



Bozhko Nataliia
February 22nd 2018

PROFICIENCY TESTING PT.UA.1.2.2016
MAIZE ANALYSIS (QUALITY)
PROFICIENCY TESTING REPORT
ROUND 2 FEBRUARY 2018(ENG)

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Status:	Final

Kyiv-2018

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2. SUMMARY

2.1. The purpose of proficiency testing in wheat testing 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.1.2.2016 Round 2 held in February 2018. This report is issued according to ISO\IEC 17043 [1] and PT.UA.1.2.2016 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 18 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 PROFECIENCY 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.1.2.2016 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 – **Maize (*Zéa máys*)** were dispatched 29.01.2018 according to schedule of proficiency testing programme PT.UA.1.2.2016 Round 2.

3.3.2. Each produced and identified sample was hermetically sealed.

3.3.3. A total of 18 participants in 2 countries received one sample. Results were returned from 18 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 ASSESMENT

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. Some Participants stated ash temperature according to ISO 2171:2007. This data is stated in clause 9 for information purposes only. Considering results of the current round and target standard deviation, Provider can state, that ash burning temperature (500°C -900°C) does not contribute a major influence on a measurement result for this method.

3.5.6. Provider did not asses results for Γ OCT Starch content, % (Expressed on dry matter) and for ISO 6493:2000 Starch content, % (Expressed as a mass fraction of the product as received) because of not enough results quantity reported by participants. This data is presented in clause 6 for information purposes only.

3.5.7. Participant #18 stated Γ OCT 26226-95 Ash yield, % (Expressed as a mass fraction of dry product) instead of ISO 2171:2007. This result was not assessed by the Provider.

3.5.8. Only 2.41% of all results in this round are considered to be unsatisfactory. No results were deemed unsatisfactory in Round 1.

4. HOMOGENITY AND STABILITY ASSESMENT

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].

4.4. ISO 6540:1980 Moisture content, %

Source of σ value to use		σ
Use(write '1')	Source	
	C>13.8%, HORWITZ	0,3802
1	120ppb<C<13.8%, HORWITZ	0,3867
	C<120 ppb	3,179770
MASS NEGATIVE POWER FOR HORWITZ EQUATION(%=2, ppb=9,ppm=6)		2
	SD	4,2700
	Trial SD	3,9800
	Target SD chosen	0,3867
	σ^2_{all}	0,013462
	Replicates	10
	F1	1,88
	F2	1,01
	Critical value	0,0268
	Between sample variance S^2_{sam}	0,0014
	Sufficient homogeneity test	PASS

4.5. Data for all analytes

Method	EN 16378:2013	EN 16378:2013	EN 16378:2013	ISO 6540:1980	ISO 20483:2013	ISO 2171:2007	ISO 6492:1999	ISO 6865:2000	FOCT 10840-64	FOCT 13586.5-93
	Broken grains, %	Grain impurities, %	Miscellaneous impurities, %	Moisture content, %	Crude protein content, % (Expressed as a mass fraction of dry product)	Ash yield, % (Expressed as a mass fraction of dry product)	Fat content, % (Expressed as a mass fraction of the product as received)	Crude fibre content, % (Expressed as a mass fraction of the product as received)	Test weight, g/l	Moisture content, %
Homogeneity and stability										
Cohran's 'C' test										
Critical value(5%,10pairs)=0,602	0,6069	0,5032	0,2900	0,5848	0,3772	0,3144	0,3147	0,4740	0,3556	0,5100
Mean Result	2,8190	1,3570	1,2405	14,4535	8,4055	1,2319	3,2415	1,9410	726,3500	14,2420
Conclusion	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS
Analytical variance test										
S ² anal	0,0297	0,1811	0,0083	0,0014	0,0144	0,0000	0,0027	0,0077	2,2500	0,0025
S ² anal	0,1722	0,4256	0,0914	0,0380	0,1201	0,0065	0,0518	0,0877	1,5000	0,0501
S ² sample	0,0277	0,2547	0,0235	0,0014	0,0000	0,0001	0,0028	0,0029	2,2111	0,0008
σ _p	0,2460	0,9300	0,3360	0,3867	0,1400	0,0477	0,1086	0,1800	3,1340	0,3819
σ _p source	Trial SD	Method Tr SD	Trial SD	Horwitz	Method Tr	Horwitz	Horwitz	Method Tr S	Trial SD	Horwitz
σ ² all	0,0054	0,0778	0,0102	0,0135	0,0018	0,0002	0,0011	0,0029	0,8840	0,0131
Critical value	0,0402	0,3293	0,0275	0,0268	0,0179	0,0004	0,0047	0,0132	3,9344	0,0272
Conclusion	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS	PASS

5. DATA SUMMARY

Method	EN 16378:2013	EN 16378:2013	EN 16378:2013	ISO 6540:1980	ISO 20483:2013	ISO 2171:2007	ISO 6492:1999	ISO 6865:2000	USDA(Grain Grading Procedures, Chapter 4 - Corn April 11, 2017)	USDA(Grain Grading Procedures, Chapter 4 - Corn April 11, 2017)	USDA (Grain Grading Procedures, Chapter 4 - Corn April 11, 2017)	USDA(Grain Grading Procedures, Chapter 1 - General Information July 30, 2013)	FOCT 10840-64	FOCT 30483-97/ДСТУ 4525:2006	FOCT 30483-97/ДСТУ 4525:2006	FOCT 13586.5-93	ДСТУ 7169:2010	FOCT 13496.15-97	FOCT 13496.2-91
	Broken grains, %	Grain impurities, %	Miscellaneous impurities, %	Moisture content, %	Crude protein content,% (Expressed as a mass fraction of dry product)	Ash yield, % (Expressed as a mass fraction of dry product)	Fat content,% (Expressed as a mass fraction of the product as received)	Crude fibre content,% (Expressed as a mass fraction of the product as received)	Broken Corn, %	Foreign Material, %	Damaged kernels, %	Test weight, lbu/bu	Test weight, g/l	Foreign impurities, %	Grain impurities, %	Moisture content, %	Crude protein content,% ((expressed on dry matter, factor for converting nitrogen content to protein content - 6,0)	Fat content,% (Expressed on dry matter)	Crude fibre content,% (Expressed on dry matter)
No of Results	12	12	12	12	10	11	9	10	8	8	8	8	13	14	14	16	10	9	8
No of Results z >3	0	0	0	0	1	0	0	0	0	0	0	0	0	1	1	0	0	2	0
No of Results z >3, %	0,000	0,000	0,000	0,000	10,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	0,000	7,143	7,143	0,000	0,000	22,222	0,000
Mean	2,913	0,953	1,358	14,281	8,226	1,246	3,418	1,865	3,180	0,929	2,843	57,511	724,692	1,731	4,339	13,877	7,986	3,864	1,938
Min	2,580	0,460	0,850	13,930	7,020	0,887	3,220	1,380	3,100	0,700	2,230	55,900	720,000	1,090	3,030	13,300	7,860	3,400	1,400
Max	3,220	1,670	1,880	14,570	8,420	1,370	3,660	2,230	3,390	1,090	4,160	59,400	732,000	4,100	9,710	14,130	8,300	4,290	2,350
SD	0,246	0,339	0,339	0,163	0,426	0,132	0,146	0,251	0,090	0,111	0,759	1,510	3,660	0,761	1,720	0,292	0,124	0,236	0,315
Median	2,830	0,890	1,415	14,310	8,360	1,270	3,380	1,820	3,150	0,940	2,430	57,595	724,000	1,585	3,840	14,025	7,955	3,850	2,000
Robust mean(assigned value)	2,913	0,930	1,356	14,292	8,367	1,289	3,437	1,807	3,158	0,940	2,800	57,511	724,111	1,630	3,994	13,919	7,979	3,822	1,933
Robust SD	0,246	0,279	0,336	0,108	0,062	0,064	0,138	0,210	0,040	0,063	0,682	1,510	3,134	0,375	0,666	0,217	0,068	0,127	0,288
SD from method(Tr.SD)	0,740	0,930	0,850	N/A	0,140	0,033	0,199	0,180	N/A	N/A	N/A	N/A	N/A	0,300	1,000	0,700	N/A	N/A	N/A
SD from Horwitz eq.	0,099	0,038	0,052	0,378	0,243	0,050	0,114	0,066	0,106	0,038	0,096	N/A	N/A	0,061	0,130	0,375	0,233	0,125	0,070
Target SD	0,246	0,930	0,336	0,378	0,140	0,050	0,114	0,180	0,106	0,090	0,976	1,387	3,134	0,300	1,000	0,375	0,233	0,125	0,288
Source of target SD of PT	Trial SD	Method Tr SD	Trial SD	Horwitz	Method Tr SD	Horwitz	Horwitz	Method Tr SD	Trial SD	Trial SD	Trial SD	Trial SD	Trial SD	Method Tr SD	Method Tr SD	Horwitz	Horwitz	Horwitz	Trial SD

6. RAW DATA

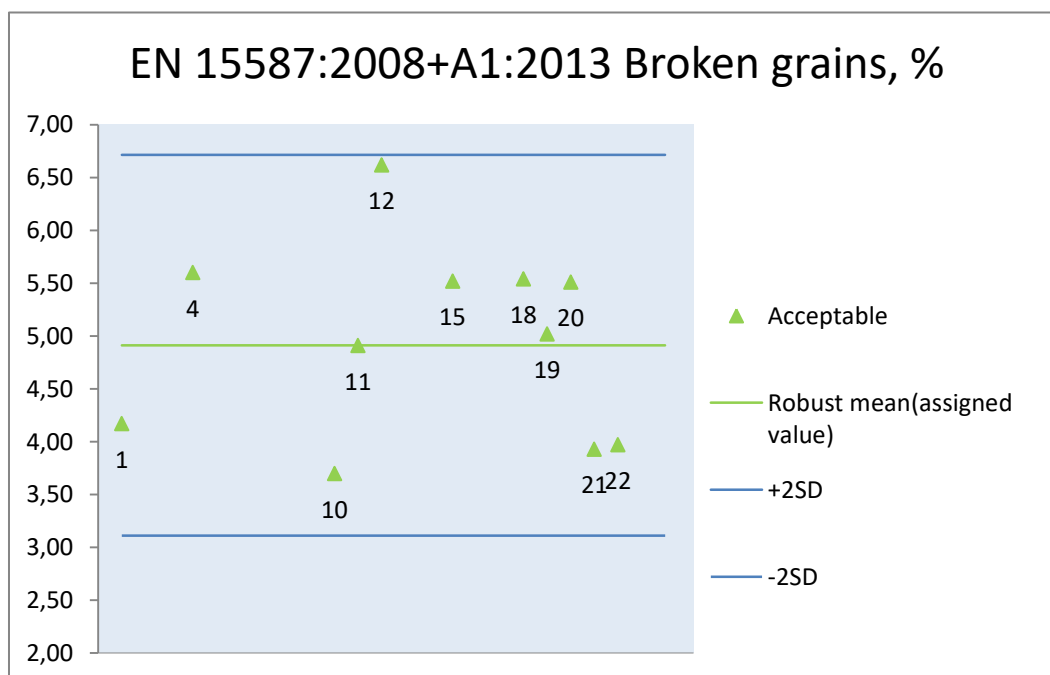
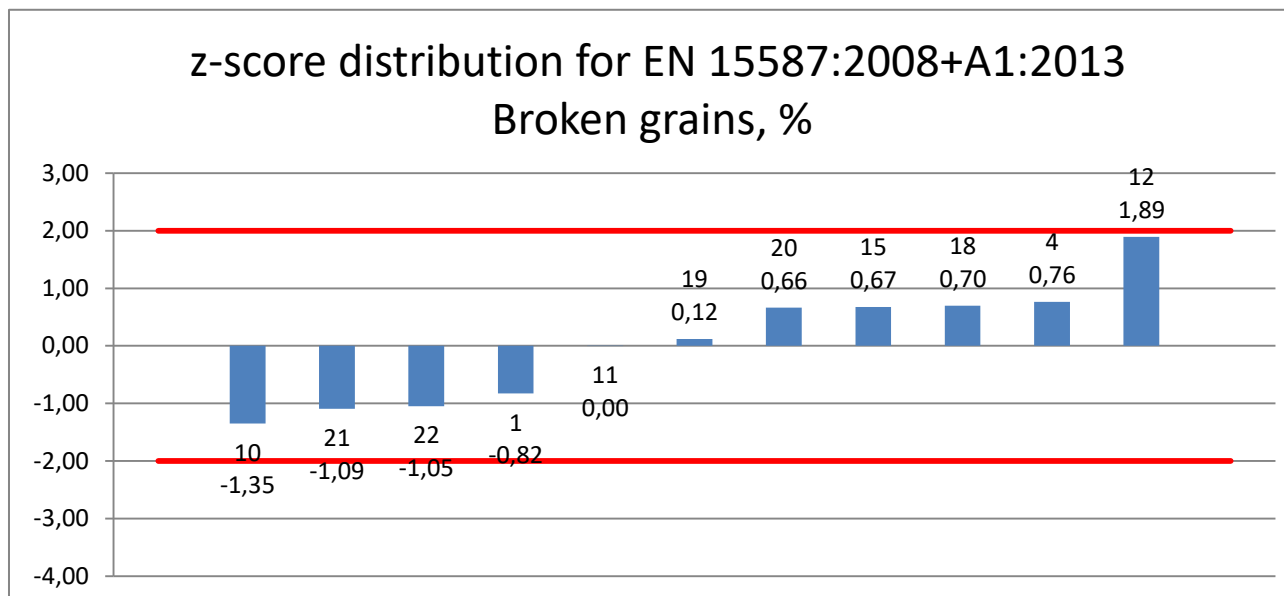
	EN 16378:2013	EN 16378:2013	EN 16378:2013	ISO 6540:1980	ISO 6493:2000	ISO 20483:2013	ISO 2171:2007	ISO 6492:1999	ISO 6865:2000	USDA(Grain Grading Procedures, Chapter 4 - Corn April	USDA(Grai n Grading Procedures, Chapter 4 - Corn April 11, 2017)	USDA (Grain Grading Procedures, Chapter 4 - Corn April	USDA(Grai n Grading Procedures, Chapter 1 - General Information	ГОСТ 10840-64	ГОСТ 30483- 97/ДСТУ 4525:2006	ГОСТ 30483- 97/ДСТУ 4525:2006	ГОСТ 13586.5-93	ГОСТ 10845-98	ДСТУ 7169:2010	ГОСТ 13496.15- 97	ГОСТ 13496.2-91
Method																					
	Broken grains, %	Grain impurities, %	Miscellaneo us impurities, %	Moisture content, %	Starch content, % (Expressed as a mass fraction of the product as received)	Crude protein content,% (Expressed as a mass fraction of dry product)	Ash yield, % (Expressed as a mass fraction of dry product)	Fat content,% (Expressed as a mass fraction of the product as received)	Crude fibre content,% (Expressed as a mass fraction of the product as received)	Broken Corn, %	Foreign Material, %	Damaged kernels, %	Test weight, lb/bu	Test weight, g/l	Foreign impurities, %	Grain impurities, %	Moisture content, %	Starch content, % (Expressed on dry matter)	Crude protein content,% ((expressed on dry matter, factor for converting nitrogen content to protein content - 6,0)	Fat content,% (Expressed on dry matter)	Crude fibre content,% (Expressed on dry matter)
Laboratory number																					
1	2,68	1,67	1,35	14,25	54,12	8,40	1,23	3,22	2,02	3,15	1,09	3,76	56,49	727,00	1,35	5,92	14,05	76,08	8,05	3,75	2,35
2	2,74	0,98	0,93	14,09										732,00	1,09	3,33	13,80				
3	3,10	0,68	1,67	14,32		8,37	1,33	3,60	1,74						1,90	3,86	14,00				
4	3,16	0,70	1,60	14,34		8,35	1,32							723,00	1,24	4,10	13,50				
5														723,00	1,86	3,82	14,10		7,94		
6	3,22	0,66	1,60	14,30										724,00	1,78	3,82	14,10				
7	2,80	0,46	1,48	14,57		8,25	1,26	3,66	1,82	3,39	0,92	4,16	59,40	724,00	1,86	3,10	14,13		7,92	3,98	2,11
8	2,73	0,80	0,87	13,93						3,15	0,88	2,30	58,80	720,00	2,07	3,65	14,10				
9	2,67	1,22	1,88	14,40		8,33	1,37	3,38	1,74	3,10	0,97	2,23	58,70								
10	2,86	1,20	1,59	14,37		8,39	1,33	3,44	1,70	3,16	0,93	2,26	58,70								
11	2,58	1,28	0,85	14,20		8,34	1,30	3,48	1,82	3,13	0,70	3,17	56,10	722,00	1,39	4,28	13,70		7,97	3,81	1,91
12	3,20	0,97	1,22	14,38	65,00	8,39	1,25	3,36	2,10	3,15	0,95	2,42	55,90	721,00	1,91	4,00	14,10		8,00	3,84	1,62
13	3,21	0,81	1,25	14,22		8,42	1,16	3,37	1,38	3,21	0,99	2,44	56,00	721,00	1,38	4,87	14,10		8,00	3,40	1,40
14														730,00	1,20	3,26	13,30		7,86	3,86	2,20
15														727,00	4,10	9,71	13,30		8,30	3,85	1,82
16							1,27		2,10								14,05		7,88	4,29	
17																	14,00		7,94	4,00	2,09
18						7,02	0,89	3,25	2,23					727,00	1,11	3,03	13,70				

