AOF Test Check program

Test Report

Round 2 2022.

Summary

1. The test materials for the AOF test check program Round 2 2022 were dispatched in June 2022. Each participant received two canola seed test samples to be analysed for a selection of parameters.
2. An assigned value was determined for each analyte and in conjunction with the standard deviation was used to calculate the z-score for each result.
3. Results for this proficiency test are summarised as follows:

Table 1 Sample 3 - Assigned values and standard deviation

Analyte	Assigned value	Standard deviation	units	No. of participating laboratories
Test weight	66.37	1.09	(kg/hL)	14
Impurities	1.46	0.74	%	14
Oil NIR	43.07	0.67	% by weight	15
Oil solvent	43.48	0.95	% by weight	9
Moisture NIR	7.06	0.34	% by weight	15
Moisture oven	7.26	0.18	% by weight	13
Oleic acid	57.77	0.99	% total fatty acids	7
Linoleic acid	19.93	0.63	% total fatty acids	7
Linolenic acid	13.62	0.19	% total fatty acids	7
Free fatty acid	0.38	0.20	% (as oleic acid)	9

Table 2 Sample 4 - Assigned values and standard deviation

Analyte	Assigned value	Standard deviation	units	No. of participating laboratories
Test weight	65.81	1.05	(kg/hL)	14
Impurities	1.15	0.34	%	14
Oil NIR	44.25	0.75	% by weight	15
Oil solvent	44.13	1.43	% by weight	9
Moisture NIR	6.47	0.34	% by weight	15
Moisture oven	6.62	0.20	% by weight	13
Oleic acid	60.59	0.81	% total fatty acids	7
Linoleic acid	20.37	0.35	% total fatty acids	7
Linolenic acid	10.25	0.20	% total fatty acids	7
Free fatty acid	0.34	0.19	% (as oleic acid)	9

1. Test Material

Preparations for this test check program were sub-contracted to organisations for sample packing and distribution as well as data analysis and reporting.

2. Statistical evaluation of results

The results submitted by participants were statistically analysed in order to provide an assigned value for each analyte. The assigned values were then used in combination with the standard deviation to calculate a Z-score for each result.

Raw data was analysed using Grubbs' test to determine any outliers. Outliers (Z-score >2) were removed and the remaining samples were used to calculate the assigned value (mean) and standard deviation results.

Participants Z-scores were calculated as:

$$Z = \frac{(participants\ result - assigned\ value)}{standard\ deviation}$$

3. Results and Z-scores

**Table 3** Results and Z-scores for test weight.

	Test weight (kg/hL)			
	Sample 3		**Sample 4**	
Lab number	**Result**	**Z-score**	**Result**	**Z-score**
P01	65.85	-0.48	64.45	-1.29
P02				
P03	63.50	-2.63	67.60	1.70
P04	67.24	0.80	65.12	-0.66
P05	68.00	1.49	66.25	0.42
P06	64.88	-1.36	64.94	-0.83
P07	65.95	-0.39	64.78	-0.98
P08	65.70	-0.61	66.70	0.85
P09	70.70	3.96	68.48	2.54
P10				
P11	67.50	1.03	66.00	0.18
P12	66.25	-0.11	65.10	-0.67
P13				
P14				
P15	68.22	1.69	67.78	1.88
P16	65.25	-1.03	65.65	-0.15
P17	65.92	-0.41	65.15	-0.63
P18	65.70	-0.61	66.00	0.18
Assigned value	66.37		65.81	
Standard Deviation	1.09		1.05	
Count	14		14	

Note - Laboratory numbers P03 and P09 Sample 3 were removed from assigned value calculation as the results were outliers

Note - Laboratory number P09 Sample 4 was removed from assigned value calculation as the result was an outlier.

Figure 1 Z-scores for test weight.
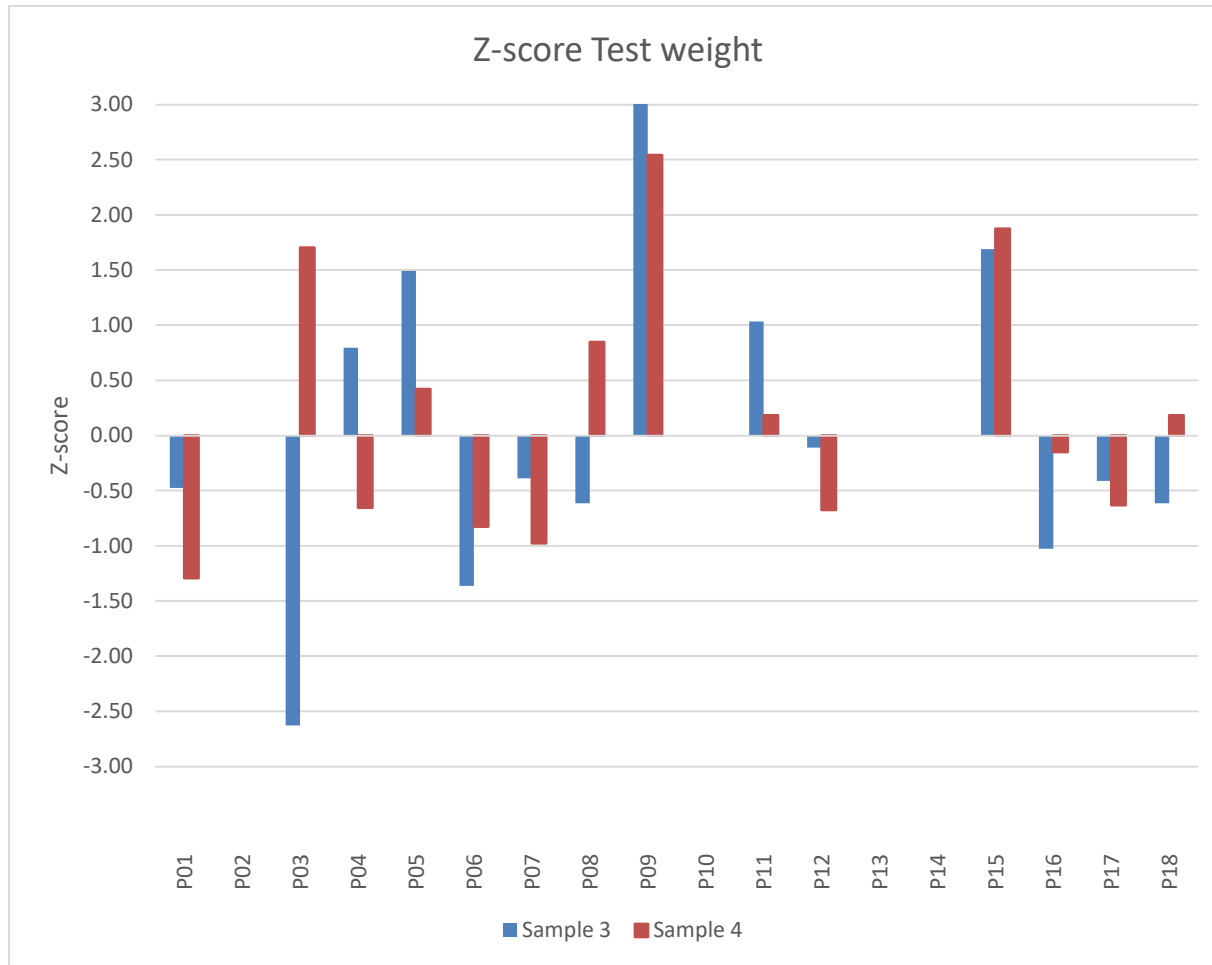
Table 4 Results and Z-scores for impurities.

