SLOVENSKI STANDARD
SIST EN 15553:2007

01-junij-2007

Naftni proizvodi in sorodni materiali - Določ evanje vrste ogljikovodikov - Adsorpcijska metoda s fluorescenč nim indikatorjem

Petroleum products and related materials - Determination of hydrocarbon types - Fluorescent indicator adsorption method

Mineralölerzeugnisse und verwandte Produkte - Bestimmung der Kohlenwasserstofftypen - Adsorptionsverfahren mit Fluoreszenz-Indikator

Produits pétroliers et produits connexes - Détermination des groups d'hydrocarbures - Méthode par adsorption en présence d'indicateur fluorescent

Ta slovenski standard je istoveten z: **EN 15553:2007**

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English Version

Petroleum products and related materials - Determination of hydrocarbon types - Fluorescent indicator adsorption method

Produits pétroliers et produits connexes - Détermination des groupes d'hydrocarbures - Méthode par adsorption en présence d'indicateur fluorescent

Mineralölerzeugnisse und verwandte Produkte - Bestimmung der Kohlenwasserstofftypen - Adsorptionsverfahren mit Fluoreszenz-Indikator

This European Standard was approved by CEN on 24 February 2007.

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Foreword

This document (EN 15553:2007) 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of an identical text or by endorsement, at the latest by October 2007, and conflicting national standards shall be withdrawn at the latest by October 2007.

This European Standard is based on IP 156/06 [1]. It is developed as an alternative method to ASTM D1319 [2], in which a de-pentanization step is described, which is not used in this European Standard and the EU environment.

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, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European Standard specifies a fluorescent indicator adsorption method for the determination of hydrocarbon types over the concentration ranges from 5 % (*V/V*) to 99 % (*V/V*) aromatic hydrocarbons, 0,3 % (*V/V*) to 55 % (*V/V)* olefinic hydrocarbons, and 1 % (*V/V*) to 95 % (*V/V*) saturated hydrocarbons in petroleum fractions that distil below 315 ºC. This method may be applicable to concentrations outside these ranges, but the precision has not been determined.

When samples containing oxygenated blending components are analysed, the hydrocarbon type results can be reported on an oxygenate-free basis or, when the oxygenate content is known, the results can be corrected to a total-sample basis.

This test method is for use with full boiling range products. Cooperative data have established that the precision statement does not apply to petroleum fractions with narrow boiling ranges near the 315 °C limit. Such samples are not eluted properly, and results are erratic.

Samples containing dark-coloured components that interfere with reading the chromatographic bands cannot be analysed.

NOTE 1 The oxygenated blending components methanol, ethanol, *tert*-butyl methyl ether (MTBE), methyl *tert*-pentyl ether *(TAME)* and *tert*-butyl ethyl ether (ETBE) do not interfere with the determination of hydrocarbon types at concentrations normally found in commercial blends. These oxygenated compounds are not detected since they elute with the alcohol desorbent. The effects of other oxygenated compounds should be individually verified.

NOTE 2 For the purposes of this European Standard, the terms "% (*m/m*)" and "% (*V/V*)" are used to represent respectively the mass fraction and the volume fraction.

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 standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 1601, *Liquid petroleum products — Unleaded petrol — Determination of organic oxygenate compounds and total organically bound oxygen content by gas chromatography (O-FID)*

EN 13132, *Liquid petroleum products — Unleaded petrol — Determination of organic oxygenate compounds and total organically bound oxygen content by gas chromatography using column switching*

EN ISO 3170, *Petroleum liquids — Manual sampling (ISO 3170:2004)*

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)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
saturates
saturated hydrocarbons
volume percentage of alkanes plus cycloalkanes

3.2
olefins
olefinic hydrocarbons
volume percentage of alkenes plus cycloalkenes plus some alkadienes

3.3
aromatics
aromatic hydrocarbons
volume percentage of monocyclic and polycyclic aromatic hydrocarbons plus aromatic olefinic hydrocarbons, some dienes, compounds containing sulfur or nitrogen, or higher-boiling oxygenated compounds

4 Principle

Approximately 0,75 ml of sample is introduced into a special glass adsorption column packed with activated silica gel. A small layer of the silica gel contains a mixture of fluorescent dyes. When all the sample has been adsorbed onto the gel, alcohol is added to desorb the sample down the column. The hydrocarbons are separated, according to their adsorption affinities, into aromatics, olefins and saturates. The fluorescent dyes are also separated selectively with the hydrocarbon types, and render the boundaries of the aromatic, olefin and saturate zones visible under ultraviolet light. The volume percentage of each hydrocarbon type is calculated from the length of each zone in the column.

5 Reagents and materials

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

5.1 Silica gel, manufactured to conform to the specifications given in Annex A.

NOTE 1 Grace Davison silica gel Grade 923 meets the requirements of this specification.

