INTERNATIONAL STANDARD

ISO 6978-1

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Natural gas — Determination of mercury —

Part 1: Sampling of mercury by chemisorption on iodine

Gaz naturel — Détermination de la teneur en mercure —
Partie 1: Échantillonnage de mercure par chimisorption sur iode

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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 6978-1 was prepared by Technical Committee ISO/TC 193, *Natural gas*, Subcommittee SC 1, *Analysis of natural gas*.

This first edition of ISO 6978-1, together with ISO 6978-2, cancels and replaces ISO 6978:1992, which has been technically revised.

ISO 6978 consists of the following parts, under the general title *Natural gas — Determination of mercury*:

— *Part 1: Sampling of mercury by chemisorption on iodine*

— *Part 2: Sampling of mercury by amalgamation on gold/platinum alloy*

Introduction

Natural gases may contain considerable amounts of mercury, which are generally present in the elemental form. Gases with high mercury content have to be purified to avoid the condensation of mercury during processing and transport as well as to be compliant with the demands of gas sales contracts. Low mercury concentrations are specified when natural gas is to be liquefied. This is to avoid severe corrosion problems, for instance in aluminium heat exchangers of liquefaction plants.

Since the presence of hydrocarbons, in particular aromatic hydrocarbons present in low concentrations in almost every natural gas, interferes in the determination of mercury by atomic absorption spectrometry (AAS) or atomic fluorescence spectrometry (AFS), mercury cannot be determined directly in natural gas. Therefore, prior to the analytical determination, mercury has to be collected and separated from aromatic hydrocarbons.

The purpose of the determination of the mercury content can be

— to monitor gas quality,

— to monitor the operation of gas treatment plants for mercury removal.

Several methods for the collection or enrichment of mercury from natural gas have been developed. The collection of mercury from dry natural gas normally poses no particular problem. However, care should be taken when sampling mercury from natural gases under nearly condensing conditions (see ISO 6570).

The two parts of ISO 6978 describe the principles of sampling and specify the general requirements for methods for sampling mercury and for determining total mercury in pipeline quality natural gas. This part of ISO 6978 specifies a method of sampling mercury by chemisorption on iodine-impregnated silica gel whereas Part 2 specifies a method of sampling mercury by amalgamation on gold/platinum alloy thread.

Natural gas — Determination of mercury —

Part 1:
Sampling of mercury by chemisorption on iodine

WARNING — The use of this part of ISO 6978 may involve hazardous materials, operations and equipment. This part of ISO 6978 does not purport to address all of the safety problems, associated with its use. It is the responsibility of the user of this part of ISO 6978 to establish appropriate safety and health practices and to determine the applicability or regulatory limitations prior to use.

1 Scope

This part of ISO 6978 specifies a method for the determination of total mercury content in natural gas using a sampling method at pressures up to 40 MPa by chemisorption on iodine-impregnated silica gel. This sampling method is suitable for the determination of mercury contents within the range of 0,1 µg/m^3 to 5 000 µg/m^3 in natural gas. This method is applicable to sampled gas volumes containing less than 20 mg hydrogen sulfide (absolute content) and less than a total liquid hydrocarbon condensate of 10 g/m^3 under the sampling conditions. The collected mercury is determined by measuring the absorbance or fluorescence of mercury vapour at 253,7 nm.

NOTE ISO 6978-2 gives a sampling method suitable for the determination of mercury content of pipeline natural gas by amalgamation of mercury on gold/platinum alloy thread at atmospheric pressure for the range of mercury from 0,01 µg/m^3 to 100 µg/m^3 and for sampling at high pressure (up to 8 MPa) from 0,001 µg/m^3 to 1 µg/m^3.

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.

ISO 6570, *Natural gas — Determination of potential hydrocarbon liquid content — Gravimetric methods*

ISO 10715, *Natural gas — Sampling guidelines*

ISO 14111, *Natural gas — Guidelines to traceability in analysis*

ISO 14532, *Natural gas — Vocabulary*

Guide to the expression of uncertainty in measurement (GUM), BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML

AISI Steel Code[1)]

1) Available from the American Iron and Steel Institute, c/o James McCarl, 131 Huron Drive, Carnegie, PA 15106, USA.

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 14532 apply.

4 Principle

The gas is passed through a glass tube containing silica gel impregnated with iodine. Mercury (Hg) present in the gas as elemental mercury or organic mercury compounds [e.g. dimethyl mercury, $Hg(CH_3)_2$, or diethyl mercury, $Hg(C_2H_5)_2$] is chemisorbed:

$$Hg + I_2 \rightarrow HgI_2$$

$$Hg(CH_3)_2 + 2\ I_2 \rightarrow HgI_2 + 2\ CH_3I$$

The mercury iodide (HgI_2) formed is then dissolved in an ammonium iodide/iodine solution (NH_4I/I_2) in the laboratory. Hydrocarbon condensates are removed by vacuum stripping at this stage. Mercury, present in the form of a water-soluble complex, is determined by reducing aliquot portions of this solution with alkaline tin(II) solution to elemental mercury. The mercury is stripped from the solution by an inert gas and the mercury vapour is transferred to a cold vapour atomic absorption spectrometer (AAS) or atomic fluorescence spectrometer (AFS) for measurement at 253,7 nm. Calibration for the final mercury determination is performed in the same way using an aqueous mercury standard solution which is matrix-matched to the sample.