	Impurities (%)			
	Sample 3		Sample 4	
Lab number	Result	Z-score	Result	Z-score
P01	0.80	-0.89	1.10	-0.16
P02				
P03	2.68	1.64	0.79	-1.08
P04	1.28	-0.25	1.54	1.14
P05	1.30	-0.22	2.75	4.69
P06	2.91	1.95	1.74	1.71
P07	2.01	0.73	1.52	1.06
P08	1.30	-0.22	0.70	-1.33
P09	1.10	-0.49	2.10	2.78
P10				
P11	1.15	-0.42	1.25	0.29
P12	1.05	-0.55	1.28	0.37
P13				
P14				
P15	0.22	-1.67	0.71	-1.32
P16	2.15	0.92	1.27	0.33
P17	0.88	-0.78	1.06	-0.27
P18	1.65	0.25	0.90	-0.74
Assigned value	1.46		1.15	
Standard Deviation	0.74		0.34	
Count	14		14	

Note - Laboratory numbers P05 and P09 Sample 4 were removed from assigned value calculation as the results were outliers

Figure 2 Z-scores for impurities.

Z-score Impurities

Table 5 Results and Z-scores for oil content (NIR).

	Oil content NIR (%)			
	Sample 3		Sample 4	
Lab number	Result	Z-score	Result	Z-score
P01	42.00	-1.61	43.08	-1.57
P02	43.52	0.68	45.47	1.63
P03	42.75	-0.48	44.10	-0.20
P04	42.94	-0.20	43.99	-0.35
P05	43.45	0.57	43.00	-1.67
P06	44.26	1.79	45.63	1.85
P07	42.36	-1.07	43.90	-0.47
P08	43.75	1.02	44.20	-0.06
P09	42.75	-0.48	44.80	0.74
P10				
P11	42.61	-0.69	43.73	-0.69
P12	44.16	1.64	44.94	0.93
P13				
P14				
P15	42.50	-0.86	44.05	-0.27
P16	42.75	-0.48	43.95	-0.40
P17	42.75	-0.48	44.40	0.20
P18	43.50	0.65	44.50	0.18
Assigned value	43.07		44.25	
Standard Deviation	0.67		0.75	
Count	15		15	

Figure 3 Z-scores for oil content by NIR.
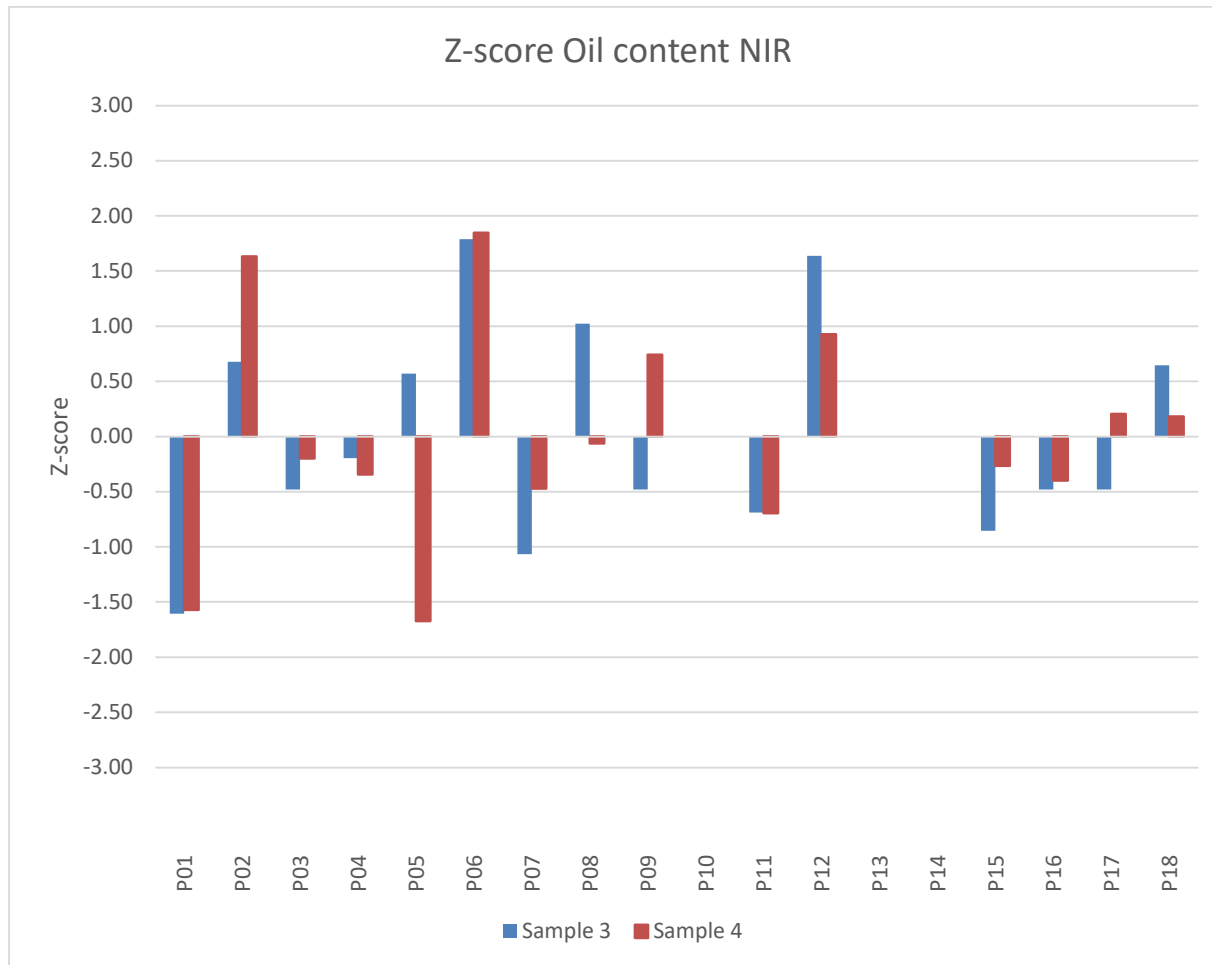
Table 6 Results and Z-scores for oil content solvent.