7. Z SCORES

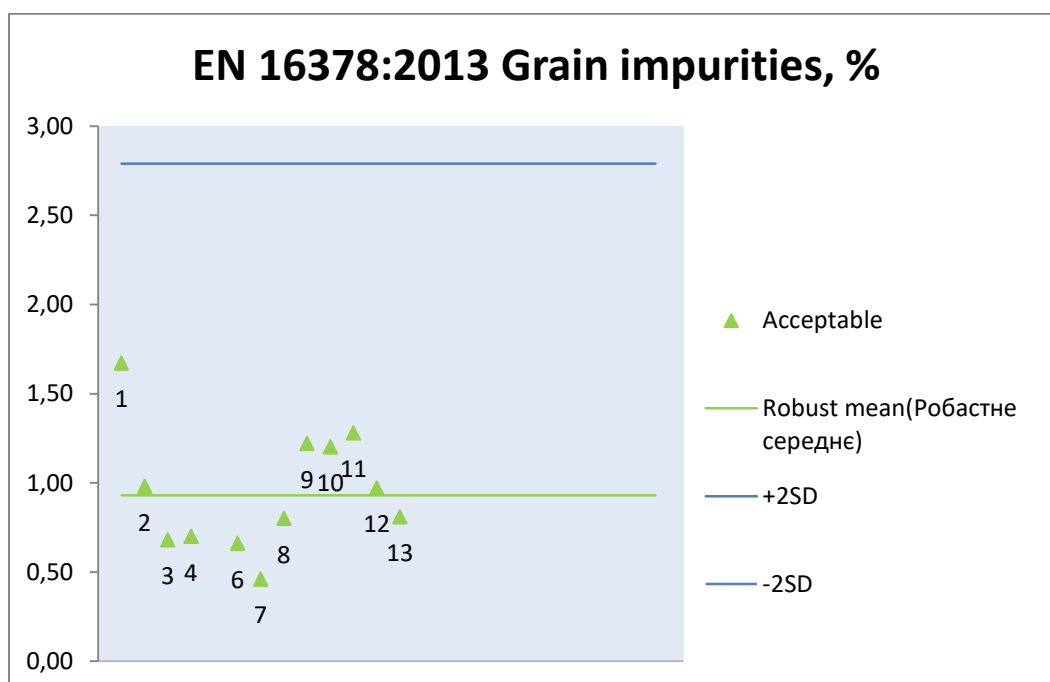
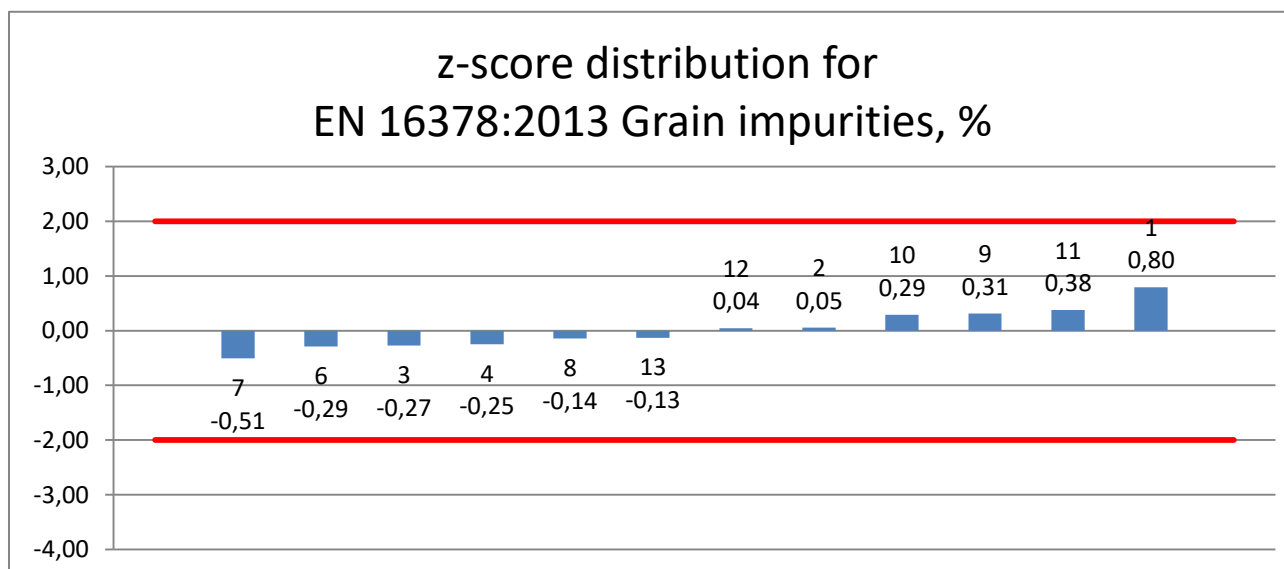
Method	EN 16378:2013	EN 16378:2013	EN 16378:2013	ISO 6540:1980	ISO 20483:2013	ISO 2171:2007	ISO 6492:1999	ISO 6865:2000	USDA(Grain Grading Procedures, Chapter 4 - Corn April 11, 2017)	USDA(Grain Grading Procedures, Chapter 4 - Corn April 11, 2017)	USDA (Grain Grading Procedures, Chapter 4 - Corn April 11, 2017)	USDA(Grain Grading Procedures, Chapter 1 - General Information July 30, 2013)	ГОСТ 10840-64	ГОСТ 30483-97/ДСТУ 4525:2006	ГОСТ 30483-97/ДСТУ 4525:2006	ГОСТ 13586.5-93	ДСТУ 7169:2010	ГОСТ 13496.15-97	ГОСТ 13496.2-91
Laboratory number	Broken grains, %	Grain impurities, %	Miscellaneous impurities, %	Moisture content, %	Crude protein content, % (Expressed as a mass fraction of dry product)	Ash yield, % (Expressed as a mass fraction of dry product)	Fat content, % (Expressed as a mass fraction of the product as received)	Crude fibre content, % (Expressed as a mass fraction of the product as received)	Broken Corn, %	Foreign Material, %	Damaged kernels, %	Test weight, lbu/bu	Test weight, g/l	Foreign impurities, %	Grain impurities, %	Moisture content, %	Crude protein content, % ((expressed on dry matter, factor for converting nitrogen content to protein content - 6,0)	Fat content, % (Expressed on dry matter)	Crude fibre content, % (Expressed on dry matter)
1	-0,95	0,80	-0,02	-0,11	0,24	-1,17	-1,91	1,18	-0,08	1,67	0,98	-0,74	0,92	-0,93	1,93	0,35	0,31	-0,58	1,45
3	0,76	-0,27	0,93	0,07	0,02	0,83	1,43	-0,37						0,90	-0,13	0,21			
4	1,01	-0,25	0,73	0,13	-0,12	0,63							-0,35	-1,30	0,11	-1,12			
5													-0,35	0,77	-0,17	0,48	-0,17		
6	1,25	-0,29	0,73	0,02									-0,04	0,50	-0,17	0,48			
7	-0,46	-0,51	0,37	0,74	-0,84	-0,57	1,95	0,07	2,19	-0,22	1,39	1,36	-0,04	0,77	-0,89	0,56	-0,25	1,26	0,61
8	-0,74	-0,14	-1,45	-0,96					-0,08	-0,67	-0,51	0,93	-1,31	1,47	-0,34	0,48			
9	-0,99	0,31	1,56	0,29	-0,26	1,63	-0,50	-0,37	-0,55	0,33	-0,58	0,86							
10	-0,21	0,29	0,70	0,21	0,16	0,83	0,02	-0,59	0,02	-0,11	-0,55	0,86							
11	-1,35	0,38	-1,51	-0,24	-0,19	0,23	0,37	0,07	-0,27	-2,67	0,38	-1,02	-0,67	-0,80	0,29	-0,59	-0,04	-0,10	-0,08
12	1,17	0,04	-0,40	0,23	0,16	-0,77	-0,68	1,63	-0,08	0,11	-0,39	-1,16	-0,99	0,93	0,01	0,48	0,09	0,14	-1,09
13	1,21	-0,13	-0,32	-0,19	0,38	-2,57	-0,59	-2,37	0,49	0,56	-0,37	-1,09	-0,99	-0,83	0,88	0,48	0,09	-3,38	-1,85
14													1,88	-1,43	-0,73	-1,65	-0,51	0,30	0,93
15													0,92	8,23	5,72	-1,65	1,38	0,22	-0,39
16						-0,37		1,63								0,35	-0,42	3,74	
17																0,21	-0,17	1,42	0,55
18					-9,62		-1,64	2,35					0,92	-1,73	-0,96	-0,59			

