Results of Proficiency Test
Virgin Olive Oil
May 2002

CONTENTS

Appendices:

1   INTRODUCTION

During the meeting of the i.i.s. advisory board in Lisbon (Portugal) in September 2001 it was decided to organise a proficiency test for the analysis of Virgin Olive Oil during the annual proficiency test program of 2001/2002. In this international interlaboratory study 18 laboratories in 11 different countries have participated. See appendix 2 for a list of participants in (alphabetical) country order.
In this report the results of the proficiency test are presented and discussed.

2   SET UP

The Institute for Interlaboratory Studies (i.i.s.) in Dordrecht, The Netherlands, was the organiser of this proficiency test.
In this study it was decided to send one Virgin Olive Oil grade Cold Press sample (labelled #0228). Participants were requested to report rounded and unrounded results. The unrounded results were used for the statistical evaluation if possible.

2.1   PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of August 1998 (iis98protocol, version 2.0).

2.2   SAMPLES

The necessary bulk material was purchased from the local Dutch market. After homogenisation in a precleaned metal drum, the Virgin Olive Oil was transferred into 27 brown glass bottles of 250 mL and labelled #0228.
The homogeneity of the subsamples was checked by determination of density in accordance with ASTM D4052:02 on 4 stratified random selected samples.

	Density @15 °C in kg/L
sample 0228-1	0.9154
sample 0228-2	0.9155
sample 0228-3	0.9155
sample 0228-4	0.9155

table 1: measured densities for homogenity of subsamples 0228.

From the density results in table 1, the repeatibility of the results was calculated by multiplication of the standard deviation by 2.8:

sample	Density @15 °C in kg/L
r (sample 0228)	0.0001
Reference test	ASTM D4052:02
r (reference test)	0.0001

table 2: repeatibility of subsamples 0228.

The repeatibility of the results of homogeneity test for density was in good agreement with the repeatibility as required by ASTM D4052:02. Therefore, homogeneity of the samples 0228 was assumed.

One 250 mL bottle of Virgin Olive Oil labelled #0228 was despatched to each of the participating laboratories on April 17, 2002.

2.3 ANALYSES

The participants were asked to determine the following tests (as mentioned in the applicable EC legislation 2568/91): Free Fatty Acid, Peroxide Value, 15 Sterols, K232 nm, K270 nm and Delta K, 16 Fatty acids (CN 14 – 24), Erythrodiol, Uvaol, Trilinolein, Total Stigmastadien, Triacylglycerols and Water, applying the analysis procedure that is routinely used in the laboratory.
To get comparable results a set of detailed report forms was sent together with each set of samples. Also a letter of instructions was sent along.

3 RESULTS

During six weeks after sample despatch, the results of the individual laboratories were gathered. The original results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code number.

Directly after the deadline the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected data are put under 'Remarks' in the result tables in appendix 1.

Results that came in after deadline, were not taken into account in the screening for suspect data and thus these participants were not requested to check the raw data for obvious errors.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test is described in the report 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of August 1998 (iis98protocol, version 2.0).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<…' or '>…' were not used in the statistical evaluation.

First the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. After removal of outliers this check was repeated. In case a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by G(0.01) for the Dixon test and by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by G(0.05) for the Dixon test and by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

Finally the reproduciblities were calculated from the standard deviations by multiplying these with a factor of 2.8.

3.2   GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the results from a sample are plotted. The corresponding laboratory numbers are under the X-axis. A straight line presents the average of the reported data. The reproducibility limits of the relevant standard, calculated as mean ± target reproducibility, are presented by two dotted lines parallel to the average line. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

3.3   Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated in accordance with:

$$z_{(target)} = (result - average\ of\ PT)\ /\ target\ standard\ deviation$$

Absolute values for z<2 are very common and absolute values for z>3 are very rare. The usual interpretation of z-scores is as follows:

$|z| < 1$ good
$1 < |z| < 2$ satisfactory
$2 < |z| < 3$ questionable
$3 < |z|$ unsatisfactory

4   EVALUATION

During the execution of this proficiency test only one problem occurred. One participant did not receive the sample and a new sample was send. It was decided to give two weeks extra for reporting the results. After six weeks from despatch all 18 participants had reported results.

In total 262 numerical results were reported. Observed were 11 outlying results, which is 4.2 % of the numerical results. In proficiency studies outlier percentages of 3 % - 7.5 % are quite normal.

Most data sets proved to have a normal distribution. However, some data sets (see result tables in appendix 1) did not have a Gaussian distribution, even not after rejection of suspect results. Therefore, the interpretation of the statistical evaluations of these components should be done with care.

4.1   EVALUATION PER TEST

In this section the results are discussed per test. All results and the statistical evaluation, reported on the several samples are summarised in appendix 1.

For several parameters too few results were reported to enable meaningful statistical evaluations and therefore are not discussed in this chapter. These parameters are: 2.4-Methylenecholesterol, Campestanol, Delta-7-campesterol, Delta-5,23-stigmasterol, trans-Oleic acid, trans-Linoleic acid, trans-Linolenic acid, Erythrodiol, Uvaol, Trilinolein, Triacylglycerols and Total stigmastadien.

FFA: This determination is not problematic. No results were outside the reproducibility limits and the calculated reproducibility is in agreement with the requirements of ISO660:96. However, when the EEC2598/91 data are evaluated separately the calculated reproducibility is smaller. Thus the large spread may be caused by the variety of methods used.

Peroxide value: This determination is not problematic. No results were outside the reproducibility limits and the calculated reproducibility is in good agreement with the requirements of ISO3960:98.

Sterols: No conclusions could be drawn, due the lack of a suitable reference method. However, when the calculated reproducibilities are compared with last years results, it is noticed that for most investigated components the calculated reproducibilities are about the same.

Fatty Acid comp.:   This determination is not problematic. Only one result was found outside the reproducibility limits. For all investigated components in the sample, the observed reproducibilities are all in good agreement with the requirements of ISO5508:90.

UV-data: No conclusions could be drawn, due the lack of a suitable reference method. However, when the calculated reproducibilities are compared with last years results, it is noticed that the prensently calculated reproducibilities are significaly smaller than last year's. When the EEC2568 data are evaluated separately, the calculated reproducibility is, except for K232 data, even smaller.

Water: This determination is problematic. Five results were found outside the reproducibility limits. The calculated reproducibility is not all in agreement with the requirements of ISO8534:96. However, when the Karl Fisher data are evaluated separately, the calculated reproducibility is much smaller. So the variety in methods used may be explain for the observed large spread.

4.2　PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and these parameters as found for the group of participating laboratories. The average results and the calculated reproducibilities are compared in the next tables with the reproducibilities, derived from literature standards (in casu the ASTM and ISO standards, see tabels in appendix 1).

Parameter	unit	n	average	$2.8 * sd_R$	R (target)
FFA as oleic acid	%M/M	17	0.513	0.080	0.077
Peroxide value	meq O_2/kg	11	10.709	3.498	8.032
Fatty acid composition					
*　Myristic acid = 14:0	%M/M	7	0.007	0.015	0.500
*　Palmitic acid = 16:0	%M/M	12	11.464	0.600	1.148
*　Palmitoleic acid = 16:1	%M/M	12	0.972	0.165	0.500
*　Heptadecanoic acid = 17:0	%M/M	9	0.083	0.067	0.500
*　Heptadecenoic acid = 17:1	%M/M	7	0.127	0.037	0.500
*　Stearic acid = 18:0	%M/M	12	2.756	0.104	0.500
*　Oleic acid = 18:1	%M/M	12	76.750	1.389	3.000
*　trans-Oleic acid = t-18:1	%M/M	4	0.007	0.014	0.500
*　Linoleic acid = 18:2	%M/M	11	6.097	0.284	0.618
*　trans-Linoleic acid = t-18:2	%M/M	4	0.008	0.015	0.500
*　Linolenic acid = 18:3	%M/M	11	0.665	0.078	0.500
*　trans-Linolenic acid = t-18:3	%M/M	4	0.004	0.013	0.500
*　Arachidic acid = 20:0	%M/M	12	0.378	0.128	0.500
*　Eicosenoic acid = 20:1	%M/M	10	0.288	0.048	0.500
*　Behenic acid = 22:0	%M/M	9	0.110	0.054	0.500
*　Lignoceric acid = 24:0	%M/M	9	0.068	0.068	0.500
Water	%M/M	8	0.087	0.045	0.016

table 3: reproducibilities of sample 0228

Without further statistical calculations it can be concluded that the calculated reproducibilities for the group of participating laboratories are in good agreement with the reproducibilities of the Standard methods for all parameters. Remarkably only the water determination was the problematic parameter. See also the discussion in paragraphs 4.1 and 5.

For the parameters not mentioned in tables 3 no precision data are known. Therefore the observed reproducibilities could not be compared with existing values. These observed reproducibilities are listed in the following table 4.

Parameter	unit	n	average	2.8 * sd_R	R(iis2001)
Sterol composition					
* Cholesterol	%M/M	6	0.30	0.56	0.36
* Brassicasterol	%M/M	5	0.04	0.10	0.07
* 2,4-Methylene cholesterol	%M/M	4	0.12	0.07	0.25
* Campesterol	%M/M	7	3.02	0.28	0.36
* Campestanol	%M/M	4	0.13	0.51	0.63
* Stigmasterol	%M/M	7	0.90	0.20	0.14
* Delta-7-Campesterol	%M/M	4	0.18	0.73	0.15
* Delta-5,23-Stigmastadienol	%M/M	4	0.08	0.27	-----
* Clerosterol	%M/M	6	0.87	0.58	0.92
* Beta-sitosterol	%M/M	7	84.91	4.89	3.27
* Sitostanol	%M/M	5	0.53	0.24	0.64
* Delta-5-Avenasterol	%M/M	7	6.87	0.56	2.22
* Delta-5,24-Stigmastadienol	%M/M	6	0.42	0.35	0.42
* Delta-7-Stigmasterol	%M/M	5	0.31	0.23	0.67
* Delta-7-Avenasterol	%M/M	7	0.48	0.31	0.07
* K 232 nm	%E.cm	9	1.810	0.097	0.377
* K 270 nm	%E.cm	8	0.118	0.008	0.027
* Delta K	%E.cm	6	-0.001	0.002	0.007
Trilinolein	%M/M		not enough results		0.33
Triacylglycerols as delta ECN42	mol%	4	0.04	0.04	0.10
Erythrodiol	%M/M		not enough results		-----
Uvaol	%M/M		not enough results		-----
Stigmastadien	%M/M		not enough results		-----

table 4: reproducibilities of sample 0228 vs iis01E02X

4.3   COMPARISON WITH RESULTS OF THE PROFICIENCY TEST OF MARCH 2001

Comparison of the group performances of the present round to last years round (2001):

2002 better than 2001	2002 the same as 2001	2002 worse than 2001
Free Fatty Acid	Sterols	Water
Peroxide value		
Fatty Acid composition		
UV data		

5.   CONCLUSIONS

A proficiency test for Virgin Olive Oil was organised for the second time. The reproducibilities, as observed in the present interlaboratory study, for the FFA determination, the peroxide determination and the fatty acids composition are in good agreement with the precision requirements of the respective ISO Standard methods. Only the results of the Water determination was not in agreement with ISO 8534:96.
The determinations of sterols and K-data gave satisfactory reproducibilities. When compared with last years reproducibilities, most components of the sterols determination showed a similar calculated reproducibility. However, the composition of last years Virgin Olive Oil was deviating on some components, so evaluation should be done with care.
For the determination the Trilinolein content, Triacylglycerols, Erythrodiol, Uvaol and Stigmastadien, the lack of a suitable method and to few results were reported for meaningful conclusions.
The overall improvement of the performance of the group is visible.

