SLOVENSKI STANDARD
oSIST prEN 17306:2022

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Tekoči naftni proizvodi - Določanje destilacijskih značilnosti pri atmosferskem tlaku - Mikrodestilacija

Liquid petroleum products - Determination of distillation characteristics at atmospheric pressure - Micro-distillation

Flüssige Mineralölerzeugnisse - Bestimmung der Destillationseigenschaften bei atmosphärischem Druck - Mikrodestillation

Produits pétroliers liquides - Détermination des caractéristiques de distillation à la pression atmosphérique - Micro-distillation

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English Version

Liquid petroleum products - Determination of distillation characteristics at atmospheric pressure - Micro-distillation

Produits pétroliers liquides - Détermination des caractéristiques de distillation à la pression atmosphérique - Micro-distillation

lüssige Mineralölerzeugnisse - Bestimmung der Destillationseigenschaften bei atmosphärischem Druck - Mikrodestillation

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

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European foreword

This document (prEN 17306:2022) 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 document is currently submitted to the CEN Enquiry.

This document will supersede EN 17306:2019.

In comparison with the previous edition, a bias correction explanation has been introduced, which has no effect on the method precision.

Introduction

The distillation (volatility) characteristics of hydrocarbons and other liquids have an important effect on their safety and performance, especially in the case of fuels and solvents. The boiling range gives information on the composition, the properties, and the behaviour of the fuel during storage and use. Volatility is the major determinant of the tendency of a hydrocarbon mixture to produce potentially explosive vapours.

The distillation characteristics are critically important for both automotive and aviation gasolines, affecting starting, warm-up and tendency to vapour lock at high operating temperature or at high altitude, or both. The presence of high boiling point components in these and other fuels can significantly affect the degree of formation of solid combustion deposits.

Distillation limits are often included in petroleum product specifications, in commercial contract agreements, process refinery/control applications, and for compliance to regulatory rules.

This test method can be applied to contaminated products or hydrocarbon mixtures. This is valuable for fast product quality screening, refining process monitoring, fuel adulteration control, or other purposes including use as a portable apparatus for field testing.

This document is as of the time of publication technically equivalent to ASTM D7345 [1], on which it is based.

This test method uses an automatic micro distillation apparatus, provides fast results using small sample volume, and eliminates much of the operator time and subjectivity in comparison to EN ISO 3405 or ASTM D1160 [2].

1 Scope

This document specifies a laboratory method for the determination of the distillation characteristics of light and middle distillates derived from petroleum and related products of synthetic or biological origin with initial boiling points above 20 °C and end-points below approximately 400 °C, at atmospheric pressure utilizing an automatic micro distillation apparatus.

This test method is applicable to such products as light and middle distillates, automotive spark-ignition engine fuels, automotive spark-ignition engine fuels containing up to 20 % (*V/V*) ethanol, aviation gasolines, aviation turbine fuels, (paraffinic) diesel fuels, FAME (B100), diesel blends up to 30 % (*V/V*) fatty acid methyl esters (FAME), special petroleum spirits, naphtha's, white spirits, kerosene's, burner fuels, and marine fuels.

The test method is also applicable to hydrocarbons with a narrow boiling range, like organic solvents or oxygenated compounds.

The test method is designed for the analysis of distillate products; it is not applicable to products containing appreciable quantities of residual material.

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

NOTE For the purpose of this document, the expression "% (*V/V*)" is used to represent the volume fraction.

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 3170, *Petroleum liquids - Manual sampling (ISO 3170)*

EN ISO 3171, *Petroleum liquids - Automatic pipeline sampling (ISO 3171)*

EN ISO 3405, *Petroleum and related products from natural or synthetic sources - Determination of distillation characteristics at atmospheric pressure (ISO 3405)*

EN ISO 4259-1, *Petroleum and related products - Precision of measurement methods and results - Part 1: Determination of precision data in relation to methods of test (ISO 4259-1)*

3 Terms and definitions

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

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

— IEC Electropedia: available at https://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
automatic apparatus
microprocessor-controlled unit that performs the procedures of automatically controlling the evaporation of a liquid specimen under specific conditions of this test method, collecting measurement data and converting this data by patented algorithm in order to predict distillation results in correlation with industry recognized reference method

3.2
corrected temperature reading
temperature readings, corrected to 101,3 kPa barometric pressure

3.3
end point
final boiling point
FBP
maximum thermometer reading (corrected) obtained during the test

Note 1 to entry: This usually occurs after the evaporation of all liquid from the bottom of the distillation flask.

