

Bozhko Natalia
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PROFICIENCY TESTING PT.UA.1.4.2017
MILLING PRODUCTS (QUALITY)
PROFICIENCY TESTING REPORT
ROUND 2 APRIL 2018(ENG)

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2. SUMMARY

2.1. The purpose of proficiency testing in milling products is to determine the characteristics of the operation (as described in ISO\IEC 17043[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.1.4.2017 Round 2 held in April 2018. This report is issued according to ISO\IEC 17043 [1] and PT.UA.1.4.2017 Round 2 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 9 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.

3. GENERAL PROTOCOL FOR PROFICIENCY TESTING

3.1. MANAGEMENT SYSTEM.

3.1.1. The functioning management system of Metrology service Ltd. (further - Provider) complies with ISO\IEC 17043:2010[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.1.4.2017 Round 2. Details of test material preparation and homogenization are 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 [2-8]. 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.

3.3. DISPATCH AND RECEIPT OF SAMPLES

3.3.1. Samples of test material – **Wheat flour** were dispatched 02.04.2018 according to schedule of proficiency testing programme PT.UA.1.4.2017 Round 2.

3.3.2. Each produced and identified sample was hermetically sealed.

3.3.3. A total of 9 participants from different regions of Ukraine received one sample. Results were returned from 9 participants.

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.

3.4.2. Surplus samples from this round are available for sale as certified reference materials(CRM) with the certified values and uncertainties. Please e-mail Provider for details.

3.5. PERFORMANCE ASSESSMENT

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

3.5.2. The assigned value for each analyte was calculated as the robust mean of the trial data using Huber H15 method [2,3]

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 $|z| \geq 3$, the results were considered to be unsatisfactory (marked red in tables). The calculations were made according to [1,3,5].

3.5.5. Only 0.84% of all results in this round are considered to be unsatisfactory. 4.7% of all results were deemed unsatisfactory in Round 1.

3.5.6. Participant number 6, 7, 8 and 9 stated ГОСТ 26361-84 Whiteness of flour, c.u. instead of ДСТУ 4870:2007 Whiteness of flour, c.u.. Provider assessed their results.

4. HOMOGENITY AND STABILITY ASSESSMENT

4.1. Samples were assessed for homogeneity and stability after blending and packing by selecting ten samples of material at random from all those produced. Six of these samples were tested in duplicate under repeatability conditions as only 40 samples were produced according to [7]. Four other samples for stability tests were stored in appropriate conditions for the period of preparation and test submission for this round. They were also tested in duplicate.

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 [3,4].

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. ISO 712:2009 Moisture, %

4.5. Data for all analytes

Method(Метод)	ISO 712:2009	ISO 20483:2013	ISO 2171:2007/ДСТУ ISO 2171:2009	ISO 3093:2009/ДСТУ ISO 3093:2009	ISO 7305:1998	ISO 21415-1:2006/ДСТУ ISO 21415-1:2009	ISO 21415-2:2015/ДСТУ ISO 21415-2:2009	ISO 21415-2:2015	ISO 21415-3:2006/ ISO 21415-4:2006	ISO 27971:2015	ISO 27971:2015	ISO 27971:2015	ISO 27971:2015	ISO 27971:2015	ISO 5529:2007	ГОСТ 27493-87	ГОСТ 27839-88	ГОСТ 27839-88	ДСТУ 4870:2007
	Moisture, %	Protein content, % (Expressed on dry matter, factor for converting nitrogen content to	Ash content, %	Falling number,s	Fat acidity, mg KOH/100 g	Wet gluten content, %	Wet gluten content, %	Gluten index	Dry gluten content, %	Deformation energy, W	Maximum pressure parameter, P	Mean abscissa at rupture, L	Index of swelling, G	Curve configuration ratio, P/L	Sedimentation index – Zeleni test, ml	Acidity, °of acidity	Wet gluten content, %	Index of gluten deformation	Whiteness of flour, c.u.

Homogeneity and stability(Гомогеність та стабільність)

Cohran's 'C' test(С-тест "Кохрана")

Critical value(5%,10pairs)=0,602	0,5199	0,3545	0,5269	0,3906	0,6577	0,3607	0,5444	0,3298	0,3902	0,1655	0,3103	0,3596	0,3130	0,1440	0,4211	0,4000	0,1712	0,2462	0,1839
Mean Result	14,2495	11,4730	0,5067	446,2000	34,0525	28,0825	24,9620	96,6590	8,2445	326,1000	103,9000	89,8000	21,0650	1,1670	46,3000	2,2700	25,7620	53,7500	59,1350
Conclusion(Висновок)	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS

Analytical variance test(тест аналітичної дисперсії)

S^2_{anal}	0,0014	0,0046	0,0004	80,0000	1,2041	0,1996	0,0053	0,0946	0,0051	59,2000	5,8000	8,9000	0,0575	0,0013	1,9000	0,0050	0,0790	3,2500	0,0435
S_{anal}	0,0372	0,0676	0,0196	8,9443	1,0973	0,4468	0,0727	0,3076	0,0716	7,6942	2,4083	2,9833	0,2398	0,0354	1,3784	0,0707	0,2810	1,8028	0,2086
S^2_{sample}	0,0103	0,0145	0,0000	61,2889	0,9569	0,0108	0,0417	0,4272	0,0132	103,0556	9,4222	6,6722	0,1541	0,0054	2,6722	0,0059	0,0000	5,0000	0,5099
σ_p	0,3775	0,3179	0,0224	11,9580	2,4510	0,5299	0,4996	1,5320	0,2401	23,0800	5,7220	6,7960	0,8000	0,1630	2,5000	0,3790	0,5076	5,0000	1,6620
σ_p source	Horwitz	Horwitz	Horwitz	Trial SD	Trial SD	Horwitz	Horwitz	Trial SD	Horwitz	Method Tr SD	Trial SD								
σ^2_{all}	0,0128	0,0091	0,0000	12,8694	0,5407	0,025274	0,0225	0,2112	0,0052	47,9418	2,9467	4,1567	0,0576	0,0024	0,5625	0,0129	0,0232	2,2500	0,2486
Critical value	0,0255	0,0217	0,0005	104,9945	2,2326	0,2491	0,0476	0,4927	0,0149	149,9225	11,3978	16,8036	0,1664	0,0058	2,9765	0,0294	0,1233	7,5125	0,5113
Conclusion(Висновок)	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS

5. DATA SUMMARY

Method	ISO 712:2009	ISO 20483:2013	ISO 2171:2007/ДСТУ ISO 2171:2009	ISO 3093:2009/ДСТУ ISO 3093:2009	ISO 7305:1998	ISO 21415-1:2006/ДСТУ ISO 21415-1:2009	ISO 21415-2:2015/ДСТУ ISO 21415-2:2009	ISO 21415-3:2006/ДСТУ ISO 21415-4:2006	ISO 27971:2015	ISO 27971:2015	ISO 27971:2015	ISO 27971:2015	ISO 5529:2007	ГОСТ 9404-88	ГОСТ 10846-91	ГОСТ 27494-87	ГОСТ 27676-88	ГОСТ 27493-87	ГОСТ 27839-88	ГОСТ 27839-88	ДСТУ 4870:2007			
Moisture, %																								
Protein content, % (Expressed on dry matter, factor for converting nitrogen content to protein content - 5.7)	Ash content, %	Falling number, s	Fat acidity, mg KOH/100 g	Wet gluten content, %	Wet gluten content, %	Gluten index	Dry gluten content, %	Deformation energy, W	Maximum pressure parameter, P	Mean abscissa at rupture, L	Index of swelling, G	Curve configuration ratio, P/L	Sedimentation index – Zeleni test, ml	Moisture content	Protein content, % (Expressed on dry matter, factor for converting nitrogen content to protein content - 5.7)	Ash content, %	Falling number, s	Acidity, ° of acidity	Wet gluten content, %	Index of gluten deformation	Whiteness of flour, c.u.			
No of Results	5	5	5	5	4	5	7	5	4	3	3	3	3	9	5	7	9	2	9	9	6			
No of Results z >3	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	0			
No of Results z >3, %	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	11,111	0,000				
Mean	14,124	11,570	0,520	448,000	35,440	29,000	25,286	96,640	8,140	284,333	94,333	85,667	20,533	1,107	43,333	14,056	11,548	0,529	451,778	2,775	25,022	54,778	58,383	
Min	14,040	11,410	0,500	435,000	32,860	27,800	25,000	94,000	7,800	233,000	86,000	81,000	19,900	1,030	42,000	13,570	11,320	0,490	431,000	2,400	23,800	50,000	56,000	
Max	14,180	11,780	0,540	461,000	38,000	30,000	25,600	98,000	8,600	323,000	99,000	95,000	21,700	1,220	45,000	14,400	11,680	0,610	462,000	3,150	26,700	75,000	60,000	
SD	0,055	0,137	0,016	11,958	2,451	0,982	0,204	1,532	0,365	46,318	7,234	8,083	1,012	0,100	1,528	0,267	0,142	0,044	11,009	0,530	0,817	8,318	1,662	
Median	14,140	11,560	0,520	451,000	35,450	29,500	25,300	97,000	8,080	297,000	98,000	81,000	20,000	1,070	43,000	14,030	11,570	0,500	458,000	2,775	25,000	50,000	58,650	
Robust mean(assigned value)	14,125	11,567	0,520	448,000	35,440	29,000	25,283	97,015	8,140	284,333	94,333	85,667	20,533	1,107	43,333	14,072	11,561	0,524	452,611	2,775	24,957	52,585	58,383	
Robust SD	0,055	0,133	0,016	11,958	2,451	0,982	0,199	1,011	0,365	46,318	7,234	8,083	1,012	0,100	1,528	0,236	0,124	0,035	9,422	0,530	0,554	4,071	1,662	
SD from method(Tr.SD)	0,160	0,176	0,022	30,480	7,000	2,010	0,900	2,000	0,860	23,080	5,722	6,796	0,800	0,163	2,500	N/A	N/A	N/A	N/A	N/A	2,000	5,000	2,000	
SD from Horwitz eq.	0,379	0,320	0,023	N/A	N/A	0,699	0,622	N/A	0,237	N/A	N/A	N/A	N/A	N/A	0,378	0,320	0,023	N/A	N/A	0,615	N/A	N/A		
Target SD	0,055	0,133	0,023	11,958	2,451	0,699	0,622	1,532	0,237	23,080	5,722	6,796	0,800	0,163	2,500	0,378	0,320	0,032	11,009	0,379	1,113	5,000	1,662	
Source of target SD of PT	Trial SD	Trial SD	Horwitz	Trial SD	Trial SD	Horwitz	Horwitz	Trial SD	Horwitz	Method Tr SD	Method Tr SD	Method Tr SD	Method Tr SD	Method Tr SD	Method Tr SD	Method Tr SD	Method Tr SD	Horwitz	Horwitz	Trial SD	Trial SD	Trial SD	Method Tr SD	Trial SD

6. RAW DATA

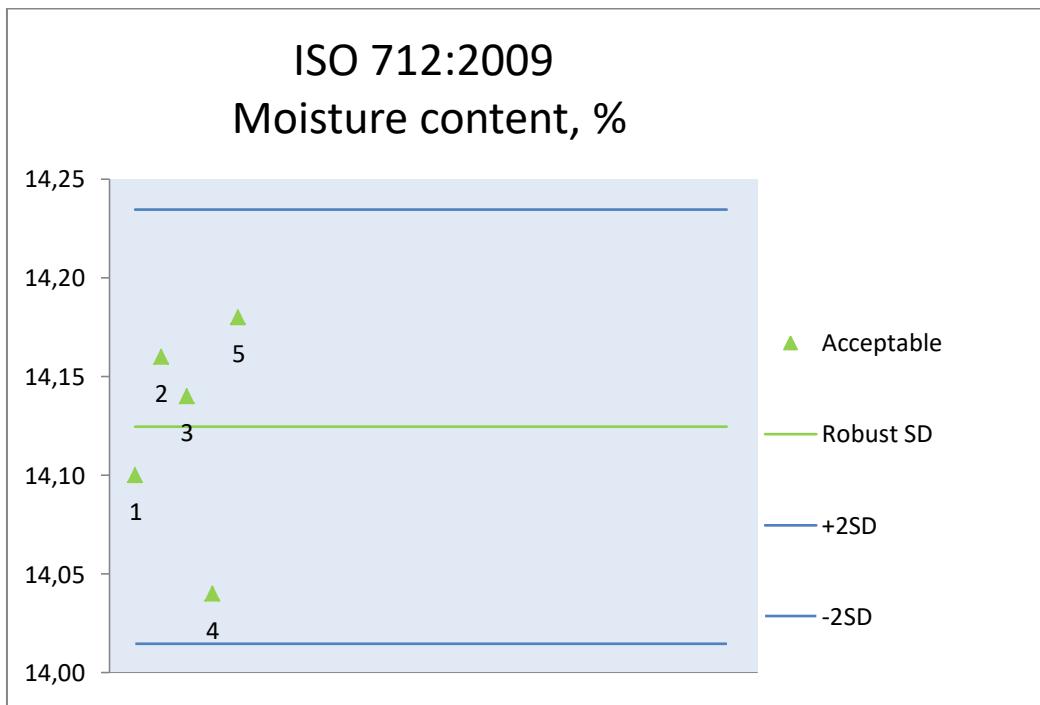
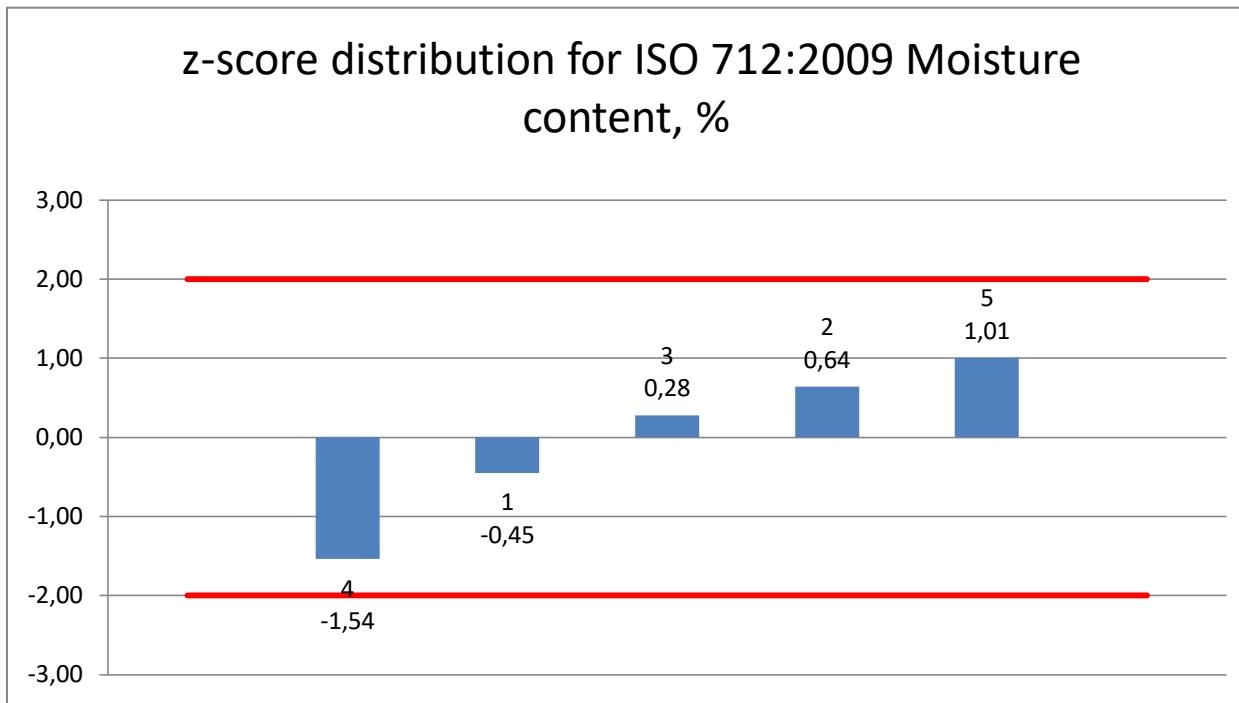
Method	ISO 712:2009	ISO 20483:2013	ISO 2171:2007/ДСТУ ISO 2171:2009	ISO 3093:2009/ДСТУ ISO 3093:2009	ISO 7305:1998	ISO 21415-1:2006/ДСТУ ISO 21415-1:2009	ISO 21415-2:2015/ДСТУ ISO 21415-2:2009	ISO 21415-3:2006/ISO 21415-4:2006	ISO 27971:2015	ISO 27971:2015	ISO 27971:2015	ISO 27971:2015	ISO 27971:2015	ISO 27971:2007	ГОСТ 9404-88	ГОСТ 10846-91	ГОСТ 27494-87	ГОСТ 27676-88	ГОСТ 27493-87	ГОСТ 27839-88	ГОСТ 27839-88	ДСТУ 4870:2007		
Laboratory number	Moisture, %	Protein content, % (Expressed on dry matter, factor for converting nitrogen content to protein content - 5.7)	Ash content, %	Falling number, s	Fat acidity, mg KOH/100 g	Wet gluten content, %	Gluten index	Dry gluten content, %	Deformation energy, W	Maximum pressure parameter, P	Mean abscissa at rupture, L	Index of swelling, G	Curve configuration ratio, P/L	Sedimentation index – Zeleni test, ml	Moisture content, %	Protein content, % (Expressed on dry matter, factor for converting nitrogen content to protein content - 5.7)	Ash content, %	Falling number, s	Acidity, % of acidity	Wet gluten content, %	Index of gluten deformation	Whiteness of flour, c.u.		
1	14,10	11,56	0,51	436,00	33,90	27,80	25,00	97,00	8,26	323,00	98,00	95,00	21,70	1,03	45,00	14,03	11,52	0,50	431,00	2,40	25,70	50,00	59,30	
2	14,16	11,60	0,50	451,00	38,00	30,00	25,30	97,00	7,90	297,00	99,00	81,00	20,00	1,22	42,00	13,90	11,65	0,56	458,00		24,40	53,00	60,00	
3	14,14	11,50	0,52	457,00	37,00	29,50	25,10	97,20	7,80								13,88	11,57	0,54	460,00		24,60	55,00	
4	14,04	11,41	0,54	461,00	32,86	28,10	25,60	94,00	8,60	233,00	86,00	81,00	19,90	1,07	43,00	13,57	11,32	0,61	462,00	3,15	26,70	75,00		
5	14,18	11,78	0,53	435,00		29,60	25,40	98,00									13,92	11,68		440,00		23,80	60,00	
6																	14,40		0,50	458,00		25,00	50,00	57,00
7								25,40									14,30		0,49	454,00		25,00	50,00	60,00
8								25,20									14,20		0,50	443,00		25,00	50,00	56,00
9																	14,30			460,00		25,00	50,00	58,00