APPENDIX 1

Determination of FFA as oleic acid in samples 0228; results in %M/M

lab	method	value	mark	Z(targ)	remarks
2100	ISO660	0.51		-0.12	
3102	ISO660	0.5		-0.48	
3103	TITR.	0.49		-0.85	
3138	AOAC940.28	0.5246		0.41	
3139	EEC2568/91	0.559		1.66	
4005	Pharma 2.5.1	0.489		-0.88	
4017	JOCS	0.4727		-1.48	
4021	IUPAC 2201	0.5		-0.48	
4037	INHOUSE	0.465		-1.76	
4076	EEC2568/91	0.53		0.61	
4078	AOCS Ca5a-40	0.53		0.61	
4082	EEC2568/91	0.577		2.32	
4084	EEC2568/91	0.490		-0.85	
4085	EEC2568/91	0.53626		0.84	
4088	AOCS Ca5a-40	0.509		-0.16	
4092	EEC2568/91	0.534		0.75	
4093	EEC2568/91	0.510742		-0.09	
4094	EEC2568/91	0.512		-0.05	

			Only EEC2568/91 data:
normality	OK		OK
n	17		7
outliers	0		0
mean (n)	0.5133		0.5246
st.dev. (n)	0.02853		0.02230
R(calc.)	0.0799		0.0625
R(ISO660:96)	0.0770		unknown
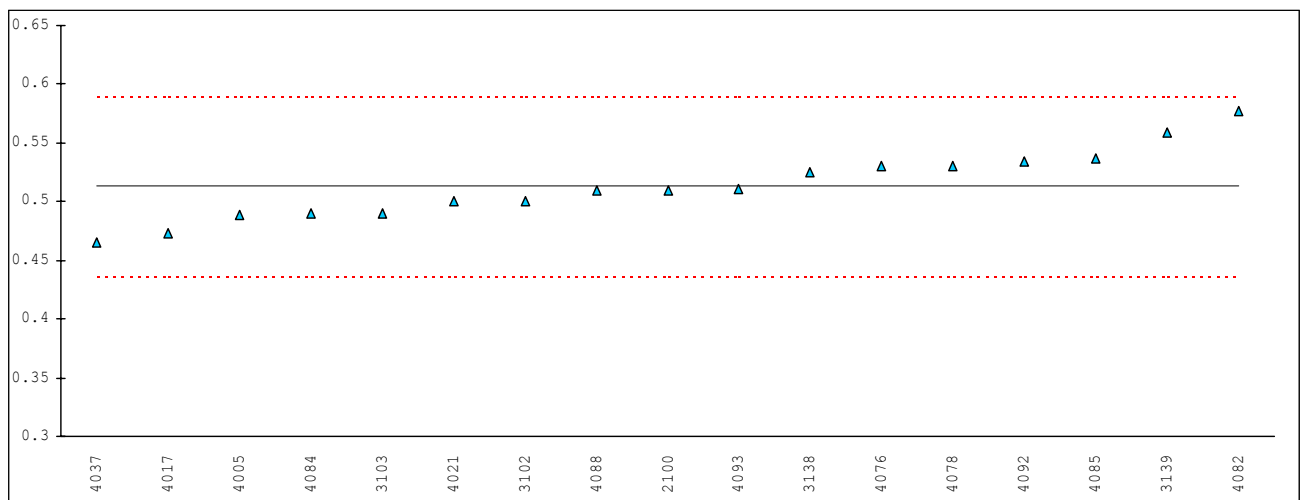
Determination of Peroxide value in samples 0228; results in meq O_2/kg

lab	method	value	mark	Z(targ)	remarks
2100	ISO3960	10.4		-0.11	
3102	ISO3960	5.97	DG(0.01)	-1.65	
3103		-----		-----	
3138	AOAC965.33	12.512		0.63	
3139	EEC2568/91	9.184		-0.53	
4005	Pharma2.5.5	11.24		0.19	
4017	JOCS	9.3208		-0.48	
4021	AOCS Cd86-90	9.1		-0.56	
4037	INHOUSE	3.50	DG(0.01)	-2.51	
4076	EEC2568/91	12.4		0.59	
4078	AOCS Cd 8-53	10.3		-0.14	
4082	EEC2568/91	10.970		0.09	
4084		-----		-----	
4085		-----		-----	
4088	AOCS Cd 8-53	10.295		-0.14	
4092		-----		-----	
4093		-----		-----	
4094	EEC2568/91	12.076		0.48	

|	
normality	OK
n	11
outliers	2
mean (n)	10.7089
st.dev. (n)	1.24919
R(calc.)	3.4977
R(ISO3960:98)	8.0317
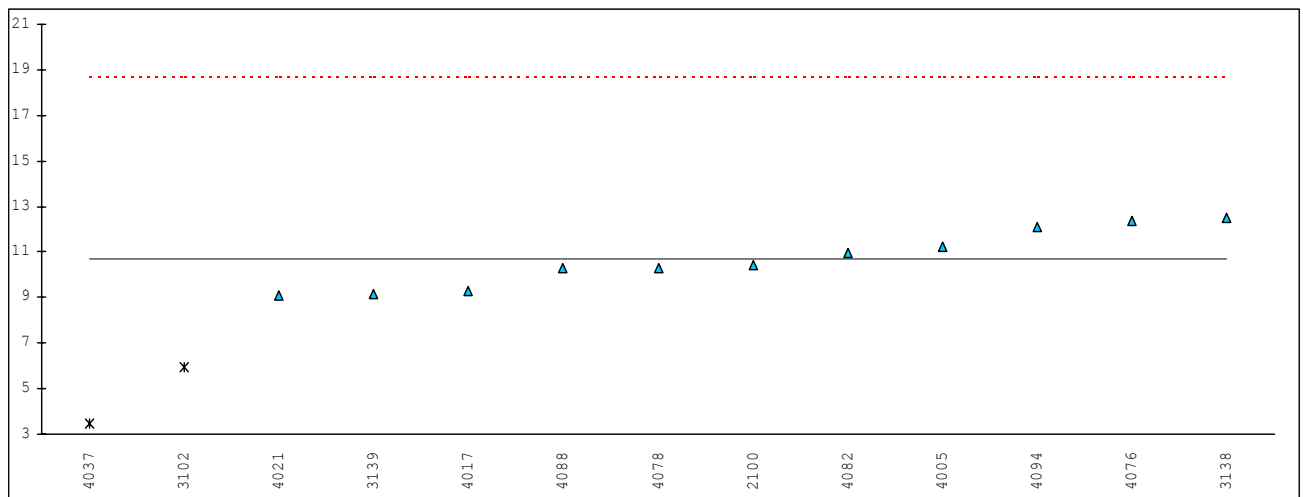
Determination of Sterols content in sample 0228; results in %M/M

| lab	Method	Chole sterol	mark	Brassica sterol	mark	2.4-methyle ne chole sterol	mark	Campe sterol	mark	Campe stanol	mark	Stigma sterol	mark
2100	ISO6799	0.5		0.08		nd		3.09		nd		0.83	
3102		-----		-----		-----		-----		-----		-----	
3103		-----		-----		-----		-----		-----		-----	
3138		-----		-----		-----		-----		-----		-----	
3139		-----		-----		-----		-----		-----		-----	
4005	inhouse	0.1831		0		0.1245		2.8521		0		0.8412	
4017	AOCS	-----		-----		-----		3.10906		-----		0.99745	
4021	GC/TLC	0.6		0.7	ex [1]	<0.1		3.0		<0.1		0.9	
4037		-----		-----		-----		-----		-----		-----	
4076	eec2568	0.221		0.04		0.13		3.121		0.40	fr 0.46	1.00	
4078	AOCS	0.21		0.02		0.08		3.04		0.05		0.85	
4082		-----		-----		-----		-----		-----		-----	
4084	eec2568	0.1		-----		0.14		2.93		0.07		0.86	
4085		-----		-----		-----		-----		-----		-----	
4088		-----		-----		-----		-----		-----		-----	
4092		-----		-----		-----		-----		-----		-----	
4093		-----		-----		-----		-----		-----		-----	
4094		-----		-----		-----		-----		-----		-----	
	normality	not OK		not OK		OK		OK		OK		OK	
	n	6		4		4		7		4		7	
	outliers	0		0		0		0		0		0	
	mean (n)	0.302		0.035		0.119		3.020		0.130		0.897	
	st.dev. (n)	0.1990		0.0342		0.0265		0.1001		0.1824		0.0729	
	R(calc.)	0.557		0.096		0.074		0.280		0.511		0.204	
	R(lit.)	unknown		unknown		unknown		unknown		unknown		unknown	
	R(iis2001)	0.360		0.068		0.246		0.355		0.625		0.142	

[1]) manual excluded, false positive result?

