SLOVENSKI STANDARD
SIST EN 15471:2017

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Nadomešča:
SIST EN 15471:2008

Utekočinjeni naftni plini - Določevanje raztopljenih ostankov - Visokotemperaturna gravimetrijska metoda

Liquefied petroleum gases - Determination of dissolved residues - High-temperature gravimetric method

Flüssiggas - Bestimmung der gelösten Rückstände - Gravimetrisches Hochtemperaturverfahren

Gaz de pétrole liquéfié - Détermination des résidus dissous - Méthode gravimétrique à haute température

Ta slovenski standard je istoveten z: **EN 15471:2017**

ICS:

75.160.30	Plinska goriva	Gaseous fuels

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EUROPEAN STANDARD

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ICS 75.160.20

Supersedes EN 15471:2007

English Version

Liquefied petroleum gases - Determination of dissolved residues - High-temperature gravimetric method

Gaz de pétrole liquéfié - Détermination des résidus dissous - Méthode gravimétrique à haute température

Flüssiggas - Bestimmung der gelösten Rückstände - Gravimetrisches Hochtemperaturverfahren

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

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European Foreword

This document (EN 15471:2017) 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of an identical text or by endorsement, at the latest by January 2018, and conflicting national standards shall be withdrawn at the latest by January 2018.

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

This document supersedes EN 15471:2007.

The changes of this second version compared to the previous version are as follows:

a) numerous changes of both technical and editorial nature have been made to add clarity to the text;

b) clarification that the use of a drying agent in the desiccator is not recommended;

c) the description of the equipment used has been improved;

d) a note to filter discs (6.3) was added;

e) references to ASTM D381 have been replaced by references to EN ISO 6246.

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

1 Scope

This European Standard specifies a method for determining the dissolved residual matter in liquefied petroleum gases (LPG), which remains after evaporation at 105 °C using the jet evaporation equipment described in EN ISO 6246.

The measurement range is from 20 mg/kg to 100 mg/kg. Higher concentrations can be determined by adjusting the sample size.

The precision data of the method have been determined from 20 mg/kg to 100 mg/kg, with samples amount from 100 g to 50 g.

NOTE An alternative European Standard, EN 15470 [1], specifies a gas chromatography method.

WARNING — The use of this standard can involve hazardous materials, operations and equipment. This standard does not purport to address all of 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 application of the standard, and fulfil statutory and regulatory requirements for this purpose.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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)*

EN ISO 6246, *Petroleum products - Gum content of fuels - Jet evaporation method (ISO 6246)*

3 Terms and definitions

For the purposes of this document, the following term and definition applies.

3.1
liquefied petroleum gas
LPG
petroleum gas that can be stored and/or handled in the liquid phase under moderate conditions of pressure and at ambient temperature, consisting predominantly of propane and butanes, with small proportions of propene, butenes and pentanes/pentenes

4 Principle

A known mass of LPG is sampled and concentrated by evaporation. The concentrate is transferred into a beaker of 100 ml capacity and then evaporated by jet evaporation under controlled conditions of temperature and airflow. The oily residue remaining after this procedure is cooled and weighed.

5 Reagents

5.1 n-heptane, analytical grade.

5.2 propan-2-ol, technical grade, for the cooling bath.

5.3 Carbon dioxide, solid for the cooling bath.

5.4 Air, supply of filtered air at a pressure not more than 34,5 kPa.

5.5 Propan-2-one (acetone), analytical grade.

6 Apparatus

6.1 Sample cylinder, of a total mass compatible with the balance (6.10) used; preferably made of stainless steel fitted with two stainless steel valves free of oily materials, conforming to EN ISO 4257.

6.2 In-line filter support, made of stainless steel and for use at suitable high pressure.

6.3 Filter discs, plain membrane with nominal pore dimension of 0,8 µm.

NOTE This method enables to quantify the total dissolved residual matter in LPG; the purpose of the filter disc is to remove any particulate matter present in the LPG sample which would contribute to the total residual matter.

6.4 Cooling coil, made by coiling 4 m of stainless steel tube of external diameter 6 mm and internal diameter 4 mm onto a mandrel of a diameter of approximately 50 mm and fitted with the necessary connections (see 3 in Figure 2).

6.5 Cooling bath, comprised of a 0,5l to 2l Dewar flask, filled with propan-2-ol (5.2) cooled by adding solid carbon dioxide (5.3), to achieve a temperature of approximately −77 °C.

6.6 Beaker, made of glass, with a capacity of 1 l.

6.7 Boiling regulating rod, made of glass and a length of about 28 cm.

6.8 Beaker, made of glass and with a capacity 100 ml.

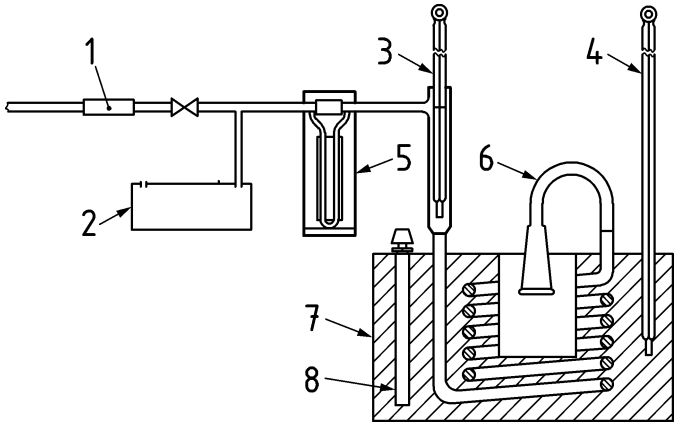
6.9 Balance for weighing the sample cylinder (6.1), capable of weighing to the nearest 1 g.