7. Z SCORES

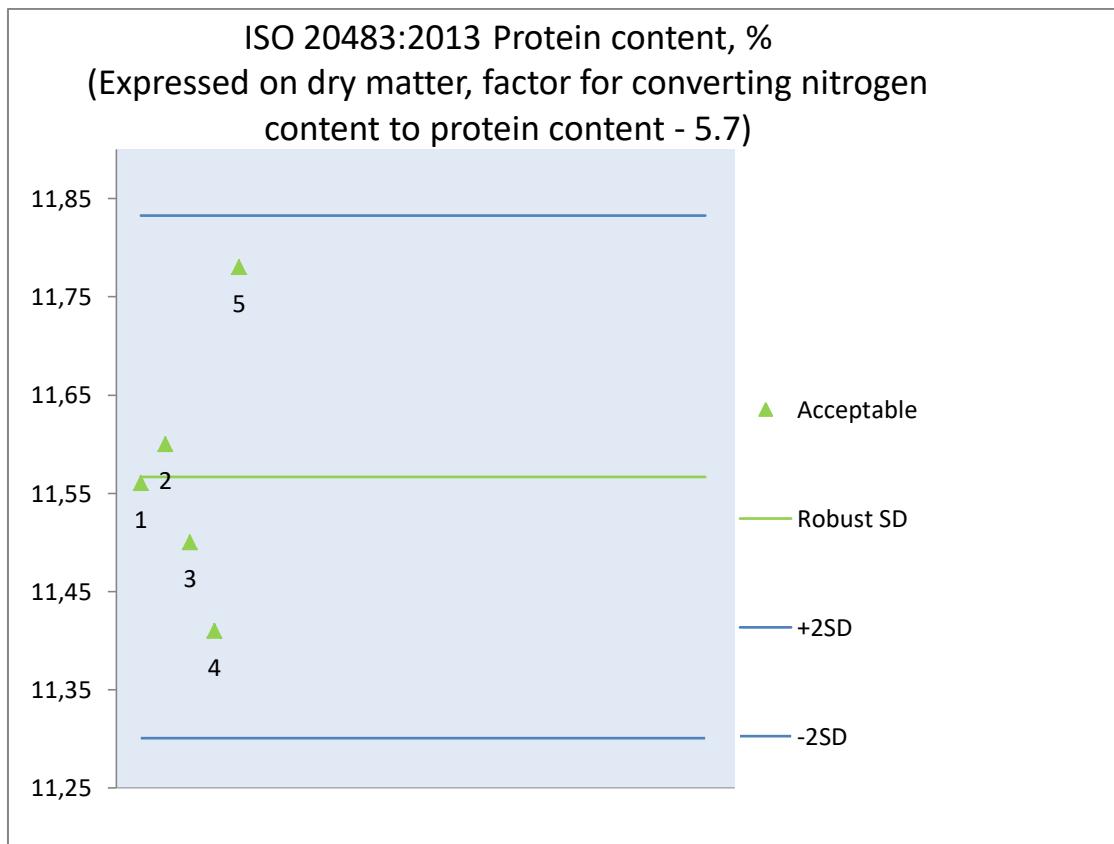
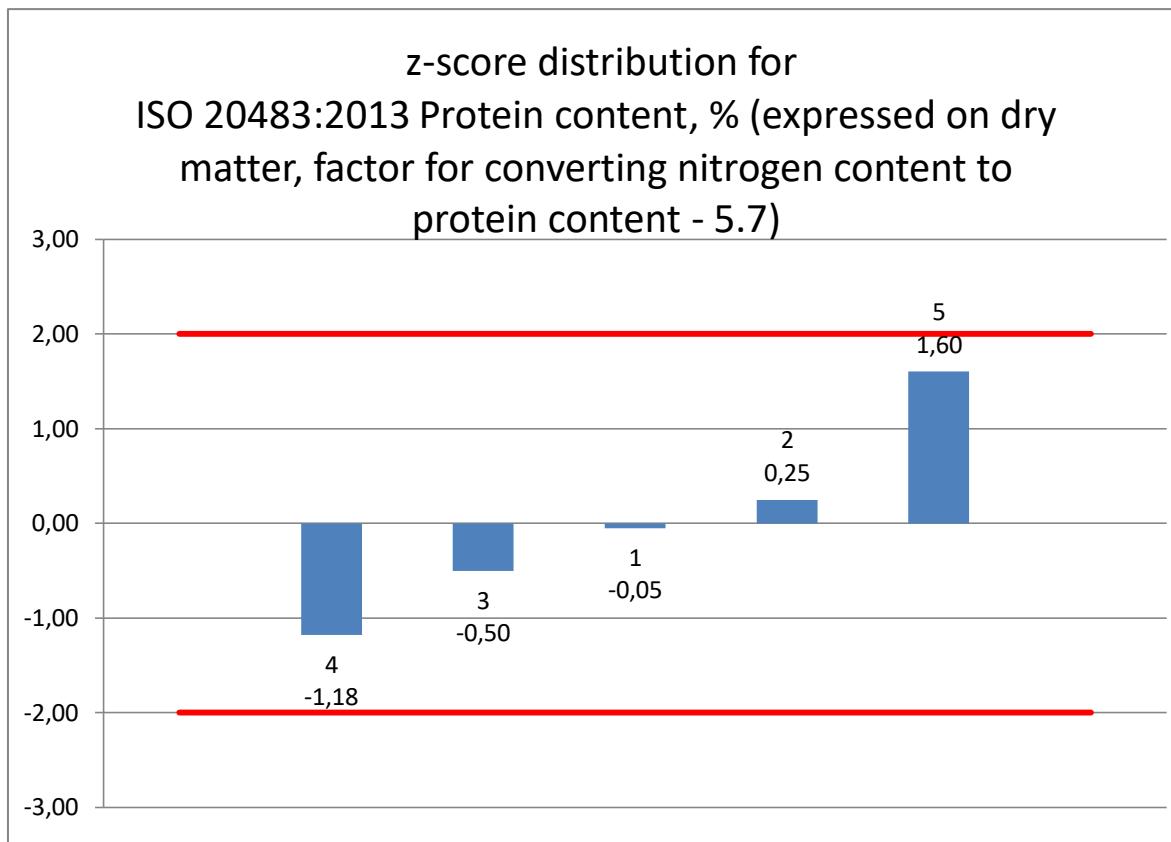
Method	ISO 712:2009	ISO 20483:2013	ISO 2171:2007/ДСТУ ISO 2171:2009	ISO 3093:2009/ДСТУ ISO 3093:2009	ISO 7305:1998	ISO 21415-1:2006/ДСТУ ISO 21415-1:2009	ISO 21415-2:2015/ДСТУ ISO 21415-2:2009	ISO 21415-3:2006/ISO 21415-4:2006	ISO 27971:2015	ISO 27971:2015	ISO 27971:2015	ISO 27971:2015	ISO 27971:2015	ISO 5529:2007	ГОСТ 9404-88	ГОСТ 10846-91	ГОСТ 27494-87	ГОСТ 27676-88	ГОСТ 27493-87	ГОСТ 27839-88	ГОСТ 27839-88	ДСТУ 4870:2007		
Laboratory number	Moisture, %	Protein content, % (Expressed on dry matter, factor for converting nitrogen content to protein content - 5.7)	Ash content, %	Falling number, s	Fat acidity, mg KOH/100 g	Wet gluten content, %	Gluten index	Dry gluten content, %	Deformation energy, W	Maximum pressure parameter, P	Mean abscissa at rupture, L	Index of swelling, G	Curve configuration ratio, P/L	Sedimentation index – Zeleni test, ml	Moisture content, %	Protein content, % (Expressed on dry matter, factor for converting nitrogen content to protein content - 5.7)	Ash content, %	Falling number, s	Acidity, ° of acidity	Wet gluten content, %	Index of gluten deformation	Whiteness of flour, c.u.		
1	-0,45	-0,05	-0,43	-1,00	-0,63	-1,72	-0,46	-0,01	0,51	1,68	0,64	1,37	1,46	-0,47	0,67	-0,11	-0,13	-0,74	-1,96	-0,99	0,67	-0,52	0,55	
2	0,64	0,25	-0,87	0,25	1,04	1,43	0,03	-0,01	0,01	0,55	0,82	-0,69	-0,67	0,69	-0,53	-0,46	0,28	1,14	0,49	-0,50	0,08	0,97		
3	0,28	-0,50	0,00	0,75	0,64	0,72	-0,29	0,12	-1,43							-0,51	0,03	0,51	0,67	-0,32	0,48			
4	-1,54	-1,18	0,87	1,09	-1,05	-1,29	0,51	-1,97	1,94	-2,22	-1,46	-0,69	-0,79	-0,22	-0,13	-1,33	-0,75	2,70	0,85	0,99	1,57	4,48		
5	1,01	1,60	0,43	-1,09		0,86	0,19	0,64							-0,40	0,37		-1,15		-1,04	1,48			
6																	0,87		-0,74	0,49		0,04	-0,52	-0,83
7									0,19								0,60		-1,05	0,13		0,04	-0,52	0,97
8									-0,13								0,34		-0,74	-0,87		0,04	-0,52	-1,43
9																	0,60		0,67		0,04	-0,52	-0,23	

8. Z SCORE PLOTS AND RESULTS CHARTS.

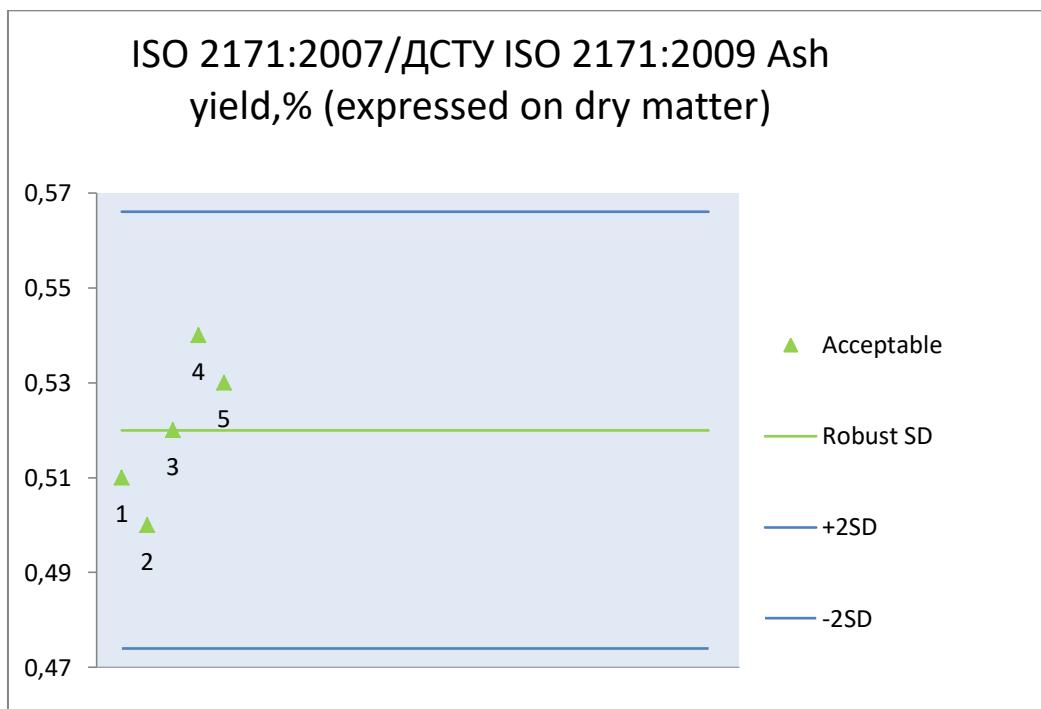
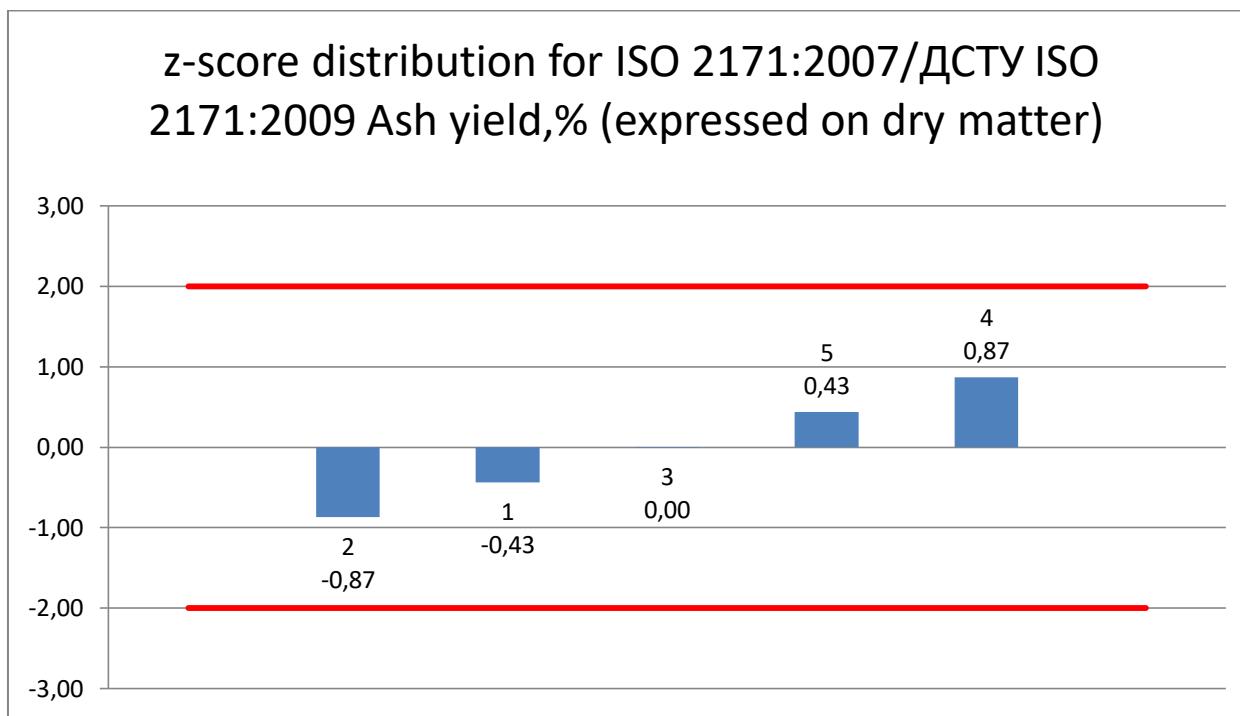
8.1. ISO 712:2009 Moisture content, %