8. Z SCORE PLOTS AND RESULTS CHARTS.

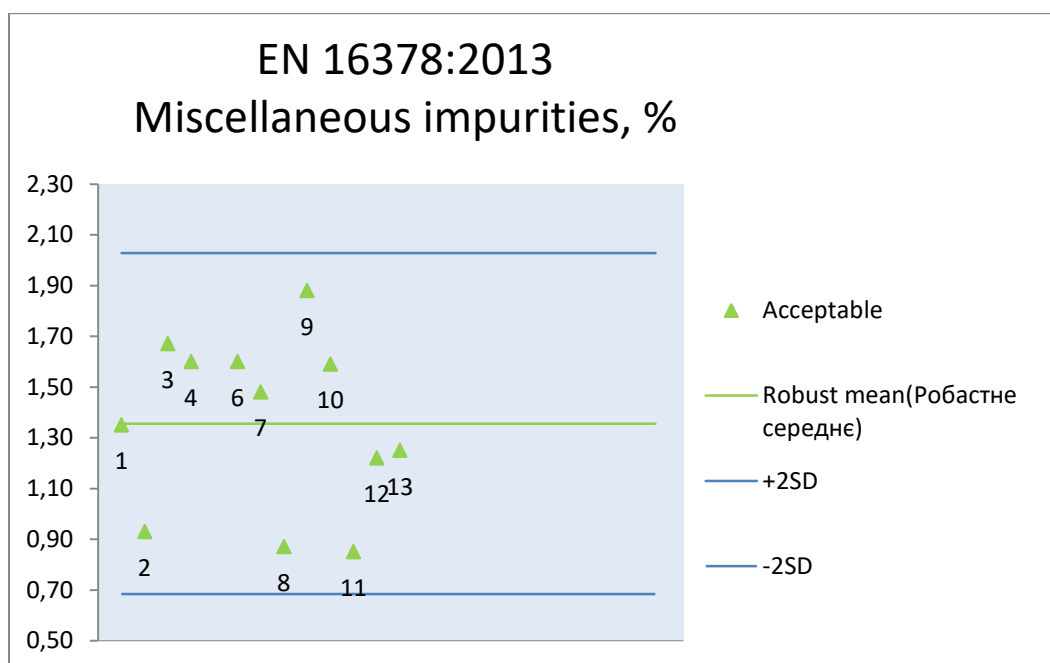
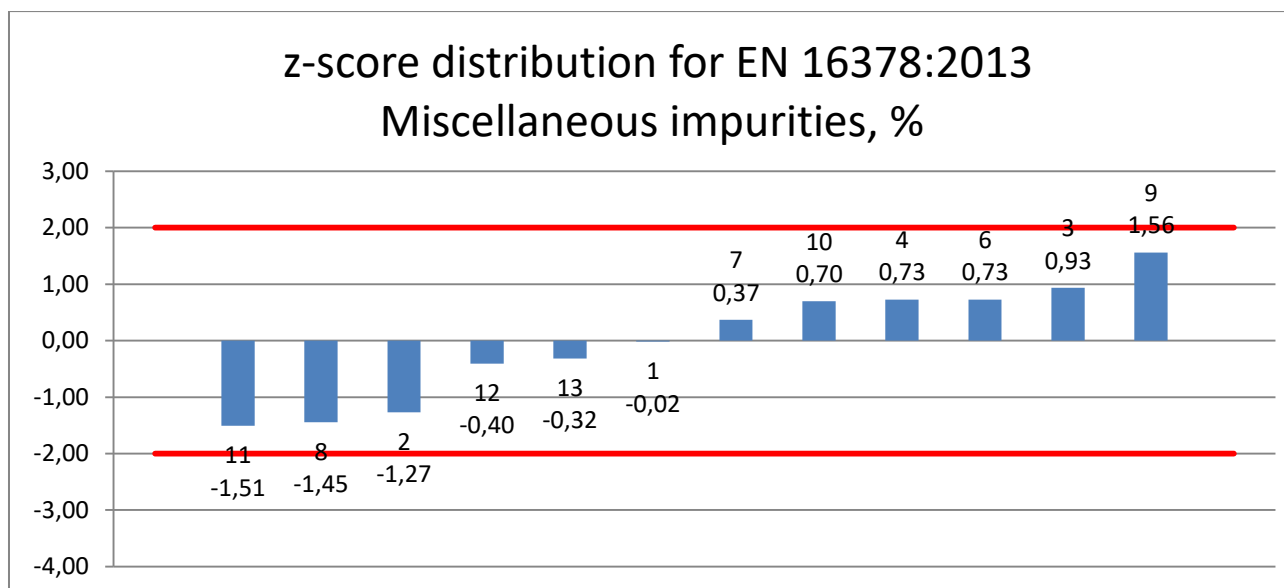
8.1. EN 15587:2008+A1:2013 Broken grains, %



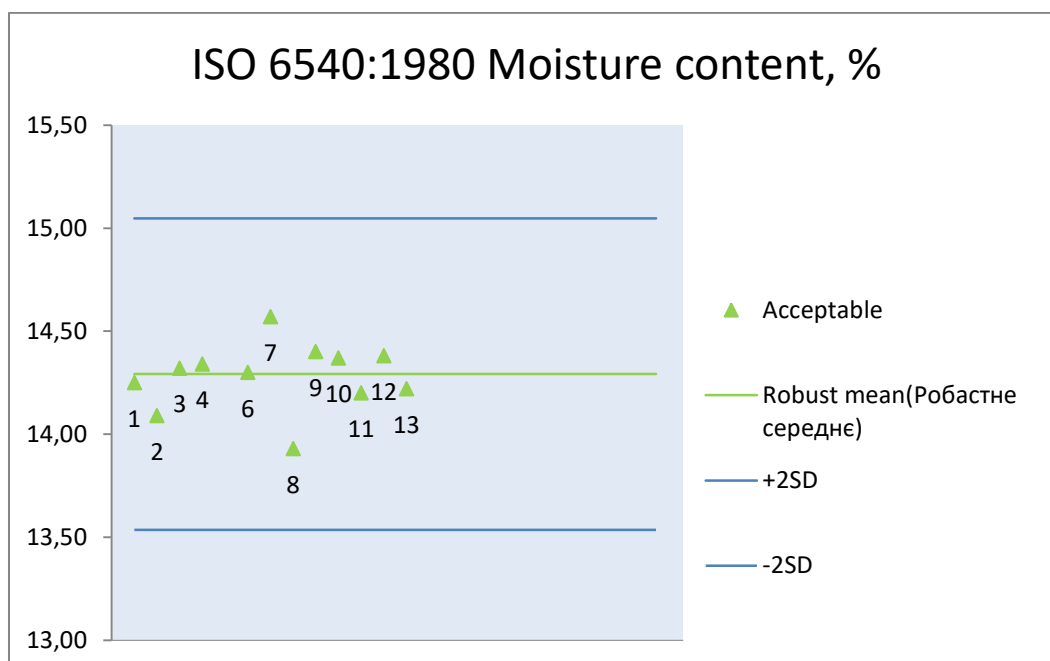
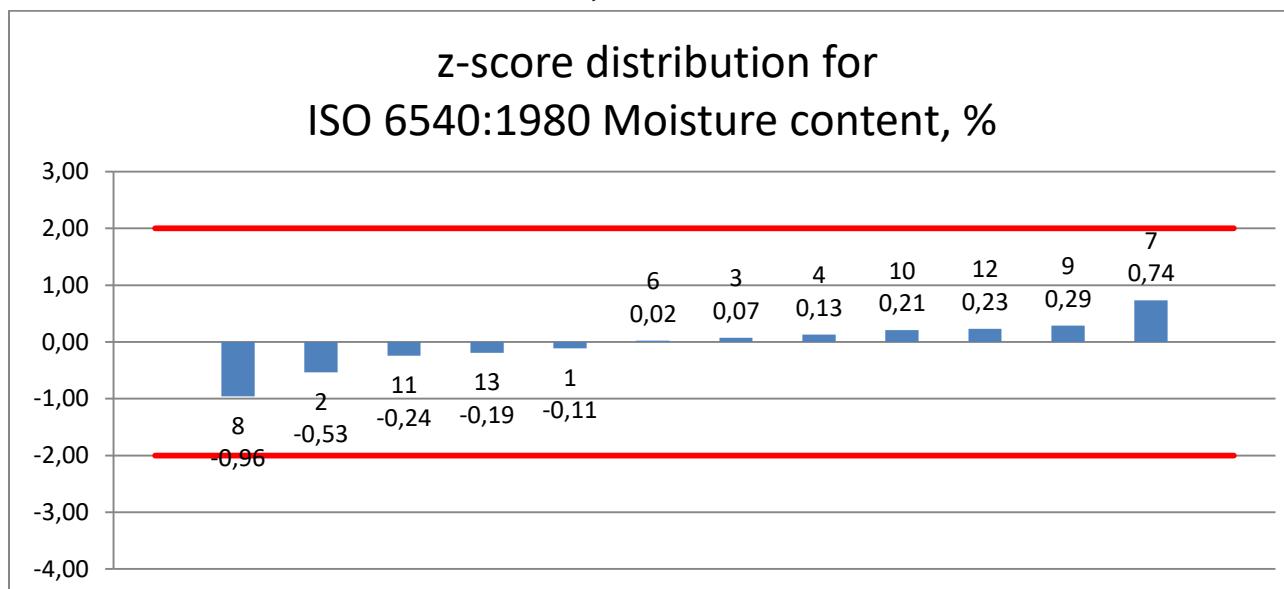
8.2. EN 16378:2013 Grain impurities, %



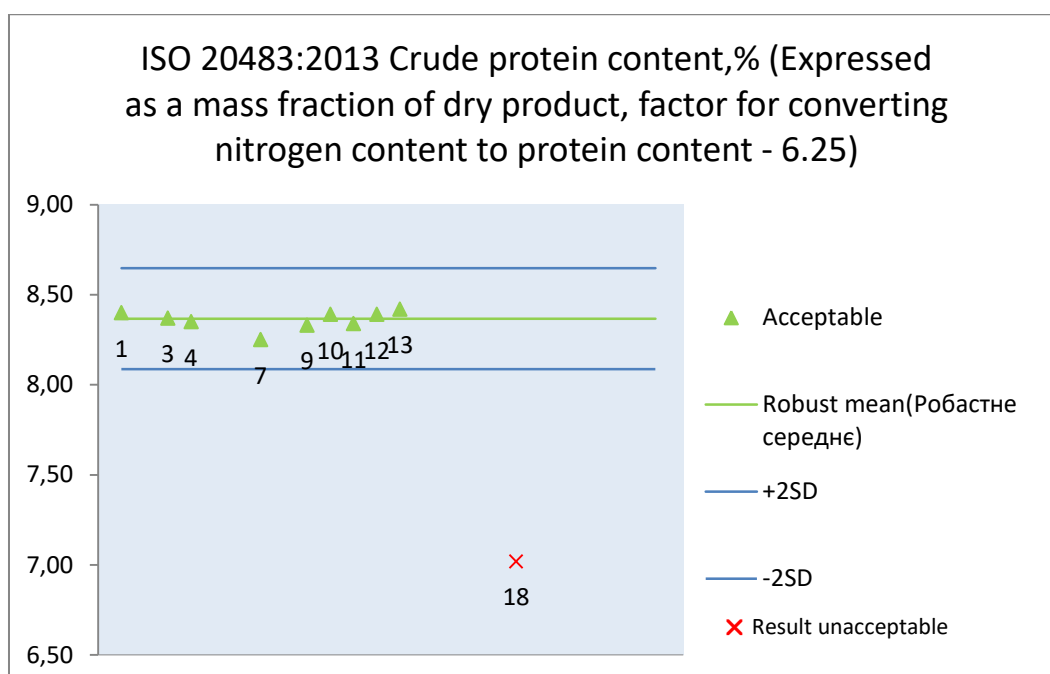
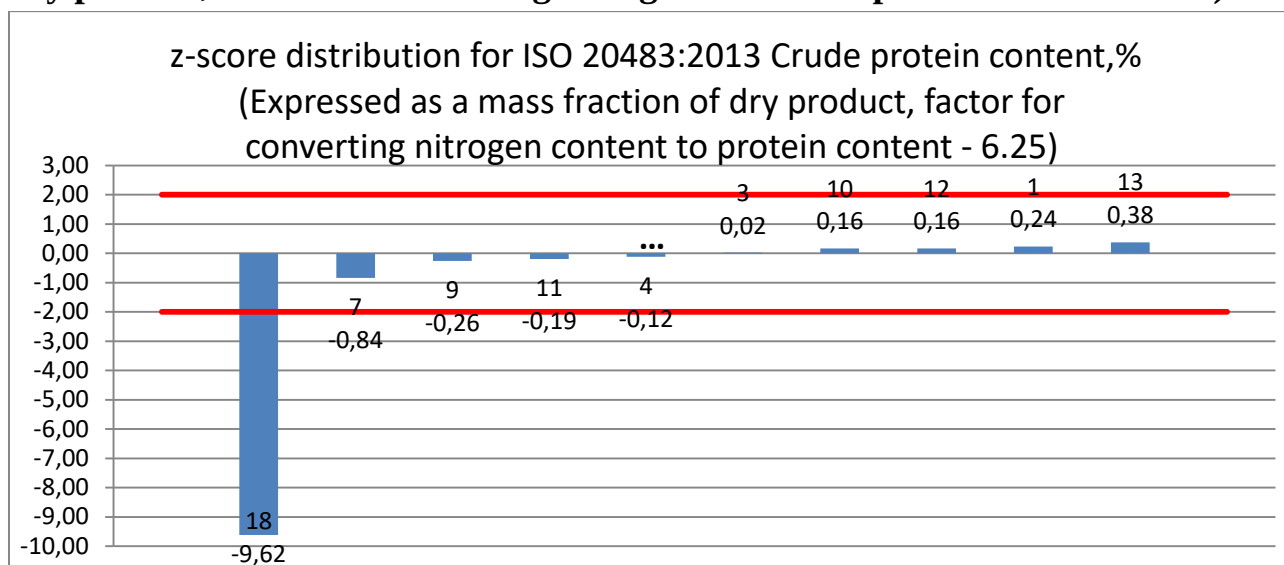
8.3. EN 16378:2013 Miscellaneous impurities, %



