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AMENDMENT 1
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Animal and vegetable fats and oils — Determination of trace elements by inductively coupled plasma optical emission spectroscopy (ICP–OES)

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Corps gras d'origines animale et végétale — Détermination des éléments traces par spectrométrie d'émission optique à plasma induit par haute fréquence (ICP–OES)

AMENDEMENT 1

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Amendment 1 to ISO/TS 21033:2011 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

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AMENDMENT 1

Page 3, 9.1.3

Most instruments no longer have a mercury lamp and do not use the procedure as written. Most modern instruments have a resolution of 0,005 nm to 0,015 nm, so a more precise indication of the appropriate lines is required. The data for P (taken directly from the AOCS Official Method Ca 20-99[7]) and S should be added to the table. Thus, this subclause and Table 1 should be replaced with the following.

9.1.3 The instrument is ignited and allowed to warm. Profile the instrument according to the manufacturer's instructions. Elements can be detected at the major emission lines (see Table 1). Additional emission lines and equipment set up instructions are given in EN 14538:2006[6].

Table 1 — Major emission lines and limits of detection

Element	Current limits of detection mg/kg	Major emission line(s) nm		
Aluminium	—[a]	167,078	308,215	
Barium	—[a]	455,404		
Boron	—[a]	249,773		
Cadmium	—[a]	226,502	214,441	228,802
Calcium	0,05	315,887	393,366	317,933
Chrome	—[a]	267,716	284,325	283,563
Copper	0,05	324,754	327,396	
Iron	0,05	259,940	238,204	261,187
Lead	—[a]	220,353		
Magnesium	0,05	285,213	279,553	280,270
Manganese	—[a]	257,611	259,373	
Molybdenum	—[a]	281,615	202,095	
Nickel	0,05	231,604	221,648	341,476
Phosphorus	0,05	213,618	178,287	177,495
Silicon	0,1	251,611	288,158	
Silver	—[a]	328,068		
Sodium	0,1	588,995		
Sulfur	1	180,731		
Tin	—[a]	242,949		
Titanium	—[a]	334,941	323,452	336,121
Vanadium	—[a]	309,311	311,071	
Zinc	—[a]	202,613	213,856	

[a] Not reported at the time of publication.

Page 4, 9.2.2

Replace the third paragraph with the following text.

> Instruments that are capable of simultaneous detection of multiple elements can achieve improved precision and accuracy by the inclusion of an internal standard in the analysis. If an internal standard is used, it should be incorporated as part of the dilution step. Typically, the resultant dilution should contain 10 mg/kg yttrium or scandium. Thus, under the dilution sequence for sampling (see 9.1.1 and 9.4), the diluent should contain 20 mg/kg internal standard yielding 10 mg/kg yttrium or scandium in the 1 + 1 dilution to accomplish this.

Page 4, Clause 10

Replace the text in this clause with the following.

> Computation is a feature of most instrument programs. Area counts from known standards are inserted into a linear regression formula versus the sample concentration. From this relationship, concentrations may be determined from the area counts of the samples.
>
> Simultaneous instruments can usually accommodate internal standard calculations. If an internal standard is used, the ratio of the counts from the standard divided by the area counts of the internal standard are inserted into the linear regression formula.
>
> It is important to include the correct dilution factor.

Page 6, Annex A

Add a new heading under the annex title.

A.1 AOCS trial, 1999

Add A.2 after Table A.1.

A.2 DGF collaborative trial, 2011

A.2.1 Data from most participating laboratories

Further precision data were established through a collaborative trial conducted in 2011 by the DGF (Deutsche Gesellschaft für Fettwissenschaft). Data are analysed according to ISO 5725.1:1994[2] and ISO 5725-2:1994[3] guidelines. In this trial, 14 laboratories from Austria (1), Germany (12), and the USA (1) participated, but not all laboratories analysed all elements.

Tables A.2 to A.9 list the different levels of Al, Ca, Cd, Na, Cr, Ni, Cu, Mg, Fe, P, Pb, Si, and Zn.