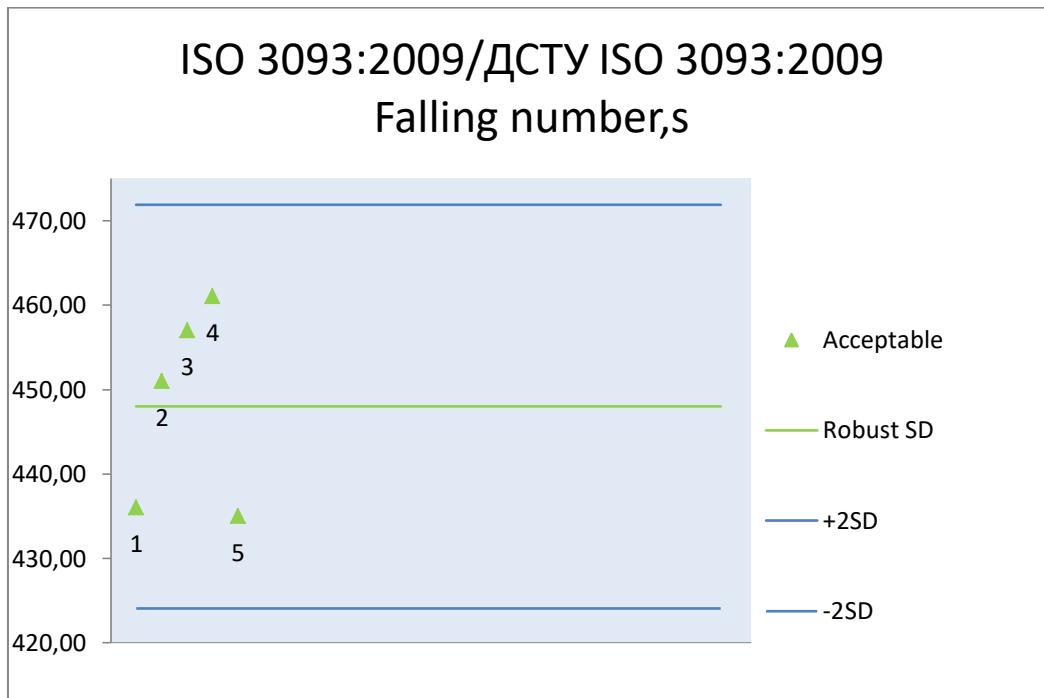
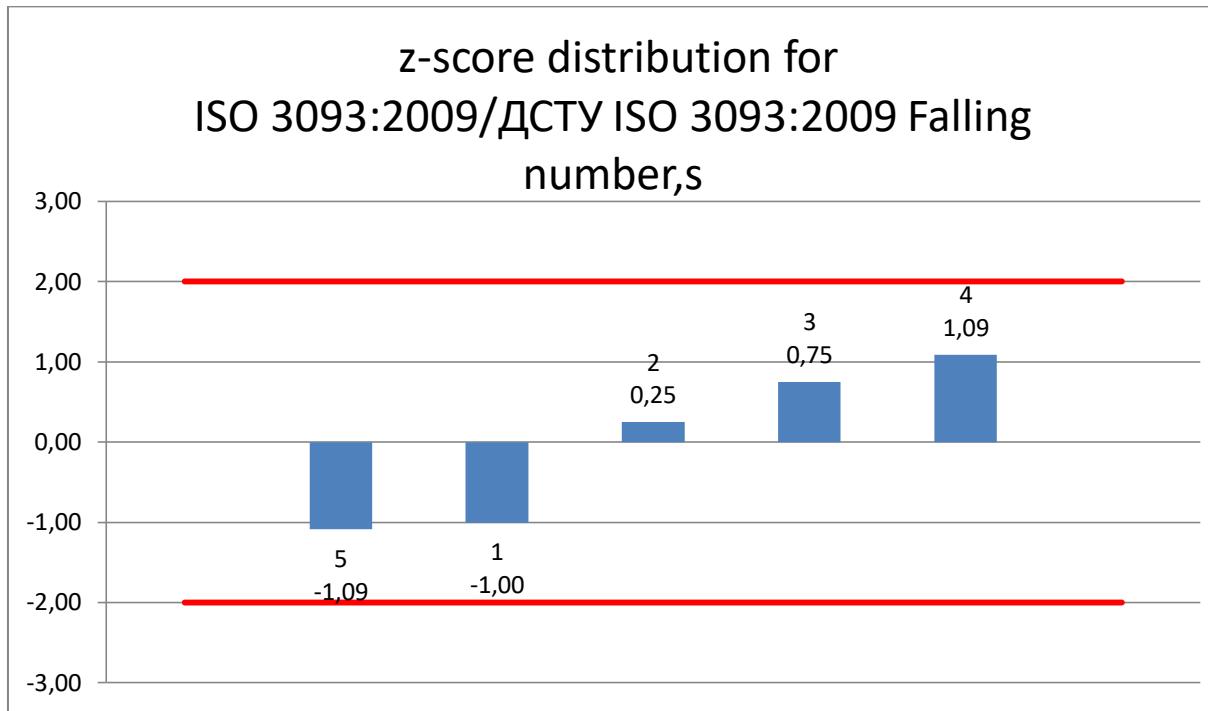
8.2. ISO 20483:2013 Protein content, % (expressed on dry matter, factor for converting nitrogen content to protein content - 5.7)



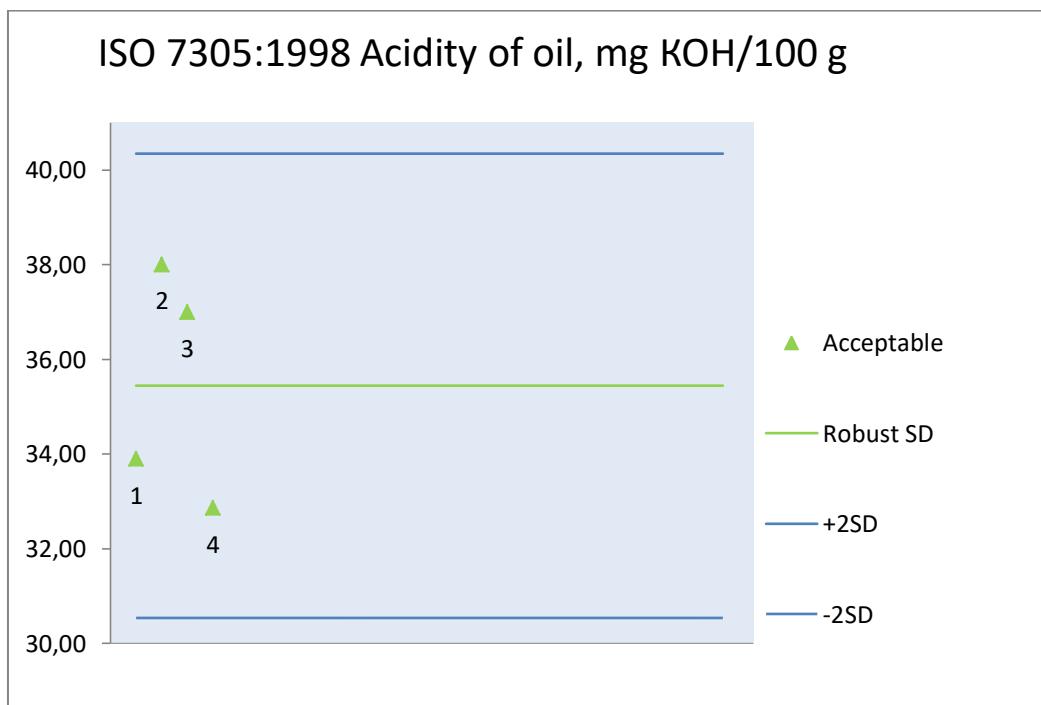
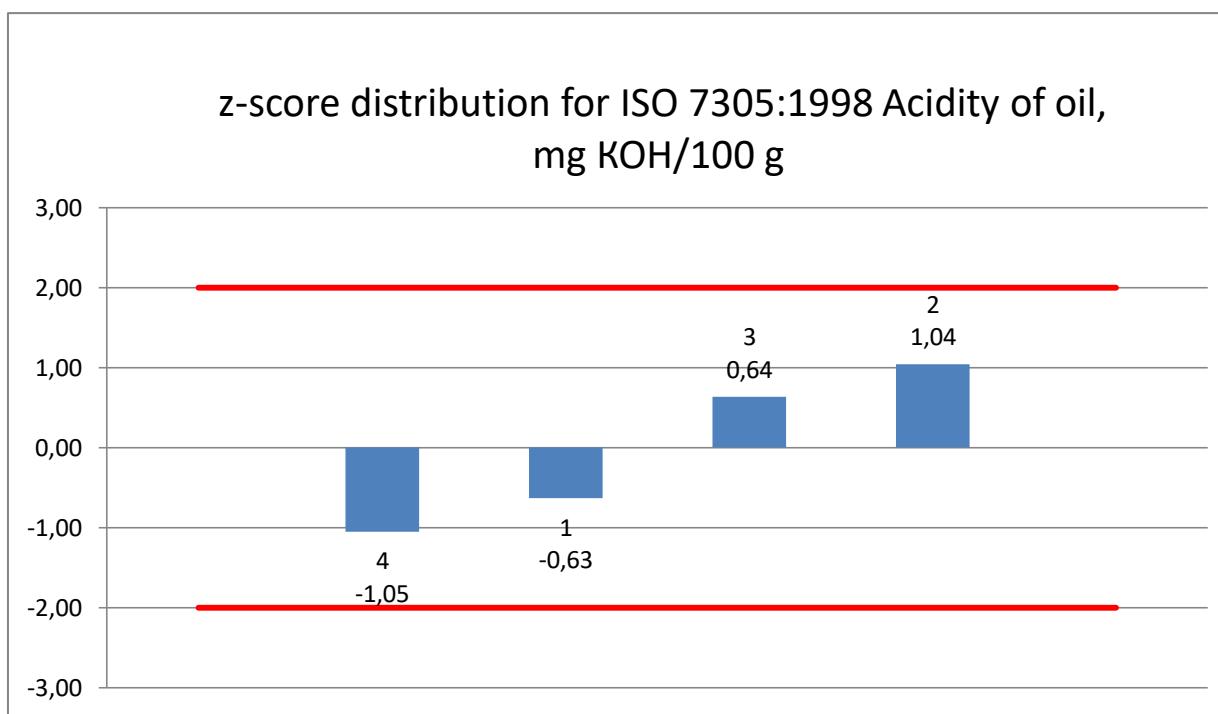
8.3. ISO 2171:2007/ДСТУ ISO 2171:2009 Ash yield, % (expressed on dry matter)



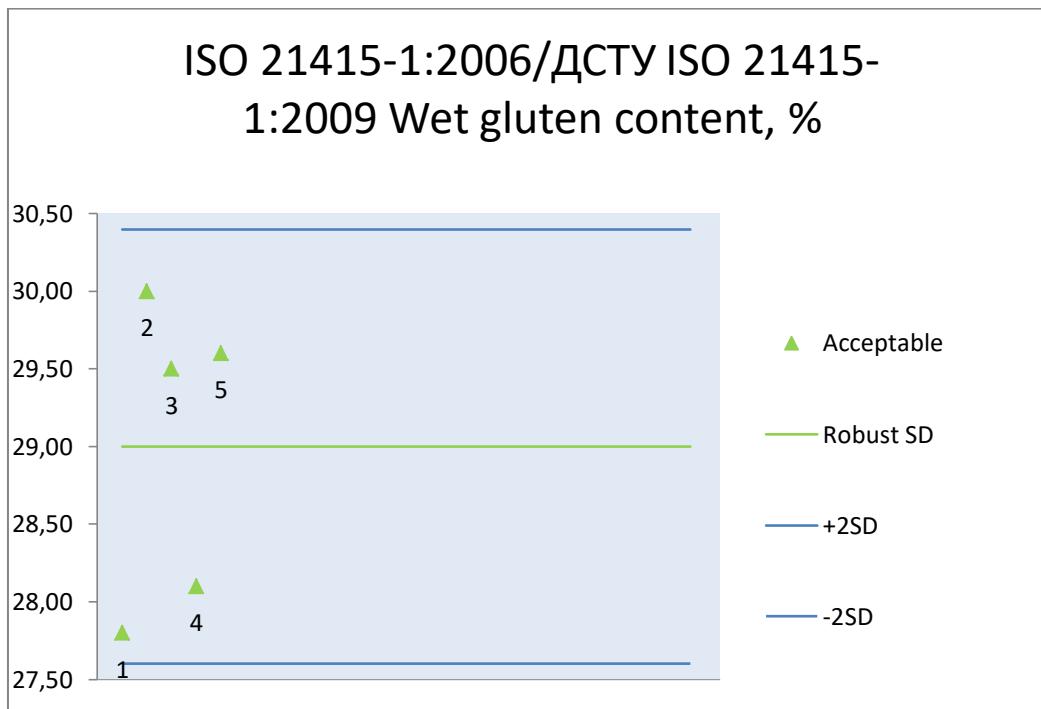
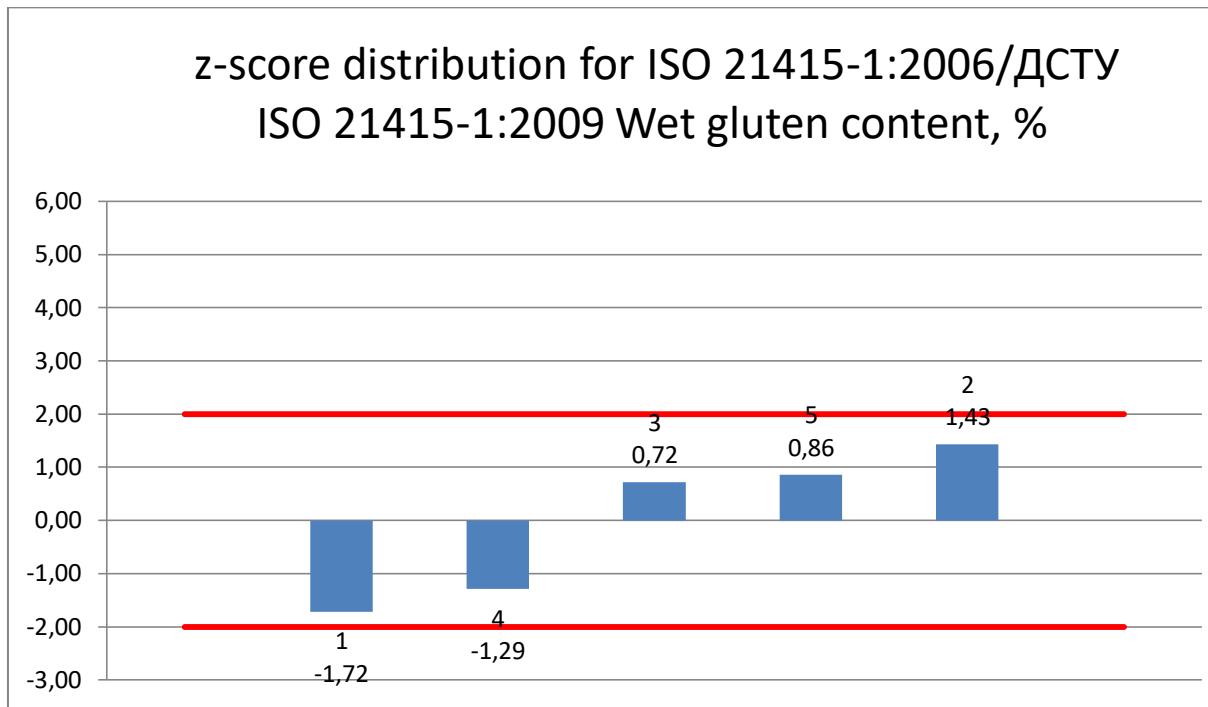
8.4. ISO 3093:2009/ДСТУ ISO 3093:2009 Falling number, s



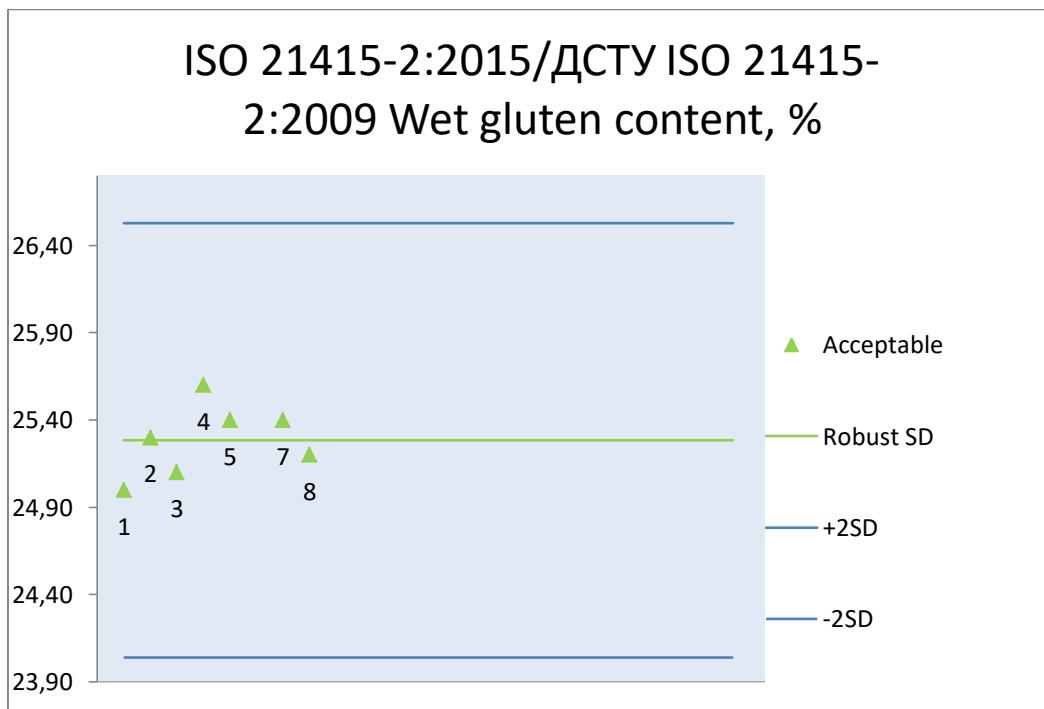
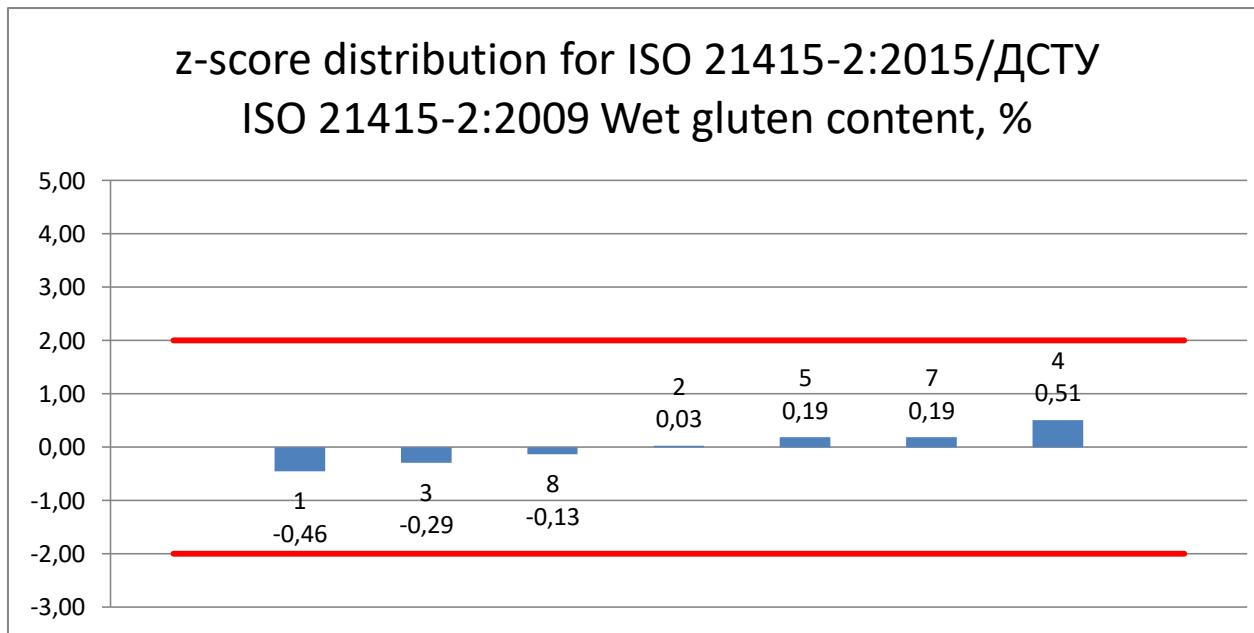
8.5. ISO 7305:1998 Acidity of oil, mg KOH/100 g



