INTERNATIONAL STANDARD

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Liquid petroleum products — Determination of hydrocarbon types — Fluorescent indicator adsorption method

Produits pétroliers liquides — Détermination des groupes d'hydrocarbures — Méthode par adsorption en présence d'indicateurs fluorescents

ISO 3837:1993

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ISO 3837:1993(E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 3837 was prepared by Technical Committee ISO/TC 28, Petroleum products and lubricants.

Annex A of this International Standard is for information only.

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Introduction

The determination of the total volume percent [% (V/V)] of saturates, olefins and aromatics in petroleum fractions is important in characterizing the quality of petroleum fractions as gasoline blending components and as feeds to catalytic reforming processes, and in characterizing petroleum fractions and products from catalytic reforming and from thermal and catalytic cracking as blending components for motor and aviation fuels. This information is also important as a measure of the quality of aviation turbine fuels.

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Liquid petroleum products — Determination of hydrocarbon types — Fluorescent indicator adsorption method

WARNING — This 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 consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International 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) olefins, and 1 % (V/V) to 95 % (V/V) saturated hydrocarbons in petroleum fractions that distill below 315 °C.

Restrictions inherent in the method and the determination of precision may limit its application as noted.

NOTES

- 1 This method may apply to concentrations outside these ranges, but the precision has not been determined.
- 2 This test method is intended 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.
- 3 The applicability of this test method to products derived from fossil fuels other than petroleum, such as coal, shale or tar sands, has not been determined and the precision statement does not apply to such products.
- 4 The precision of this test method has not been determined with oxygenated fuels and thus does not apply to automotive gasolines containing lead anti-knock mixtures.
- 5 The oxygenated blending components methanol, ethanol, methyl tert-butyl ether, tert-amyl methyl ether and ethyl tert-butyl ether do not interface 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 al-

cohol desorbent. Other oxygenated compounds must be individually verified. When samples containing oxygenated blending components are analyzed, 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.

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

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 756-1:1981, Propan-2-ol for industrial use — Methods of test — Part 1: General.

ISO 3171:1988, Petroleum liquids — Automatic pipeline sampling.

ASTM D 3663-84, Test method for surface area of catalysts.

ASTM D 4815-89, Test method for analysis of C_1 to C_4 alcohols and MTBE in gasoline by gas chromotography.

3 Definitions

For the purposes of this International Standard, the following definitions apply.

- **3.1 saturates:** Volume percent [% (*V/V*)] of alkanes plus cycloalkanes.
- **3.2 olefins:** Volume percent [% (*V/V*)] of alkenes plus cycloalkenes plus some alkadienes.
- **3.3 aromatics:** Volume percent [% (*V/V*)] of condensed monocyclic and polycyclic aromatic hydrocarbons plus aromatic olefinic hydrocarbons, some dienes, compounds containing sulfur and nitrogen, or higher-boiling oxygenated compounds (excluding those listed in Note 5).

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 [% (V/V)] of each hydrocarbon type is calculated from the length of each zone in the column.

NOTE 7 Ferrors leading to high saturate values and low aromatic and low olefin values can result if the sample contains C_3 or lighter hydrocarbons, or more than 5 % C_4 hydrocarbons, or more than 10 % C_4 and C_5 hydrocarbons. Such samples should be depentanized as specified in ANSI/ASTM D2001 (see annex A).

5 Apparatus

5.1 Adsorption columns, either with precision bore tubing, as shown on the right in figure 1, made of glass and consisting of a charger section with a capillary neck, a separator section, and an analyzer section; or with standard wall tubing, as shown on the left in figure 1.

The inside diameter of the analyzer section for the precision bore tubing shall be 1,60 mm to 1,65 mm and an approximately 100 mm thread of mercury shall not vary by more than 0,3 mm in any part of the analyzer section. In glass-sealing the various sections to each other, long-taper connections shall be made instead of shouldered connections. Support the silicated 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 have a 2 mm inside diameter. Clamp the ball and socket together and 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.

For convenience, adsorption columns with standard wall tubing, as shown on the left in figure 1, may be used. When using standard wall tubing for the analyzer section, it is necessary to select tubing of uniform bore and to provide a leakproof connection between the separator and the analyzer sections. Calibrations of standard wall tubing would be impractical; however, any variations of 0,5 mm or greater, as measured by ordinary calipers, in the outside diameter along the tube may be taken as an indication of irregularities in the inside diameter and such tubing should not be used. Draw out one end of the tubing selected for the analyzer section to a fine capillary to retain the gel. Connect the other end of the analyzer section to the separator section with a 30 mm length of polyvinyl tubing, making certain that the two glass sections touch. To ensure a leakproof glass-topolyvinyl seal with the analyzer section, it is necessary to heat the upper end of the analyzer section until it is just hot enough to melt the polyvinyl, then insert the upper end of the analyzer section into the polyvinyl sleeve. Alternatively, this seal can be made by securing the polyvinyl sleeve to the analyzer section by wrapping it tightly with soft wire.

5.2 Zone-measuring-device.

The zones may be marked with glass-writing pencil and the distances measured with a metre rule, with the analyzer section lying horizontally. Alternatively, the metre rule may be fastened adjacent to the column. In this case, it is convenient to have each rule fitted with four movable metal index clips (figure 1) for marking zone boundaries and measuring the length of each zone.

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

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

- **5.4 Electric vibrator**, for vibrating the individual columns or for vibrating the frame supporting multiple columns.
- **5.5 Hypodermic syringe**, of capacity 1 ml, graduated to 0,01 ml or 0,02 ml, with needle 102 mm in length, with an inside diameter of 0,7 mm to 1,2 mm.

Needles of No. 18, 20 or 22 gauge are satisfactory.