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**Natural gas — Determination of water  
by the Karl Fischer method —**

**Part 2:  
Volumetric procedure**

*Gaz naturel — Dosage de l'eau par la méthode de Karl Fischer —*

*Partie 2: Méthode volumétrique*

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Published in Switzerland

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

This document was prepared by Technical Committee ISO/TC 193, *Natural Gas*, Subcommittee SC 1, *Analysis of natural gas*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 238, *Test gases, test pressures, appliance categories and gas appliance types*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 10101-2:1993), which has been technically revised.

The main changes are as follows:

- Clause 2 and Bibliography were revised;
- New fixed structure numbering inserted;
- Clause 5 was modified;
- Clause 9 was modified;
- 10.2 was modified.

A list of all parts in the ISO 10101 series can be found on the ISO website.

## Introduction

Water vapour may be present in natural gas due to, for example, natural occurrence in the well production stream, the storage of gas in underground reservoirs, transmission or distribution through mains containing moisture or other reasons.

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# Natural gas — Determination of water by the Karl Fischer method —

## Part 2: Volumetric procedure

WARNING — Local safety regulations should be taken into account, when the equipment is located in hazardous areas.

### 1 Scope

This document specifies a volumetric procedure for the determination of water content in natural gas. Volumes are expressed in cubic metres at a temperature of 273,15 K (0 °C) and a pressure of 101,325 kPa (1 atm). It applies to water concentrations between 5 mg/m<sup>3</sup> and 5 000 mg/m<sup>3</sup>.

### 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 383, *Laboratory glassware — Interchangeable conical ground joints*

ISO 10101-1, *Natural gas- Determination of water by the Karl Fischer method – Part 1- Introduction*

ISO 14532, *Natural gas — Vocabulary*

### 3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

### 4 Principle

A measured volume of gas is passed through a cell containing a relatively small volume of absorbent solution. Water in the gas is extracted by the absorbent solution and subsequently titrated with Karl Fischer reagent. The design of the cell and the absorbent solution are chosen to ensure efficient collection of the water at the high flowrates necessary.

The principle and chemical reactions of the Karl Fischer method are given in ISO 10101-1:2020, Clauses 4 and 5; interferences are also described in ISO 10101-1:2020, Clause 5.

ISO 10101-1:2020, Clause 5 describes interfering substances which may be present in natural gas and corrections for the interference of hydrogen sulfide and mercaptans.

## 5 Reagents

**5.1 Karl Fischer reagent**, of which the water equivalent is approximately 5 mg/ml.

NOTE For most applications, commercially available Karl Fischer (KF) reagent with a water equivalent of approximately 5 mg/ml has been found adequate.

The reagent can be bought as a one-component reagent, which contains all the necessary reagents (iodine, sulfur dioxide and the base (e.g. imidazole)) dissolved in an anhydrous solvent (methanol or 2-methoxyethanol) or it can be provided as two-component reagent, i.e. a solvent reagent and a titrant reagent which are mixed before use.

The solvent reagent contains sulfur dioxide and a base (e.g. an alkali or alkaline earth metal benzoate, ammonia, imidazole). The titrant reagent contains iodine. The two-component reagent provides a stable titre as long as any moisture is prevented from entering into the reagents and a better shelf life.

If required, it may be prepared following the procedure in [5.1.1](#).

### 5.1.1 Preparation of Karl Fischer reagent

#### 5.1.1.1 Components

**5.1.1.1.1 Methanol, with a water content of less than 0,01 % (mass fraction)**. Use commercially available dry methanol, anhydrificated in the lab by one of the following procedures

**5.1.1.1.1.1** Place 2 l of methanol in a two-neck 3 l flask and add 10 g of magnesium turnings. Add a crystal of iodine, connect the flask to a reflux condenser and leave overnight. Next day, add a further 5 g of magnesium turnings and reflux for 1 h. Connect the top of the reflux condenser to a still head, a double surface condenser and a collection flask. Disconnect the water flow through the condenser originally used for reflux and distil the contents of the flask. Discard the first 150 ml of condensate. Distil the rest into dried 1 litre flasks. Vent the system through a drying tube during distillation.

**5.1.1.1.1.2** Dry the methanol over a freshly activated molecular sieve.

**5.1.1.1.2 2-Methoxyethanol**, with a water content of less than 0,01 % (mass fraction).

NOTE This can be used as an alternative to methanol ([5.1.1.1.1](#)) with a lower vapour pressure and therefore less losses due to evaporation during sampling of the gas

**5.1.1.1.3 Imidazol**, anhydrous.

**5.1.1.1.4 Sulfur dioxide**, liquefied and dry.

**5.1.1.1.5 Iodine**.

#### 5.1.1.4 Preparation

Measure 300 ml of dry methanol ([5.1.1.1.1](#)) or 2-methoxyethanol ([5.1.1.1.2](#)) and 110 ml of anhydrous imidazole ([5.1.1.1.3](#)) into a 750 ml conical flask. Slowly pass liquid sulfur dioxide ([5.1.1.1.4](#)) into this solution, mixing carefully until the increase in weight is 43 g. Cool this solution in a freezing mixture. When cool, add sufficient iodine ([5.1.1.1.5](#)) to give a permanent light brown colour. Then add 63 g of iodine and swirl until dissolved. Make up to 500 ml with dry methanol or 2-methoxyethanol. Leave standing in the stoppered conical flask for 24 h before use. Commercial reagents, when aged, may give a slow response near the end point.



