METROLOGY SERVICE

Director
Metrology service Ltd.

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PROFICIENCY TESTING PT.UA.13.2.2026
SWEETENERS, PRESERVATIVES, AND CAFFEINE IN FOOD PRODUCTS
PROFICIENCY TESTING PROGRAMME – ROUND 1 MAY 2026

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2. SUMMARY

2.1. The purpose of proficiency testing in food product is to demonstrate the laboratory's competence (as described in ISO/IEC 17043:2023[1]) and improve the reliability of test results.

2.2. This proficiency testing involves the use of inter-laboratory comparisons to confirm the performance of individual laboratories' abilities and/or identify areas of improvement.

2.3. This is the final report on the PT.UA.13.2.2025 ROUND 1 held in April-May 2026. This report is issued according to ISO/IEC 17043[1] and PT.UA.13.2.2025 ROUND 1 Programme. The report is issued in two languages – Ukrainian and English. English should be considered as the basic language of the report. Both versions of this report can be found at: http://www.metrologyservice.com.ua

2.4. A total of 12 participants have reported. Their results are presented in the next clauses.

2.5. Technical experts list and/or subcontractors for this round can be provided to the Participant by request.

2.6. Any calculations, formulas, raw and intermediate data used in this round can be provided to the Participant by request, except confidential information about other participants and information that may contain commercial secret.

2.7. If the Participant does not agree with the proficiency testing results or has any comments on the Provider's work, one can submit a complaint or appeal within 10 days. More information on the complaint procedure can be found at https://www.metrologyservice.com.ua/ or by contacting the Provider.

2.8. The Provider declares that all results presented in this report are confidential. Each participant is identified by a unique number assigned to them based on their registration application for each round of the program separately. This number is confidential information and may only be disclosed at the participant's request

2.9. Where applicable, the metrological traceability of assigned values is ensured, as confirmed by the use of measurement equipment properly calibrated in accordance with current EA and NAAU policies.

2.10. The uncertainty of the assigned values (for quantitative evaluation) may be provided upon the participant's request.

2.11. All users of this report are prohibited from copying or reproducing it, in whole or in part, without the written consent of the Provider.

3. GENERAL PROTOCOL FOR PROFECIENCY TESTING

3.1. MANAGEMENT SYSTEM.

3.1.1. The functioning management system of Metrology service Ltd. (further - Provider) complies with ISO/IEC 17043[1] requirements and covers all aspects of proficiency testing (further - PT) for all proficiency tests.

3.2. SAMPLES PREPARATION, HOMOGENITY AND STABILITY

3.2.1. Provider has used a validated procedure and appropriate technical experts and contractors for the samples' selection, production, homogenization and division designs that is proved

to be satisfactory for the purposes of PT programme PT.UA.13.2.2025 ROUND 1. Details of test material preparation and homogenization is not published in the report, though can be provided to the Participant by request. Tests, required to prove (validate) homogeneity and stability of samples were performed by competent contracting laboratories according to [1]. These results with statistics are published in the report.

3.2.2. Participants may contact the Provider to request details of test material selection, preparation, homogenization and division of those test material samples, for which they tested in PT. Such information can be provided to the Participant in confidence and only if it cannot compromise other Participants and/or is not a commercial secret.

3.2.3. During sample preparation, all necessary procedures (where applicable) were performed. A sample preparation report can be provided upon request.

3.3. DISPATCH AND RECEIPT OF SAMPLES

3.3.1. Samples of test material – **Approximately 50±5 g of Apple puree** were dispatched 28.04.2026 according to schedule of proficiency testing programme PT.UA.13.2.2025 ROUND 1.

3.3.2. Each produced and identified sample was sealed in a plastic foil bag.

3.3.3. A total of 12 participants received one sample. Results were returned from 12 participants.

3.3.4. The samples were shipped to participants via the commercial delivery service “Nova Poshta” LLC (or another option).

3.4. FOLLOW-UP SERVICES

3.4.1. If a participant wishes to obtain advice/consultation on any aspect of their performance, one should contact the Provider. Provider can (with agreement with Participant) pass on the Participant’s inquiry to a technical expert and/or contracting laboratory.

