BRITISH STANDARD

BS
2000-182:2006
IP 182:2006

Methods of test for petroleum and its products —

Part 182: Determination of inorganic acidity of petroleum products — Colour indicator titration method

(Identical with IP 182:2006)

ICS 75.080

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BSI
British Standards

National foreword

This British Standard reproduces verbatim IP 182:2006 and implements it as the UK national standard. It supersedes BS 2000-182:2005 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum testing and terminology, which has the responsibility to:

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— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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Further information is available from:
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Summary of pages

This document comprises a front cover, an inside front cover, pages 1 to 4, an inside back cover and a back cover.

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Determination of inorganic acidity of petroleum products — Colour indicator titration method

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

1 Scope

This standard specifies a method for determining the water soluble inorganic (strong) acid content of used and unused lubricating oils, fuel oils, and petrolatum.

NOTE 1 - Oils containing additives that can be extracted with water may give erroneous results.

NOTE 2 - The results may not be numerically identical with those obtained when other colour indicators or techniques are used.

2 Normative references

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Principle

The inorganic acids in the oil are extracted with boiling water in a specified apparatus. After separation of the aqueous and oil layers an extract of the aqueous layer is titrated with standardized alkali using bromophenyl blue indicator.

4 Reagents and materials

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

4.1 Potassium hydrogen phthalate

4.2 Potassium hydroxide solution 0,1 mol/l

Either prepare a solution in accordance with 4.2.1 or use a commercially available potassium hydroxide solution of equivalent concentration and purity.

4.2.1 Dissolve 6 g potassium hydroxide in 1 l in water and standardize using potassium hydrogen phthalate in accordance with 4.2.2. The reagent shall be protected against carbon dioxide absorption and restandardized frequently enough to detect concentration changes of 0,0005 mol/l.

4.2.2 Dry a quantity of potassium hydrogen phthalate (4.1) in an oven at approximately 120 °C for approximately 2 h. Place in a desiccator and allow to cool. Weigh between 0,1 g to 0,2 g to the nearest 0,1 mg into a 250 ml flask and record this mass. Add approximately 50 ml of carbon dioxide free water and swirl to dissolve. Add 2 drops of bromophenol blue solution (4.3) and titrate to neutral end point with the potassium hydroxide solution (4.2). Carry out a blank determination using the same volume of carbon dioxide free water. Calculate the concentration C, in moles per litre, of the potassium hydroxide solution from the equation:

$$C = \frac{1\,000\,m}{204{,}23\,(V_1 - V_0)}$$

where

m is the mass of potassium hydrogen phthalate;

V_1 is the volume of potassium hydroxide solution for the titration;

V_0 is the volume of potassium hydroxide used for the blank.

4.3 Bromophenol Blue 0,04% solution

Dissolve 0,4 g bromophenol blue in 200 ml 95% ethanol (4.4). Add 6 ml 0,1 mol/l potassium hydroxide solution (4.2) and make up to 1 l with water.

4.4 Ethanol, approximately 95% *V/V*

4.5 Toluene, general purpose reagent grade.

4.6 Water, carbon dioxide free, see note 3.

NOTE 3 - A suitable way of preparing carbon dioxide free water is to place approximately 100 ml of water (conforming to ISO 3696 Grade 3) in a 250 ml conical flask (5.3), heat to boiling on either a hot plate or gas burner and boil for 2 min to 3 min. Remove the flask and its contents from the heat and insert a guard tube (5.7) filled with soda lime (4.7) and cool to ambient temperature before use.

4.7 Soda lime, for the guard tube.

5 Apparatus

5.1 Extraction apparatus, made glass, conforming to the dimensions given in Figure 1, and consisting of the following component parts:

5.1.1 Boiling flask, capacity approximately 500 ml.

5.1.2 Hopkins reflux condenser, having a vapour outlet connected by a rubber tube to an outside vent or to a suction hood.

5.1.3 Thistle tube, capacity approximately 70 ml, with a line indicating the approximate 50 ml level.

5.1.4 Heating tube, containing a chimney for increasing convection in the liquid.

5.1.5 Heating coil, 250 W, consisting of a suitable gauge of nichrome wire.

5.1.6 Rheostat, of suitable resistance and capacity, for regulating the heater.

5.2 Measuring cylinder, 100 ml capacity.

5.3 Conical flask x 3, glass stoppered approximately 250 ml capacity.

5.4 Filter paper, course filtration grade, approximately 125 mm diameter.

5.5 Filter funnel

5.6 Burette, 10 ml capacity

5.7 Glass guard tube, with ground glass joint to fit the conical flask (5.3).

6 Procedure

6.1 Using the measuring cylinder (5.2) place 100 ml distilled water into the extraction apparatus switch on and bring to the boil. Switch off the heater.

6.2 Weigh 25g ± 0,1 g of the test material into a conical flask (5.3) and add approximately 75 ml toluene (4.5) and swill until dissolved.

6.3 Add the toluene solution to the water in the extraction apparatus. Switch on and bring to the boil. Using the rheostat adjust the heater such that the mixture boils vigorously. After 15 min turn off the heater and allow the mixture to cool and to separate.

6.4 Place the filter funnel (5.5) fitted with a filter paper (5.4) into a 250 ml flask (5.3) and locate beneath the stopcock of the extraction apparatus.

