SLOVENSKI STANDARD
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Zemeljski plin - Določevanje vode po Karl-Fischerjevi metodi - 1. del: Uvod (ISO/DIS 10101-1:2020)

Natural gas - Determination of water by the Karl Fischer method - Part 1: Introduction (ISO/DIS 10101-1:2020)

Erdgas - Bestimmung des Wassergehaltes nach Karl Fischer - Teil 1: Einführung (ISO/DIS 10101-1:2020)

Gaz naturel - Dosage de l'eau par la méthode de Karl Fischer - Partie 1: Introduction (ISO/DIS 10101-1:2020)

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

Part 1: Introduction

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

Partie 1: Introduction

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Contents

Page

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).

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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 *[or Project Committee]* ISO/TC *[or ISO/PC]* 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 of this part of ISO 10101 is for information only.

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.

The Karl Fischer method for the determination of moisture has several practical advantages compared to other methods for moisture determination, such as accuracy, speed and selectivity.

KF is selective for water, because the titration reaction itself consumes water.

The Karl Fischer titration can be divided into two basic techniques

— depending on the application range – volumetric and coulometric KF titration. The two analysis techniques differ in the mode of iodine addition or generation.

Karl Fischer titration is essentially based on the Bunsen reaction used for the determination of sulphur dioxide in aqueous solution:

$$2H_2O + SO_2 + I_2 \rightarrow H_2SO_4 + 2HI$$

If an excess of sulphur dioxide with simultaneous neutralization of the sulphuric acid formed shift the reaction equilibrium to the right, the Bunsen reaction can also be used for the determination of water. Karl Fischer used pyridine as (neutralization) base, thus developing the classical Karl Fischer reagent. This was a solution of iodine and sulphur dioxide in a solvent mixture of pyridine and methanol [4]. The fact that the pyridine contained in the reagent has a strong unpleasant odour and toxicity and the reaction runs stoichiometrically only within a certain pH range led to the revision of the KF reagents [4]. Scholz formulated the following KF reaction based on imidazole:

$$CH_3OH + SO_2 + RN \rightarrow [RNH]SO_3CH_3 \text{ with RN = Base}$$

$$H_2O + I_2 + [RNH]SO_3CH_3 + 2RN \rightarrow [RNH]SO_4CH_3 + 2[RNH]I$$

The Karl Fischer titration can be divided into two basic techniques – depending on the application range –: volumetric and coulometric KF titration. The two analysis techniques differ in the mode of iodine addition or generation. Volumetric KF titration is preferably used for the determination of large amounts of water in the range of 1 to 100 mg [5]. Coulometry, however, is a micro-method which is particularly well suited for determination of quantities of water from 10 µg to 10 mg [5]. In coulometric water determination, iodine is not added in the form of a titrating solution but rather directly produced from a iodine-containing solution by an anodic oxidation reaction [4]. The high analytic precision at low absolute water quantities makes coulometric KF titration particularly well suited for determination of the water content in aqueous gases.

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

Part 1: Introduction

1 Scope

This part of ISO 10101 specifies general requirements for the determination of water in natural gas using the Karl Fischer method (see [1]).

ISO 10101-2 and ISO 10101-3 specify two individual methods of determination, a titration procedure and a coulometric procedure, respectively.

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 10101-2, *Natural gas — Determination of water by the Karl Fischer method — Part 2: Volumetric procedure*

ISO 10101-3, *Natural gas — Determination of water by the Karl Fischer method — Part 3: Coulometric procedure*

ISO 10715, *Natural gas — Sampling guidelines*

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 Method basics

4.1 General

Reaction of water present in the test sample with iodine and sulfur-dioxide in a methanol mixture (Karl Fischer reagent commercially available).

The oxidation of the alkylsulfite to alkylsulfate in reaction (3) uses the water that theoretically is in the sample. Since water and iodine are used in a stoichiometric ratio 1:1, the quantity of water in the sample is calculated considering the iodine needed for the complete reaction. The iodine is measured by titration or by coulometry.

4.2 Principle of the titration method (ISO 10101-2)

A measured volume of gas is passed through a cell containing a relatively small volume of absorbent solution. Water in the gas is dissolved in the absorbent solution and subsequently titrated with Karl Fischer reagent, the endpoint being detected voltametrically.

4.3 Principle of the coulometric method (ISO 10101-3)

A measured volume of gas is passed through a cell containing anhydrous, previously titrated, anodic-solution. The iodine required for the titration of the dissolved water is coulometrically produced from the iodide present in the solution by the reaction:

$$2I^- \rightarrow I_2 + 2e^- \qquad (1)$$

5 Reactions and interferences

$$ROH + SO_2 + RN \rightarrow [RNH]SO_3R \qquad (2)$$

$$H_2O + I_2 + [RNH]SO_3CH_3 + 2RN \rightarrow [RNH]SO_4R + 2[RNH]I \qquad (3)$$

where

ROH = alcohol, typically methanol (or 2-methoxyethanol)

RN = basic nitrogen compound

Various gas components react with the Karl Fischer reagent and can give rise to erroneous results. Such components are oxidizing and reducing agents, e.g. hydrogen sulfide, mercaptans and certain basic nitrogenous substances.

Hydrogen sulfide and mercaptans are present in some natural gases. If their concentration is less than 20 % of the water content, interference due to their presence shall be corrected as follows:

$$\rho(H_2O)_a = \rho(H_2O) - \frac{9\rho(SasH_2S)}{16} - \frac{9\rho(SasRSH)}{32} \qquad (4)$$

where

$\rho(H_2O)_a$ is the actual water content, in milligrams per cubic meter at 273.15 K (0°C) and 101.325 kPa (1 atm)

$\rho(H_2O)$ is the observed or measured water content, in milligrams per cubic meter at 273.15 K (0°C) and 101.325 kPa (1 atm)

$\rho(SasH_2S)$ is the measured sulfur as hydrogen sulfide, in milligrams per cubic meter at 273.15 K (0°C) and 101.325 kPa (1 atm)

$\rho(S\ as\ RSH)$ is the measured sulfur as mercaptans in the gas, in milligrams per cubic meter at 273.15 K (0°C) and 101.325 kPa (1 atm)

At higher contents of H2S and RSH this method is not applicable.

NOTE The sulfur present as hydrogen sulfide and mercaptans can be determined by gaschromatography (see ISO 19739 [2]) or by any other suitable method.