Before use, dry the gel in a shallow vessel at 175 °C for at least 3 h. Transfer the dried gel to an airtight container while still hot, and protect it from atmospheric moisture.

NOTE 2 Some batches of silica gel that otherwise meet specifications have been found to produce olefin-boundary fading. The exact reason for this phenomenon is unknown but will affect accuracy and precision.

5.2 Fluorescent indicator-dyed gel, a standard dyed gel, consisting of a mixture of re-crystallized Petrol Red AB4 and purified portions of olefin and aromatic dyes obtained by chromatographic adsorption following a definite, uniform procedure, and deposited on silica gel[1)]. Store the dyed gel in a dark place under an atmosphere of nitrogen.

NOTE When stored under these conditions, dyed gel can have a shelf life of at least five years. It is recommended that portions of the dyed gel be transferred as required to a smaller working vial from which the dyed gel is routinely taken for analyses.

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1) A list of suppliers is available from the Energy Institute, London.

5.3 Propan-2-ol, 99 % (*V/V*)

5.4 3-methylbutan-1-ol, 99 % (*V/V*) (optional)

5.5 Acetone, reagent grade

5.6 Pressurizing gas, air (or nitrogen) capable of being delivered to the top of the column at controllable pressures over the range from 0 kPa gauge to 103 kPa gauge.

NOTE A special valve with possibility to regulate this pressure to ± 2 kPa. (see **Error! Reference source not found.**.2) is advised as common valves do not always have this possibility.

6 Apparatus

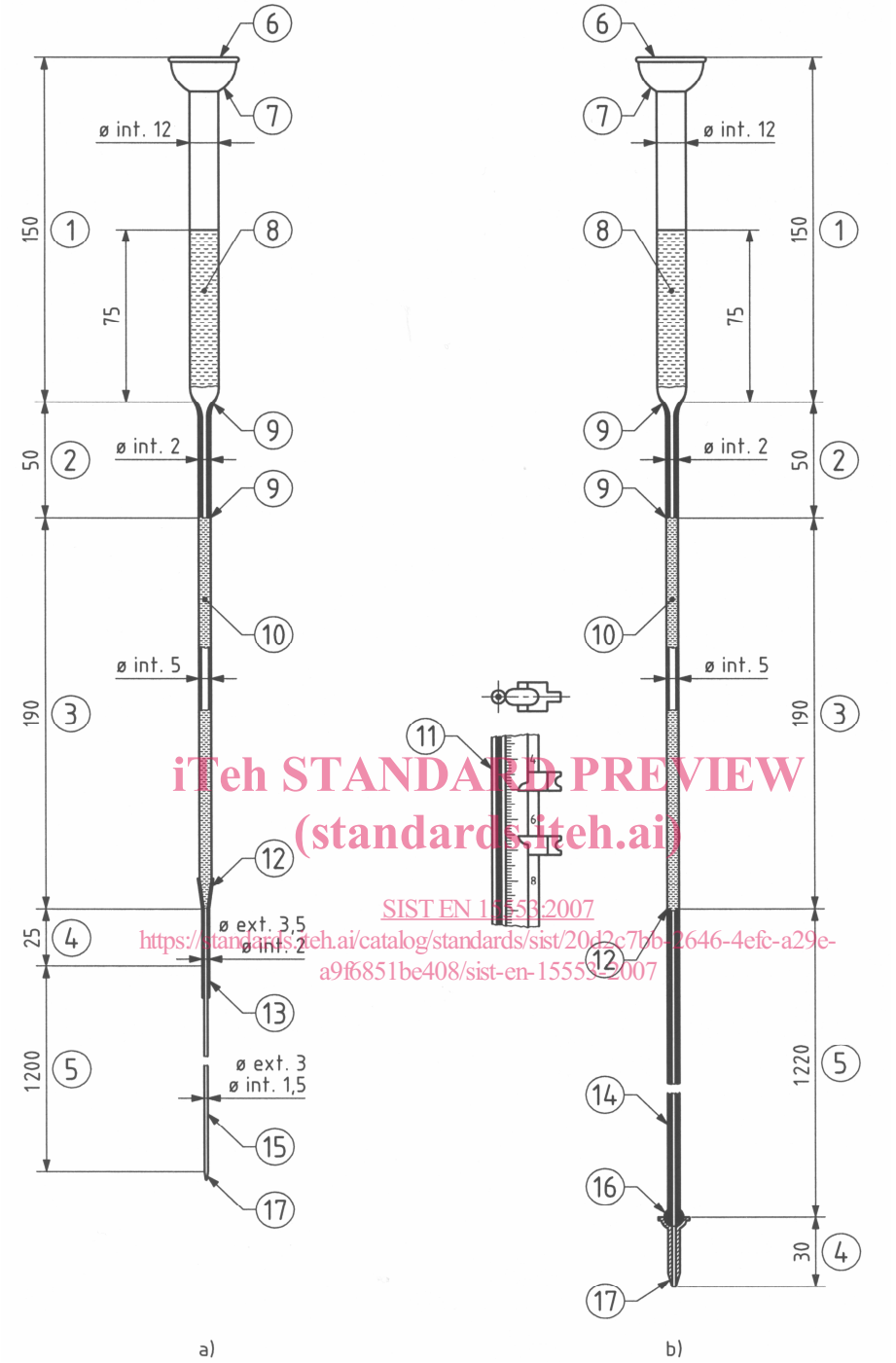
6.1 Adsorption column, with precision bore tubing conforming to the specification given in Table 1 and as shown in Figure 1, part b), made of glass and consisting of a charger section with a capillary neck, a separator section and an analyzer section.