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

6.11 Desiccator, (a drying agent is not recommended).

6.12 Apparatus, for determining evaporation residues by air jet evaporation according to Figure 1 (for further information, see EN ISO 6246).

6.13 Oven, static type (without fan assisted circulation), explosion-proof, capable of heating to (105 ± 5) °C.



Key

1	dry air supply	5	flow indicator
2	dry and clean steam supply	6	removable adaptor
3	thermometer and well (optional)	7	heating block
4	thermometer	8	thermo-regulator

Figure 1 — Apparatus for determining oily residues by jet evaporation

7 Sampling

Samples shall be taken as described in EN ISO 4257 and/or in accordance with the requirements of national standards or regulations for the sampling of automotive LPG.

8 Procedure

8.1 Apparatus assembling

Assemble the apparatus, as shown in Figure 2, and operate the following instructions:

a) connect the lower valve of the sample cylinder to the in-line filter (6.2);

b) purge and fill the in-line filter and the cooling coil with the LPG to be analysed;

c) close the lower valve of the sample cylinder;

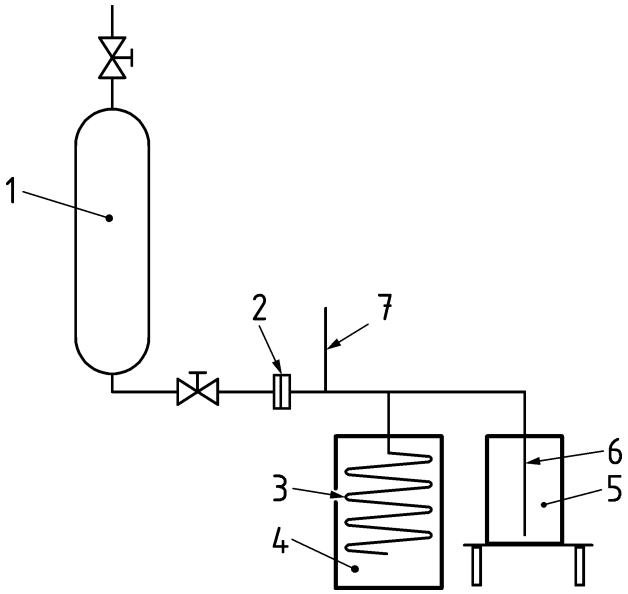
d) disconnect the in-line filter from the sample cylinder and weigh the cylinder to obtain its mass m_1 in grams;

e) reconnect the sample cylinder to the purged in-line filter and cooling coil;

f) position the 1 l beaker (6.6) and open the lower valve of the sample cylinder to obtain a steady flow of LPG sample into the beaker, until a mass of approximately 250 g is obtained (corresponding to approximately 500 ml);

g) close the lower valve of the sample cylinder;

h) disconnect the in-line filter from the sample cylinder and weigh the sample cylinder once more to obtain the post sample cylinder mass m_2 in grams.



Key

1 sample cylinder with two valves (6.1)
2 in-line filter support (6.2)
3 stainless steel cooling coil (6.4)
4 Dewar flask filled as stipulated in 6.5
5 1 l beaker (6.6)
6 boiling regulating rod (6.7)
7 earthing

Figure 2 — Apparatus assembling

The test sample mass, m_s, in grams, is determined by subtraction by the following formula:

$$m_s = m_1 - m_2 \quad (1)$$

8.2 Evaporation of the LPG

Place the beaker with the sample (see 8.1) into an explosion-proof hood. Leave to evaporate until there is no visible volume of liquid left in the beaker.

8.3 Jet evaporation procedure

The 100 ml beaker (6.8) is washed/rinsed with acetone (5.5) and distilled water and then dried at 105 °C for 30 min.

When found necessary one may also immerse the beaker in a mildly detergent solution or, for a deeper cleaning action, use an oxidative acid cleaning solution. After a few hours soaking period, rinse the beaker with distillated water and dry.

After cooling, this 100 ml beaker is placed in a desiccator (6.11) for 30 min, and then weighed to the nearest 0,1 mg to obtain the mass m_3. After evaporation of the sample, rinse the walls of the 1 l beaker (6.6) twice very carefully with approximately 20 ml n-heptane (5.1) each time and transfer the contents into the previously prepared 100 ml beaker. Place the 100 ml beaker into the jet evaporation apparatus (6.12) as shown in Figure 1 and let the contents evaporate for a period of 30 min at 105 °C with an air stream of 18 l/min to 24 l/min. Remove the beaker from the jet evaporation apparatus and place it in a desiccator for between 30 min and 1 h. Finally, weigh the beaker to the nearest 0,1 mg to obtain the mass m_4. The difference between this mass and the tare mass of the beaker gives the mass m_r of the dissolved residue obtained during the test.