|	Oil content solvent (%)				
	Sample 3			Sample 4	
Lab number	Result	Z-score		Result	Z-score
P01					
P02					
P03	43.99	0.53		44.79	0.46
P04					
P05	44.34	0.91		42.71	-0.99
P06	44.59	1.16		45.86	1.21
P07					
P08					
P09	42.25	-1.29		43.87	-0.18
P10					
P11					
P12	44.46	1.03		45.14	0.71
P13					
P14					
P15	43.70	0.23		44.57	0.31
P16	42.98	-0.53		44.70	0.40
P17	40.20	-3.46		41.48	-1.84
P18	42.40	-1.14		42.60	-1.06
Assigned value	43.48			44.13	
Standard Deviation	0.95			1.43	
Count	9			9	

Note - Laboratory number P17 Sample 3 was removed from assigned value calculation as the result was an outlier.

Note - Laboratory number P05 results were excluded from assigned value calculations. Despite the average value being within the statistically acceptable range, the raw data duplicate values were significantly different from each other. Discussions with the laboratory confirmed the raw data values were recorded correctly.

**Figure 4** Z-scores for oil content by solvent extraction.

Z-score Oil content solvent
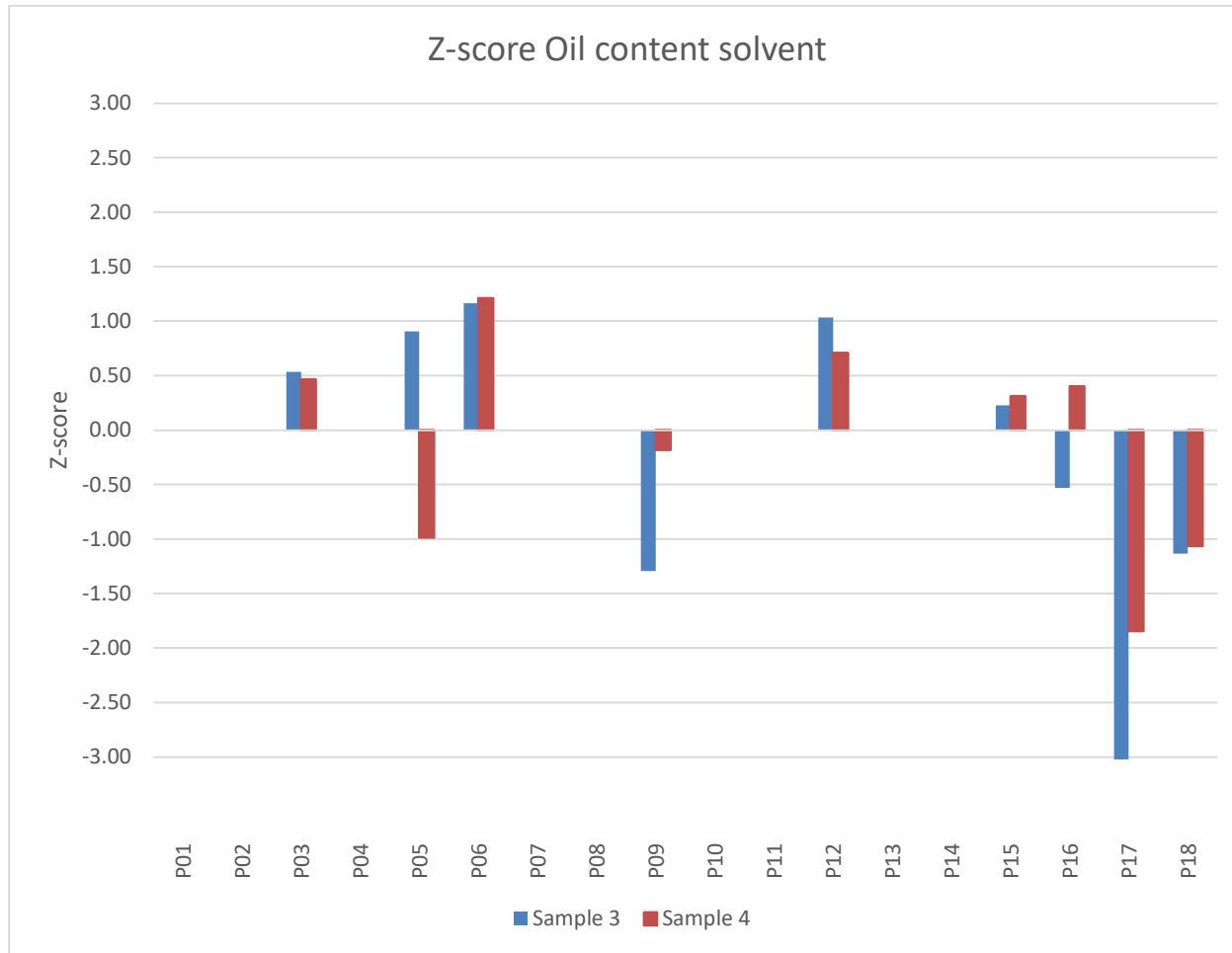
Figure 5 Absolute difference between oil content (NIR result minus wet chemistry)