3.5. PERFORMANCE ASSESSMENT

3.5.1. Provider expressed Participant’s results as traditional z-scores according to [1].

3.5.2. A certified reference value was chosen as the assigned value for each parameter.

3.5.3 The target standard deviation for each analyte was chosen from either the appropriate form of the Horwitz equation, method trial standard deviation (if stated in the method from inter-laboratory comparisons), standard deviation from the previous trials (PT rounds), or the robust trial standard deviation, after the removal of outliers. The choice was made using current industry practices used in other collaborative trials and proficiency testing schemes.

3.5.4. z-Scores were deemed satisfactory if $|z| \leq 2$. z-Scores were deemed questionable if $2 < |z| \leq 3$ (marked yellow in tables). If $|z| > 3$, the results were considered to be unsatisfactory (marked red in tables). The calculations were made according to [1,2,3]. Provider recommends corrective actions if $|z| > 3$ and preventive actions if $2 < |z| \leq 3$.

3.5.5. All results in this round were considered to be satisfactory.

3.5.6. Participant №4 stated result for «Mass fraction of sorbic acid, mg/kg» according to the «LI 00.031-4 BENZOIC & SORBIC ACIDS, PARABENS BY HPLC (Nordic Committee on Food Analysis: NMKL method N° 124, 2. Ed., 1997)» instead of the «ДСТУ 5050:2008» method proposed

by the Provider. This result was assessed by the Provider but was not taken into account when calculating robust mean and robust SD

3.5.7. Participant №5 stated the additional method «M 04-58-2009» near result for «Mass fraction of sorbic acid, mg/kg» of the «ДСТУ 5050:2008». This result was assessed by the Provider and was taken into account when calculating robust mean and robust SD.

3.5.8. Participant №6 stated result for «Mass fraction of sorbic acid, mg/kg» according to the «ДСТУ EN 12856:2003» instead of the «ДСТУ 5050:2008» method proposed by the Provider. This result was assessed by the Provider but was not taken into account when calculating robust mean and robust SD.

3.5.9. Participant №10 stated result for «Mass fraction of sorbic acid, mg/kg» according to the «ДСТУ 4948:2008» instead of the «ДСТУ 5050:2008» method proposed by the Provider. This result was assessed by the Provider but was not taken into account when calculating robust mean and robust SD.

4. HOMOGENITY AND STABILITY ASSESSMENT

4.1. Samples were assessed for homogeneity and stability after blending and packing by selecting five samples of material at random from all those produced. Three samples were tested in duplicate under repeatability conditions as only 19 samples were produced according to [6]. Samples for stability tests were stored in appropriate conditions for the period of preparation and test submission for this round.

4.2. Statistical analysis of the resulting data for homogeneity and stability was carried out using the industry standard Cochran's 'C' test and analytical variance test for 'sufficient homogeneity' according to [3] or Annex B.2[4].

4.3. Produced samples were found to be sufficiently homogeneous and stable for every analyte according to programme, except for those that can be considered equivalent or homogeneity can be assumed from other analyte homogeneity.

4.4. «Mass fraction of sorbic acid, mg/kg» (ДСТУ 5050:2008)

Mass fraction of sorbic acid, mg/kg				ДСТУ 5050:2008						
Дослідження гомогенності/Homogenity test										
Аналіз викидів за тестом Кохрана(C -тест)/Cohran's C test for outliers						Аналіз на 'достатню однорідність'/Test for 'sufficient homogenity'				
Номер зразку/ Sample number	Результат/ Result A	Результат/ Result B	Average	SD²		Номер зразку /Sample number	Результат/ Result A	Результат/ Result B	SUM	Difference²
1	98,50	101,50	100,000	4,5000	0,00	1	98,50	101,50	200,00	9,0000
2	99,80	100,50	100,150	0,2450	0,00	2	99,80	100,50	200,30	0,4900
3	101,30	102,60	101,950	0,8450	0,00	3	101,30	102,60	203,90	1,6900
										11,1800
Mean	100,700		Worst pair	4,5000		Mean	100,700			
Max	102,60		SUM of SD²	5,5900		Max	102,60			
Min	98,50		C	0,8050		Min	98,50			
			Ccr, 5%	0,9669						
			Ccr, 1%	0,9933		Analytical variance S²an	1,8633	SD	1,4353	
			Conclusion			Sanal	1,3650	RSDR	1,4253	
			5%	PASS		Ssums	4,7100			
			1%	PASS		MSb	2,3550			
					Between sample variance S²sam		0,2458			
Remarks										
1. Cohran's C test is described in ISO 5727-2 and ISO 13528:2022										
2. Test for 'sufficient homogenity' is performed according to Annex B ISO 13528:2022										