lab	Method	Delta-7-Campe sterol	mark	Delta-5,23-Stigma sterol	mark	Clero sterol	mark	Beta-sisto sterol	mark	Sito stanol	mark	Delta-5-Avena sterol	mark
2100	ISO6799	nd		nd		nd		82.28		-----		6.88	
3102		-----		-----		-----		-----		-----		-----	
3103		-----		-----		-----		-----		-----		-----	
3138		-----		-----		-----		-----		-----		-----	
3139		-----		-----		-----		-----		-----		-----	
4005	inhouse	0		0		0.9574		85.7708		1.3215	G(0.01)	6.9861	
4017	AOCS	-----		-----		0.98710		86.31115		0.53839		6.75444	
4021	GC/TLC	<0.1		0.2		0.5	DG(0.01)	83.0		0.6		6.7	
4037		-----		-----		-----		-----		-----		-----	
4076	eec2568	0.15		0.10		1.021		84.37		0.53		7.26	
4078	AOCS	0		0		0.73	DG(0.01)	86.94		0.39		6.69	
4082		-----		-----		-----		-----		-----		-----	
4084	eec2568	0.55		-----		1.00		85.71		0.59		6.84	
4085		-----		-----		-----		-----		-----		-----	
4088		-----		-----		-----		-----		-----		-----	
4092		-----		-----		-----		-----		-----		-----	
4093		-----		-----		-----		-----		-----		-----	
4094		-----		-----		-----		-----		-----		-----	
	normality	OK		OK		OK		OK		OK		OK	
	n	4		4		4		7		5		7	
	outliers	0		0		2		0		1		0	
	mean (n)	0.175		0.075		0.991		84.912		0.530		6.873	
	st.dev. (n)	0.2598		0.0957		0.0266		1.7475		0.0839		0.2005	
	R(calc.)	0.727		0.268		0.075		4.893		0.235		0.561	
	R(lit.)	unknown		unknown		unknown		unknown		unknown		unknown	
