



PRECISION VALUES OF THE METHODS OF ANALYSIS ADOPTED BY THE INTERNATIONAL OLIVE COUNCIL

In compliance with the decision taken by the International Olive Council (IOC) at its 93rd session (November 2005), the Executive Secretariat set up a working group which was given the brief of reviewing the precision values of the physico-chemical testing methods drawn up and adopted by the IOC and inserted in the trade standard for olive oils and olive-pomace oils.

The working group, which met at the IOC offices on 14 November 2006, drew up this document for consideration at the chemists' meetings in 2007.

The document reports:

- (a) The precision values for the following methods bearing the reference COI/T.20:
- COI/T.20/Doc. no. 11 – Determination of stigmastadienes in vegetable oils;
 - COI/T.20/Doc. no. 17 – Determination of *trans* unsaturated fatty acids by capillary column gas chromatography;
 - COI/T.20/Doc. no. 10 – Determination of the composition and content of sterols by capillary column gas chromatography;

In these cases, the precision values were calculated on the basis of the interlaboratory test conducted in 1998 by 19 IOC-recognised laboratories from eight countries, reported in the document referenced T.20/Doc. no. 42-2 of 9 March 2000.

- (b) The precision values for COI/T.20/Doc. no. 24 – Preparation of the fatty acid methyl esters from olive oil and olive-pomace oil;

- (c) The precision values for the following methods :
- COI/T.20/Doc. no. 20 – Determination of the difference between actual and theoretical content of triacylglycerols with ECN₄₂;
 - COI/T.20/Doc. no. 16 – Determination of sterenes in refined vegetable oils;
 - COI/T.20/Doc. no. 10 – Determination of the composition and content of sterols by capillary column gas chromatography (relative solely to brassicasterol);
 - Determination of free acidity;
 - Determination of erythrodiol + uvaol content;

- Determination of the peroxide value;
- COI/T.20/Doc. no. 24 – Preparation of the fatty acid methyl esters (relative solely to heptadecanoic acid and heptadecenoic acid)
- COI/T.20/Doc. no. 18 – Determination of wax content by capillary column gas liquid chromatography;
- COI/T.20/Doc. no. 19 – Spectrophotometric analysis in the ultraviolet.
- COI/T.20/Doc. no. 23 – Determination of the percentage of 2-glyceryl monopalmitate (2007 data);
- COI/T.20/Doc. no. 26 – Determination of aliphatic alcohols content by capillary gas chromatography;

These have been calculated from the data for 2000–2006 supplied by the laboratories of 13 countries for earning entitlement under the IOC recognition scheme. The results underwent statistical analysis according to ISO 5725 “Accuracy (trueness and precision) of measurement methods and results” and with the aid of the AOAC Statistical Manual (W.J. Youden, E.H. Steiner). Outliers were detected by applying the Ranking, Cochran and Grubbs tests to the laboratory results for all the samples (replicates a and b) .

The tables on the next pages report the following data for each parameter studied:

n	number of laboratories which participated in the test
outliers	number of laboratories with outlying values
mean	mean of the accepted results
r	repeatability
S_r	repeatability standard deviation
RSD_r(%)	repeatability coefficient of variation ($S_r \times 100 / \text{mean}$)
R	reproducibility
S_R	reproducibility standard deviation
RSD_R(%)	reproducibility coefficient of variation ($S_R \times 100 / \text{mean}$)

Table 1. Waxes (mg/kg)

	A	B	C	D	E	F	G	H
n	44	48	48	48	49	49	49	53
outliers	2	7	1	4	1	2	6	8
mean	2607.7	87.9	1564.1	167.8	660.4	738.1	63.88	825.8
r	100.6	9.4	80.4	12.0	24.9	24.5	6.7	20.4
S_r	35.9204	3.3663	28.7059	4.2734	8.9020	8.7594	2.3812	7.2883
RSD_r(%)	1.4	3.8	1.8	2.6	1.4	1.2	3.7	0.9
R	679.4	34.3	311.96	49.9	164.8	163.6	25.6	122.7
S_R	242.6280	12.2568	111.41	17.8225	58.8404	58.4183	9.1457	43.8244
RSD_R(%)	9.3	13.9	7.1	10.6	8.9	7.9	14.3	5.3

