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BSI Standards Publication

Automotive fuels — Determination of manganese and iron content in unleaded petrol — Inductively coupled plasma optical emission spectrometry (ICP OES) method

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum Testing and Terminology.

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

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Automotive fuels - Determination of manganese and iron content in unleaded petrol - Inductively coupled plasma optical emission spectrometry (ICP OES) method

Carburants pour automobiles - Détermination des teneurs en fer et en manganèse dans les essences sans plomb - Méthode spectrométrique optique par plasma à couplage inductif (ICP OES)

Kraftstoffe für Kraftfahrzeuge - Bestimmung des Gehaltes an Mangan und Eisen in unverbleitem Ottokraftstoff - Optische Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP OES)

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Foreword

This document (EN 16136:2015) 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of an identical text or by endorsement, at the latest by August 2015, and conflicting national standards shall be withdrawn at the latest by August 2015.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 16136:2011.

The major updates are the lowering of the manganese content to allow a specification setting of 2 mg/l of manganese in line with the FQD requirement per 2014-01-01, and the introduction in the scope of determination of iron content, which can be added into petrol as ferrocene.

This document answers requirements originating from the amended Fuels Quality Directive (FQD, [2]).

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard specifies a method based on inductively coupled plasma optical emission spectrometry (ICP OES) for the determination of manganese content from about 0,5 mg/l to about 7,5 mg/l and of iron content from about 1,4 mg/l to about 6,0 mg/l in unleaded petrol containing up to 3,7 % (m/m) oxygen.

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 to determine the applicability of regulatory limitations prior to use.

NOTE 1 Manganese as MMT and iron as ferrocene are added to petrol to increase anti-knock properties.

NOTE 2 Solutions of MMT in petrol are unstable when exposed to light. Low and erratic results are expected if petrol samples are exposed to light prior the analysis.

Iron and manganese contents higher than 6,0 mg/l and 7,5 mg/l respectively may be measured after preliminary dilution of the sample with a suitable solvent. However, the precision has not been established for such a procedure. Further work regarding automotive ethanol (E85) fuel is on-going in CEN.

NOTE 3 For the purposes of this European Standard, the terms "% (m/m)" and "% (V/V)" are used to represent the mass fraction (μ) and the volume fraction (φ) of a material respectively.

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)*

EN ISO 3675, *Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method (ISO 3675)*

EN ISO 12185, *Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method (ISO 12185)*

3 Principle

A petrol sample is diluted with a hydrocarbon solvent. The solution is introduced directly into the plasma of an ICP OES spectrometer. Iron and manganese contents are calculated by comparison with calibration solutions prepared from suitable iron and manganese compounds. An internal standard is employed to correct viscosity and vapour pressure effects.

4 Reagents

Unless specified otherwise, only chemicals which are known to have a high degree of purity shall be used.

Some ready-made commercial multi-element Standard solutions may be used instead of the single element Standard solution (4.4 and 4.5).

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

4.1 Kerosene, boiling range between 150 °C and 250 °C, analytical reagent grade.

Other grades of kerosene with analyte concentrations below the detection limit of the instrument for the elements under investigation may be used. In this case, perform a wavelength scan for analyte elements to check spectral interferences.

4.2 Heptane, analytical reagent grade.

4.3 Solvent, add 25 ml heptane (4.2) to a 500 ml HDPE bottle (5.1.2) and fill to 500 ml with kerosene (4.1).

4.4 Manganese Standard solution, commercially available in oil, c(Mn) = 100 mg/kg.

4.5 Iron Standard solution, commercially available in oil, c(Fe) = 100 mg/kg.

Some commercial element Standard solutions are furnished with higher content on the market. Those solutions may be used instead of the required solutions, but an initial mass to mass dilution has to be done according to recommendations given in 7.4.

4.6 Element Standard solution, of one of the elements cobalt, scandium, yttrium, etc. commercially available in oil (analyte free), for example with 1 000 mg/kg per element, available as single element standards.

NOTE The internal Standard solutions are commonly available as single element standards with various element contents.

