



**SLOVENSKI STANDARD**  
**oSIST prEN 15553:2021**  
**01-april-2021**

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**Naftni proizvodi in sorodni materiali - Določevanje vrste ogljikovodikov - Adsorpcijska metoda s fluorescenčnim 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 groupes d'hydrocarbures - Méthode par adsorption en présence d'indicateur fluorescent

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**ICS:**

75.080      Naftni proizvodi na splošno      Petroleum products in general

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**en**

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**DRAFT**  
**prEN 15553**

April 2021

ICS 75.160.20

Will supersede EN 15553:2007

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 draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 19.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

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## European foreword

This document (prEN 15553:2021) 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 document is currently submitted to the CEN Enquiry.

This document will supersede EN 15553:2007. It is originally based on IP 156/06 [1]. It is intended as an alternative method to ASTM D1319 [2], which requires a de-pentanization step.

In comparison with the previous edition, the following technical modification has been made:

— lot numbers of the to be used reformulated dyed gel (5.2) that are questionable in their use have been excluded and clarification of correct references to the gel have been included.

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## 1 Scope

This document 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) olefins, and 1 % (V/V) to 95 % (V/V) saturated hydrocarbons in petroleum fractions that distil below 315 °C. This method can apply 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 petroleum blends. These oxygenated compounds are not detected since they elute with the alcohol desorbent. The effects of other oxygenated compounds are individually verified.

NOTE 2 For the purposes of this document, the terms “% (m/m)” and “% (V/V)” are used to represent respectively the mass fraction and the volume fraction.

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

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 - Determination of organic oxygenate compounds and total organically bound oxygen content in unleaded petrol - Method 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)*

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

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

### 3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

#### 3.1

##### **saturates**

volume percentage of alkanes plus cycloalkanes

#### 3.2

##### **olefins**

volume percentage of alkenes plus cycloalkenes plus some alkadienes

#### 3.3

##### **aromatics**

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

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### 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.

NOTE See NOTE 1 under Clause 1 for aromatics that the described method cannot measure.

### 5 Reagents and materials

Use only chemicals and reagents of recognized 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.

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**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<sup>1)</sup>.

IMPORTANT — In analysing any sample type by this document, users shall not report results obtained using any of the following lot numbers of fluorescent indicator dyed gel: 3000000975, 3000000976, 3000000977, 3000000978, 3000000979, and 3000000980, 3000000981, 3000000982. Current production of batches of the dyed gel will have lot numbers starting with 3000000983.

NOTE Early in 2018, a key component of the dyed gel became unavailable. An alternative dye was substituted, but the reformulated dyed gel was later found to be unsuitable for the analysis of diesel fuel and spark-ignition engine fuel. See [4] for further details.

Store the dyed gel in a dark place under an atmosphere of nitrogen.

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.

**5.3 Propan-2-ol**, 99 %

**5.4 3-methylbutan-1-ol**, 99 % (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.

## 6 Apparatus

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**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 analyser section.

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 shall not vary in length by more than 0,3 mm in any part of the analyser 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 analyser 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 analyser section during the packing and subsequent use of the column.

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<sup>1)</sup> At the moment of publication of this document, Honeywell UOP is the only supplier known of the dyed gel. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN or CENELEC of the producer named. Equivalent products may be used if they can be shown to lead to the same results, e.g. by an interlaboratory study.



**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
Analyser section	
Inside diameter	1,60 mm to 1,65 mm
Overall length	1,200mm ± 30 mm
Tip	
Overall length	30 mm ± 5 mm

