INTERNATIONAL STANDARD

ISO 6978-2

First edition
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Natural gas — Determination of mercury —

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

Gaz naturel — Détermination de la teneur en mercure —

Partie 2: Échantillonnage du mercure par amalgamation sur alliage or/platine

Reference number
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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 6978-2 was prepared by Technical Committee ISO/TC 193, *Natural gas*, Subcommittee SC 1, *Analysis of natural gas*.

This first edition of ISO 6978-2, together with ISO 6978-1, 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 amalgamation on gold/platinum alloy thread whereas Part 1 specifies a method of sampling mercury by chemisorption on iodine-impregnated silica gel.

Natural gas — Determination of mercury —

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

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 of pipeline quality natural gas using a sampling method by amalgamation on gold/platinum (Au/Pt) alloy thread. This method is applicable to the sampling of raw natural gas when no condensation is present. At atmospheric pressure, this method is suitable for the determination of mercury content within the range of 0,01 µg/m^3 to 100 µg/m^3 in natural gas samples. At higher pressures (up to 8 MPa), this sampling method is suitable for the determination of mercury contents within the range of 0,001 µg/m^3 to 1 µg/m^3. The collected mercury is determined by measuring the absorbance or fluorescence of mercury vapour at 253,7 nm.

NOTE ISO 6978-1 gives a sampling method suitable for the determination of mercury contents in natural gas by chemisorption on iodine-impregnated silica gel for the working range of 0,1 µg/m^3 to 5 000 µg/m^3 for sampling at pressures up to 40 MPa.

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 10715, *Natural gas — Sampling guidelines*

ISO 14532, *Natural gas — Vocabulary*

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

3 Terms and definitions

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

4 Principle

Sampling is performed at a temperature at least 10 °C higher than the dewpoint of the gas sampled. The gas is passed through two quartz glass sampling tubes in series containing fine gold/platinum alloy thread. The mercury is collected on the gold/platinum alloy thread by amalgamation. Subsequently, each sampling tube is separately heated to 700 °C to desorb the mercury from the amalgam. The released mercury is transferred by

a stream of air onto an analytical tube filled with gold/platinum alloy thread (double amalgamation). This second quartz glass tube is then heated to 800 °C and the mercury is transferred to an AAS or AFS for measurement at 253,7 mm.

This sampling method is suitable for the determination of mercury content in natural gas in the range of 0,01 µg/m^3 to 100 µg/m^3 for sampling at atmospheric pressure and 0,001 µg/m^3 to 1 µg/m^3 for sampling at high pressure. To avoid diffusion of mercury from the surface into the gold/platinum alloy thread, which would reduce the recovery of mercury under the specified transfer conditions, it is necessary to determine the amount of collected mercury within one week after sampling.

Other sorption materials such as gold-impregnated silica with a high specific surface may be used instead of fine gold/platinum alloy thread, provided they show equivalent method performance in the natural gas matrix.

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

NOTE The comparability of the two sampling techniques has been demonstrated by interlaboratory tests at two different concentration levels.

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).

5.1.1 Apparatus for sampling at atmospheric pressure, typically comprising the following:

5.1.1.1 Heated bypass valve.

5.1.1.2 Heated flow control valve (needle type).

5.1.1.3 Three-way valve, for the second bypass.

5.1.1.4 Aluminium block, capable of being heated to ⩽ 100 °C (see Figure 2) and dividable into two halves with a central bore coated with a layer (about 2 mm) of silicon rubber for keeping the quartz glass sampling tubes filled with gold/platinum alloy thread (see Figure 3) during sampling at elevated temperature (if necessary) and equipped with a temperature gauge (not shown in Figure 1).

5.1.1.5 Flow meters (three):

— one for flowrates ⩽ 50 l/min;

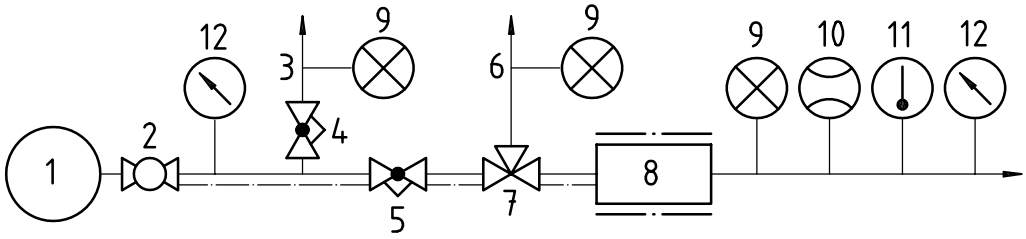
— two for flowrates ⩽ 5 l/min.

5.1.1.6 Gas meter, suitable for measuring flowrates of ⩽ 5 l/min, capable of allowing adjustment of the bypass flow and the volume measurement and equipped with the following:

a) **pressure gauge**;

b) **temperature gauge**, for measuring temperatures between 0 °C to 40 °C.

