



SLOVENSKI STANDARD
SIST EN 13131:2000

01-julij-2000

HY_c]'bUzb]dfc]nj cX]'8 c`c Yj Ub^b]_`U]b]j UbUX]U!'5 hca g_UUVgcf dW]g_U
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Liquid petroleum products - Determination of nickel and vanadium content - Atomic
absorption spectrometric method

Flüssige Mineralölerzeugnisse - Bestimmung des Gehaltes an Nickel und Vanadium -
Atomabsorptionsspektrometrie

Produits pétroliers liquides - Détermination de la teneur en nickel et en vanadium -
Méthode par spectrométrie d'absorption atomique

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Ta slovenski standard je istoveten z: EN 13131:2000

ICS:

75.080	Naftni proizvodi na splošno	Petroleum products in general
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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 13131

March 2000

ICS 75.080

English version

Liquid petroleum products - Determination of nickel and vanadium content - Atomic absorption spectrometric method

Produits pétroliers liquides - Détermination de la teneur en nickel et en vanadium - Méthode par spectrométrie d'absorption atomique

Flüssige Mineralölzeugnisse - Bestimmung des Gehaltes an Nickel und Vanadium - Atomabsorptionsspektrometrie

This European Standard was approved by CEN on 14 February 2000.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products", the secretariat of which is held by NNI.

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of an identical text or by endorsement, at the latest by September 2000, and conflicting national standards shall be withdrawn at the latest by September 2000.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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1 Scope

This European Standard specifies a method for the determination of nickel and vanadium by atomic absorption spectrometry in liquid petroleum products with nickel and vanadium contents greater than 5 mg/kg.

NOTE For the purposes of this European Standard, the term “% (m/m)” is used to represent the mass fraction.

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

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN ISO 3170, *Petroleum liquids - Manual sampling (ISO 3170:1988, including Amendment 1:1998)*

EN ISO 3171, *Petroleum liquids - Automatic pipeline sampling (ISO 3171:1988)*

ISO 648, *Laboratory glassware - One-mark pipettes.*

ISO 4797, *Laboratory glassware - Flasks with conical ground joints.*

3 Principle

The sample is diluted with an organic solvent, the solution aspirated into the flame of an atomic absorption spectrometer, and the absorbance of the characteristic radiation of nickel or vanadium is measured. An acetylene/air flame is used for nickel and a nitrous oxide/acetylene flame for vanadium. Calibration curves are obtained from measurements on calibration solutions of fuels with a known content of nickel and/or vanadium in the same solvent or from measurements on calibration solutions of organometallic compounds in the same solvent.

4 Reagents and materials

Use only reagents of recognized analytical grade.

4.1 Xylene, isomer mixture.

4.2 Propan-2-ol.

4.3 Standard fuel oils, or organometallic standards in oil, with known contents of nickel and/or vanadium.

4.4 Base oil, free of nickel and vanadium.

5 Apparatus

Usual laboratory apparatus and glassware, together with the following.

5.1 Flame atomic absorption spectrometer, suitable for measurements at wavelengths of 352,5 nm (for nickel) and 318,4 nm (for vanadium), fitted with a gas burner with provisions for the aspiration of organic liquids into the flame, and with burner heads capable of burning air-acetylene and nitrous oxide-acetylene mixtures.

NOTE For some spectrometers it may be better to use a wavelength of 232,0 nm for nickel. At this wavelength, there may be some nonlinearity. The precision data quoted in clause 10 for nickel is based on analyses using a wavelength of 352,5 nm.

5.2 Nickel hollow-cathode lamp

5.3 Vanadium hollow-cathode lamp

5.4 One-mark pipettes, conforming to ISO 648.

5.5 Flasks, of suitable capacity, conforming to ISO 4797.

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 standards or regulations for the sampling of the product under test.

7 Procedure

7.1 Preparation of calibration solutions and the blank solution

7.1.1 Preparation of calibration solutions of nickel

Prepare a set of calibration solutions with nickel contents of 0,5 mg/kg, 1 mg/kg, 2 mg/kg, 4 mg/kg and 7 mg/kg respectively.

Either

a) heat standard fuels with known nickel contents (4.3) to $60\text{ °C} \pm 5\text{ °C}$ and agitate vigorously i.e. by shaking by hand for at least 1 min. Dilute appropriately by mass with xylene (4.1). Prepare fresh solutions every day;

or

b) dilute organometallic standards in oil (4.3) by mass with nickel free base oil (4.4) to obtain standards with nickel contents of 5 mg/kg, 10 mg/kg, 20 mg/kg, 40 mg/kg and 70 mg/kg. Further dilute 1+9 by mass with xylene (4.1). Prepare freshly every day.

A stabilizer, supplied by the manufacturer of the organometallic standards in oil, may be used. In this case, the base oil diluted intermediate solutions are stable up to three months and need not be made freshly every day.

If the sample contains more than 1 % (*m/m*) water, use as solvent a mixture of xylene (4.1) and propan-2-ol (isopropyl alcohol) (4.2) in the ratio of about 9:1, by mass or by volume instead of xylene only.

NOTE A simple and efficient test to the presence of water can be executed by heating a few droplets of the sample in a test tube. In the presence of water, a clearly audible crackling noise will appear.

7.1.2 Preparation of calibration solutions of vanadium

Prepare a set of calibration solutions with vanadium contents of 1 mg/kg, 2 mg/kg, 4 mg/kg, 10 mg/kg and 20 mg/kg respectively.

