

Detailed plan for ILC/PT

1. Identification and address of the PT provider

The organizer of the project is PT Provider UCLSB Ltd, 5300 Gabrovo, 6 Ivan Dimov Str., BG 206627780.

2. General data for the Technical Project

2.1. The interlaboratory technical project MC 06/2023 is organized by PT provider USLSB – expert team, consisting of:

- Project leader – eng. Ilian Iliev
- Project coordinator – assoc. prof. Tony Venelinov
- Materials' Engineer – prof. Dr. eng. Ivan Rostovski
- Metrologist – eng. Ilian Iliev
- Expert in data collection and coding – eng. Ilian Iliev
- Technical expert for data evaluation – assoc. prof. Dr. eng. Liubomir Brakalov

The members of the expert team of the PT provider have the necessary qualification (specialized training), skills and experience in organizing interlaboratory comparisons and proficiency tests.

2.2. The procedure for conducting Interlaboratory Technical Project MC 06/2023 includes organizing, conducting and evaluating test material (raw material, product) through the participation of laboratories in interlaboratory comparison and proficiency testing according to established conditions, approved program, confirmed invitation and application for participation and execution of the provided instruction.

Along with the assessment of competence, the project aims to examine the ability of laboratories to use the assessment of test uncertainty in order to present more authoritatively, reliably and qualitatively in the measurement (determination) of indicators (characteristics) that are characterized by significant scattering (uncertainty) inherent in natural and artificial building materials.

2.3. The technical project is carried out according to EN ISO/IEC 17043:2010, as follows:

- Split sample testing scheme: The starting material will be distributed simultaneously among the participating test laboratories. Sufficient material will be retained to verify homogeneity, possible subsequent testing and sample stability.
- Homogeneity will be determined by appropriate testing by laboratories where sampling, homogenization, reduction and distribution of samples are performed in the presence of an expert team of the PT provider and according to its procedure. After evaluation of the results by the technical expert and verification of homogeneity, the samples will be sent to the participants. Otherwise, a new homogenization is performed according to the current procedures of the PT provider.
- An identification code will be assigned to each of the laboratories, which will be marked in the upper right corner of the instruction for confidentiality. The applied PT

PROFICIENCY TESTING PLAN

Page 2 of 7

scheme is one-time depending on the frequency and at the same time multifaceted depending on the number of participants.

- Laboratories accept as “true value” (accepted value) and its uncertainty the value obtained by the independent expert through statistical processing of all equal results of laboratories.

2.4. The statistical processing is applied in compliance with the requirements of the following statutory documents:

- [1] BDS EN ISO/IEC 17025:2018 (General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2017))
- [2.1] BDS 17397:2005 Part 1 INTERNATIONAL VOCABULARY OF BASIC AND GENERAL METROLOGICAL TERMS
- [2.2] ISO/IEC GUIDE 99:2008 International vocabulary of metrology — Basic and general concepts and associated terms (VIM), 3th edition, 2012 (2008 with minor changes)
- [3] BAS QR 18, v. 3:2013, PROCEDURE FOR INTERLABORATORY COMPARISONS AND PROFICIENCY TESTINGS.
- [4] EN ISO/IEC 17043:2010: Conformity assessment General requirements for proficiency testing.
- [5.1] [://www.nist.gov/pml/wmd/labmetrology/roundrobins.cfm](http://www.nist.gov/pml/wmd/labmetrology/roundrobins.cfm)
- [5.2] [://www.nist.gov/pml/wmd/labmetrology/upload/pt-ilc-report-terminology-guidance-8-4-2011-2.doc](http://www.nist.gov/pml/wmd/labmetrology/upload/pt-ilc-report-terminology-guidance-8-4-2011-2.doc) - 2011-08-04
- [6] IUPAC: The International Harmonized Protocol for Proficiency Testing of Analytical Chemistry Laboratories, 2006, Pure Appl. Chem., 78, No 1, pp. 145-196
- [7] ISO 13528:2022 Statistical methods for use in proficiency testing by interlaboratory comparisons
- [8.1] ISO 5725:2019 Accuracy (trueness and precision) of measurements methods and results. Part 2. Basic methods for determination of repeatability and reproducibility of a standard measurement method
- [8.2] Bozhanov E., Vuchkov I., Statistical methods of modeling and optimization of multi-factor objects, S., Technika, 1983
- [8.3] ISO 21748:2018 (ISO 21748:2017), Guidance to the use of repeatability, reproducibility and trueness estimates in measurement uncertainty evaluation
- [8.4] http://en.wikipedia.org/wiki/Welch%27s_t_test (Welch – Aspin test)
- [9] Harvey D., Modern analytical chemistry, McGraw-Hill, 2000
- [10] Дёрффель К., Статистика в аналитической химии, М., Мир, 1994
- [11] Eurachem / CITAC Guide, Quantifying uncertainty in analytical measurement, Third Edition, 2012
- [12] Bituminous /asphalt/ mixtures standards
- [12.1.] EN 1426:2015 Bitumen and bituminous binders - Determination of needle penetration
- [12.2.] Determination of Softening Point according to EN 1427:2015 Bitumen and bituminous binders - Determination of the softening point - Ring and Ball method.
- [12.3.] EN 12593:2015 Bitumen and bituminous binders - Determination of the Fraass breaking point.
- [12.4.] EN ISO 2592:2017 Petroleum and related products - Determination of flash and fire points - Cleveland open cup method (ISO 2592:2017).
- [12.5.] EN 15326:2007+A1:2009 Bitumen and bituminous binders - Measurement of density and specific gravity - Capillary-stoppered pyknometer method.

