

Foam producing solutions for leak detection on gas installations

The European Standard EN 14291:2004 has the status of a
British Standard

ICS 23.040.99

National foreword

This British Standard is the official English language version of EN 14291:2004.

The UK participation in its preparation was entrusted to Technical Committee PSE/2, Jointing materials and compounds, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

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

Additional information

Attention is drawn to the fact that in the United Kingdom concerns have been raised over the possibility of corrosion being caused as a result of certain constituents that are permitted in leak detection solutions complying with this standard.

There is concern over the corrosion of thin-wall stainless steel tube when coming into contact with solutions containing the levels of halogens permitted by the standard and the following recommendations should be considered when applying the standard.

“Leak detection solutions containing more than 30 parts per million of halogens shall not be used on stainless steel components.”

Similarly there is concern over the corrosion of brass and copper components when coming into contact with solutions containing ammonia and the following recommendations should be considered when applying the standard.

“Leak detection solutions containing ammonia shall not be used on brass or copper components; the absence of ammonia being demonstrated by the fluid having a pH not exceeding 7.0.”

It is similarly recommended that the Instructions for Use shall include the instruction “After using this leak detecting solution, the tested joint shall be washed clean to ensure the removal of any residual solution which could result in the corrosion of the pipework or fittings.”

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the *BSI Catalogue* under the section entitled “International Standards Correspondence Index”, or by using the “Search” facility of the *BSI Electronic Catalogue* or of British Standards Online.

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English version

Foam producing solutions for leak detection on gas installations

Solution pour le détection des fuites aux installations de
gazSchaumbildende Lösungen zu Lecksuche an
Gasinstallationen

This European Standard was approved by CEN on 23 September 2004.

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

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

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Foreword

This document (EN 14291:2004) has been prepared by Technical Committee CEN/TC 108 “Sealing materials and lubricants for gas appliances and gas equipment”, 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 2005, and conflicting national standards shall be withdrawn at the latest by May 2005.

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

Introduction

There are different methods to detect and localize leaks on gas installation and gas equipment. One commonly used method is the application of surface-active aqueous solutions of detergents that produce foam on leaking gas installations. This standard specifies several important requirements for foam producing solutions especially for the detection of small leaks e.g. surface tension, foam stability, corrosiveness, and compatibility with sealing materials and lubricants.

It should be mentioned that excessive and repeated use of these aqueous detergent solutions on old threaded joints sealed with natural fibres (e. g. hemp or flax) and unsuitable sealing materials can temporarily seal a leaking joint by swelling of the natural fibres.

Since some plastic and copper pipe materials may be attacked by surface-active aqueous solutions of detergents used for leak detection, it is generally recommended to rinse plastic and copper pipes with water after leak detection by foam producing solutions according to this standard.

1 Scope

This document specifies requirements and test methods of foam producing solutions for leak detection (hereafter called leak detection solution) used for leak detection of combustible gases of the 1st family (town gas), 2nd family (natural gas), and 3rd family (liquefied petroleum gases (LPG)) (see EN 437) within the temperature range of -15 °C or 0 °C to 50 °C. It is applicable for leak detection solutions, which are delivered as ready to use solutions, e.g. in spray cans.

Note The leak detection solution covered by this document may also be used for other compressed gases. In this case the producer has to be consulted.

2 Normative references

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 377, *Lubricants for applications in appliances and associated controls using combustible gases except those designed for use in industrial processes.*

EN 437, *Test gases - Test pressures - Appliance categories.*

EN 456, *Paints, varnishes and related products - Determination of flashpoint - Rapid equilibrium method (ISO 3679:1983, modified).*

EN 751 - 2, *Sealing materials for metallic threaded joints in contact with 1st, 2nd, and 3rd family gases and hot water - Part 2: Non-hardening jointing compounds.*

EN 1412, *Copper and copper alloys - European numbering system.*

EN 10027 - 1, *Designation systems for steel - Part 1: Steel names, principal symbols*

EN ISO 10304 - 1:1995, *Water quality - Determination of dissolved fluoride, chloride, nitrite, orthophosphate, bromide, nitrate and sulfate ions, using liquid chromatography of ions - Part 1: Method for water with low contamination (ISO 10304-1:1992).*

ISO 304, *Surface active agents - Determination of surface tension by drawing up liquid films.*

ISO 696, *Surface active agents - Measurement of foaming power - Modified Ross-Miles-method.*