NOTE For routine/non-specification compliance analysis adsorption columns with standard wall tubing conforming to the specification given in Annex B and as shown in Figure 1, part a), may be used.

In addition, the length of a thread of liquid approximately 100 mm long, which shall not vary in length by more than 0,3 mm in any part of the analyzer section.

Table 1 — Precision bore column dimensions and tolerance limits

Charger section	
Inside diameter	12 mm ± 2 mm
Overall length	150 mm ± 5 mm
Neck section	
Inside diameter	2 mm ± 0,5 mm
Overall length	50 mm ± 5 mm
Separator section	
Inside diameter	5 mm ± 0,5 mm
Overall length	190 mm ± 5 mm
Analyzer section	
Inside diameter	1,60 mm to 1,65 mm
Overall length	1 200mm ± 30 mm
Tip	
Overall length	30 mm ± 5 mm



Key

1 charger
2 neck
3 separator
4 tip
5 analyzer
6 pressuring gas
7 spherical joint S29
8 pack gel to this level
9 long taper
10 dyed gel
11 zone measuring device (optional)
12 long taper
13 polyvinyl tubing
14 precision bore capillary tubing
15 standard wall tubing
16 spherical joint S13
17 tip drawn out to fine capillary

Figure 1 — Adsorption columns with a) standard wall and b) precision bore tubing in analyzer section

Glass-sealing of the various sections to each other shall be done with long-taper connections rather than shouldered connections. The silica gel shall be supported with a small piece of glass wool located between the ball socket of the 12/2 spherical joint and covering the analyzer outlet. The column tip attached to the 12/2 socket shall be approximately 2 mm inside diameter. The ball and socket joints shall be clamped together to ensure that the tip does not tend to slide from a position in a direct line with the analyzer section during the packing and subsequent use of the column.

6.2 Zone-measuring-device

Either a metre rule mounted adjacent to the column, fitted with four movable metal index clips, for measuring the length of each zone, see Figure 1 and 9.9, or glass-writing pencils for marking zone boundaries and metre rule for measuring the length of each zone.

6.3 Ultraviolet light source, with radiation predominantly at wavelength 365 nm.

NOTE A convenient arrangement consists of one or two units 915 mm or 1,220 mm in length mounted vertically alongside the apparatus adjusted to give the best fluorescence.

6.4 Electric vibrator, for vibrating the individual columns or for vibrating the frame supporting multiple columns.

6.5 Hypodermic syringe, capacity 1 ml, graduated to 0,01 ml or 0,02 ml fitted with a needle 102 mm ± 2 mm in length having an inside diameter of 0,7 mm to 1,2 mm.

NOTE Needles of No. 18 gauge, 20 gauge or 22 gauge have been found to be satisfactory.

6.6 Glass funnel, with a stem of less than 10 mm external diameter.

7 Sampling and sample storage

7.1 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 unleaded petrol.

7.2 Store the sample in the dark at a temperature of 2 ºC to 4 ºC until ready for analysis.

8 Apparatus preparation

8.1 Mount the apparatus assembly in a room or area darkened to facilitate observations of zone boundaries. For multiple determinations, assemble an apparatus that includes the ultraviolet source, a rack to hold the columns and a gas manifold system with spherical joints to connect to the desired number of columns.

8.2 Freely suspend the column from a loose-fitting clamp placed immediately below the spherical joint of the charger section. Place the glass funnel (6.6) in the column. Using the vibrator (6.4) vibrate the column along its entire length and add small increments of silica gel (5.1) through the glass funnel into the charger section until the separator section is half full. Stop the vibrator and add a 3 mm to 5 mm layer of dyed gel (5.2). Re-start the vibrator and vibrate the column while adding silica gel until the tightly packed gel extends 75 mm ± 5 mm into the charger section. To aid packing by removing static electricity wipe the length of the column with a damp cloth while vibrating the column. Vibrate the column for about 4 min after filling is completed.

NOTE More than one column can be prepared simultaneously by mounting several on a frame or rack to which an electric vibrator is attached.

8.3 Attach the filled column to the apparatus assembly (see **Error! Reference source not found.** for advice).