

INTERNATIONAL STANDARD

**ISO
6618**

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Petroleum products and lubricants — Determination of acid or base number — Colour-indicator titration method

*Produits pétroliers et lubrifiants — Détermination de l'indice d'acide ou de
l'indice de base — Méthode par titrage en présence d'un indicateur coloré*



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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 6618 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 6618:1987), which has been technically revised.

Annexes A and B form an integral part of this International Standard.

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Petroleum products and lubricants — Determination of acid or base number — Colour-indicator titration method

WARNING — The use of this International 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 International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a colour-indicator titration method for the determination of acidic or basic constituents in petroleum products and lubricants soluble in mixtures of toluene and propan-2-ol. It is applicable for the determination of acids or bases whose dissociation constants in water are greater than 10^{-9} ; extremely weak acids or bases whose dissociation constants are less than 10^{-9} do not interfere. Salts react if their hydrolysis constants are greater than 10^{-9} .

NOTE 1 In new and used oils, the constituents considered to have acidic characteristics include organic and inorganic acids, esters, phenolic compounds, lactones, resins, salts of heavy metals, and additives such as inhibitors and detergents. Similarly, constituents considered to have basic properties include organic and inorganic bases, amino compounds, salts of weak acids (soaps), basic salts of polyacidic bases, salts of heavy metals, and additives such as inhibitors and detergents.

The method is not suitable for measuring the basic constituents of many basic additive-type lubricating oils, for which ISO 3771 can be used.

This International Standard indicates relative changes that occur in an oil during use under oxidizing conditions. Although the titration is made under definite equilibrium conditions, the method does not measure an absolute acidic or basic property that can be used to predict performance of an oil under service conditions.

NOTES

2 No general relationship between bearing corrosion and acid or base number is known.

3 Oils, such as many cutting oils, rustproofing oils, and similar compounded oils, or excessively dark-coloured oils, that cannot be analysed by this method owing to obscurity of the colour-indicator end-point, can be analysed in accordance with ISO 6619. The acid or base numbers obtained by this colour-indicator method may or may not be numerically the same as those obtained by ISO 6619, but they are generally of the same order of magnitude.

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 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 3771:1994, *Petroleum products — Determination of base number — Perchloric acid potentiometric titration method*.

ISO 6619:1988, *Petroleum products and lubricants — Neutralization number — Potentiometric titration method*.

NOTES

1 ISO 3771 employs the use of chlorobenzene as a solvent. Alternatives to this solvent are being sought.

2 ISO 6619 is under review, and the scope will be restricted to acid number.

3 Definitions

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

3.1 acid number: Quantity of base, expressed in milligrams of potassium hydroxide (KOH) per gram of sample, that is required to titrate the acid constituents present in 1 g of sample when titrated under prescribed conditions.

3.2 strong acid number: Quantity of base, expressed in milligrams of potassium hydroxide (KOH) per gram of sample, that is required to titrate the strong acid constituents present in 1 g of sample when titrated under prescribed conditions.

3.3 base number: Quantity of acid, expressed in terms of the equivalent number of milligrams of potassium hydroxide (KOH) per gram of sample, that is required to titrate the base constituents present in 1 g of sample when titrated under prescribed conditions.

4 Principle

To determine the acid number or base number, the test portion is dissolved in a mixture of toluene and propan-2-ol containing a small amount of water, and the resulting single-phase solution is titrated at room temperature with standard alcoholic base or alcoholic acid solution, respectively, to the end-point indicated by the colour change of added *p*-naphtholbenzein solution (orange in acid and green-brown in base).

To determine the strong acid number, a separate portion of the sample is extracted with boiling water and the aqueous extract is titrated with potassium hydroxide solution using methyl orange as indicator.

5 Reagents and materials

During the analysis, use only reagents of recognized analytical grade, and only water in accordance with Grade 3 of ISO 3696.

5.1 Propan-2-ol, anhydrous, maximum water content 0,9 % (V/V) (volume fraction 0,9 %).

5.2 Toluene.

5.3 Titration solvent.

Add 500 ml of toluene (5.2) and 5 ml of water to 495 ml of propan-2-ol (5.1).

5.4 Hydrochloric acid, $c(\text{HCl}) = 0,1 \text{ mol/l}$ standard volumetric alcoholic solution.

Prepare in accordance with 5.4.1 or use commercially available solution. Standardize in accordance with 5.4.2.

5.4.1 Preparation

Mix 9 ml of concentrated HCl [35,4 % (m/m) (mass fraction 35,4 %)] with 1 l of propan-2-ol (5.1).

5.4.2 Standardization

Standardize frequently enough to detect changes of 0,000 5 mol/l, preferably by electrometric titration of approximately 8 ml (accurately measured) of the 0,1 mol/l alcoholic KOH solution (5.5) diluted with 125 ml of carbon dioxide-free water.

5.5 Potassium hydroxide, $c(\text{KOH}) = 0,1$ mol/l standard volumetric alcoholic solution.

Prepare in accordance with 5.5.1 or use commercially available solution. Standardize in accordance with 5.5.2.

5.5.1 Preparation

Add 6 g of solid KOH to approximately 1 l of propan-2-ol (5.1) in a 2-litre conical flask. Boil the mixture gently for 10 min to 15 min, stirring to prevent solids from forming a cake on the bottom.

Add at least 2 g of barium hydroxide $[\text{Ba}(\text{OH})_2]$ and again boil gently for 5 min to 10 min.

Allow to cool to room temperature, and then to stand for several hours. Filter the supernatant liquid through a fine sintered-glass or porcelain filtering funnel; avoid unnecessary exposure to carbon dioxide during filtration.

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 absorbent¹⁾. Glass bottles are not recommended for storage.

5.5.2 Standardization

Standardize frequently enough to detect changes of 0,000 5 mol/l, preferably against 0,1 g to 0,15 g of pure potassium acid phthalate (5.9) weighed with an accuracy of 0,000 2 g and dissolved in approximately 100 ml of carbon dioxide-free water, using phenolphthalein (5.8) to detect the end-point.

NOTES

- 1 To simplify calculations, both the standard potassium hydroxide and hydrochloric acid solutions may be adjusted so that 1 ml is equivalent to 5 mg of KOH.
- 2 Because of the relatively large coefficient of cubic expansion of organic liquids such as propan-2-ol, the standard alcoholic solutions should be standardized at temperatures close to those employed in the titrations of sample, and close to 20 °C.

5.6 Methyl orange, indicator solution.

Dissolve 0,1 g of methyl orange in 100 ml of water.

5.7 *p*-naphtholbenzein indicator solution, meeting the specifications given in annexes A and B. Dissolve 1,0 g of *p*-naphtholbenzein in 100 ml of titration solvent (5.3).

NOTE — In a study in 1992, only one commercially available source of this indicator met the specification given in annex A²⁾. Careful adherence to this requirement is therefore recommended.

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

2) Solid *p*-naphtholbenzein meeting the specification given in annex A is available from Distillation Products Industries, Eastman Organic Chemicals Dept., Rochester 3, New York, USA. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.