NIR minus wet chemistry (% Oil content)
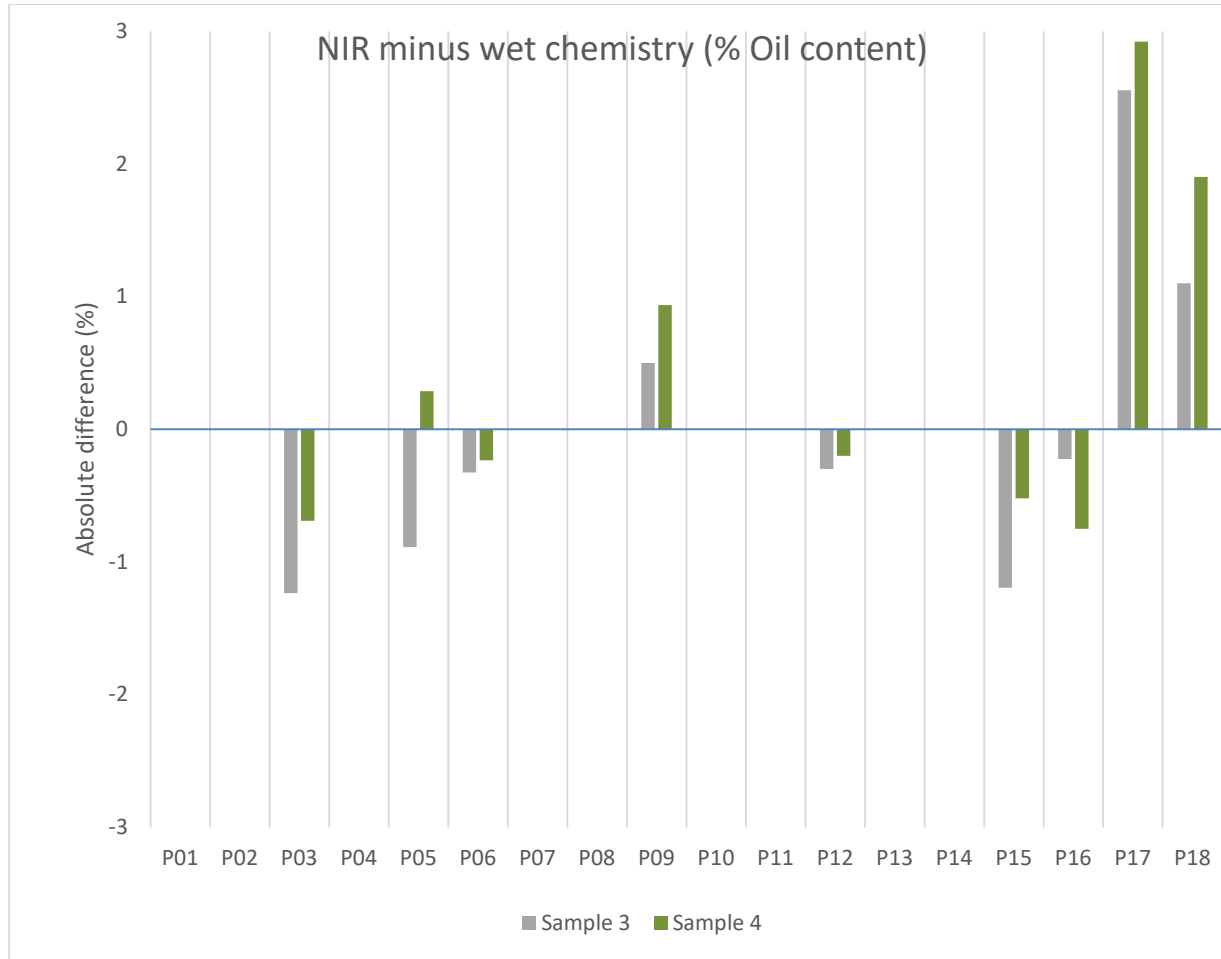
Table 7 Results and Z-scores for moisture content (NIR).

	Moisture NIR (% by weight)			
	Sample 3		Sample 4	
Lab number	Result	Z-score	Result	Z-score
P01	7.65	1.71	7.01	1.60
P02	6.85	-0.62	6.30	-0.50
P03	6.95	-0.33	6.20	-0.80
P04	7.21	0.43	6.60	0.40
P05	6.65	-1.20	7.05	1.74
P06	6.71	-1.03	6.06	-1.22
P07	7.45	1.13	6.87	1.19
P08	6.80	-0.77	5.95	-1.54
P09	6.95	-0.33	6.20	-0.80
P10				
P11	7.28	0.63	6.49	0.07
P12	6.40	-1.93	6.19	-0.83
P13				
P14				
P15	7.45	1.13	6.75	0.84
P16	7.15	0.25	6.40	-0.20
P17	7.20	0.40	6.45	-0.05
P18	7.25	0.54	6.50	0.10
Assigned value	7.06		6.47	
Standard Deviation	0.34		0.34	
Count	15		15	

Figure 6 Z-scores for moisture content by NIR.
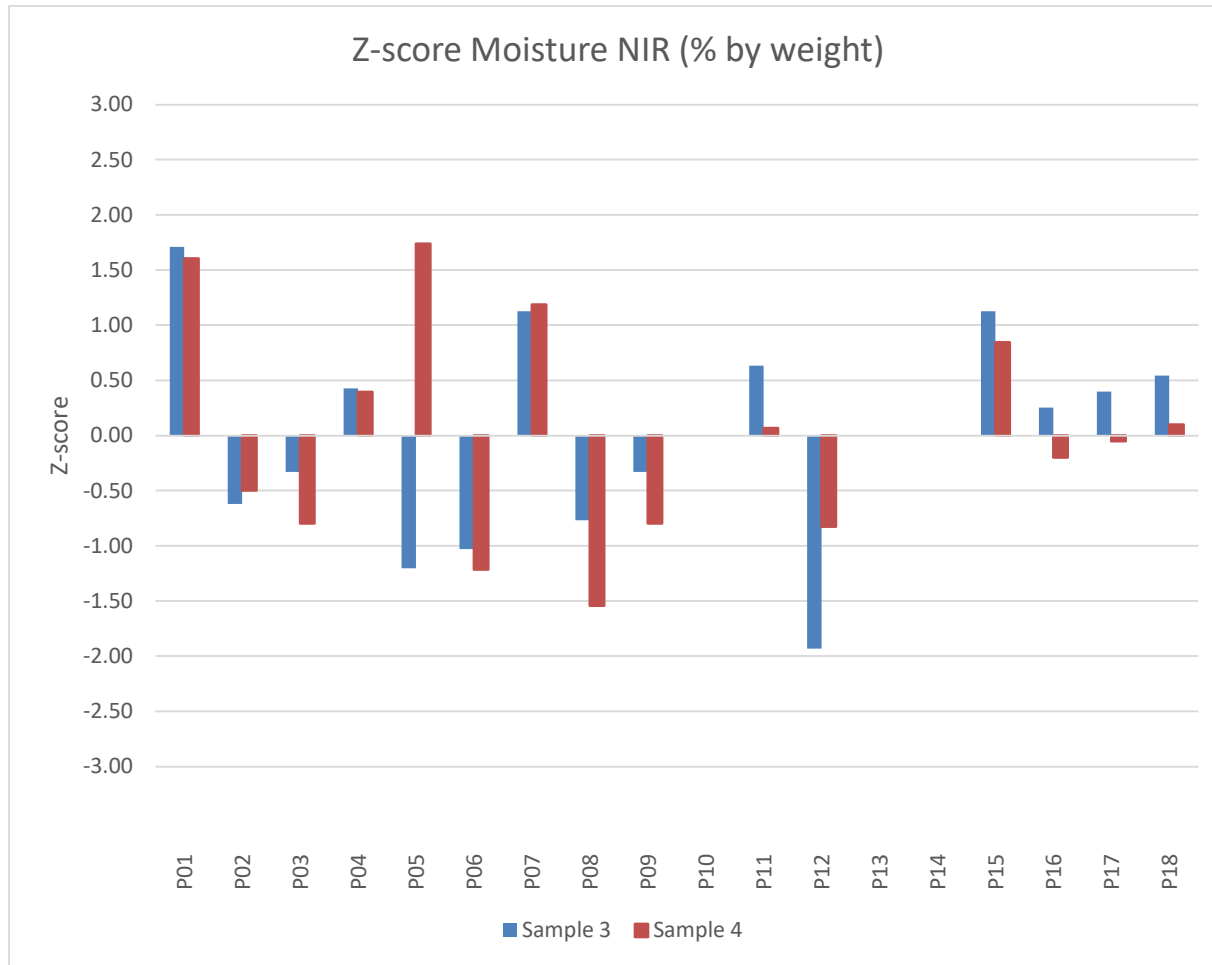
Table 8 Results and Z-scores for moisture content by oven.

