change to the protocol may be made without prior agreement of all Participants and the TC.

2.2 Participants' details

Table 1. Participant's details

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

Participant 2	State Enterprise All-Ukrainian State Research and Production Centre for Standardization, Metrology, Certification and Consumers Rights Protection. Ukraine	Ukrmetrteststandart	Andriy B. Glebov Nataliya M. Parkhomenko	Tel.: +38 (044) 526-36-98 Fax: +38 (044) 526-36-98 E-mail: optic@ukrcsm.kiev.ua natapar@mail.ru
Participant 3	National Institute of Advanced Industrial Science and Technology. Japan	AIST	Yasuaki Hori	Tel: +81-29-861-4211 Fax: +81-29-861-4080 E-mail: y-hori@aist.go.jp
Participant 4	Istituto Nazionale di Ricerca Metrologica. Italy	INRiM	<u>Marco Pisani</u> , Milena Astrua	Tel +39 011 3919 961 Fax +39 011 3919 959 E-mail: m.pisani@inrim.it
Participant 5	Kazakh Institute of Metrology RSE. Kazakhstan	KazInMetr	Kuanbayev Ch. B. Suyeubayeva G.A.	Tel: +7 (7172) 79-33-70, +7 701 4068890 Fax: +7 (7172) 24-32-97 E-mail: chin_as@mail.ru , gulaikhan-api@mail.ru

2.3 Form of comparison

The comparison will cover 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 will be carried out in the following sequence:

Pilot → Participant 1 → Participant 2 → Participant 3 → Participant 4 → Participant 5 → Pilot

The Pilot will measure the artefacts for the first time and send the set to the Participant 1. The Participant 1 will carry out measurements of the refractive index for each transfer standard

and will send the set to the next Participant and then will send the measurement results and uncertainty budget to the Pilot by e-mail and etc. Finally the comparison standards will return to the Pilot. The Pilot will measure the artefacts for the second time.

All results are to be communicated directly to the Pilot as soon as it possible.

Each Participant has 1 month for measurements and transportation the set of transfer standards to the next Participant. With its confirmation to participate, each Participant has confirmed that it is capable to perform the measurements during the time allocated to it. This ensures that the relatively short timetable to complete the comparison is met.

To prevent delays of customs formalities and insure the artefacts from damages during the transportation from one Participant to another Participant, it is recommended to carry the artefacts by hand or send them by an express-courier (for ex. UPS, DHL etc.).

If, for some reasons, the measurement facility is not ready or take place an unplanned damage of any facility, or customs clearance takes too much time in a country the Participant should contact the Pilot immediately to discuss further details and changes in the measurement timetable. However, if the delay is too long, such changes might not be possible. If this is the case, the Participant and their results might be excluded from the final report. Exclusion may also occur if the results are not available at the prescribed time.

Each Participant will bear the costs incurred himself, including the costs for the transport and insurance of the comparison standard. No additional costs will be incurred by the Participants.

2.4 Timetable

For the time being, only relative statements are made with respect to the time schedule. The exact dates will be fixed after the comparison programme has been confirmed by the Participants and the date fixed on which the comparison will be started.

Table 2. Timetable of the Comparisons

	Activity	Date
•	Start of comparisons Pilot measures comparison standards and sends comparison standards to Participant 1	August – September 2010
•	Participant 1 measures comparison standards, sends measurement protocol and uncertainty budgets to Pilot by e-mail, sends comparison standards to Participant 2	October 2010
•	Participant 2 measures comparison standards, sends measurement protocol and uncertainty budgets to Pilot by e-mail, sends comparison standards to Participant 3	November 2010
•	Participant 3 measures comparison standards, sends measurement protocol and uncertainty budgets to Pilot by e-	December 2010 – January 15 th , 2011

