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Liquefied petroleum gases — Detection of hydrogen sulfide — Lead acetate method

*Gaz de pétrole liquéfiés — Détection de l'acide sulfhydrique — Méthode
à l'acétate de plomb*



Reference number
ISO 8819:1993(E)

Foreword

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International Standard ISO 8819 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 8819:1987), which has been technically revised.

Annex A of this International Standard is for information only.

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Liquefied petroleum gases — Detection of hydrogen sulfide — Lead acetate method

WARNING — The use of this International Standard may 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 the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the detection of hydrogen sulfide in liquefied petroleum gases.

The lower limit of detectability is 4 mg of hydrogen sulfide in 1 m³ of liquefied petroleum gas. Methyl mercaptan, if present, produces a transitory yellow stain on lead acetate paper which, however, fades completely in less than 5 min. Other sulfur compounds present in liquefied petroleum gas do not interfere with the test.

NOTE 1 Liquefied petroleum gases and the products of their combustion should not be unduly corrosive or reactive to materials with which they come into contact. It is therefore important that the presence of any hydrogen sulfide, a highly reactive substance, be detected. Additionally, the odour of hydrogen sulfide is unacceptable in some applications of liquefied petroleum gases (e.g. lighter fuel).

2 Principle

The vaporized sample is passed over moist lead acetate paper under controlled conditions. Hydrogen sulfide reacts with lead acetate to form lead sulfide and thus produces a coloration on the paper which will vary from yellow to black, depending upon the amount of hydrogen sulfide present.

3 Apparatus

3.1 Apparatus for detecting hydrogen sulfide in liquefied petroleum gas, as shown in figure 1.

3.2 Lead acetate test paper, either prepared by dipping strips of smooth filter paper into an aqueous 50 g/l solution of lead acetate, withdrawing the strips and removing excess solution from them with clean filter paper, or commercially available test paper if it is of a type that has been shown to give similar results to paper prepared as above.

The strips of the test paper shall be approximately 51 mm long by 9,5 mm wide and have a 3,5 mm diameter hole near to one end. This hole shall permit the strip to hang freely in the test apparatus.

3.3 Flow indicator, comprising a wet test meter or variable area flowmeter, which measures gas flow rates in the range of 2 l/min to 3 l/min.

4 Sampling

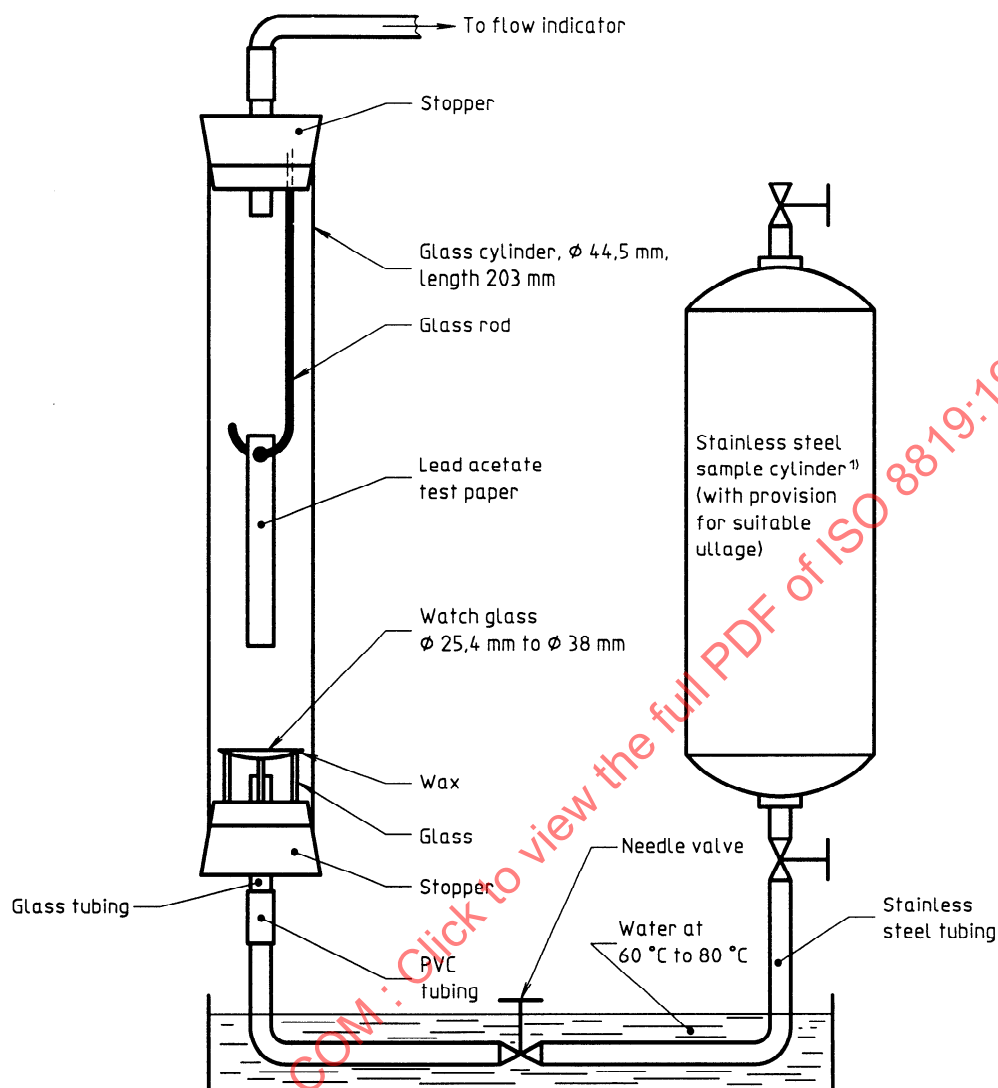
Information on constraints to the use of sample containers and to laboratory testing is given in annex A.