Table 2. Δ ECN₄₂

	A	B	C	D	E
n	29	29	29	29	29
outliers	1	3	7	1	3
mean	0.05	0.70	0.18	0.26	0.49
r	0.02	0.03	0.02	0.04	0.04
S_r	0.0074	0.0122	0.0084	0.0136	0.0147
RSD_r(%)	14.6	1.7	4.6	5.3	3.0
R	0.08	0.35	0.14	0.15	0.13
S_R	0.0269	0.1236	0.0515	0.0530	0.0459
RSD_R(%)	52.8	17.7	28.2	20.5	9.3

Table 3. Stigmastadienes (mg/kg)

	A	B	C	D	E
n	19	19	19	19	19
outliers	3	5	7	2	5
mean	0.01	0.8	9.5	0.22	7.6
r	0.01	0.1	0.4	0.05	0.5
S_r	0.004	0.03	0.14	0.01	0.17
RSD_r(%)	32.4 _(not sig.)	3.7	1.5	8.4	2.3
R	0.03	0.2	2	0.1	2
S_R	0.012	0.05	0.59	0.025	0.57
RSD_R(%)	98.6 _(not sig.)	6.7	6.3	11.5	7.6

Table 4. Sterenes (mg/kg)

	A	B	C	D	E
n	31	31	31	31	31
outliers	4	4	5	4	4
mean	9.5	31	46	9.0	11.4
r	0.2	1	1	0.3	0.5
S_r	0.0730	0.3276	0.3924	0.0984	0.1778
RSD_r(%)	0.8	1.0	0.9	1.1	1.6
R	2	5	12	1	1
S_R	0.5802	1.6758	4.1960	0.5293	0.4974
RSD_R(%)	6.1	5.3	9.1	5.9	4.4

Table 5. C18:1T (%)

	A	B	C	D	E
n	17	17	17	17	17
outliers	5	2	4	0	0
mean	0.01	0.02	0.02	0.02	0.05
r	0.01	0.01	0.01	0.01	0.02
S_r	0.003	0.003	0.004	0.004	0.007
RSD_r(%)	22.6	20.2	32.1	22.0	14.1
R	0.01	0.02	0.02	0.02	0.03
S_R	0.004	0.008	0.008	0.008	0.011
RSD_R(%)	31.0	46.6	32.1	50.0	21.7

Table 6. C18:2T + C18:3T(%)

	A	B	C	D	E
n	16	17	16	17	17
outliers	5	2	2	4	0
mean	0.01	0.03	0.01	0.04	0.30
r	0.01	0.01	0.01	0.01	0.04
S_r	0.004	0.004	0.003	0.004	0.015
RSD_r(%)	34.9	13.4	19.0	9.9	5.1
R	0.02	0.02	0.02	0.02	0.1
S_R	0.006	0.007	0.006	0.008	0.047
RSD_R(%)	47.7	25.6	42.8	22.4	15.4

Table 7. Total sterols (mg/kg)

	A	B	C	D	E
n	19	19	19	19	19
outliers	5	3	4	5	3
mean	1547	1720	1618	1498	1578
r	86	75	57	52	61
S_r	30.57	26.81	20.35	18.68	21.72
RSD_r(%)	2.0	1.6	1.3	1.3	1.4
R	95	182	157	164	155
S_R	34.09	64.94	56.02	58.66	55.38
RSD_R(%)	2.2	3.8	3.5	3.9	3.5

Percentage composition of sterol fraction

Table 8. Cholesterol (%)

