



SLOVENSKI STANDARD

SIST ISO 3771:2012

01-maj-2012

Nadomešča:
SIST ISO 3771:1996

Naftni proizvodi - Določevanje baznega števila - Metoda potenciometrične titracije s perklorovo kislino

Petroleum products - Determination of base number - Perchloric acid potentiometric titration method

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Produits pétroliers - Détermination de l'indice de base - Méthode par titrage potentiométrique à l'acide perchlorique **IST ISO 3771:2012**

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Ta slovenski standard je istoveten z: ISO 3771:2011

ICS:

75.080 Naftni proizvodi na splošno Petroleum products in general

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en

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INTERNATIONAL STANDARD

ISO
3771

Third edition
2011-09-15

Petroleum products — Determination of base number — Perchloric acid potentiometric titration method

*Produits pétroliers — Détermination de l'indice de base — Méthode par
titrage potentiométrique à l'acide perchlorique*

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3771 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This third edition cancels and replaces the second edition (ISO 3771:1994), which has been technically revised.

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Petroleum products — Determination of base number — Perchloric acid potentiometric 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 method for the determination of basic constituents in petroleum products by potentiometric titration with perchloric acid in glacial acetic acid.

The constituents that can be considered to have basic characteristics include organic and inorganic bases, amino compounds, salts of weak acids (e.g. soaps), basic salts of polyacid bases, and salts of heavy metals.

The ranges of base number values for which precision values for the method have been established are:

- unused oils: base numbers from 3 to 45;
- additive concentrates: base numbers from 5 to 45;
- used oils: base numbers from 3 to 30.

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2 Normative references

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The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

base number

w_{BN}

quantity of perchloric acid, expressed in terms of the equivalent number of milligrams of potassium hydroxide, required to titrate 1 g of sample dissolved in the specified solvent to a well-defined inflection point, as specified in this International Standard

4 Principle

The test sample is dissolved in an essentially anhydrous mixture of toluene, acetone and glacial acetic acid, and titrated with a standard volumetric solution of perchloric acid in glacial acetic acid using a potentiometric titrimeter. A combination pH-Ag/AgCl glass electrode (6.2) is used. The meter readings are plotted against the corresponding volumes of titrating solution, and the endpoint is taken as the last inflection in the resulting curve.

5 Reagents

During the analysis, use only reagents of recognized analytical grade, and water equivalent to Grade 3 of ISO 3696.

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5.1 **Acetic acid** (CH_3COOH), glacial.

5.2 **Acetic anhydride** [$(\text{CH}_3\text{CO})_2\text{O}$].

5.3 **Toluene**.

5.4 **Acetone**.

5.5 **Tris (hydroxymethyl)-aminomethane** (Tris) ($\text{C}_4\text{H}_{11}\text{NO}_3$).

5.6 **Potassium hydrogen phthalate** ($\text{KHC}_8\text{H}_4\text{O}_4$).

5.7 **Tetraethylammoniumbromide 0,4 mol**, electrolyte. Use an appropriate non-aqueous electrolyte in the electrode. Tetraethylammoniumbromide (TEABr in ethylene glycol, 0,4 mol/l) is an example of such an electrolyte. In general, follow the manufacturer's instructions for electrolyte use in the electrode. Prepare a solution (0,4 mol/l) of TEABr in ethylene glycol.

5.8 **Lithium chloride** (LiCl): electrode manufacturers recommend alternatively a saturated solution of LiCl in ethanol (96 % denatured). Dissolve 70 g LiCl in 500 ml ethanol at ambient temperature, stirring for several hours.

5.9 **Titration solvent**: add six volumes of toluene (5.3) to three volumes of acetic acid (5.1) to one volume of acetone (5.4).

5.10 **Perchloric acid** (HClO_4), 0,1 mol/l standard volumetric acetic solution.

5.10.1 A 0,1 mol/l acetic acid perchloric acid mixture may be obtained commercially. If necessary this may be prepared using concentrated perchloric acid (HClO_4), acetic acid and acetic anhydride. Perchloric acid is available in different concentrations, the amount of acetic anhydride required depends on the concentration. Three typical possibilities are found in Table 1. When preparing the mixture, initially dissolve the appropriate amount of perchloric acid in 500 ml of acetic acid, add the appropriate amount of acetic anhydride and dilute to 1 l with acetic acid.

Store this solution for at least 24 h before standardization.

WARNING — This perchloric acid solution is not harmful under the test conditions, but concentrated perchloric acid is a powerful oxidant when dry or heated. Take great care to avoid contact with organic matter under conditions that may result in subsequent drying or heating. Wash spills immediately and thoroughly with water.

NOTE Avoid adding excess acetic anhydride to prevent acetylation of any primary or secondary amines which may be present in the sample being tested.

Table 1 — Perchloric acid and acetic anhydride dilution volumes based on perchloric acid concentrations

Perchloric acid concentration % (m/m) ^a	Perchloric acid addition ml	Acetic anhydride ml
70 to 72	8,5	30
60 to 62	10,2	35
57	11,8	40

^a For the purposes of this International Standard, the term "% (m/m)" is used to represent the mass fraction of a material.

5.10.2 Standardization may be conducted using either tris (hydroxymethyl)-aminomethane (5.5) or potassium hydrogen phthalate (5.6), as described below. Commercially available certified solutions may be used in place of standardization.

a) Procedure A using tris (hydroxymethyl)-aminomethane (5.5)

Dry tris (hydroxymethyl)-aminomethane over silica gel in a desiccator (minimum of 24 h at room temperature). Measure approximately 0,05 g of tris(hydroxymethyl)-aminomethane to 0,1 mg accuracy and dissolve in 60 ml of acetic acid. Titrate the solution with perchloric acid (5.10) using the electrode system and methods given in 8.1 to 8.4 and 9.3 to 9.6. Detect the end point using the same method as used for base number determination (10.1). The titer shall be adjusted each time a new titration solution is prepared or minimum once a week.

b) Procedure B using potassium hydrogen phthalate (5.6)

Dry a quantity of potassium hydrogen phthalate (5.6) for 2 h in an oven at 120 °C and allow it to cool in a desiccator. Dissolve 0,1 g to 0,2 g of potassium hydrogen phthalate weighed to the nearest 0,1 mg and in 60 ml of acetic acid (5.1). Titrate with the perchloric acid solution (5.10.1) using the appropriate method (see Procedure A above). The titer shall be adjusted each time a new titration solution is prepared or minimum once a week.

5.10.3 Calculation of the concentration is carried out as described below.

a) Calculation procedure A using tris (hydroxymethyl)-aminomethane (5.5)

Calculate the concentration, $c_{0(\text{Tris})}$, expressed in moles per litre, of the perchloric acid solution from Equation (1):

$$c_{0(\text{Tris})} = \frac{m_{\text{Tris}} \times f_c}{121,14 \times V_{\text{pa}}} \quad (1)$$

where

m_{Tris}

is the mass of tris (hydroxymethyl)-aminomethane (5.5), expressed in milligrams;

f_c

is the correction factor calculated from the concentration given in the certificate divided by 100;

121,14

is the molar mass of tris (hydroxymethyl)-aminomethane (5.5), expressed in milligrams per millimole;

V_{pa}

is the volume of perchloric acid solution used, expressed in millilitres.

b) Calculation procedure B using potassium hydrogen phthalate (5.6)

Calculate the concentration, $c_{0(\text{KHP})}$, expressed in moles per litre, of the perchloric acid solution from Equation (2):

$$c_{0(\text{KHP})} = \frac{m_{\text{KHP}} \times f_c}{204,22 \times V_{\text{pa}}} \quad (2)$$

where