BRITISH STANDARD

BS EN 15469:2007

Petroleum products — Test method for free water in liquefied petroleum gas by visual inspection

The European Standard EN 15469:2007 has the status of a British Standard

ICS 75.160.30

British Standards

National foreword

This British Standard is the UK implementation of EN 15469:2007.

The UK participation in its preparation was entrusted to Technical Committee PTI/15, Natural gas and gas analysis.

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

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 December 2007

ISBN 978 0 580 56655 4

Amendments issued since publication

Amd. No.	Date	Comments

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15469

November 2007

ICS 75.160.20

English Version

Petroleum products - Test method for free water in liquefied petroleum gas by visual inspection

Produits pétroliers - Détermination de l'eau libre dans les gaz de pétrole liquéfiés par inspection visuelle

Mineralölerzeugnisse - Bestimmung von freiem Wasser in Flüssiggas durch visuelle Begutachtung

This European Standard was approved by CEN on 7 October 2007.

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Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 15469:2007: E

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Foreword

This document (EN 15469:2007) 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.

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1 Scope

This test method covers the use of a pressure cylinder to determine the presence of free water in liquefied petroleum gas (LPG) by visual inspection below 0 °C.

2 Terms and definitions

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

2.1
liquefied petroleum gases (LPG)
petroleum gas, which 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

3 Principle

An LPG sample is transferred into a pressure transparent cylinder filled to 50 % of its capacity. The sample is left below 0 °C for not less than 1 h; then it is observed for the presence of ice lumps.

The results of the test shall be shown as *pass*, if ice is not present, or *fail*, if ice is present.

The LPG sample, containing dissolved water and/or free water, in condition of saturated vapour pressure and above 0 °C, is clear and does not become cloudy, which makes it difficult to distinguish the two liquid phases. Indeed, as both LPG and water are colourless, it is rather difficult to locate the separation surface between the two liquids at laboratory temperature, and it is only by using a transparent container and under optimal lighting conditions that it could be realised. Alternatively, since ice is far less transparent than water and can be better observed, the visibility of the water can be improved if it is frozen.

4 Significance and use

The test is used to detect liquid water transported with LPGs. Excess water content in LPGs can cause corrosive conditions degrading equipments. It can also separate and freeze, causing blockages of valves, pumps and regulators.

5 Apparatus

5.1 The sample cylinder will be fitted, preferably with a bottom valve, the stem of which will be short enough so as not to retain possible free water during the transfer of the sample to the test cylinder (5.2). If it is not possible to get such an equipment, use a classical sample cylinder with procedure 7.1.2.

5.2 The test cylinder will be a transparent container with a capacity of 250 ml and suitable for the pressure of the product to be tested with a suitable thermometer in contact with the liquid to be tested. It is fitted with a bottom valve and a top vent valve.

NOTE A transparent container made of plastic or glass as described in test method EN ISO 3993 [1] or any other transparent LPG container provided of a bottom and a top vent valve may be used.

5.3 A freezer capable of easily maintaining a temperature below 0 °C.

NOTE A cooling bath is not appropriate to perform this test method.

6 Preparation of apparatus

Before cleaning, the test cylinder (5.2) will be discharged from the previous LPG sample by removing the liquid phase, rather than the vapour phase, to prevent there being any residual residue left from the LPG on the walls.

The test cylinder (5.2) is carefully cleaned with a suitable solvent (for instance ethanol, or isopropanol), dried with nitrogen or dry air. The nitrogen or dry air is then removed by applying a vacuum to the cylinder. The vacuum is released by introducing dry vaporized butane into the test cylinder. The test cylinder is closed and stored in dry conditions before use. It shall not be suspected to retain some liquid (water or solvent) inside the bottom valve.

7 Procedure

7.1 Introduction of the Sample

7.1.1 Leave the test cylinder in the freezer (5.3) for at least 30 min before transferring the sample to the test cylinder.

7.1.2 If there is a possibility, due to the shape of the stem of the bottom (outlet) valve of the sampling cylinder, that liquid water already decanted could stay in it, then vigorously shake the sample for dispersing water droplets in the liquid.

7.1.3 Immediately connect the bottom valve of the sample cylinder (standing vertically), containing the product to be tested, to the inlet valve of the test cylinder by suitable tubing and fittings. The tubing will have a small diameter, in order not to introduce too much air in the test cylinder. Ascertain that these connections are free from leaks.

Open the sample cylinder bottom valve.

7.1.4 Open the test cylinder inlet valve and allow the product to flow inside it. If necessary, slightly open the vent valve of the test cylinder. Fill the test cylinder to approximately 50 % of its capacity.

7.1.5 Close all valves and disconnect the test cylinder from the sample cylinder.

7.1.6 With all valves closed, examine the test cylinder for leaks; if leaks are detected, discard the sample and repeat the sampling with a repaired test cylinder.

7.2 Testing

7.2.1 Place the test cylinder, filled with the sample as specified in 7.1, horizontally in the freezer (5.3) for not less than 1 h.

NOTE More time than 1 h may be required to cool the sample depending on the mass of the test cylinder and the quantity of product to be cooled.

7.2.2 Remove the test cylinder from the freezer, make sure that the temperature is below 0 °C and immediately observe the liquid to see if small ice lumps are visible at the lower generatrix or at the bottom of the test cylinder.

7.2.3 Because of air humidity, the external walls of the cylinder could be rapidly covered with a frozen layer of moisture making it impossible to observe the content. If so, wipe the ice off as fast as possible and observe again.

7.2.4 Record any presence of ice.

NOTE As self-training for the operator, it can be useful to simulate a failed test by adding 1 ml of water into the test cylinder and then follow the entire test procedure. In this condition it is certain that the ice lumps will appear and they will be easily distinguished; so the test performer will be aware of what he/she is looking for.

Perform the same test by introducing 1 ml of water in the sample cylinder, in order to check whether water contained in the sample cylinder is not trapped.

8 Report

The results of the test shall be shown as *pass*, if ice is absent, or *fail*, if ice is present.

9 Precision

It is not practicable to specify the precision of the procedure because the test is a pass - fail test, not a quantitative measurement.

Bibliography

[1] EN ISO 3993, *Liquefied petroleum gas and light hydrocarbons - Determination of density or relative density- Pressure hydrometer method (ISO 3993:1984).*

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

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