INTERNATIONAL STANDARD

ISO 6978-1

First edition
2003-10-15

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

Reference number
ISO 6978-1:2003(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

© ISO 2003

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS

Not for Resale

Contents

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.

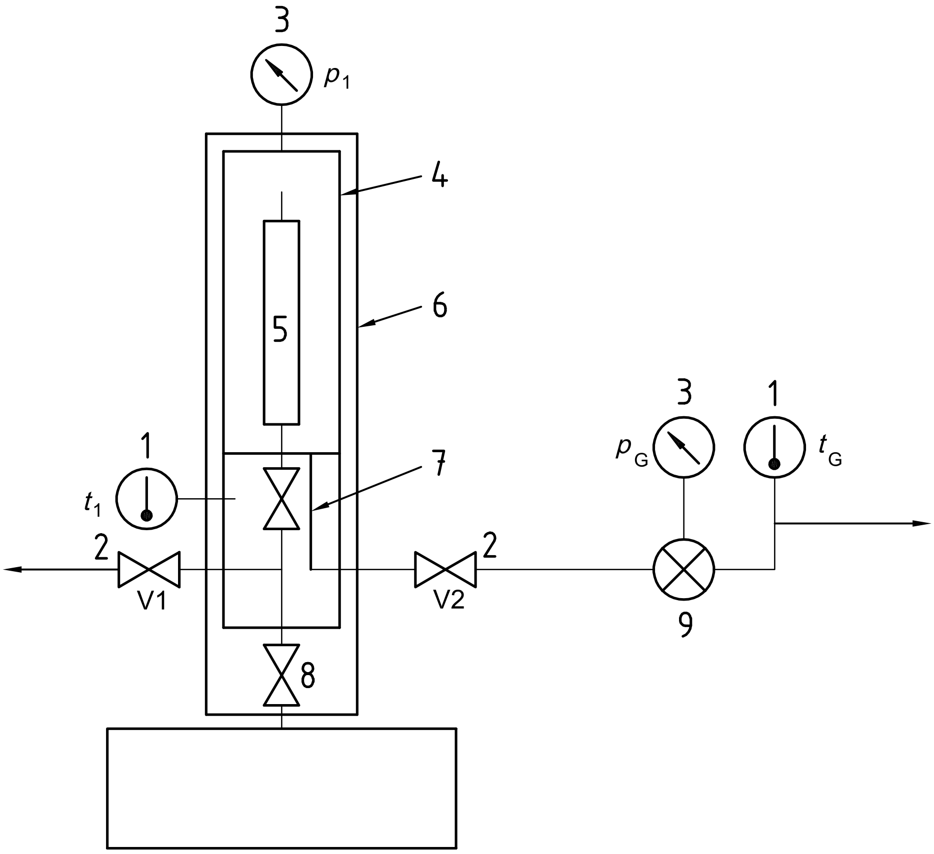
5.3.2 Gas inlet tube.

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.