Note 2 to entry: The term maximum temperature is a frequently used synonym.

3.4
flask internal pressure
pressure within the distillation flask obtained during the test by a differential pressure sensor of automatic apparatus

Note 1 to entry: The flask internal pressure data recorded during the test is automatically converted to the volume percent recovered or evaporated data by patented algorithm employed by automatic apparatus.

3.5
initial boiling point
IBP
corrected temperature readings that corresponds to the instant of the flask internal pressure rise observed

3.6
liquid temperature
temperature of the liquid specimen in the distillation flask during the test obtained by a liquid temperature measuring device of automatic apparatus

3.7
percent recovered
volume of condensate observed by the automatic apparatus at any point in the distillation, expressed as a percentage of the charge volume, in connection with a simultaneous temperature reading

3.8
percent recovery
recovery predicted by the automatic apparatus and expressed as a percentage of the charge volume

3.9
percent residue
volume of residue in the distillation flask expressed as a percentage of the charge volume

3.10
reference method
test method or its analogues which is widely used for expression of the distillation characteristics of petroleum products in industry

3.11
temperature reading
adjusted vapour and liquid temperature by using an algorithm of the automatic apparatus to mimic the same temperature lag and emergent stem effects as would be seen when using a liquid-in-glass thermometer to determine the distillation characteristics

3.12
vapour temperature
temperature of the vapour in the distillation flask during the test obtained by a vapour temperature measuring device of automatic apparatus

4 Principle

A sample is transferred into the distillation flask, the distillation flask is placed into position on the automatic apparatus, and heat is applied to the bottom of the distillation flask.

The automatic apparatus measures and records sample vapour and liquid temperatures, and pressure in the distillation flask as the sample gradually distils under atmospheric pressure conditions. Automatic recordings are made throughout the distillation and the data stored into the apparatus memory.

At the conclusion of the distillation, the collected data are treated by the data processing system, converted to distillation characteristics and corrected for barometric pressure.

Test results are commonly expressed as percent recovered or evaporated versus corresponding temperature in compliance with industry recognized standard form and reference method either in a table or graphically, as a plot of the distillation curve.

5 Reagents and materials

5.1 Cleaning solvents, suitable for cleaning and drying the test flask such as; petroleum naphtha and acetone.

5.2 Toluene, 99,5 % purity.

5.3 n-Hexadecane, 99 % purity.

5.4 Chemicals of at least 99 % purity shall be used in the calibration procedure (see 9.3).

5.5 Granular pumice stones, clean and dry fine grade pumice stones of diameter 0,8 mm to 3,0 mm, approximately 10 grains are necessary for each test.

5.6 Sample drying agent, Anhydrous sodium sulphate has been found to be suitable.

6 Apparatus

6.1 Micro distillation unit

The basic components of the micro distillation unit are the distillation flask, a condensate recovery area with waste beaker, an enclosure for the distillation flask with the heat source and flask support, the specimen liquid temperature measuring device, the specimen vapour temperature measuring device, the distillation flask internal pressure measuring device, the ambient pressure measuring device, the control systems for regulating the distillation process, and the data processing system for converting recorded information into typical industry recognized standard report form.

A detailed description of the apparatus is given in Annex A.

6.2 Barometer for calibration

A pressure measuring device capable of measuring local station pressure with an accuracy of 0,1 kPa (or better, at the same elevation relative to sea level where the apparatus is located.

WARNING — The barometer is only required for periodic calibration of the ambient pressure measuring devices. Do not take readings from ordinary aneroid barometers, such as those used at weather stations and airports, since these are pre-corrected to give sea level readings.)

6.3 Sampling device

Glass or plastics syringe capacity (10 ± 0,3) ml or constant volume dispenser capacity (10 ± 0,3) ml.

