SLOVENSKI STANDARD
SIST EN 237:1998

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Tekoļ i naftni proizvodi - Bencin - Doloļ evanje nizkih vsebnosti svinca z atomsko absorpcijsko spektrometrijo

Liquid petroleum products - Petrol - Determination of low lead concentrations by atomic absorption spectrometry

Flüssige Mineralölerzeugnisse - Ottokraftstoff - Bestimmung von niedrigen Bleigehalten durch Atomabsorptionsspektrometrie

Produits pétroliers liquides - Essence - Détermination des basses teneurs en plomb par spectrométrie d'absorption atomique

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ICS:

75.160.20	Tekoļ a goriva	Liquid fuels

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English version

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The European Standards exist 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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CEN

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 the 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of an identical text or by endorsement, at the latest by July 1996, and conflicting national standards shall be withdrawn at the latest by July 1996.

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

This European Standard is based on DIN 51 769 Part 8: "Determination of the lead content (total lead) of gasolines with a mass concentration of lead of 5 mg/l to 25 mg/l, direct determination by atomic absorption" (October 1981). DIN 51 769 Part 8 is technically equivalent with ASTM D 3237-79 "Standard Test Method for Lead in Gasoline By Atomic Absorption Spectroscopy".

1 Scope

This European Standard specifies an atomic absorption spectrometric method for the determination of the total lead content of petrol with a lead content of 5 mg/l to 25 mg/l. This method is independent of the lead alkyl type.

WARNING : The use of this European Standard may 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 3696	Water for analytical laboratory use - Specification and test methods
ISO 385-1	Laboratory glassware - Burettes - Part 1: General requirements
ISO 648	Laboratory glassware - One-mark pipettes
ISO 1042	Laboratory glassware - One-mark volumetric flasks
ISO 3170	Petroleum liquids - Manual sampling
ISO 3171	Petroleum liquids - Automatic pipeline sampling

3 Principle

The sample, diluted with propan-2-ol (isopropyl alcohol) and treated with iodine, is aspirated into the air/acetylene flame of an atomic absorption spectrometer. The absorbance is measured at a wavelength of 217,0 nm and is compared with that of calibration solutions of known lead concentrations.

4 Reagents and materials

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

4.1 Concentrated lead standard stock solution, c(Pb) = 1000 mg/l.

Dissolve 183,1 mg lead (II) acetate 3-hydrate in a mixture of 5 ml acetic acid and 5 ml water in a volumetric flask (5.4) at approximately 20 °C. Dilute to the mark with propan-2-ol (4.3).

NOTE : The concentrated lead standard stock solution is stable for at least 6 months.

4.2 Diluted lead standard stock solution, c(Pb) = 50 mg/l.

Pipette (see 5.5), 5,0 ml of the concentrated lead standard stock solution (4.1) into a volumetric flask (5.4), and dilute to the mark with propan-2-ol (4.3).

4.3 Propan-2-ol

4.4 Toluene

4.5 2,2,4-Trimethylpentane (iso-octane)

4.6 Iodine solution. Dissolve 20 g of sublimated iodine in propan-2-ol (4.3). Make up to 1 litre with propan-2-ol (4.3).

4.7 Methyltrioctylammonium chloride solution. Dissolve 10 g of methyltrioctylammonium chloride in propan-2-ol (4.3). Make up to 1 litre with propan-2-ol (4.3).

4.8 Air, oil free, under pressure in a steel cylinder, or compressed air.

4.9 Acetylene, under pressure in a steel cylinder.

WARNING : Compressed gases shall be stored outside the laboratory.

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1 Flame atomic absorption spectrometer, suitable for measurements at a wavelength of 217,0 nm, and fitted with a burner feed with acetylene and air, suitable for use with organic solutions.

5.2 Lead hollow-cathode lamp.

5.3 Micro-burette, capacity 5 ml, conforming to class A of ISO 385-1.

5.4 One-mark volumetric flasks, capacity 100 ml, conforming to class A of ISO 1042.

5.5 One-mark pipettes, capacity 10 ml, 5 ml, and 1 ml, conforming to class A of ISO 648, with suction ball.

6 Sampling

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

7 Procedure

7.1 General

Measure all volumes at a temperature within 5 °C of the temperature at which the volumetric glassware was calibrated.