ISO 3819, *Laboratory glassware - Beakers.*

ISO 6974-4, *Natural gas- Determination of composition with defined uncertainty by gas chromatography- Part 4 Determination of nitrogen, carbon dioxide and C1 to C5 and C6+ hydrocarbons for a laboratory and on-line measuring system using two columns.*

ASTM D 1177, *Standard test method for freezing point of aqueous engine coolants.*

3 Terms and definitions

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

3.1 foam producing leak detection solution
aqueous solution with low surface tension, which indicates small gas leaks by formation of foam bubbles

3.2 gas family
for further information on types of gases see EN 437

4 Requirements

4.1 Surface tension

Leak detection solutions shall enable the detection of small gas leaks, therefore the surface tension shall be ≤ 30 mN/m.

4.2 Foam stability

Foam produced according to 6.2 shall be a maximum of 50 % decomposed 10 min after preparation.

4.3 Corrosiveness

When testing the corrosion attack on steel pipe sections according to 6.3.1 the loss in mass by corrosion shall be lower than 20 mg. Leak detection solutions shall contain less than 200 mg/l halogens (F^- and Cl^-), less than 1000 mg/l f sulfates (SO_4^{2-}) and shall not corrode the containers, they are filled in.

4.4 pH-value

The pH-value shall be within the range of 6 to 8.

4.5 Compatibility with non-hardening sealing materials and lubricants

Leak detection solutions shall not attack soft white paraffin jelly (white Vaseline) used for medical treatment as replacement for non-hardening sealing materials according to EN 751-2 and lubricants according to EN 377) or remove it from the metal surface when tested according to 6.5.

4.6 Flammability

The flash point determined according to 6.6 shall be higher than 55 °C.

The propellant used in spray cans shall not be flammable.

4.7 Freezing point

The freezing point of leak detection solutions for low temperature application determined according to 6.7 shall be equal or lower than -15 °C.

4.8 Toxicological harmlessness

The formulation of the leak detection solutions shall be toxicologically harmless.

5 Test samples and documentation

5.1 Test samples

The manufacturer or supplier shall submit 5 spray cans or 2 l leak detection solution in its original container(s) for the tests.

5.2 Documentation

The following documentation shall be submitted:

- a) Appropriate health and safety data sheets;
- b) Information on propellants used;
- c) Application and handling instructions.

6 Test methods

6.1 Determination of surface tension

The determination of the surface tension of leak detection solutions is performed according to ISO 304 with a platinum ring at $(20 \pm 0,5) ^\circ\text{C}$. The instrument is calibrated with double distilled water and toluene. The measured surface tension is corrected according to Harkins and Jordan by subtracting 1,9 mN/m from the measured value.

6.2 Determination of foam stability

The determination of foam stability is performed according to ISO 696 but with a 250 ml measuring cylinder, 150 ml leak detection solution and at a test temperature of $(23 \pm 2) ^\circ\text{C}$. 15 ml of the leak detection solution is poured into the measuring cylinder before starting the test.

The volume of foam is registered according to ISO 696, 30 s after the solution is poured into the cylinder and again after 10 min. The foam stability is calculated as the arithmetic mean value of 3 individual determinations on samples taken from different spray cans or containers.

6.3 Determination of corrosiveness

6.3.1 Corrosion attack on steel

Three precision steel pipe sections 32 mm x 3 mm (steel code S235G2T according to EN 10027-1), length $(20 \pm 0,5)$ mm, and three pipe sections of copper/zinc alloy (brass) CuZn₃₇ according to EN 1412 25 mm x 1,5 mm, length $(25 \pm 0,5)$ mm are cleaned first with toluene and then with acetone to remove any adhering corrosion protection means or cutting oil. Afterwards the parts are treated for (15 ± 1) min at (23 ± 2) °C with 100 ml 4 mol/l hydrochloric acid containing 1 g hexamethylene tetramine per litre as inhibitor and 1 % m/m of sodium dodecylsulfate as detergent to remove scales and corrosion products. Thereafter the parts are carefully washed with distilled water until chloride is no longer detectable in the washing water when adding a few drops of silver nitrate solution (precipitate when containing chloride), rinsed with acetone, dried in a desiccator for $(15 + 5)$ min at reduced pressure (≤ 25 mbar) over anhydrous calcium chloride. The steel pipe sections are weighed to the nearest 1 mg (values a_1 , a_2 and a_3). The cleaned and dried pipe sections shall no longer be touched with bare hands.

