

British Standard Methods of test for

Petroleum and its products

Part 156. Hydrocarbon types in liquid petroleum products by fluorescent indicator absorption

(Identical with IP 156/84)

Méthodes d'essai du pétrole et des produits pétroliers

Partie 156. Types de hydrocarbures dans les produits pétroliers liquides
par absorption de l'indicateur fluorescent

Verfahren zur Prüfung von Erdöl und Erdölprodukten

Teil 156. Schwefelkohlenstoffarten in flüssigen Erdölprodukten
durch Fluoreszenz-Indikator-Absorption

WITHDRAWN

Foreword

BS 2000 comprises a series of tests for petroleum and its products that have been published previously by the Institute of Petroleum (IP) and have been accorded the status of British Standards. Each method should be read in conjunction with BS 2000 : Part 0 'General introduction', which gives details of the BSI/IP agreement for publication of the series, lists more than 60 methods published in this way in 1982 and 1983, and provides general information on safety precautions, sampling and other matters. It is envisaged that Part 0 will be revised from time to time to take account of the publication of additional methods.

The numbering of the other Parts follows that of the corresponding methods published in 'IP Standards for petroleum and its products, Part 1 Methods for analysis and testing'. BS 2000 : Part 156 is thus identical with IP 156/84.

The previous edition of this Part* was identical with IP 156/70 (78) and previously this IP method was published in A5 format as BS 4712, which was withdrawn in 1983. In the present revision the significant changes include the following.

- (a) The scope clause has been revised so as to state the ranges over which the method applies and notes have been added setting out limitations on the fields of application.
- (b) A clause has been added setting out the significance and use of the method.
- (c) The description of the adsorption columns in 4.1 has been revised and the note relating to the use of standard wall tubing has been included in the body of the standard unchanged.
- (d) The instructions for reporting the results have been reworded so that they are the same for both precision bore columns and standard wall columns.
- (e) Revised precision data have been introduced as the result of a recent test programme.

It is intended to issue further revisions when necessary in line with any changes that may be made in subsequent editions of this IP method.

At the time of publication of this British Standard, no corresponding international standard exists, but a draft proposal for the method is under consideration in Technical Committee 28, Petroleum products and lubricants, of the International Organization for Standardization (ISO).

Caution. Attention is drawn to the safety precautions mentioned in Part 0 of this British Standard.

NOTE. The titles of the publications referred to in this Part of this British Standard are listed on the back cover.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

*Published in 1983 and now withdrawn.

Method (identical with IP 156/84¹)**1. SCOPE**

1.1. This method covers the determination of hydrocarbon types over the concentration ranges of 5 to 95 volume per cent aromatics, 0.3 to 55 volume per cent olefins, and 1 to 95 volume per cent saturates in petroleum fractions that distil below 600°F (315°C). This method may apply to concentrations outside these ranges but the precision has not been determined.

NOTE 1: A more accurate procedure for chromatographically determining the aromatic content of olefin-free gasoline or gasoline fractions appears in ASTM Method D936.

NOTE 2: This method is intended for use with full boiling range products. Cooperative data have established that the precision statement does not apply to narrow boiling point fractions near the 600°F (315°C) limit. Such samples are not eluted properly, and results are erratic.

NOTE 3: The applicability of this 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 may or may not apply to such products.

NOTE 4: The precision statement for this method has been determined with unleaded fuels that do not contain oxygenates. It may or may not apply to automotive gasolines containing lead anti-knock mixtures and/or oxygenates.

NOTE 5: For the determination of olefins below 0.3 volume per cent, other methods are available such as IP 299-ASTM D2710, Bromine Index of Petroleum Hydrocarbons by Electrometric Titration.

NOTE to note 1. There is at present no IP or BS equivalent to ASTM D936 'Test for aromatic hydrocarbons in olefin-free gasolines by silica gel adsorption'.

NOTE to note 5. The method in BS 5089 is identical with ISO 3839 and technically equivalent to IP 299.

2. SUMMARY OF METHOD

2.1. Approx 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 on the gel, alcohol is added to desorb the sample and force it down the column. The hydrocarbons are separated according to their adsorption affinities into aromatics (including some diolefins and aromatics with olefinic side chains plus any sulphur, nitrogen, and oxygen compounds), olefins, and saturates. The fluorescent dyes are also separated selectively, with the hydrocarbon types, and make 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.

3. SIGNIFICANCE AND USE

3.1. This method provides information on the total volume per cent of saturates, olefins, and aromatics in petroleum fractions. This information is important in characterizing the quality of petroleum fractions as gasoline blending components and as feeds to catalytic reforming processes. This information is also important 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 fuels, such as specified in ASTM Specification D1655.