| Lab number | Moisture Oven (% by weight) | | | | |
| | Sample 3 | | Sample 4 | |
	Result	Z-score	Result	Z-score
P01	7.25	-0.08	6.60	-0.11
P02				
P03	7.48	1.19	6.73	0.52
P04	7.28	0.09	6.66	0.19
P05	6.41	-4.73	6.98	1.81
P06	6.95	-1.76	6.27	-1.81
P07	7.23	-0.21	6.47	-0.77
P08				
P09	7.37	0.56	6.64	0.09
P10				
P11	7.52	1.39	6.85	1.15
P12	6.32	-5.23	6.05	-2.93
P13				
P14				
P15	7.18	-0.46	6.49	-0.67
P16	7.44	0.97	6.77	0.75
P17	7.21		6.61	
P18	7.01	-1.40	6.41	-1.08
Assigned value	7.26		6.62	
Standard Deviation	0.18		0.20	
Count	13		13	

Note - Laboratory number P05 and P12 Sample 3 were removed from assigned value calculation as the results were outliers.
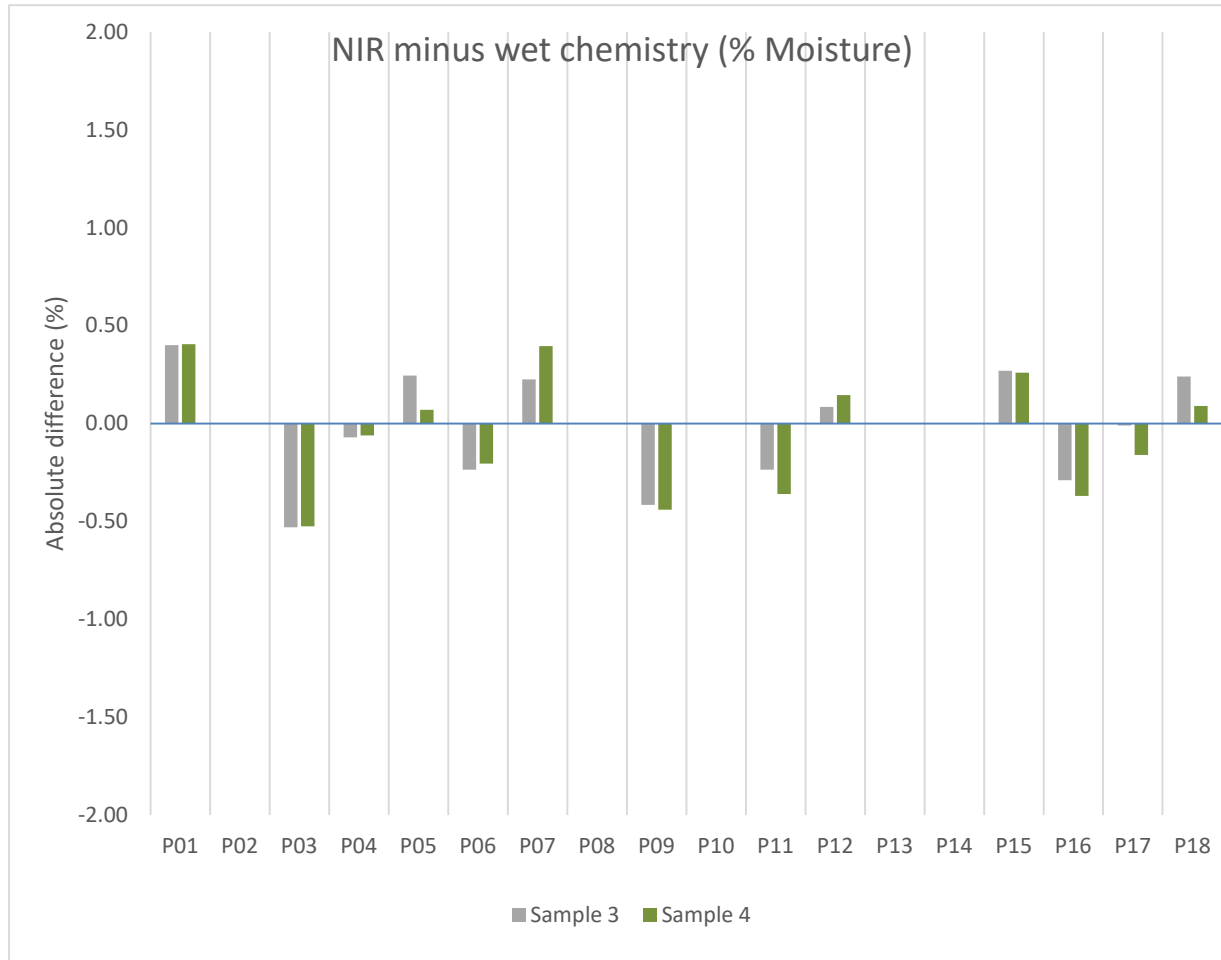**Note** - Laboratory number P12 Sample 4 was removed from assigned value calculation as the result was an outlier.

Figure 7 Z-scores for moisture content by oven.