	R(iis2001)	0.153		unknown		0.924		3.272		0.639		2.222	

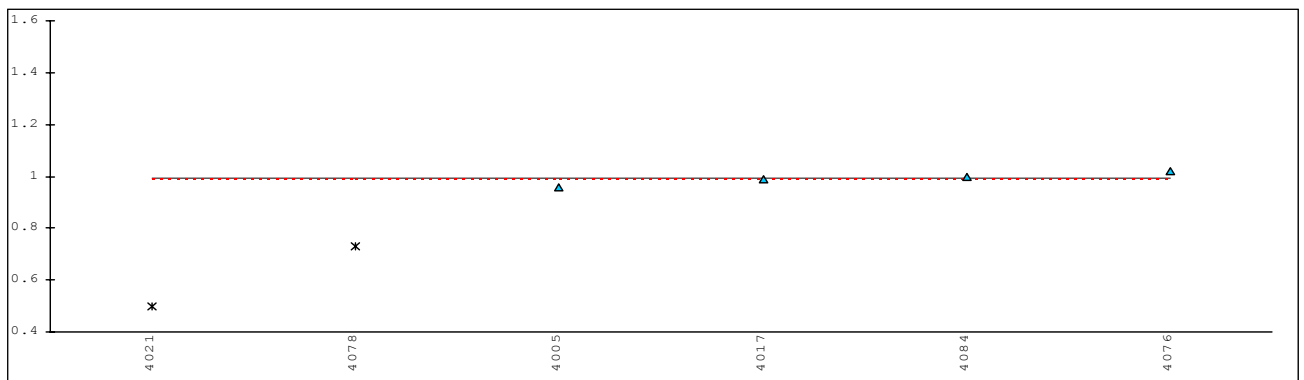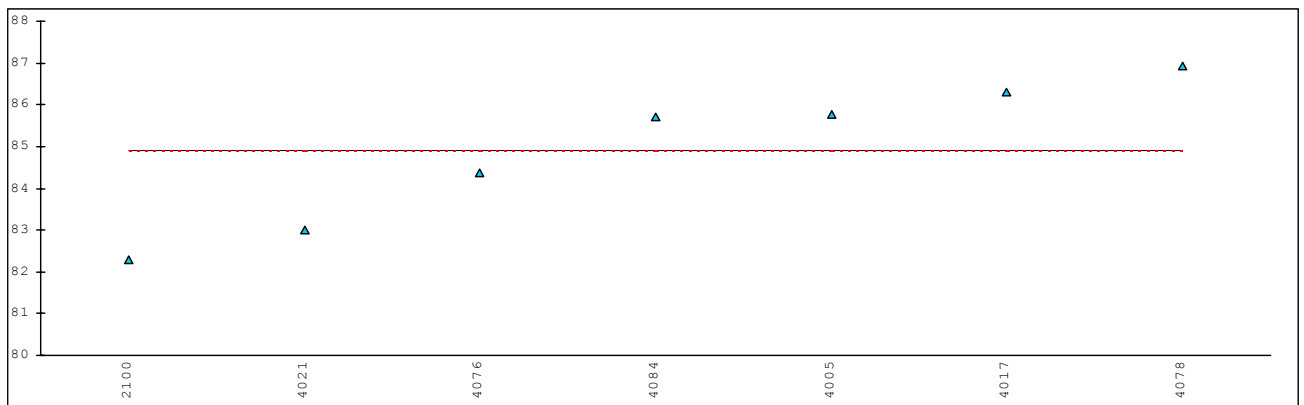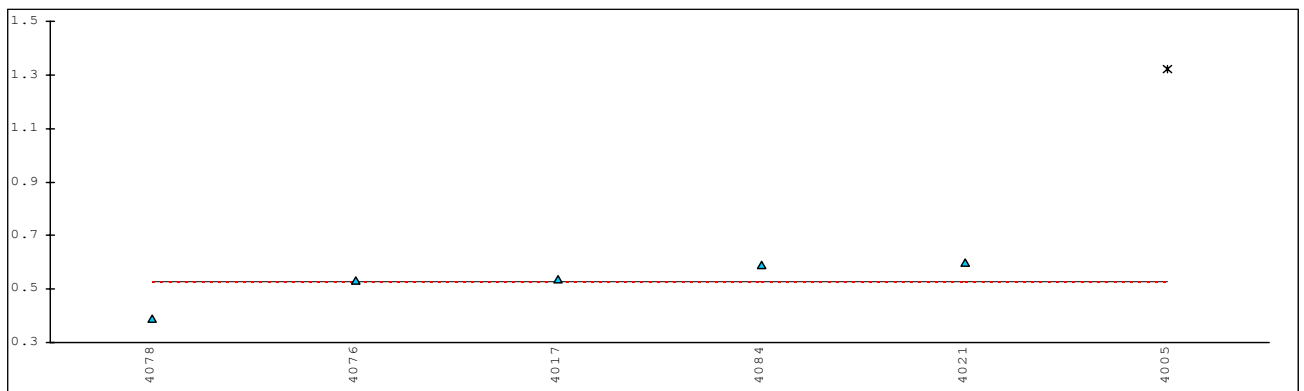| lab	Method	Delta-5,24-Stigmastadientol	mark	Delta-7-Stigmasterol	mark	Delta-7-Avenasterol	mark
2100	ISO6799	-----		1.38	DG(0.01)	0.35	
3102		-----		-----		-----	
3103		-----		-----		-----	
3138		-----		-----		-----	
3139		-----		-----		-----	
4005	inhouse	0.2729		0.2342		0.4461	
4017	AOCS	0.36297		0.32114		0.48599	
4021	GC/TLC	0.4		0.9	DG(0.01)	0.7	
4037		-----		-----		-----	
4076	eec2568	0.64		0.44		0.52	
4078	AOCS	0.35		0.25		0.40	
4082		-----		-----		-----	
4084	eec2568	0.48		0.28		0.45	
4085		-----		-----		-----	
4088		-----		-----		-----	
4092		-----		-----		-----	
4093		-----		-----		-----	
4094		-----		-----		-----	
	normality	OK		OK		OK	
	n	6		5		7	
	outliers	0		2		0	
	mean (n)	0.418		0.305		0.479	
	st.dev. (n)	0.1282		0.0824		0.1121	
	R(calc.)	0.359		0.231		0.314	
	R(lit)	unknown		unknown		unknown	
	R(iis2001)	0.418		0.673		0.067	
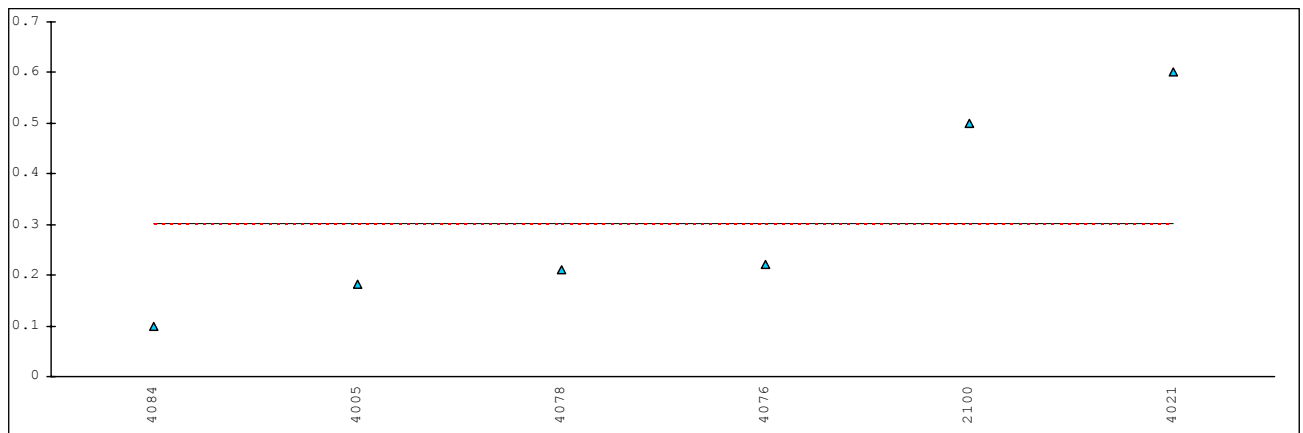
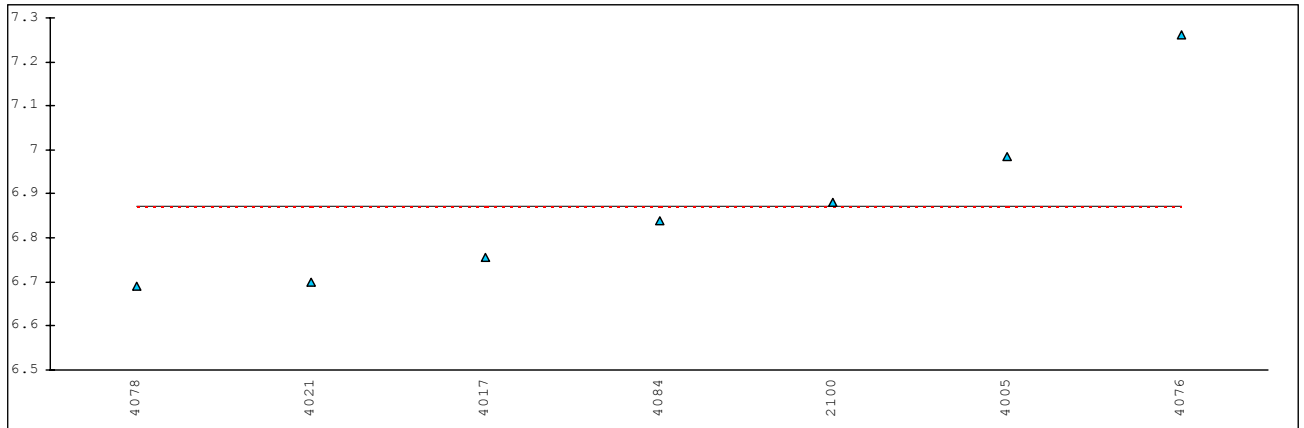Cholesterol
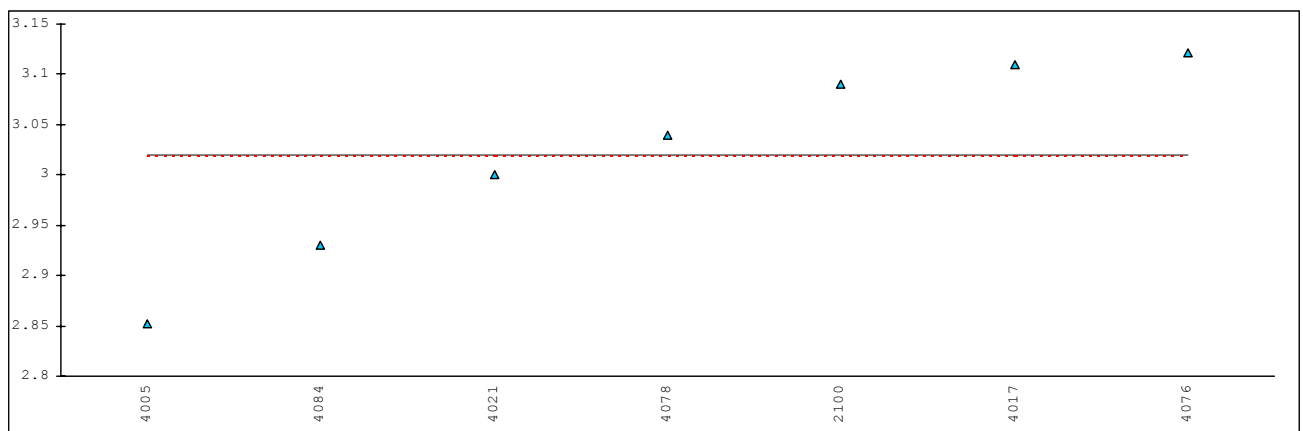
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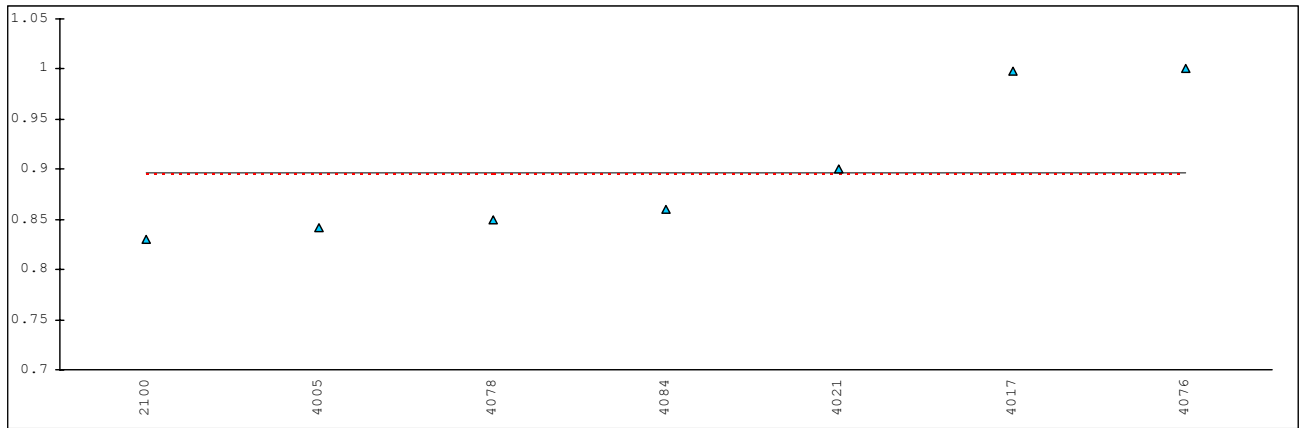
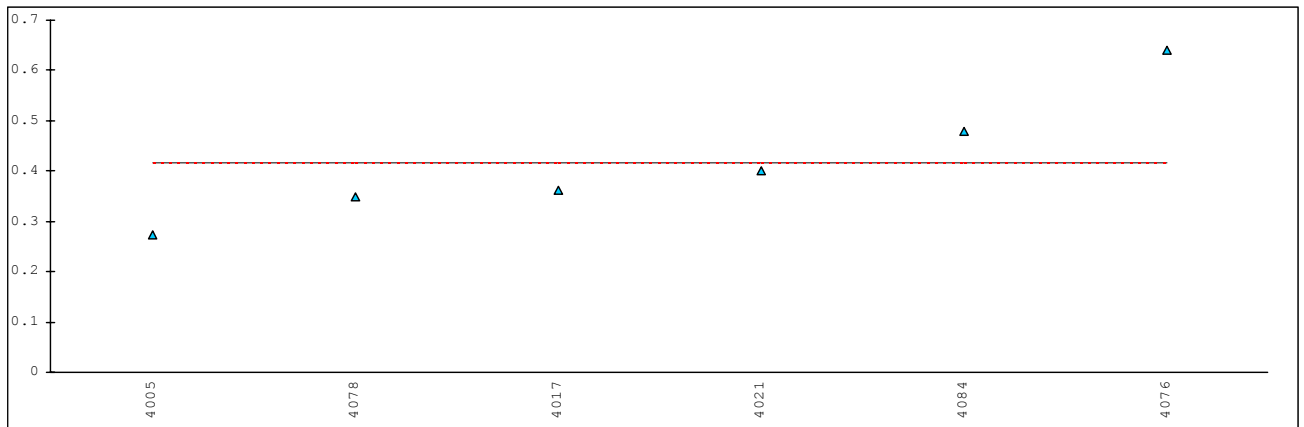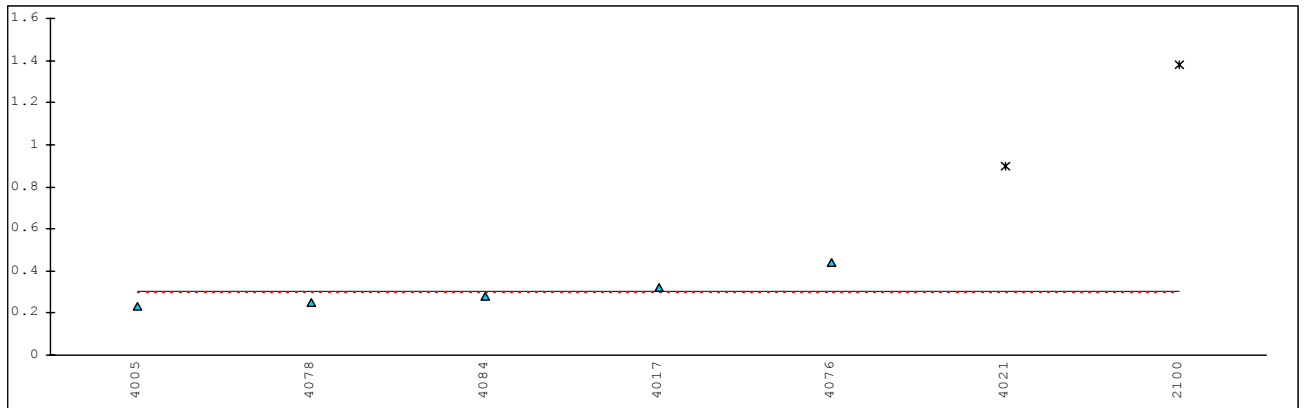Stigmasterol