4. APPARATUS

4.1. *Adsorption Columns* – with precision bore ('true bore' IP designation) tubing as shown on the right in Fig. 1, made of glass and consisting of a charger section with a capillary neck, separator section, and an analyser section; or with standard wall tubing, as shown on the left in Fig. 1.

4.1.1. The inner diameter of the analyser section for the precision bore tubing shall be 1.60 to 1.65 mm and an approx 100-mm thread of mercury shall not vary by more than 0.3 mm in any part of the analyser section. In glass-sealing the various sections to each other, long-taper connexions shall be made instead of shouldered connexions. Support the silica gel by means of a 6-mm diameter portion of phosphor-bronze or equivalent 200-mesh (aperture size 75 µm) wire gauze (BS 410:1976) located between the ball and socket of the 12/2 spherical joint and covering the analyser outlet. The column tip attached to the 12/2 socket

NOTE to 4.1. A spherical joint complying with BS 2761 is suitable.

¹ Under the standardization procedure of the Society, this method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants.

In the IP, this method is under the jurisdiction of the Standardization Committee.

² *Annual Book of ASTM Standards*, Part 23.

shall have a 2-mm internal 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 analyser section during the packing and subsequent use of the column.

4.1.2. For convenience, adsorption columns with standard wall tubing, as shown on the left in Fig. 1 may be used. In case of dispute, however, precision-bore tubing is required. When using standard wall tubing for the analyser section, it is necessary to select tubing of uniform bore and to provide a leak-proof connexion between the separator and the analyser 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 inner diameter and such tubing should not be used. Draw out one end of the tubing selected for the analyser section to a fine capillary to retain the gel. Connect the other end of the analyser section to the separator section with a 3-cm length of vinyl tubing, making certain that the two glass sections touch. To ensure a leak-proof glass-to-vinyl seal with the analyser section, it is necessary to heat the upper end of the analyser section until it is just hot enough to melt the vinyl, then insert the upper end of the analyser section into the vinyl sleeve. Alternatively, this seal can be made by securing the vinyl sleeve to the analyser section by wrapping it tightly with soft wire.

4.2. *Zone-Measuring Device* – the zones may be marked with a glass-writing pencil and the distances measured with a meter rule, with the analyser section lying horizontally. Alternatively, the meter 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 (Fig. 1) for marking zone boundaries and measuring the length of each zone.

4.3. *Ultraviolet Light Source* – with radiation predominantly at 365 nm is required. A convenient arrangement consists of one or two 915 mm (36 in) or 1220 mm (48 in) units mounted vertically along the apparatus. Adjust to give the best fluorescence.

4.4. *Electric Vibrator* – for vibrating individual columns or the frame supporting multiple columns.

4.5. *Hypodermic Syringe* – 1 ml, graduated to 0.01 or 0.02 ml, with needle 102 mm (4 in) in length. Needles of No. 18, 20, or 22 gauge, 0.7, 0.9 or 1.2 mm external dia, are convenient to use.

NOTE to 4.5. A syringe and needles, complying with BS 1263 and BS 3522 respectively, are suitable.

5. MATERIALS

5.1. *Silica Gel* – 100 to 200 mesh (150 to 75 μm particle size),³ manufactured to ensure minimum olefin polymerization and to conform to the sieve test requirements shown in Table 1. It is necessary to screen the gel to make certain that the gel size specification is met and the gel is homogenous throughout the container. Before use, dry the gel in a shallow vessel at 177°C (350°F) for 3 h. Transfer the dried gel to an airtight container while still hot, and protect it thereafter from atmospheric moisture.

TABLE 1. Sieve acceptance tests of silica gel

Sieve size ^a		Particles passing through sieve, wt%
Sieve No. ^a	μm	
80	180	100
100	150	95 min
200	75	15 max

^a Detailed requirements for these sieves are given in ASTM Specifications E11 or Sieves for Testing Purposes,⁴ and BS 410:1976.

³ Available from W. R. Grace and Co., Davison Chemical Div., Baltimore, Md. 21203 by specifying Code 923, or from Joseph Crosfield and Sons Ltd., Bank Quay, Warrington WA5 1AB by specifying Code 5G/0(100–200).

A list of suppliers in the U.K. can be obtained from the Institute of Petroleum. The precision statement does not apply to the second of these two sources.

The precision for this method is currently under review.

⁴ *Annual Book of ASTM Standards*, Parts 14, 18, 26, 30 and 41.

NOTE to footnote 3. A list of suppliers of suitable silica gel is available also on application, enclosing a stamped addressed envelope, to Enquiry Section (London), British Standards Institution, 2 Park Street, London W1A 2BS.