Z-score Moisture oven (% by weight)
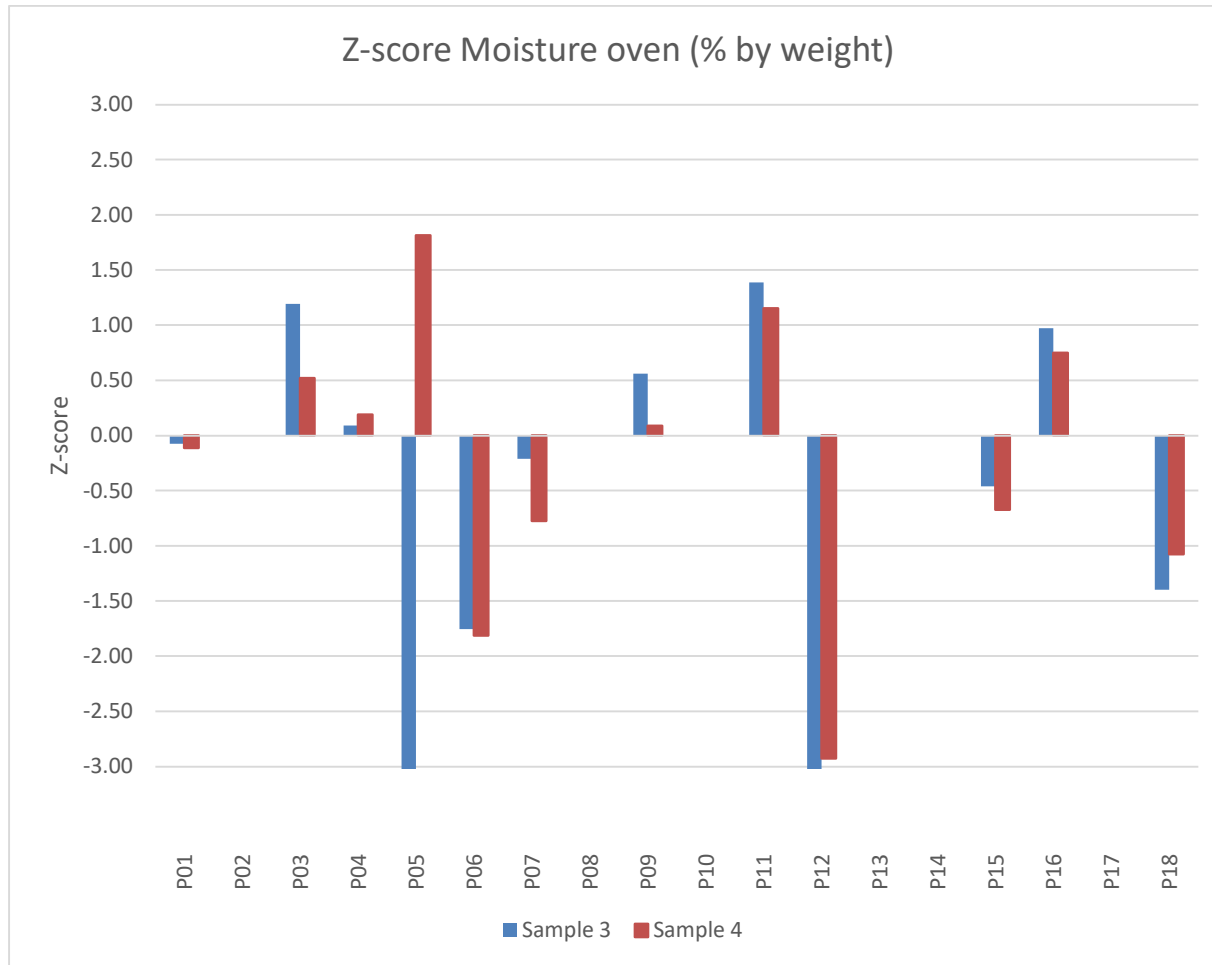
Figure 8 Absolute difference between moisture content (NIR result minus wet chemistry)

Table 9 Results and Z-scores for oleic acid.

Lab number	Sample 3 Result	Sample 3 Z-score	Sample 4 Result	Sample 4 Z-score
P01				
P02				
P03	56.43	-1.36	59.42	-1.44
P04				
P05	62.57	4.87	60.40	-0.24
P06	57.25	-0.52	60.12	-0.58
P07				
P08				
P09	59.10	1.35	61.81	1.50
P10				
P11				
P12	57.82	0.05	60.53	-0.08
P13				
P14				
P15				
P16	57.33	-0.45	60.40	-0.24
P17	58.68	0.92	61.48	1.09
P01				
Assigned value	57.77		60.59	
Standard Deviation	0.99		0.81	
Count	7		7	

**Note** - Laboratory number P05 Sample 3 was removed from assigned value calculation as the result was an outlier.

Figure 9 Z-scores for oleic acid content.

Z-score Oleic acid (% total fatty acids)

Table 10 Results and Z-scores for linoleic acid.

| | Linoleic acid (% of total fatty acids) | | | |
| | Sample 3 | | Sample 4 | |
Lab number	Result	Z-score	Result	Z-score
P01				
P02				
P03	19.88	-0.09	20.49	0.35
P04				
P05	20.81	1.38	19.90	-1.34
P06	19.85	-0.14	20.44	0.22
P07				
P08				
P09	19.38	-0.88	20.32	-0.15
P10				
P11				
P12	19.00	-1.48	19.93	-1.27
P13				
P14				
P15				
P16	20.05	0.17	20.74	1.08
P17	20.59	1.04	20.75	1.11
P01				
Assigned value	19.93		20.37	
Standard Deviation	0.63		0.35	
Count	7		7	

Figure 10 Z-scores for linoleic acid content.

