

Method for

Determination of mercury in natural gas

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Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Petroleum Standards Policy Committee (PTC/-) to Technical Committee PTC/15, upon which the following bodies were represented:

- British Compressed Gases Association
- British Gas plc
- Department of Trade and Industry (Gas and Oil Measurement Branch)
- Department of Trade and Industry (National Engineering Laboratory)
- Institute of Petroleum

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National foreword

This British Standard has been prepared under the direction of the Petroleum Standards Policy Committee. It is identical with ISO 6978:1992 *Natural gas — Determination of mercury*, published by the International Organization for Standardization (ISO).

In deciding to adopt ISO 6978 as a British Standard, the Technical Committee considered that, although mercury determination is not carried out frequently on natural gas in the United Kingdom, the method described in ISO 6978 is the preferred method for this and has applications for other gases, in particular those produced electrolytically using a mercury electrode.

Cross-references

International standard	Corresponding British Standard
ISO 641:1975	BS 2761:1983 <i>Specification for spherical ground joints</i> (Technically equivalent)
ISO 3696:1987	BS 3978:1987 <i>Specification for water for laboratory use</i> (Identical)
ISO 4793:1980	BS 1752:1983 <i>Specification for laboratory sintered or fritted filters including porosity grading</i> (Identical)

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

WARNING — Mercury presents a health hazard if incorrectly handled. Avoid prolonged inhalation of the vapour. Spillages of mercury should be removed immediately, even from places which are difficult to access.

1 Scope

This International Standard specifies two methods, A and B, for the determination of mercury, including elemental mercury, dimethyl mercury and diethyl mercury, in natural gas.

Method A prescribes sampling at atmospheric pressure, using absorption in potassium permanganate solution followed by reduction of mercury ions and subsequent analysis by flameless atomic absorption spectrometry. The lower detection limit is $0,05 \mu\text{g}/\text{m}^3$ for a sampling time of 2 h. Aromatic hydrocarbons may interfere. If aromatic hydrocarbons are present, method B is recommended.

Method B prescribes sampling at atmospheric or higher pressure using adsorption of mercury on silver/gold followed by desorption and subsequent analysis by flameless atomic absorption spectrometry. The lower detection limit is $3 \times 10^{-4} \mu\text{g}/\text{m}^3$ at a minimum pressure of 3 MPa (30 bar) and for a sampling time of 2 h.

Method A is recommended for natural gases with a high mercury content ($> 0,5 \mu\text{g}/\text{m}^3$) while method B is preferred for lower mercury concentrations ($10^{-3} \mu\text{g}/\text{m}^3$ to $1 \mu\text{g}/\text{m}^3$).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 641:1975, *Laboratory glassware — Interchangeable spherical ground joints.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

3 Principle

3.1 Method A: Determination of mercury by sampling at atmospheric pressure

The gas is passed through a gas washing bottle filled with a potassium permanganate-sulfuric acid solution. The mercury present in the gas is oxidized to mercury(II) ions. The excess of permanganate is reduced by a hydroxylamine hydrochloride solution, and the mercury(II) ions are reduced by a tin(II) chloride solution to form elemental mercury which is drawn into nitrogen stream. The nitrogen stream is passed through an absorption cell (optical path length 175 mm) which is placed in an atomic absorption spectrometer. The mercury is determined by measuring the absorbance of the mercury resonance line at 253,7 nm.

3.2 Method B: Determination of mercury by sampling at atmospheric or high pressure

The gas is passed through a silica tube containing silver lint placed in a stainless steel high-pressure vessel. Mercury present in the gas is quantitatively trapped by the silver and is subsequently released by heating the tube in an oven at 850 °C whilst passing a stream of air. The mercury vapour is trapped in a second silica tube filled with gold thread, whilst hydrocarbons and other contaminants are oxidized and passed through. The tube containing gold thread is heated in an oven at 850 °C and, by means of an air stream, the mercury vapour is passed through an absorption cuvette and determined by flameless atomic absorption spectrometry at a wavelength of 253,7 nm.

4 Method A: Determination of mercury by sampling at atmospheric pressure

4.1 Reagents and materials

All reagents shall be of recognized analytical grade and have a mercury content as low as possible. For all purposes where water is mentioned, water of a suitable purity shall be used; various types of water are described in ISO 3696.

4.1.1 Aqua regia, cleaning solution

Mix 3 volumes of concentrated hydrochloric acid ($\rho_{20} = 1,19 \text{ g/ml}$) with 1 volume of concentrated nitric acid ($\rho_{20} = 1,40 \text{ g/ml}$).

4.1.2 Sulfuric acid solution, 65 % (m/m).

Cautiously add 1 volume of concentrated sulfuric acid ($\rho_{20} = 1,84 \text{ g/ml}$) to 1 volume of water.