SLOVENSKI STANDARD
SIST EN ISO 14597:1999

01-november-1999

Naftni proizvodi ï Določ evanje vanadija in niklja ï Valovno disperzivna rentgenska fluorescenč na spektrometrija (ISO 14597:1997)

Petroleum products - Determination of vanadium and nickel content - Wavelength-dispersive X-ray fluorescence spectrometry (ISO 14597:1997)

Mineralölerzeugnisse - Bestimmung des Vanadium- und Nickelgehaltes - Wellenlängedispersive Röntgenfluoreszenz-Analyse (ISO 14597:1997)

Produits pétroliers - Dosage du vanadium et du nickel - Spectrométrie de fluorescence X dispersive en longueur d'onde (ISO 14597:1997)

Ta slovenski standard je istoveten z: **EN ISO 14597:1999**

ICS:

75.080	Naftni proizvodi na splošno	Petroleum products in general

SIST EN ISO 14597:1999 **en**

EUROPEAN STANDARD
NORME EUROPÉENNE
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EN ISO 14597

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ICS 75.080

Descriptors: see ISO document

English version

Petroleum products - Determination of vanadium and nickel content - Wavelength-dispersive X-ray fluorescence spectrometry (ISO 14597:1997)

Produits pétroliers - Dosage du vanadium et du nickel - Spectrométrie de fluorescence X dispersive en longueur d'onde (ISO 14597:1997)

Mineralölerzeugnisse - Bestimmung des Vanadium- und Nickelgehaltes - Wellenlängedispersive Röntgenfluoreszenz-Analyse (ISO 14597:1997)

This European Standard was approved by CEN on 20 December 1998.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN ISO 14597:1999 E

Foreword

The text of the International Standard from Technical Committee ISO/TC 28 "Petroleum products and lubricants" of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products", the secretariat of which is held by NNI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 1999, and conflicting national standards shall be withdrawn at the latest by July 1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 14597:1997 has been approved by CEN as a European Standard without any modification.

NOTE: Normative references to International Standards are listed in annex ZA (normative).

Annex ZA (normative)
Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

Publication	Year	Title	EN	Year
ISO 3170	1988	Petroleum liquids - Manual sampling	EN ISO 3170	1998

INTERNATIONAL STANDARD

ISO 14597

First edition
1997-07-15

Petroleum products — Determination of vanadium and nickel content — Wavelength-dispersive X-ray fluorescence spectrometry

Produits pétroliers — Dosage du vanadium et du nickel — Spectrométrie de fluorescence X dispersive en longueur d'onde

Reference number
ISO 14597:1997(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 14597 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants.*

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Petroleum products — Determination of vanadium and nickel content — Wavelength-dispersive X-ray fluorescence spectrometry

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 vanadium and nickel in liquid petroleum products. It may also be applied to semi-solid and solid petroleum products that are either liquefied by moderate heating or completely soluble in the specified organic solvent mixture. The method is applicable to products having vanadium contents in the range 5 mg/kg to 1 000 mg/kg, and nickel contents in the range 5 mg/kg to 100 mg/kg, although precision data have only been determined up to 100 mg/kg for vanadium and 60 mg/kg for nickel; higher contents may be determined by appropriate dilution.

Barium at concentrations above approximately 300 mg/kg interferes with the determination of vanadium, and iron at concentrations above approximately 500 mg/kg interferes with the determination of nickel. Other elements at concentrations above approximately 500 mg/kg may affect precision and accuracy due to spectral line overlap or absorption.

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 3170:1988, *Petroleum liquids — Manual sampling.*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling.*

3 Principle

The test portion and a manganese solution as internal standard are mixed in a given mass ratio and exposed, in a sample cell, to the primary radiation of an X-ray tube.

The count rates of excited metal and reference material are measured, and the ratio of these count rates calculated. The vanadium and nickel contents of the sample are determined from calibration curves prepared on the basis of calibration standards.

4 Reagents and materials

4.1 White oil (light paraffin oil), high purity grade, sulfur content 1 mg/kg maximum.

4.2 Xylene or **mixed xylenes,** analytical reagent grade.

4.3 Solvent mixture, 1 part by volume of white oil (4.1) mixed with 2 parts by volume of xylene (4.2).

4.4 Acetyl acetone, minimum purity 99 % (*m/m*).

NOTE — For the purposes of this International Standard, the expressions "% (*m/m*)" and "% (*V/V*)" are used to represent the mass and volume fractions respectively.

4.5 2-ethylhexanoic acid, minimum purity 98 % (*m/m*).