Either

a) heat standard fuels with known vanadium contents (4.3) to $60\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ and agitate vigorously i.e. by shaking by hand for at least 1 min. Dilute appropriately by mass with xylene (4.1). Prepare freshly every day;

or

b) dilute organometallic standards in oil (4.3) by mass with vanadium free base oil (4.4) to obtain standards with vanadium contents of 10 mg/kg, 20 mg/kg, 40 mg/kg, 100 mg/kg and 200 mg/kg. Further dilute 1+9 by mass with xylene (4.1). Prepare freshly every day.

A stabilizer, supplied by the manufacturer of the organometallic standards in oil, may be used. In this case, the base oil diluted intermediate solutions are stable up to three months and need not be made freshly every day.

If the sample contains more than 1 % (*m/m*) water, use as solvent a mixture of xylene (4.1) and propan-2-ol (isopropyl alcohol) (4.2) in the ratio of about 9:1, by mass or by volume, instead of xylene only (see note to 7.1.1).

7.1.3 Preparation of the blank solution

Prepare the blank solution by diluting the base oil (4.4) 1+9 by mass with xylene (4.1).

If the sample contains more than 1 % (*m/m*) water, use as solvent a mixture of xylene (4.1) and propan-2-ol (isopropyl alcohol) (4.2) in the ratio of about 9:1, by mass or by volume, instead of xylene only (see note to 7.1.1).

7.2 Preparation of the test solution

Heat the sample to $60\text{ °C} \pm 5\text{ °C}$ and agitate vigorously, i.e. by shaking by hand for at least 1 min. Filter the heated sample through a glass fibre filter to remove solid parts. Weigh approximately 5 g to the nearest 0,05 g in a suitable flask (5.5) and dilute 1+9 by mass with xylene (4.1).

NOTE 1 In the presence of particulates or insoluble matter it may be necessary to filter the sample in order to avoid blockage of the nebulizer capillary. If this filtration step is required, a subsample of sufficient mass to carry out the analysis may be used.

If the sample contains more than 1 % (*m/m*) water, use as solvent a mixture of xylene (4.1) and propan-2-ol (isopropyl alcohol) (4.2) in the ratio of about 9:1, by mass or by volume, instead of xylene only (see note to 7.1.1).

NOTE 2 A simple and efficient test to the presence of water can be executed by heating a few droplets of the sample in a test tube. In the presence of water, a clearly audible crackling noise will appear.

7.3 Preparation of the apparatus

Follow the manufacturer's instructions for the operation of the atomic absorption spectrometer. Install the hollow-cathode lamp (5.2) for the element to be determined (see table 1) in the spectrometer (5.1) and allow the apparatus to become stable.

Adjust the lamp current, the attenuation and the slit, to suit the characteristics of the apparatus.

Set to the appropriate wavelength (see table 1) and adjust in order to obtain maximum intensity.

Install the burner head for the appropriate flame (see table 1), ignite the flame, ensure that the flame is stable and adjust the height of the burner's head to obtain maximum response.

Aspirate the calibration solution containing 4 mg/kg of nickel or the calibration solution containing 10 mg/kg of vanadium, according to the element to be determined. Slightly move the burner head horizontally to obtain maximum absorbance. Adjust the flow rates of the gas mixture for the flame so as to achieve an aspiration flow of the calibration solution of nickel or of vanadium of 1 ml/min to 3 ml/min.

WARNING Proper operation procedures are required for safety. An explosion can occur from flame flashback unless the correct burner head and operating sequence are used.

Table 1 - Apparatus conditions for nickel and vanadium

Element	Wavelength nm	Flame
Nickel	352,5	air/acetylene
Vanadium	318,4	nitrous oxide/acetylene

7.4 Determination

Take readings only after a stable flame has been achieved. Set the spectrometer to a reading time of a minimum of 3 s and to a minimum of 3 readings. Aspirate the blank solution (7.1.3) and set the absorbance to zero. Aspirate the series of five calibration solutions (7.1.1 or 7.1.2) with increasing contents successively into the flame, and record the absorbance of each. Aspirate the test solution (7.2) and record the absorbance value. Take care to keep the rate of aspiration constant throughout the measurements. Aspirate the blank solution between every two other solutions in order to minimize memory effects.

If the absorbance value of the test solution exceeds the absorbance value of the calibration solution with the highest content of nickel or vanadium, further dilute the test solution to obtain an absorbance value falling within the calibration range. The oil of the diluted sample shall be adjusted with the metal-free base oils during the dilution step in order to maintain the 1+9 ratio.

If a number of test solutions is to be measured, aspirate the blank solvent in order to check the zero and aspirate one calibration solution in order to check the calibration every five tests. Select this calibration solution whose metal content is expected to be in the working range of the analysis.

8 Calculation

Calculate the arithmetic mean of the three readings for each of the calibration solutions and for the test solution. Plot a graph having the contents of nickel or of vanadium in the calibration solutions as abscissae and the corresponding absorbances as ordinates.

Determine the calibration graph and the parameters for the regression line by applying the linear regression method using the metal contents of nickel or vanadium as abscissae and the corresponding absorbances as ordinates. This can be done manually or with the help of a computer.

Some instruments will provide the results directly in terms of the requested metal content. In that case it has to be assured that the software used performs according to the manual calculation given in this clause.

Calculate the nickel or vanadium content of the sample, W_{Me} , expressed in milligrams per kilogram, using the equation:

$$W_{Me} = (m_s + m_o) / m_s \times (A - K) / H$$

where:

- m_s is the mass of the test portion, in grams
- m_o is the mass of the diluent, in grams
- A is the absorbance value of the sample solution
- K is the ordinate intercept
- H is the slope of the regression line