SLOVENSKI STANDARD
SIST EN 17178:2019

01-november-2019

Tekoči naftni proizvodi - Določevanje skupne vsebnosti hlapnega žvepla v utekočinjenih naftnih plinih z ultravijolično fluorescentno spektroskopijo

Liquid petroleum products - Determination of the total volatile sulfur content in liquefied petroleum gases by ultraviolet fluorescence spectroscopy

Flüssige Mineralölerzeugnisse - Bestimmung des Gesamtgehaltes an flüchtigem Schwefel in Flüssiggas (LPG) durch Ultraviolettfluoreszenz-Spektroskopie

Produits pétroliers liquides - Détermination de la teneur en soufre volatil dans les gaz de pétrole liquéfiés par spectroscopie de fluorescence ultra-violette

Ta slovenski standard je istoveten z: **EN 17178:2019**

ICS:

75.160.20	Tekoča goriva	Liquid fuels

SIST EN 17178:2019 **en,fr,de**

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 17178

September 2019

ICS 75.160.20

English Version

Liquid petroleum products - Determination of the total volatile sulfur content in liquefied petroleum gases by ultraviolet fluorescence spectroscopy

Produits pétroliers liquides - Détermination de la teneur en soufre volatil dans les gaz de pétrole liquéfiés par spectroscopie de fluorescence ultra-violette

Flüssige Mineralölerzeugnisse - Bestimmung des Gesamtgehaltes an flüchtigem Schwefel in Flüssiggas (LPG) durch Ultraviolettfluoreszenz-Spektroskopie

This European Standard was approved by CEN on 20 June 2019.

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 CEN-CENELEC Management Centre 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 CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Ref. No. EN 17178:2019 E

Contents

Page

European foreword

This document (EN 17178:2019) has been prepared by Technical Committee CEN/TC 19 “Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin”, the secretariat of which is held by NEN.

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 March 2020 and conflicting national standards shall be withdrawn at the latest by March 2020.

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

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

The sulfur content of LPG used for fuel purposes contributes to SO_x emissions and can lead to corrosion in engine and exhaust systems. Sulfur also acts as catalyst poison, blocking and damaging catalysts like catalytic converters in cars and process catalysts in the chemical industry where LPG is used as feedstock.

Due to the need for lower sulfur limits, this document was developed in order to solve precision issues with existing test methods [3, 4].

1 Scope

This document specifies an ultraviolet (UV) fluorescence test method for the determination of the sulfur content of liquefied petroleum gases (LPG) containing up to 0,35 % (m/m) halogens, and having sulfur contents in the range of 2 mg/kg to 50 mg/kg.

This test method does not detect sulfur compounds that do not vaporize under the conditions of the test.

NOTE 1 LPG is defined as low pressure liquefied gas composed of one or more light hydrocarbons which are assigned to UN 1011, 1075, 1965, 1969 or 1978 only and which consists mainly of propane, propene, butane, butane isomers, butenes with traces of other hydrocarbon gases.

NOTE 2 For the purposes of this document, the terms "% (m/m)" and "% (V/V)" are used to represent respectively the mass fraction, μ, and the volume fraction, φ.

WARNING — The use of this standard can involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of users of this standard to take appropriate measures to ensure the safety and health of personnel prior to the application of the standard, and fulfil statutory and regulatory requirements for this purpose.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

EN ISO 4257, *Liquefied petroleum gases — Method of sampling (ISO 4257)*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

This test method is used to determine sulfur in process feeds and in finished products and can also be used for compliance determinations when acceptable to a regulatory authority.

An appropriate sample is taken from the sample container and introduced into the analyser. The gaseous sample then enters a high temperature (900 °C – 1 100 °C) combustion tube where sulfur is oxidized to sulfur dioxide (SO_2) in an oxygen rich atmosphere.

Water produced during the sample combustion is removed and the remaining sample combustion gases are exposed to UV light. The SO_2 absorbs the energy from the UV light and is converted to excited sulfur dioxide (SO_2*). The fluorescence emitted from the excited SO_2* molecules as they return to a stable state SO_2 is detected by a photomultiplier tube and the resulting signal is a measure of the sulfur contained in the sample.

5 Reagents and materials

5.1 Inert gas, argon or helium, high purity grade with a minimum purity of 99,9 % (*V*/*V*).

5.2 Oxygen, high purity grade with a minimum purity of 99,75 % (*V*/*V*), maximum moisture content of 5 mg/kg preferably dried over an additional molecular sieve.

CAUTION — Oxygen vigorously accelerates combustion.

5.3 Calibration standards.

Certified LPG calibration standards from commercial sources or calibration gases prepared using certified permeation tube devices are recommended. Table 1 lists the sulfur source material and diluent matrices proven to give good results.