	A	B	C	D	E
n	17	17	17	17	17
outliers	2	2	4	5	3
mean	0.2	0.2	0.16	0.1	0.16
r	0.1	0.1	0.04	0.06	0.04
S_r	0.03	0.03	0.01	0.02	0.01
RSD_r(%)	19.4	17.4	11.1	17.6	10.5
R	0.2	0.2	0.1	0.1	0.2
S_R	0.07	0.08	0.05	0.04	0.05
RSD_R(%)	43.4	42.9	31.8	35.3	34.2

Table 9. Brassicasterol (%)

	A	B	C	D	E
n	35	35	35	35	35
outliers	3	2	2	6	6
mean	1.76	4.6	0.07	0.07	0.02
r	0.04	0.1	0.02	0.02	0.01
S_r	0.0135	0.0376	0.0064	0.0068	0.0032
RSD_r(%)	0.8	0.8	9.8	9.9	19.0
R	0.1	0.3	0.1	0.1	0.1
S_R	0.0442	0.1122	0.0377	0.0427	0.0208
RSD_R(%)	2.5	2.5	57.4	62.1	123.1

Table 10. Campesterol (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	0	0	0	0	0
mean	4.1	5.0	4.0	4.0	4.3
r	0.1	0.2	0.2	0.2	0.1
S_r	0.05	0.07	0.07	0.05	0.04
RSD_r(%)	1.3	1.5	1.8	1.4	1.0
R	0.3	0.3	0.3	0.2	0.3
S_R	0.09	0.12	0.09	0.07	0.10
RSD_R(%)	2.3	2.5	2.5	1.8	2.4

Table 11. Stigmasterol (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	1	0	0	1	0
mean	1.0	2.3	1.1	1.4	2.4
r	0.1	0.1	0.1	0.1	0.2
S_r	0.02	0.04	0.03	0.03	0.05
RSD_r(%)	1.9	1.9	30.6	2.2	2.3
R	0.1	0.2	0.1	0.1	0.2
S_R	0.04	0.07	0.04	0.04	0.07
RSD_R(%)	3.9	3.1	4.6	2.9	3.1

Table 12. Δ -7-Stigmastenol (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	0	0	0	1	0
mean	0.1	2.3	0.2	0.5	0.6
r	0.1	0.2	0.1	0.1	0.1
S_r	0.03	0.06	0.02	0.03	0.03
RSD_r(%)	24.7	2.7	15.3	6.4	5.3
R	0.1	0.3	0.1	0.1	0.1
S_R	0.04	0.09	0.04	0.04	0.04
RSD_R(%)	30.7	4.3	22.6	7.9	8.5

Table 13. Apparent β -sitosterol (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	0	1	0	0	0
mean	93.9	88.7	93.9	93.2	91.6
r	0.3	0.4	0.4	0.3	0.3
S_r	0.10	0.13	0.13	0.10	0.12
RSD_r(%)	0.1	0.2	0.1	0.1	0.1
R	0.9	0.9	0.7	0.9	0.9
S_R	0.31	0.32	0.25	0.32	0.32
RSD_R(%)	0.3	0.4	0.3	0.4	0.4

Spectrophotometric analysis in the ultraviolet

Table 14. K₂₇₀

	A	B	C	D	E	F	G	H
n	44	48	48	49	51	51	52	51
outliers	5	3	4	3	2	4	1	1
mean	0.12	0.14	0.12	0.24	0.20	0.39	0.40	0.46
r	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.02
S_r	0.0026	0.0024	0.0018	0.0025	0.0024	0.0035	0.0047	0.0054
RSD_r(%)	2.4	1.7	1.6	1.0	1.2	0.9	1.2	1.2
R	0.03	0.03	0.03	0.03	0.02	0.03	0.06	0.05
S_R	0.0090	0.0092	0.0090	0.0101	0.0067	0.0095	0.0207	0.0162
RSD_R(%)	8.3	6.7	7.8	4.2	3.3	2.5	5.2	3.5