4.7 Argon, with a purity of ≥ 99,995 % (V/V).

Small amounts of oxygen (purity ≥ 99,995 % (V/V)) may be added, for instance in accordance with the operating instructions of the equipment manufacturer, to the argon gas stream using a metering valve (30 ml/min to 100 ml/min) to prevent carbon deposits in the area of the plasma torch.

5 Apparatus

5.1 Laboratory equipment:

All glassware shall be cleaned carefully before use.

5.1.1 Glassware, usual laboratory glassware, together with the following:

5.1.1.1 Beakers, 50 ml.

5.1.1.2 Volumetric flasks, 20 ml, 50 ml and 500 ml according to EN ISO 1042, with taper sleeve and plug.

5.1.2 Bottles, 50 ml and 500 ml, with screw caps, high-density polyethylene (HDPE).

5.1.3 Graduated pipettes or variable volume automatic pipettes, fitted with disposable polypropylene tips capable to measure up to the nearest 0,01 ml.

CAUTION — Attention shall be paid with air displacement pipettes in the presence of volatile solvents or petrol samples.

5.2 Analytical balance, capable of weighing to the nearest 0,1 mg.

5.3 ICP OES spectrometer:

ICP OES spectrometer equipped for the analysis of organic liquids, with a high-frequency generator and a nebulizer suitable for organic solvents. The use of a feed pump for sample introduction into the nebulizer is required. Both setup and operation of the ICP OES spectrometer shall be done in accordance with operating instructions of the manufacturer.

A cooled spray chamber may be used, provided that the temperature is controlled ± 1 °C.

Table 1 gives the recommended wavelengths. As the magnitude of the background signal highly depends on spectral structures caused by the sample's nature, only net intensities are to be recorded.

Table 1 — Recommended wavelengths

Element	Wavelength nm
Manganese	257,610 259,372 260,569 279,482 279,827 293,931
Iron	234,350 238,204 240,488 259,940 261,187
Cobalt	238,892
Scandium	361,383
Yttrium	224,306 360,073 371,029

6 Sampling

IMPORTANT — The laboratory shall receive a sample which is truly representative and was not damaged or altered during transport or storage.

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.

The samples shall be stored in clean, opaque containers.

7 Preparation of solutions

7.1 General

In order to avoid inhomogeneity, iron and manganese standard solutions (4.4 and 4.5) shall be shaken vigorously before use. It is strongly advised to use freshly prepared calibration solutions.

7.2 Preparation of the internal standard solution

Weigh 2,00 g of cobalt, scandium or yttrium stock solution (4.6) with a precision of 0,01 g in a 50 ml volumetric flask (5.1.1.2)

Fill up to 50 ml with kerosene (4.1).

This prepared solution shall be homogenized by vigorous shaking.

The same standard batch shall be used for all samples and calibration standards.

7.3 Preparation of the manganese intermediate solution

Weigh 1,50 g ± 0,01 g of manganese standard solution (4.4) into a 50 ml HDPE bottle (5.1.2). Add solvent (4.3) to 15,00 g ± 0,01 g. In case manganese standard solutions (4.4) with different manganese content are used, the mass of standard solution shall be adjusted accordingly to achieve 10 mg/kg manganese content.

7.4 Preparation of the iron intermediate solution

Weigh 1,50 g ± 0,01 g of iron standard solution (4.5) into a 50 ml HDPE bottle (5.1.2). Add solvent (4.3) to 15,00 g ± 0,01 g. In case iron standard solutions (4.5) with different iron content are used, the mass of standard solution shall be adjusted accordingly to achieve 10 mg/kg iron content.

7.5 Preparation of the calibration solutions

The calibration solutions shall be prepared as indicated in Table 2. Each mass of manganese intermediate dilution solution (7.3) and iron intermediate dilution solution (7.4), shall be weighed to the nearest 0,001 g into a 20 ml volumetric flask (5.1.1.2). Add exactly 1,00 ml of the internal standard solution (7.2). Fill with solvent (4.3) to the mark.

NOTE These concentrations in Table 2 seem odd compared to the scope of determination as given in Clause 1, but in 9.1 a dilution step for the sample is introduced.

All solutions thus prepared shall be homogenized by vigorous shaking.