Table A.2 — Results for different levels of aluminium and calcium

Parameter	Sample					
	Al	Ca				
Number of participating laboratories, N_1	8	12	12	12	12	12
Number of laboratories retained after eliminating outliers, n	8	11	10	11	12	12
Number of individual test results of all laboratories on each sample	16	22	20	22	24	24
Mean value, $\bar{w}$, mg/kg	**0,120**	**0,064**	**0,082**	**0,114**	**1,384**	**25,237**
Repeatability standard deviation, s_r, mg/kg	0,009	0,003	0,006	0,006	0,043	0,397
Repeatability coefficient of variation, $C_{V,r}$, %	7,2	4,1	7,3	4,9	3,1	1,6
Repeatability limit, r ($s_r \times 2{,}8$), mg/kg	**0,024**	**0,007**	**0,017**	**0,016**	**0,122**	**1,110**
Reproducibility standard deviation, s_R, mg/kg	0,049	0,029	0,016	0,017	0,098	1,360
Reproducibility coefficient of variation, $C_{V,R}$, %	40,7	45,0	19,7	14,8	7,1	5,4
Reproducibility limit, R ($s_R \times 2{,}8$), mg/kg	**0,137**	**0,080**	**0,045**	**0,047**	**0,273**	**3,809**

Table A.3 — Results for different levels of cadmium and sodium

Parameter	Sample					
	Cd				Na	
Number of participating laboratories, N_1	9	9	9	9	11	10
Number of laboratories retained after eliminating outliers, n	8	9	9	9	8	9
Number of individual test results of all laboratories on each sample	16	18	18	18	16	18
Mean value, $\bar{w}$, mg/kg	**0,037**	**0,045**	**0,081**	**0,117**	**0,807**	**1,026**
Repeatability standard deviation, s_r, mg/kg	0,004	0,004	0,004	0,006	0,016	0,111
Repeatability coefficient of variation, $C_{V,r}$, %	11,7	9,1	4,3	4,9	2,0	10,8
Repeatability limit, r ($s_r \times 2{,}8$), mg/kg	**0,012**	**0,011**	**0,010**	**0,016**	**0,046**	**0,310**
Reproducibility standard deviation, s_R, mg/kg	0,006	0,011	0,014	0,017	0,232	0,252
Reproducibility coefficient of variation, $C_{V,R}$, %	16,6	25,1	16,8	14,5	28,8	24,6
Reproducibility limit, R ($s_R \times 2{,}8$), mg/kg	**0,017**	**0,032**	**0,038**	**0,047**	**0,650**	**0,707**

Table A.4 — Results for different levels of chromium and nickel

Parameter	Sample					
	Cr				Ni	
Number of participating laboratories, N_1	9	9	9	9	9	9
Number of laboratories retained after eliminating outliers, n	9	7	9	9	8	9
Number of individual test results of all laboratories on each sample	18	14	18	18	16	18
Mean value, $\bar{w}$, mg/kg	**0,049**	**0,053**	**0,098**	**0,314**	**0,054**	**0,097**
Repeatability standard deviation, s_r, mg/kg	0,003	0,000	0,000	0,008	0,003	0,006
Repeatability coefficient of variation, $C_{V,r}$, %	6,8	0,0	0,0	2,6	4,6	6,4
Repeatability limit, r ($s_r \times 2{,}8$), mg/kg	**0,009**	**0,000**	**0,000**	**0,023**	**0,007**	**0,017**
Reproducibility standard deviation, s_R, mg/kg	0,007	0,013	0,012	0,019	0,012	0,017
Reproducibility coefficient of variation, $C_{V,R}$, %	14,2	23,7	12,3	5,9	21,9	17,7
Reproducibility limit, R ($s_R \times 2{,}8$), mg/kg	**0,019**	**0,035**	**0,034**	**0,052**	**0,033**	**0,048**

Table A.5 — Results for different levels of copper and magnesium

Parameter	Sample					
	Cu		Mg			
Number of participating laboratories, N_1	12	12	13	13	13	13
Number of laboratories retained after eliminating outliers, n	12	12	12	13	13	13
Number of individual test results of all laboratories on each sample	24	24	24	26	26	26
Mean value, $\bar{w}$, mg/kg	**0,060**	**0,066**	**0,070**	**0,102**	**0,458**	**5,794**
Repeatability standard deviation, s_r, mg/kg	0,004	0,007	0,004	0,005	0,016	0,124
Repeatability coefficient of variation, $C_{V,r}$, %	5,9	10,2	5,1	4,7	3,4	2,1
Repeatability limit, r ($s_r \times 2{,}8$), mg/kg	**0,010**	**0,019**	**0,010**	**0,013**	**0,044**	**0,348**
Reproducibility standard deviation, s_R, mg/kg	0,026	0,028	0,028	0,027	0,038	0,270
Reproducibility coefficient of variation, $C_{V,R}$, %	43,1	41,6	39,6	27,1	8,3	4,7
Reproducibility limit, R ($s_R \times 2{,}8$), mg/kg	**0,073**	**0,077**	**0,077**	**0,077**	**0,107**	**0,756**

