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Liquid petroleum products - Determination of sodium content - Atomic absorption spectrometric method

Flüssige Mineralölerzeugnisse - Bestimmung des Natriumgehaltes - Atomabsorptionsspektrometrie

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

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**Ta slovenski standard je istoveten z: EN 241:2000**

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**ICS:**

75.160.20      V^[\ æ\ [ !ãæ      Liquid fuels

**SIST EN 241:2000**      **en**

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

EN 241

March 2000

ICS 75.160.20

English version

## Liquid petroleum products - Determination of sodium content - Atomic absorption spectrometric method

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

Flüssige Mineralölerzeugnisse - Bestimmung des Natriumgehaltes - Atomabsorptionspektrometrie

This European Standard was approved by CEN on 18 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.

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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of an identical text or by endorsement, at the latest by September 2000, and conflicting national standards shall be withdrawn at the latest by September 2000.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 the sodium content of crude oils, liquid petroleum products, heating oils, residual oils and mineral oil distillates by means of atomic absorption spectrometry after incineration of the products.

NOTE For the purpose 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)*.

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*.

ISO 385-1, *Laboratory glassware - Burettes - Part 1: General requirements*.

ISO 1042, *Laboratory glassware - One-mark volumetric flasks*.

## 3 Principle

The sample is carefully burnt in a platinum crucible (or platinum dish) and the carbon residue incinerated in a muffle furnace at 550 °C. The residue is taken up with water free from sodium and the sodium content of the aqueous solution is determined by means of atomic absorption spectrometry at a wavelength of 589,0 nm.

## 4 Reagents and materials

Use only reagents of recognized analytical grade and water conforming to grade 3 of EN ISO 3696.

**4.1 Hydrochloric acid**,  $c(\text{HCl}) =$  approximately 1 mol/l.

**4.2 Hydrofluoric acid**, 38 % (*m/m*) to 40 % (*m/m*), sodium free.

**WARNING** Great care shall be taken in the handling of hydrofluoric acid as it causes severe burns and permanent injury. Consult the manufacturer's handling instructions. Gloves and

eye-protection shall be worn when handling the acid solutions. If the acid does come in contact with the skin, wash the affected area with a continuous stream of water and seek immediate medical assistance.

**4.3 Sulfuric acid, 10 % (m/m), sodium free.**

**4.4 Air**, oil free, under pressure in a steel cylinder or pipe.

**4.5 Acetylene**, under pressure in a steel cylinder or pipe.

**4.6 Aqueous standard sodium solution**,  $c(\text{Na}) = 0,1 \text{ g/l}$  ( $w(\text{Na})=100 \text{ mg/kg}$ ).

Either

a) dry sodium chloride for at least 10 h at  $(105 \pm 5) \text{ }^\circ\text{C}$  and store it in a desiccator. Dissolve  $(2,543 \pm 0,001) \text{ g}$  of the dried sodium chloride in water in a 1000 ml volumetric flask (5.5) and make up to the mark. Transfer 10 ml of this solution into a 100 ml volumetric flask (5.5), add 1 ml of hydrochloric acid (4.1) and make up to the mark with water;

or

b) transfer 434,8 ml of a sodium chloride solution,  $c(\text{NaCl}) = 0,01 \text{ mol/l}$ , into a 1000 ml volumetric flask (5.5), add 10 ml of the hydrochloric acid (4.1) and make up to the mark with water;

or

c) as an alternative, commercially available and certified standards may also be used.

**4.7 Blank solution**, water (see clause 4), to which 1 ml of the hydrochloric acid (4.1) per 100 ml water has been added.

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## 5 Apparatus

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Usual laboratory apparatus and glassware, together with the following:

**5.1 Flame atomic absorption spectrometer**, suitable for measurements at a wavelength of 589,0 nm, and fitted with a burner feed for acetylene and air.

**5.2 Sodium hollow-cathode lamp.**

**5.3 Incineration vessel**, either a platinum crucible, capacity approximately 50 ml, diameter approximately 35 mm, or a platinum dish, capacity approximately 130 ml, diameter approximately 75 mm, freed from sodium by rinsing with the hydrochloric acid (4.1).

**5.4 Muffle furnace**, electrically heated and capable of maintaining a temperature of  $(550 \pm 25) \text{ }^\circ\text{C}$ .

**5.5 One-mark volumetric flasks**, capacity 50 ml, 100 ml, 250 ml and 1000 ml, conforming to class A of ISO 1042.

**5.6 Polyethene bottles**, for storage of the calibration solutions.

**5.7 Burette**, capacity 50 ml, graduated in 0,1 ml, conforming to class A of ISO 385-1. Automatic burettes or pipettes may be used, provided that they meet the same requirements.

## 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.

Prepare test samples by pouring a suitable quantity of sample into clean containers of glass, polypropene (PP) or polytetrafluoroethene (PTFE) that have been freed from sodium by rinsing twice with the hydrochloric acid (4.1), rinsed with water and dried.

## 7 Procedure

### 7.1 Preparation of the calibration graph

#### 7.1.1 Preparation of a set of calibration solutions

Prepare a set of calibration solutions with a sodium content as given in table 1 as follows.

Using a burette (5.7), transfer volumes of the standard sodium solution (4.6) as given in table 1 into a series of eight one-mark 250 ml volumetric flasks (5.5). Make each up to the mark with water and mix well. Transfer to polyethene bottles (5.6) for storage. The prepared calibration solutions are stable for a maximum of 8 weeks and shall not be used for longer periods.