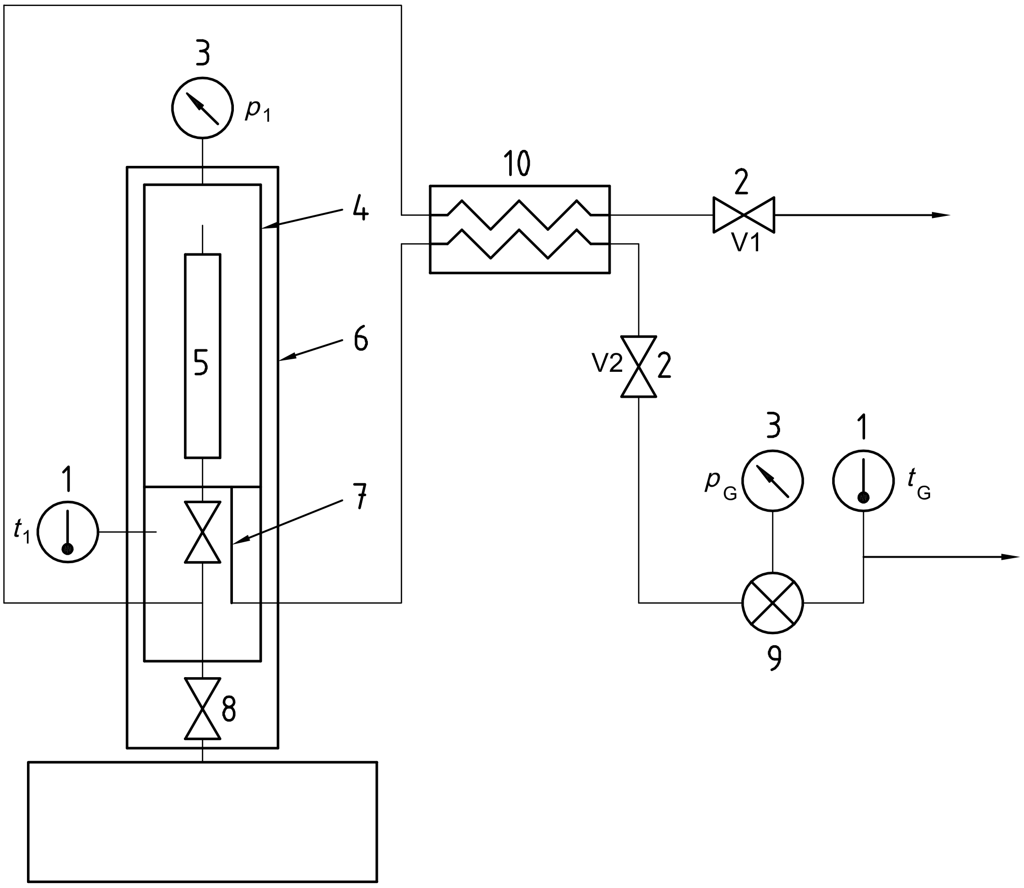
It is essential to make sure that the flowrate transporting the mercury through the analytical system remains constant.



