

INTERNATIONAL STANDARD

ISO
6293-1

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Petroleum products — Determination of saponification number —

Part 1: Colour-indicator titration method

*Produits pétroliers — Détermination de l'indice de saponification —
Partie 1: Méthode par titrage avec indicateur coloré*



Reference number
ISO 6293-1:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 6293-1 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This first edition cancels and partially replaces the first edition of ISO 6293:1983, which has been technically revised.

ISO 6293 consists of the following parts, under the general title *Petroleum products — Determination of saponification number*:

- *Part 1: Colour-indicator titration method*
- *Part 2: Potentiometric titration method*

Petroleum products — Determination of saponification number —

Part 1:

Colour-indicator titration method

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

1 Scope

This part of ISO 6293 specifies a method for the determination, by colour-indicator titration, of the amount of constituents in petroleum products that will saponify under the conditions of the test.

The method is applicable to materials having saponification numbers in the range 2 mg/g KOH to 200 mg/g KOH.

Compounds of sulfur, phosphorus, halogens and some other compounds interfere by reacting with the alkali and acids under the test conditions.

NOTES

1 The results on used crankcase and turbine oils, and on oils containing the interfering compounds above as additive constituents, should be interpreted with care, bearing in mind the possible higher values obtainable due to these additional reactants.

2 The interfering compounds above are contained in extraneous materials including certain organic acids and most nonalkali soaps. The odour of hydrogen sulfide near the end of the back-titration step is an indication of the presence of certain reactive sulfur compounds, but other reactive sulfur compounds, as well as those of chlorine, phosphorus and other interfering materials, give no simple indication during the test. A gravimetric determination of fatty acid content is an alternative procedure for the estimation of such compounds.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 6293. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 6293 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 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 6353-2:1983, *Reagents for chemical analysis — Part 2: Specifications — First series.*

ISO 6353-3:1987, *Reagents for chemical analysis — Part 3: Specifications — Second series.*

3 Definitions

For the purposes of this part of ISO 6293, the following definitions apply.

3.1 saponify: To hydrolyze a fat with alkali to form an alcohol and the salt of a fatty acid.

3.2 saponification number: The number of milligrams of potassium hydroxide consumed by 1 g of a sample under the specified conditions of this test.

4 Principle

A test portion of petroleum product of known mass, dissolved in butan-2-one, is reacted by heating with a known amount of alcoholic potassium hydroxide solution. The excess alkali is titrated with standard volumetric hydrochloric acid solution, using phenolphthalein as indicator, and the saponification number calculated.

5 Reagents and materials

During the analysis, use only reagents specified in ISO 6353-2 and ISO 6353-3, if listed there, or if not, of recognized analytical grade. Use only distilled water or water to grade 3 of ISO 3696.

5.1 Ethanol, 95 % (V/V) ethanol, or a solution comprising 90 % (V/V) of 95 % (V/V) ethanol and 10 % (V/V) of methanol, or absolute ethanol.

NOTE — Studies have shown that 99 % (V/V) propan-2-ol can be substituted for the ethanol in routine analysis with equivalent sensitivity and precision, but should not be used in referee tests.

5.2 Potassium hydroxide, $c(\text{KOH}) = 0,5 \text{ mol/l}$, standard volumetric alcoholic solution, prepared in accordance with 5.2.1 or obtained commercially, and standardized in accordance with 5.2.2.

5.2.1 Preparation

Add approximately 29 g of solid KOH to 1 l of ethanol (5.1) in a 2-litre conical flask. Boil gently with stirring for 10 min to 15 min. Add at least 2 g of barium hydroxide $[\text{Ba}(\text{OH})_2]$ and boil gently for a further 5 min to 10 min.

Allow to cool and stand at room temperature for at least 24 h in the dark. Transfer to the storage container by filtration or pressure displacement under inert gas conditions (carbon dioxide-free).

Store the solution in a chemically resistant dispensing bottle out of contact with cork, rubber or saponifiable stopcock lubricant, and protected by a guard tube containing soda lime or nonfibrous soda silicate absorbent (Ascarite, Carbosarb or Indecarb)¹⁾. Glass bottles are not recommended for storage.

1) Ascarite, Carbosarb and Indecarb are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 6293 and does not constitute an endorsement by ISO of these products.