Table 15. ΔK

	A	B	C	D	E	F	G	H
n	17	18	18	18	18	48	49	51
outliers	1	2	0	1	1	6	5	2
mean	0.000	0.020	0.010	0.003	0.04	0.002	0.006	0.019
r	-	0.001	0.003	0.002	0.007	0.001	0.002	0.003
Sr	-	0.001	0.001	0.01	0.003	0.0004	0.0006	0.0010
RSDr(%)	-	7.7	17.9	25.7	6.4	26.8	9.1	5.0
R	-	0.004	0.01	0.004	0.02	0.003	0.004	0.012
SR	-	0.002	0.002	0.002	0.006	0.0009	0.0015	0.0042
RDS_R(%)	-	8.3	37.0	50.2	14.	58.3	24.3	21.9

Table 16. Glyceryl monopalmitate (%)

	A	B	C	D	E	F	G	H
n	12	12	12	12	12			46
outliers	0	0	0	0	0			2
mean	0.5	0.8	0.9	1.8	2.8			2.0
r	0.1	0.1	0.2	0.1	0.3			0.1
Sr	0.0408	0.0408	0.0612	0.0354	0.0935			0.0532
RSDr(%)	8.9	5.	6.8	2.0	3.3			2.7
R	0.1	0.	0.3	1	1			0.4
SR	0.0508	0.950	0.0919	0.2006	0.3068			0.1357
RDS_R(%)	11.1	12.7	10.2	11.1	10.9			6.8

Table 17. Aliphatic alcohols (mg/kg)

	A	B	C	D	E	F	G	H
n				47	48	47	53	50
outliers				6	3	3	6	4
mean				128.50	173.85	105.44	1441.57	241.29
r				10.70	9.57	10.74	98.55	15.64
S_r				3.8209	3.4194	3.8341	35.1963	5.5844
RSD_r(%)				3.0	2.0	3.6	2.4	2.3
R				49.05	54.87	36.46	472.88	56.51
S_R				17.5175	19.5964	13.0222	168.8854	20.1832
RSD_R(%)				13.6	11.3	12.4	11.7	8.4

Table 18. Acidity (%)

	A	B	C	D	E
n	35	35	35	35	35
outliers	2	2	2	0	1
mean	0.41	0.20	3.91	1.32	0.98
r	0.02	0.01	0.04	0.02	0.02
S_r	0.0064	0.0043	0.0152	0.0071	0.0069
RSD_r(%)	1.6	2.1	0.4	0.5	0.7
R	0.05	0.05	0.2	0.1	0.1
S_R	0.0162	0.0177	0.0785	0.0330	0.0209
RSD_R(%)	4.0	8.7	2.0	2.5	2.1

Table 19. Erythrodiol + uvaol

	A	B	C	D	E
n	30	30	30	30	30
outliers	4	0	5	1	4
mean	6.6	5.9	4.5	2.0	1.9
r	0.2	0.3	0.1	0.1	0.1
S_r	0.0674	0.0988	0.0378	0.0475	0.0338
RSD_r(%)	1.0	1.7	0.9	2.3	1.8
R	1	1	1	1	1
S_R	0.3927	0.3645	0.4428	0.2842	0.1846
RSD_R(%)	6.0	6.2	10.0	14.0	10.0

Table 20. Peroxide value (meqO₂/kg)

	A	B	C	D	E	F	G	H
n	49	48	48	49	49	51	51	52
outliers	5	9	0	1	2	4	3	3
mean	10.48	3.32	9.45	12.55	9.03	17.75	3.55	3.10
r	0.43	0.32	0.51	0.47	0.38	0.49	0.26	0.33
S_r	0.1542	0.1137	0.1836	0.1672	0.1355	0.762	0.0915	0.1163
RSD_r(%)	1.5	3.4	1.9	1.3	1.5	1.0	2.6	3.8
R	2.29	2.24	3-28	2.82	2.04	3.19	1.85	1.78
S_R	0.8165	0.7996	1.1697	1.0065	0.7288	1.1389	0.6610	0.6354
RSD_R(%)	7.8	24.1	12.4	8.0	8.1	6.4	18.6	20.5

