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

National foreword

This British Standard is the UK implementation of ISO 21033:2016. It supersedes PD ISO/TS 21033:2011+A1:2012 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee AW/307, Oilseeds, animal and vegetable fats and oils and their by-products.

A list of organizations represented on this committee can be obtained on request to its secretary.

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

Corps gras d’origines animale et végétale — Détermination des éléments traces dans les corps gras par spectrométrie d’émission optique à plasma induit par haute fréquence (ICP-OES)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This first edition cancels and replaces ISO/TS 21033:2011, which has been technically revised. It also incorporates the Amendment ISO/TS 21033:2011/Amd 1:2012. The scope has been revised to state that the document is not applicable to milk and milk fat products.

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

1 Scope

This International Standard specifies an inductively coupled plasma optical emission spectroscopic method (ICP-OES) for the determination of the trace element content in oils. Depending on the dilution solvent used, most types of vegetable oils can be analysed (crude, degummed, refined, bleached, deodorized and hardened oils) and nearly all types of lecithins and phosphatides. Milk and milk products (or fat coming from milk and milk products) are excluded from the scope of this International Standard.

This method is only suitable when the elements are present in a solubilized form. Fine particles, such as bleaching earth, metal particles and rust, can result in poor recovery of the trace elements present as nebulization and atomization problems affect the ICP-OES analysis.

NOTE The only suitable non-ashing direct method for samples containing fine particles is graphite furnace atomic absorption spectrometry.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 661, *Animal and vegetable fats and oils — Preparation of test sample*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 6353-3, *Reagents for chemical analysis — Part 3: Specifications — Second series*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
trace element
element present in very low concentrations

Note 1 to entry: Trace element content is expressed as a mass fraction in milligrams per kilogram.

4 Principle

Solvent-diluted vegetable oils are analysed for trace elements by direct aspiration. Liquid samples are nebulized and carried into the excitation source by a flowing gas. Atoms are quantified by measuring the specific emission lines produced by atoms decaying from high energy levels.

5 Reagents

WARNING — Attention is drawn to national regulations that specify the handling of hazardous substances and users' obligations thereunder. Technical, organizational and personal safety measures shall be followed.

Unless stated otherwise, use reagents specified in ISO 6353-2 and ISO 6353-3, if listed there; if not, then use reagents of recognized analytical grade.

5.1 1-Butanol, ISO 6353-3.

5.2 Kerosene.

5.3 Xylene, ISO 6353-3.

5.4 Standard elements, present in solution as an organic soluble material.1) Multi-element standards may be used.

5.5 Base oil,2) may be used to check the blank oil used and for the dilution of the standard solutions as needed.

5.6 Blank oil, typically, refined and bleached soya bean or other oil, which has been shown to be free of trace elements.

5.7 Mixture of the blank oil (5.6) with 1-butanol (5.1), mass fraction $w_{\text{(blank oil)}}$ = 50 g/100 g.

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 Inductively coupled plasma optical emission spectrometer.

6.2 Analytical balance, readability 0,000 1 g, weighing precision 0,001 g.

6.3 Oven, capable of maintaining a temperature of (60 ± 2) °C.

6.4 Tilt table mixer.

6.5 Volumetric flasks, capacity 100 ml.

7 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555.[1]

It is important that the laboratory receive a truly representative sample which has not been damaged or changed during transport or storage.

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1) SPEX products are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

2) Base 20 Oil or Base 75 Oil from Accu-Standard are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

8 Preparation of test sample

Prepare the test samples in accordance with ISO 661, except that the samples should not be clarified.

9 Procedure

9.1 General

9.1.1 Melt solid samples at approximately 10 °C above their melting point and homogenize prior to dilution. Keep the diluted sample warm and monitor throughout the analysis to ensure it remains in solution. The maximum temperature for the analysis of hardened fats is 60 °C.

9.1.2 All samples, standards and blanks are diluted (equal volume fractions) with 1-butanol (kerosene or xylene) to reduce the viscosity of the oil for better nebulization. Some samples are more soluble in 1-butanol than others.

1-Butanol is preferred because it has better moisture tolerance and allows a higher flow rate with higher pressure than kerosene without extinguishing the torch. The increased moisture tolerance permits the analysis of crude oils and lecithins without phase separation. The higher flow rate provides for improved detection limits.

If kerosene or xylene is used, all instrumental operating conditions, e.g. pump flow rate, change from those set for 1-butanol. Therefore, the analysis shall be standardized and all analyses shall be run with all standards, blank and samples dissolved in the same solvent.

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[5] and ISO 10540-3.[4]

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		

[a] Not reported at the time of publication.

Table 1 *(continued)*

Element	**Current limits of detection** mg/kg	**Major emission line(s)** nm		
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.

