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

Part 1: General requirements

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

Partie 1: Exigences générales

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Foreword

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This second edition cancels and replaces the first edition (ISO 10101-1:1993), which has been technically revised.

The main changes are as follows:

— Clause 2 and Bibliography were revised;

— New fixed structure numbering inserted.

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

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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.

The Karl Fischer method is selective for water, because the titration reaction itself consumes water.

The Karl Fischer (KF) 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.

KF 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 KF reagent. This was a solution of iodine and sulphur dioxide in a solvent mixture of pyridine and methanol[9]. 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[9]. Scholz formulated the following KF reaction based on imidazole:

$$CH_3OH + SO_2 + RN \rightarrow [RNH]SO_3CH_3$$

where RN = Base.

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

Volumetric KF titration is preferably used for the determination of large amounts of water in the range of 1 mg to 100 mg[10]. Coulometry, however, is a micro-method which is particularly well suited for determination of quantities of water from 10 µg to 10 mg[10]. 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[9]. 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.

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

Part 1: General requirements

1 Scope

This document specifies general requirements for the determination of water in natural gas using the Karl Fischer method (see Reference [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 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:

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

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

4 Principle

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 given by Formula (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

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, see ISO 10101-2.

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