BRITISH STANDARD

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

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ICS 75.160.30

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Flüssiggas - Bestimmung der gelösten Rückstände - Gravimetrisches Hochtemperaturverfahren

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Foreword

This document (EN 15471:2007) 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of an identical text or by endorsement, at the latest by May 2008, and conflicting national standards shall be withdrawn at the latest by May 2008.

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1 Scope

This Standard specifies a method, for determining the residual matter in liquefied petroleum gases (LPG), which remains after evaporation at 105 °C. This material represents those products deposited in car LPG vaporizers that are subject to a temperature equal to or greater than the boiling temperature of water. The range of determination extends from 50 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.

This method has been developed as a potential replacement of the commonly used method EN ISO 13757 [1], The advantages of the method are that a small quantity of LPG (100 ml) is required.

NOTE An alternative European Standard, EN 15470 [2], with the same scope, specifies a gas chromatography method with slightly better fidelity.

WARNING — Use of this method involves hazardous materials and operations. It is the responsibility of the user to establish appropriate safety and health precautions. All handling must be performed in a fume hood.

2 Normative reference

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.

EN ISO 4257, *Liquefied petroleum gases - Method of sampling (ISO 4257:2001)*

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, 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 **2-propanol**, technical grade, for the cooling bath.

5.3 **Solid carbon dioxide**, for the cooling bath.

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

5.5 **propanone (acetone)**

6 Apparatus

6.1 **Sample cylinder,** made of stainless steel, fitted with two stainless steel valves conforming to EN ISO 4257 and having a maximum service pressure of 3 MPa and a minimum capacity of 1 l. This cylinder is used to sample the LPG, of which the dissolved residues are to be determined.

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.

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**, comprising a Dewar flask, three-quarters filled with an appropriate liquid, e.g. 2-propanol (5.2) cooled with solid carbon dioxide, to achieve a temperature of about - 60 °C.

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

6.7 **Ebullition 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 **Balances**

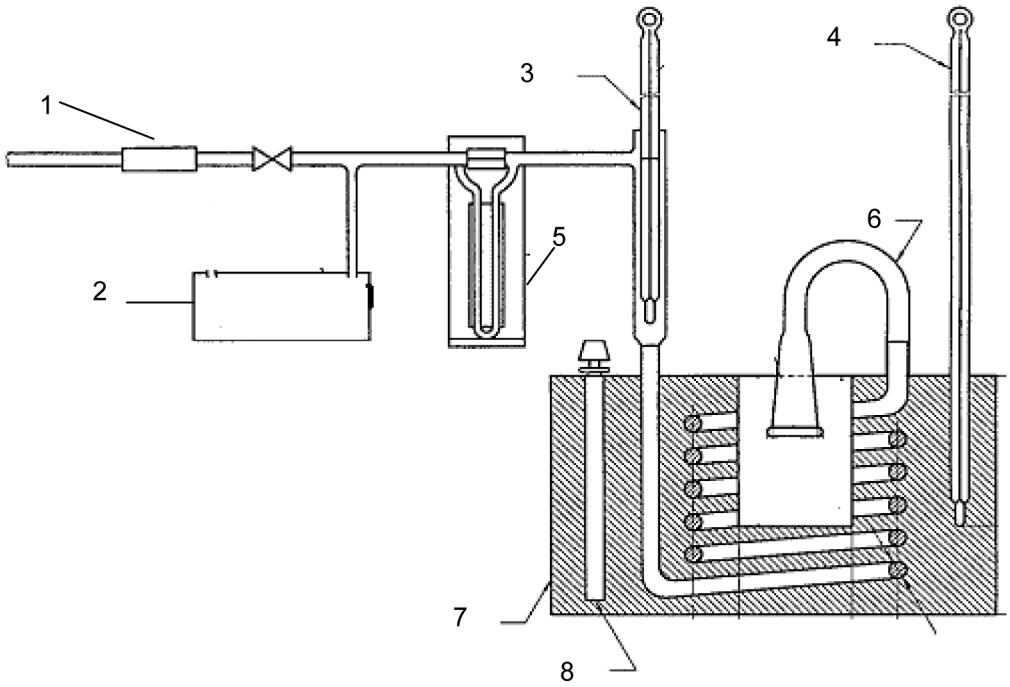
6.9.1 For weighing the sample cylinder (6.1), balance with a range of 10 kg minimum, accurate to within ± 1 g or better.

6.9.2 For weighing the beaker (6.8), balance with a range of 200 g, accurate to within ± 0,1 mg or better.

6.10 **Cabinet drier,** suitable for the preparation of the 100 ml beaker (6.8).

6.11 **Desiccator.**

6.12 **Apparatus**, for determining evaporation residues by air jet evaporation according to Figure 1 (for further information see ASTM D 381 [3]).