5.1.1.7 Barometer, required for measuring the ambient air pressure.

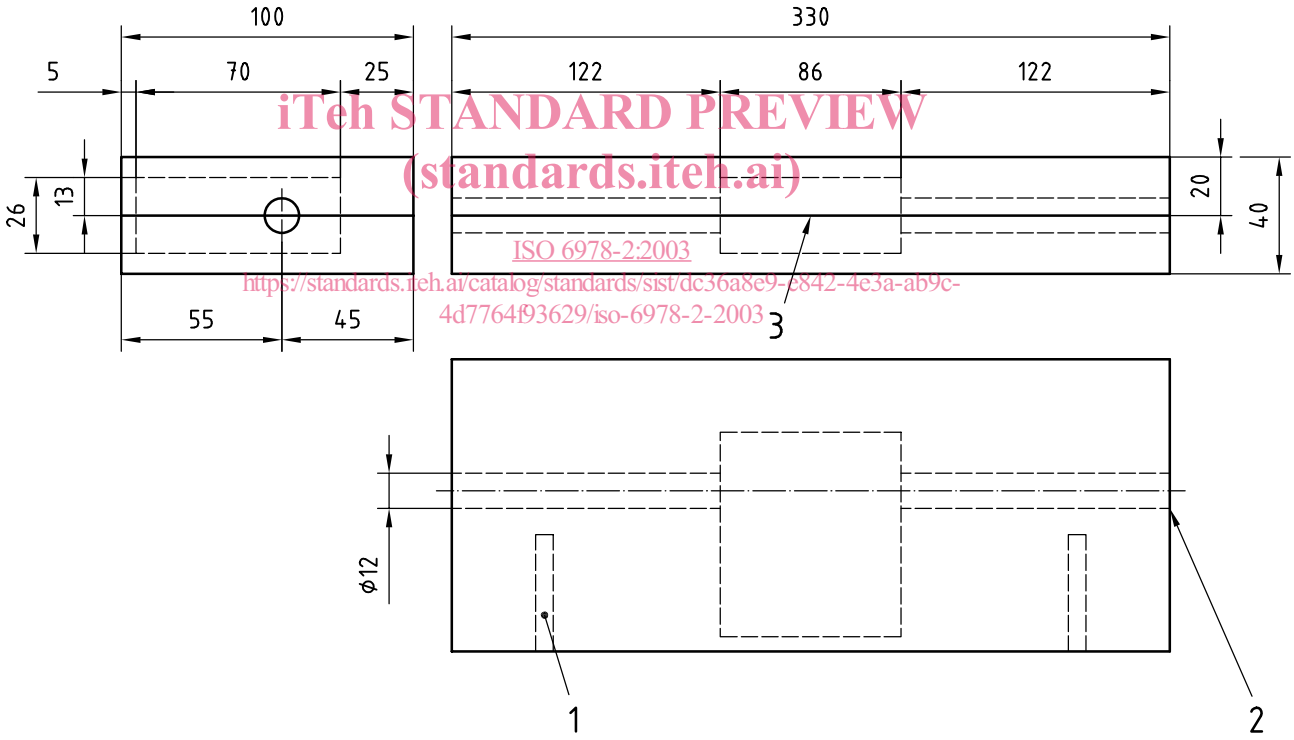


Key

1 pipeline
2 sampling valve
3 first bypass
4 bypass valve
5 flow control valve
6 second bypass
7 three-way valve
8 aluminium block for heating
9 flow indicator
10 gas meter
11 temperature gauge
12 pressure gauge