9.1.4 Standardize the instrument as specified in 9.3 and scan all the samples in triplicate.

NOTE Calibration drift has been noted. It can result from carbon build-up on the injector tip.

9.2 Preparation of standards

9.2.1 Blank

Typically, refined and bleached soya bean or other oil, which has been shown to be free of trace elements, is used. Blank oil is diluted (1:1) as described in the sampling procedure. Base oil (5.5) is used as an absolute reference blank to determine that the blank oil is free of trace elements.

9.2.2 Standards

The standard is prepared from commercially available single element organic-based standards. Weigh accurately the standard and add enough blank oil to total 50,00 g. Add 50,00 g of solvent (1-butanol, kerosene or xylene) to achieve a (1:1) dilution.

One standard concentration works; however, up to four multi-level, multi-element standards provide a better calibration for linearity and accuracy. Levels should include 2,5 mg/kg, 5 mg/kg and 10 mg/kg standards depending on the range of values expected.

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.

9.3 Standardization

Run the blank oil standard and diluted base oil (5.5) at the specified wavelength for the element(s) of interest.

Run the standard solutions (see 9.2.2) at the wavelength(s) chosen.

Blanks, samples and standards are scanned in triplicate for trace element(s) and are averaged.

Standards and the blank are run every 10 samples or fewer and the instrument is re-standardized as needed. For accuracy, use a narrow range of standardization (0 mg/kg to 25 mg/kg of each element). Although the linearity is somewhat greater, test samples should be diluted to keep the trace element(s) content within the range of standardization.

9.4 Preparation of samples

Weigh 2,5 g ± 0,02 g of sample into an auto-sampler tube and dilute with 2,5 g of 1-butanol (kerosene or xylene) delivered from an automatic pipette. Cap the tube and invert 40 times to 50 times on a mixing table.

Dilute 0,2 g lecithins (up to 100 % acetone insoluble) to 5,0 g with blank soybean oil (dilution factor 25) and then to 10 g with 1-butanol (5.6), dilution factor 50. Mix the samples on a tilt table mixer for 1 h. Then dilute 1 part with 10 parts of the mixture of the blank oil and 1-butanol (5.7). The total dilution factor is 500.

10 Calculation

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 is inserted into the linear regression formula.

It is important to include the correct dilution factor.

11 Precision

11.1 Interlaboratory test

Details of interlaboratory tests on the precision of the method are summarized in Annex A. Additional data are given in Annex B. The values derived from these interlaboratory tests may not be applicable to concentration ranges and matrices other than those given.

11.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will not be greater than the repeatability limit, *r*, as shown in Annex A.

11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will not be greater than the reproducibility limit, *R*, as shown in Annex A.

12 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, with reference to this International Standard, i.e. ISO 21033;

d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

e) the test result(s) obtained;

f) the final quoted result obtained if repeatability has been checked.

Annex A
(informative)

Results of interlaboratory studies

The precision of the method has been established for high levels of Cu, Fe and Ni in soybean oil by an international study conducted in 1999 by AOCS[6] (see Table A.1).

Further precision data were established through a collaborative trial conducted in 2011 by the DGF (Deutsche Gesellschaft für Fettwissenschaft).[7] In this trial, 14 laboratories from Austria (1), Germany (12) and the USA (1) participated, but not all laboratories analysed all elements. Table A.2 to Table A.9 contain the summary of different levels of aluminium, calcium, cadmium, sodium, chrome, nickel, copper, magnesium, iron, phosphorus, lead, silicon and zinc.

Additional precision data for high and low levels of phosphorus in oil from an international study conducted in 1998/1999 by AOCS and FOSFA International are also given in ISO 10540-3:2002, Tables A.1 and A.2.[4]

Data are analysed according to ISO 5725-1[2] and ISO 5725-2[3] guidelines.

Table A.1 — ICP analysis of high levels of trace element(s) in oil: Summary of analyses from 11 laboratories