## 5.2 Absorbent solution.

### 5.2.1 Components

5.2.1.1 **Ethylene glycol**, with a water content less than 0,1 % (mass fraction).

5.2.1.2 **Sulfur dioxide**, liquefied and dry.

5.2.1.3 **Imidazol**, anhydrous

5.2.1.4 **Karl Fischer reagent** (see 5.1).

### 5.2.2 Preparation of the absorbent solution

Slowly add 20 g of sulfur dioxide (5.2.1.2) to 180 ml of anhydrous imidazole (5.2.1.3), while mixing carefully (solution A).

To prepare the absorbent solution, add 55 ml of dry ethylene glycol (5.2.1.1), 55 ml of Karl Fischer reagent (5.2.1.4) and 73 ml of solution A to a round bottomed flask. Boil under reflux for 10 min with a drying tube on the condenser, and then cool.

5.3 **Reference solution**, e.g. water and methanol mixture, with a water content of 5,0 mg/l  $\pm$  0,2 mg or 10,0 mg/l  $\pm$  0,4 g. Keep this solution in a flask sealed with a septum.

NOTE There are reference solutions commercially available. They consist of stable solvent mixtures with specific composition and precisely determined water content, supplied in airtight glass ampoules to ensure quality when opened by the end user. The exact amount of water is given on the certificate of analysis. Typically, the reference solutions are filled under Argon in 4 ml or 8 ml glass ampoules.

## 6 Apparatus

6.1 **Karl Fischer apparatus**, as described in Annex A.

6.2 **Wet-test gas meter**, accurate to  $\pm 1$  % of the volume passed.

6.3 **Guard tube, or Durand bottle**, packed with anhydrous calcium chloride (or another suitable drying agent).

NOTE This is used to prevent back diffusion of water vapour from the gas meter to the titration cell.

6.4 **Titration cell**, as shown in Figure A.1.

6.5 **Glass syringe**, of 20 ml.

NOTE Absorbent solution is most easily added to and removed from the cell by means of a 20 ml graduated syringe with a 6 % (Luer) fitting and hypodermic needles of suitable length and 1 mm to 2 mm bare.

6.6 **Syringe**, with a fixed needle, of 10  $\mu$ l, for standardization of the Karl Fischer reagent.

## 7 Determination of the water equivalent of the Karl Fischer reagent

7.1 Determine the water equivalent of the Karl Fischer reagent daily or before use, as appropriate, in line with 7.2 to 7.5.

7.2 Using a dry syringe, introduce sufficient absorbent solution (5.2) to cover the electrodes in the apparatus (5.1). Switch on the apparatus and start the stirrer motor. Add the Karl Fischer reagent (5.1) until the needle settles down at a position near zero. When this point is reached, cease additions, since additions of large amounts of reagent will only move the electrometer about 0,02 V. To achieve maximum sensitivity at this first stable point, adjust the zero control until the electrometer needle is at zero. Shake the cell several times so that all the internal surfaces are wetted. Once again, adjust to the zero position by adding more reagent. Repeat the procedure until the needle remains steady at the zero position for at least 30 s.

NOTE The meter needle will remain at zero for at least 30 s when the titration end point is reached.

7.3 Using the 10 µl syringe (6.6), add exactly 10 µl of reference solution (5.3) to the contents of the cell (6.4) (with the syringe needle below the surface of the absorbent solution) titrate to the zero position and note the volume of reagent used. Once again, shake the cell several times and if the electrometer needle shifts, titrate back to zero. Ignore this additional volume of Karl Fischer reagent; it represents any water which may have entered the cell as vapour while the 10 µl of reference solution was being added

7.4 Add a further 10 µl of water to the cell, and again titrate to the zero position. Take an average of the two titrations. If the variation is greater than 2 %, discard the contents of the cell. Introduce a further portion of absorbent solution into the cell and repeat the standardization procedure. If the titration for two further 10 µl portions of distilled water still varies by more than 2 %, it is likely that the Karl Fischer reagent has aged and needs replacing with fresh reagent.

Check the delivery of the 10 µl syringe by weighing it, using a balance capable of weighing to ±0,1 mg. Ensure that the weighings are within 1 %.

7.5 Calculate the water equivalent  $T$ , expressed in milligrams of water per millilitre, of the Karl Fischer reagent using the following Formula (1):

$$T = m / V \quad (1)$$

where

$m$  is the mass, in milligrams, of water added;

$V$  is the volume, in millilitres, of Karl Fischer reagent required for the titration of the added water.

A commercially available water standard may be used instead of distilled water.

## 8 Sampling

Use the procedure described in accordance with ISO 10101-1.

## 9 Procedure

9.1 The apparatus may be used in the laboratory, or outside on the processing plant, with appropriate precautions.

NOTE The differences between these approaches are described in 9.4. Because of the difficulty of sampling gas streams without altering their water content, the equipment is likely to be used more frequently outside the laboratory.

Whether the determination is executed inside or outside the laboratory, the first determination is likely to be erroneous, due to the uncertainties associated with connecting the sample stream and purging the sampling lines. For this reason, repeat determinations are necessary on a sample stream flowing continuously at 1 l/min.