
**Petroleum products — Determination
of saponification number —**

Part 2:
Potentiometric titration method

Produits pétroliers — Détermination de l'indice de saponification —

Partie 2: Méthode par titrage potentiométrique



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 6293-2 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This first edition, together with ISO 6293-1, cancels and replaces 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*

Annex A forms an integral part of this part of ISO 6293.

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Petroleum products — Determination of saponification number —

Part 2:

Potentiometric titration method

WARNING — The use of this part of ISO 6293 may involve hazardous materials, operations and equipment. This part of ISO 6293 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 6293 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 potentiometric titration, of the amount of constituents in petroleum products that will saponify under the conditions of the test. ISO 6293-1 specifies a determination by colour indicator titration.

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

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

NOTES

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

2 These extraneous materials include certain organic acids and most non-alkali 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 6293-1:1996, *Petroleum products — Determination of saponification number — Part 1: Colour-indicator titration method*.

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 that is consumed by 1 g of a sample under the specified conditions of this test

4 Principle

A test portion of known mass, dissolved in butan-2-one, is heated with a known amount of alcoholic potassium hydroxide solution. The excess alkali is potentiometrically titrated with standard volumetric hydrochloric acid solution and the saponification number is 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 according to grade 3 of ISO 3696.

5.1 Ethanol, 95 % (V/V) ethanol, or 9 parts of 95 % (V/V) ethanol to which has been added 1 part of methanol or absolute alcohol.

NOTES

1 For the purposes of this part of ISO 6293, the expressions “% (m/m)” and “% (V/V)” are used to represent the mass and volume fractions of a material respectively.

2 For routine analysis, 99 % (V/V) propan-2-ol can be substituted for ethanol without compromising the sensitivity or precision of the method. Ethanol should always be used for referee tests.

5.2 Potassium hydroxide, $c(\text{KOH}) = 0,5 \text{ mol/l}$, standard volumetric alcoholic solution.

Prepare in accordance with 5.2.1 or use a commercially available solution. Standardize in accordance with 5.2.2.

5.2.1 Preparation

Add approximately 29 g of solid KOH to 1 litre of ethanol (5.1) in a 2 litre conical flask. Boil gently while 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.

CAUTION — Barium hydroxide is strongly alkaline and toxic if ingested. Use protective clothing to avoid severe irritation caused by contact with the skin.

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 non-fibrous soda silicate absorbent. Glass bottles are not recommended for storage.