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**Table 1 - Preparation of calibration solutions**

Number of calibration solution	Standard sodium solution (4.6) ml	w(Na) mg/kg
0*	0	0
1	2,5	1
2	5	2
3	7,5	3
4	10	4
5	12,5	5
6	15	6
7	17,5	7
* Blank solution		

#### 7.1.2 Spectrometric measurements

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 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 the wavelength to 589,0 nm and adjust in order to obtain maximum intensity. For some spectrometers, it may be better to use a wavelength of 589,6 nm instead of 589,0 nm because of sensitivity reasons. Install the burner head for acetylene-air and ignite the flame, ensure that the flame is stable, and adjust its height to obtain maximum response. Adjust the flow rates of the acetylene (4.5) and of the air (4.4).

Take readings only after a stable flame has been achieved. Aspirate the series of eight calibration solutions (7.1.1) with increasing contents successively into the flame, and measure the absorbance of each. Aspirate water through the burner after each measurement. Take care to keep the rate of aspiration constant throughout the preparation of the calibration graph.

Establish zero point by aspirating the blank solution (4.7) between every two calibration solutions in order to check the zero point.

### 7.1.3 Calibration graph

If the spectrometer does not automatically produce the calibration graph, plot a graph having the sodium content, in milligrams per kilogram, in the calibration solutions as abscissae and the corresponding absorbance values as ordinates.

If it has been previously established that the calibration curve in the specified working range is a straight line, it may be plotted using three calibration solutions, one at the low end of the range, one in the middle and one at the high end.

NOTE If the spectrometer output is linear in absorbance, the net absorbance is given by the difference between the absorbance for the calibration solution and the absorbance for the blank. If the spectrometer output is proportional to transmission (i.e. to light intensity) then the net absorbance is given by  $\lg(\alpha_0/\alpha)$ , where the readings are  $\alpha_0$  when the blank is aspirated and  $\alpha$  when the calibration or test solution is aspirated.

## 7.2 Preparation of test solution SIST EN 241:2000

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7.2.1 Heat a test sample to approximately 50 °C and shake it vigorously for at least one minute or use a highspeed mixer in order to disperse any sediment and to render viscous samples homogenous.

NOTE When using a high speed mixer, ensure that it is cleaned between consecutive uses in order to avoid contamination of samples.

In the presence of components which boil below 50 °C, the test sample has to be weighed before and after homogenization in order to account for any potential loss of these components.

Assess by a suitable method the range of the sodium content of the sample (see table 2) and weigh a test portion in accordance with table 2 into a clean dry incineration vessel (5.3).

**Table 2 - Mass of test portion**

Range of the sodium content of the sample mg/kg	Approximate mass of test portion g	Weigh to the nearest g
up to 10	25	0,05
greater than 10 and up to 25	10	0,02
greater than 25 and up to 50	5	0,01
greater than 50	3	0,005



If the sensitivity of the atomic absorption spectrometer varies over the absorption range 0,1 to 0,6, the mass of the test portion shall be adjusted to give an absorbance in the straight section of the calibration curve.

**7.2.2** Heat the incineration vessel with a bunsen burner on a triangle under a hood until the contents ignite and burn slowly. Adjust the burner to obtain smooth combustion until the oil has carbonized. Handle the vessel with platinum tipped crucible tongs of forceps.

NOTE 1 It is important to make sure that foaming is avoided in the process of burning the sample.

Ash the carbonized residue by heating in the muffle furnace (5.4) at a temperature of  $(550 \pm 25)$  °C until all the carbon has been burned. Remove the incineration vessel from the furnace and allow to cool to room temperature in a desiccator.

If the silicon content of the ash is expected to be 10 mg/kg or less, the ashing is herewith completed.

If the silicon content of the ash is expected to be more than 10 mg/kg, add 1 ml of the sulfuric acid (4.3) and 1 ml of hydrofluoric acid (4.2). Evaporate to dryness under a hood, transfer to the muffle furnace (5.4) and heat for 15 min at a temperature of  $(550 \pm 25)$  °C. Remove the incineration vessel from the furnace and allow to cool to room temperature in a desiccator.

NOTE 2 If a high content of silicon is present, a low result may be obtained as some of the sodium may be retained in the insoluble silica.

NOTE 3 If hydrofluoric acid is used, a full system blank should be carried out.

**7.2.3** Add a few millilitres of water and 1 ml of the hydrochloric acid (4.1). Heat gently, allow to cool and transfer the contents of the incineration vessel quantitatively to a 50 ml volumetric flask (5.5), make up to the mark with water and mix well.

### 7.3 Determination

Following the procedure as given in 7.1.2, aspirate the blank solution and set the instrument to zero, then aspirate the test solution and measure the absorbance. Carry out these measurements a minimum of three times and calculate the arithmetic mean of the test solution measurements. Using this arithmetic mean, read from the calibration curve the sodium content, in milligrams per kilogram, of the test solution. If a number of tests are to be carried out, check zero and one calibration sample every five tests.

If the sodium content of the test portion exceeds the range of the calibration curve, dilute it accordingly, and repeat the determination.

## 8 Calculation

Calculate the sodium content of the sample,  $W_{Na}$ , expressed in milligrams per kilogram, using the equation:

$$W_{Na} = \frac{V \times C}{m}$$

where:

$C$  is the sodium content read from the calibration curve, in milligrams per kilogram;