Unless otherwise specified, gas volumes are expressed in cubic metres (m^3) at 273,15 K and 1 013,25 hPa.

5 Apparatus

The parameters influencing the measurement shall be traceable to national or International Standards. The uncertainty of the volume measurement (volume, temperature and pressure of the gas as well as ambient air pressure) directly contributes to the uncertainty of the determined mercury content of the gas. Therefore suitable measuring equipment, calibrated against a commonly accepted reference, shall be used to minimize the uncertainty of the volume measurement to less than 1 %.

5.1 Sampling apparatus [see Figure 1 a) for sampling up to 10 MPa and Figure 1 b) for up to 40 MPa], comprising the following:

5.1.1 High-pressure sampling cell (see Figure 1 with details given in Figure 2), made of NiMo16Cr15W (material no. 2.4819 in accordance with the Steel Code), which includes a temperature gauge and a pressure gauge and the following components.

5.1.1.1 Acid-washed glass wool.

5.1.1.2 Sampling tube (see Figure 1 with details given in Figure 3), made of borosilicate glass and fitted with a screw cap.

Fit the sampling tube (Figure 3) first with a layer, 0,5 cm thick, of acid-washed glass wool (5.1.1.1) to retain the absorbent. Then fill the tube with 4,00 g of iodine-laden silica gel (6.2.3) to obtain a 5 cm absorption layer. Then add another layer, 0,5 cm thick, of glass wool (5.1.1.1) to retain the top of the absorbent layer. Close the tube with a screw cap.

5.1.2 Temperature gauges or **thermometers** (see Figure 1 with details given in Figure 2).

5.1.3 Pressure gauges (see Figure 1 with details given in Figure 2).

5.1.4 Pressure relief valves (see Figure 1) which are either

a) **electrically heated,** for sampling pressure up to 10 MPa [see Figure 1 a)], or

b) **coupled with a heat exchanger** (5.1.5), for sampling pressure up to 40 MPa [see Figure 1 b)].

When positioned in a hazardous area, the electrically heated pressure relief valve shall comply with local safety regulations.

5.1.5 Heat exchanger [see Figure 1 b)], with sufficient capacity to heat sample and bypass gas to 90 °C.

When positioned in a hazardous area, the heat exchanger shall comply with local safety regulations.

5.1.6 Gas meter (see Figure 1), equipped with a thermometer (5.1.2) and a pressure gauge (5.1.3).

5.1.7 Barometer, required for measuring the ambient air pressure.

5.2 Glass apparatus for dissolution (see Figure 4), for the dissolution of mercury iodide and consisting of the following.

5.2.1 Reservoir, for the solution.

5.2.2 Graduated receiver.

5.2.3 Stop cocks.

5.2.4 Three-way valve.

5.3 Glass apparatus for charging of silica gel with iodine (see Figure 5), comprising the following.

5.3.1 Round bottom flask, of 2 l capacity.

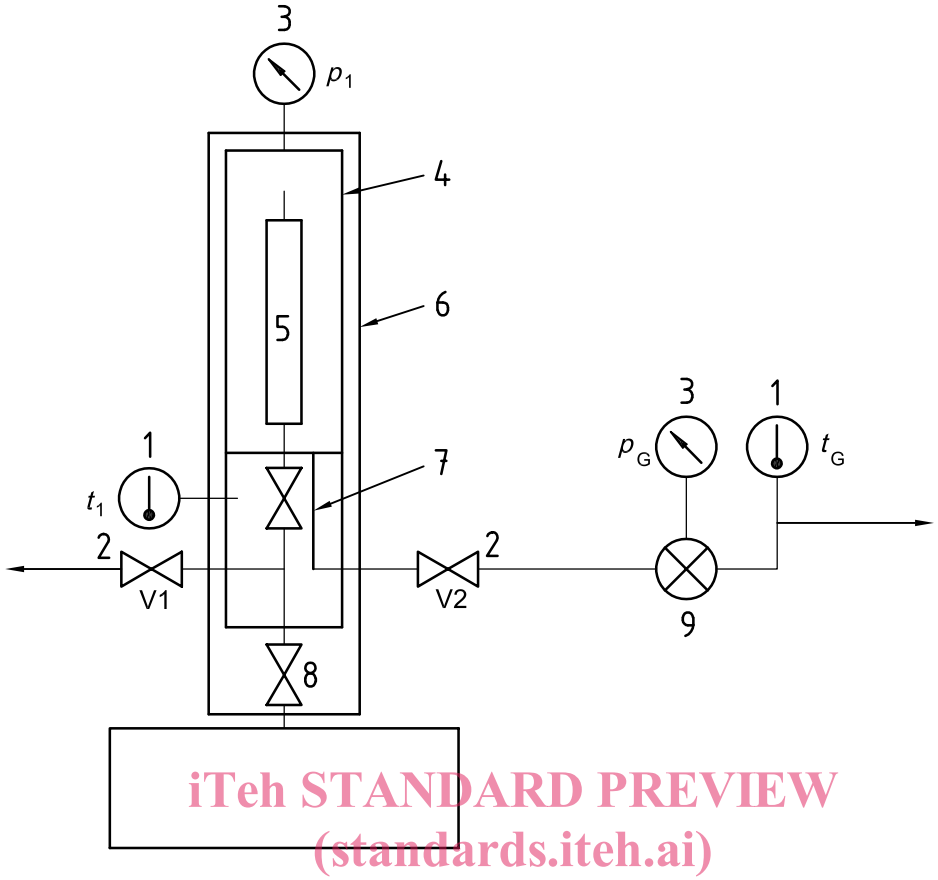
5.3.2 Gas inlet tube.