NOTE : This temperature is usually 20 °C.

Prepare the calibration solutions and the test solution on the same day and measure on that day.

7.2 Preparation of the calibration graph

7.2.1 Preparation of a set of calibration solutions

Prepare a set of calibration solutions with a lead content of 0,5 mg/l, 1,0 mg/l, 1,5 mg/l, 2,0 mg/l and 2,5 mg/l as follows.

Using a microburette (5.3), transfer 1,0 ml, 2,0 ml, 3,0 ml, 4,0 ml, and 5,0 ml respectively of the diluted lead standard stock solution (4.2) into a series of five 100 ml one mark volumetric flasks (5.4) each containing approximately 50 ml of propan-2-ol (4.3) and 10,0 ml of a mixture of 50 % (*V/V*) of toluene (4.4) and 50 % (*V/V*) iso-octane (4.5). Add 1 ml of iodine solution (4.6), agitate and allow to stand for 1 minute. Add 5 ml of methyltrioctylammonium chloride solution (4.7) and dilute to the mark with propan-2-ol (4.3).

7.2.2 Preparation of the blank solution

Follow the procedure given in 7.2.1 without adding the lead standard stock solution.

7.2.3 Spectrometric measurements

Install the hollow-cathode lamp (5.2) in the spectrometer (5.1) and leave the apparatus switched on for the time necessary to achieve stability.

Adjust the lamp current, the attenuation and the slit, to suit the characteristics of the apparatus. Adjust the wavelength to the region of 217,0 nm in order to obtain the maximum intensity. Install the burner-head for acetylene-air and ignite the flame. Adjust the flow rates of the acetylene (4.9) and of the air (4.8). Set the acetylene-air mixture as lean as possible, i.e. so that the flame remains just attached to the sides of the burner.

Readings shall only be taken after a stable flame has been achieved. Aspirate the blank solution (7.2.2) and the set of calibration solutions (7.2.1), with increasing concentration 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 (7.2.2) between every two calibration solutions in order to check the zero point.

7.2.4 Calibration graph

If the spectrometer does not automatically produce the calibration graph, plot a graph having the lead concentration, in milligrams per litre, in the calibration solutions as abscissae and the corresponding absorbance values, reduced by the value for the blank solution, as ordinates.

7.3 Preparation of the test solution

Transfer approximately 50 ml of propan-2-ol (4.3) into a volumetric flask (5.4) and add 10,0 ml of the sample by means of a pipette, using the suction ball (5.5). When handling the sample with the suction ball (5.5), ensure that no vapour bubbles are formed while filling the pipette. Add 1 ml of the iodine solution (4.6), agitate and allow to stand for 1 minute. Add 5 ml of the methyltrioctylammonium chloride solution (4.7) and dilute to the mark with propan-2-ol (4.3).

If less than 10 ml of sample are used, make up the volume to 10 ml using the mixture of 50 % (*V/V*) toluene (4.4) and 50 % (*V/V*) iso-octane (4.5) (see 7.2.1).

7.4 Determination

Following the procedure as given in 7.2.3 measure the test solution and then the calibration solution that shows the absorbance closest to that of the test solution.

8 Calculation

Calculate the lead content of the sample, ρ(Pb), expressed in milligrams per litre, using the equation:

$$\rho(\mathrm{Pb}) = \frac{A \times \rho B(\mathrm{Pb}) \times 10}{A_1}$$

where :

A is the absorbance of the test solution;

ρB(Pb) is the mass concentration of lead in the calibration solution in milligrams per litre.

A_1 is the absorbance of the calibration solution that shows the closest absorbance to the test solution;

If sample volumes less than 10,0 ml are used, the factor 10 in the above equation shall be changed to $100/V_s$, where V_s is the volume of the sample in millilitres.

9 Expression of results

Report the lead concentration to the nearest 1 mg/l.

NOTE : If results are required in grams per litre, divide by 1000.