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BSI Standards Publication

Liquid petroleum products — Determination of Sodium, Potassium, Calcium, Phosphorus, Copper and Zinc contents in diesel fuel — Method via Inductively Coupled Plasma Optical Emission Spectrometry (ICP OES)

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National foreword

This British Standard is the UK implementation of EN 16476:2014.

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

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Liquid petroleum products - Determination of Sodium, Potassium, Calcium, Phosphorus, Copper and Zinc contents in diesel fuel - Method via Inductively Coupled Plasma Optical Emission Spectrometry (ICP OES)

Produits pétroliers liquides - Détermination des concentrations en sodium, potassium, calcium, phosphore, cuivre et zinc dans le gazole - Méthode par spectrométrie d'émission atomique par plasma à couplage inductif (ICP OES)

Flüssige Mineralölerzeugnisse - Bestimmung des Gehalts an Natrium, Kalium, Calcium, Phosphor, Kupfer, Zink in Dieselkraftstoffen - Direkte Bestimmung durch optische Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP OES)

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Foreword

This document (EN 16476:2014) has been prepared by Technical Committee CEN/TC 19 “Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2014 and conflicting national standards shall be withdrawn at the latest by November 2014.

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Introduction

This document specifies a method for determining concentration of the six most essential ash forming elements. The number of six was chosen in order to limit the complexity of the test and to be able to determine a decent method precision. The six elements were specified by the vehicle manufacturers. The test provides additional information to the more common ash concentration determination methods which do aim towards filter problems. Metals have more impact on the durability of modern DPF exhaust gas after-treatment systems in diesel vehicles than ash in general.

1 Scope

This European Standard specifies an inductively coupled plasma optical emission spectrometry (ICP OES) method for the determination of sodium, potassium, calcium, phosphorus, copper and zinc concentrations of diesel fuels, including those containing up to 30 % (*V/V*) fatty acid methyl ester (FAME), in the range detailed in Table 1. These six elements are considered as the most essential ash forming elements.

Table 1 — Application ranges for ash forming elements

Element	**Range** mg/kg
Sodium	1,2 to 2,5
Potassium	0,9 to 2,5
Calcium	0,3 to 2,5
Phosphorus	0,9 to 2,5
Copper	0,2 to 2,5
Zinc	0,2 to 2,5

NOTE For the purposes of this European Standard, the term “% (*V/V*)” is used to represent the volume fraction, φ.

WARNING — The use of this European Standard may involve hazardous materials, operations and equipment. This European Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this European Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

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.

EN ISO 1042, *Laboratory glassware - One-mark volumetric flasks (ISO 1042)*

EN ISO 3170, *Petroleum liquids - Manual sampling (ISO 3170)*

EN ISO 3171, *Petroleum liquids - Automatic pipeline sampling (ISO 3171)*

3 Principle

A weighed amount of diesel fuel sample is diluted with an organic solvent. The solution is then introduced directly into an ICP OES spectrometer. Sodium, potassium, calcium, phosphorous, copper and zinc concentrations are determined by comparison with calibration solutions. An Internal Standard is employed to correct viscosity effects.

4 Reagents

If not specified otherwise, only chemicals of a known high degree of purity shall be used.

4.1 Kerosene, boiling range between 175 °C and 250 °C, elements free. The solvent shall contain less than 0,1 mg/kg of each of the element under test.

NOTE In case of unstable plasma or in case of special laboratory requirements, kerosene can be substituted by other suitable solvents, provided that they are free of analytes.

4.2 Sodium Standard solution, commercially available in oil, containing, e.g. 1 000 mg/kg sodium.

4.3 Potassium Standard solution, commercially available in oil, containing, e.g. 1 000 mg/kg potassium.

4.4 Calcium Standard solution, commercially available in oil, containing, e.g. 1 000 mg/kg calcium.

4.5 Phosphorus Standard solution, commercially available in oil, containing, e.g. 1 000 mg/kg phosphorus.

4.6 Copper Standard solution, commercially available in oil, containing, e.g. 1 000 mg/kg copper.

4.7 Zinc Standard solution, commercially available in oil, containing, e.g. 1 000 mg/kg zinc.

4.8 Sodium, potassium, calcium, phosphorus, copper and zinc intermediate working Standard (50 mg/kg each).

Weigh (1,00 g ± 0,01) g of each element Standard solution (4.2), (4.3), (4.4), (4.5), (4.6), (4.7) into a 50 ml bottle (5.2). Add kerosene (4.1) to (20,00 ± 0,20) g. Each mass shall be weighed to the nearest 0,001 g.

In the case of using several mono-element Standard solutions, attention-shall be paid to ensure that they are free of other analyte elements.

