INTERNATIONAL STANDARD

ISO 10540-3

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Animal and vegetable fats and oils — Determination of phosphorus content —

Part 3: Method using inductively coupled plasma (ICP) optical emission spectroscopy

Corps gras d'origines animale et végétale — Détermination de la teneur en phosphore —

Partie 3: Méthode par spectrométrie d'émission optique avec plasma induit par haute fréquence

Reference number
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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 10540-3 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

ISO 10540 consists of the following parts, under the general title *Animal and vegetable fats and oils — Determination of phosphorus content*:

— *Part 1: Colorimetric method*

— *Part 2: Method using graphite furnace atomic absorption spectrometry*

— *Part 3: Method using inductively coupled plasma (ICP) optical emission spectroscopy*

Animal and vegetable fats and oils — Determination of phosphorus content —

Part 3: Method using inductively coupled plasma (ICP) optical emission spectroscopy

1 Scope

This part of ISO 10540 specifies a method for the quantification of phosphorus in oil, which is limited to oils with low phosphorus content, without any turbidity or visible sediment.

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. This method is only suitable when phosphorus is present in a solubilized form. When it is present as fine particles, such as bleaching earth, ICP-OES analysis results in poor recovery as a result of nebulization and atomization problems. The only suitable non-ashing direct method for these samples is graphite furnace atomic absorption spectrometry.

ISO 10540-1 is the reference (colorimetric) method for the determination of the phosphorus content in fats and oils.

2 Normative references

The following referenced documents are indispensable for the application of this document. 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*

3 Terms and definitions

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

3.1
phosphorus content
dissolved phosphorus measured according to the method specified in this part of ISO 10540

NOTE It is measured in milligrams per kilogram.

4 Principle

Solvent-diluted vegetable oils are analysed for phosphorus 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

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Butanol.

5.2 Kerosene.

5.3 Xylene.

5.4 Standard element, present in solution as an organic soluble material[1)].

5.5 Base oil (Base 20 or Base 75 Oil)[1)], used to check the blank oil used and for the dilution of the standard solutions as needed.

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 Inductively coupled plasma optical emission spectrometer.

6.2 Analytical balance, capable of weighing to the nearest 0,001 g and displaying 0,000 1 g.

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

6.4 Tilt table mixer.

6.4 Volumetric flasks, of 100 ml capacity.

7 Sampling

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

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

8 Preparation of test sample

Prepare the test samples in accordance with ISO 661, except that the samples should not be clarified. If the sample is not clear when liquid, this method should not be used.

1) SPEX, 203 Norcross Ave., Metuchen, NJ 08840 (908/549-7144) and Base 20 Oil or Base 75 Oil and Constan brand standards available from Accu-Standard, 25 Science Park, New Haven, CT 06511 (800-442-5290) are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 10540 and does not constitute an endorsement by ISO of these products.

9 Procedure

9.1 General

9.1.1 Dilute all samples, standards and blanks 1:1 (by mass) with 1-butanol (or kerosene/xylene) to reduce the viscosity of the oil for better nebulization. Some samples are more soluble in 1-butanol than others.

NOTE 1 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.

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

9.1.3 Precise operating conditions will vary from instrument to instrument. Operate your instrument according to the manufacturer's directions and specifications. Conditions used for identical instruments from the same manufacturer will vary depending on the type of nebulizer and pumping system used.

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

NOTE 2 Some of the conditions used in the collaborative study are given in Annex B.

9.1.4 Ignite the instrument and allow it to warm up. It is profiled on an internal Hg lamp. Phosphorus may be detected at the major emission lines of 178,2 nm, 213,6 nm or 214,9 nm. Limits of detection are approximately 0,5 mg/kg.

9.1.5 Standardize the instrument as described (9.3) and scan all the samples in triplicate.

NOTE 3 Calibration drift has been noted. It may 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 another oil that has been shown to be free of trace elements, is used. Blank oil is diluted 1:1 as described in 9.1.1. Base 20 or Base 75 oil (5.5) is used as an absolute reference blank to determine that the blank oil is free of trace elements.

9.2.2 Phosphorus standard solution

The phosphorus standard is prepared from commercially available single-element organic-based standard. 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.

Although one standard concentration will work, up to four standards will provide a better calibration for linearity and accuracy. Levels should include 2,5 mg/kg, 5,0 mg/kg and 10,0 mg/kg standards depending on the range of values expected.

If an internal standard is used, it may be weighed as part of the elements or incorporated as part of the dilution solvent to yield 10 mg/kg to match that amount added to the sample.

9.3 Standardization

Run the blank oil standard and diluted Base 20 or Base 75 oils at the specified wavelength for phosphorus.

Run the phosphorus standard solutions (9.2.3) at the phosphorus wavelength chosen.

Scan blanks, samples and standards in triplicate for phosphorus and average them.

