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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

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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

1 Scope and field of application

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 the 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 — 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 should 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 the figure.

3.2 Lead acetate test paper. Prepare the test paper by dipping strips of smooth filter paper into an aqueous 50 g/l solution of lead acetate, withdraw the strips and remove excess solution from them with clean filter paper.

Commercially available test papers may be used as an alternative, if they are of a type that has been shown to give similar results to papers prepared as above.

Use strips of the test paper approximately 51 mm long by 9,5 mm wide and having 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. A wet test meter or a variable area flowmeter, which measures gas flow rates in the range of 2 to 3 l/min.

4 Sampling

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 the spot, 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.

5 Procedure

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

NOTE — 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 for about 1 min. Fill the water reservoir with water at a temperature of 60 to 80 °C. By use of the needle valve, adjust the rate of gas flow to $2,3 \pm 0,2$ l/min.

NOTE — Any restriction in the downstream flow indicator, or rapid opening of the needle valve, may result in overpressuring 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 $2,3 \pm 0,2$ 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 a 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 :

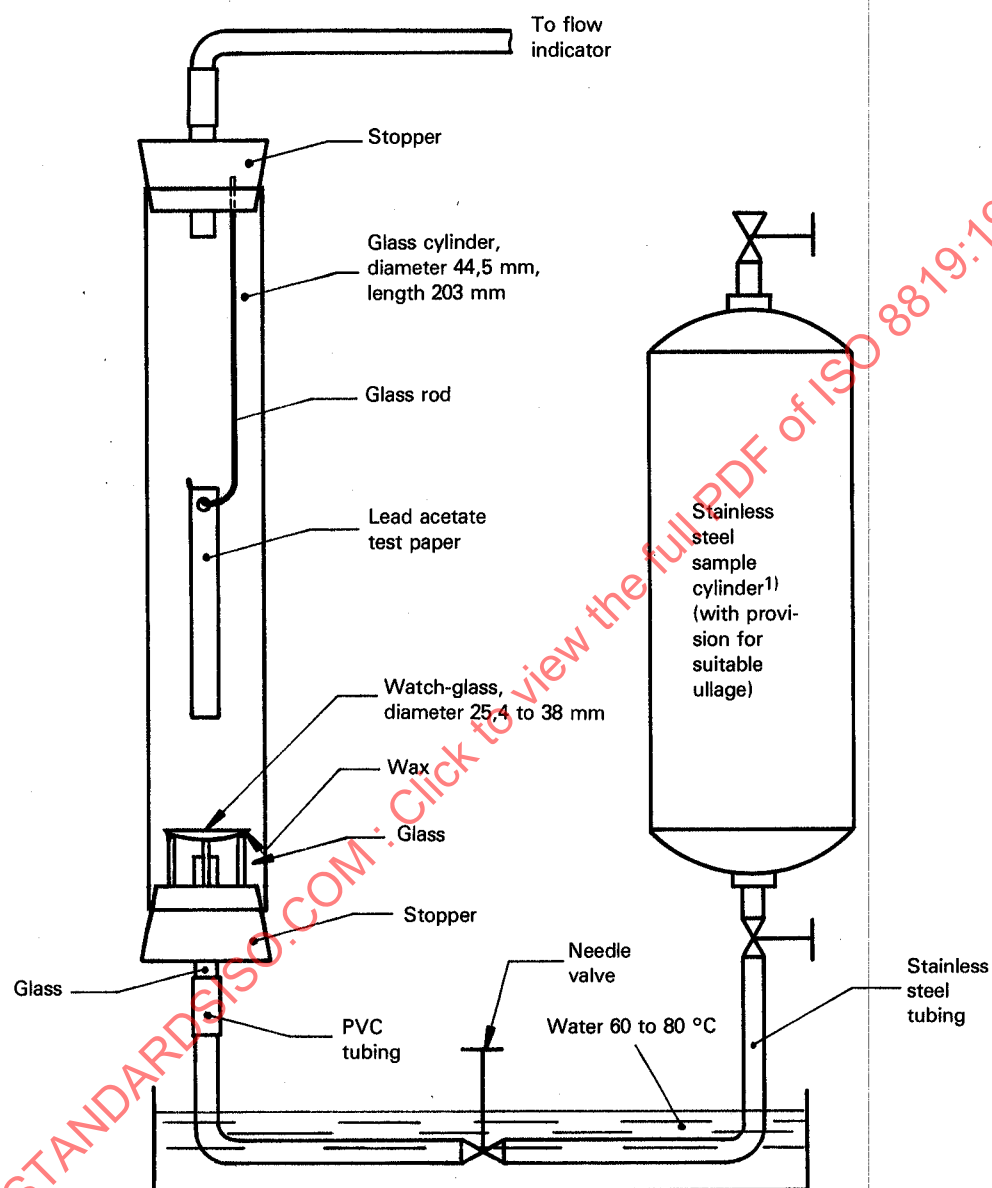
- a) if the distinct coloration has persisted, report hydrogen sulfide positive;
- b) if initially there was a distinct yellow coloration that is no longer present after 5 min, report methyl mercaptan present.

NOTE — 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) the type and 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.



1) It is preferable to replace the sample cylinder by a direct connection to the sample source (see clause 4).

Figure — Apparatus for detecting hydrogen sulfide in liquefied petroleum gas

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