## 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, 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.

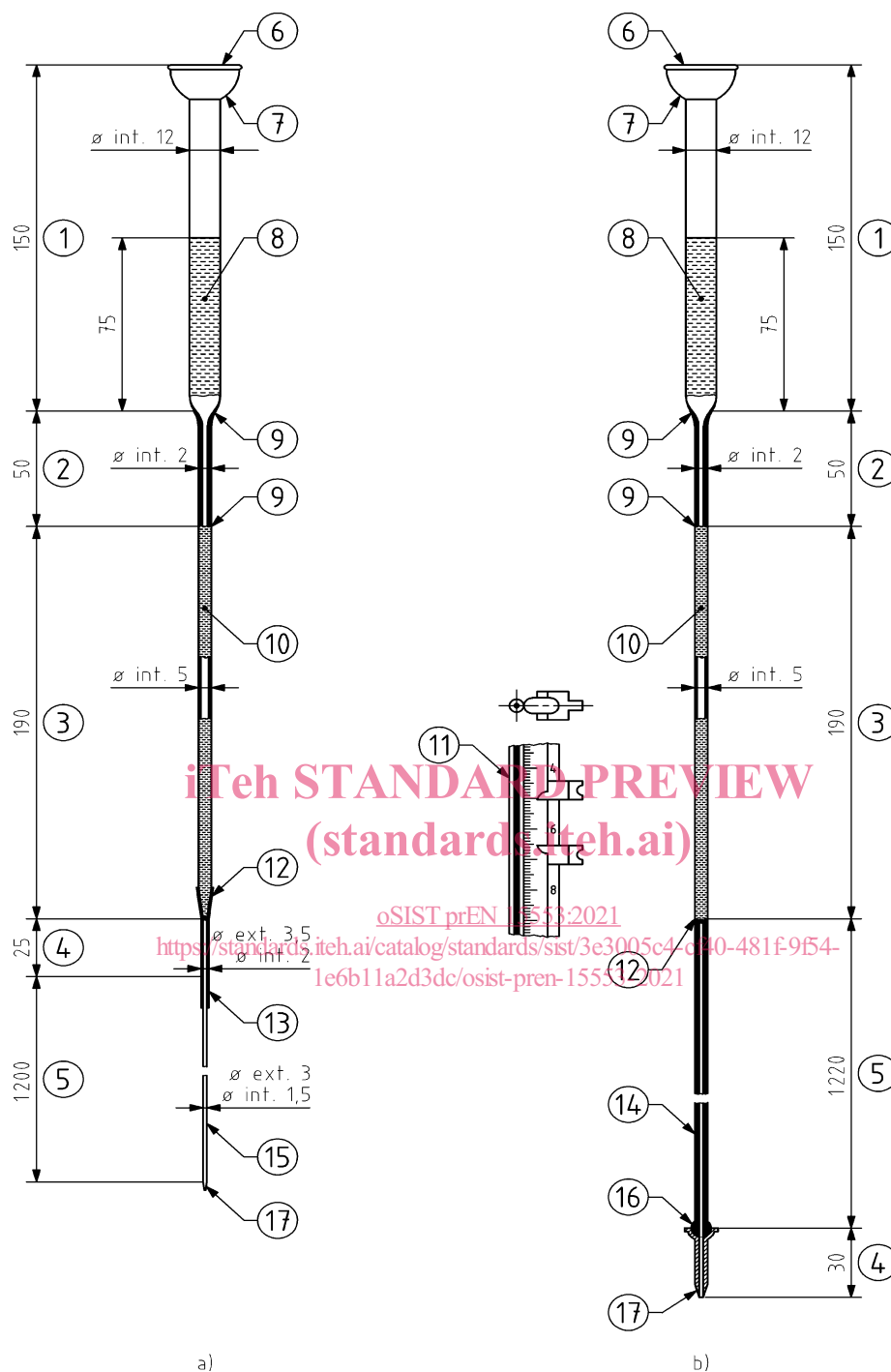
NOTE A convenient arrangement consists of one or two units 915 mm or 1 220 mm in length mounted vertically alongside the apparatus adjusted to give the best fluorescence.

## 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 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.



**Key**

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

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

## 7 Sampling and sample handling

7.1 Samples shall be taken as described in EN ISO 3170 or EN ISO 3171, whereas requirements of national standards or regulations for the sampling of unleaded petrol should be taken into account.

7.2 Store the sample in the dark at a temperature of 2 °C to 4 °C 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  $75 \text{ mm} \pm 5 \text{ mm}$  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.<sup>1</sup>

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## 9 Procedure

9.1 Bring the sample and a hypodermic syringe (6.5) to a temperature of 2 °C to 4 °C. Draw a  $0,75 \text{ ml} \pm 0,05 \text{ ml}$  test portion into the syringe and inject it approximately 30 mm below the surface of the gel in the charger section.

9.2 Fill the charger section to the spherical joint with propan-2-ol (5.3).

NOTE 1 For samples containing substantial amounts of material boiling above 205 °C, the use of 3-methylbutan-1-ol (5.4) instead of propan-2-ol can improve elution.

Connect the column to the gas manifold (6.4) and supply  $14 \text{ kPa} \pm 2 \text{ kPa}$  gauge gas pressure for  $2,5 \text{ min} \pm 0,5 \text{ min}$  to move the liquid front down the column. Increase the pressure to  $34 \text{ kPa} \pm 2 \text{ kPa}$  gauge for another  $2,5 \text{ min} \pm 0,5 \text{ min}$  and then adjust the pressure required to give a column transit time of about 1 h.

NOTE 2 Usually 28 kPa to 69 kPa gauge gas pressure is needed for gasoline type material and 69 kPa to 103 kPa gauge gas pressure for kerosine and aviation turbine fuel. The pressure required will depend on the tightness of packing of the gel and the molecular mass of the sample.

A transit time of 1 h is optimum; however, high-molecular mass samples may require longer transit times.