Table A.6 — Results for different levels of iron

Parameter	Sample				
	Fe				
Number of participating laboratories, N_1	12	12	12	12	12
Number of laboratories retained after eliminating outliers, n	11	11	12	10	11
Number of individual test results of all laboratories on each sample	22	22	24	20	22
Mean value, $\bar{w}$, mg/kg	**0,055**	**0,060**	**0,095**	**0,213**	**0,317**
Repeatability standard deviation, s_r, mg/kg	0,000	0,005	0,008	0,007	0,008
Repeatability coefficient of variation, $C_{V,r}$, %	0,0	8,8	8,4	3,4	2,5
Repeatability limit, r ($s_r \times 2{,}8$), mg/kg	**0,000**	**0,015**	**0,022**	**0,020**	**0,022**
Reproducibility standard deviation, s_R, mg/kg	0,025	0,022	0,014	0,018	0,016
Reproducibility coefficient of variation, $C_{V,R}$, %	45,2	36,6	14,5	8,5	5,1
Reproducibility limit, R ($s_R \times 2{,}8$), mg/kg	**0,070**	**0,061**	**0,038**	**0,051**	**0,045**

Table A.7 — Results for different levels of phosphorus

Parameter	Sample				
	P				
Number of participating laboratories, N_1	11	11	11	11	11
Number of laboratories retained after eliminating outliers, n	11	10	11	11	11
Number of individual test results of all laboratories on each sample	22	20	22	22	22
Mean value, $\bar{w}$, mg/kg	**0,977**	**3,335**	**3,464**	**6,235**	**43,150**
Repeatability standard deviation, s_r, mg/kg	0,032	0,092	0,076	0,098	0,500
Repeatability coefficient of variation, $C_{V,r}$, %	3,3	2,7	2,2	1,6	1,2
Repeatability limit, r ($s_r \times 2{,}8$), mg/kg	**0,090**	**0,256**	**0,212**	**0,273**	**1,401**
Reproducibility standard deviation, s_R, mg/kg	0,236	0,197	0,343	0,463	3,203
Reproducibility coefficient of variation, $C_{V,R}$, %	24,2	5,9	9,9	7,4	7,4
Reproducibility limit, R ($s_R \times 2{,}8$), mg/kg	**0,661**	**0,553**	**0,961**	**1,298**	**8,970**

Table A.8 — Results for different levels of lead and silicon

Parameter	Sample				
	Pb	Si			
Number of participating laboratories, N_1	8	9	9	9	9
Number of laboratories retained after eliminating outliers, n	8	8	8	7	9
Number of individual test results of all laboratories on each sample	16	16	16	14	18
Mean value, $\overline{w}$, mg/kg	**0,090**	**0,035**	**0,043**	**0,088**	**0,515**
Repeatability standard deviation, s_r, mg/kg	0,003	0,004	0,003	0,007	0,033
Repeatability coefficient of variation, $C_{V,r}$, %	3,5	10,9	6,6	7,5	6,4
Repeatability limit, r ($s_r \times 2{,}8$), mg/kg	**0,009**	**0,011**	**0,008**	**0,019**	**0,093**
Reproducibility standard deviation, s_R, mg/kg	0,043	0,018	0,021	0,010	0,067
Reproducibility coefficient of variation, $C_{V,R}$, %	47,3	51,3	49,0	11,8	13,0
Reproducibility limit, R ($s_R \times 2{,}8$), mg/kg	**0,119**	**0,051**	**0,058**	**0,029**	**0,187**

Table A.9 — Results for different levels of zinc

Parameter	Sample				
	Zn				
Number of participating laboratories, N_1	9	9	9	9	9
Number of laboratories retained after eliminating outliers, n	8	7	9	8	9
Number of individual test results of all laboratories on each sample	16	14	18	16	18
Mean value, $\overline{w}$, mg/kg	**0,038**	**0,039**	**0,072**	**0,079**	**0,162**
Repeatability standard deviation, s_r, mg/kg	0,000	0,000	0,006	0,003	0,003
Repeatability coefficient of variation, $C_{V,r}$, %	0,0	0,0	8,7	3,2	2,1
Repeatability limit, r ($s_r \times 2{,}8$), mg/kg	**0,000**	**0,000**	**0,017**	**0,007**	**0,009**
Reproducibility standard deviation, s_R, mg/kg	0,015	0,019	0,016	0,022	0,022
Reproducibility coefficient of variation, $C_{V,R}$, %	39,7	48,3	22,6	27,5	13,5
Reproducibility limit, R ($s_R \times 2{,}8$), mg/kg	**0,042**	**0,052**	**0,045**	**0,061**	**0,061**

A.2.2 Data from seven participating laboratories only

Tables A.10 to A.12 contain further data for Ag, Mn, Mo, Pb, Ti, Sn, and V, but only from seven laboratories. All of these data are free from outliers.