In each steel pipe section a brass pipe section is inserted and eccentrically fixed by pressing a small plastic plug (e.g. PE) between the pipe sections. The shorter steel pipe section is symmetrically fixed on the longer brass pipe section. The pipe sections are laid in beakers according to ISO 3819, capacity 250 ml, each containing about 45 ml leak detection solution. The pipe sections are three times rolled to and fro to wet the surface evenly. The pipe sections are then placed in the middle of the beaker – pipe axis in upright position. The depth of immersion of $(12,5 \pm 0,5)$ mm may need correction by adding or removing a few ml of the leak detection solution. The beakers are then stored for (96 ± 2) h at (23 ± 2) °C in a climate cabinet (cupboard) or a large desiccator over saturated sodium chloride solution with precipitates to guarantee constant relative humidity of about 75 % to 80 %.

After the storage period the pipe sections are dismantled and the steel pipe sections cleaned for $5 \text{ min} \pm 5 \text{ s}$ in an ultrasonic bath with a 1 % m/m solution of sodium dodecylsulfate to remove organic deposits of the leak detection solution from the pipe sections and the corrosion products. Afterwards the corrosion products are removed from the steel pipe sections by immersing them for $5 \text{ min} \pm 5 \text{ s}$ in inhibited hydrochloric acid as mentioned above. The steel pipe sections are weighed again after washing and drying as described above (values b_1 , b_2 and b_3).

Three further steel pipe sections are to be treated as described in the first paragraph (weight values a_4 to a_6). Subsequently these sections are immersed for $5 \text{ min} \pm 5 \text{ s}$ in inhibited hydrochloric acid. After that the three sections are weighed again after washing and drying (values b_4 to b_6).

The corrosive value d is calculated as follows:

$$d = [(a_1 - b_1) + (a_2 - b_2) + (a_3 - b_3) - (a_4 - b_4) - (a_5 - b_5) - (a_6 - b_6)] / 3$$

6.3.2 Determination of anions

The concentration of fluoride, chloride and sulfate is determined according to EN ISO 10304-1 using liquid chromatography of ions. Detergents and corrosion inhibitors are removed by passing the sample through a pre-column (e. g. PP C18 C macro porous divinylbenzene resin) according to clause 7 of EN ISO 10304-1:1995).

6.3.3 Corrosion attack on spray can

For testing the attack of leak detection solution on the spray can two half emptied spray cans are stored for four weeks at (50 ± 2) °C in the oven. Thereafter the leak detection solution shall show no visible change. The spray cans, opened after having been completely emptied, shall show no signs of corrosion.

6.4 Determination of pH-value

The determination of the pH-value is performed by a glass electrode and a pH-meter according to the instructions supplied by the manufacturer of the device. Leak detection solutions from spray cans are measured 1 h after 25 ml have been sprayed into an open 100 ml beaker. The contents shall be stirred several times to remove dissolved propellants.

6.5 Test of the compatibility with non-hardening sealing materials and lubricants

Two steel sheets (75 mm x 25 mm x 0,5 mm) after polishing and cleaning are half covered on both sides with soft white paraffin jelly (white vaseline) in about 1 mm thickness. These steel strips are put for 5 h \pm 10 min in two beakers (50 ml) filled with the leak detection solution. After the test period the leak detection solution shall not have dissolved or removed the paraffin from the steel. Discoloration is not evaluated.

6.6 Determination of inflammability

The determination of inflammability is performed by the determination of the flash point with reference to EN 456 by testing at 55 °C, whether the leak detection solution contains inflammable admixtures. The leak detection solution under test is heated in the closed cup to 55 °C, and then the ignition is tested.

Propellants are tested by gas chromatography according to ISO 6974-4 for inflammable components.

6.7 Determination of freezing point

The determination of the freezing point of leak detection solutions for low temperature suitability is performed according to ASTM D 1177 using for example a mixture of crushed ice with calcium chloride or dry ice (solid CO₂) in alcohol as a cooling bath.

7 Marking

The containers of leak detection solutions shall be clearly and indelibly marked in the language of the country of destination with the following information:

- a) Manufacturer's or supplier's name and/or registered trade mark;
- b) Designation of leak detection solution;
- c) Essential instructions for use;
- d) Appropriate handling and safety labelling;
- e) Unique identification mark providing traceability (e. g. a batch number) and production date or "use by" date (if applicable);
- f) An instruction that leak detection solutions shall be rinsed off by water when used on plastic or copper pipes.

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