Clerosterol

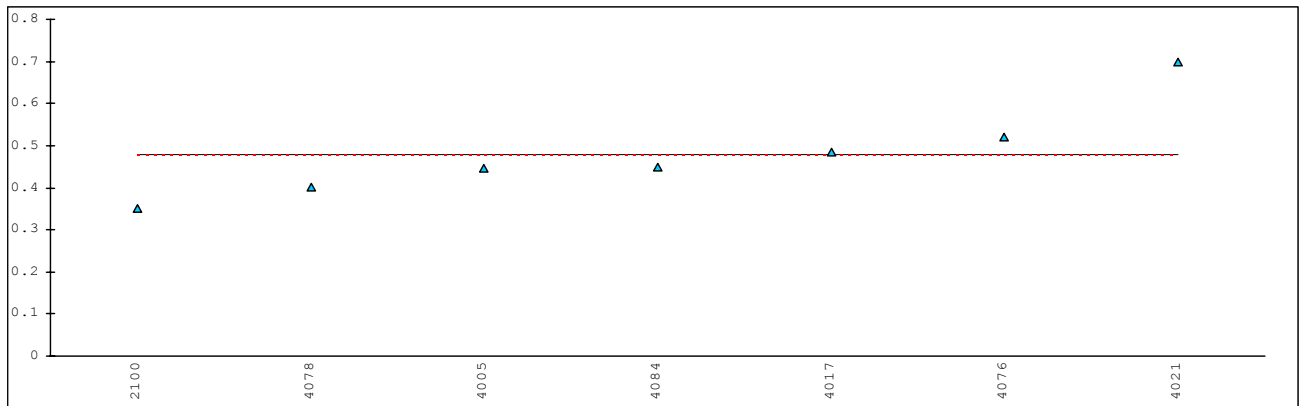Betasistosterol

Sitostanol

Delta-5-avenasterol

Delta-5,24-stigmasitadientol

Delta-7-stigmasterol

Delta-7-avenasterol

Determination of Total Fatty Acids composition in sample 0228: results %M/M

| lab	Methods	Myristic acid C14:0	mark	Z(targ)	Palmitic acid C16:0	mark	Z(targ)	Palmitol eic acid C16:1	mark	Z(targ)	Heptade canoic acid C17:0	mark	Z(targ)
2100	ISO5508/9	0		-0.04	11.0		-1.13	1.0		0.16	0.1		0.11
3102		-----		-----	-----		-----	-----		-----	-----		-----
3103	ISO5508/9	-----		-----	11.38		-0.21	0.97		-0.01	0.13		0.27
3138		-----		-----	-----		-----	-----		-----	-----		-----
3139	eec2568	0.007		0.00	11.701		0.58	0.991		0.11	0.071		-0.06
4005	inhouse	0		-0.04	11.7378		0.67	0.9829		0.06	0.0670		-0.08
4017	AOCS	-----		-----	11.538		0.18	1.0547		0.47	-----		-----
4021	AOCS RP2A-94	<0.1		-----	11.3		-0.40	0.9		-0.40	0.1		0.11
4037	inhouse	-----		-----	11.6978		0.57	0.888319		-0.47	-----		-----
4076	eec2568	0.015		0.04	11.50		0.09	0.91		-0.35	0.07		-0.06
4078	AOCS291	0.01		0.02	11.38		-0.38	1.03		0.59	0.06		-0.12
4082	ISO5508/9	-----		-----	11.29		-0.42	0.93		-0.23	-----		-----
4084	eec2568	0.009		0.01	11.56		0.23	0.96		-0.07	0.06		-0.12
4085	eec2568	0.009		0.01	11.535		0.17	0.993		0.12	0.071		-0.06
4088		-----		-----	-----		-----	-----		-----	-----		-----
4092		-----		-----	-----		-----	-----		-----	-----		-----
4093		-----		-----	-----		-----	-----		-----	-----		-----
4094		-----		-----	-----		-----	-----		-----	-----		-----
	normality	OK			OK			OK			not OK		
	n	7			12			12			9		
	outliers	0			0			0			0		
	mean (n)	0.0071			11.4641			0.9717			0.0810		
	st.dev. (n)	0.00546			0.21412			0.05889			0.02378		
	R(calc.)	0.0153			0.5995			0.1649			0.0666		
	R(D5508:90)	0.5000			1.1476			0.5000			0.5000		

lab	Methods	Heptade canoic acid C17:1	mark	Z(targ)	Stearic acid C18:0	mark	Z(targ)	Oleic acid C18:1	mark	Z(targ)	trans-Oleic acid C18:1	mark	Z(targ)
2100	ISO5508/9	0.1		-0.15	2.75	fr 2.4	-0.03	76.9		0.14	-----		-----
3102		-----		-----	-----		-----	-----		-----	-----		-----
3103	ISO5508/9	-----		-----	2.78		0.14	76.51		-0.22	nd		-----
3138		-----		-----	-----		-----	-----		-----	-----		-----
3139	eec2568	0.129		0.01	2.768		0.07	76.781		0.03	-----		-----
4005	inhouse	0.1341		0.04	2.7491		-0.04	76.7806		0.03	0		-0.04
4017	AOCS	-----		-----	2.7615		0.03	76.6249		-0.12	-----		-----
4021	AOCS RP2A-94	<0.1		-----	2.7		-0.31	76.5		-0.23	<0.1		-----
4037	inhouse	-----		-----	2.691121		-0.36	76.30121		-0.42	-----		-----
4076	eec2568	0.14		0.08	2.77		0.08	76.00		-0.70	0.011		0.02
4078	AOCS291	0.13		0.02	2.74		-0.09	78.06		1.22	0.01		0.02
4082	ISO5508/9	-----		-----	2.83		0.42	76.74		-0.01	-----		-----
4084	eec2568	0.12		-0.04	2.74		-0.09	77.00		0.23	0.006		0.00
4085	eec2568	0.133		0.04	2.785		0.17	76.800		0.05	-----		-----
4088		-----		-----	-----		-----	-----		-----	-----		-----
4092		-----		-----	-----		-----	-----		-----	-----		-----
4093		-----		-----	-----		-----	-----		-----	-----		-----
4094		-----		-----	-----		-----	-----		-----	-----		-----
	normality	OK			OK			OK			unknown		
	n	7			12			12			4		
	outliers	0			0			0			0		
	mean (n)	0.1266			2.7554			76.7498			0.0068		
	st.dev. (n)	0.01320			0.03717			0.49592			0.00499		
	R(calc.)	0.0370			0.1041			1.3886			0.0140		
	R(D5508:90)	0.5000			0.5000			3.0000			0.5000		

| lab	Methods	Linoleic acid C18:2	mark	Z(targ)	trans-Linoleic acid C18:2	mark	Z(targ)	Linolenic acid C18:3	mark	Z(targ)	trans-Linolenic acid C18:3	mark	Z(targ)
2100	ISO5508/9	6.3		0.92	-----		-----	0.7		0.20	-----		-----
3102		-----		-----	-----		-----	-----		-----	-----		-----
3103	ISO5508/9	6.09		-0.03	nd		-----	0.64		-0.14	-----		-----
3138		-----		-----	-----		-----	-----		-----	-----		-----
3139	eec2568	6.104		0.03	-----		-----	0.664		0.00	-----		-----
4005	inhouse	6.0789		-0.08	0		-0.04	0.6465		-0.10	0		-0.02
4017	AOCS	6.1378		0.19	-----		-----	0.6402		-0.14	-----		-----
4021	AOCS RP2A-94	6.1		0.02	<0.1		-----	0.7		0.20	<0.1		-----
4037	inhouse	7.052358	G(0.01)	4.33	-----		-----	1.027577	G(0.01)	2.03	-----		-----
4076	eec2568	6.05		-0.21	0.009		0.01	0.621		-0.24	0.005		0.01
4078	AOCS291	5.87		-1.03	0.01		0.01	0.70		0.20	0.01		0.04
4082	ISO5508/9	6.06		-0.17	-----		-----	0.65		-0.08	-----		-----
4084	eec2568	6.11		0.06	0.011		0.02	0.68		0.09	0		-0.02
4085	eec2568	6.161		0.29	-----		-----	0.669		0.02	-----		-----
4088		-----		-----	-----		-----	-----		-----	-----		-----
4092		-----		-----	-----		-----	-----		-----	-----		-----
4093		-----		-----	-----		-----	-----		-----	-----		-----
4094		-----		-----	-----		-----	-----		-----	-----		-----
	normality	OK			unknown			OK			unknown		
	n	11			4			11			4		
	outliers	1			0			1			0		
	mean (n)	6.0965			0.0075			0.6646			0.0038		
	st.dev. (n)	0.10133			0.00507			0.02767			0.00479		
	R(calc.)	0.2837			0.0142			0.0775			0.0134		
	R(D5508:90)	0.6176			0.5000			0.5000			0.5000		