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

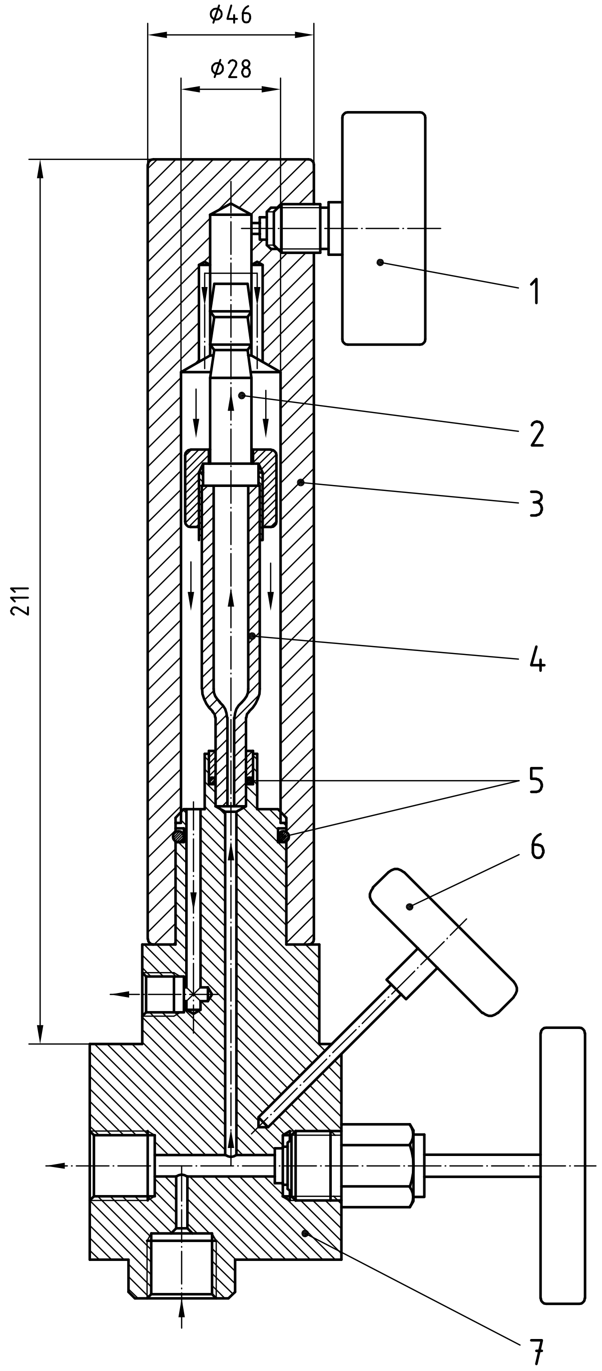
Key

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

Dimensions in millimetres



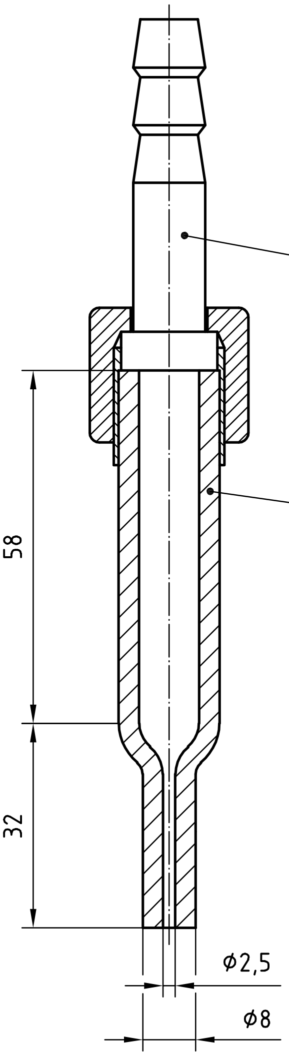
Key

1 pressure gauge
2 connection tube
3 high-pressure cell body
4 sampling tube
5 O-ring
6 temperature gauge
7 bottom part of cell with integrated valve

NOTE See 7.2.

Figure 2 — High-pressure sampling cell

Dimensions in millimetres



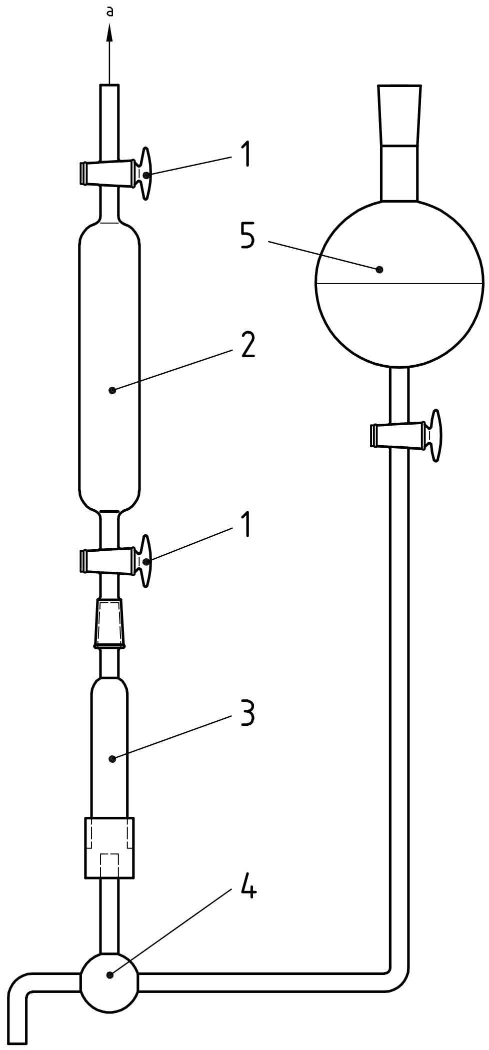
Key

1 connection tube

2 sampling tube

NOTE See 7.2.

Figure 3 — Sampling tube (borosilicate glass)



Key

1 stop cock

2 graduated receiver

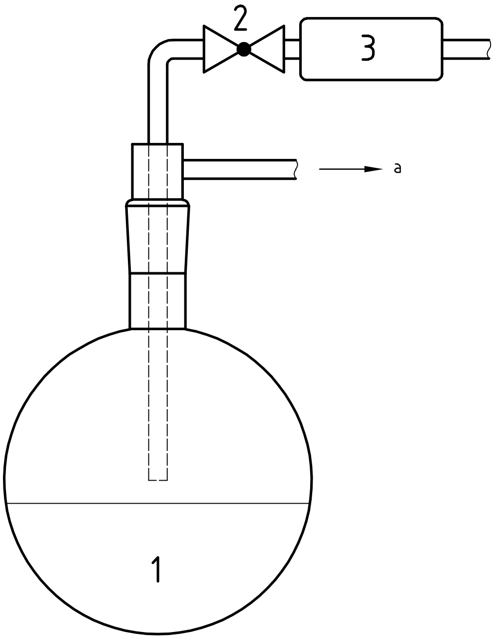
3 sampling tube

4 three-way valve

5 round-bottom reservoir

[a] To vacuum.