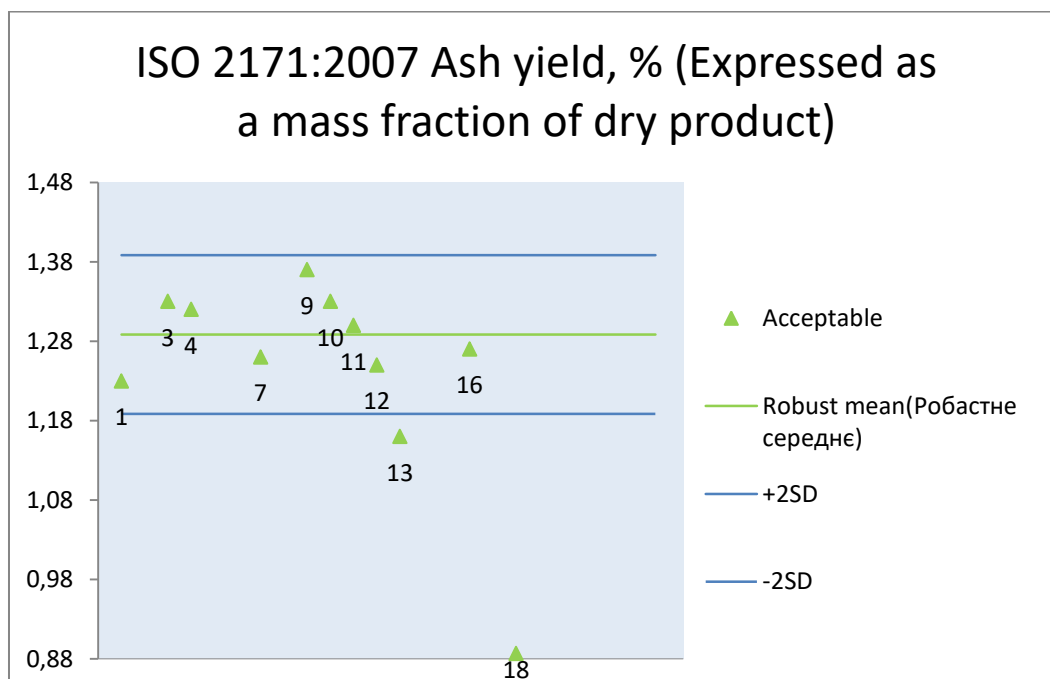
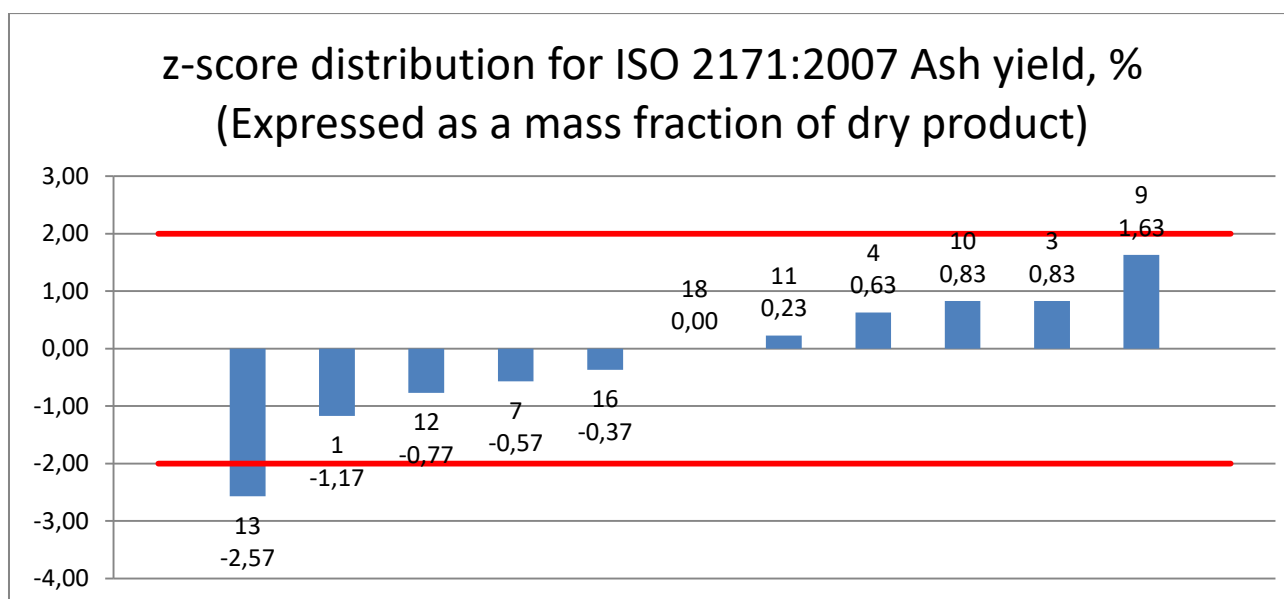
8.4. ISO 6540:1980 Moisture content, %



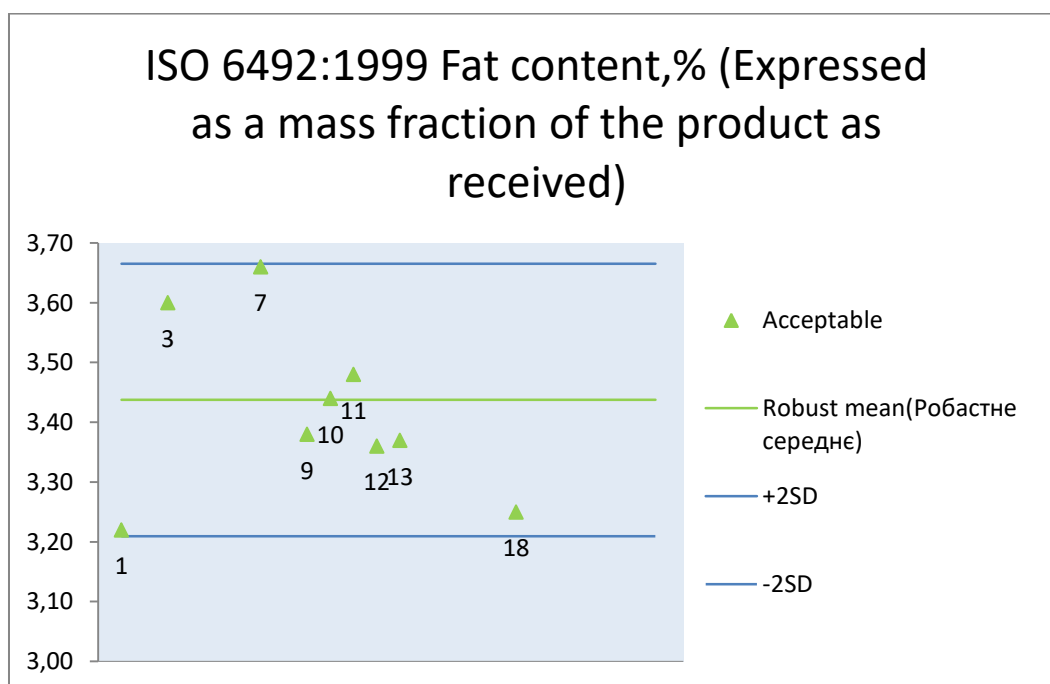
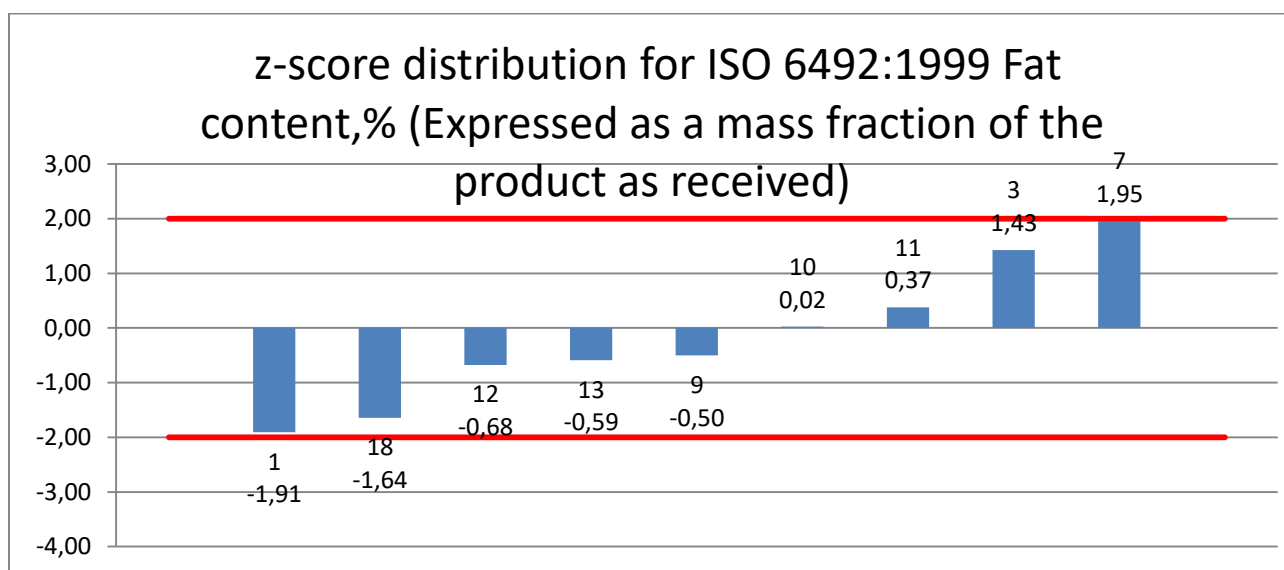
8.5. ISO 20483:2013 Crude protein content,% (Expressed as a mass fraction of dry product, factor for converting nitrogen content to protein content - 6.25)



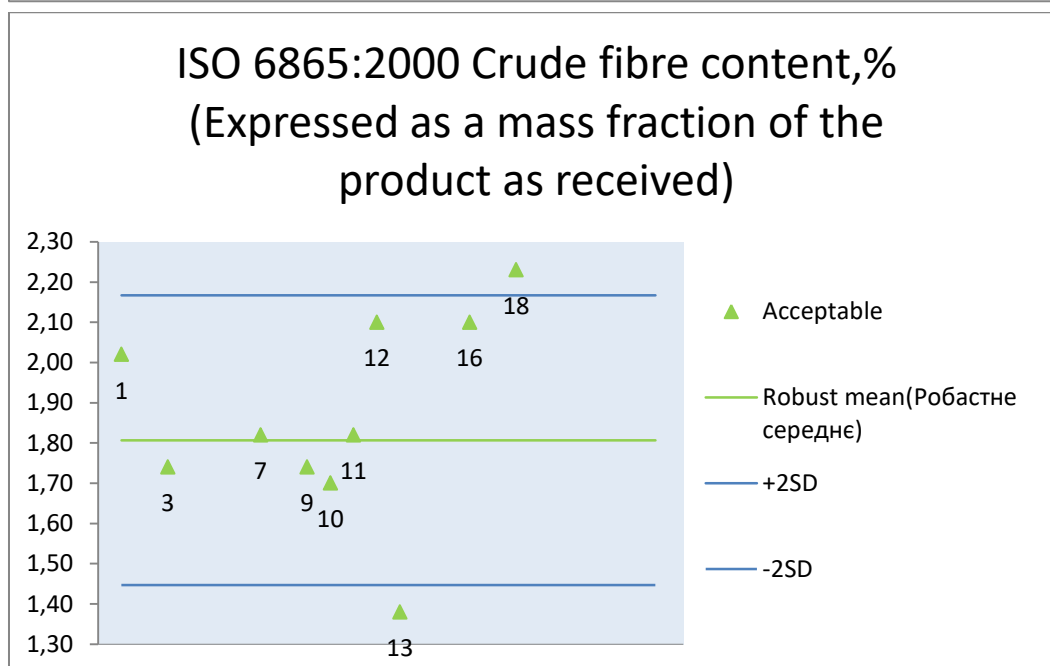
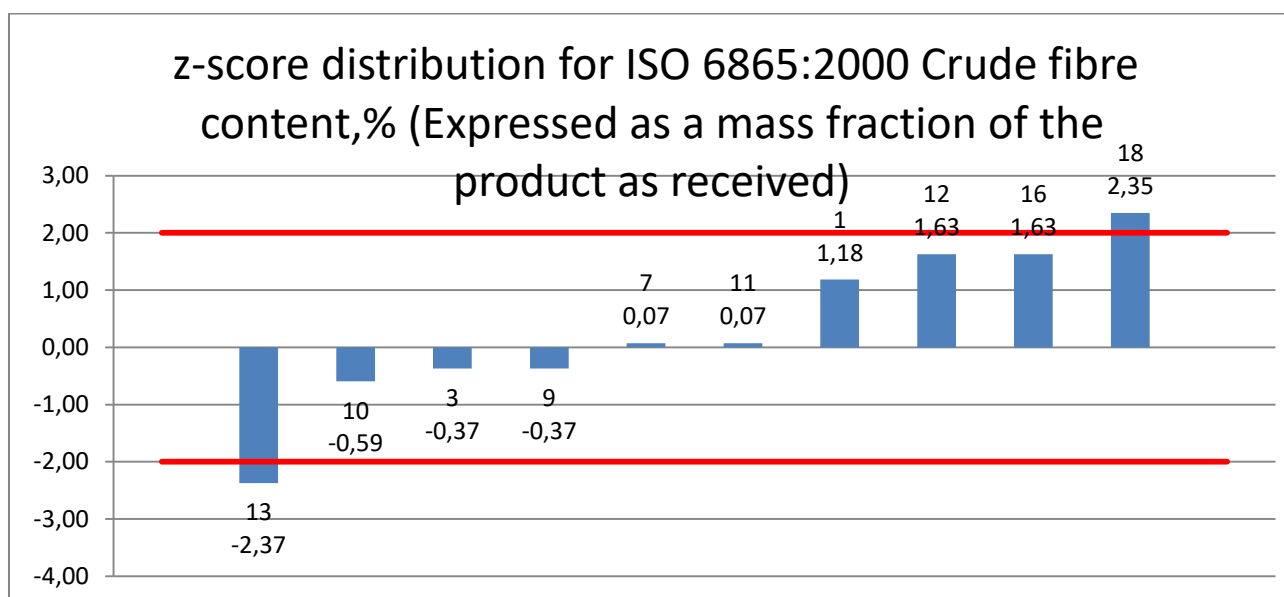
8.6. ISO 2171:2007 Ash yield, % (Expressed as a mass fraction of dry product)