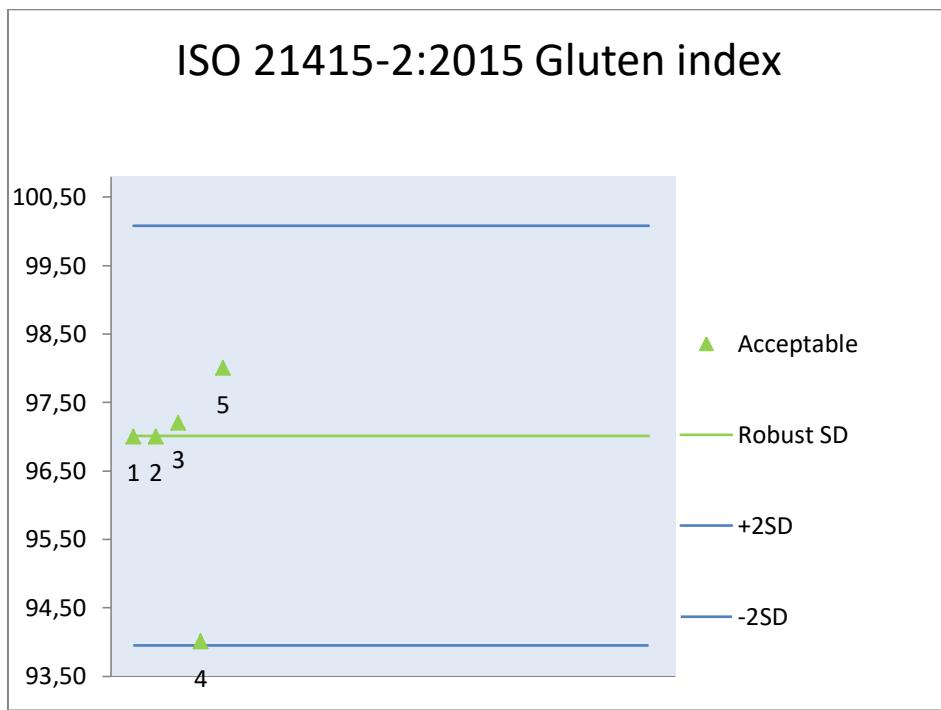
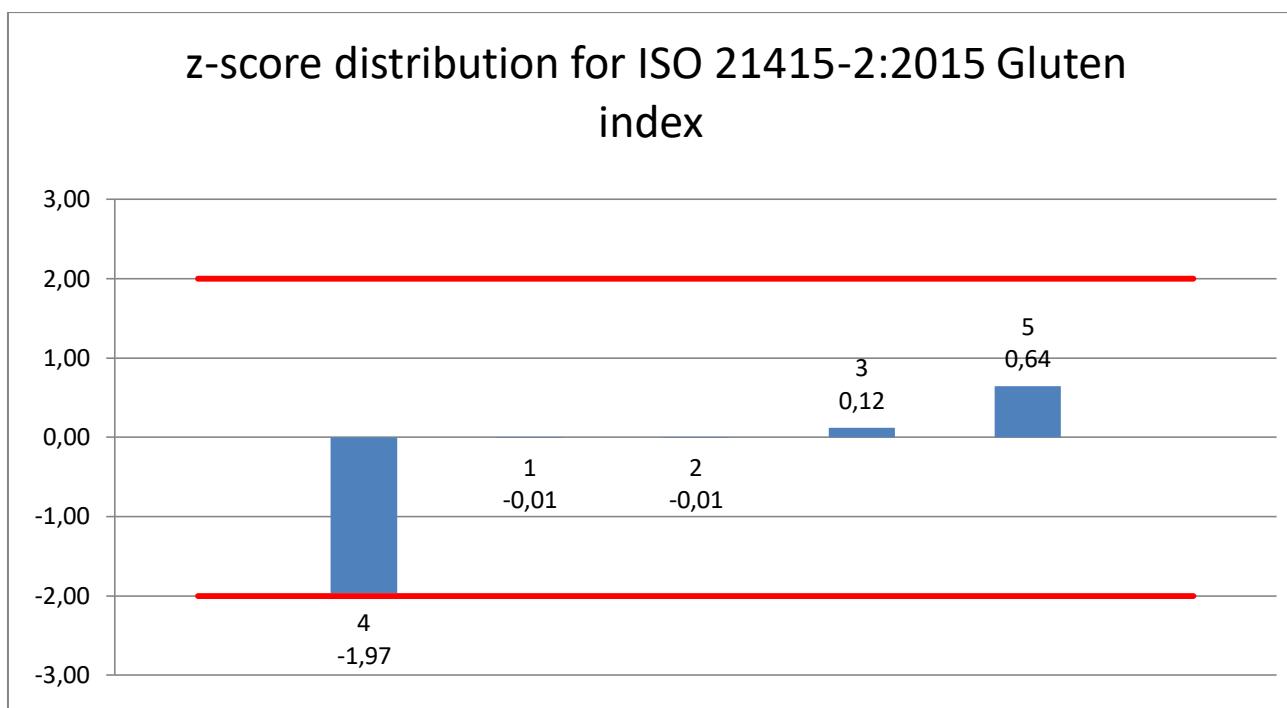
8.6. ISO 21415-1:2006/ДСТУ ISO 21415-1:2009 Wet gluten content, %



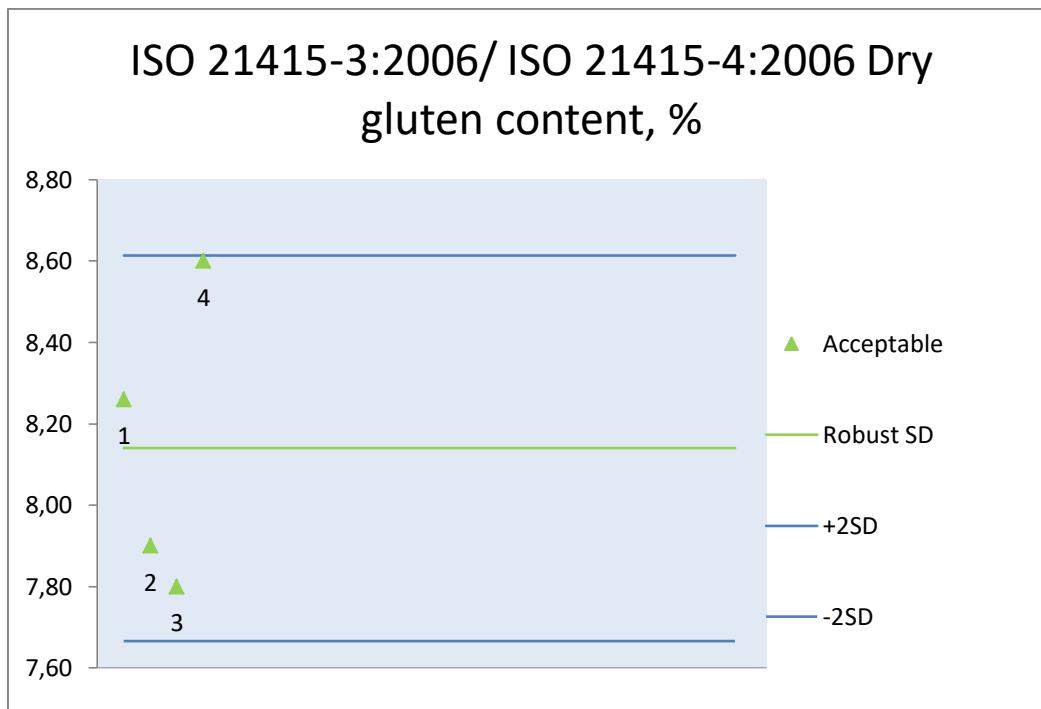
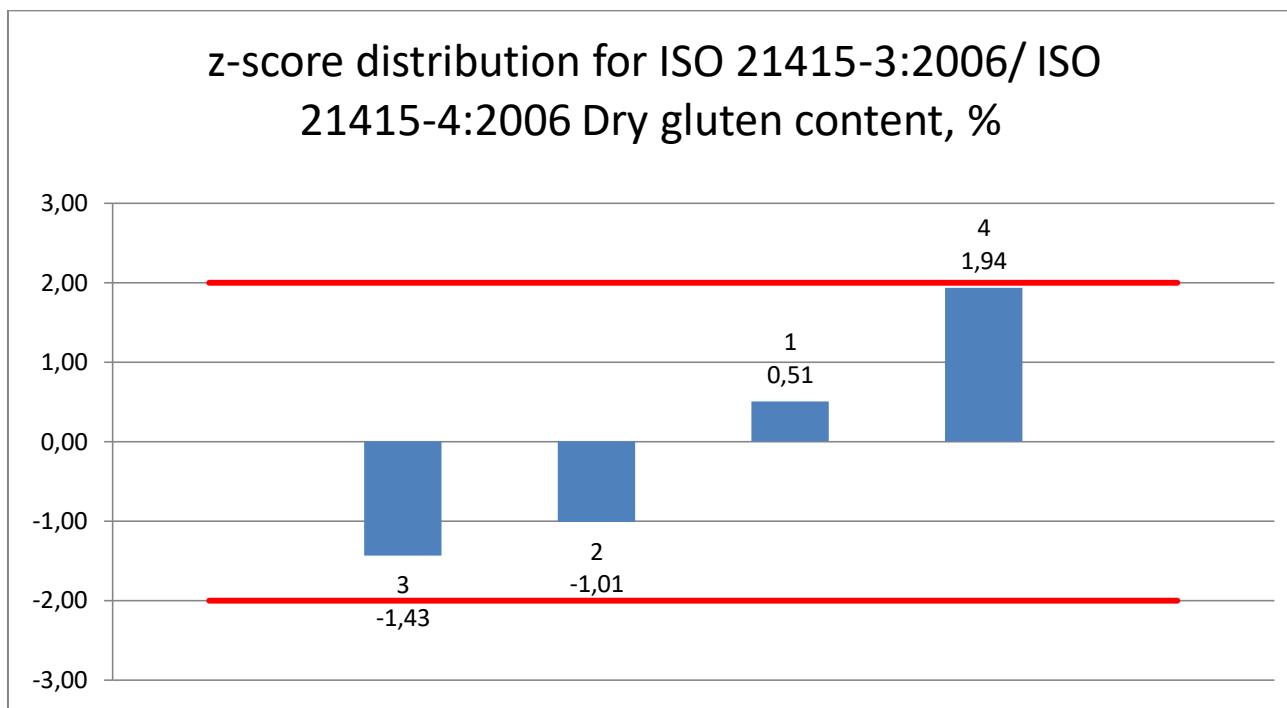
8.7. ISO 21415-2:2015/ДСТУ ISO 21415-2:2009 Wet gluten content, %



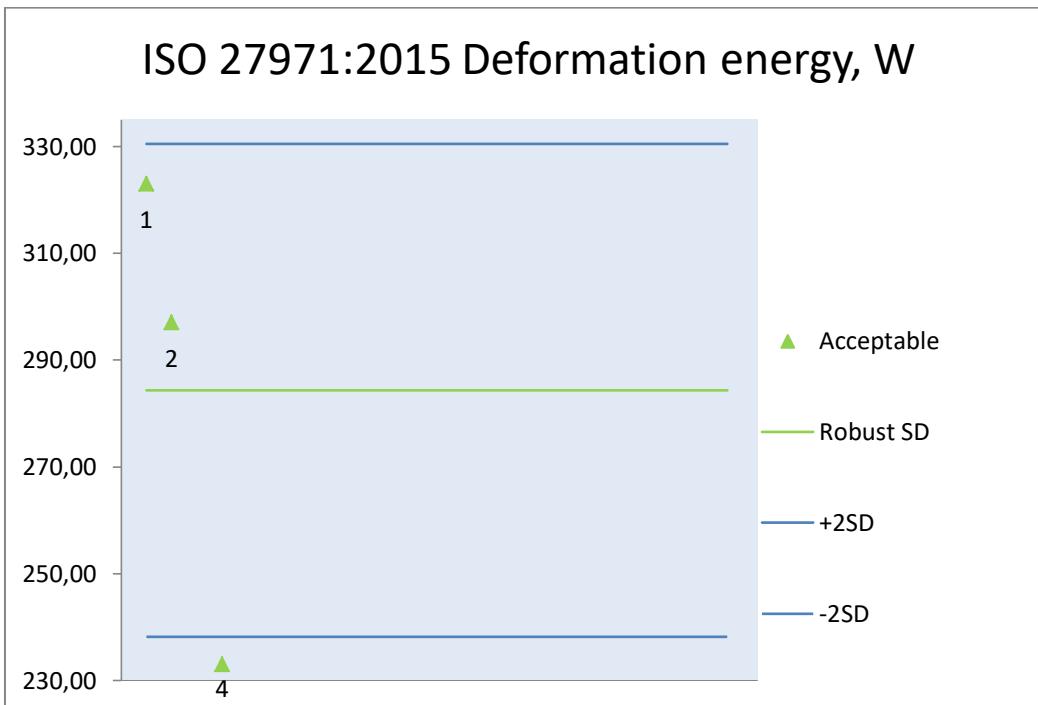
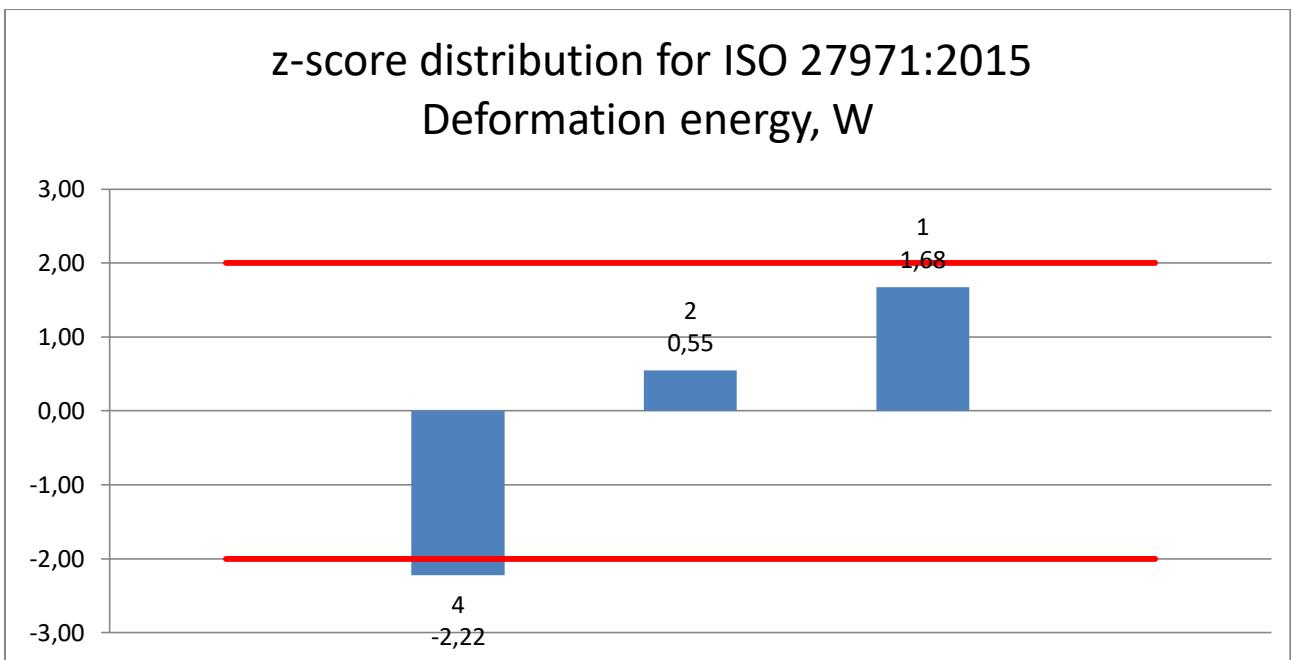
8.8. ISO 21415-2:2015 Gluten index