Z-score Linoleic acid (% total fatty acids)
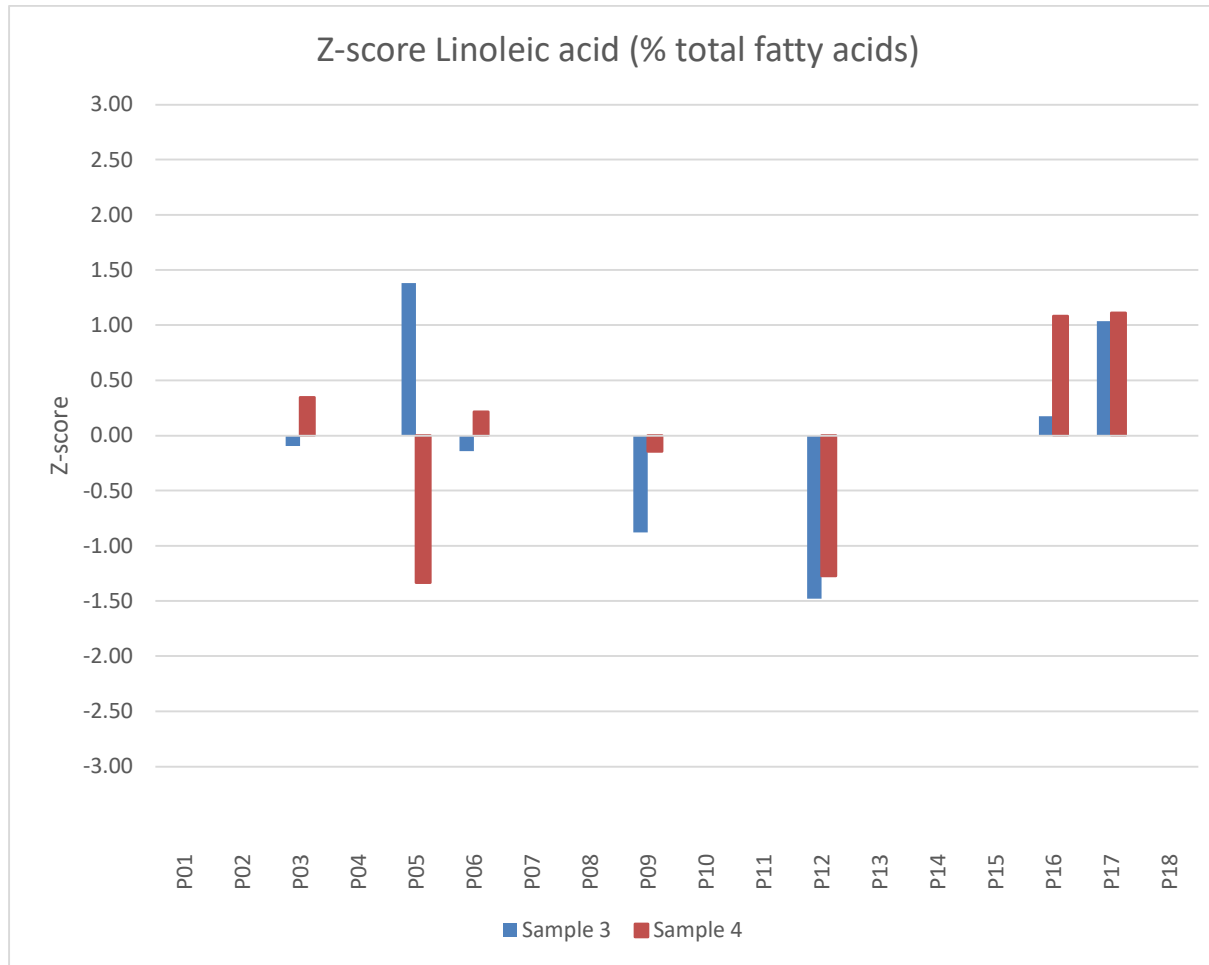
Table 11 Results and Z-scores for linolenic acid.

| Lab number | Linolenic acid (% of total fatty acids) | | | | |
| | Sample 3 | | Sample 4 | |
	Result	Z-score	Result	Z-score
P01				
P02				
P03	13.41	-1.13	10.08	-0.87
P04				
P05	9.07	-23.57	12.00	8.94
P06	13.85	1.14	10.58	1.68
P07				
P08				
P09	13.59	-0.18	10.09	-0.81
P10				
P11				
P12	13.57	-0.28	10.14	-0.56
P13				
P14				
P15				
P16	13.87	1.27	10.38	0.66
P17	13.47	-0.82	10.23	-0.10
P18				
Assigned value	13.62		10.25	
Standard Deviation	0.19		0.20	
Count	7		7	

Note - Laboratory number P05 Sample 3 was removed from assigned value calculation as the result was an outlier.

Note - Laboratory number P05 Sample 4 was removed from assigned value calculation as the result was an outlier.

Figure 11 Z-scores for linolenic acid content.
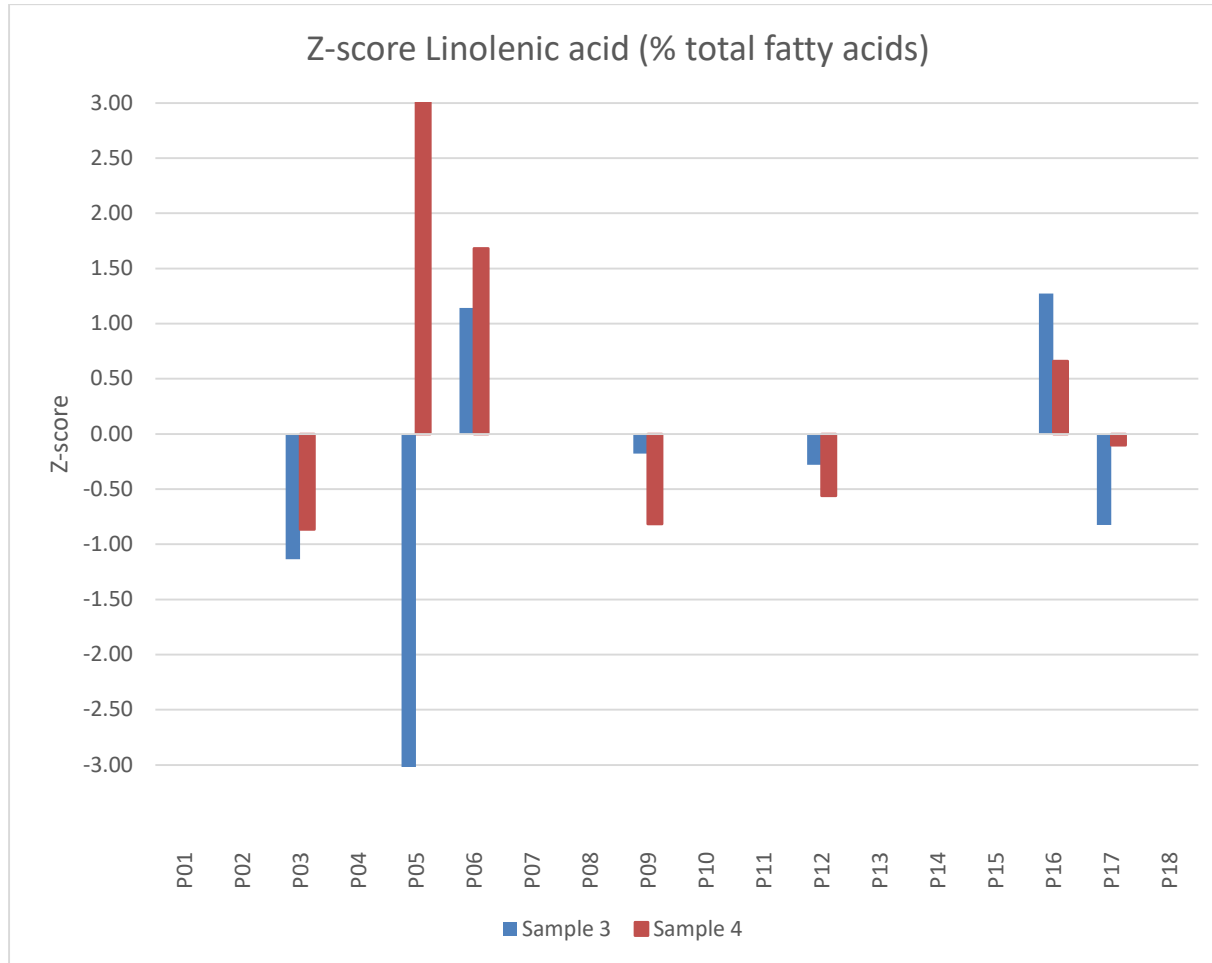
Table 12 Results and Z-scores for free fatty acids.