Figure 4 — Glass apparatus for dissolution



Key

1 round-bottom flask (2 l)
2 glass valve
3 air filter, filled with activated carbon

a To vacuum.

Figure 5 — Glass apparatus for charging silica gel with iodine

6 Reagents

6.1 **Water**, distilled, containing less than 1 µg/l of Hg.

6.2 **Reagents for chemisorption**.

6.2.1 **Iodine** (I_2).

6.2.2 ***n*-Pentane**.

6.2.3 **Iodine-laden silica gel**, having a grain size of 0,2 mm to 0,5 mm and prepared as follows.

In a 2-l round bottom flask (5.3.1), dissolve 12 g of iodine (6.2.1) in 1 l of *n*-pentane (6.2.2). To this solution, add 0,5 kg silica gel in portions under swirling. After a reaction time of some 30 min, fit the flask with the assembly shown in Figure 5 featuring an air filter (5.3.3), filled with sulfur-impregnated activated charcoal or another suitable absorbent material. By drawing air through the filter and flask and occasionally swirling the flask contents, gradually evaporate the *n*-pentane. Finally, evaporate the remaining *n*-pentane at a reduced pressure of approximately 30 hPa by closing the glass valve for a short time. Do not remove *n*-pentane completely so as to avoid evaporating the iodine. Keep the I_2-laden silica gel in a brown ground-glass container to protect it against light. It then has a shelf life of more than 6 months.

6.3. Reagents for dissolution.

6.3.1 Iodine (I_2).

6.3.2 Ammonium iodide (NH_4I).

6.3.3 Ammonium iodide solution, $c(NH_4I)$ = 1 mol/l.

6.3.4 Ammonium iodide/iodine (NH_4I/I_2) **reagent solution**, prepared by first dissolving 10 g of iodine in 1 l of the NH_4I solution (6.3.3), then diluting this solution tenfold with water (6.1).

Keep both the NH_4I/I_2 reagent solution and the NH_4I solution in brown flasks to protect them against light. Both solutions then have a shelf life of more than 6 months.

6.4 Reagents for reduction solution.

6.4.1 Potassium hydroxide (KOH) **solution**, 15 % by mass.

6.4.2 Tin(II) chloride ($SnCl_2$).

6.4.3 Reduction solution, prepared by dissolving, in portions and stirring, 5 g of tin(II) chloride (6.4.2) into 95 g of the potassium hydroxide solution (6.4.1).

This solution may have to be filtered and has a maximum shelf life of two days.

6.5 Calibration solutions.

6.5.1 Mercury stock solution, mass concentration $\rho(Hg)$ = 1,000 g/l, traceable to a national or international primary standard in accordance with ISO 14111.

6.5.2 Mercury calibration solution, prepared by diluting the mercury stock solution (6.5.1) with the NH_4I/I_2 solution (6.3.4) to an appropriate mass concentration suitable for the working range of the instrumentation.

The mercury calibration solution, when kept in brown flasks, will be stable for more than one year.

7 Sampling

DANGER — Mercury presents a health hazard if incorrectly handled. Avoid inhalation of the vapour.

SAFETY PRECAUTIONS — Sampling shall follow all relevant safety regulations in accordance with ISO 10715. All the equipment used should comply with the specific local safety regulations.

7.1 General

7.1.1 Precautions

In natural gas, mercury is found at extremely low concentration levels. Consequently, to determine mercury at such low levels, it is necessary to take certain precautions in order to obtain reliable results. Such trace analysis is strongly affected by the following:

a) the sorption of mercury by the sampling equipment, resulting in false, lower levels of mercury;

b) background mercury contamination within the equipment and chemicals or of the air in the laboratory, resulting in false, higher levels of mercury.

Therefore, it is necessary to check all the equipment and chemicals used in the method prior to use.