5.3.3 Air filter, filled with a suitable absorbent, such as sulfur-impregnated activated charcoal, to remove mercury from the air.

5.4 Cold vapour atomic absorption spectrometer (AAS) or **atomic fluorescence spectrometer** (AFS).

A cold vapour technique AAS with background correction capability or an AFS with a mercury unit, capable of detecting at least 1 ng Hg, standard laboratory equipment and polyvinyl acetate (PVA) hoses are required. However, hoses of other suitable plastic material, such as polytetrafluoroethylene (PTFE) or polyamide (PA), may be used.

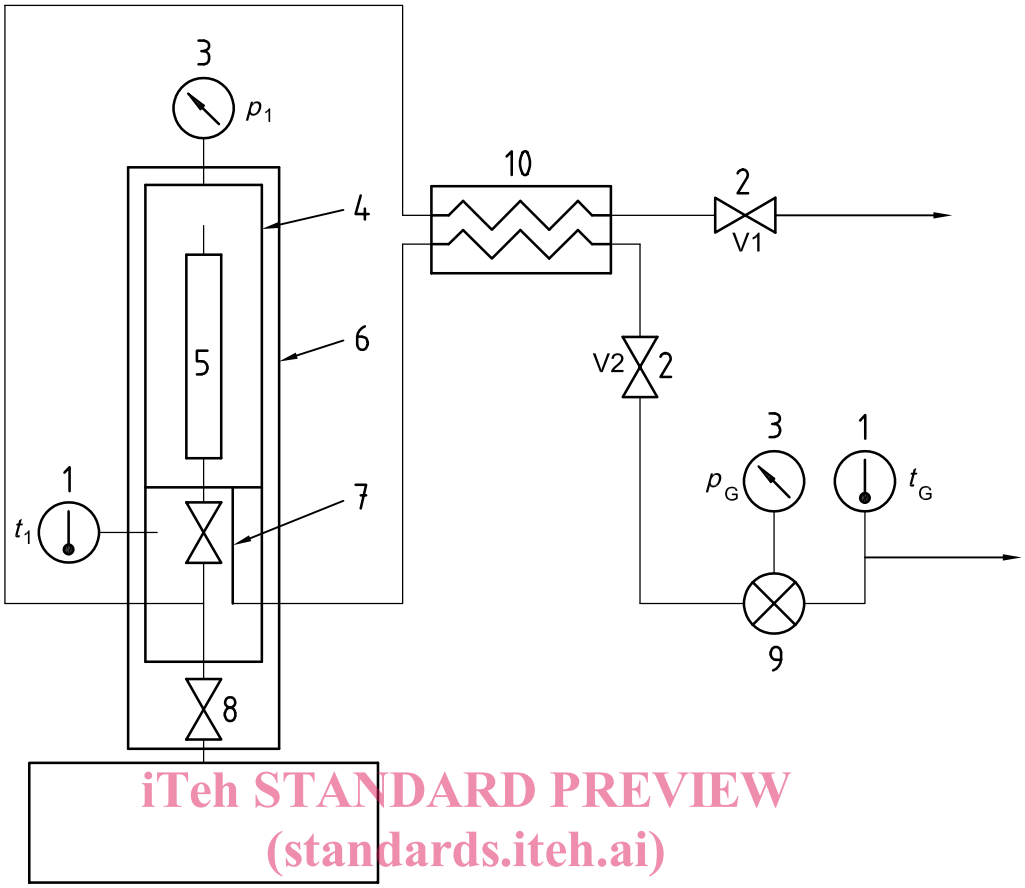
It is essential to make sure that the flowrate transporting the mercury through the analytical system remains constant.



a) Apparatus for pressures up to 10 MPa

Key

1 temperature gauges (t_1, t_G)
2 pressure relief valves (V1, V2)
3 pressure gauges (p_1, p_G)
4 high-pressure sampling cell
5 sampling tube
6 insulation
7 cell valve
8 sampling valve
9 gas meter



b) Apparatus for pressures up to 40 MPa

Key

1	temperature gauges (t_1, t_G)	6	insulation
2	pressure relief valves (V1, V2)	7	cell valve
3	pressure gauges (p_1, p_G)	8	sampling valve
4	high-pressure sampling cell	9	gas meter
5	sampling tube	10	heat exchanger

NOTE See 7.2.

Figure 1 — High pressure sampling apparatus