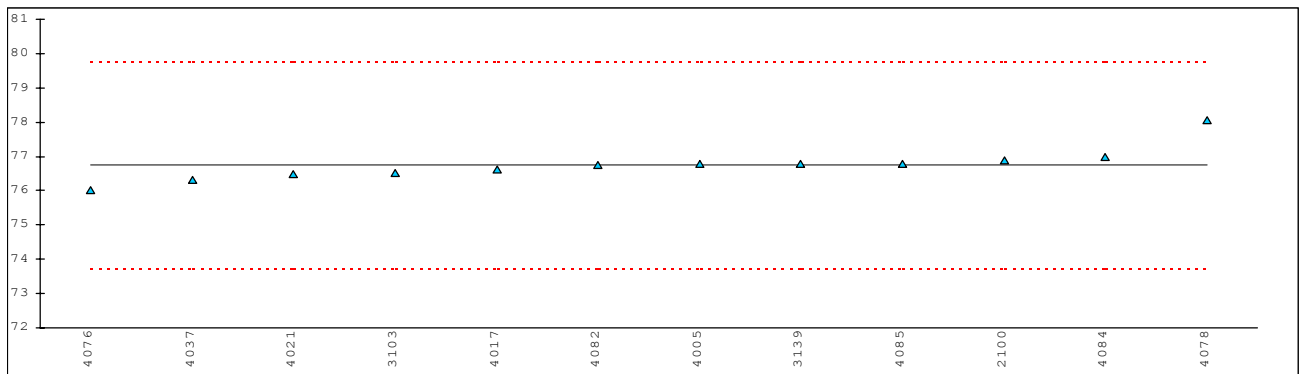| lab	methods	Arachidic acid C20:0	mark	Z(targ)	Eicosenoic acid C20:1	mark	Z(targ)	Behenic acid C22:0	mark	Z(targ)	Lignoceric acid C24:0	mark	Z(targ)
2100	ISO5508/9	0.3		-0.44	0.3		0.07	0.1		-0.06	0.1		0.18
3102		-----		-----	-----		-----	-----		-----	-----		-----
3103	ISO5508/9	0.39		0.07	-----		-----	-----		-----	-----		-----
3138		-----		-----	-----		-----	-----		-----	-----		-----
3139	eec2568	0.379		0.01	0.268		-0.11	0.108		-0.01	0.052		-0.09
4005	inhouse	0.3831		0.03	0.2820		-0.03	0.1079		-0.01	0.0500		-0.10
4017	AOCS	0.3352		-0.24	0.2791		-0.05	-----		-----	-----		-----
4021	AOCS RP2A-94	0.4		0.12	0.3		0.07	0.1		-0.06	0.1		0.18
4037	inhouse	0.341587		-0.20	-----		-----	-----		-----	-----		-----
4076	eec2568	0.38		0.01	0.31		0.13	0.10		-0.06	0.05		-0.10
4078	AOCS291	0.39		0.07	0.31		0.13	0.11		0.00	0.05		-0.10
4082	ISO5508/9	0.49		0.63	0.28		-0.04	0.16		0.28	0.10		0.18
4084	eec2568	0.36		-0.10	0.26		-0.15	0.10		-0.06	0.05		-0.10
4085	eec2568	0.388		0.06	0.287		0.00	0.105		-0.03	0.056		-0.06
4088		-----		-----	-----		-----	-----		-----	-----		-----
4092		-----		-----	-----		-----	-----		-----	-----		-----
4093		-----		-----	-----		-----	-----		-----	-----		-----
4094		-----		-----	-----		-----	-----		-----	-----		-----
	normality	OK			OK			not OK			not OK		
	n	12			10			9			9		
	outliers	0			0			0			0		
	mean (n)	0.3781			0.2876			0.1101			0.0676		
	st.dev. (n)	0.04574			0.01706			0.01915			0.02441		
	R(calc.)	0.1281			0.0478			0.0536			0.0683		
	R(D5508:90)	0.5000			0.5000			0.5000			0.5000		
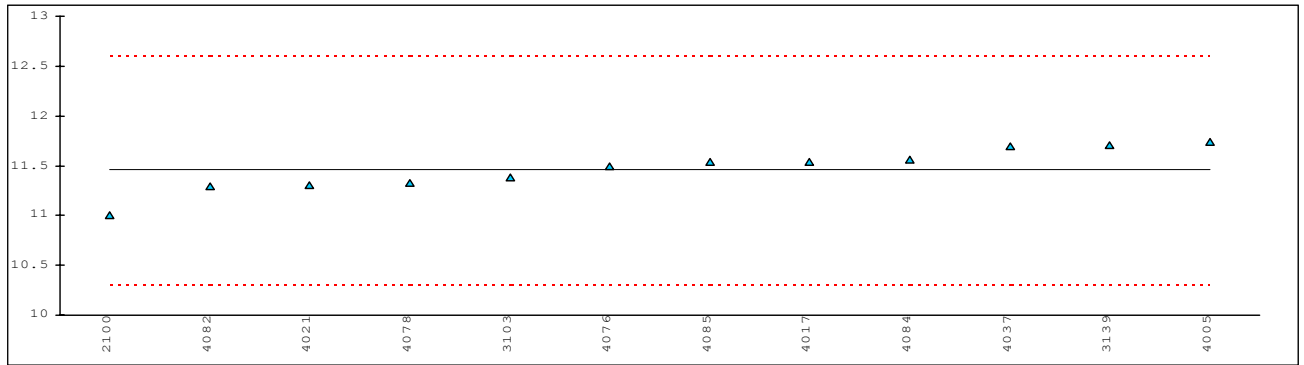
Palmitric acid =16:0
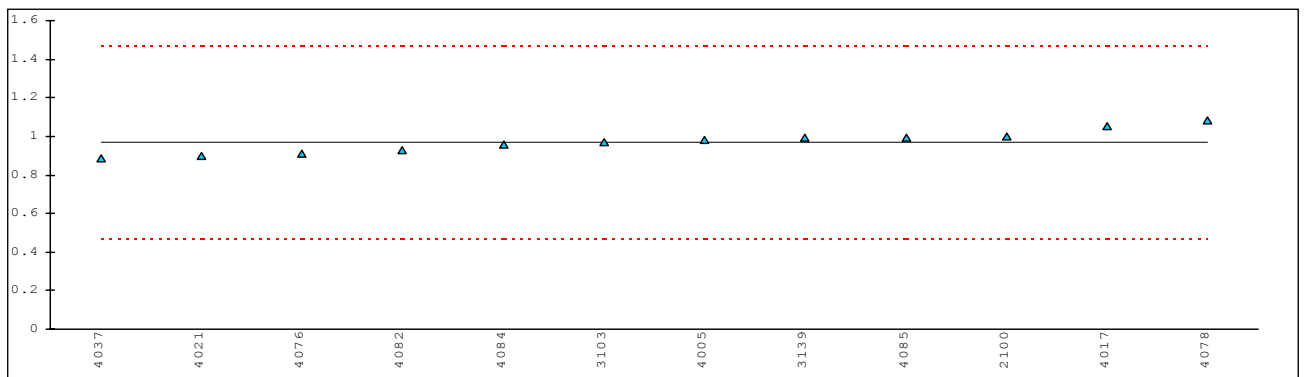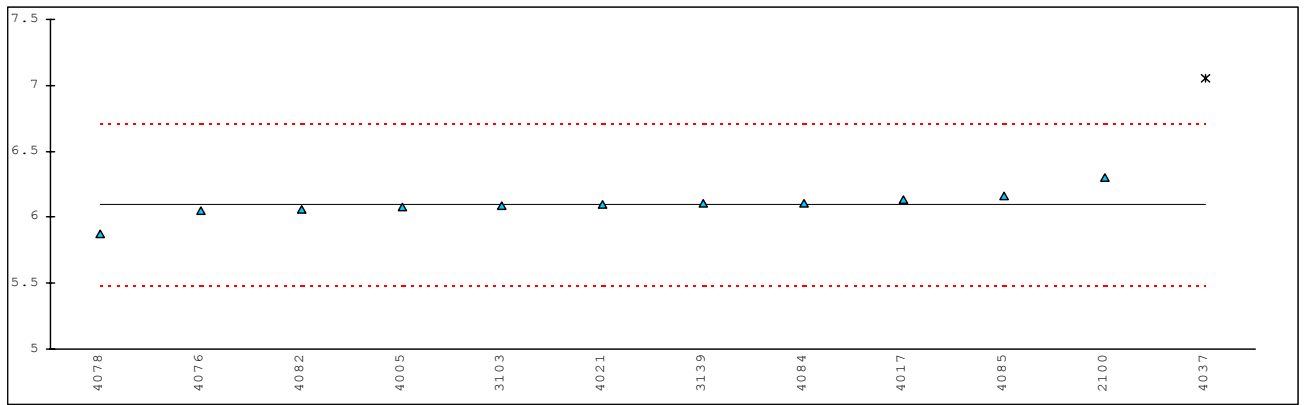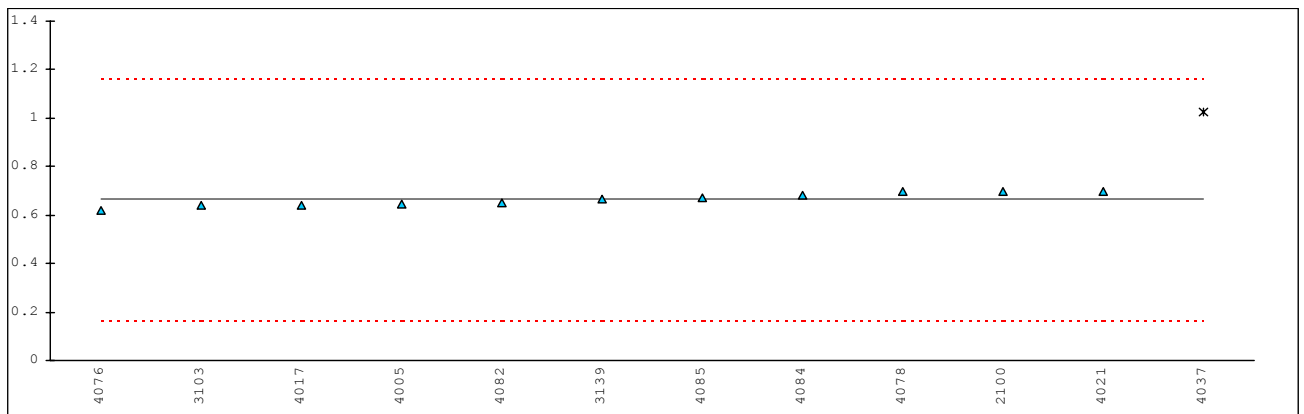

Palmitoleic acid =16:1
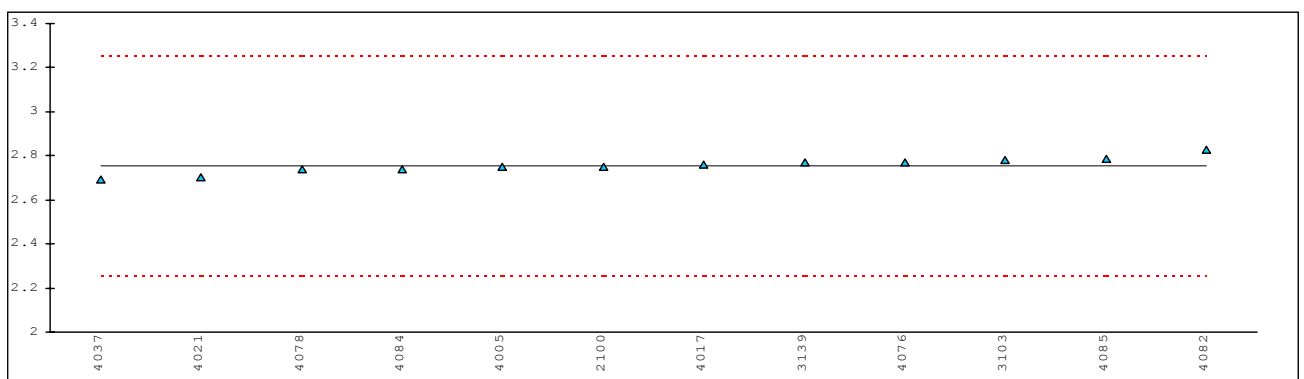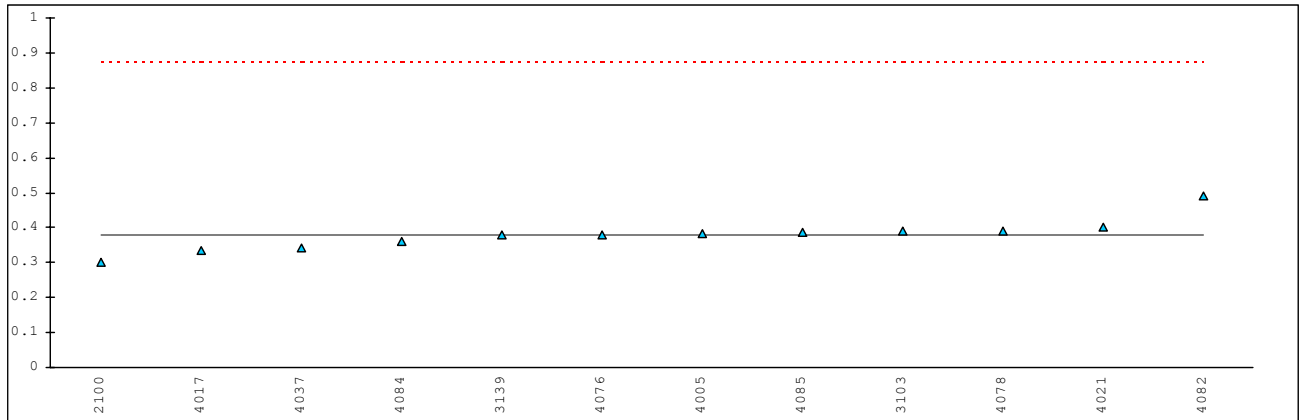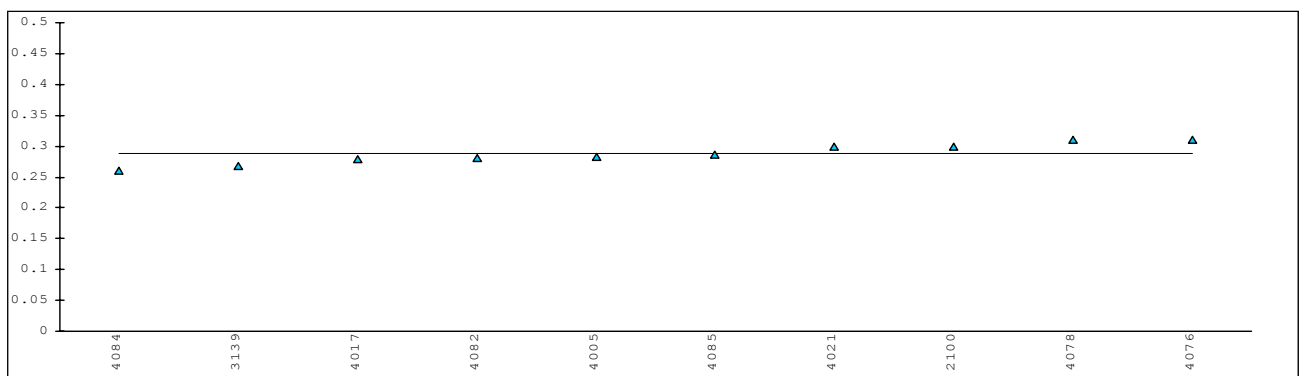

