SLOVENSKI STANDARD
SIST CR 13840:2000

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Naftni proizvodi - Določanje policikličnih aromatskih ogljikovodikov - Ultravijolična (UV) spektrometrijska metoda

Petroleum products - Determination of polycyclic aromatic hydrocarbons - Ultraviolet (UV) spectrometric method

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ICS

English version

Petroleum products - Determination of polycyclic aromatic hydrocarbons - Ultraviolet (UV) spectrometric method

Produits pétroliers - Détermination des hydrocarbures aromatiques polycycliques - Méthode par spectrométie ultraviolette (UV)

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This CEN Report has been prepared by Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products", the Secretariat of which is held by the Netherlands Standardization Institute (NNI)).

1. Scope

This CEN Report describes a method of test which can be used for the determination of polycyclic aromatic hydrocarbons (and compounds of similar structure) in diesel fuels and petroleum distillates in the concentration range 1 g/l to 40 g/l.

The method described is applicable to all hydrocarbon mixtures that are completely soluble in cyclohexane and is not restricted to a particular boiling range.

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

2. Normative references

This CEN Report 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 CEN Report only when incorporated in it by amendment of revision. For undated references the latest edition of the publication referred to applies.

EN ISO 3170, *Petroleum liquids - Manual sampling.*

EN ISO 3171, *Petroleum liquids - Automatic pipeline sampling.*

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

EN ISO 4259, *Petroleum products - Determination and application of precision data in relation to methods of test.*

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

3. Principle

The absorbance due to polycyclic aromatic hydrocarbons in solution in cyclohexane is measured using an ultraviolet (UV) spectrometer and the concentration of these determined by reference to the optical density (OD) at 260 nm ± 10 nm.

4. Reagents

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

4.1 Cyclohexane.

CAUTION: Care should be taken when using cyclohexane as a solvent.

5. Apparatus

5.1 UV spectrometer, double beam, capable of measuring absorption over the wavelength range 240 nm to 340 nm and providing a scale covering optical densities from 0,1 to 1,0.

NOTE: The manufacturer's instructions for optimumum set-up and operating conditions for the spectrometer should be followed.

5.2 Quartz cells, matched pairs for reference and sample, between 1 mm and 50 mm optical path length.

NOTE: The reference cell and the sample cell pair should have the same optical path length selected to give an absorption of between 0,5 OD and 1,0 OD at 260 nm for the sample solution under examination.

5.3 Analytical balance, capable of weighing to an accuracy of 0,0001 g.

5.4 Volumetric flask, 100 ml capacity, class A in accordance with ISO 1042.

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 Weigh, to the nearest 0,001 g, between 0,1 g and 4,0 g of sample into a 100 ml volumetric flask (5.4) and make up to the mark with cyclohexane (4.1). Shake thoroughly to dissolve, using an ultrasonic bath for 5 min to facilitate dissolution, if necessary.

NOTE. An appropriate sample size of between 0,1 g and 4,0 g should be selected to ensure an absorption of between 0,5 OD and 1,0 OD for cells of optical pathlength between 1 mm and 50 mm (see NOTE to 5.2).

7.3 Fill the reference and sample cells (5.2) with cyclohexane and scan in the spectrometer over the range of wavelengths 240 nm to 340 nm to determine the baseline.

7.4 Empty the sample cell, refill it with sample solution and scan again in the spectrometer over the range of wavelengths 240 nm to 340 nm. Display the resulting spectrum overlaid onto the same spectrum as that for the baseline (see 7.3).

7.6 Measure the absorption (OD) at 260 nm (or at the maximum in the 250 nm to 270nm range) between the sample spectrum and the baseline obtained at the same wavelength.

8. Expression of results

Express the result as the absorbance, A, of a solution at 1 g/l concentration of sample and for a 1 mm optical path length, to four decimal places, using the following equation:

$$A = \frac{OD}{10cl}$$

Where:

OD is the optical density of the sample solution at 260 nm (or at the maximum in the 250 nm to 270 nm range);
c is the mass of the sample in 100 ml of sample solution, in grams (see 7.1);
l is the optical path length, in millimetres (see NOTE to 5.2).

9. Precision

Precision has been estimated following interlaboratory tests by 10 laboratories using 9 samples and determined by statistical examination according to EN ISO 4259 as $R = 0{,}090m$ and $r = 0{,}045m$ for m in the range (0,0000-1,5000), as the mean of two results being compared.

9.1 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 of *r* as given in clause 9 only in one case of twenty.

9.2 Reproducibility

The difference between two single and independent 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 of *R* as given in clause 9 only in one case of twenty.

10. Test report

The test report shall contain at least the following information:

a) the type and identification of the product under test;
b) a reference to this CEN Report;
c) the sampling procedure used (see clause 6);
d) the result of the test (see clause 8);
e) any deviation from the procedure described;
f) the date of the test.