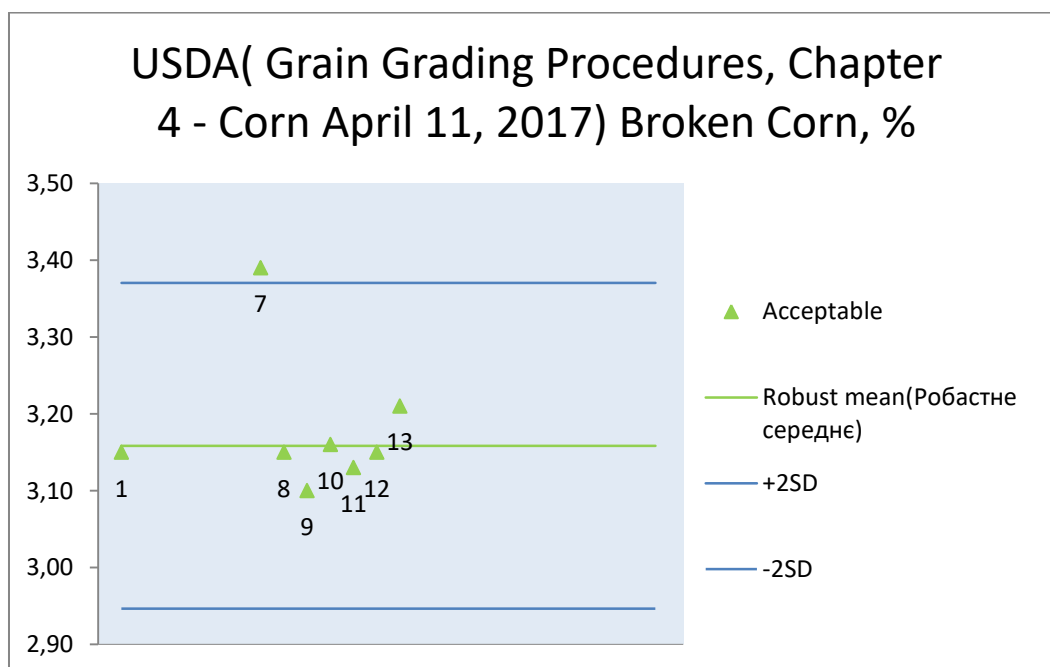
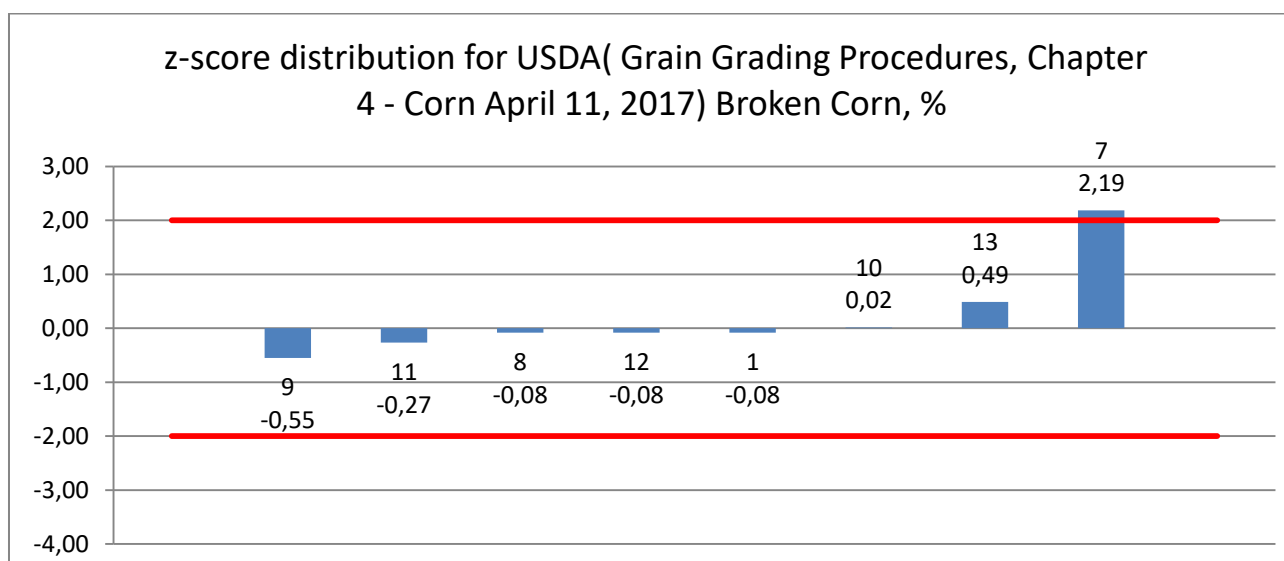
8.7. ISO 6492:1999 Fat content,% (Expressed as a mass fraction of the product as received)



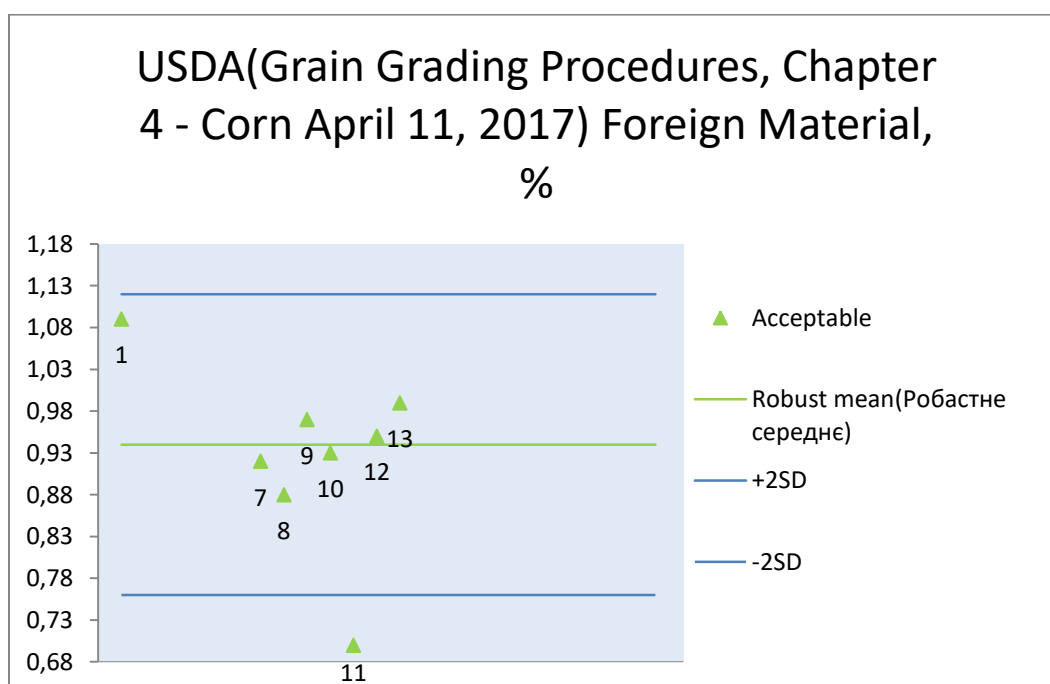
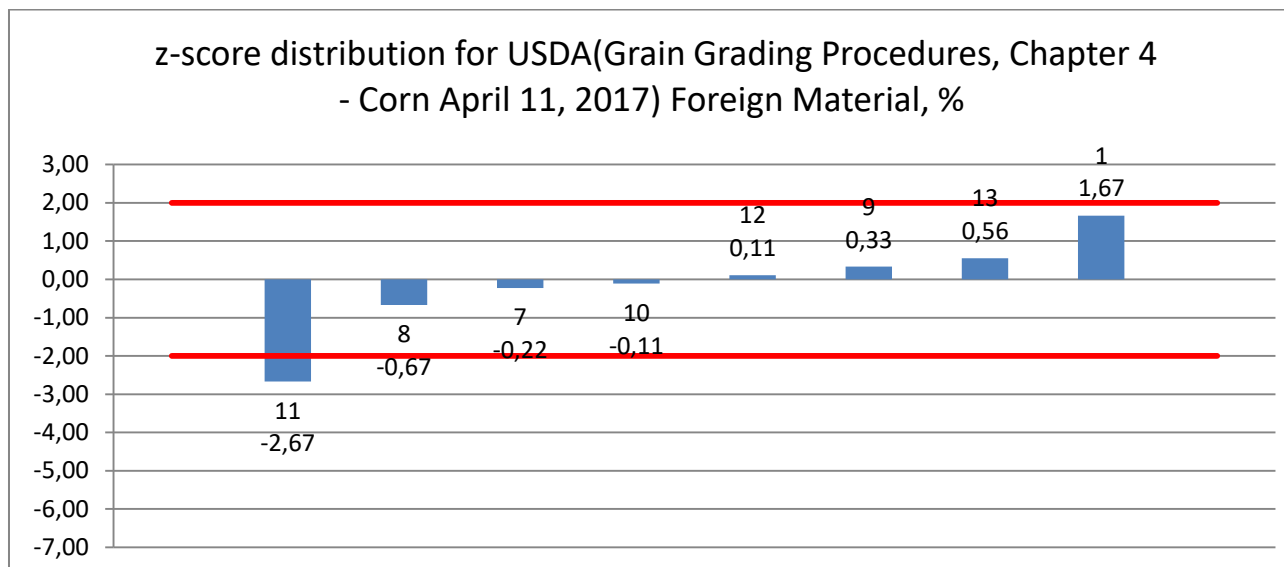
8.8. ISO 6865:2000 Crude fibre content,% (Expressed as a mass fraction of the product as received)



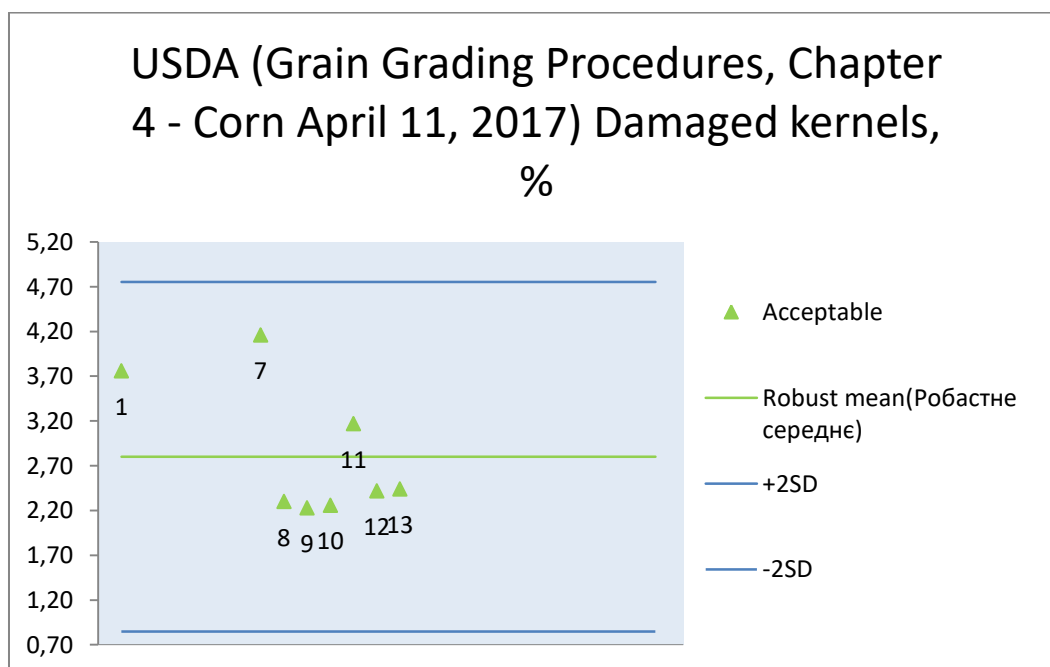
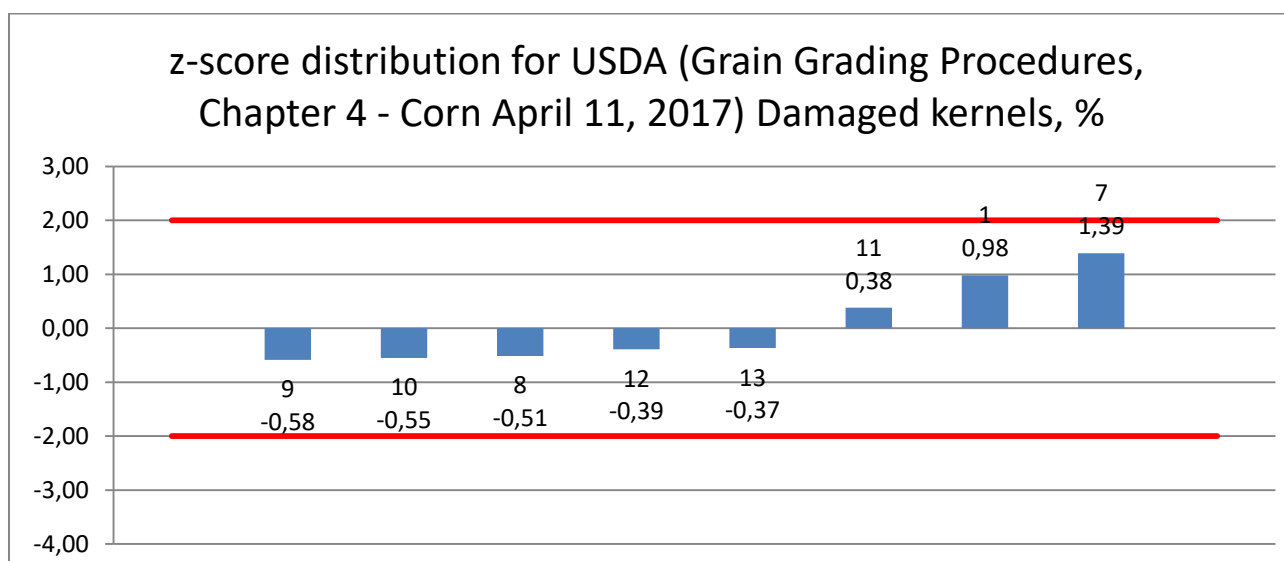
8.9. USDA(Grain Grading Procedures, Chapter 4 - Corn April 11, 2017) Broken Corn, %