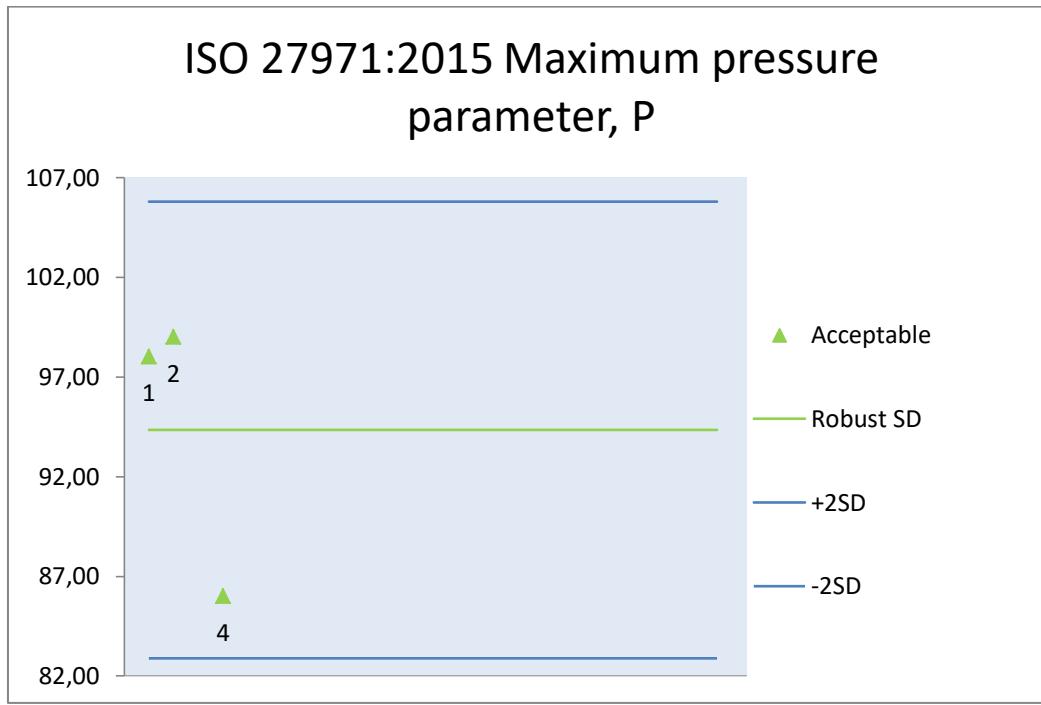
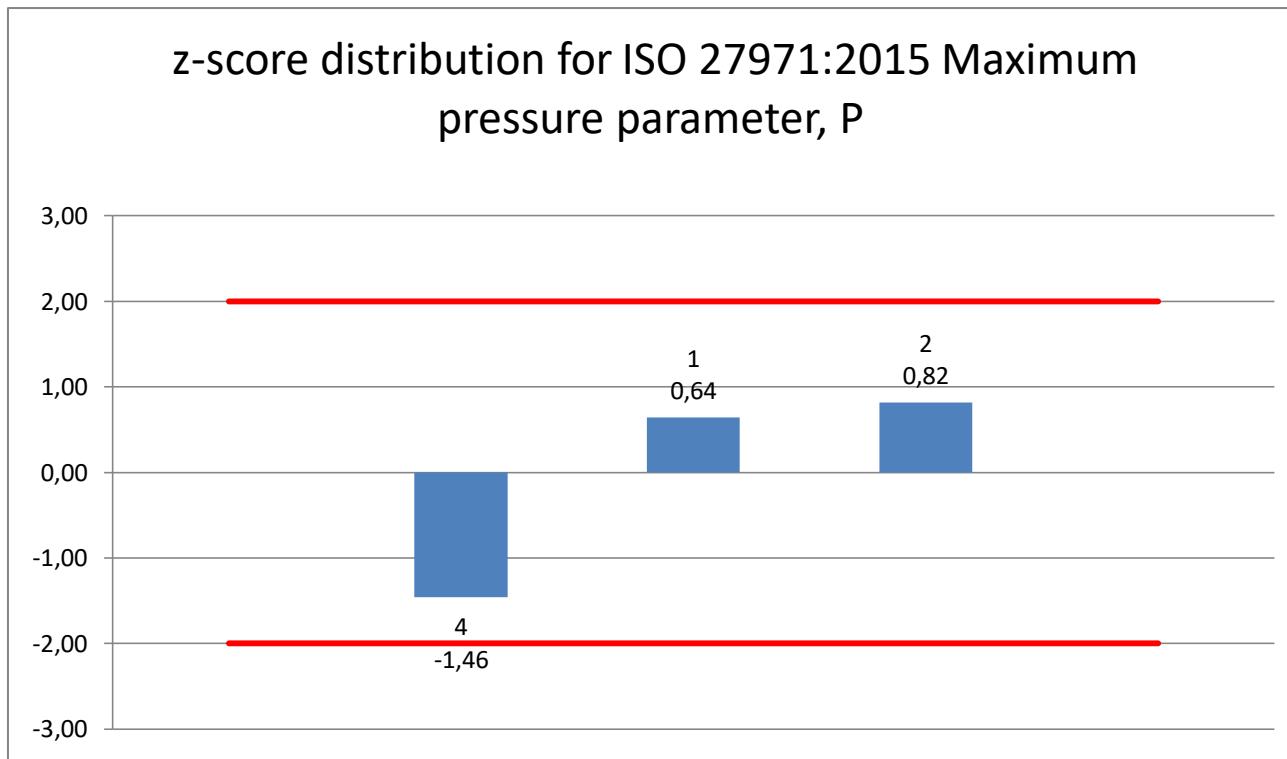
8.9. ISO 21415-3:2006/ ISO 21415-4:2006 Dry gluten content, %



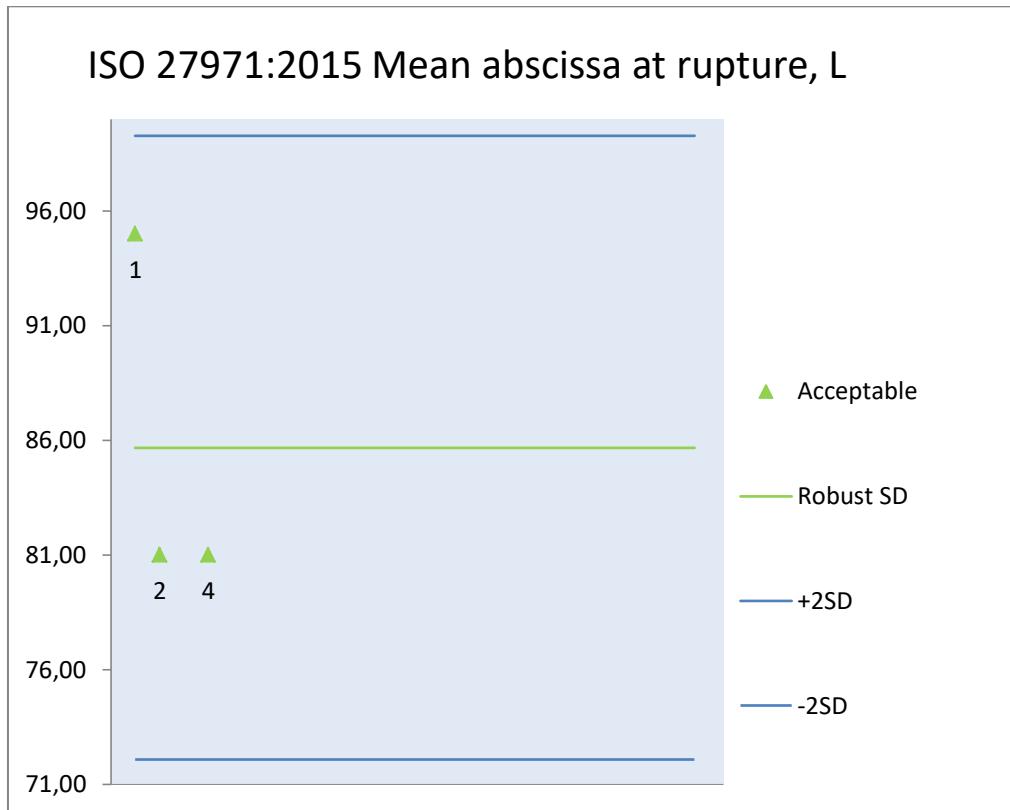
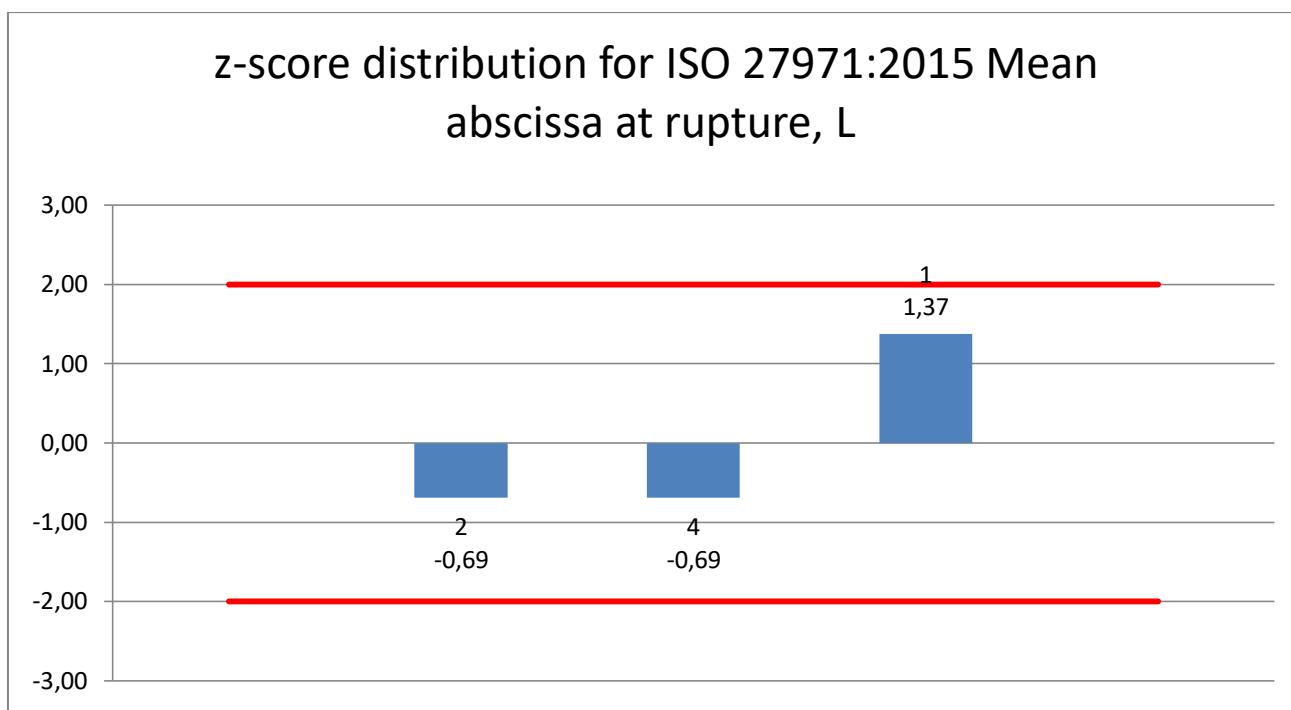
8.10. ISO 27971:2015 Deformation energy, W



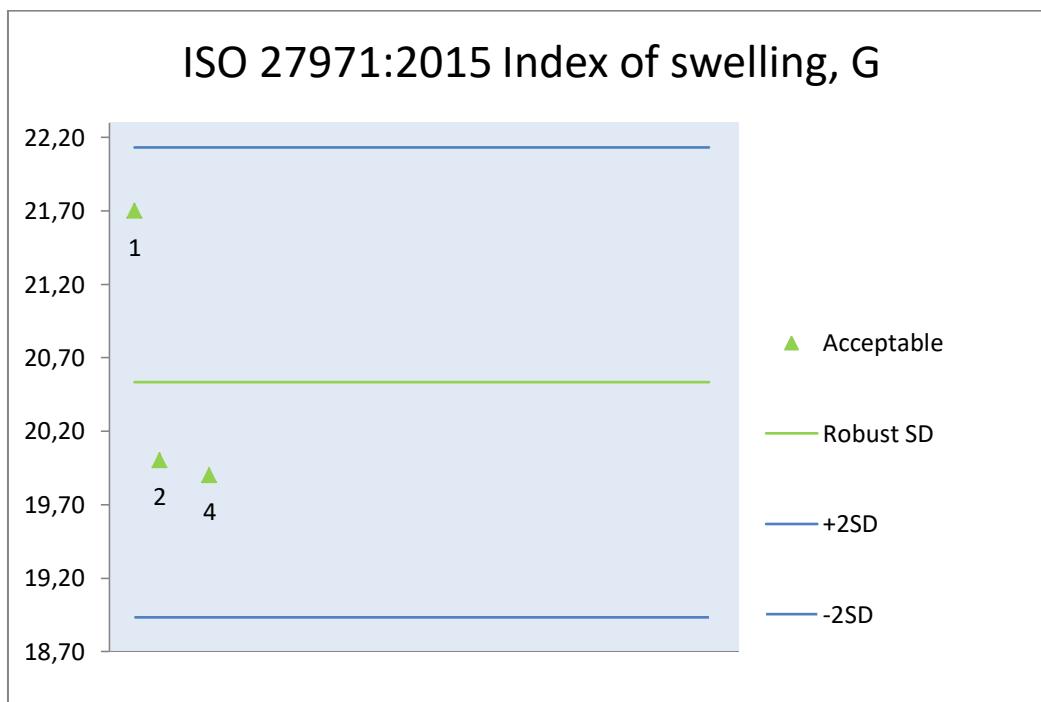
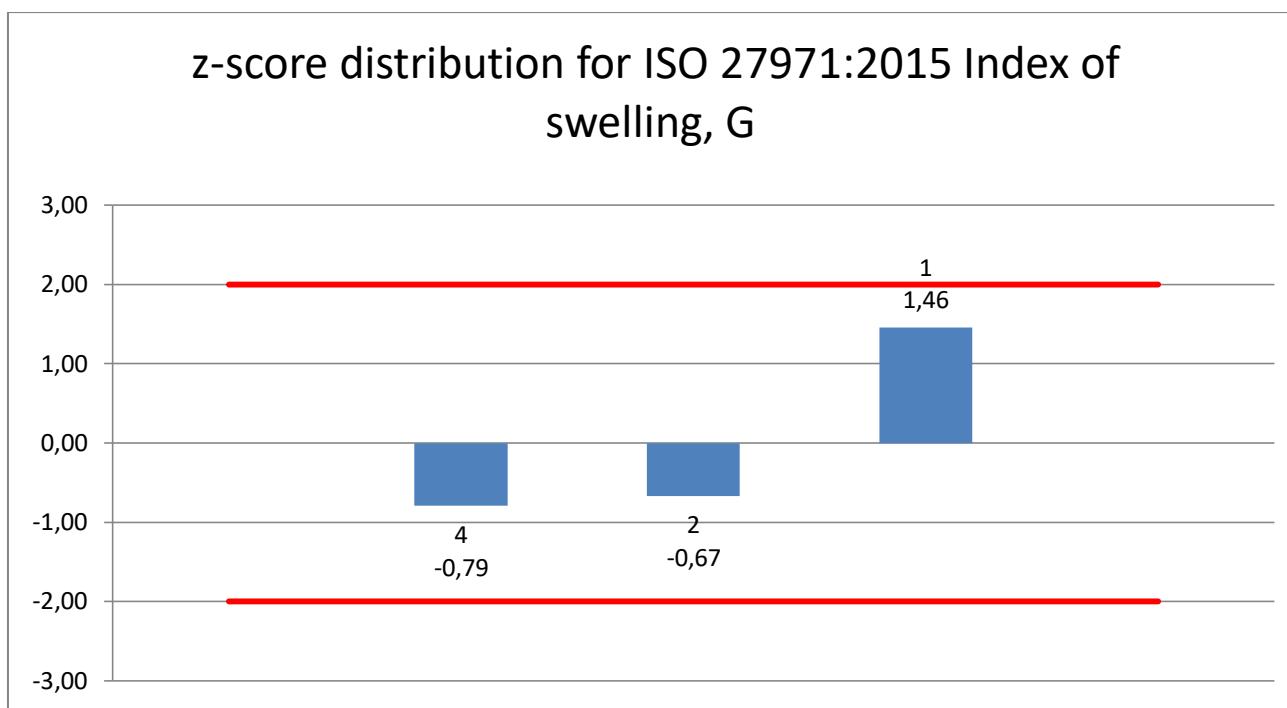
8.11. ISO 27971:2015 Maximum pressure parameter, P