Source of σp value to use Use(write '1')	Source	σp
	C>13.8%, HORWITZ	100,3494
1	120ppb<C<13.8%, HORWITZ	8,0464
	C<120 ppb	22,1540
MASS NEGATIVE POWER FOR HORWITZ EQUATION(%=2, ppb=9,ppm=6)		6
	SD	1,3102
	Trial SD	8,0690
	Target SD chosen	8,0464
	σ²all	5,8271
	Replicates	3
	F1	2,996
	F2	4,276
	Critical value	25,4240
	Between sample variance S²sam	0,2458
	Sufficient homogenity test	PASS

5. DATA SUMMARY

Method	ДСТУ 5050:2008
	Mass fraction of sorbic acid, mg/kg
No of Results	12
No of Results |z|>3	0
No of Results |z|>3, %	0,000
Mean	100,340
Min	90,200
Max	110,000
SD	6,141
Median	99,455
Robust mean (assigned value)	100,222
Robust SD	5,667
Spiked level	100,000
Recovery, %	100,222
SD from method (Tr.SD)	N/A
SD from Horwitz eq.	8,014
Target SD	8,014
Source of target SD of PT	Horwitz

6. RAW DATA

Method	ДСТУ 5050:2008
Laboratory number	Mass fraction of sorbic acid, mg/kg
1	99,0
2	90,2 ± 2,1
3	110,00
4	106,1 мг/кг
5	102,25
6	93,02 ± 0,97
7	109,66
8	97,241
9	99,11
10	102,3
11	99,8 мг/кг ± 14,9 мг/кг
12	95,40