Some commercial element Standard solutions are furnished with higher concentration on the market. Those solutions may be used instead of the required solutions, but in this case an initial mass to mass dilution shall be done according to recommendations given in 7.1.

Ready-made commercial multi-element Standard solutions may be used instead of the single element Standard solutions (4.2, 4.3, 4.4, 4.5, 4.6 and 4.7) as the method is designed to avoid interference at the wavelengths specified.

4.9 Internal Standard solution (cobalt, scandium, yttrium), commercially available in oil (analyte free), for example with 1 000 mg/kg per element, available as single element Standard.

NOTE The internal Standard solutions are commonly available as single element Standards with various element concentrations.

4.10 Argon, with a mass fraction *w* (Ar) ≥ 99,996 %.

Small amounts of oxygen may be added to the argon gas stream using a metering valve (for example 30 ml/min to 100 ml/min) to prevent carbon deposits in the area of the plasma torch.

5 Apparatus

5.1 Volumetric flasks, 25 ml and 250 ml, according to EN ISO 1042, with taper sleeve and plug.

To avoid contamination of the test samples, all solutions shall be prepared in plastic containers. Surfaces which can come in contact with the solutions shall not be touched by hand.

To avoid phosphorus contamination due to the phosphates contained in the detergents used for washing the flasks, rinse the latter at least twice with an approximate 5 mol/l solution of nitric acid. Then rinse with distilled water and dry.

The use of borosilicate glass containers is not recommended due to the risk of sodium contamination.

5.2 Bottles, 50 ml, with screw caps, polyethylene PE.

To avoid phosphorus contamination due to the phosphates contained in the detergents used for washing the bottles, rinse the latter at least twice with an approximate 5 mol/l solution of nitric acid. Then rinse with distilled water and dry.

5.3 Analytical balance, capable of weighing to the nearest 0,000 1 g.

5.4 ICP OES spectrometer

5.4.1 General

ICP OES spectrometer equipped for the analysis of organic liquids. The use of a feed pump for sample introduction into the nebuliser is required. Pump tubing shall be suitable for organic use. Both setup and operation of the ICP OES spectrometer shall be done in accordance with operating instructions of the manufacturer.

The use of vertically oriented plasma, radially observed ICP OES is recommended to avoid carbon residue formation and minimize matrix interference due to the presence of carbon in the plasma.

5.4.2 Recommended wavelengths

The recommended wavelengths of sodium, potassium, calcium, phosphorus, copper and zinc, cobalt, scandium and yttrium are given in Table 2.

Table 2 — Recommended wavelengths

Element	**Wavelength** [a] nm			
Sodium	589,592	588,995		
Potassium	766,491	769,897		
Calcium	317,933	184,006 (183,944)	315,887	396,847 393,366
Phosphorus	177,499 (177,434)	178,287 (178,222)	213,618 [b]	214,914
Copper	324,754	327,396	224,700	
Zinc	206,200	213,856		202,548 [b]
Cobalt	238,892	258,033		
Scandium	361,383			
Yttrium	224,306	371,029	360,073	

[a] Wavelengths are expressed as vacuum lines or as air lines (in brackets) according to the expression of the different manufacturers of ICP OES spectrometers.

[b] Can be slightly interfered by Cu at the same wavelength.

6 Sampling

Unless otherwise specified in the commodity specification, samples shall be taken as described in EN ISO 3170 or EN ISO 3171 and/or in accordance with the requirements of national regulations for the sampling of the product under test.

Collect and store samples in sample bottles made of plastic material (HDPE, FEP, PP, or PFA) or metallic epoxy coated containers.

7 Preparation of calibration solutions

7.1 General

In order to avoid inhomogeneity, the intermediate working Standard (4.8) shall be thoroughly shaken before use.

The masses given correspond to a nominal element concentration of 1 000 mg/kg in the single element solutions (4.2, 4.3, .4.4, 4.5, 4.6 and 4.7) and the element Internal Standard solution (4.9). If solutions with other element concentrations are used, these masses shall be adjusted accordingly in order to establish the given nominal concentrations as closely as possible. Calculate the exact concentrations of the calibration solutions, taking into account the exact masses.

It is strongly advised to use freshly prepared calibration solutions.

7.2 Internal Standard solution

Weigh approximately 5 g of cobalt, scandium or yttrium stock solution (4.9) in a 250 ml volumetric flask (5.1). Fill with kerosene (4.1) to the mark.

The solution shall be homogenized by vigorous shaking.

The same Standard batch shall be used for all samples and calibration Standards.

7.3 Calibration solutions

Prepare the calibration solutions as indicated in Table 3. Each intermediate mass of elements for the working Standards (4.8) shall be weighed to the nearest 0,001 g into a 25 ml volumetric flask (5.1). Add 5,0 ml of the Internal Standard solution (7.2). Fill with kerosene (4.1) to the mark.