PROFICIENCY TESTING PLAN

Page 3 of 7

- [12.6.] EN 12592:2014 Bitumen and bituminous binders - Determination of solubility.
- [13] Provider's procedures
- [13.1] QP 4.6.1 HANDLING THE TEST OBJECTS
- [13.2] QP 4.7.2 ASSESSMENT OF THE ASSIGNED VALUE OF PARAMETERS AND ENSURING ITS QUALITY AND ASSESSMENT OF THE PARTICIPANT'S PERFORMANCE IN ILC.
- [14] Other documents related to the tests and ILC
- [14.1] Accreditation and Quality Assurance (2006) 11: 23–28, Validation of the Volhard method for chloride determination in food, M. Naceur Haouet, M. Serena Altissimi, Marisa Framboas, Roberta Galarini
- [14.2] EURACHEM / CITAC Guide CG 4 Quantifying Uncertainty in Analytical Measurement, Third Edition, (Appendix F),
- [14.3] The Fitness for Purpose of Analytical Methods A Laboratory Guide to Method Validation and Related Topics,
https://www.eurachem.org/images/stories/Guides/pdf/MV_guide_2nd_ed_EN.pdf
- [15.1] Davis, J.R. Metals Handbook Desk Edition . ASM, 1998.
- [15.2] Callister. W.D, JR. (2007). Materials Science and Engineering: An Introduction. 7th edition. John Wiley & Sons, Inc.

3. Subcontracted activities

Accredited PT provider (organizer) UCLSB for this technical project will use the services of subcontractor Construction Laboratory "BKS - Gorna Oryahovitsa" at "BKS - Gorna Oryahovitsa" EAD Gorna Oryahovitsa for Bituminous but only for sampling, homogenization, reduction and splitting of samples under the control (supervision) of the PT provider.

Present persons during the process of sample preparation will be the Project leader – eng. Ilian Iliev, the project coordinator – assoc. prof. Dr. Tony Venelinov and the materials engineer – prof. Dr. eng. Ivan Rostovski.

Homogeneity will be determined by appropriate tests by the subcontracted laboratories the presence of PT provider and according to its procedures. After assessing the results by the technical expert and authentication (verification of the) homogeneity, the samples will be sent to participants. Otherwise, we proceed to a new homogenization under the current procedures of PT provider.

The stability of the samples will be determined by the subcontractors at the request of the PT provider through the same tests that were performed to determine the level of homogeneity.

The assessment of the level of homogeneity and stability of the samples will be performed by the technical expert of the PT provider and for this purpose will be used the statistical methods and information contained in EN ISO/IEC 17043:2010, EN ISO/IEC 13258:2022, standard methods included in this suitability test, etc.

4. Criteria for participation and adequacy assessment

Accredited laboratories are invited to participate in the project, those that are in the process of accreditation or have many years of practical experience.

The processing, analysis and evaluation of the obtained results will be performed by the technical expert according to the documents specified in item 2.4.

PROFICIENCY TESTING PLAN

Page 4 of 7

Only assessments that are appropriate for the purposes of the PT are included in the project.

5. Number and type of expected participants in the proficiency testing scheme

Based on the accumulated experience of the organizer (PT provider ULSSB) in conducting PT schemes, the expected number of laboratories is about 20 and all are from the field of construction. The minimum number of participants in MC 06/2023 is set at 5.