	Copper				**Iron**				**Nickel**			
	S-1	**S-2**	**S-3**	**S-4**	**S-5**	**S-6**	**S-7**	**S-8**	**S-9**	**S-10**	**S-11**	**S-12**
No. of participating laboratories	11	11	11	11	11	11	11	11	11	11	11	11
No. of laboratories retained after eliminating outliers	9	8	8	9	11	11	11	10	9	10	8	10
No. of test results in all laboratories	18	16	16	18	22	22	22	20	18	20	16	20
Mean value, mg/kg	2,05	9,91	8,14	9,94	2,05	2,07	8,29	10,08	4,94	5,64	7,64	2,10
Repeatability standard deviation, s_r, mg/kg	0,05	0,10	0,12	0,10	0,06	0,05	0,25	0,19	0,09	0,23	0,37	0,05
Repeatability coefficient of variation, CV(r), %	2,29	1,03	1,48	1,03	3,10	2,49	3,06	1,91	1,86	4,06	4,88	2,62
Repeatability limit, r ($s_r \times 2{,}8$), mg/kg	0,13	0,29	0,34	0,29	0,18	0,14	0,71	0,54	0,26	0,64	1,04	0,15
Reproducibility standard deviation, s_R, mg/kg	0,06	0,13	0,31	0,43	0,20	0,19	0,56	0,41	0,25	0,41	0,46	0,20
Reproducibility coefficient of variation, CV(R), %	2,85	1,32	3,81	4,37	9,62	9,38	6,70	4,10	4,97	7,18	5,98	9,51
Reproducibility limit, R ($s_R \times 2{,}8$), mg/kg	0,16	0,37	0,87	1,22	0,55	0,54	1,56	1,16	0,69	1,13	1,28	0,56

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

Sample	Al	Ca				
Number of participating laboratories	8	12	12	12	12	12
Number of laboratories retained after eliminating outliers	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, 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, $CV(r)$, %	7,2	4,1	7,3	4,9	3,1	1,6
Repeatability limit, r (s_r × 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, $CV(R)$, %	40,7	45,0	19,7	14,8	7,1	5,4
Reproducibility limit, R (s_R × 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

Sample	Cd				Na	
Number of participating laboratories	9	9	9	9	11	10
Number of laboratories retained after eliminating outliers	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, 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, $CV(r)$, %	11,7	9,1	4,3	4,9	2,0	10,8
Repeatability limit, r (s_r × 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, $CV(R)$, %	16,6	25,1	16,8	14,5	28,8	24,6
Reproducibility limit, R (s_R × 2,8), mg/kg	**0,017**	**0,032**	**0,038**	**0,047**	**0,650**	**0,707**

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

Sample	Cr				Ni	
Number of participating laboratories	9	9	9	9	9	9
Number of laboratories retained after eliminating outliers	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, 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, $CV(r)$, %	6,8	0,0	0,0	2,6	4,6	6,4
Repeatability limit, r (s_r × 2,8), mg/kg	**0,009**	**0,000**	**0,000**	**0,023**	**0,007**	**0,017**

Table A.4 *(continued)*

Sample	**Cr**				**Ni**	
Reproducibility standard deviation, s_R, mg/kg	0,007	0,013	0,012	0,019	0,012	0,017
Reproducibility coefficient of variation, $CV(R)$, %	14,2	23,7	12,3	5,9	21,9	17,7
Reproducibility limit, R (s_R × 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

Sample	**Cu**		**Mg**			
Number of participating laboratories	12	12	13	13	13	13
Number of laboratories retained after eliminating outliers	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, 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, $CV(r)$, %	5,9	10,2	5,1	4,7	3,4	2,1
Repeatability limit, r (s_r × 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, $CV(R)$, %	43,1	41,6	39,6	27,1	8,3	4,7
Reproducibility limit, R (s_R × 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

Sample	**Fe**				
Number of participating laboratories	12	12	12	12	12
Number of laboratories retained after eliminating outliers	11	11	12	10	11
Number of individual test results of all laboratories on each sample	22	22	24	20	22
Mean value, 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, $CV(r)$, %	0,0	8,8	8,4	3,4	2,5
Repeatability limit, r (s_r × 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, $CV(R)$, %	45,2	36,6	14,5	8,5	5,1
Reproducibility limit, R (s_R × 2,8), mg/kg	**0,070**	**0,061**	**0,038**	**0,051**	**0,045**

Table A.7 — Results for different levels of phosphorus

Sample	**P**				
Number of participating laboratories	11	11	11	11	11
Number of laboratories retained after eliminating outliers	11	10	11	11	11
Number of individual test results of all laboratories on each sample	22	20	22	22	22
Mean value, 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, $CV(r)$, %	3,3	2,7	2,2	1,6	1,2
Repeatability limit, r (s_r × 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, $CV(R)$, %	24,2	5,9	9,9	7,4	7,4
Reproducibility limit, R (s_R × 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

Sample	**Pb**	**Si**			
Number of participating laboratories	8	9	9	9	9
Number of laboratories retained after eliminating outliers	8	8	8	7	9
Number of individual test results of all laboratories on each sample	16	16	16	14	18
Mean value, 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, $CV(r)$, %	3,5	10,9	6,6	7,5	6,4
Repeatability limit, r (s_r × 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, $CV(R)$, %	47,3	51,3	49,0	11,8	13,0
Reproducibility limit, R (s_R × 2,8), mg/kg	**0,119**	**0,051**	**0,058**	**0,029**	**0,187**