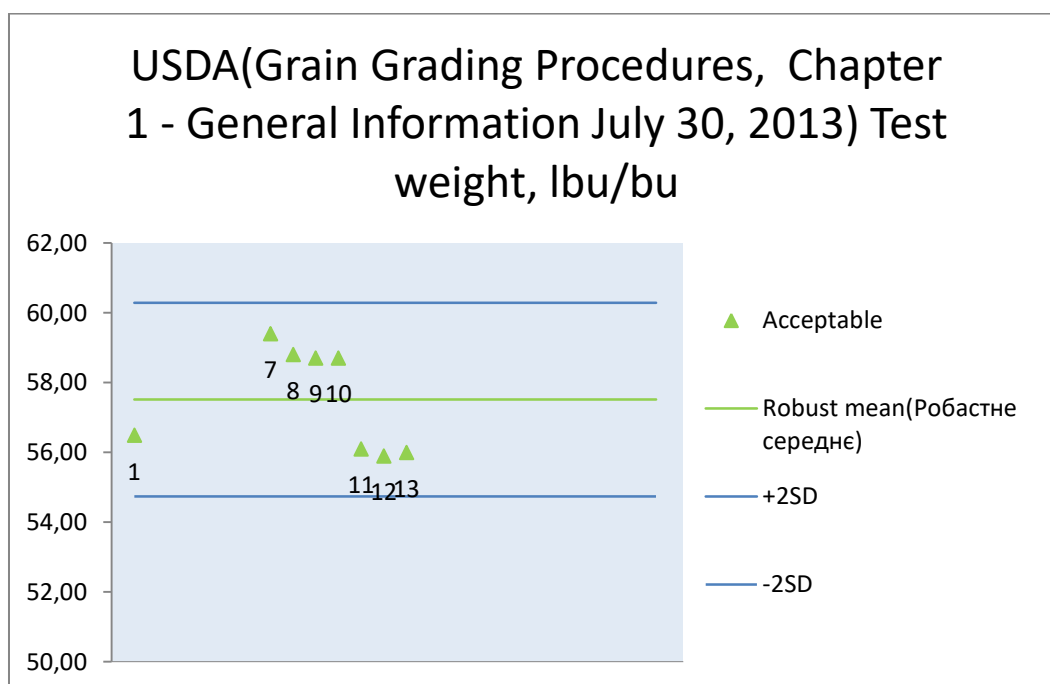
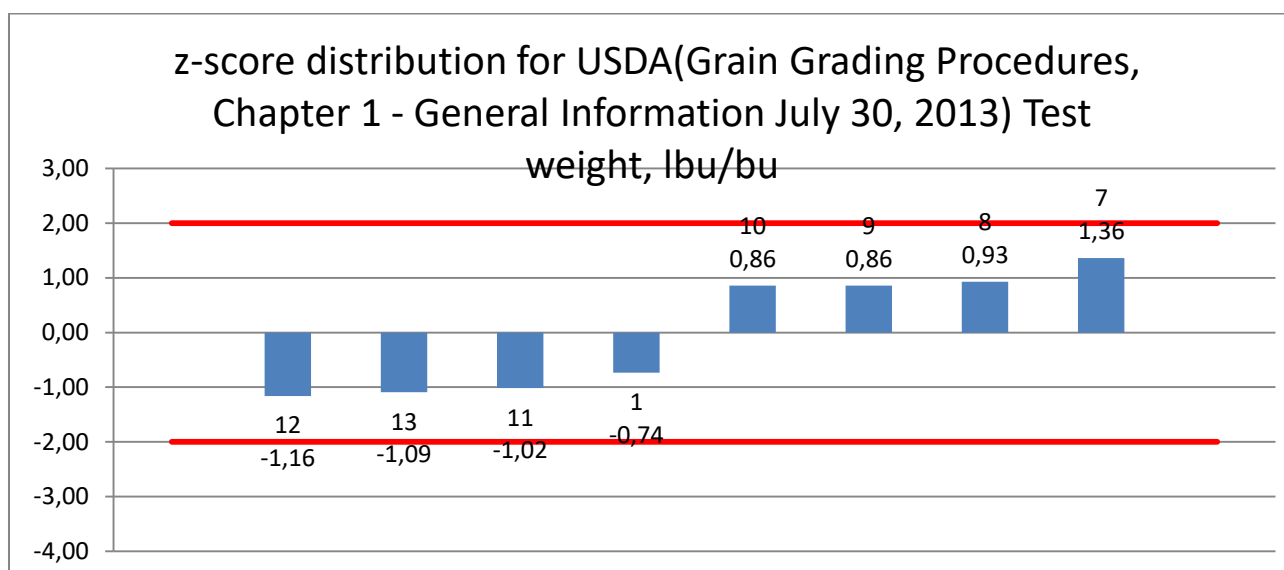
8.10. USDA(Grain Grading Procedures, Chapter 4 - Corn April 11, 2017) Foreign Material, %



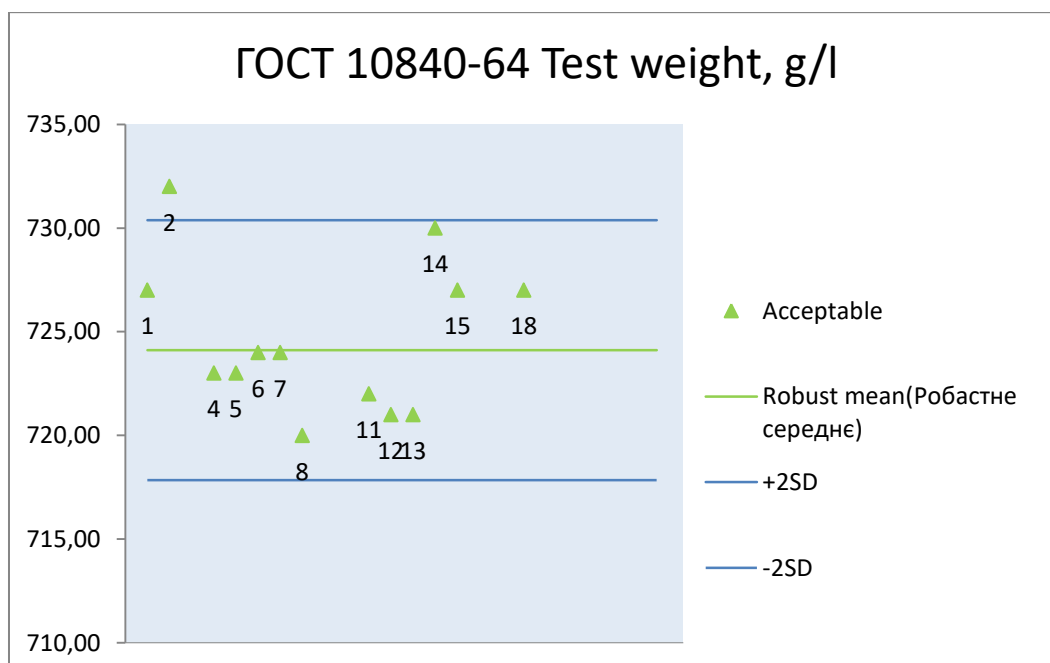
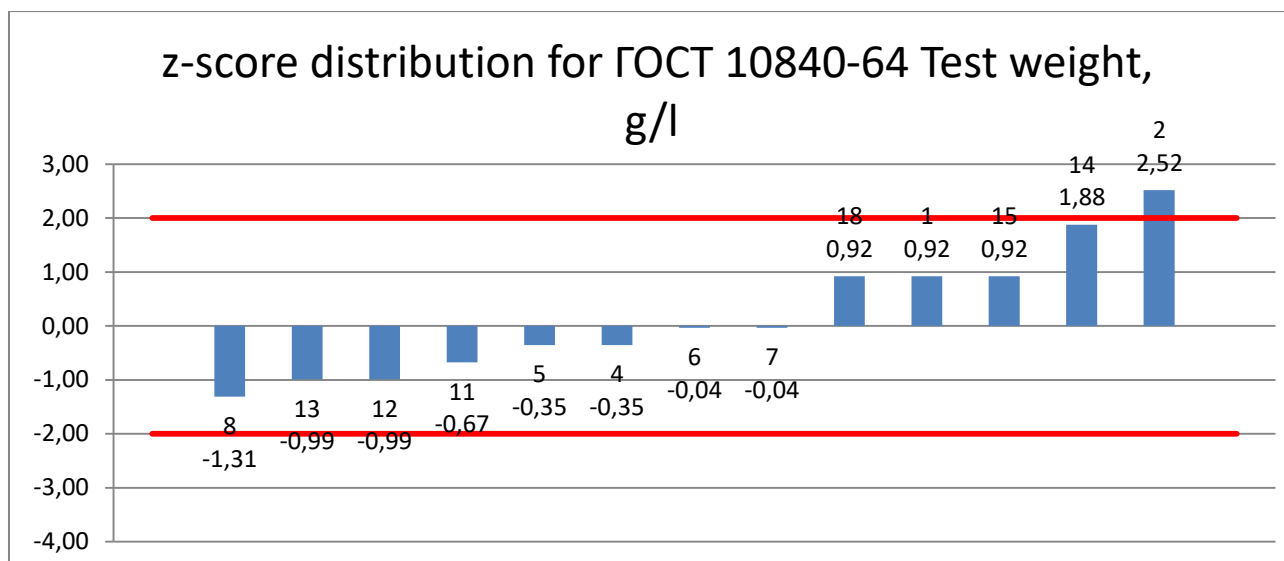
8.11. USDA (Grain Grading Procedures, Chapter 4 - Corn April 11, 2017) Damaged kernels, %