	Free fatty acid (% as oleic acid)			
	Sample 3		Sample 4	
Lab number	Result	Z-score	Result	Z-score
P01	0.43	0.27	0.37	0.10
P02				
P03	0.72	1.72	0.52	0.92
P04				
P05	0.20	-0.88	0.45	0.53
P06	0.20	-0.88	0.10	-1.27
P07				
P08				
P09	0.34	-0.18	0.68	1.74
P10				
P11				
P12	0.19	-0.95	0.12	-1.19
P13				
P14				
P15				
P16	0.47	0.47	0.27	-0.39
P17	0.21	-0.83	0.20	-0.75
P18	0.63	1.25	0.41	0.31
Assigned value	0.38		0.34	
Standard Deviation	0.20		0.19	
Count	9		9	

Figure 12 Z-scores for free fatty acid content.

Z-score Free fatty acid (% as oleic acid)
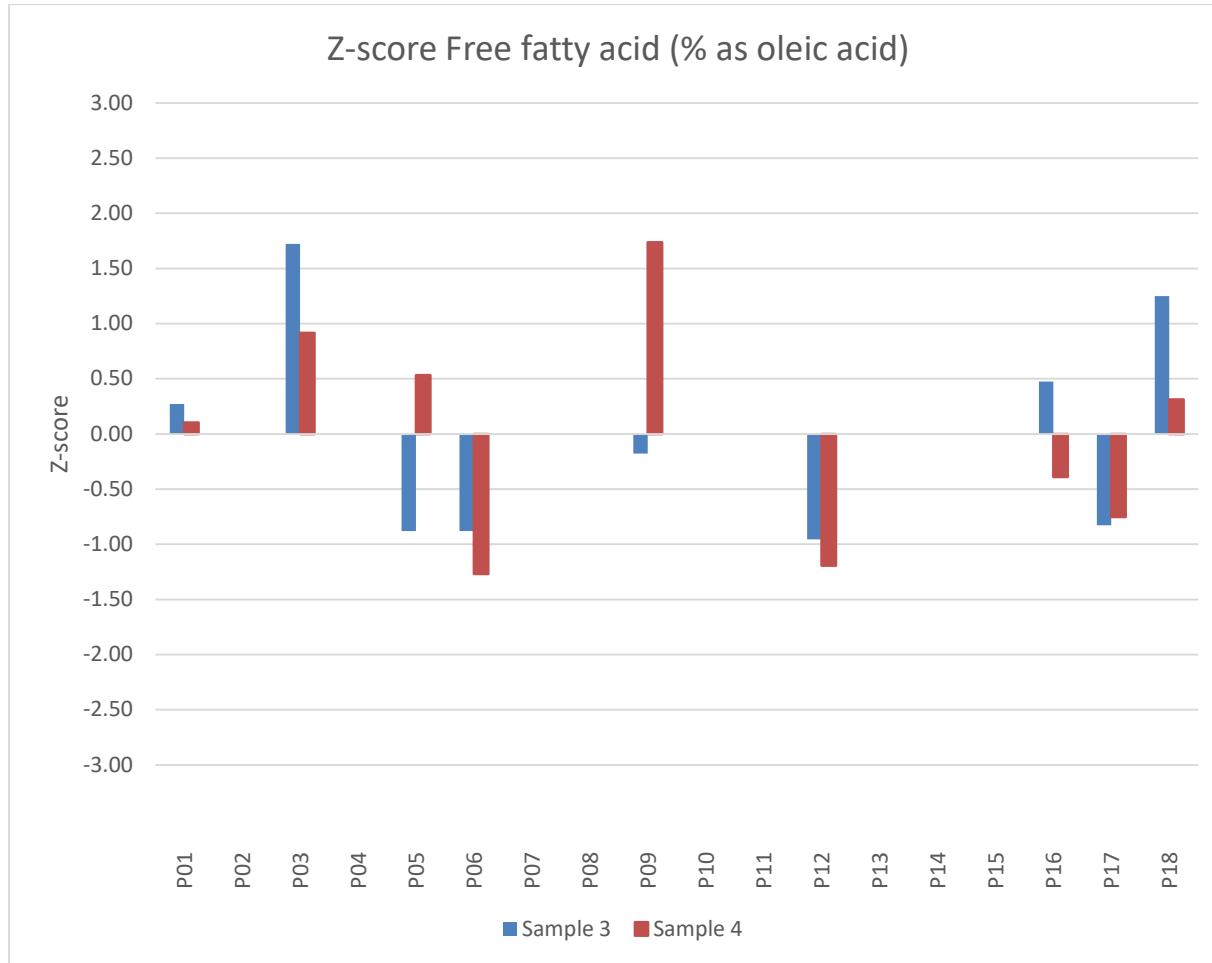
Appendix

Analytical methods used

Participating laboratories were asked to indicate which analytical methods were used for each determination. Information is summarised below (number of laboratories using method in brackets):

<u>Test weight</u>

Chrondrometer (4), half litre measure (2), Test weight cup (1), not indicated (5), MS55-measurement of grain density by CBH chrondrometer (1), GAFTA 25.0.

<u>Impurities</u>

AOF 4-1.2(b)(2), AOF 4-1.3 (6), not indicated (4), ISO658 (2).

<u>Oil content (NIR)</u>

Calibration based on ISO659 (1), NIR (2), FOSS NIR (1), Infratec 1241 (1), ISO 10565 (NMR) (1), not indicated (9).

<u>Oil content (solvent)</u>

ISO659:2009 (4), extract for 4,2,2 hours with regrind in between (1), AOF 4-1.24a (2), Not indicated (2).

<u>Moisture (NIR)</u>

Calibration based on ISO665 (1), FOSS NIR (1), NIR (2), Infratec 1241 (1), NMR (1), not indicated (9).

<u>Moisture (oven)</u>

AOF 4-1.5 (130°C for 1 hour) (6), ISO665 (103°C for 3 hours, then 1 hour, 5g) (2), 105°C for 2 hours (1), ISO662 (1) not indicated (3).

<u>Fatty acids (oleic, linoleic and linolenic acid)</u>

IOC doc no. 24 (1), GC (1), AOCS Ce 1h-05 (1), ISO588 (1), not indicated (3).

<u>Free fatty acids</u>

AOCS Ac 5-41 (3), AOCS Ca 5a-40 (1), ISO660 (2), not indicated (3).