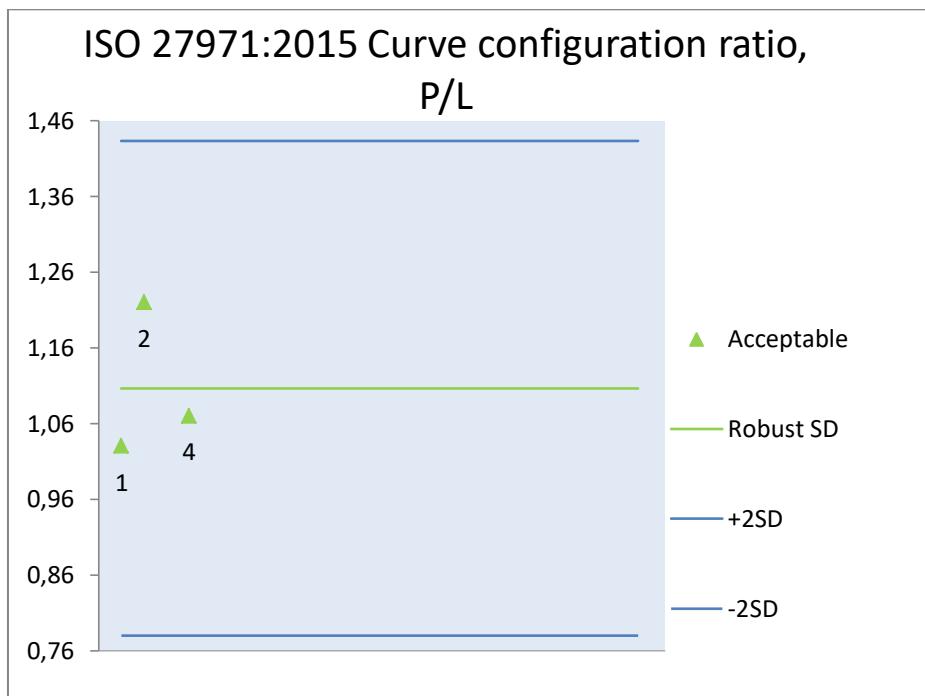
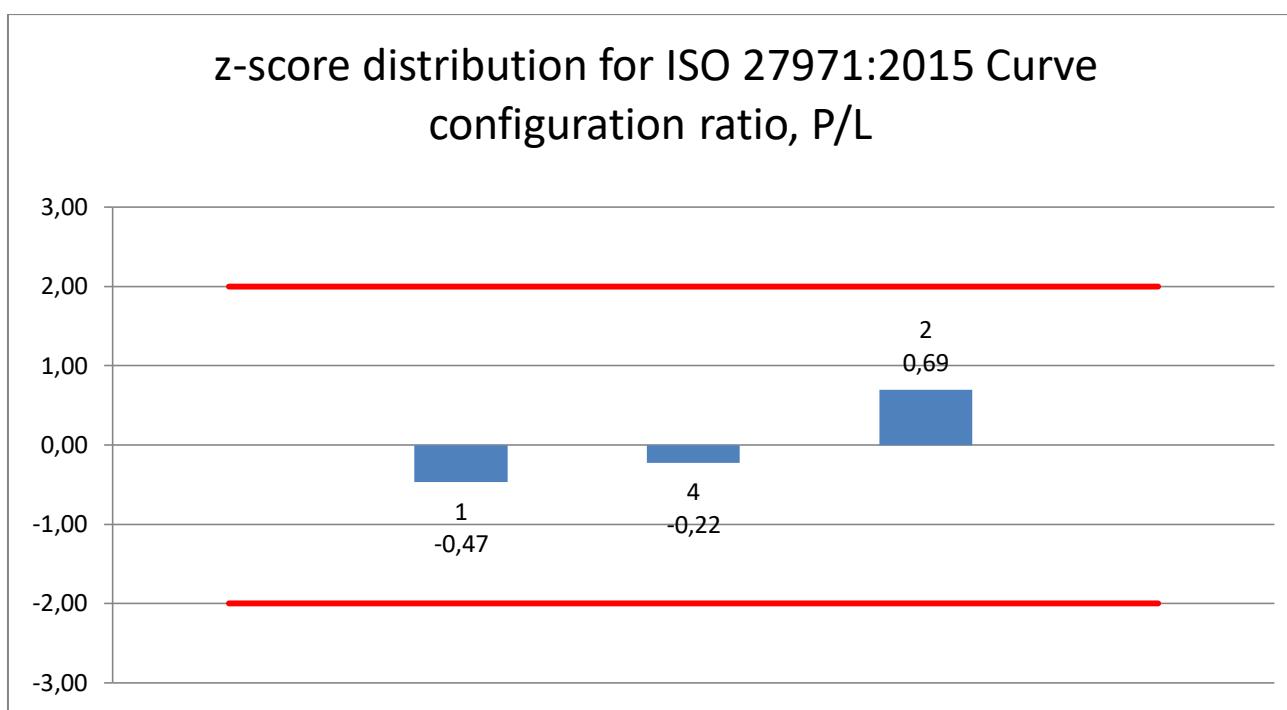
8.12. ISO 27971:2015 Mean abscissa at rupture, L



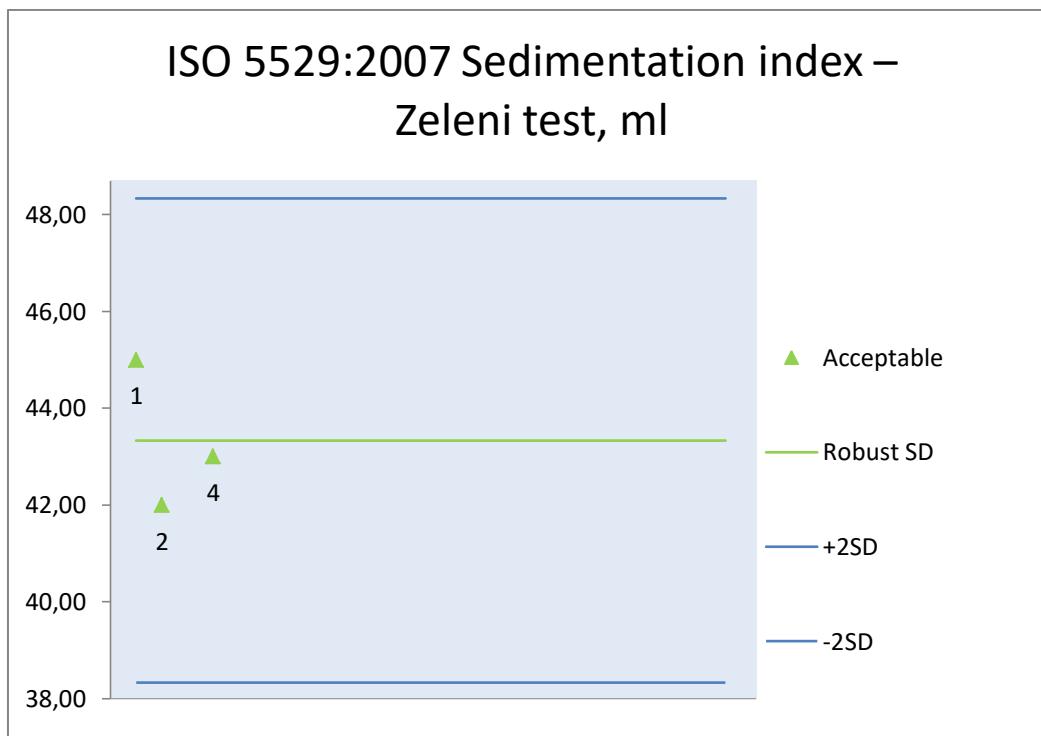
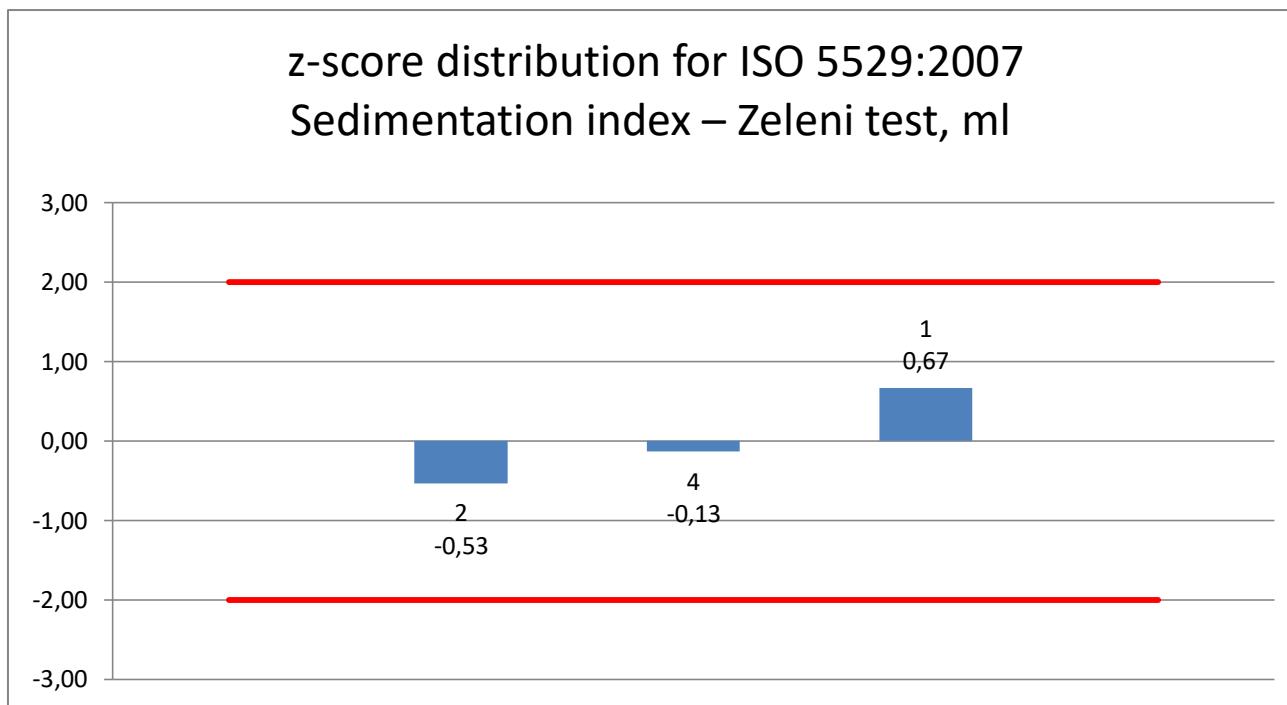
8.13. ISO 27971:2015 Index of swelling, G



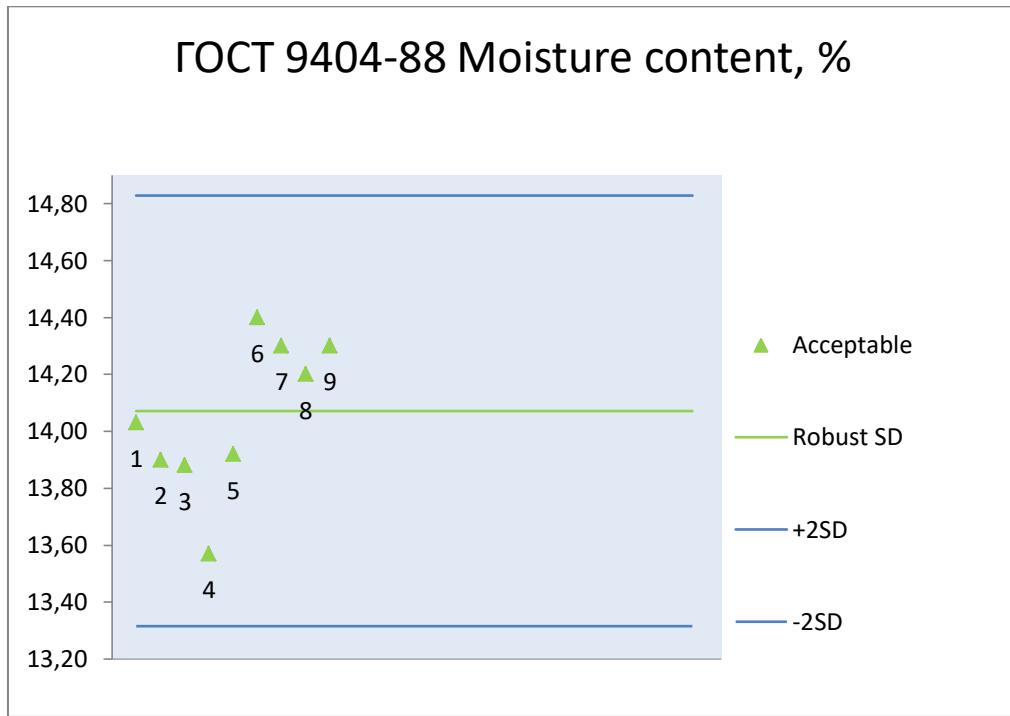
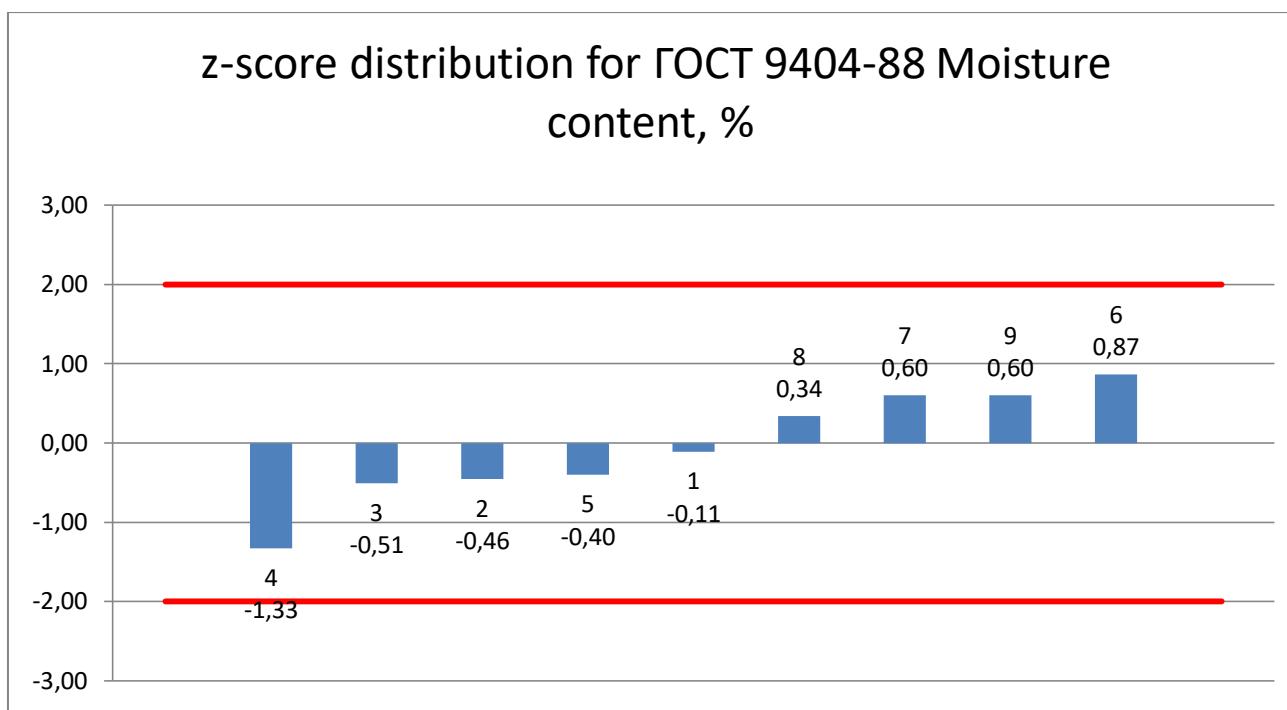
8.14. ISO 27971:2015 Curve configuration ratio, P/L



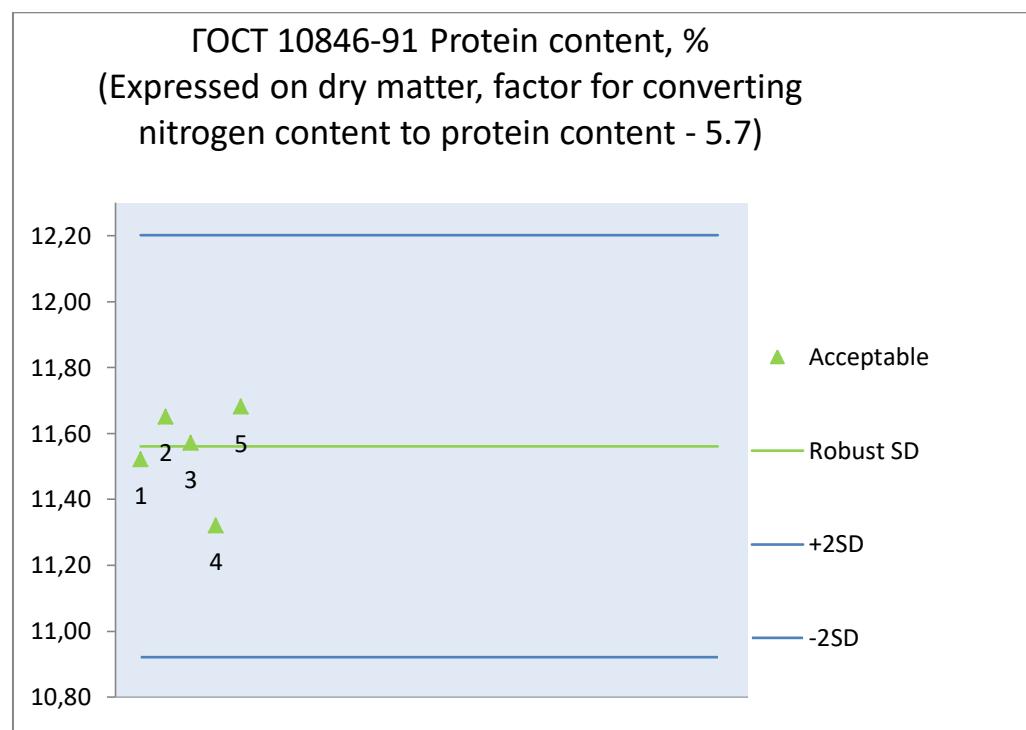
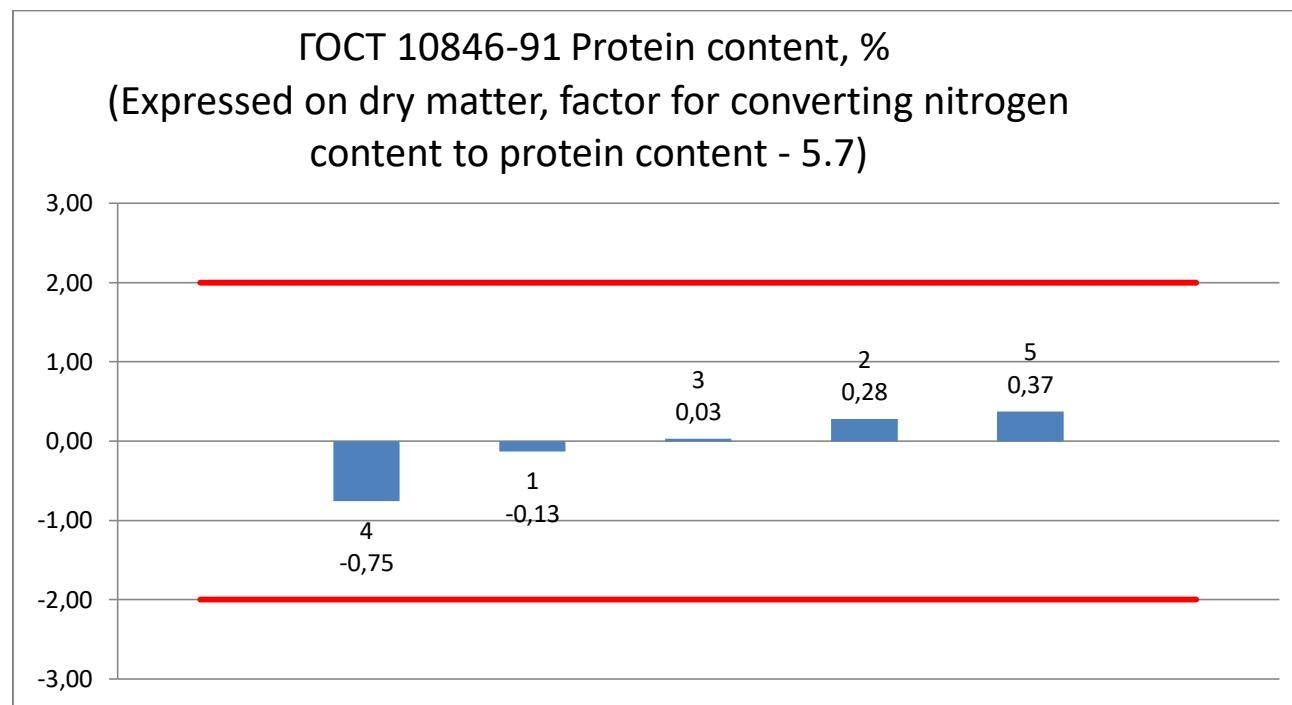
8.15. ISO 5529:2007 Sedimentation index – Zeleni test, ml