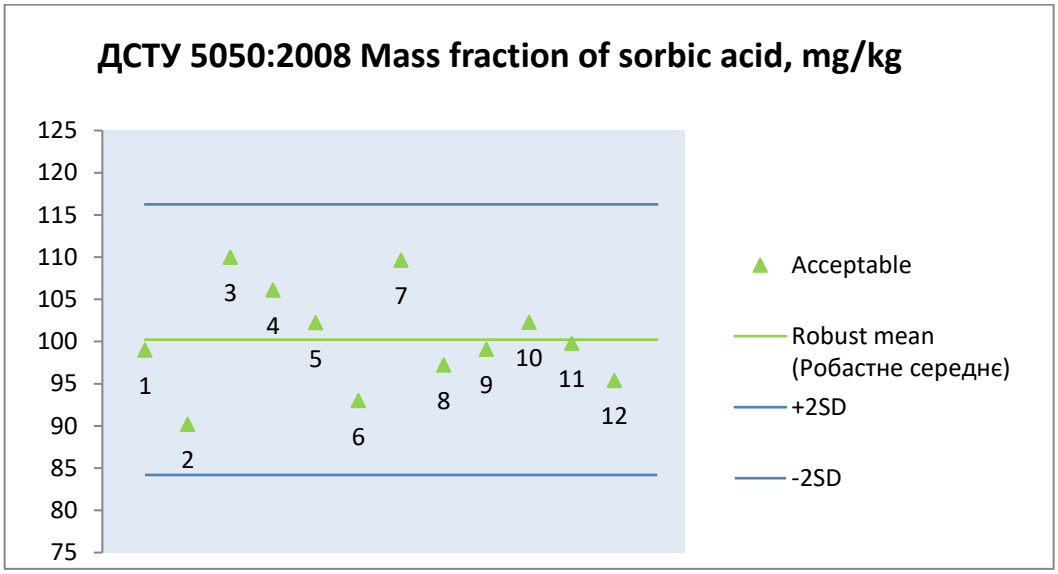
7. Z SCORES

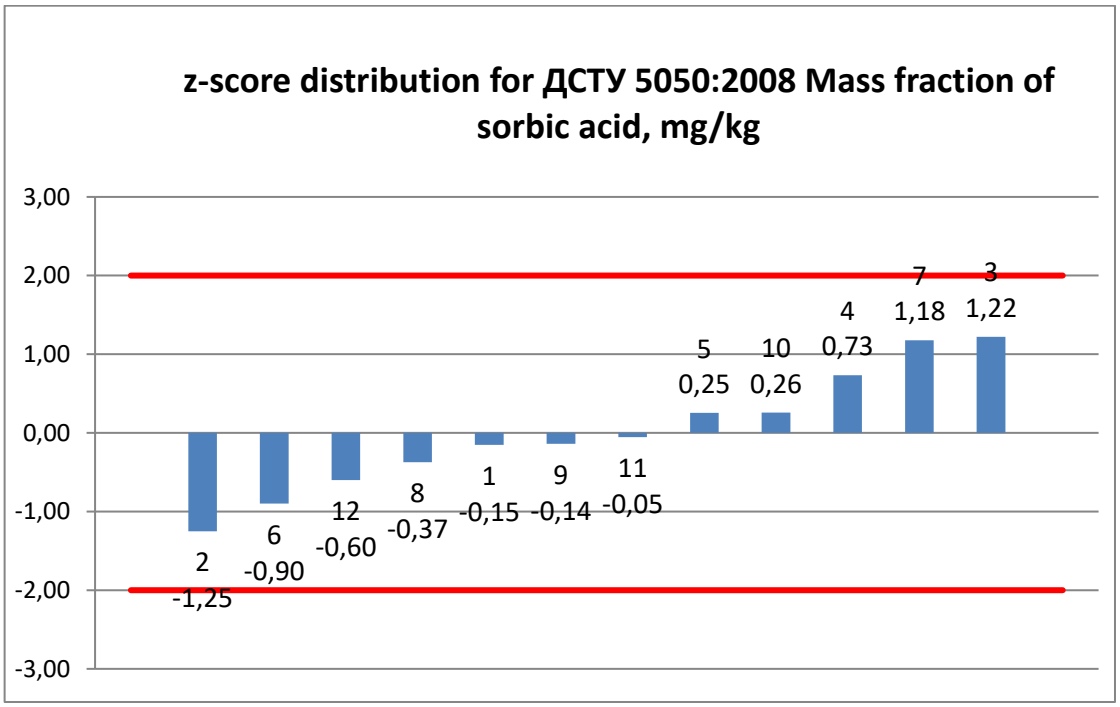
Method	ДСТУ 5050:2008
Laboratory number	Mass fraction of sorbic acid, mg/kg
1	-0,15
2	-1,25
3	1,22
4	0,73
5	0,25
6	-0,90
7	1,18
8	-0,37
9	-0,14
10	0,26
11	-0,05
12	-0,60

Remarks:
1. Green colored cells contain results that are considered to be satisfactory.
2. Red colored cells contain results that are considered to be not satisfactory.
3. Results that are considered to be questionable are marked by yellow colored cell.
4. Blank cell – results were not reported by the Participants.

8. Z SCORE PLOTS AND RESULTS CHARTS.

8.1. ДСТУ 5050:2008 Mass fraction of sorbic acid, mg/kg





10. NORMATIVE REFERENCE

1. ISO/IEC 17043:2023 Conformity assessment – General requirements for the competence of proficiency testing providers.
2. Analytical Methods Committee, Robust Statistics – How not to reject outliers Part 1. Basic Concepts, Analyst, 1989, 114, 1693-1697.
3. Fearn, T. and Thompson, M, A new test for ‘sufficient homogeneity’, Analyst, 2001, 126, 1414-1417.
4. ISO 13528:2022 Statistical methods for use in proficiency testing by interlaboratory comparison.
5. ISO 33405:2024 Reference materials — Approaches for characterization and assessment of homogeneity and stability.
6. ILAC Discussion Paper on Homogeneity and Stability Testing, April 2008.