Percentage fatty acid composition

Table 21. Myristic acid C14:0 (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	0	0	1	1	3
mean	0.01	0.01	0.01	0.01	0.02
r	0.01	0.01	0.01	0.01	0.01
S_r	0.002	0.003	0.004	0.004	0.002
RSD_r(%)	20	20	36	38	11
R	0.01	0.02	0.02	0.01	0.02
S_R	0.0041	0.0059	0.0062	0.0047	0.0058
RSD_R(%)	45	47	52	42	32

Table 22. Palmitic acid C16:0 (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	2	3	1	0	0
mean	8.0	10.3	10.4	10.5	9.7
r	0.1	0.2	0.4	0.3	0.4
S_r	0.04	0.06	0.15	0.10	0.14
RSD_r(%)	0.5	0.6	1.5	1.0	1.4
R	1	0.4	1	1	1
S_R	0.24	0.16	0.33	0.46	0.45
RSD_R(%)	3.0	1.5	3.2	4.4	4.7

Table 23. Palmitoleic acid C16:1 (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	0	2	0	1	1
mean	0.50	0.68	0.7	0.91	0.64
r	0.04	0.03	0.1	0.03	0.04
S_r	0.014	0.010	0.026	0.012	0.014
RSD_r(%)	2.9	1.4	3.6	1.3	2.3
R	0.1	0.1	0.1	0.1	0.1
S_R	0.034	0.027	0.047	0.44	0.046
RSD_R(%)	6.8	4.1	6.4	4.9	7.2

Table 24. Heptadecanoic acid C17:0 (%)

	A	B	C	D	E
n	25	25	25	25	25
outliers	1	1	1	2	2
mean	0.18	0.06	0.11	0.14	0.12
r	0.01	0.01	0.01	0.01	0.01
S_r	0.0048	0.0041	0.0035	0.0033	0.0033
RSD_r(%)	2.7	6.9	3.1	2.3	2.7
R	0.02	0.02	0.02	0.02	0.03
S_R	0.0073	0.0074	0.0087	0.0075	0.0096
RSD_R(%)	4.1	12.6	7.7	5.2	7.8

Table 25. Heptadecenoic acid C17:1 (%)

	A	B	C	D	E
n	29	29	29	29	29
outliers	3	2	2	3	2
mean	0.26	0.09	0.24	0.22	0.19
r	0.01	0.01	0.01	0.01	0.01
S_r	0.0037	0.0036	0.0049	0.0048	0.0043
RSD_r(%)	1.4	3.8	2.0	2.2	2.2
R	0.03	0.03	0.04	0.03	0.03
S_R	0.0110	0.0095	0.0147	0.0107	0.0110
RSD_R(%)	4.3	10.1	6.1	4.9	5.7

Table 26. Stearic acid C18:0 (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	2	0	0	0	1
mean	2.9	2.49	2.6	3.5	3.1
r	0.1	0.03	0.1	0.1	0.1
S_r	0.032	0.012	0.030	0.034	0.038
RSD_r(%)	1.1	0.5	1.1	1.0	1.2
R	0.2	0.3	0.2	0.4	0.3
S_R	0.061	0.092	0.088	0.131	0.117
RSD_R(%)	2.1	3.7	3.4	3.8	3.8

Table 27. Oleic acid C18:1 (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	0	0	1	1	0
mean	79.4	74.6	75.6	76.1	75.8
r	0.4	0.3	0.4	0.2	0.5
S_r	0.15	0.11	0.14	0.08	0.16
RSD_r(%)	0.2	0.2	0.2	0.1	0.2
R	1	1	1	1	2
S_R	0.49	0.45	0.45	0.47	0.64
RSD_R(%)	0.6	0.6	0.6	0.6	0.9