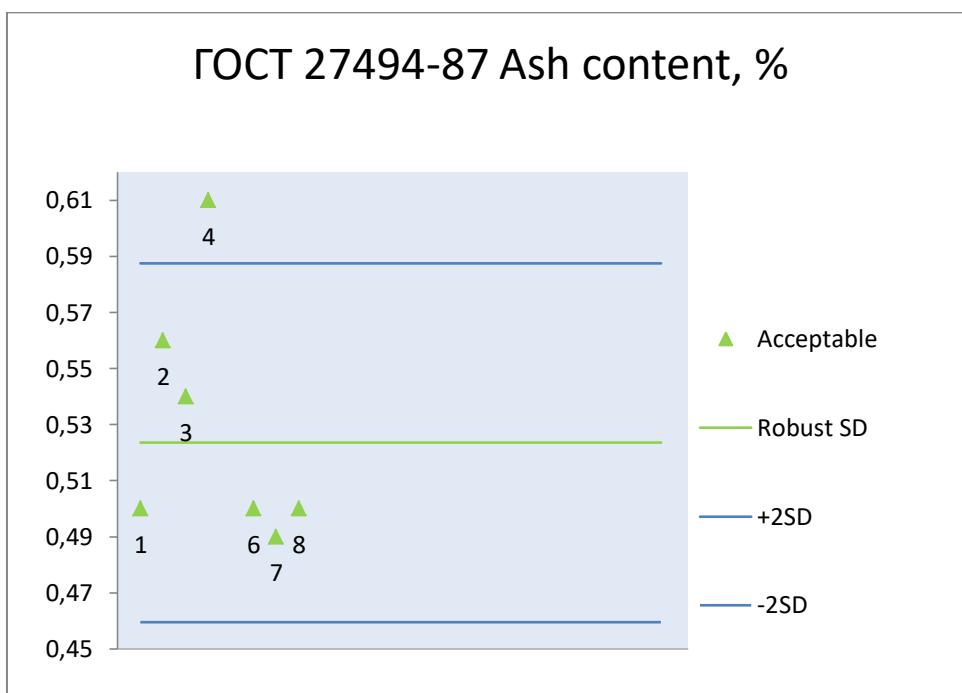
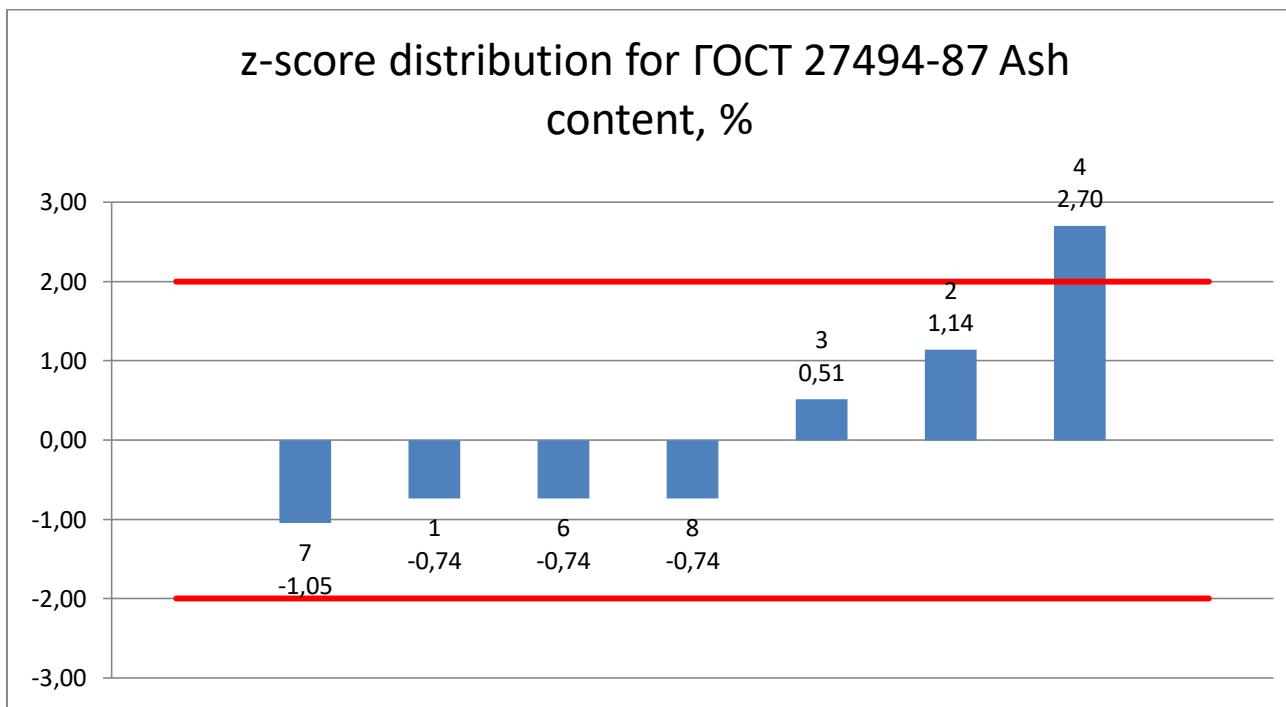
8.16. ГОСТ 9404-88 Moisture content, %



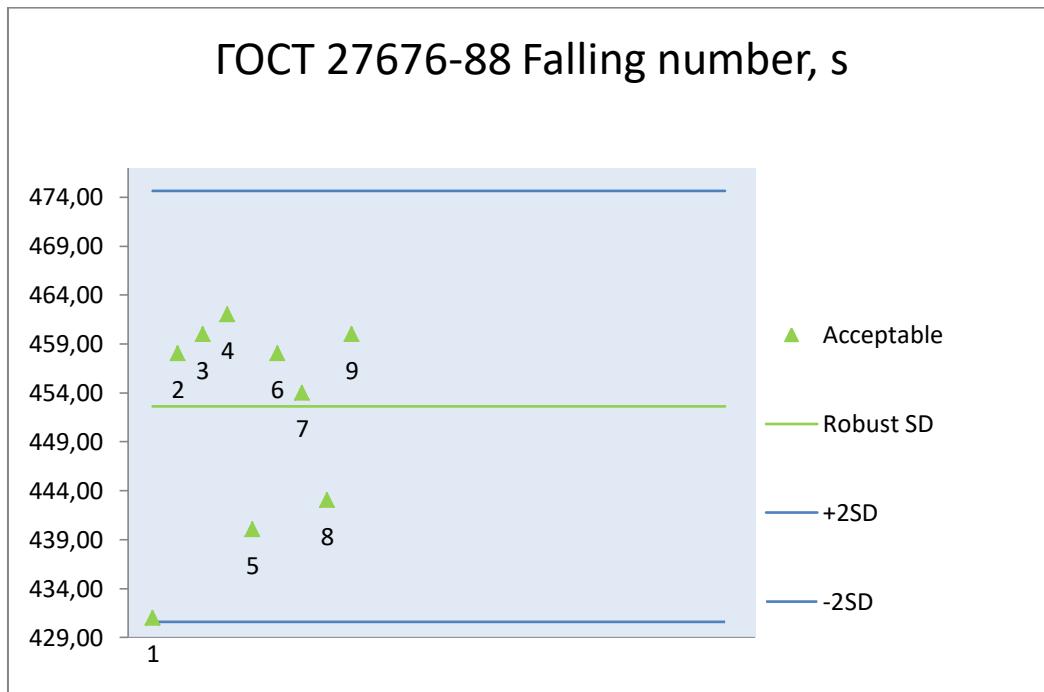
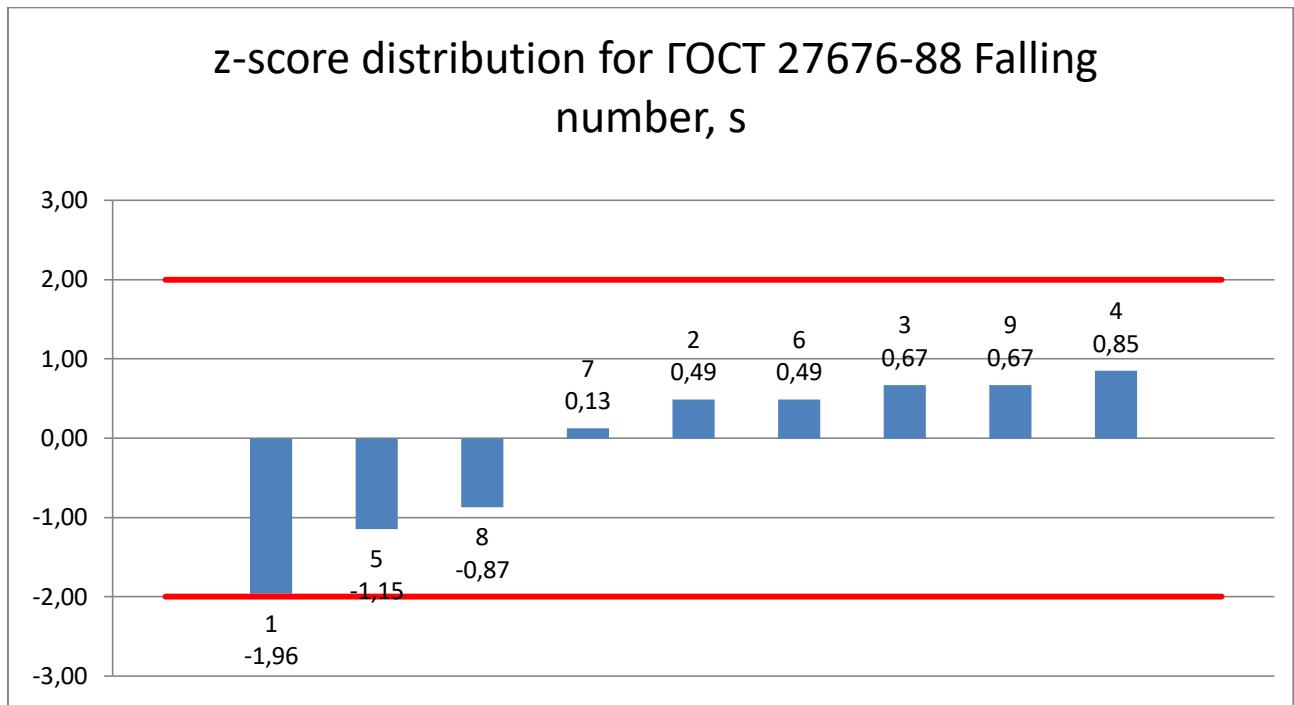
8.17. ГОСТ 10846-91 Protein content, % (Expressed on dry matter, factor for converting nitrogen content to protein content - 5.7)



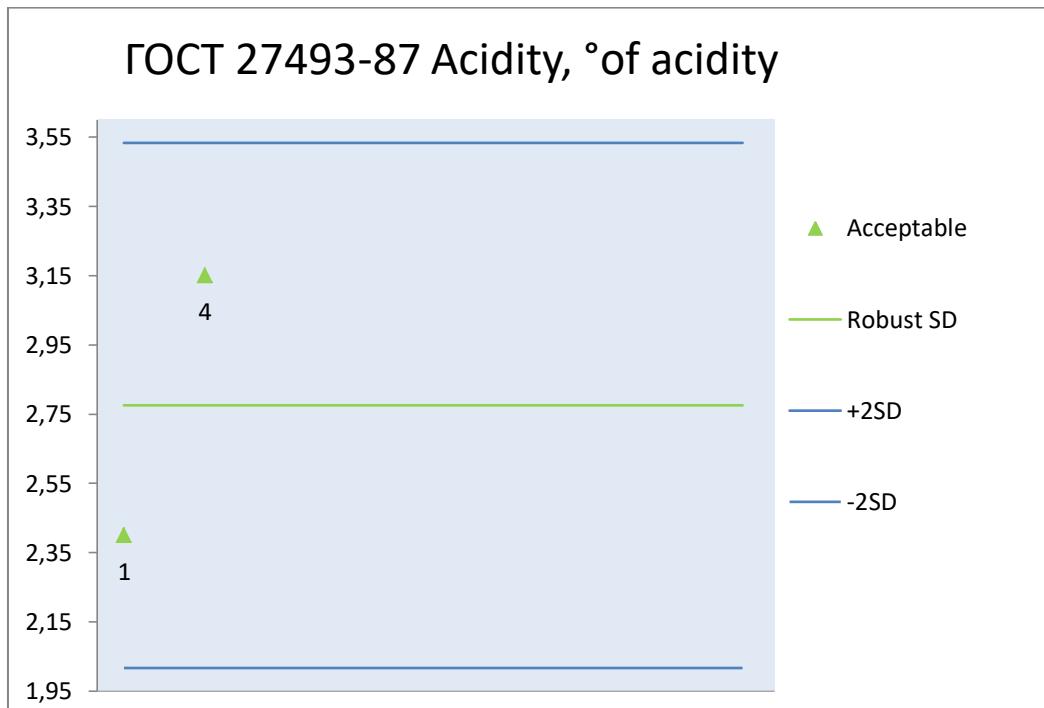
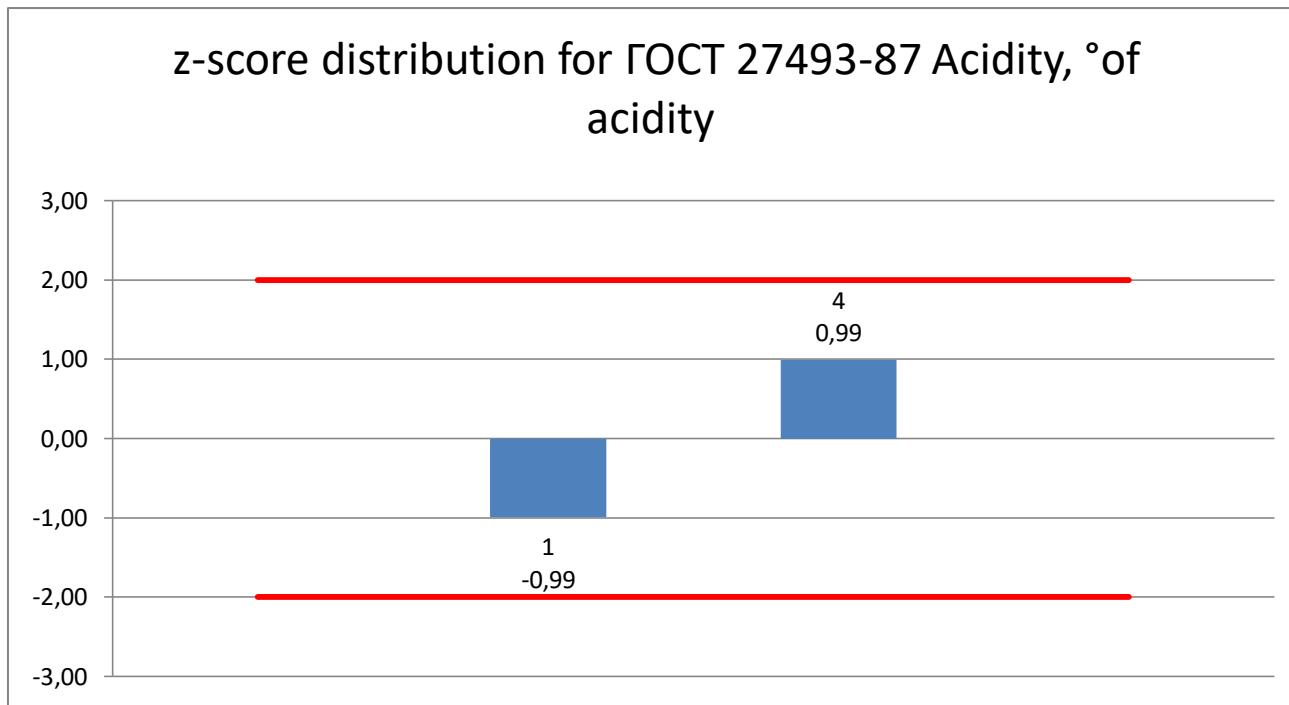
8.18. ГОСТ 27494-87 Ash content, %