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

Table 2 — Concentration of manganese and iron in the calibration solutions

Calibration solutions	**Manganese intermediate solution (7.3)** g	**Iron intermediate solution (7.4)** g	**Manganese concentration** mg/l	**Iron concentration** mg/l
Blank	0,00	0,00	0,00	0,00
1	0,05	0,05	0,025	0,025
2	0,20	0,20	0,10	0,10
3	0,50	0,50	0,25	0,25
4	0,80	0,80	0,40	0,40

7.6 Preparation of quality control solution

A 0,15 mg/l quality control (QC) solution shall be prepared to verify sensitivity and accuracy of the calibration curve.

Standard solutions (4.4 and 4.5) used for the preparation of the calibration solution (7.5) shall be sourced from a different batch, lot or supplier than those used for the preparation of quality control solutions.

Weigh 0,075 g of manganese standard solution (4.4) and 0,075 g of iron standard solution (4.5) to the nearest 0,1 mg into a 50 ml volumetric flask (5.1.1.2). Add exactly 2,50 ml of the internal standard solution (7.2). Fill with the solvent (4.3) to the mark.

The mass given is based on manganese and iron contents of 100 mg/kg. In case a standard solution with different element content is used, the mass shall be adjusted accordingly to obtain the specified element content.

All solutions prepared shall be homogenized by shaking.

8 Calibration

8.1 General

The ICP OES spectrometer set up and instrument check are performed according to the instructions from the manufacturer. Follow the manufacturer's instructions for setting up the instrument with organic solutions.

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

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. Some instruments are equipped with software which allows the automatic correction of the background.

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 (7.5) using three replicates. At least one of the wavelengths recommended in Table 1 shall be used. It is important to 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 (7.5).

For each calibration solution and for each element, measure the net emission intensity of iron, I_{Fe}, and of manganese, I_{Mn} and the net emission intensity of the internal standard, I_{IS}, at the chosen wavelengths.

Calculate the intensity ratio of iron, F_{Fe}, and of manganese, F_{Mn}, of each calibration solution using the following formulae:

$$F_{\mathrm{Fe}} = \frac{I_{\mathrm{Fe}}}{I_{\mathrm{IS}}} \tag{1}$$

$$F_{\mathrm{Mn}} = \frac{I_{\mathrm{Mn}}}{I_{\mathrm{IS}}} \tag{2}$$

A calibration curve for iron and a calibration curve for manganese are constructed using linear regression with concentration of the element in the calibration solutions (7.5) as independent variable (X) and the

corresponding mean intensity ratio F of iron or of manganese as dependent variable (Y) according to the formula for Procedure A:

$$Y_A = m_A \cdot X_A + b_A \quad (3)$$

where

m_A is the slope of the calibration line;

b_A is the intercept.

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

8.4 Procedure B

Conduct the aspiration of the blank solution to measure the net emission intensity of the internal standard (I_{Blank}).

Conduct the aspiration of each following calibration solution (7.5) to measure the net emission intensity of iron I_{Fe} and of manganese I_{Mn}, and of internal standard I_{IS}.

Calculate the correction factor F_C of each calibration solutions using the formula:

$$F_c = \frac{I_{Blank}}{I_{IS}} \quad (4)$$

Calculate the corrected intensity of iron I_{FeCor} and of manganese I_{MnCor} of each calibration solutions (7.5) using the formulae:

$$I_{FeCor} = F_c \cdot I_{Fe} \quad (5)$$

$$I_{MnCor} = F_c \cdot I_{Mn} \quad (6)$$

A calibration curve for iron and a calibration curve for manganese are constructed using linear regression with concentration of the element in the calibration solutions (7.5) as independent variable (X) and the corrected intensity of iron and manganese as dependent variable (Y) according to the formula for Procedure B:

$$Y_B = m_B \cdot X_B + b_B \quad (7)$$

where

m_B is the slope of the calibration curve;

b_B is the intercept.

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

8.5 Check of calibration

The calibration curves shall be checked at regular intervals to verify sensitivity and accuracy of the calibration curve using a QC solution (see 7.6).

If the manganese or iron content of the QC solution differs from the reference value by more than 10 %, prepare a new QC solution as in 7.6. If manganese or iron contents of the new QC solution still differs from the reference values by more than 10 %, a new calibration shall be established. All samples which have been analysed since the last acceptable QC should be repeated with the new calibration.