All prepared solutions shall be homogenized by vigorous shaking.

The exact concentration of each element in the calibration solution shall be calculated considering the exact weighed portion.

Table 3 — Calibration solutions

Calibration solution	**Sodium concentration** mg/l	**Potassium concentration** mg/l	**Calcium concentration** mg/l	**Phosphorus concentration** mg/l	**Copper concentration** mg/l	**Zinc concentration** mg/l	**Intermediate working Standard** g
1 (blank)	0	0	0	0	0	0	-
2	0,25	0,25	0,25	0,25	0,25	0,25	0,125
3	0,50	0,50	0,50	0,50	0,50	0,50	0,25
4	1,00	1,00	1,00	1,00	1,00	1,00	0,50
5	2,00	2,00	2,00	2,00	2,00	2,00	1,00

7.4 Calibration check solution

Prepare a calibration check solution as indicated in Table 4 to verify sensitivity and accuracy of the calibration curve, using the methodology indicated in 7.3.

Table 4 — Calibration check solution

	Sodium concentration mg/l	**Potassium concentration** mg/l	**Calcium concentration** mg/l	**Phosphorus concentration** mg/l	**Copper concentration** mg/l	**Zinc concentration** mg/l
Calibration check solution	0,75	0,75	0,75	0,75	0,75	0,75

Standard solutions (4.2, 4.3, 4.4, 4.5, 4.6 and 4.7) used for the preparation of the calibration check solution shall be sourced from a different batch, lot, or supplier than those used for the preparation of calibration solutions (7.3).

Ready-made commercial multi-element Standard solutions may be used instead of the single element Standard solutions.

8 Calibration

8.1 General

The ICP OES spectrometer setup and instruments checks are performed according to the manufacturer's recommendations.

The choice of the instrumental parameters is made to obtain the best signal/background ratio for all elements.

As the magnitude of the background signal highly depends on spectral structures caused by the sample nature and origin, only net intensities have to be recorded. To keep background noise as low as possible, signal and background shall be measured simultaneously.

Net intensity of analytical lines shall be calculated by subtracting the intensity measured at appropriate background wavelengths. Most instruments are equipped with software which allows the automatic correction of the background.

A separate calibration function for each element under investigation shall be established.

8.2 Calibration of the ICP OES spectrometer

The calibration of the ICP OES spectrometer shall be done by the measurement of the blank solution and of the calibration solutions (7.3) using three replicates. For the determination of the elements, the wavelengths recommended in Table 2 shall be used. The background subtraction shall be performed at wavelengths not affected by other lines.

IMPORTANT — Ensure that the wavelengths used in calibration also match exactly the ones used in the sample measurement.

Depending on the spectrometer software, follow either procedure A or B.

8.3 Procedure A

For each element under investigation, conduct the aspiration of the calibration solutions (7.3).

For each calibration solution and for each element, measure the net emission intensity of the element, I_E, and the net emission intensity of the Internal Standard, I_{IS}, at the chosen wavelengths.

Calculate the intensity ratio of each element, R_E, of each calibration solution~~s~~ using the following formula:

$$R_{\mathrm{E}} = \frac{I_{\mathrm{E}}}{I_{\mathrm{IS}}} \tag{1}$$

Construct a calibration curve for each element using linear regression with concentration of the element in the calibration solutions (7.3) as independent variable (X) and the corresponding mean intensity ratio R of the element as dependent variable (Y) according to the formula:

$$Y = m \cdot X + b \tag{2}$$

This regression may be performed with built in software of the spectrometer.

8.4 Procedure B

Conduct the aspiration of calibration solutions 1 (blank solution) to measure the net emission intensity of the Internal Standard (I_{Blank}).

Conduct the aspiration of each following calibration solution~~s~~ to measure the net emission intensity of each element, I_E, and of Internal Standard, I_{IS}.

Calculate the correction factor R of each calibration solution~~s~~ using the formula:

$$R = \frac{I_{\mathrm{Blank}}}{I_{\mathrm{IS}}} \tag{3}$$

Calculate the corrected intensity of each element I_{ECor} of each calibration solution (7.3) using the formula:

$$I_{\mathrm{ECor}} = R \cdot I_{\mathrm{E}} \tag{4}$$

Construct a calibration curve for each element using linear regression with concentration of the element in the calibration solution (7.3) as independent variable (X) and the corrected intensity of the element as dependent variable (Y) according to the formula:

$$Y = m \cdot X + b \tag{5}$$

This regression may be performed with built in software of the spectrometer.