5 Procedure

5.1 Connect the test apparatus (3.1) to the sample source with a minimum length of clean stainless steel tubing.

NOTE 2 The use of rubber hoses, stoppers, etc., should be avoided since hydrogen sulfide has an affinity for rubber which will result in erroneous test results.

Flush the line and apparatus with the sample gas for approximately 1 min. Fill the water reservoir with water at a temperature of 60 °C to 80 °C. By use of the needle valve, adjust the rate of gas flow to 2,3 l/min $\pm$ 0,2 l/min.



1) It is preferable to avoid the use of a sample cylinder by instead using a direct connection to the sample source (see clause 4).

Figure 1 Apparatus for detecting hydrogen sulfide in liquefied petroleum gas

NOTE 3 Any restriction in the downstream flow indicator, or rapid opening of the needle valve, may result in over-pressuring of the glass cylinder.

Immediately place a single strip of lead acetate paper on the hook in the glass cylinder of the apparatus (3.1) so that the paper, moistened with distilled water, is held midway between the watch glass and the bottom of the upper stopper. Expose the moist paper

for exactly 2 min to the gas flow maintained at $2,3 \text{ l/min} \pm 0,2 \text{ l/min}$. Remove the test paper and proceed as described in 5.2.

5.2 Compare the exposed test paper with a moistened test paper that has not been exposed.

5.2.1 If the distinct coloration is absent, report hydrogen sulfide negative.

5.2.2 If a distinct coloration is present, wait 5 min and again compare the exposed test paper with the moistened test paper that has not been exposed and record the test result as follows:

- a) if the distinct coloration has persisted, report hydrogen sulfide positive;
- b) if initially there was a distinct yellow coloration that is no longer present after 5 min, report methyl mercaptan present.

NOTE 4 In the case of pass-fail data or results from other qualitative tests, no generally accepted method for determining precision is currently available.

6 Test report

The test report shall contain at least the following information:

- a) sufficient details for complete identification of the product tested;
- b) a reference to this International Standard;
- c) the result of the test;
- d) any deviation, by agreement or otherwise, from the procedure specified;
- e) the date of the test.

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Annex A

(informative)

Use of sample cylinders

Because of the chemical activity and physical adsorptive properties of hydrogen sulfide, it is highly desirable to connect the test apparatus directly to the sample source and perform the test on site, rather than transferring sample into a sample cylinder for testing in the laboratory.

Experience has demonstrated that the hydrogen sulfide concentration in liquefied petroleum gas samples collected in scrupulously clean sample cylinders is

depleted even though the cylinder is made of stainless steel. The exact opposite, an increase in hydrogen sulfide concentration, has been found when samples of liquefied petroleum gas containing no hydrogen sulfide are transferred into sample cylinders that have not been properly cleaned and have been in use with samples containing hydrogen sulfide. This indicates that data obtained on samples tested for hydrogen sulfide in the laboratory are unreliable.

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