Table 28. Linoleic acid C18:2 (%)

	A	B	C	D	E
N	15	15	15	15	15
Outliers	2	1	0	1	0
Mean	7.3	9.7	8.5	7.2	8.8
R	0.1	0.1	0.2	0.1	0.1
S_r	0.02	0.03	0.06	0.04	0.05
RSD_r(%)	0.3	0.3	0.7	0.6	0.6
R	0.3	0.5	0.5	0.5	0.6
S_R	0.12	0.19	0.18	0.16	0.21
RSD_R(%)	1.7	1.9	2.1	2.2	2.4

Table 29. Linolenic acid C18:3 (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	2	0	0	0	4
mean	0.73	0.90	0.86	0.74	0.8
r	0.04	0.05	0.03	0.04	0.1
S_r	0.013	0.017	0.010	0.014	0.020
RSD_r(%)	1.8	1.9	1.2	1.9	2.6
R	0.1	0.1	0.1	0.1	0.1
S_R	0.029	0.041	0.036	0.028	0.041
RSD_R(%)	3.9	4.6	4.2	3.8	5.4

Table 30. Arachidic acid C20:0 (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	1	0	0	1	0
mean	0.39	0.44	0.44	0.42	0.43
r	0.04	0.05	0.04	0.04	0.05
S_r	0.015	0.018	0.013	0.013	0.019
RSD_r(%)	3.8	4.0	3.0	3.1	4.4
R	0.1	0.1	0.1	0.1	0.1
S_R	0.029	0.032	0.031	0.042	0.036
RSD_R(%)	7.3	7.2	7.0	9.8	8.6

Table 31. Eicosenoic acid C20:1 (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	1	1	1	0	1
mean	0.37	0.39	0.37	0.28	0.3
r	0.03	0.03	0.04	0.05	0.1
S_r	0.009	0.011	0.013	0.017	0.026
RSD_r(%)	7.8	3.0	3.5	6.0	8.9
R	0.1	0.1	0.1	0.1	0.1
S_R	0.029	0.034	0.023	0.028	0.027
RSD_R(%)	7.9	8.7	6.2	10	9.3

Table 32. Behenic acid C22:0 (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	0	1	1	1	3
mean	0.11	0.14	0.14	0.12	0.19
r	0.02	0.04	0.04	0.05	0.04
S_r	0.008	0.013	0.014	0.016	0.013
RSD_r(%)	7.0	9.6	10.0	14.0	6.9
R	0.04	0.04	0.05	0.1	0.04
S_R	0.014	0.016	0.018	0.020	0.015
RSD_R(%)	12.0	12.0	13.0	17.0	8.3

Table 33. Lignoceric acid C24:0 (%)

	A	B	C	D	E
n	15	15	15	15	15
outliers	1	0	0	0	3
mean	0.04	0.06	0.06	0.05	0.08
r	0.02	0.02	0.03	0.03	0.04
S_r	0.006	0.005	0.012	0.012	0.014
RSD_r(%)	15.0	8.9	20.0	24.0	19.0
R	0.1	0.1	0.1	0.1	0.04
S_R	0.020	0.026	0.026	0.019	0.014
RSD_R(%)	49	42	45	39	19

3. References

ISO 5725 – Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions.

ISO 5725 - Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.

ISO 5725 – Accuracy (trueness and precision) of measurement methods and results – Part 5: Alternative methods for the determination of the precision of a standard measurement method.

ISO 5725 – Accuracy (trueness and precision) of measurement methods and results – Part 6: Use in practice of accuracy values.

AOAC - Statistical Manual of the Association of Official Analytical Chemists.
W.J. Youden. E.H. Steiner