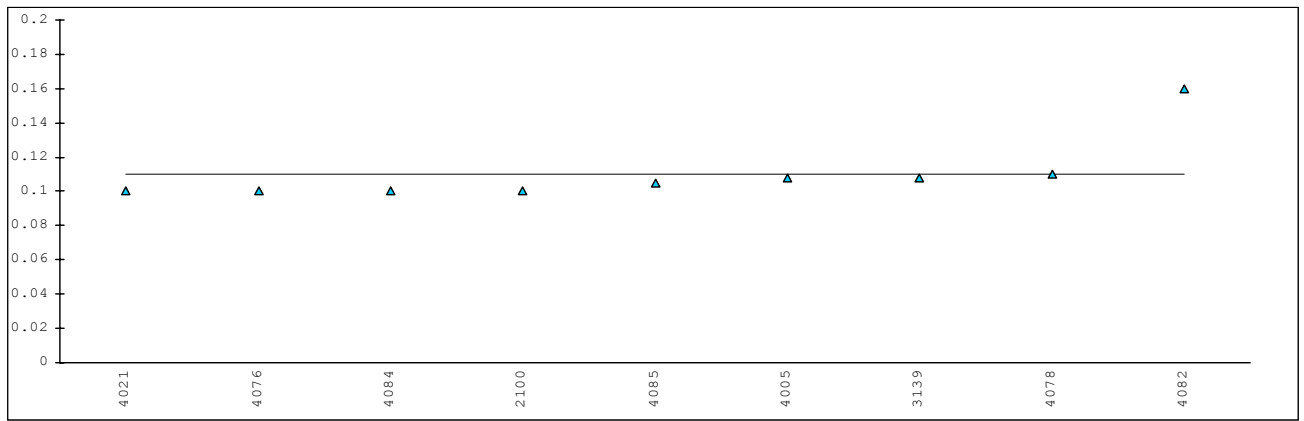Stearic acid =18:0

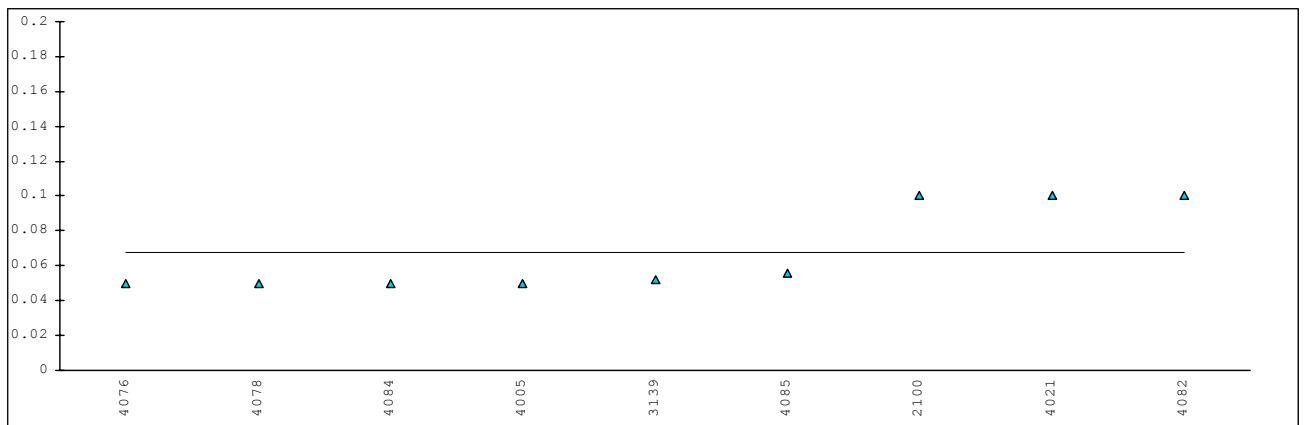Oleic acid =18:1

Linoleic acid =18:2

Linolenic acid =18:3

Arachidic acid =20:0

Eicosenic acid =20:1

Behenic acid =22:0


Lignoceric acid =24:0

Determination of UV spectrophotometric data in sample 0228 ; results in %E.cm

lab	method	K232	mark	Z(targ)	K272	mark	Z(targ)	Delta K	mark	Z(targ)
2100		-----		-----	-----		-----	-----		-----
3102		-----		-----	-----		-----	-----		-----
3103		-----		-----	-----		-----	-----		-----
3138		-----		-----	-----		-----	-----		-----
3139		-----		-----	-----		-----	-----		-----
4005	EP 2.2.25	1.77	fr 0.056	-----	0.116		-----	0.005	G(0.01)	-----
4017		-----		-----	-----		-----	-----		-----
4021	iupac2505	1.81		-----	0.104	G(0.05)	-----	<0.05		-----
4037		-----		-----	-----		-----	-----		-----
4076	eec2568	1.840		-----	0.123		-----	0		-----
4078	AOCS	1.780		-----	0.114		-----	-0.002		-----
4082		-----		-----	-----		-----	-----		-----
4084	eec2568	1.792		-----	0.119		-----	0		-----
4085	eec2568	1.84733		-----	0.12002		-----	-----		-----
4088		-----		-----	-----		-----	-----		-----
4092	eec2568	1.8695		-----	0.1170		-----	-0.0011		-----
4093	eec2568	1.78363		-----	0.119062		-----	-0.00123		-----
4094	eec2568	1.798		-----	0.117		-----	-0.001		-----
			only eec2598 data:			only eec2598 data:			only eec2598 data:	
	normality	OK	OK		OK	OK		OK	OK	
	n	9	6		8	6		6	5	
	outliers	0	0		1	0		1	1	
	mean (n)	1.8101	1.8217		0.1181	0.1192		-0.0009	-0.0007	
	st.dev. (n)	0.03455	0.03513		0.00275	0.00223		0.00077	0.00061	
	R(calc.)	0.0965	0.0984		0.0077	0.0062		0.0022	0.0017	
	R(lit)	unknown	unknown		unknown	unknown		unknown	unknown	
	R(iis2001)	0.377	-----		0.0269	------		0.0068	-----	
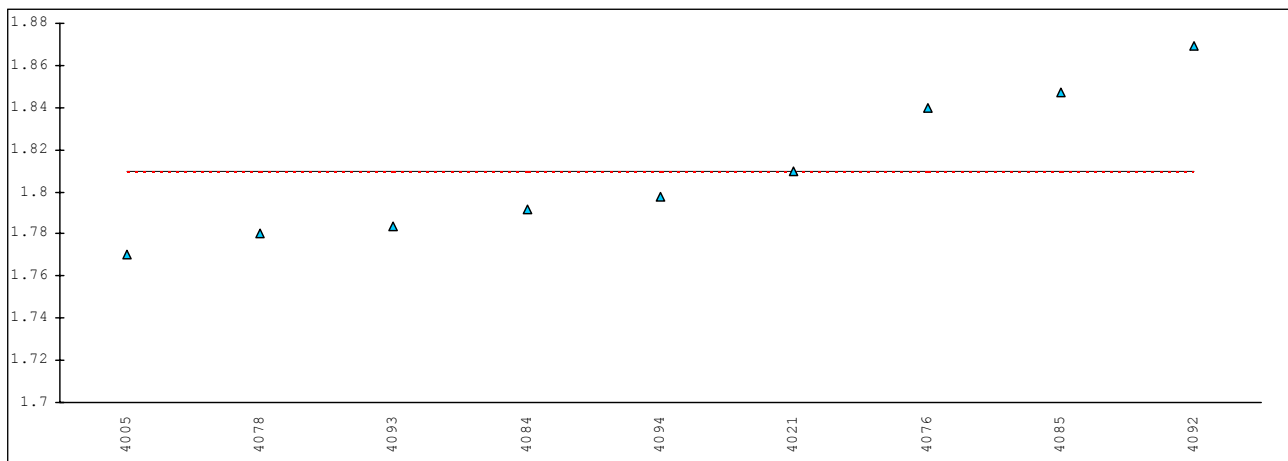
K232
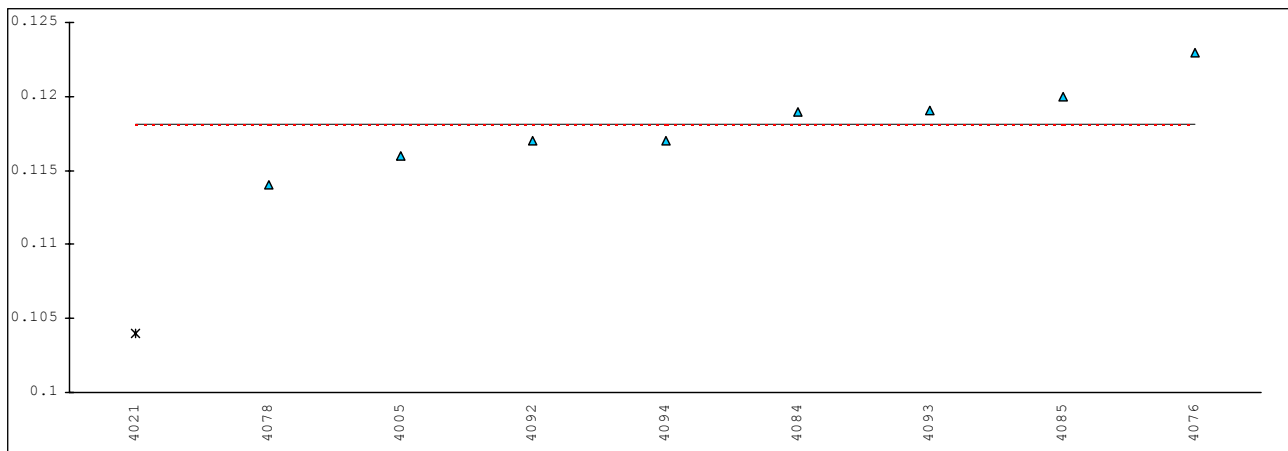
K272

