SLOVENSKI STANDARD
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Zemeljski plin - Določevanje vode po Karl-Fischerjevi metodi - 2. del: Volumetrijski postopek (ISO/DIS 10101-2:2020)

Natural gas - Determination of water by the Karl Fischer method - Part 2: Volumetric procedure (ISO/DIS 10101-2:2020)

Erdgas - Bestimmung des Wassergehaltes nach Karl Fischer - Teil 2: Volumetrisches Verfahren (ISO/DIS 10101-2:2020)

Gaz naturel - Dosage de l'eau par la méthode de Karl Fischer - Partie 2: Méthode volumétrique (ISO/DIS 10101-2:2020)

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

Part 2:
Volumetric procedure

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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).

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).

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.

This document was prepared by Technical Committee ISO/TC 193, *Natural Gas*, Subcommittee SC 1, *Analysis of Natural Gas.*

ISO 10101 consists of the following parts, under the general title Natural gas — Determination of water by the Karl Fischer method:

— *Part 1: Introduction*

— *Part 2: Volumetric procedure*

— *Part 3: Coulometric procedure*

Annex A forms an integral part of this part of ISO 10101.

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.

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

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

Part 2: Volumetric procedure

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^3 and 5 000 mg/m^3.

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 383:1976, *Laboratory glassware — Interchangeable conical ground joints*

ISO 10101-1:2020, *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:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

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 clause 5 of ISO 10101-1:2020.

Clause 5 of ISO 10101-1:2020 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 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 KF reagent

5.1.1.1 Components

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

5.1.1.1.1.1 Place 2 litres of methanol in a two-neck 3 litres 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 % (m/m).

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

5.1.1.2 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. 5 Commercial reagents, when aged, may give a slow response near the end point.