8.5 Check of calibration

Verify the calibration before the sample series and after every ten samples by using the calibration check solution prepared in 7.4. If the element concentrations of the calibration check solution differ from the reference values by more than 10 %, prepare a new calibration check solution. If the element concentrations of the new calibration check solution still differ from the reference values by more than 10 %, prepare a new calibration curve. All samples which have been analysed since the last acceptable check standard shall be repeated with the new calibration.

9 Sample analysis

9.1 Sample preparation

Before starting the preparation, the sample shall be homogenized by shaking vigorously.

Weigh (12,50 ± 0,01) g of sample to the nearest 0,001 g in a 25 ml volumetric flask (5.1). Add exactly 5,0 ml of Internal Standard solution (7.2) and fill with kerosene (4.1) to the mark.

Record the exact mass of sample solution to calculate the dilution factor which is nominally 2 (25 ml/12,50 g). Homogenize the sample solution by shaking vigorously.

Do not use a volume/volume automatic diluter.

NOTE The dilution factor is expressed in ml/g.

9.2 Sample measurement

Perform the analysis of the elements with the same instrumental parameters and wavelengths used for the calibration of the spectrometer (8.2).

As the magnitude of the background signal highly depends on spectral structures caused by the sample nature and origin, only net intensities shall be recorded. To keep background noise as low as possible, signal and background shall be measured simultaneously.

Net intensity of analytical lines shall be calculated by subtracting the intensity measured at appropriate background wavelengths.

The background subtraction shall be performed at wavelengths not affected by other lines.

Perform three measurements of the net emission intensity of each element, I_{E}, and of Internal Standard, I_{IS}, at the chosen wavelengths and calculate the mean values.

The *RSD* of I_{IS} shall be lower than 3 %, otherwise that might indicate nebulizer problems. Check and solve this trouble before continuing.

A maximum variation of 20 % of the intensity of the Internal Standard from the first calibration blank is accepted. A value greater than 20 % may indicate a problem with the instrument (blocked nebulizer) or with the sample solution. Check and solve this trouble before continuing.

For procedure A (8.3), calculate the intensity ratio R_{E} of each element using the corresponding Formula (1).

For procedure B (8.4), calculate the corrected intensity of each element using Formula (4) and the corresponding Formula (3).

Allow sufficient rinsing time after each sample analysis as a memory effect may occur in cases where high concentrations of elements are introduced.

The drift of the spectrometric system shall be checked before and at the end of the sample series or at least between every ten samples using the calibration check solution (7.4). If one of the element concentrations of the calibration check solution differs from the reference values by more than 10 %, proceed as indicated in 8.5.

10 Calculation

The concentration of each element in the sample solution is calculated from the intensity ratio of each element (9.2) using Formula (2) if Procedure A is used or Formula (5) if Procedure B is used. This may be done either manually, or with use of the appropriate software functions of the ICP OES spectrometer.

The concentration of each element in the diesel fuel sample is calculated using the formula:

$$C_{\mathrm{E}} = c_{\mathrm{E}} \times DF \tag{6}$$

where

C_E is the concentration of element of the diesel fuel sample, expressed in mg/kg;

c_E is the concentration of element of the sample solution, expressed in mg/l;

DF is the dilution factor.

11 Expression of result

Report the concentration of each element (sodium, potassium, calcium, phosphorus, copper and zinc) in mg/kg rounded to the nearest 0,01 mg/kg.

12 Precision

12.1 General

The precision given was derived from statistical analysis by EN ISO 4259 [1] of the results of a round robin test.

12.2 Repeatability

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the value given in Table 5 in only one case in 20.

12.3 Reproducibility

The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the value given in Table 5 in only one case in 20.

Table 5 — Method precision

Element	Repeatability *r*	Reproducibility *R*
Sodium	0,061 4 *X* + 0,116 1	0,190 8 *X* + 0,360 9
Potassium	0,012 4 *X* + 0,145 1	0,034 2 *X* + 0,401 0
Calcium	0,043 9 *X* + 0,041 0	0,097 9 *X* + 0,091 4
Phosphorus	0,128	0,424
Copper	0,053 4 *X* + 0,014 1	0,163 5 *X* + 0,043 2
Zinc	0,057 9 *X* + 0,029 5	0,127 3 *X* + 0,064 8
NOTE *X* is the average of the two results being compared, in mg/kg.		

13 Test report

The test report shall contain at least the following information:

a) reference to this European Standard, i.e. EN 16476;

b) type and complete identification of the product tested;

c) used method of sampling (see Clause 6);

d) used calibration procedure (A or B, see Clause 8);

e) result of the test (see Clause 11);

f) any deviation, by agreement or otherwise, from the procedure specified;

g) date of the test.

Bibliography

[1] EN ISO 4259:2006, *Petroleum products - Determination and application of precision data in relation to methods of test (ISO 4259:2006)*

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