Mercury is easily sorbed by most materials commonly available for sampling. Therefore, to obtain acceptable results, use quartz glass, borosilicate glass and stainless steel. Depending on the length of the lines, the material used and the flowrate, preconditioning of the sampling system is recommended. For the transfer of mercury vapour to the spectrometer, polyvinyl acetate (PVA) hose connections are recommended. However, other suitable plastics, for instance polytetrafluoroethylene (PTFE) or polyamide (PA), may be used.

7.1.2 Conditions for representative sampling

For general aspects of natural gas sampling, see ISO 10715.

Due to the sorption phenomena mentioned in 7.1.1, only perform direct sampling.

Purge the sampling system (sample probe and sampling valve) prior to sampling so as to remove any deposits or impurities. Avoid any excessive purging because it can possibly cause the sampling valve to freeze as a result of gas expansion (Joule-Thomson effect).

The sampling and transfer lines should be as short as possible.

It is strongly recommended to use a fast loop to bypass the sorption tube. This guarantees high gas velocity in the sampling system (sample probe and sampling valve) and minimizes any sorption phenomena.

7.1.3 Sampling at high pressure

High-pressure sampling (above atmospheric pressure) allows a rather large volume of gas to be passed through the sampling tube in a short time without changing the pressure and temperature of the gas. This may also prevent retrograde condensation of hydrocarbons. Heat exchangers are required to heat the gas prior to pressure reduction so that the gas sample volume can be measured accurately; the bypass should be reintroduced either to a low-pressure pipeline or to a flared and/or vented outlet for safety reasons.

7.1.4 Applicability

This sampling method is suitable for the determination of mercury content in the range of 0,1 µg/m^3 to 5 000 µg/m^3 in natural gas. The sampled gas volume shall contain less than 20 mg hydrogen sulfide (H_2S) (absolute content) and less than 10 g/m^3 liquid hydrocarbon condensate determined in accordance with ISO 6570 under the sampling conditions.

7.2 Sampling procedure (see Figures 1 and 2)

Carry out direct sampling (i.e. discontinuously) in accordance with ISO 10715 at operating pressure.

Open the sampling valve (see Figure 1) to blow out any deposits or impurities. Take care to prevent excessive blowing out, otherwise the Joule-Thomson effect can cause the valve to ice up.

Keep the sample line to the high-pressure sampling cell (see Figure 1) as short as possible to prevent losses by adsorption on the inside surface of the sampling line as well as by a temperature drop.

Connect the bottom part of the high-pressure sampling cell (see Figure 2) in such a way so as to fix it in a vertical position. If tube connections are required, use stainless steel tubing.

After replacing the screw cap on the sampling tube (see Figure 3) by a connecting tube, insert the sampling tube into the bottom part of the cell (see Figure 2), where it is sealed by an O-ring. Screw the high-pressure cell body (see Figure 2) onto the bottom part and tighten it by hand (O-ring seal).

To prevent condensation and icing-up of the pressure relief valves (see Figure 1), heat the pressure relief valve [for sampling pressure up to 10 MPa, see Figure 1 a)], or heat the gas using a heat exchanger [see Figure 1 b)] upstream to the pressure relief valves, which are connected by two lines (NiMo16Cr15W steel, material No. 2.4819 as specified by the Steel Code, or PTFE-coated high-pressure hoses) to the high-pressure sampling cell [for sampling pressure up to 40 MPa, see Figure 1 b)].

When the temperature of the heated relief valves has reached approximately 50 °C, or the temperature t_2 of the heat exchanger has reached approximately 90 °C, gradually open the sampling valve and the pressure relief valve V1 (see Figure 1). Depending on the diameter and length of the sampling line and the upstream pressure, adjust the volumetric flow from the system through the bypass to 10 m^3/h.

The sampling nozzle with the sampling valve (see Figure 1) and the bottom part of the cell (see Figure 2) become heated to the temperature of the gas. Measure the temperature t_1 using the temperature gauge (see Figure 1). When a constant temperature has been reached (i.e. the measured drift is less than 2 K/10 min), begin sampling.

Pressurize the sampling cell, by gradually opening the cell valve (see Figure 1). Take care not to open the valve too quickly, as this may cause the sampling tube to burst.