4.6 Vanadium compound, bis (1-phenylbutane-1, 3-dionato)-oxo-vanadium(IV) or any other oil-soluble vanadium compound. The vanadium content shall be accurately known to the nearest 0,01 % (*m/m*).

4.7 Nickel compound, cyclohexane butanoic acid-nickel salts or any other oil-soluble nickel compound. The nickel content shall be accurately known to the nearest 0,01 % (*m/m*).

4.8 Manganese compound, manganese octoate or any other oil-soluble manganese compound. The manganese content shall be approximately 10 % (*m/m*).

NOTE — Manganese compounds may contain insoluble impurities entrapped during manufacture, e.g. oxides. If this is evident, the compound should be cleaned by dissolution in petroleum spirit, boiling range 60 °C to 80 °C, followed by filtration and evaporation.

4.9 Manganese solution.

Dissolve the manganese compound (4.8) in a solution of 95 % (*V/V*) solvent mixture (4.3) and 5 % (*V/V*) 2-ethylhexanoic acid (4.5) to produce a manganese content of approximately 500 mg/kg [0,05 % (*m/m*)]. Store the solution in a tightly-stoppered brown glass bottle protected from light within the temperature range of 18 °C to 28 °C.

NOTE — Under these conditions, the solution is stable for at least 3 months.

4.10 Certified reference standards, use materials from a national standards body or accredited suppliers, if available.

5 Apparatus

5.1 Wavelength-dispersive X-ray fluorescence spectrometer, use any suitable X-ray spectrometer capable of being operated under the conditions in table 1 and of measuring the wavelengths in table 2, or other giving equivalent results. It shall be set up according to the manufacturer's instructions.

5.2 Analytical balance, capable of weighing to the nearest 0,1 mg.

5.3 Homogenizer, of non-aerating, high speed shear type or **heatable magnetic** or **ultrasonic stirrer**.

5.4 Flasks, of 50 ml capacity, narrow-necked, conical, made of borosilicate glass and fitted with ground-glass stoppers.

Table 1 — General requirements of spectrometer

Component	Requirement
Anode	Any tube anode may be used provided that the counting times be adjusted to achieve the required precision[1)]
Tube voltage and current	Set to provide maximum sensitivity for the lines in table 2 and within the power rating of the spectrometer
Analyzing cristal	Lithium fluoride (LiF) or any other crystal suitable for the dispersion of the wavelengths in table 2 within the angular range of the spectrometer
Optical path	Helium
Detector	Gas proportional detector with pulse-height analyser

1) If a chromium anode is used, either measure the Mn-K_β line (0,191 0 nm) and I_{UMn} at 0,188 5 nm, or measure the Mn-K_α line (0,210 3 nm) with a suitable tube filter to eliminate spectral interference from the Cr-K_β line and I_{UMn} at 0,219 0 nm.

6 Samples and sampling

6.1 Unless otherwise specified, samples shall be taken by the procedures described in ISO 3170 or ISO 3171.

6.2 Test portions from the samples shall be drawn after thorough mixing subdivision. Heat viscous, opaque, semi-solid or solid samples to a temperature which renders the sample liquid and homogenize using the homogenizer (5.3).

NOTE — Stratification in the sample cell, either of water or asphaltenic material, can lead to erroneous results.

7 Calibration solutions

7.1 General

Use either certified reference materials (4.10) or primary standards prepared from metal compounds (4.6 and 4.7) prepared as described in 7.2 as a basis for the preparation of stock solutions.

7.2 Preparation of stock solutions

7.2.1 Vanadium stock solutions

Weigh, to the nearest 0,1 mg, a quantity (m') of vanadium compound (4.6) to prepare stock solutions of approximately 1 000 mg/kg [0,10 % (m/m)] and 200 mg/kg [0,02 % (m/m)] vanadium content. Dissolve each of these in a mixture of 98,5 % (V/V) solvent mixture (4.3) and 1,5 % (V/V) acetyl acetone (4.4) and then weigh the solution to the nearest 0,1 mg ($m' + m''$). Mix the contents thoroughly using a homogenizer (5.3) and transfer to a tightly-stoppered brown glass bottle.

Calculate the exact vanadium content, W_{V2}, in mg/kg, to the nearest 1 mg/kg, from the mass of vanadium compound and mass of liquid using the following equation:

$$W_{V2} = \frac{m' \times W_{V1}}{m' + m''} \quad \ldots (1)$$

where

m' is the mass, in grams, of the vanadium compound;