	mail, sends comparison standards to Participant 4	
•	Participant 4 measures comparison standards, sends measurement protocol and uncertainty budgets to Pilot by e-mail, sends comparison standards to Pilot. Pilot sends comparison standards to Participant 5	January 16 th – February 2011
•	Participant 5 measures comparison standards, sends measurement protocol and uncertainty budgets to Pilot by e-mail, sends comparison standards to Pilot	March 2011
•	Pilot measures comparison standards Pilot describes the analysis process in details to all Participants	April – May 2011
•	Pilot sends uncertainty budgets to all participants (pre-draft A Process 1)	June 2011
•	Pilot sends relative data to each participant and reported values to Participants for checking (pre-draft A Process 2)	July 2011
•	Responses to Relative Data from all participants due	August 2011
•	Comments on the uncertainty budgets closed	September 2011
•	Responses to comments on uncertainty budgets and revision of uncertainty closed	October 2011
•	Draft A distributed	November 2011
•	Comments on draft A due	December 2011 – January 2012
•	Draft A-2 sent to Participants for approval	February 2012
•	Draft B sent to COOMET-PR Committee	March 2012
•	Draft B approved	April – May 2012
•	Draft B submitted to CCPR	June 2012
•	Final Report published	July 2012

2.5 Handling of transfer standards

Participants shall inform the Pilot about the fact of receiving the transfer standards.

The transfer standards must be examined immediately up on receipt. The condition of the artefacts and associated packaging should be noted and reported to the Pilot. Please use the form in Appendix A.3.

The transfer standards should only be handled by authorized persons and stored in such a way to prevent damage.

Working surfaces of the artefacts should be cleaned by pure alcohol as required. If a transfer standard is damaged the Participant should contact the Pilot immediately so that an appropriate decision can be made as regard replacement or partial use of results. If any damages of the artefacts occur due to the fault of Participant, the Participant will cover the costs to the Pilot.

However, appropriate insurance should be taken out by Participants to cover the cost of such a replacement if the damage occurred during transportation.

Please inform the Pilot and the next Participant by e-mail when the measurements are completed to arrange a suitable date for dispatch.

When the measurements are completed, the transfer standards should be re-packaged in their original transit cases. Ensure that the content of the package is complete before shipment. Always use the original packaging.

2.6 Transportation of transfer standards

It is of utmost importance that the transfer standards will be transported in a manner in which they will not be lost, damaged or handled by un-authorized persons.

Packaging for the transfer standards has been made which should be suitably robust to protect the artefacts from being damaged during transportation.

The preferable way of transportation is hand luggage, either by personal road transport, train, or in an aircraft cabin. However, recognising that this may result in high financial costs to some Participants it is possible to send the artefacts by express courier (for ex. DHL, UPS etc.). Package must be marked as 'Fragile'.

The transfer standards should be accompanied by a suitable customs carnet (where appropriate) or documentation identifying the items uniquely. Pilot will prepare ATA-Carnet. The packaging will be lockable e.g. by clasp, but is easy to open with minimum delay to allow customs inspections to take place.

Transportation is each Participant's responsibility. Each Participant covers the costs for its own measurements, transportation, shipment insurance and any customs charges as well as for any damages that may have occurred within its country or in transit. The total cost of the transfer standards is EUR 6 113.00 (Six thousand one hundred thirteen Euro). The overall costs for the organisation of the comparison are covered by the Pilot. The Pilot has no insurance for any loss or damage of the artefacts during transportation.

3. Description of transfer standards

The transfer standard is a set of three prismatic samples made of different types of glass. Prisms have identification numbers: 01, 02 and 03. Each prim has different refractive index and different temperature coefficients of the refractive index Parameters of the prisms are listed in the Table 3.

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.