Then check the system for tightness by performing a bubble leak test. Replace the O-ring in the bottom part of the cell, if it leaks. If the gas temperature is higher than the ambient temperature during sampling, insulate the entire high-pressure sampling cell and the sampling valve including the sampling nozzle with adequate insulation material.

It is recommended to carry out the procedure once before sampling begins to pre-heat the sampling cell. Discard the sampling tube used for this purpose.

Take a pressure reading p_1 at the pressure gauge (see Figure 1).

Record the initial value V_i at the gas meter (see Figure 1), then adjust the pressure relief valve V2 to give the following gas flow:

- for system pressures of $p > 6{,}0$ MPa gas flow = 6 m^3/h to 8 m^3/h
- for system pressures of $p \leqslant 6{,}0$ MPa gas flow = numerical value of p in m^3/h

Take readings at the gas meter for the gas temperature t_G and the gauge pressure p_G at regular intervals. At minimum, take readings at the beginning and at the end of sampling.

At the same time, take a reading at the barometer of the ambient pressure p_a.

Once the required sampling volume (0,15 m^3 to 2 m^3) has passed through the system, gradually close the cell valve (see Figure 1) and take the final reading V_f at the gas meter.

After each sampling procedure, replace the sampling tube with a new, clean tube as quickly as possible so as to avoid heat loss.

If the absorption layer inside the sampling tube is almost fully discoloured during the sampling, i.e. the iodine has evaporated or the sampling tube is saturated, insert a new sampling tube and repeat the procedure using a smaller sample volume.

7.3 Sample treatment

Fix the sampling tube in the dissolution apparatus (see Figure 4). Evacuate the sampling tube through the upper stop cock of the graduated 50 ml receiver using a vacuum pump and keeping the three-way valve closed. After closing the lower stop cock of the graduated receiver, open the three-way valve to allow all the silica gel in the sampling tube to come into full contact with the NH_4I/I_2 solution fed from the round-bottom reservoir.

Slightly open the lower stop cock of the graduated receiver so as to draw the NH_4I/I_2 solution into the graduated receiver. Allow 50 ml of this solution to flow through the sampling tube into the receiver within approximately 10 min.

During this process, keep the upper stop cock of the graduated receiver connected to the vacuum pump open. Owing to the vacuum still present, the absorbed hydrocarbons are mostly evaporated, avoiding any adverse effect on subsequent testing.

Close both stop cocks of the graduated receiver and the three-way valve and remove the connections. Shake the receiver containing the NH_4I/I_2 solution a few times and transfer the contents to a brown sample flask.

8 Mercury determination

WARNING — Mercury presents a health hazard if incorrectly handled. Avoid inhalation of the vapour.

8.1 Transfer of mercury to AAS or AFS instrument

Perform the analytical determination using an AAS (5.4) with background correction capability or an AFS (5.4) instrument with a reduction unit. Batch, flow injection and continuous flow methods may be used as specified in the following procedure.

Pour 20 ml of a potassium hydroxide solution (6.4.1) and 20 ml of water (6.1) into the reduction receptacle of the mercury unit of the AAS or AFS instrument. Then add 10 µL to 1 000 µL of the sample solution. Ensure the mercury content remains within the linear range of the AAS or AFS. Dilute samples with a higher mercury content with the NH_4I/I_2 solution (6.3.4).

Add 1,25 ml of the reduction solution (6.4.3) in order to reduce the mercury complex to elemental mercury. Strip off the mercury with inert gas and use the AAS or AFS instrument for detection. When using the flow injection and continuous flow system, use the same reduction solution. The presence of condensate in the solution may influence the mercury determination unless background correction is used with AAS.

8.2 Calibration

Calibrate the integrator of the AAS or AFS instrument by analysing known amounts of mercury calibration solution (6.5.2) in the same way as described in 8.1.

Repeat this procedure until the relative standard deviation for three consecutive measurements is less than 3 %. At least two calibrations at different levels within the measuring range are necessary for a first order calibration curve passing through the origin and at least three calibrations for curves not passing through the origin.

The appropriate response factor for the measured signal of the sample is derived from the calibration curve.

The measured signal for the mercury calibration solution may be used to construct control charts for the purpose of indicating whether the equipment and the method are working satisfactorily.