6. Selection of measurements or characteristics for complete identification of methods

Interlaboratory technical project MC 06/2023 shall cover the following product: Hardened concrete, according to the indicated standards and indices in the following table:

Test subject	Code of the standard	Name of the standard	Tested parameters (characteristics)
Bitumen and bitumen products	EN 1426	Bitumen and bituminous binders - Determination of needle penetration.	Needle penetration ¹⁰⁰
	EN 1427	Bitumen and bituminous binders - Determination of the softening point - Ring and Ball method.	Softening point - Ring and Ball method ¹⁰⁰
	EN 12593	Bitumen and bituminous binders - Determination of the Fraass breaking point.	Fraass breaking point ¹⁰⁰
	EN ISO 2592	Petroleum and related products - Determination of flash and fire points - Cleveland open cup method (ISO 2592).	Flash point ¹⁰⁰
	EN 15326	Bitumen and bituminous binders - Measurement of density and specific gravity - Capillary-stoppered pyknometer method.	Density ¹⁰⁰
	EN 12592	Bitumen and bituminous binders - Determination of solubility.	(Determination of) Solubility ¹⁰⁰

Note: All products and features are within the scope of accreditation of PT Provider.

All the tests and sample preparations shall be performed under the latest editions of the standards.

7. Potential main sources of errors that may hinder good performance in MC 06/2023.

- Ignorance of procedures and relevant methods for sampling, testing and reduction.
- Improper application of procedures according to the relevant methods of sampling, testing and reduction.
- Use of inappropriate technical means.
- Use of defective or uncalibrated technical devices
- Incorrect estimation of measurement uncertainty
- Wrong transfer of data and information in the forms provided by the PT provider.

8. Instructions and directions to participants (including levels of data protection, confidentiality), encoding of participants both individually and in the report.

Electronically, as well as from the website of the PT provider USLSB <https://ptprovider.sslsb.org/bg/pt-schemes/> laboratories receive "Invitation to participate in inter-laboratory comparison and proficiency testing" – QF 4.4-3 and "Application" for participation in interlaboratory comparison and proficiency testing" – QF 4.4-4. After

confirming their participation and after the expiration of the deadlines for applications, the laboratories receive their samples. Electronically, each requested laboratory receives a package of documents containing "Sampling Protocol" – QF 4.4-6 and "Instructions for conducting interlaboratory comparison and proficiency testing" – QF 4.4-5, the laboratories perform in the instructions term individual definitions of each characteristic/indicator selected by them. The deadlines for confirming the participation of the laboratories, for receiving and testing the samples, as well as the deadlines for feedback with the results obtained and the presentation of the report will be mentioned in the invitation and instructions for conducting (only the participating laboratories), which will accompany each test specimen submitted with a unique sample and laboratory identification code.

Upon receipt of the test samples, the laboratories also receive "test result sheets" – QF 4.4-7, which indicate the unique code of the respective laboratory, as well as the sample code known only to the project coordinator. The heads of the relevant conformity assessment bodies undertake to complete QF 4.10 -1 "Privacy Statement".

The confidentiality of the results until their final presentation is achieved as follows. The coordinator informs the CAB that it is sending a test sample so that it is ready to receive it. The coded sample is transported to the CAB packed in a way that ensures identity, integrity, anonymity and metrology and stability. Upon acceptance, the CAB completes QF 4.4-6, which is returned to the coordinator. After completion of the tests and assessment of the uncertainty, the CAB fills in the form with the results Annex QF 4.4-7, which contain only the minimum necessary information and the participant's code. Annex/Annexes QF 4.4-7 are sent to the data collection and coding expert responsible for information only. The expert in data collection and coding eng. Iliyan Iliev collects the results and transmits them in the form of QL 4.4-3 "Coded list of results" and QL 4.4-1 "List of participants" participating in MC 06/2023 and transmits them to the technical expert for data evaluation – assoc. prof. Dr. eng. Lyubomir Brakalov for processing and general and individual evaluation. The Technical Expert has signed a privacy statement stating that he is not allowed to disseminate the information – QF 4.10-1. However, in principle, he does not have any access to primary information on CABs and tests.

As can be seen from the above, there is a separation of information flow and movement of samples, which ensures anonymity and impartiality of the participants and the independent expert.

For their part, the participating CABs and coordinators undertake not to disseminate information from the Interlaboratory Technical Project MC 06/2023 report and results in any form, unless requested to do so by SNAS or another authorized state body. When presenting results to such a body, CABs undertake to inform it of the confidential nature of the interlaboratory comparison. The results can also be used in a CAB audit, but this should also be ensured by a privacy statement from the auditor. That is why all participants in MC 06/2023 also sign declarations of confidentiality, which are an integral part of the Interlaboratory Technical Project MC 06/2023.

9. Transport of samples:

The samples will be transported by the organizers in appropriate packaging from approved courier service providers (Bulgarian Post and Speedy).

10. Performing the test

During the test, participants undertake not to use external information that would suggest results from testing in other laboratories. For this reason, it is necessary to perform the tests as soon as possible. The heads of the relevant conformity assessment bodies undertake to complete QF 4.10-1 "Privacy Statement". For gross violations, participants can be removed from the scheme.