Table 3. Parameters of the prisms used as the comparisons artefacts

Prism No.	Glass type, (Schott)	Nominal Refractive index, n_d	Temperature coefficient of refractive index $\Delta n_{\text{abs}}/\Delta T [10^{-6}/\text{K}]$			Dispersion $n_F - n_C$	Apex angles (approximate)
			1060,0	e	g		
01	N-BAF 10	1,670	2.4	3.5	4.5	0,014222	60°; 60°; 60°
02	N-BK 7	1,517	1.1	1.6	2.1	0,008054	55°; 65°; 60°
03	SF 1	1,717	3.6	6.4	9.8	0,024307	53°; 67°; 60°

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

The following apexes and surfaces must be 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 procedure

Each Participant should measure absolute refractive index of the transfer standards using its own method of measurement and facilities.

Before starting measurements the Participant must inspect the transfer standards for damage. Any damage must be documented using the appropriate form in Appendix A.3. The appropriate photos and drawings must be attached. The Pilot must be informed immediately by e-mail.

Each Participant determines the absolute refractive index of the prisms using his measuring facility and applying the measurement and adjustment criteria established by him.

The refractive index is to be determined at the following discrete wavelengths (or at several wavelengths selected from this list): Cd 480; 509; Hg 546; Na 589.3; He+Ne 632.8; Cd 644 nm.

If the Participant is not able to carry out measurements at the listed wavelength it's allowed to calculate refractive indexes for these wavelengths using the results measured at other wavelengths.

The temperature of the prism during the measurements should be within the limits of 19,9 °C to 20,1 °C. Each Participant uses its own method of temperature measurement.

In case of goniometric measurements, the refractive index is to be determined in the minimum of the deflection, if possible; other measuring methods may be used if good reasons are stated for this.

The measuring light beam should be circular. Participants can 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 is 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, must be recorded and reported. The refractive indexes must 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%.

When the measurements are completed, thy working surfaces of the prisms should be cleaned by pure alcohol as required.

5. Reporting of results

On completion of the measurements by the Participant the results of these measurements should be sent to the Pilot by e-mail as soon as it possible after measurements. The signed report must also be sent in paper form by mail. In case of any differences, the paper forms are considered to be the definitive version.

Pilot will reconfirm these values to the Participant to ensure that the correct Participant values are being used by the Pilot and that no mix-up has occurred.

As soon as possible after measurements have been made the Participant should provide a full measurement report. The report should include: description of method and procedure of independent realization of the quantity, or route of traceability to another named laboratory; description and schematic diagram of the measuring facility; and full uncertainty analysis.

6. Measurement uncertainties

The uncertainties of measurement shall be estimated according to the ISO “Guide to the Expression of Uncertainty in Measurement” (GUM 1995). The uncertainty budget (table of uncertainty components and uncertainty contributions), as well as description of measurement technique and facility must be submitted from each laboratory together with their results. The overall uncertainty values alone will not be sufficient. An example of the uncertainty budget is presented in the table in Appendixes A.2. All uncertainty components should be given as standard uncertainties ($k=1$), and the expanded uncertainty should be given for a coverage factor of $k = 2$.

7. Preparation of the comparison report

Before preparation the comparison report itself, the Pilot organises the Pre-Draft A processes.

7.1. Pre-Draft A Process 1: Verification of reported results

After the results have been submitted by all the Participants the Pilot, within two months, sends to each Participant, individually, their reported values as received by the Pilot for verification.

Each Participant reviews their reported results received from the Pilot and examine if there are any errors. If any errors are found, the Participant should correct their results at this stage.

Each participant must respond to the Pilot within three weeks from receiving the verification data, to confirm that there is no problem in their data or to request any corrections.

After this process period is over, any corrections of participants' reported results due to errors or misinterpretation by pilot lab are not allowed.

7.2. Pre-Draft A Process 2: Review of uncertainty budgets

The Pilot distributes to all Participants the uncertainty budgets of all the Participants to allow them to review other labs' uncertainty budgets. This is done within two months.