Table 1 — Standard materials

Sulfur source	Diluent
Dimethyl sulfide	Mixture of n-butane, iso-butane, propylene, propane

Other sulfur sources and diluent materials may be used if precision and accuracy are not degraded.

NOTE Calibration standards are typically re-mixed and re-certified on a regular basis depending upon frequency of use and age. These calibration standards have a useful shelf life of about 6 to 12 months.

Due to the reactive nature of the sulfur components, it is recommended to measure the calibration standards shortly after they have been prepared and pour these into a suitably passivated LPG sampling cylinder.

5.4 Quality control samples, preferably portions of one or more gas or LPG materials, stable and representative of the samples of interest.

6 Apparatus

Figure 1 illustrates the basic components of an UVF analyser.

6.1 Furnace, comprising an electric device, capable of maintaining a temperature sufficient to pyrolyze the entire sample and oxidize sulfur to sulfur dioxide.

6.2 Combustion tube, constructed to allow the direct injection of the sample into the heated oxidation zone of the furnace (6.1). Check with the instrument manufacturer for specific combustion tube requirements.

The combustion tube shall have side arms for the introduction of oxygen and carrier gas. The oxidation section shall be large enough to ensure complete combustion of the sample. It may be set either in a horizontal or vertical position.

6.3 Flow controllers, capable of maintaining a constant supply of oxygen and carrier gas.

6.4 Vapour drier, capable of removing water vapour formed during combustion prior to measurement by the detector (6.5).

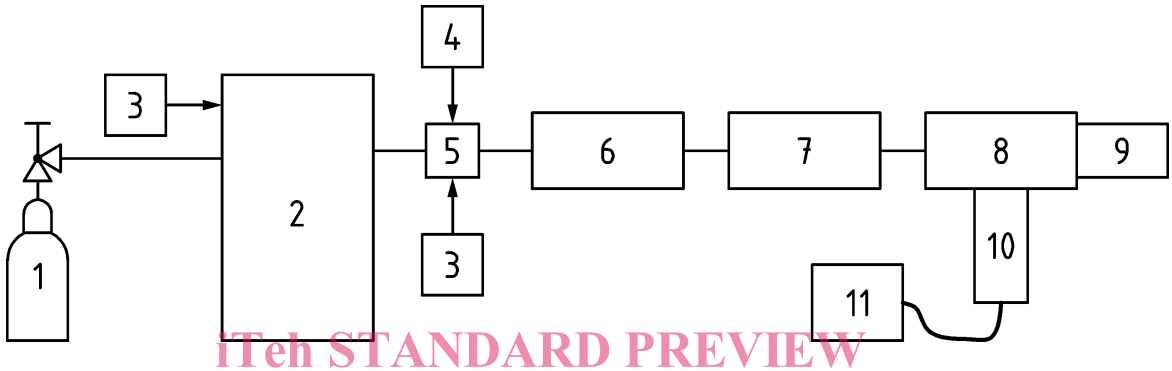
6.5 UV fluorescence detector, selective and quantitative, capable of measuring light emitted from the fluorescence of sulfur dioxide by UV light.

WARNING — Exposure to excessive quantities of UV light is injurious to health. The operator shall avoid exposing any part of his/her person, especially his/her eyes, not only to direct UV light, but also to secondary or scattered radiation that may be present.

6.6 Sample inlet system

The system provides a heated gas-sampling valve, or a LPG-sampling valve, or both, with a heated expansion chamber, connected to the inlet of the oxidation area. The system is swept by an inert carrier gas and shall allow the quantitative delivery of the material to be analysed into the oxidation zone at a controlled and repeatable rate of approximately 30 ml/min.

NOTE Special inert treatment of the sample inlet system might be required to ensure precision on samples containing low levels of sulfur.



Key

1	sample container	7	water separator
2	gas/LPG autosampler	8	sulfur detector
3	carrier gas	9	UV source
4	O_2	10	photomultiplier tube (PMT)
5	mixing chamber	11	PC
6	furnace		

Figure 1 — Synopsis of the apparatus

7 Sampling

7.1 Obtain a sample in accordance with EN ISO 4257.

7.2 Samples should be analysed as soon as possible after removal from bulk supplies, to prevent loss of sulfur or contamination due to exposure or contact with the sample container.

7.3 If the sample is not used immediately, thoroughly mix it in its container prior to taking a test portion. The use of sulfur-inert cylinders and fittings can help to reduce contamination and to improve the stability of the samples.

8 Calibration

8.1 Calibration range

Table 2 shows the calibration range. Preferably a sulfur compound and a diluent type representative of the samples to be analysed shall be used.