6.5 Undo the stopcock and run off the separated aqueous layer through the filter paper into the 250 ml flask.

6.6 Using the measuring cylinder (5.2) measure 50 ml of the filtrate into a clean dry 250 ml flask.

NOTE 4 - If separation of the oil and water mixture is poor and the stipulated volume of extract is not obtainable a smaller volume may be used for the titration, though results may be less precise.

6.7 Add 2 drops of bromophenol blue indicator (4.3) and using the burette (5.6) titrate with standard potassium hydroxide solution (4.2) to a neutral end point. Record the volume of potassium hydroxide solution used.

7 Calculation

7.1 Calculate the inorganic acidity of the sample using the following equation:

$$\text{Inorganic acidity (mg KOH/g)} = 4{,}5\ M\ T$$

where

M is the molarity of potassium hydroxide solution;

T is the volume of potassium hydroxide solution in ml required to neutralize 50 ml of filtered extract.

NOTE 5 - Where less than 50 ml of extract is titrated, see Note 4, the result must be multiplied by 50/V, where V is the volume of the extract that is titrated in ml.

8 Expression of results

Report the result to the nearest 0,01 mg KOH/g .

9 Precision

The precision of this method has not been determined.

DIMENSIONS IN MILLIMETRES

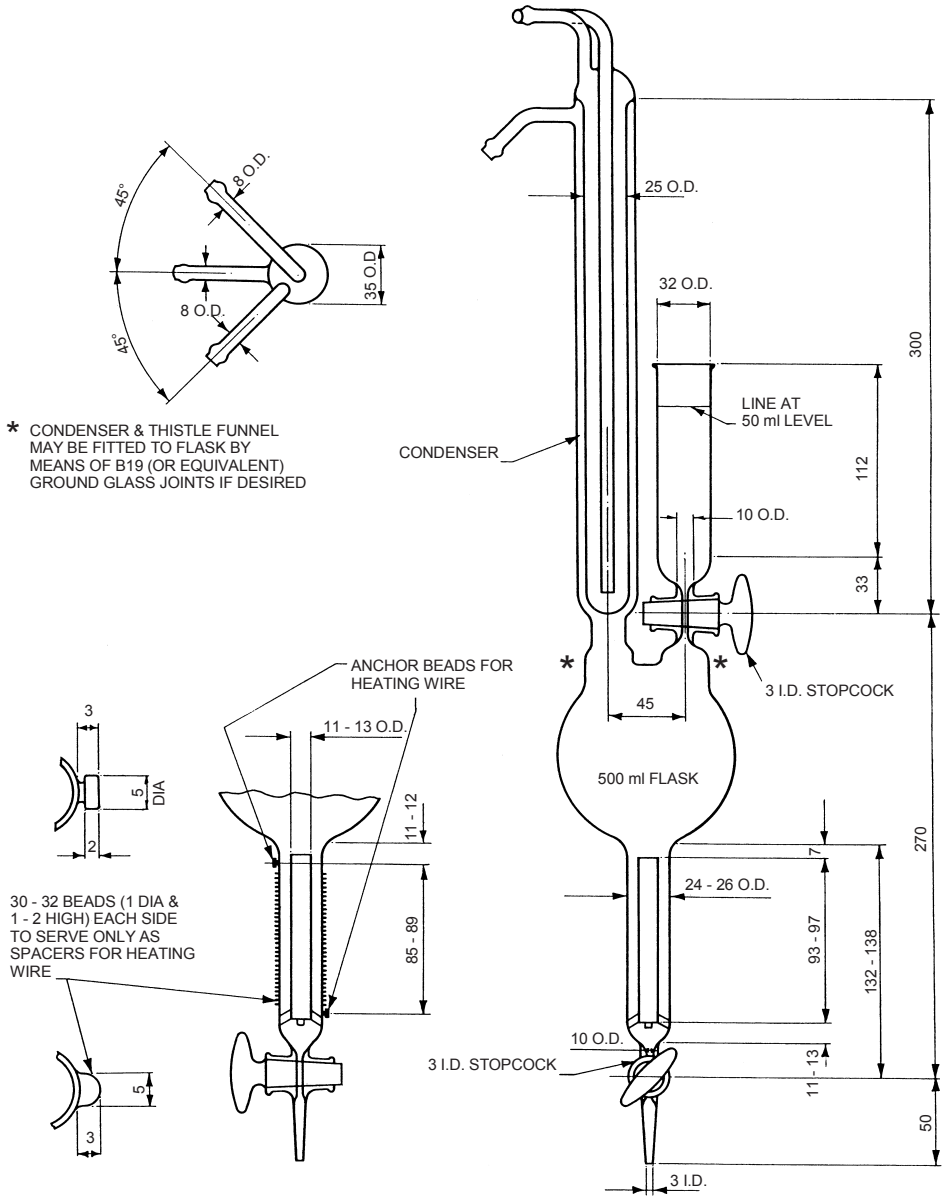

Figure 1. Extraction apparatus

10 Test report

The test report shall contain at least the following information:

a) a reference to this standard;

b) the type and complete identification of the product tested;

c) the result of the test (see clause 8);

d) any deviation, by agreement or otherwise, from the standard procedures specified;

e) the date of the test.

blank

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