Any Participants including the Pilot can send questions or comments on other Participant's uncertainty budgets and ask for further information, for example, if a Participant's uncertainty is considered unusually small, or if some important uncertainty components are missing. Comments/questions from any Participants are accepted within six weeks from distribution of the uncertainty budgets. Comments/questions should be sent to the Pilot, who will then forward the comments anonymously to the Participant being asked and copied to all other Participants. The Pilot takes the records of all communication.

The Participants who received comments must respond promptly and, if necessary, can revise their uncertainty budget. This, however, does not force the Participant to revise it. At this stage, any Participants can submit correction of their uncertainty budget, even without receiving comments. However, revision of uncertainty components is allowed only in the direction to increase the overall uncertainty.

Responses to comments and revisions of uncertainty budgets (if any) are accepted within two months from distribution of the uncertainty budgets.

Replies to comments should go to the Pilot. If any correction or changes of the uncertainty budget is submitted in this stage, the changes of values and the reason will be reported in the appendix of the comparison report.

7.3. Pre-Draft A Process 3: Review of Relative Data

After the results have been submitted from all the participants and the measurements of the pilot lab have been completed, within two months, the pilot lab prepares "Relative Data" of each participant, which are the data reduced to show only the internal consistency of all the transfer standards measured at each participant lab. The Relative Data can be obtained by calculating the ratios of values of all transfer standards measured by the Participant and by the Pilot, and normalizing the ratios to their mean. This normalization removes any relationship of the Participant's absolute scale to the pilot lab, and leaves only internal consistency information. For spectral data, the normalization is done at each wavelength. Any data reflecting the relationship of the absolute scales between participant and pilot are not allowed to be disclosed in this Pre-Draft A process.

The Pilot distributes the Relative Data of all Participants to all Participants. The Participants review the Relative Data and examine the internal consistency. If significant inconsistency in any of the transfer standards is identified, the Participant can propose removal of the data of the particular transfer standards. Removal of results is discussed and agreed by the Participant and the Pilot, and all Participants will be informed of such decisions.

Each Participant must respond to the Pilot within one month from distribution of Relative Data, to confirm that there is no problem in their data or to request any corrections.

If a Participant finds any errors (clerical, technical, or any other reasons) in their reported values, from any anomalous feature in their Relative Data, the Participant can submit a correction of the results at this stage. But, it is the Participants' responsibility to identify any anomalous feature of their Relative Data that imply errors.

If the pilot lab finds obvious anomalous results of any participant that cannot be identified from Relative Data, a warning may be sent to all participants (without specific information).

If any corrections of data are submitted from participants in this stage, the changes of values and the reason will be reported in the appendix of the report of comparison. If data of any transfer standards are removed, the fact will be stated in the report of comparison.

Note: Processes 1, 2, and 3 above can proceed simultaneously.

7.4. Preparation, Distribution and Review of Draft A

After the Pre-Draft A processes are complete, the Pilot prepares and distributes Draft A to all the Participants, which discloses the absolute results of the comparison with identification of all the participating labs. The Draft A should tabulate all the results. The Draft A should be distributed within six months after completion of all the measurements of the comparison.

The default method for calculating comparison reference values (CRV) is the weighted mean with cut-off. Use of other methods can be discussed only when the Pilot finds serious problems in using the default method, and should be discussed before distribution of Draft A.

The cut-off value for the uncertainty, as a default, is determined as the average of the uncertainty values of those Participants that reported uncertainties smaller than or equal to the median of all the Participants. (For example, if there are 10 participants, the cut-off value will be the average of the 5 smallest values of uncertainty.)

The weights are determined based on the participants' reported uncertainties adjusted by the cut-off, combined with the transfer uncertainty of the comparison (reproducibility of measurements at the pilot lab and other components associated with difference in measurement conditions between Pilot and Participants, etc.).

Preparing the Draft A the Pilot should follow the "Guidelines for CCPR Comparison Report Preparation"

Each participating lab carefully reviews all the data presented in Draft A, and reports to the Pilot if they find any clerical errors made by the Pilot or send any other comments. Comments should be sent within two months from distribution of Draft A.