Table A.9 — Results for different levels of zinc

Sample	**Zn**				
Number of participating laboratories	9	9	9	9	9
Number of laboratories retained after eliminating outliers	8	7	9	8	9
Number of individual test results of all laboratories on each sample	16	14	18	16	18
Mean value, 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, $CV(r)$, %	0,0	0,0	8,7	3,2	2,1
Repeatability limit, r (s_r × 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, $CV(R)$, %	39,7	48,3	22,6	27,5	13,5
Reproducibility limit, R (s_R × 2,8), mg/kg	**0,042**	**0,052**	**0,045**	**0,061**	**0,061**

Annex B
(informative)

Additional data

Tables B.1 to B.3 contain further data for silver, manganese, molybdenum, lead, titanium, tin and vanadium, but only from seven laboratories (not fully validated). As all these data are free from outliers, they are reported for information only.

Table B.1 — Results for different levels of silver and manganese

Sample	**Ag**			**Mn**			
Number of participating laboratories	7	7	7	7	7	7	7
Number of laboratories retained after eliminating outliers	7	7	7	7	7	7	7
Number of individual test results of all laboratories on each sample	14	14	14	14	14	14	14
Mean value, m, mg/kg	**0,036**	**0,060**	**0,121**	**0,041**	**0,084**	**0,101**	**0,188**
Repeatability standard deviation, s_r, mg/kg	0,004	0,007	0,004	0,004	0,004	0,004	0,006
Repeatability coefficient of variation, $CV(r)$, %	10,6	12,3	3,1	9,2	4,5	3,7	3,2
Repeatability limit, r ($s_r \times 2{,}8$), mg/kg	**0,011**	**0,021**	**0,011**	**0,011**	**0,011**	**0,011**	**0,017**
Reproducibility standard deviation, s_R, mg/kg	0,013	0,019	0,018	0,014	0,015	0,012	0,016
Reproducibility coefficient of variation, $CV(R)$, %	35,5	31,0	15,0	33,4	17,2	11,9	8,7
Reproducibility limit, R ($s_R \times 2{,}8$), mg/kg	**0,035**	**0,052**	**0,051**	**0,039**	**0,041**	**0,034**	**0,046**

Table B.2 — Results for different levels of molybdenum, lead and titanium

Sample	**Mo**		**Pb**	**Ti**		
Number of participating laboratories	7	7	7	7	7	7
Number of laboratories retained after eliminating outliers	7	7	7	7	7	7
Number of individual test results of all laboratories on each sample	14	14	14	14	14	14
Mean value, m, mg/kg	**0,034**	**0,042**	**0,168**	**0,046**	**0,053**	**0,096**
Repeatability standard deviation, s_r, mg/kg	0,004	0,007	0,009	0,000	0,004	0,000
Repeatability coefficient of variation, $CV(r)$, %	11,0	16,8	5,3	0,0	7,2	0,0
Repeatability limit, r ($s_r \times 2{,}8$), mg/kg	**0,011**	**0,020**	**0,025**	**0,000**	**0,011**	**0,000**
Reproducibility standard deviation, s_R, mg/kg	0,012	0,013	0,047	0,013	0,014	0,013
Reproducibility coefficient of variation, $CV(R)$, %	35,0	32,0	28,1	27,8	27,2	13,3
Reproducibility limit, R ($s_R \times 2{,}8$), mg/kg	**0,034**	**0,038**	**0,132**	**0,036**	**0,040**	**0,036**

Table B.3 — Results for different levels of barium, tin and vanadium

Sample	**Ba**		**Sn**		**V**	
Number of participating laboratories	7	7	7	7	7	7
Number of laboratories retained after eliminating outliers	7	7	7	7	7	7
Number of individual test results of all laboratories on each sample	14	14	14	14	14	14
Mean value, m, mg/kg	**0,093**	**0,115**	**0,105**	**0,360**	**0,026**	**0,044**
Repeatability standard deviation, s_r, mg/kg	0,004	0,009	0,008	0,008	0,004	0,000
Repeatability coefficient of variation, $CV(r)$, %	4,1	7,7	7,6	2,1	14,7	0,0
Repeatability limit, r (s_r × 2,8), mg/kg	**0,011**	**0,025**	**0,022**	**0,021**	**0,011**	**0,000**
Reproducibility standard deviation, s_R, mg/kg	0,029	0,029	0,024	0,023	0,011	0,016
Reproducibility coefficient of variation, $CV(R)$, %	30,9	24,9	22,5	6,5	40,9	36,5
Reproducibility limit, R (s_R × 2,8), mg/kg	**0,080**	**0,080**	**0,066**	**0,065**	**0,029**	**0,045**

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