11. Completion of the interlaboratory comparison and proficiency test

The deadline for submission of complete documentation will be mentioned in the instructions to be sent by the organizer. Test reports will be sent to the e-mail specified by the organizer in the instructions.

12. Reporting and evaluation of ILC / PT results

The final report QF 4.8-1 will be prepared by the Technical Expert of data evaluation – assoc. prof. Dr. eng. Lyubomir Brakalov within the deadline specified in the instructions and will contain statistical processing of results and conclusions on the effectiveness of the process.

The statistical analysis used will be in accordance with EN 17043:2010., ISO 13528:2022 and others.

In accordance with the rules for participation, CABs accept as "true value" (accepted, assigned or assigned value) and its uncertainty their estimates obtained by the technical expert through standard statistical processing of all peer laboratory results.

To eliminate outliers, this MC will use the Grubbs criterion, supplemented by limiting the data used by the three-sigma criterion and the Interquartile Range Method. The Kolmogorov-Smirnov (KS) test is used to verify that all data lie within the normal distribution interval with a confidence level of at least 0.95.

The assessment of the reliability of the data in this MC will be used to assess the accepted value and its uncertainty made with the Kolmogorov and Smirnov criteria.

The report will include the following assessments and criteria for the performance of the participants: bias-deviation; z - assessment; ζ - assessment, according to the requirements of EN 13528:2022.

A document (Certificate QF 4.9-1) on the participation of laboratories in interlaboratory comparison and suitability testing will be attached.

13. Terms of the proficiency testing scheme and information for participants

- Applications for participation (QF 4.4-4) – **until 31.08.2023.**
- Dispatch of the test objects – **by 30.09.2023.**
- Sending the sample tracking code together with the package of documents ("Instructions for conducting interlaboratory comparison and suitability testing" – QF 4.4-5, "Sampling protocol" – QF 4.4-6, "Declaration (for confidentiality) for protection of industrial and trade secrets" – QF 4.10-1, "Test results sheets" – (QF 4.4-7) and "Feedback sheet" (QF 5.7-1) of the requested participants by e-mail: **by 30.09.2023 at the latest.**
- Conducting the test by the participants **01.11-05.11.2023 r.**
- Presentation of test results (QF 4.4-7) of the results sheets from **06.11.2023 r.** for all indicators.

PROFICIENCY TESTING PLAN

Page 7 of 7

- Preparation of a final report **by 12.12.2023**.
- Sending a final report with a certificate to the participants **by 30.12.2023**.

Detailed information will be provided to the participants through the "Instructions for conducting interlaboratory comparison and suitability testing" - QF 4.4-5 which will be sent together with the package of documents by e-mail.

14. Actions in case of lost or damaged test object

- The test sites within the Republic of Bulgaria reach from 24 to 48 hours after their dispatch and from 5 to 20 days abroad.
- All participants are notified by e-mail about the sending of samples. In the attached file of this e-mail is sent the full package of documents of the provider ("Instructions for interlaboratory comparison and proficiency testing" – QF 4.4-5, "Protocol for sampling" – QF 4.4-6, "Declaration (for confidentiality) for protection of industrial and commercial secrecy" – QF 4.10-1, "Test results sheets" – (QF 4.4-7) and "Feedback sheet" (QF 5.7-1)). For the participants from abroad they also receive the tracking number.
- In case the sample is unusable or damaged, the participant sends objective evidence/photos, together with the protocol for acceptance of the sample. A member of the PT provider's team sends a new sample of the backup samples provided.
- In case of loss of a test object, regardless of whose fault it is on the PT provider, participant or courier service provider, a member of the provider's team sends a new sample of the backup samples provided for that purpose.

Important for the participants: Upon receipt of the samples and documents, the participants are obliged to fill in electronically and send immediately form QF 4.4-6 "Protocol for acceptance of samples" for each sample by e-mail: ms2010@abv.bg to be assured of their integrity and suitability for testing.

15. Feedback and communication with participants

QF 5.7-1 "Feedback sheet" is filled in. It is sent by e-mail together with the other documents accompanying the test samples. The return of QF 5.7-1 "Feedback sheet" is also made via e-mail to the provider within the time limit specified in QF 4.4-5 Instructions for conducting a proficiency test. Throughout the period of the Interlaboratory Technical Project MC 06/2023, the head of the respective participating laboratory or other authorized person maintains direct contact with the Project Manager and / or Coordinator. mail and.

16. Planning or participating in technical meetings with participants

If necessary and desired by the participant, a meeting is planned to discuss the method of sample preparation, the method of sampling and/or testing, the primary and secondary results, the final results of the evaluations and the final report. This is usually necessary when the results are significantly distracted. The meeting can also be held online through various platforms such as Zoom, Skype and others.

Approved by:
Manager of PT provider UCLSB:

/ eng. Ilian Iliev/