6.4 Waste beaker

Glass approximately 200 ml capacity, outside diameter approximately 70 mm and height approximately 130 mm fitted with a cover to reduce evaporation. The cover design shall allow the beaker to remain open to atmospheric pressure.

7 Sampling

7.1 Unless otherwise specified, samples shall be taken as described in EN ISO 3170 or EN ISO 3171, whereas requirements of national regulations for the sampling of the product under test should be taken into account. At least 50 ml of sample is recommended.

7.2 The extreme sensitivity of volatility measurements to losses through evaporation and the resulting changes in composition is such as to require the utmost precaution in the drawing and handling of volatile product samples.

7.3 Sample shall be free from any suspended solids or other insoluble contaminations. Obtain another sample or remove solid particle by filtration. During filtration operation take care to minimize any loss of light ends.

7.4 All samples shall be stored in a tightly closed and leak-free container away from direct sunlight or sources of direct heat.

Protect samples containing light materials having expected initial boiling point lower than 100 °C from excessive temperatures prior to testing. This can be accomplished by storage of the sample container in an appropriate ice bath or refrigerator at a temperature below 10 °C. Other samples can be stored at ambient or lower temperature.

7.5 If the sample has partially or completely solidified during storage, it shall be carefully heated to a temperature when it is completely fluid. It shall be vigorously shaken after melting, prior to opening the sample container, to ensure homogeneity.

7.6 Wet samples of materials that visibly contain water are not suitable for testing by this test method. If the sample is not dry, obtain another sample that is free from suspended water.

If such a sample cannot be obtained, remove any free water by placing approximately 30 ml of the sample to be tested in a glass conical flask containing approximately 10 g of the drying agent. Stopper and shake gently. Allow the mixture to settle for approximately 15 min. Once the sample shows no visible signs of water, use a decanted portion of the sample for the analysis. It is recommended to filter the test portion from the residual or suspended drying agent. During this drying and filtration operations take care to minimize any loss of light ends. Report that the sample has been dried by the addition of a drying agent.

8 Apparatus preparation

Install the analyser for operation in accordance with the manufacturer's instructions.

This instrument shall be located away from direct sunlight, sources of direct heat or air draft.

Turn on the main power switch of the analyser.

9 Calibration, verification and quality control

9.1 General

Calibrate and verify the apparatus at each of the following occasions:

— after it is installed and commissioned;

— after replacement of critical parts or components;

— whenever QC sample determinations are not in statistical control, and the reasons for QC non-compliance have been suitably addressed.

9.2 Calibration

9.2.1 Follow the manufacturer's instructions for verifying the correct operation of the apparatus.

9.2.2 Temperature measurement system

At intervals of not more than six months or after the system has been replaced or repaired, or both, following the apparatus instruction manual, check the calibration of the liquid and vapour temperature measuring sensors by distilling of pure compounds, like toluene and n-hexadecane. If the sample is solid, heat it to about 25 °C and wait until all the material is liquid before starting the test.

NOTE The melting point of n-hexadecane is 18 °C.

9.2.3 Ambient pressure measuring device

At intervals of not more than six months, or after the system has been replaced or repaired, or both, the ambient pressure measuring device reading of the apparatus shall be verified against a barometer (6.3).

9.2.4 Differential pressure measuring device

At intervals of not more than six months, or after the system has been replaced or repaired, or both, the differential pressure measuring device reading of the apparatus shall be verified in accordance with the manufacturer's instructions.

9.3 Instrument verification

9.3.1 To verify the temperature measurement system, distil high purity toluene in accordance with this test method and comparing the temperature reading at 50 % distilled. If the temperature reading differs more than 0,5 °C from the expected temperature of 109,3 °C (see 9.2.2), then check the instrument calibration (see 9.2).

9.3.2 To verify the temperature measurement system at elevated temperatures, use *n*-hexadecane and record the temperature at 50 % distilled. If the temperature reading differs more than 1,0 °C from the expected temperature of 278,6 °C (see 9.2.2), then check the instrument calibration (see 9.2).