Determination of Total Erythrodiols content in sample 0228: results in %M/M

lab	method	Erythrodiol	mark	Z(targ)	Uvaol	mark	Z(targ)
2100		-----		-----	-----		-----
3102		-----		-----	-----		-----
3103		-----		-----	-----		-----
3138		-----		-----	-----		-----
3139		-----		-----	-----		-----
4005		-----		-----	-----		-----
4017		-----		-----	-----		-----
4021		-----		-----	-----		-----
4037		-----		-----	-----		-----
4076	EEC2568	1.53		-----	0.56		-----
4078	AOCS	1.22		-----	0.21		-----
4082		-----		-----	-----		-----
4084	EEC2568	1.39		-----	0.06		-----
4085		-----		-----	-----		-----
4088		-----		-----	-----		-----
4092		-----		-----	-----		-----
4093		-----		-----	-----		-----
4094		-----		-----	-----		-----

normality	unknown		unknown
n	3		3
outliers	0		0
mean (n)	1.380		0.277
st.dev. (n)	0.1552		0.2576
R(calc.)	0.435		0.718
R(lit)	unknown		unknown

Determination of Trilinolein in sample 0228; results in %M/M

lab	method	value	mark	Z(targ)	remarks
2100		-----		-----	
3102		-----		-----	
3103		-----		-----	
3138		-----		-----	
3139		-----		-----	
4005		-----		-----	
4017		-----		-----	
4021		-----		-----	
4037		-----		-----	
4076	EEC2568	0.32		-----	
4078	AOCS Ce5b	0.12		-----	
4082		-----		-----	
4084		-----		-----	
4085	EEC2568	0.169		-----	
4088		-----		-----	
4092		-----		-----	
4093		-----		-----	
4094		-----		-----	

normality	unknown
n	3
outliers	0
mean (n)	0.203
st.dev. (n)	0.1043
R(calc.)	0.292
R(lit)	unknown

Determination of Triacylglycerols as delta ECN42 in sample 0228; results in mol%

lab	method	value	mark	Z(targ)	remarks
2100		-----		-----	
3102		-----		-----	
3103		-----		-----	
3138		-----		-----	
3139		-----		-----	
4005		-----		-----	
4017		-----		-----	
4021		-----		-----	
4037		-----		-----	
4076	EEC2568	0.05		-----	
4078	EEC2472	0.05		-----	
4082		-----		-----	
4084	EEC2472	0.02		-----	
4085	EEC2568	0.041		-----	
4088		-----		-----	
4092		-----		-----	
4093		-----		-----	
4094		-----		-----	

|	
normality	unknown
n	4
outliers	0
mean (n)	0.040
st.dev. (n)	0.0142
R(calc.)	0.040
R(lit)	unknown

Determination of Total Stigmastadien content in samples 0228 results in %M/M

lab	method	value	mark	Z(targ)	remarks
2100		-----		-----	
3102		-----		-----	
3103		-----		-----	
3138		-----		-----	
3139		-----		-----	
4005		-----		-----	
4017		-----		-----	
4021		-----		-----	
4037		-----		-----	
4076	EEC2568	0.0221		-----	
4078	EEC2568	0.04		-----	
4082		-----		-----	
4084		-----		-----	
4085		-----		-----	
4088		-----		-----	
4092		-----		-----	
4093		-----		-----	
4094		-----		-----	

normality	n.a.
n	2
outliers	0
mean (n)	n.a.
st.dev. (n)	n.a.
R(calc.)	n.a.
R(lit)	unknown

Determination of Water in sample 0228; results in %M/M

lab	method	value	mark	Z(targ)	remarks
2100	ISO8534	0.11	C	4.01	first reported <0.01
3102		-----		-----	
3103		-----		-----	
3138		-----		-----	
3139		-----		-----	
4005	KF	0.086	C	-0.19	first reported 0.863
4017	KF	0.09557		1.49	
4021	AOCS Ca2e-84	0.09		0.51	(KF)
4037	KF	0.105		3.14	
4076	AOCS Ca2c-25	0.07		-2.99	(Oven)
4078	AOCS	0.07		-2.99	
4082		-----		-----	
4084		-----		-----	
4085		-----		-----	
4088	AOCS Ca2a-45	0.07		-2.99	(Dist.)
4092		-----		-----	
4093		-----		-----	
4094		-----		-----	

			Only KF data:
normality	OK		OK
n	8		5
outliers	0		0
mean (n)	0.0871		0.0973
st.dev. (n)	0.01605		0.01006
R(calc.)	0.0449		0.0282
R(ISO8534:95)	0.0160		0.0160
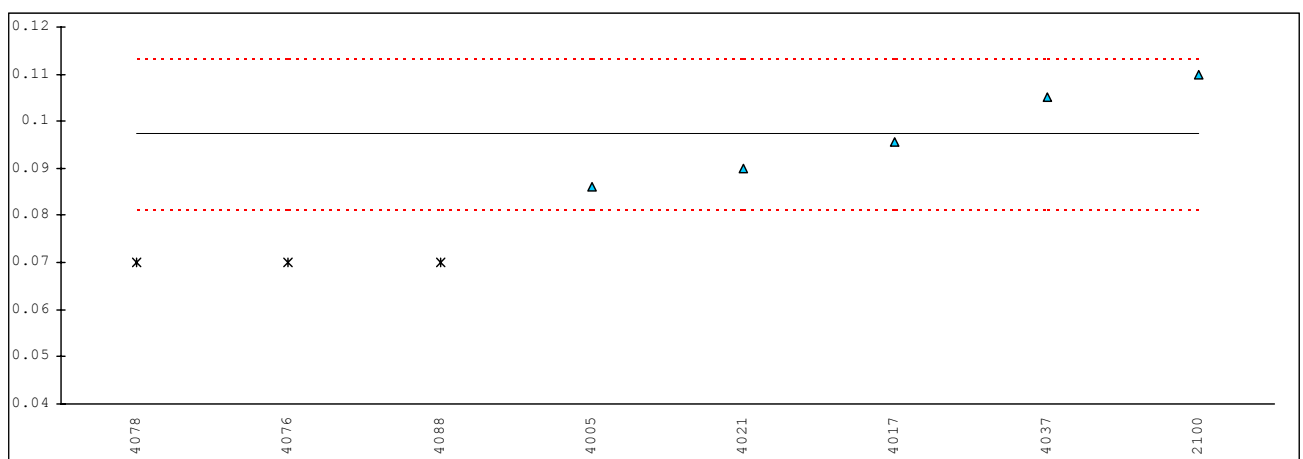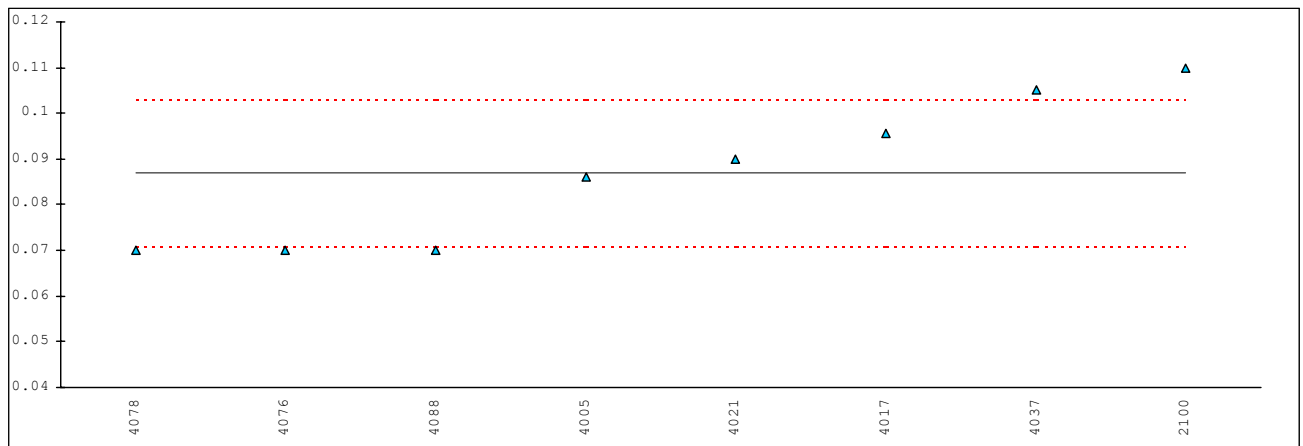
All original data
Only Karl Fisher data (KF)

APPENDIX 2

List of participating countries

Labs	Country
1 lab in	BELGIUM
1 lab in	FRANCE
6 labs in	GREECE
1 lab in	HONG KONG
1 lab in	IRELAND
1 lab in	ITALY
1 lab in	JAPAN
1 lab in	KENYA
1 labs in	PORTUGAL
1 lab in	SWEDEN
3 labs in	THE NETHERLANDS

APPENDIX 3

Abbreviations:

C	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
E	= error in calculations
ex	= excluded from calculations
n.a.	= not applicable
fr	= first reported

Literature:

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10   ISO 9936:97

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