If a participating lab has found error(s) that they made in their measurements or in data analysis that affected the reported results, the fact should be reported to the Pilot. The corrections are documented in the appendix of the report. In this case, under the Pilot lab's decision, the lab's results (or part of the results) may be excluded from the CRV calculation, with the fact stated in the report.

If comments are made by one or more participants, these comments should be circulated to all participants, and if they are significant, the Pilot can discuss with participants whether and how changes are to be made for the next Draft A version. If necessary, further data can be distributed as Supplement to Draft A.

When changes are made to address comments, the revised draft will be called Draft A-2 and are distributed again to all the participants for approval. In this case, the revised draft should be distributed within two months from closing comments. If further comments are made to the revised draft, the process can be repeated (Draft A-3, ...) or the Pilot can consult TC in case of dispute.

Draft A is considered as confidential for only the participants. The data in Draft A should not be distributed or presented to general public.

7.5. Draft B

When the final version of Draft A has been agreed by all participants, it becomes Draft B. The Pilot submits Draft B to TC for approval within four months from distribution of Draft A (if no further version of Draft A needs to be prepared).

Draft B must include tables of unilateral Degrees of Equivalence. The tables can be in the main body or an Appendix of the report.

Draft B will be reviewed by TC (and no longer by participants). As the result of review, changes in Draft B may be requested to the Pilot lab. If a revision is produced, it is called Draft B-2 (B-3, ... if repeated) and reviewed again by TC. Participants do not participate in this process unless some major revision is proposed by TC.

When Draft B-x is approved by TC, it will be submitted to CCPR WG-KC for comments only, not for approval. If there are comments, the COOMET TC Chair might be requested to revise the report.

If there are no comments, the Draft B will be forwarded to CCPR Executive Secretary for distribution in CCPR for approval. If there are comments, the RMO PR TC Chair will be requested to revise the report. If there are no comments, the Draft-B becomes the Final Report.

7.6. Publication of Final Report

The final report of the comparison will be published in the *Technical Supplement of Metrologia* (electronic media on the website).

After a final report has been published, the Pilot lab sends to all participants, with copies to the RMO P&R TC chairs, a reminder to check the consistency of their CMCs with the comparison results and to report to the participant's RMO TC chair (with a copy to the pilot) about their evaluation and any proposed actions in case of inconsistency, within two months from the reminder.

From the Pilot lab:

Svetlana Kornysheva



COOMET PR TC Chair



Boris Khlevnoy

A.1 Measurement protocol

The attached measurement summary should be completed for each comparison standard.

The following is to be stated in the description:

Participant _____

Date _____

1. Description of the measuring facility (as detailed as possible) :
 - 1) Type
 - 2) the method applied to measure the refractive index
 - 3) the procedures of evaluation of uncertainty components
 - 4) Diameter of aperture of measuring beam
2. Description of alignment of the transfer standards
3. Measurement results

Identification of comparison standard	Absolute refractive index reduced to standard conditions	Total standard Uncertainty ($k = 1$)
Wavelength $\lambda_1 = \dots$		
1		
2		
3		
Wavelength $\lambda_i = \dots$		
1		
2		
3		

A.2 Uncertainty budget form

Standard uncertainty	Standard Uncertainty Type A	Standard Uncertainty Type B
Contribution due to:		
Combined standard uncertainty		
Total combined standard uncertainty		
Expanded uncertainty ($k=2$)		

A.3 Inspection of the transfer standards

**1) Has the standard transportation package been opened during transit?
e.g. Customs...(Y/N) If Yes please give details.**

2) Is there any damage to the transportation package? (Y/N) If Yes, please give details.

3) Are there any visible signs of damage to the standards? (Y/N) If Yes, please give details.

Laboratory:

.....

Date: **Signature:**