Figure 1 — Sampling apparatus

Dimensions in millimetres

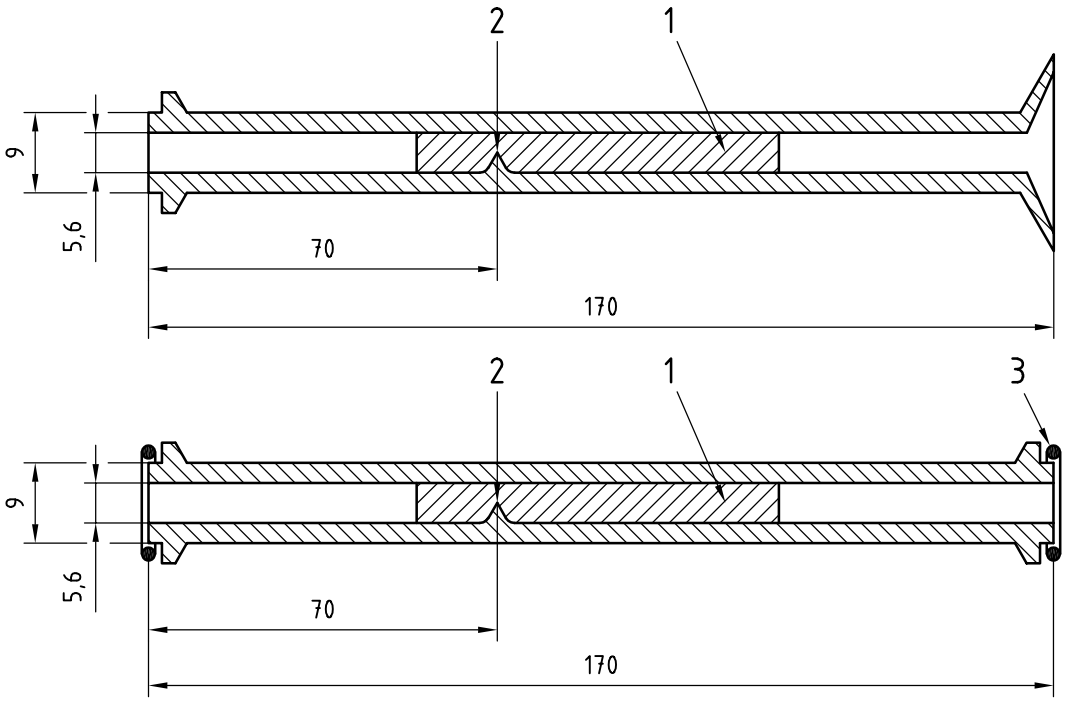


Key

1 heating plug
2 silicon rubber (glued)
3 division line

Figure 2 — Heating box (heatable aluminium box)

Dimensions in millimetres



Key

1 gold/platinum wire (350 Au/150 Pt alloy)
2 indentation
3 O-ring

Figure 3 — Quartz glass sampling and analytical tubes

5.1.2 Apparatus for sampling at high pressure (see Figure 4), comprising the following:

5.1.2.1 Pressure gauge, suitable for measuring pressures from 0 MPa to 25 MPa.

5.1.2.2 Valve.

5.1.2.3 Bypass valve.

5.1.2.4 Pressure-reducing valves (two).

5.1.2.5 Three-way valve.

5.1.2.6 Pressure relief valves (two), set at pressures of 10 MPa and 4 kPa, respectively, to protect the high-pressure vessel and the gas meter against over pressure.

5.1.2.7 Pressure gauge, suitable for measuring pressures from 0 MPa to 10 MPa in the high-pressure vessel.

5.1.2.8 Flow indicator, for adjusting the gas flow through the high-pressure vessel.

5.1.2.9 Heating tape, for wrapping around the assembly except for the relieve valves and the flow indicator.

5.1.2.10 Gas meter, for measuring sample volumes and suitable for a gas flowrates of $\leqslant$ 50 l/min and equipped with the following:

a) **pressure gauge**;

b) **temperature gauge**, for measuring temperatures between 0 °C to 40 °C.

5.1.2.11 **Barometer**, required for measuring the ambient air pressure.

5.1.2.12 **High-pressure vessel** (see detail in Figure 4 for the construction), of which all parts consist of stainless steel.

The high-pressure sampling apparatus specified in ISO 6978-1 may also be used by adapting it for two sampling tubes. The high-pressure apparatus may also be used for sampling at atmospheric pressure.

5.1.2.13 **Connections**, between the inlet of the vessel and the sampling tubes consisting of a ball socket with screw cap and dividable screw.

5.2 **Desorption station** (see Figure 5), comprising the following:

5.2.1 **Tube oven**, for the thermal desorption of mercury from the quartz glass sampling tubes or quartz glass analytical tubes.

The length of the heating zone of the oven should be (120 ± 20) mm to cover the part of the tubes filled with Au/Pt-alloy thread. The inner diameter shall allow the free passage of the tube sockets. The heating capacity of the oven shall be capable of reaching 800 °C within ⩽ 2 min.

5.2.2 **Quartz glass analytical tubes** (see Figure 3).

5.2.3 **Mercury trap** (see Figure 5), filled with sulfur impregnated activated charcoal or any other suitable mercury adsorbent such as gold/platinum alloy.

5.2.4 **Air pump**, capable of delivering a flow rate between 0,5 l/min and 2 l/min.

5.2.5 **Flow meter**, for measuring flow rates ranging between 1 l/min and 5 l/min.

5.2.6 **Polyvinyl acetate** (PVA) **tubing**, having an internal diameter of 3 mm.

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

A cold vapour AAS or an AFS with an integrator and a mercury unit, capable of detecting at least 0,05 ng Hg, standard laboratory equipment and polyvinyl acetate (PVA) tubing 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.

5.4 **Calibration set** (see Figure 6), for the preparation of mercury-saturated air (6.6), consisting of the elements given in 5.4.1 to 5.4.7.

5.4.1 **Bottles**, of 500 ml capacity.

5.4.2 **Screw caps with polytetrafluoroethylene (PTFE)-coated silicone rubber septa**.

5.4.3 **PVA tubing**.

5.4.4 **Stainless steel syringe needles**.

5.4.5 **Thermometer**, with a range from 10 °C to 40 °C and graduated to 0,1 °C.

5.4.6 **Insulated box**.

5.4.7 **Gastight glass syringe**, equipped with a **PTFE plunger,** with a **stainless steel needle,** capable of delivering a volume of 0,5 ml to 5 ml.

5.5 **Sampling and analytical tubes** (see Figure 3).