Key

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

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

a) slowly shake the sample cylinder (6.1) containing the LPG to be analysed, in order to mix the contents;

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

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

d) close the lower valve of the sample cylinder;

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

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

g) 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;

h) close the lower valve of the sample cylinder;

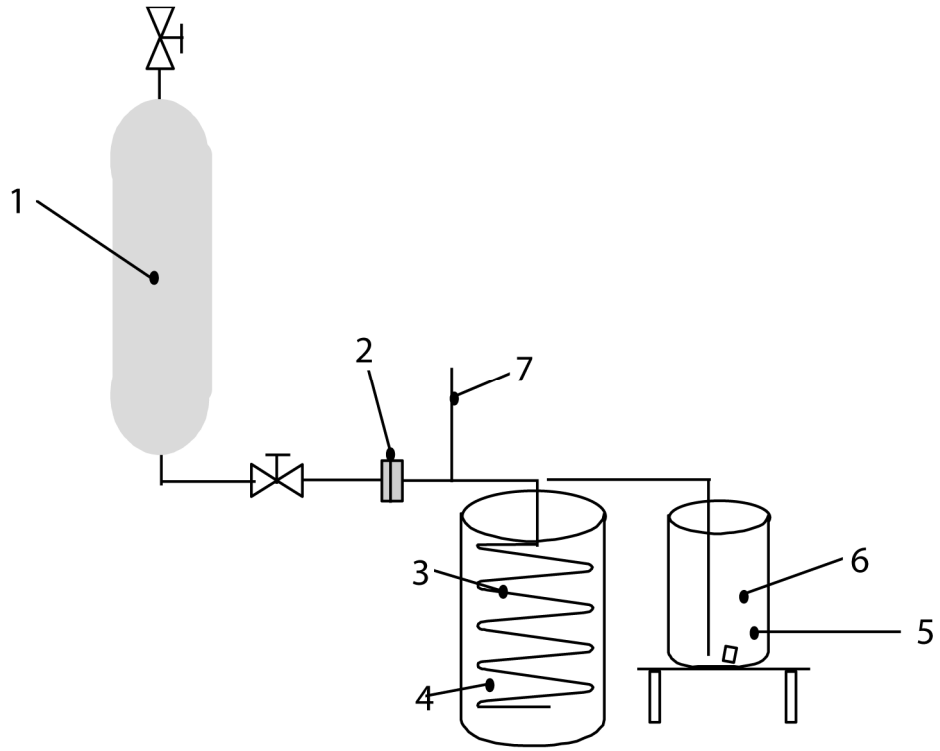
i) disconnect the in-line filter from the sample cylinder and weigh the sample cylinder once more to obtain the post sample cylinder mass m_2 in grams.

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

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

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.



Key

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

Figure 2 — Sampling assembly

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 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. 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.

$$m_r = (m_4 - m_3) \tag{2}$$

where

m_r is the mass, in milligrams, of residue obtained during the test;

m_3 is the tare mass, in milligrams, of the 100 ml beaker;

m_4 is the mass, in milligrams, of the 100 ml beaker plus residue.

9 Calculation

Calculate the dissolved residue of the sample, *ER*, after evaporation, in milligrams per kilogram, using the following equation:

$$ER = \frac{m_r \cdot 1000}{m_s} \tag{3}$$

where

m_r is the mass, in milligrams, of residue obtained during the test;

m_s is the mass, in grams, of the LPG sample.

10 Expression of results

Report the result to the nearest 1 mg/kg.

11 Precision

11.1 General

The precision of this method, established during interlaboratory tests relating to LPG samples with oily residue contents between 20 mg/kg and 100 mg/kg, and determined on the basis of statistical examination of interlaboratory test results [4], is as follows:

11.2 Repeatability, *r*

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on nominally identical test material would, in the long run, in the normal and

correct operation of the test method, exceed the value as given in Table 1 in only one case in 20 (see Table 2 for further details).

11.3 Reproducibility, *R*

The difference between two single and independent results obtained by different operators working in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of the test method, exceed the value as given in Table 1 in only one case in 20 (see Table 2 for further details).

Table 1 — Repeatability and reproducibility

r (mg/kg)	**R** (mg/kg)
$r = 0{,}081X + 4{,}25$ mg/kg	$R = 0{,}204\ X + 10{,}7$ mg/kg

where

X = the average of two results being compared, in mg/kg.

Table 2 — Calculated precision at different levels

Level (mg/kg)	**r** (mg/kg)	**R** (mg/kg)
20	6	15
50	8	21
75	10	26
100	12	31

Bibliography

[1] EN ISO 13757:1996, *Liquefied petroleum gases - Determination of oily residues - High-temperature method (ISO 13757:1996)*

[2] EN 15470, *Liquefied petroleum gases - Determination of dissolved residues - High temperature Gas chromatographic method*

[3] ASTM D381 *Standard test method for Gum Content in Fuels by Jet Evaporation*

[4] EN ISO 4259, *Petroleum products - Determination and application of precision data in relation to methods of test (ISO 4259:2006)*

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