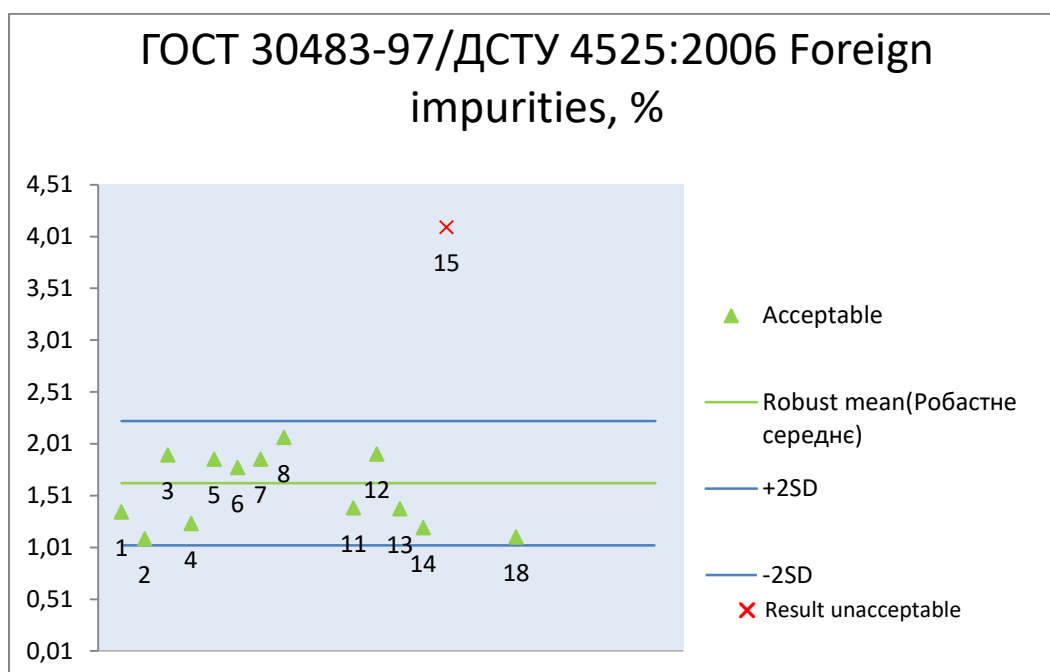
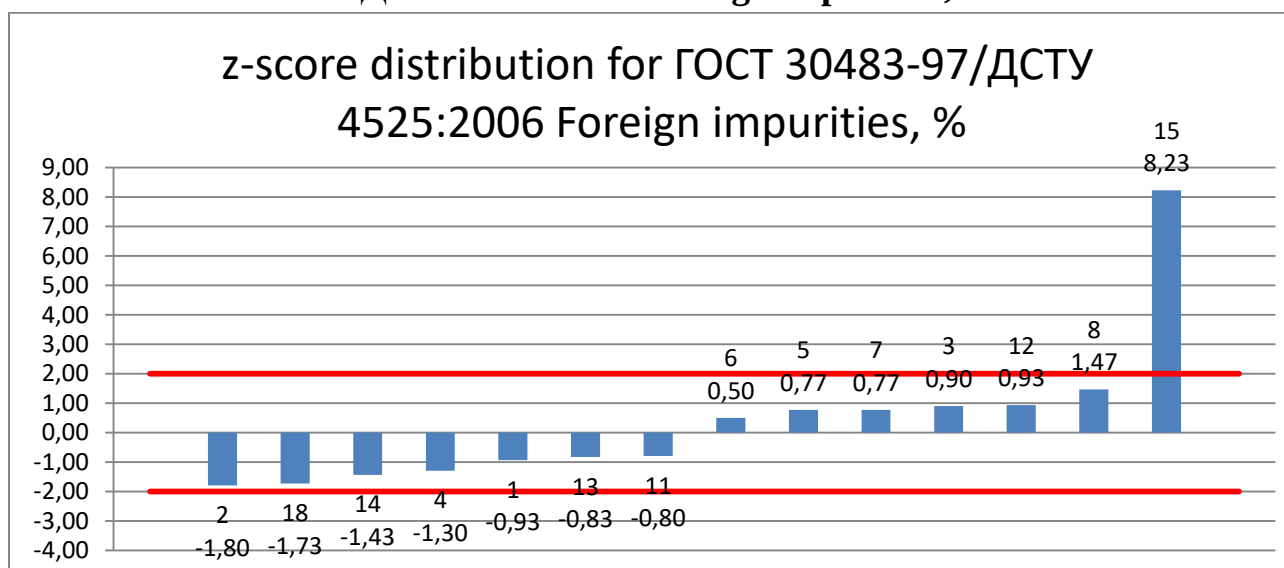
8.12. USDA(Grain Grading Procedures, Chapter 1 - General Information July 30, 2013) Test weight, lbu/bu



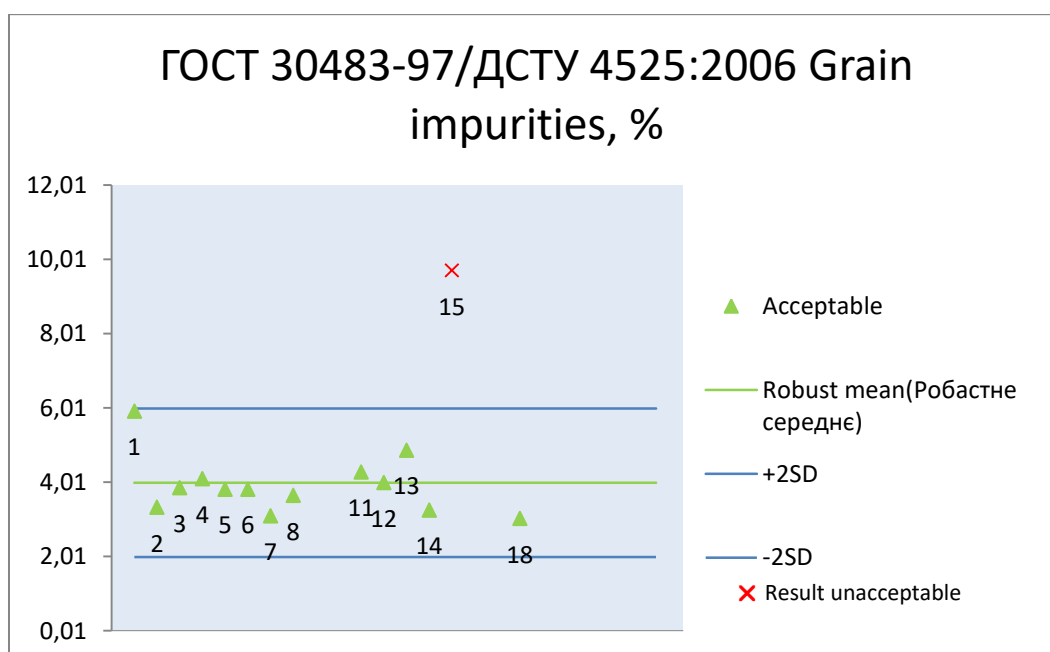
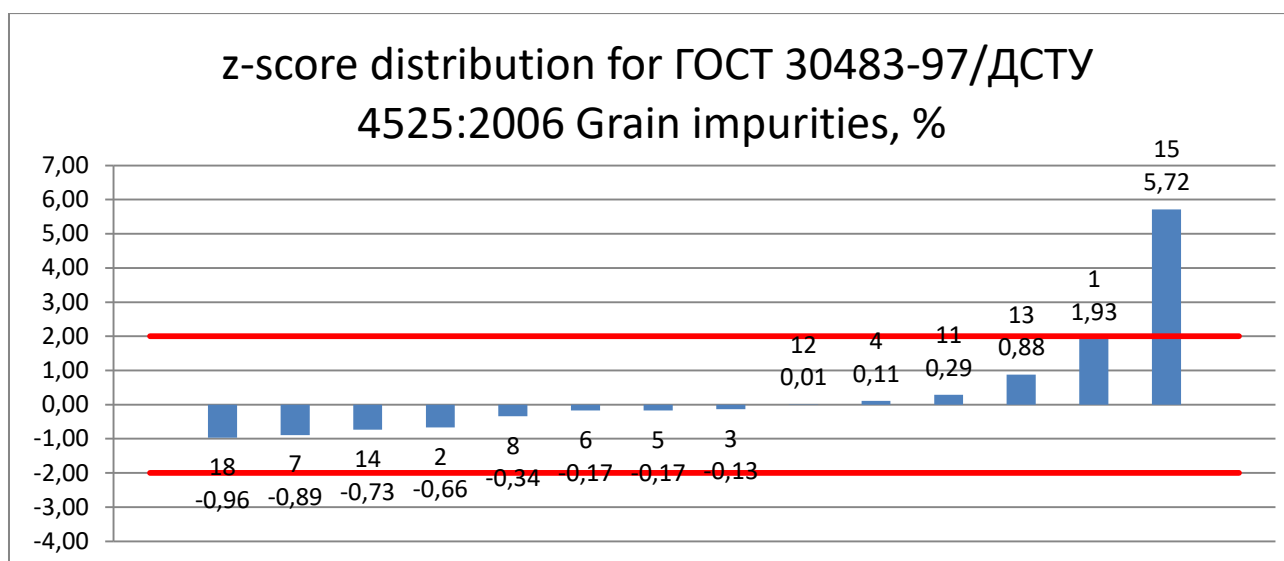
8.13. ГOCT 10840-64 Test weight, g/l



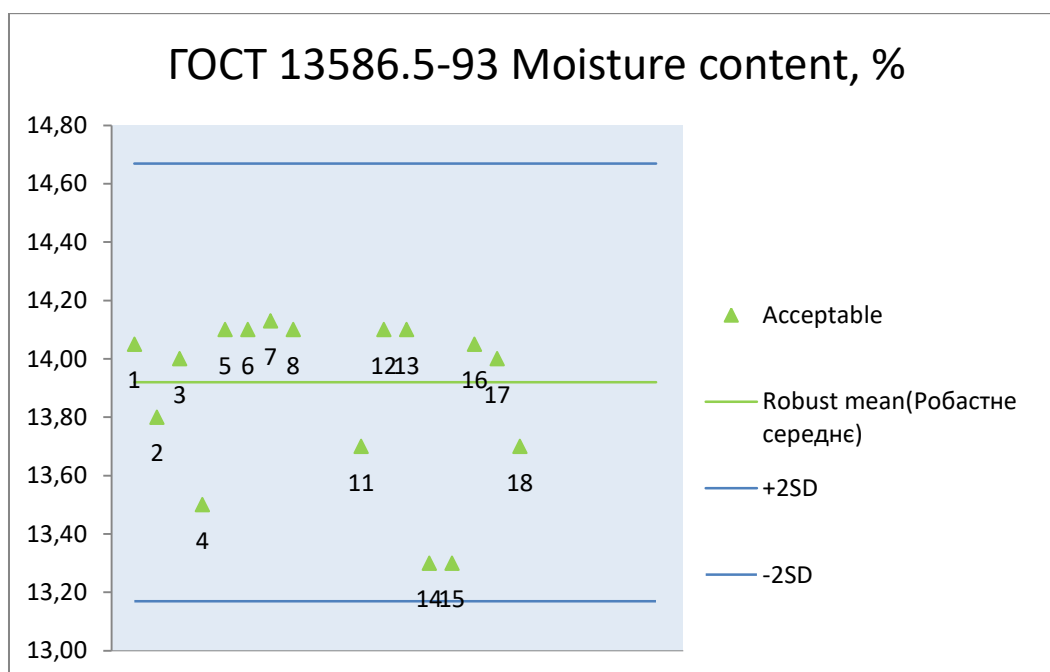
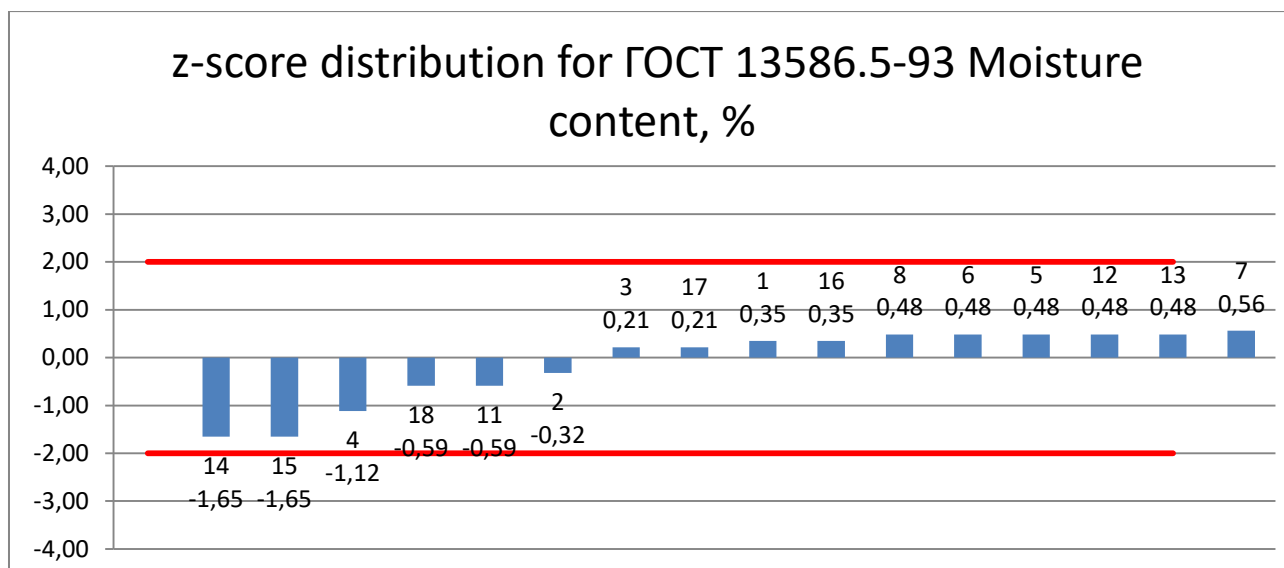
8.14. ГОСТ 30483-97/ДСТУ 4525:2006 Foreign impurities, %



