
**Petroleum products — Determination of
acid number — Semi-micro colour-indicator
titration method**

*Produits pétroliers — Détermination de l'indice d'acide — Méthode de titrage
semi-micro par indicateur coloré*



Foreword

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International Standard ISO 7537 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 7537:1989), which has been technically revised.

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Petroleum products — Determination of acid number — Semi-micro 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 International Standard specifies a semi-micro colour-indicator method for the determination of acidic constituents in new or used petroleum products and lubricants soluble in mixtures of toluene and propan-2-ol, or capable of existing as suspensions in such mixtures, and where the suspended material is sufficiently dissociated that its acidic components can be titrated.

The method is especially intended for applications in which the amount of sample available to be analysed is too small to allow accurate analysis by ISO 6619 or ISO 6618. It is applicable to the determination of acids having dissociation constants in water larger than 10^{-9} . Extremely weak acids having dissociation constants smaller than 10^{-9} do not interfere. Salts titrate if their hydrolysis constants are larger than 10^{-9} .

NOTE 1 This method may be used to indicate relative changes in acid number 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 property that can be used to predict performance of an oil under service conditions. No general relationship between bearing corrosion and acid number is known.

NOTE 2 Since this test method requires substantially less sample than ISO 6618 or ISO 6619, it provides an advantageous means of monitoring an oxidation test by changes in acid number by:

- a) minimizing test sample depletion for acid number analyses and thus minimizing the disturbance of the test, and
- b) allowing additional acid number analyses to be made while maintaining the same test sample depletion, and thus providing additional data.

NOTE 3 Some oils, such as many cutting oils, rustproofing oils, and similar compounded oils, or excessively dark-coloured oils, may be more difficult to analyse by this method due to obscurity of the colour-indicator end-point. These oils can be analyzed by ISO 6619 provided sufficient sample is available. However, this situation is much less likely using this International Standard than using ISO 6618, since the sample is more dilute during the titration and the end-point colour change is more stable. The acid numbers obtained by this method may or may not be numerically the same as those obtained by ISO 6619, but they should be of the same order of magnitude.

NOTE 4 The results obtained using this method have been found to be numerically the same as those obtained using ISO 6618, within the precision of the two methods, for new or oxidized lubricants of the type primarily intended for hydraulic or steam turbine type service. The oxidized lubricants were obtained using ISO 4263.

This correlation is shown by the correlation coefficient $r = 0,989$ with slope $s = 1,017$ and intercept $y = +0,029$, calculated using the acid numbers obtained using both titration methods for the samples used for the precision statement (see clause 11).

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 listed 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 6618:1997, *Petroleum products and lubricants — Determination of acid and base number — Colour-indicator titration method*.

3 Definition

For the purposes of this International Standard, the following definition applies:

3.1 acid number: The quantity of potassium hydroxide (KOH), expressed in milligrams, required to titrate acids or salts present in one gram of sample to a specified end-point, when the dissociation or hydrolysis constants are larger than 10^{-9} .

4 Principle

The sample is dissolved in a toluene/propan-2-ol solvent containing a small amount of water, and the resulting single-phase solution is titrated at room temperature with standard alcoholic potassium hydroxide solution under a nitrogen atmosphere. The end-point is the stable green colour of the added *p*-naphtholbenzein indicator.

5 Reagents and materials

During the analysis, unless otherwise stated, use only reagents of recognised analytical grade. All references to water shall be understood to mean freshly distilled (carbon dioxide free) water conforming to grade 3 of ISO 3696.

NOTE 5 For the purposes of this International Standard, the term “% (V/V)” is used to represent the volume fraction of a material.

5.1 *p*-Naphtholbenzein indicator solution, conforming to the specifications given in annexes A and B of ISO 6618:1997. Prepare a solution containing 10 g of *p*-naphtholbenzein per litre of titration solvent.

NOTE 6 In a study in 1992, only one commercially available source of this indicator met the specification now given in annex A of ISO 6618:1997. Careful adherence to this requirement is therefore required.

5.2 Nitrogen, of minimum purity 99,5 %, dry and carbon dioxide free.

NOTE 7 In order to obtain repeatable results and a stable end-point colour change, it is especially important that the nitrogen purge gas be free of carbon dioxide. Prepurified grade nitrogen has been found to be satisfactory.

5.3 Phenolphthalein indicator solution.

5.3.1 Preparation

Dissolve 0,10 g of pure, solid phenolphthalein in 50 ml of water and 50 ml of 95 % (V/V) ethanol.