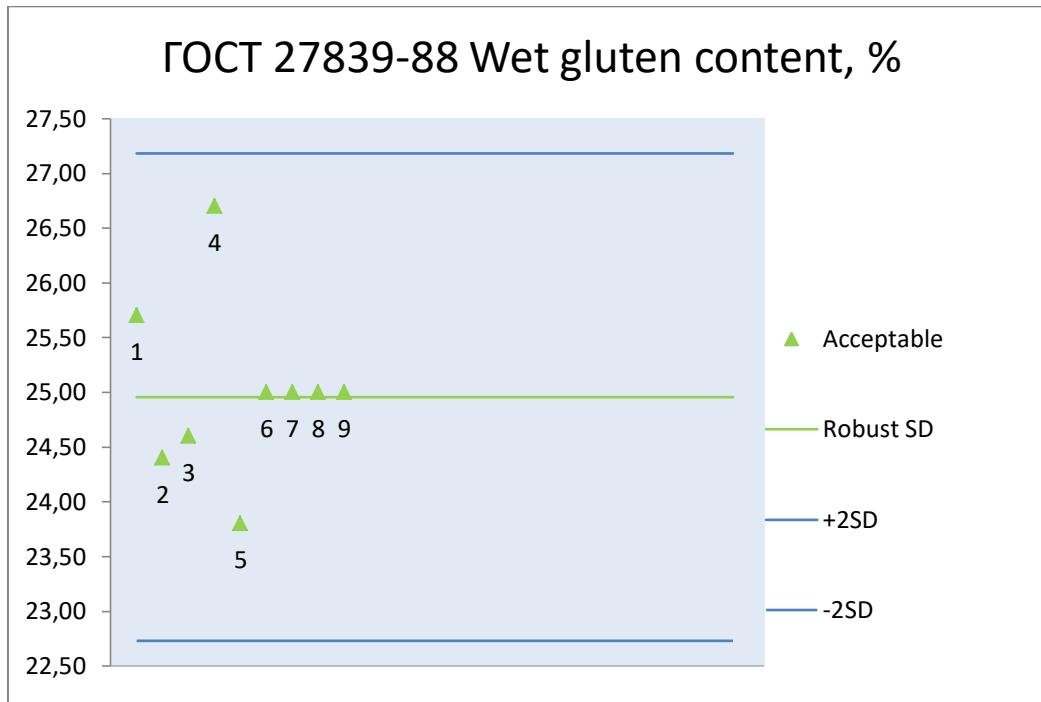
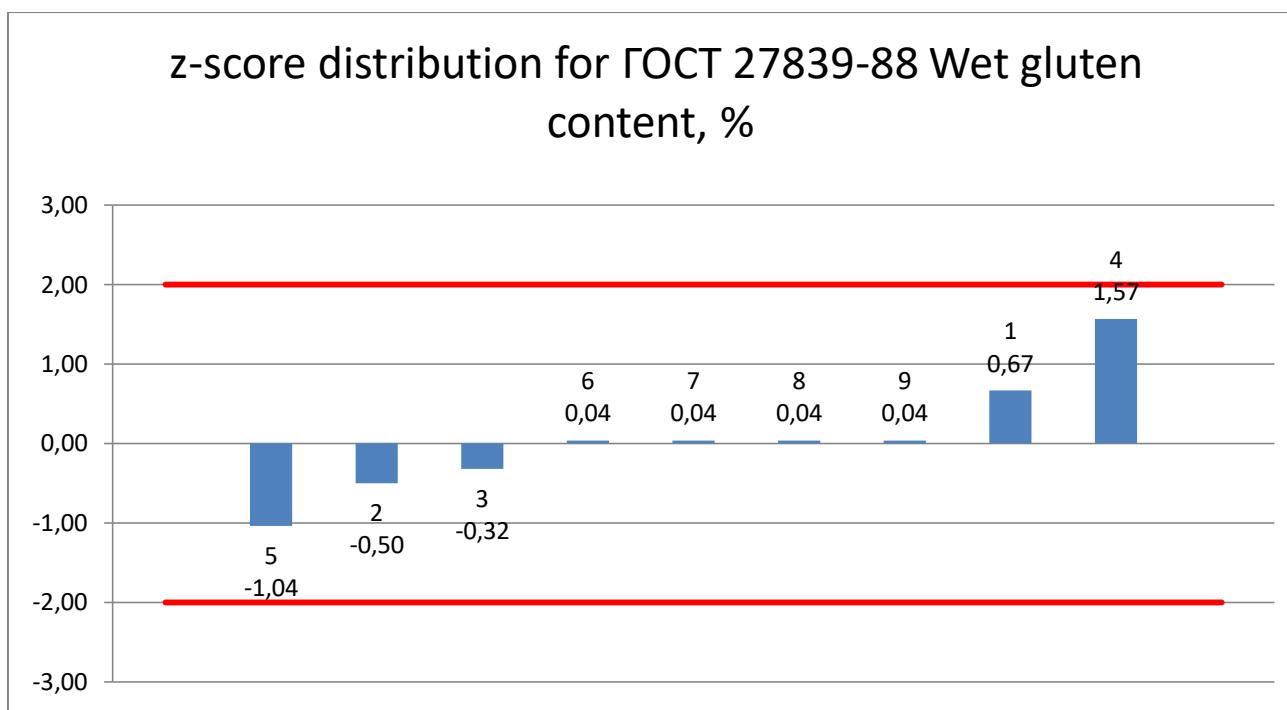
8.19. ГОСТ 27676-88 Falling number, s



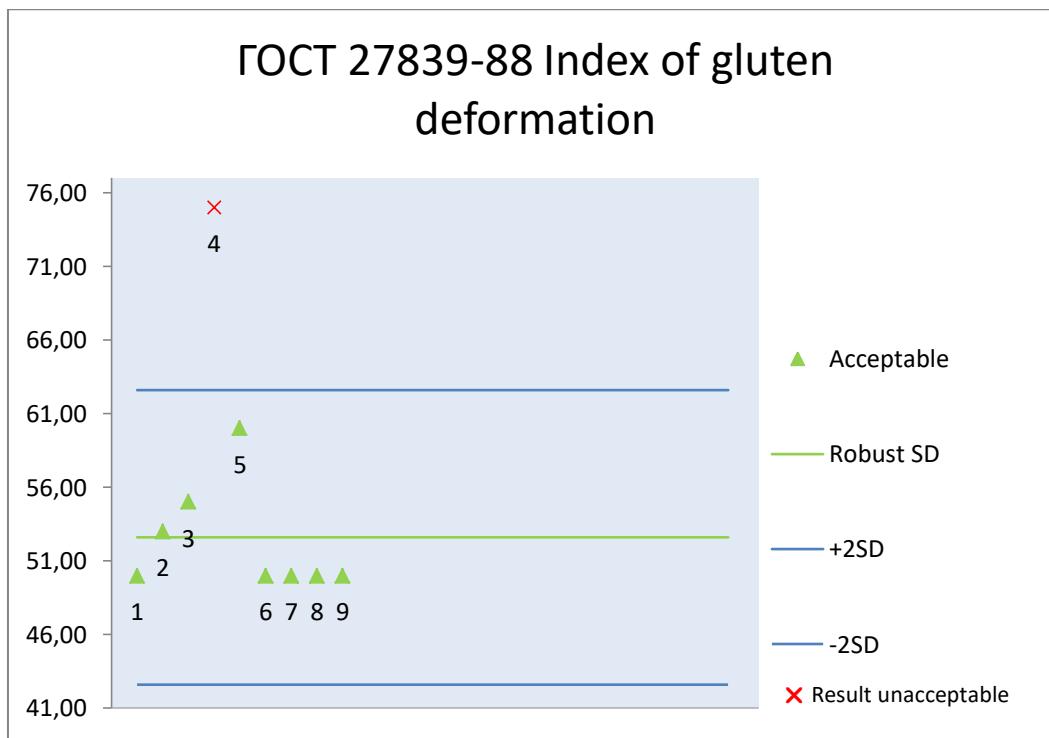
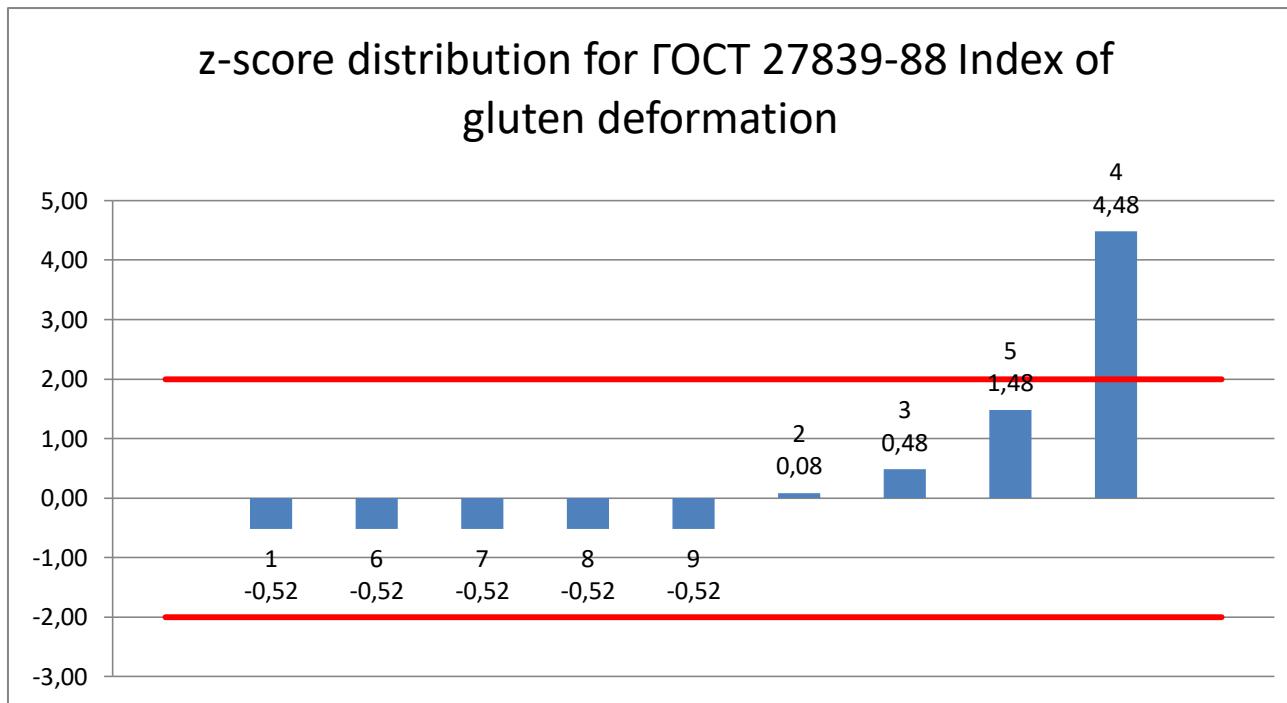
8.20. ГОСТ 27493-87 Acidity, °of acidity



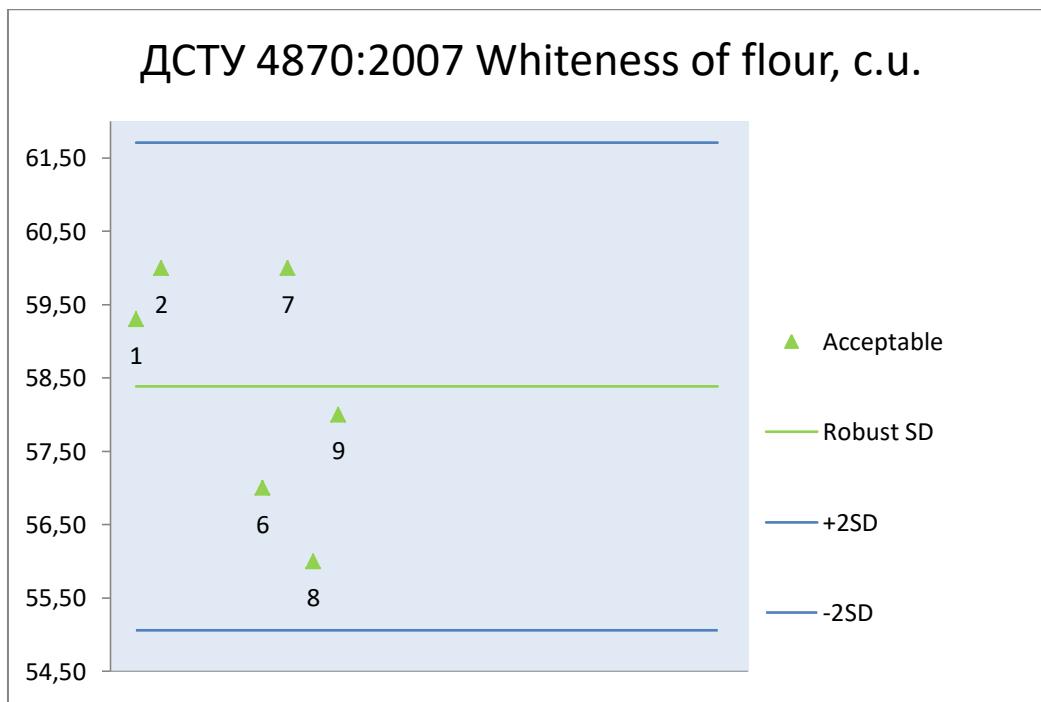
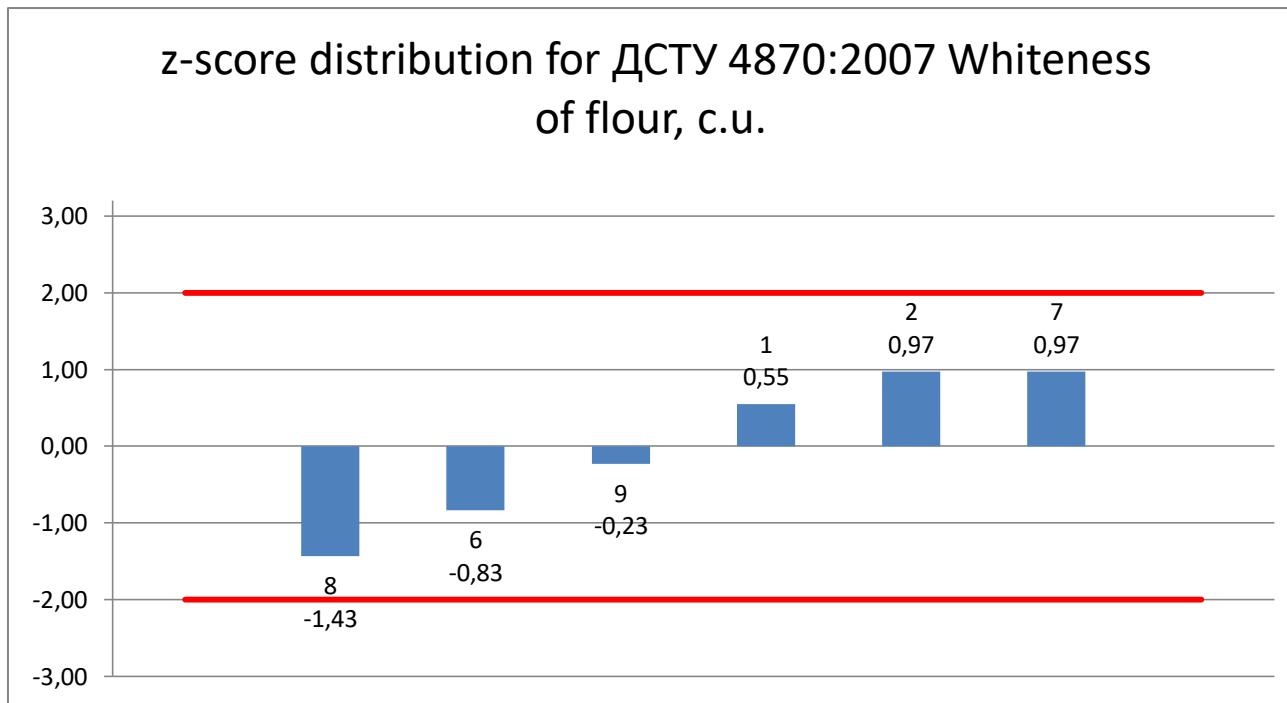
8.21. ГОСТ 27839-88 Wet gluten content, %



8.22. ГОСТ 27839-88 Index of gluten deformation



8.23. ДСТУ 4870:2007 Whiteness of flour, c.u.



9. NORMATIVE REFERENCE

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2. Analytical Methods Committee, Robust Statistics – How not to reject outliers Part 1. Basic Concepts, *Analyst*, 1989, 114, 1693-1697
3. FOOD ANALYSIS PERFORMANCE ASSESSMENT SCHEME (FAPAS). Protocol for the organization and analysis of data, sixth edition, 2002.
4. Fearn, T. and Thompson, M, A new test for ‘sufficient homogeneity’, *Analyst*, 2001, 126, 1414-1417
5. ISO 13528:2015 Statistical methods for use in proficiency testing by interlaboratory comparisons
6. ISO Guide 35:2006 Reference materials -- General and statistical principles for certification
7. ILAC Discussion Paper on Homogeneity and Stability Testing, April 2008.
8. ISO 17034:2016 General requirements for the competence of reference material producers