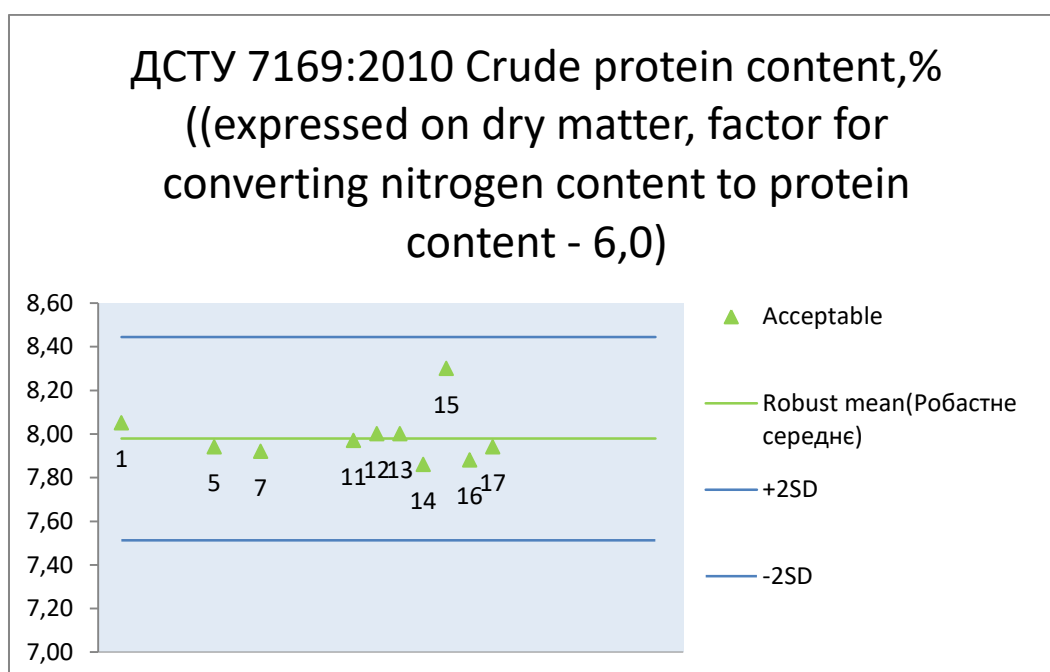
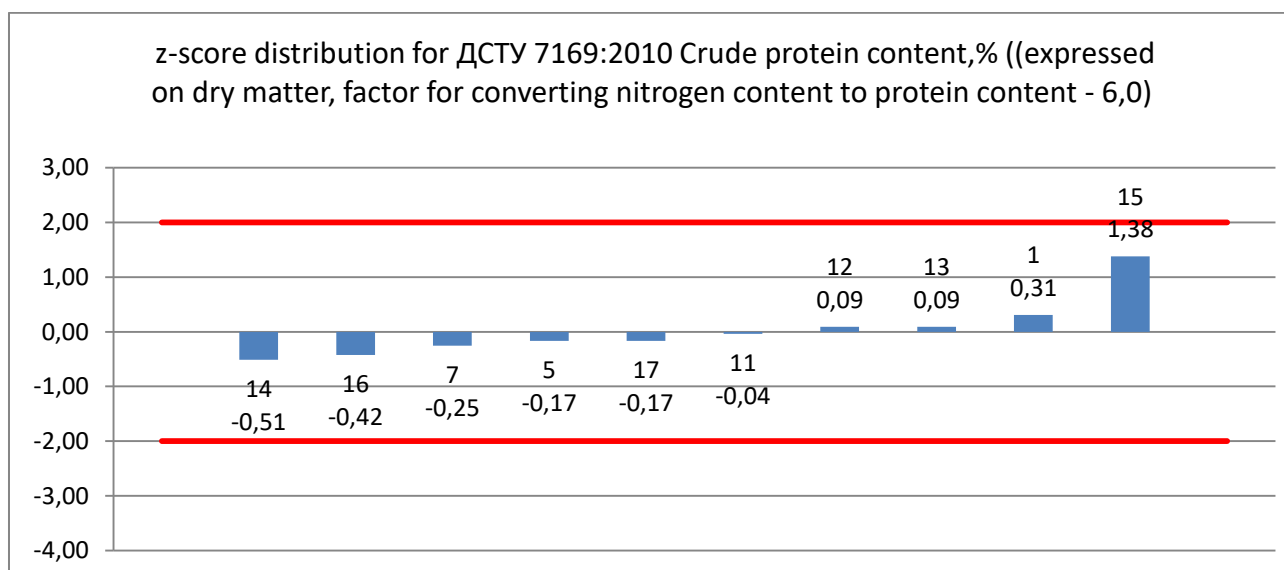
8.15. ГОСТ 30483-97/ДСТУ 4525:2006 Grain impurities, %



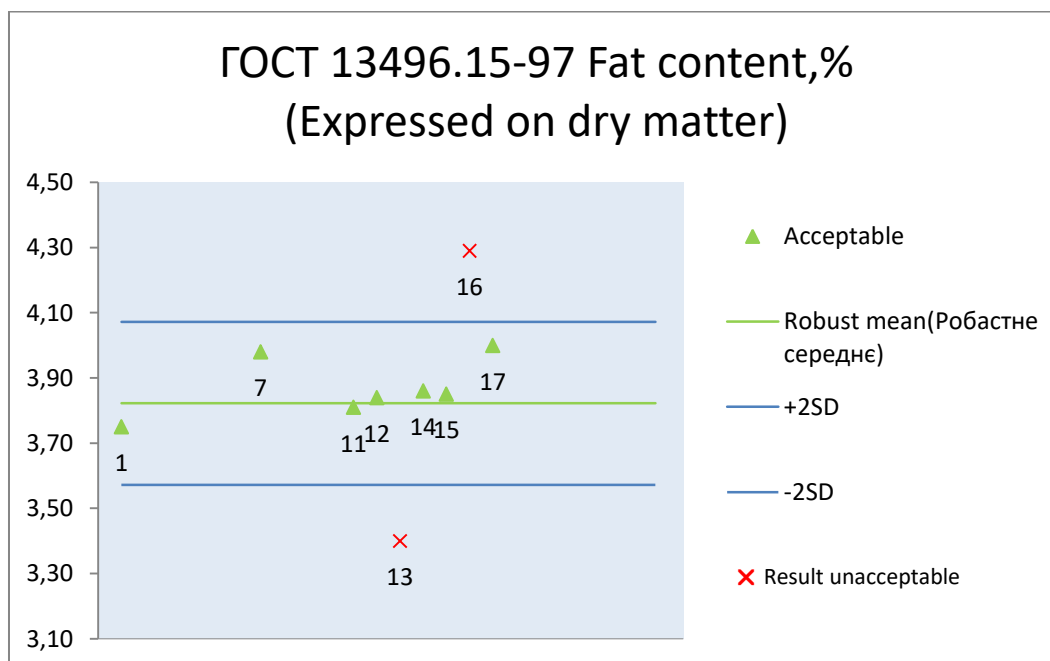
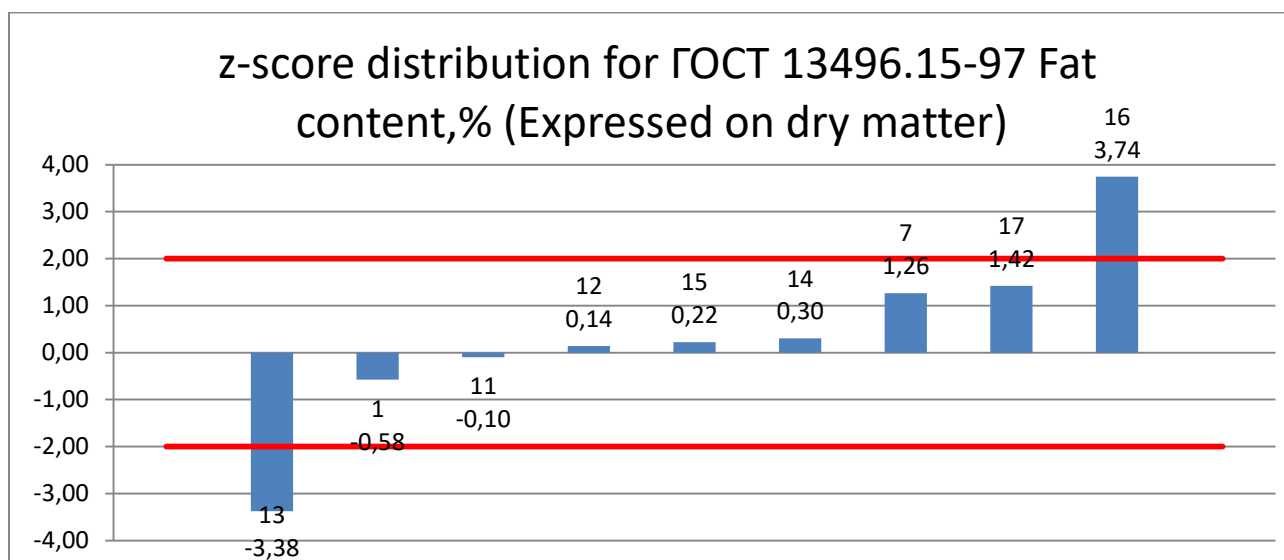
8.16. ГOCT 13586.5-93 Moisture content, %



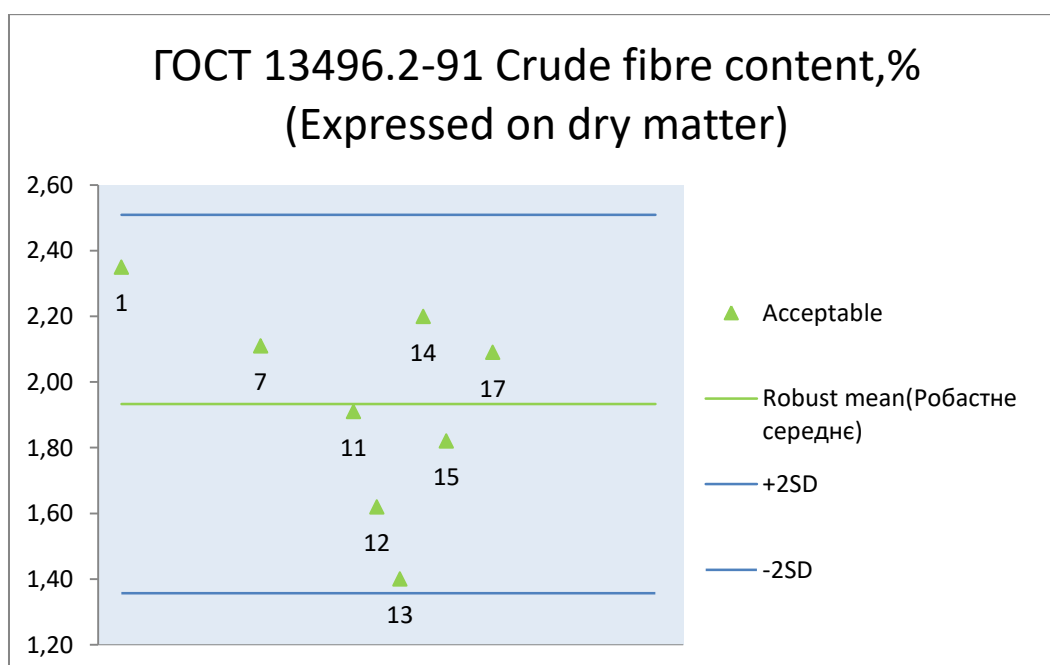
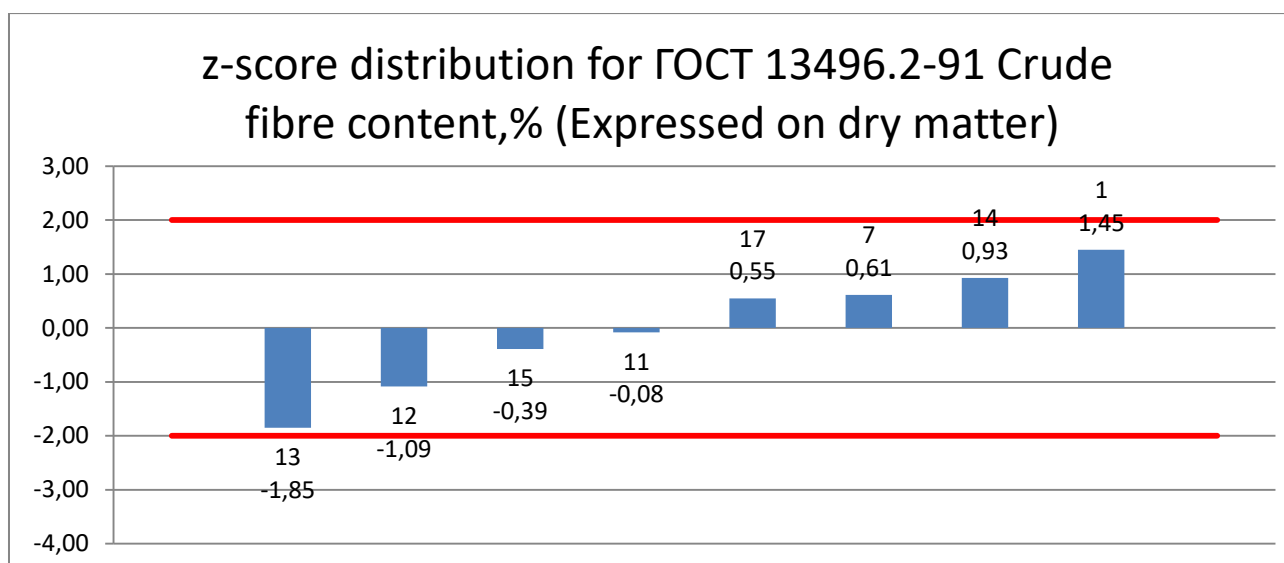
8.17. ДСТУ 7169:2010 Crude protein content,% ((expressed on dry matter, factor for converting nitrogen content to protein content - 6,0)



8.18. ГOCT 13496.15-97 Fat content,% (Expressed on dry matter)



8.19. ГOCT 13586.1-68 Index of gluten deformation



9. STATED ASH BURNING TEMPERATURES (FOR INFORMATION ONLY)

Laboratory number	
1	При визначенні вмісту золи за метом ISO 2171 наважка зразка спалювалась при температурі 900 °C
2	
3	
4	
5	Температура спалювання золи 550°C
6	
7	t = 550C
8	t = 550C
9	Ash yield - 550 ± 10 °C
10	Ash was determinated at temperature 550 oC
11	Температура при визначення золи в муфельній печі 900°C
12	Температура спалювання золи - 500°C
13	Вміст золи при -Т 550*С
14	
15	
16	температура спалювання для золи - 550°C
17	
18	

10. NORMATIVE REFERENCE

1. ISO/IEC 17043:2010 Conformity assessment -- General requirements for proficiency testing
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