9 Sample analysis

9.1 Sample solution preparation

Homogenize the sample by vigorous shaking. Transfer 1 ml of sample into a 20 ml volumetric flask (5.1.1.2) by means of a pipette (5.1.3). Weigh this sample volume, m_s, to the nearest 0,1 mg using the analytical balance (5.2), add exactly 1,00 ml of the internal standard solution (7.2) and fill to the mark with kerosene (4.1).

The solution shall be thoroughly homogenized by shaking.

The solutions shall be analysed just after the preparation.

9.2 Sample solution measurement

The determination of iron and manganese is performed with the same instrumental parameters and wavelengths used for the calibration of the spectrometer (8.2).

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 manganese, I_{Mn}, and of iron, I_{Fe}, at the chosen wavelengths and calculate the mean values.

The relative standard deviation (RSD) of I_{IS} shall not be higher than 3 %, otherwise that might indicate nebuliser problem.

A maximum variation of 20 % of the intensity of the internal standard from the first calibration blank is accepted. In the case this variation is bigger; it might indicate problems with the instrument (blocked nebulizer) or with the sample solution.

For procedure A (8.3), calculate the intensity ratio, R_I, of each element using the corresponding Formulae (1) and (2).

For procedure B (8.4), calculate the corrected intensity of each element using Formula (4) and the corresponding Formulae (5) and (6).

Allow sufficient rinsing time after each sample analysis.

The drift of the spectrometric system has to be checked before and at the end of the sample series or at least between every 10 samples using the control solution sample (7.6). If manganese content or iron content of the control solution differs from the reference value by more than $10\ \%$, proceed as indicated in 8.5.

10 Calculation

The content of iron and manganese of the sample solution are calculated from the intensity ratio of each element using Formula (3) (if Procedure A is used) or from the corrected intensity of each element using Formula (7) (if Procedure B is used). This is done with use of the appropriate software functions of the ICP spectrometer.

The content of iron or of manganese of the petrol sample is calculated according to Formula (8):

$$C_{\text{Element}} = \frac{S_{\text{Element}} \times \rho \times 20}{m_s} \tag{8}$$

where

$C_{Element}$ is the content of iron or manganese of the petrol sample, expressed in mg/l;

$S_{Element}$ is the content of iron or manganese of the sample solution, expressed in mg/l;

ρ is the density of the petrol sample (measured by EN ISO 12185 or EN ISO 3675 at 15 °C and corrected to the temperature at which the analysis is done), expressed in g/ml;

20 is the volume of the tested solution in the flask (9.1), expressed in ml;

m_S is the mass of sample volume used (9.1), expressed in g.

11 Expression of results

Report the content of manganese and iron in mg/l, rounded to the nearest 0,01 mg/l.

12 Precision

12.1 General

The precision given in 12.2 and 12.3 was determined by statistical examination of interlaboratory test results in accordance with EN ISO 4259 [1].

12.2 Repeatability, *r*

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 following value in only one case in twenty.

Iron $r = 0{,}034\,4\,X + 0{,}107\,3$ (9)

Manganese $r = 0{,}068\,2\,X + 0{,}065\,6$ (10)

where

X represents the mean of the two results expressed in mg/l.

12.3 Reproducibility, *R*

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 following value in only one case in twenty.

Iron $R = 0{,}158\,1\,X + 0{,}492\,8$ (11)

Manganese $R = 0{,}169\,0\,X + 0{,}162\,6$ (12)

where

X represents the mean of the two results expressed in mg/l.

13 Test report

The test report shall specify:

a) the reference to this European Standard, i.e. EN 16136;

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

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

d) the procedure used (see Clause 8)

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

f) the test result (see Clause 11);

g) the date of the test.

Bibliography

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

[2] *Directive 2009/30/EC of the European Parliament and of the Council of 23 April 2009 amending Directive 98/70/EC as regards the specification of petrol, diesel and gas-oil and introducing a mechanism to monitor and reduce greenhouse gas emissions and amending Council Directive 1999/32/EC as regards the specification of fuel used by inland waterway vessels and repealing Directive 93/12/EEC*, OJ L 140, 5.6.2009, p. 88-113

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