Run standards and the blank every 10 samples or less and restandardize the instrument as needed. For accuracy use a narrow range of standardisation (0 mg/kg to 25 mg/kg of phosphorus). Although the linearity is somewhat greater, test samples should be diluted to keep the phosphorus content within the range of standardization.

9.4 Preparation of test samples

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

Dilute 0,2 g of lecithins (up to 100 % acetone-insoluble) to 5,0 g with blank soybean oil and then to 10 g with 1-butanol. Mix the samples on a tilt table mixer for 1 h and then dilute 1:10 with 1:1 blank oil/1-butanol to give a total dilution of 1:500.

10 Calculation

Computation is a feature of most instrument programs. Area counts from known standards are inserted into a linear regression formula. From this relationship, concentrations may be determined from the area counts of the samples.

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. 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 with 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 in not more than 5 % of cases be greater than the repeatability limit (r) as shown in Annex A.

11.3 Reproducibility

The absolute difference between two single test results, obtained with the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than the reproducibility limit (R) as shown in Annex A.

12 Test report

The test report shall specify:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, with reference to this part of ISO 10540;

— all operating details not specified in this part of ISO 10540, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

— the test result obtained;

— if repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Results of interlaboratory tests

The precision of the method has been established for high and low levels of phosphorus in oil by an international study conducted in 1998/1999 by AOCS and FOSFA International. Data were analysed according to ISO 5725-2:1994. Examples of instrument configurations used in the interlaboratory test are reported in Annex B.

Table A.1 — ICP analysis of high levels of phosphorus in oil — Summary of analyses from 15 laboratories

Sample	A	B	C	D	E	F	G	H
Number of participating laboratories after eliminating outliers	13	13	13	13	13	12	13	13
Total number of replicates	26	26	26	26	26	24	26	26
Mean value, mg/kg	497,13	334,60	147,37	101,72	56,36	29,11	12,01	305,38
Repeatability standard deviation (s_r)	9,22	7,82	1,92	1,34	0,97	0,41	0,30	6,22
Coefficient of variation of repeatability, %	1,85	2,34	1,30	1,32	1,73	1,42	2,49	2,04
Repeatability limit ($2{,}8 \times s_r$)	25,81	21,89	5,37	3,75	2,72	1,16	0,84	17,41
Reproducibility standard deviation (s_R)	48,42	39,32	22,26	12,13	4,73	1,74	1,32	36,32
Coefficient of variation of reproducibility,%	9,74	11,75	15,10	11,92	8,39	5,99	10,96	11,89
Reproducibility limit ($2{,}8 \times s_R$)	135,56	110,10	62,32	33,96	13,24	4,88	3,68	101,70

NOTE The same samples were also tested using a colorimetric method as specified in ISO 10540-1, and using an atomic absorption spectrometry (AAS) method as specified in ISO 10540-2. Statistical results related to the colorimetric and AAS methods are presented in ISO 10540-1 and ISO 10540-2, respectively.

Table A.2 — ICP analysis of low levels of phosphorus in oil — Summary of analyses from 9 laboratories

Sample	A	B	C	D	E
Laboratories returning results	8	8	9	8	9
Number of participating laboratories after eliminating outliers	8	8	9	8	9
Total number of replicates	16	16	18	16	18
Mean value, mg/kg	7,51	3,40	1,60	3,42	14,66
Repeatability standard deviation (s_r)	0,22	0,05	0,08	0,05	0,96
Coefficient of variation of repeatability, %	2,93	1,59	5,07	1,33	6,53
Repeatability limit ($2{,}8 \times s_r$)	0,62	0,15	0,23	0,13	2,68
Reproducibility standard deviation (s_R)	0,59	0,44	0,29	0,42	0,96
Coefficient of variation of reproducibility, %	7,86	13,03	18,24	12,22	6,53
Reproducibility limit ($2{,}8 \times s_R$)	1,65	1,24	0,81	1,17	2,68

Annex B
(informative)

Instrument configurations

Table B.1 — Instrument configurations reported in the interlaboratory tests

Instrument	Grating configuration	Nebulizer
Unicam 7000	Radial	V-groove
Leeman PS 1000	Radial	V-groove
Thermo-JA	Axial	Meinhard TR 30-K3
Spectroflame Modular OES	Radial	Cross flow
Spectroflame	Radial	Meinhard
P-E Plasma 400	Axial	High solids
ARL 3410	Radial	Glass concentric
Thermo-JA Iris	Radial	Modified Lichte
P-E Optima 3000 DV	Axial	External mount gemcone

Table B.2 — Examples of some instrument conditions

Settings	Thermo-JA IRIS	Unicam 7000
Power	1 350 W	1 200 W
Uptake time		99 s
Integration time		3 × 1 s
Nebulizer flow	20,06 psi	40 psi
Pump flow rate	1,85 ml/min	1,5 ml/min
Auxiliary flow	1 500 ml/min	600 ml/min
Coolant flow		16 l/min