8.3 Blank test

Reagents used can contain traces of mercury. Correct the values of the sample by subtracting a blank from the determined total mercury. Check the blank value for each new batch of reagents used. Use the same procedure as described in 7.3 and 8.1 using at least three unused sampling tubes. The blank should contain less than 1 µg/l of Hg in the final solution used for the determination specified in 8.1.

9 Calculation

Calculate the mass, expressed in nanograms, of mercury, m_{Hg}, collected on the sampling tube using Equation (1):

$$m_{\mathrm{Hg}} = \frac{A \times V_1}{R_{\mathrm{f}} \times V_2} \tag{1}$$

where

A is the integrated signal, expressed in arbitrary units, indicated by the AAS or AFS;

R_{f} is the response factor, expressed in arbitrary units per nanogram, derived from the calibration curve;

V_1 is the volume, expressed in millilitres, of the total sample solution;

V_2 is the volume, expressed in millilitres, of the measured sample solution.

Correct the mass of mercury for reagent contribution using Equation (2):

$$m_{\mathrm{Hg,corr}} = m_{\mathrm{Hg}} - m_{\mathrm{blank}} \tag{2}$$

where

$m_{\mathrm{Hg,corr}}$ is the corrected mass , expressed in nanograms, of mercury in the sample;

m_{blank} is the mass, expressed in nanograms, of mercury in the blank, calculated according to Equation (1).

Calculate the gas volume sampled, expressed in litres, V_{G}, at 273,15 K and 1 013,25 hPa using Equation (3):

$$V_{\mathrm{G}} = \frac{\left(V_{\mathrm{f}} - V_{\mathrm{i}}\right) \times \left(p_{\mathrm{a}} + p_{\mathrm{G}}\right) T_{\mathrm{N}}}{p_{\mathrm{N}}\left(T_{\mathrm{N}} + t_{\mathrm{G}}\right)} \tag{3}$$

where

V_{f} is the final volume reading, expressed in litres, of the gas meter;

V_{i} is the initial volume reading, expressed in litres, of the gas meter;

p_{a} is the ambient air pressure, expressed in hectopascals;

p_{G} is the gauge pressure, expressed in hectopascals, of the gas meter (averaged for duration of sampling);

t_{G} is the gas temperature, expressed in degrees Celsius, inside the gas meter (averaged for duration of sampling);

T_{N} is the normal temperature equal to 273,15 K;

p_{N} is the normal pressure equal to 1 013,25 hPa.

Calculate the mercury content as mass concentration, β_{Hg}, expressed in nanograms per litre (ng/l), and equal to micrograms per cubic metre (µg/m^3) at normal conditions according to Equation (4):

$$\beta_{\mathrm{Hg}} = \frac{m_{\mathrm{Hg\ corr}}}{V_{\mathrm{G}}} \tag{4}$$

10 Precision

10.1 General

The precision of the method, as obtained by statistical examination of interlaboratory test results, is as follows.

10.2 Repeatability and reproducibility

10.2.1 Repeatability

Little statistical data is available. However, it is expected that replicate test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test gas would be expected to be ± 3 % (95 % confidence limits).

10.2.2 Reproducibility

Little statistical data is available. However, it is expected that two single and independent replicate results, obtained by different operators working in different laboratories on identical test gas, under normal and correct operation of the test method, would be expected to be ± 7 % (95 % confidence limits).

10.3 Uncertainty

The uncertainty based on interlaboratory comparison of measurements of the amount of mercury in the sample solution is typically better than ± 20 %, including both bias and uncertainty of measurement.

11 Test report

The test report shall include the following information:

- a reference to this part of ISO 6978, i.e. ISO 6978-1.
- the date and time of sampling;
- the place where the sample was taken;
- the conditions of the sampled gas, e.g. temperature, pressure, throughput;
- the sampling method;
- the analytical method (AAS or AFS instrument);
- the total mercury content, expressed in micrograms per cubic metre (µg/m^3), with calculated uncertainty in accordance with the GUM;
- the calibration data;
- the corresponding blank measurement;
- any unusual features noted during sampling and determination.

Bibliography

[1] ISO 6978-2:2003, *Natural gas — Determination of mercury — Part 2: Sampling of mercury by amalgamation on gold/platinum alloy*

ICS 75.060

Price based on 16 pages