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

Part 3:
Coulometric procedure

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

Partie 3: Méthode coulométrique

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Foreword

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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 10101-3:1993), which has been technically revised.

The main changes are as follows:

- Clause 2 and Bibliography were revised;
- new fixed structure numbering inserted;
- Subclause 9.2 Measurement of uncertainty was added.

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

Modern KF coulometers cover a range from 10 µg to 200 mg of water. Usually a resolution of 0,1 µg of water is achieved.

In coulometric water determination, iodine is not added in the form of a titrating solution but rather directly produced from an iodine-containing solution by an anodic oxidation reaction. 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.

Coulometric KF titration can be subdivided according to two distinct designs of the analysis cell: Cells with and without diaphragm. In both variants, the measuring cells are made of a titration vessel tightly sealed to prevent moisture ingress. The sample gas is passed directly through a glass frit into the KF titration cell. Thus, absorption of moisture from the environment is prevented and the gas finely dispersed. The fine distribution of the gas in the hygroscopic KF solution provides a large surface for material exchange, so that the water contained in the gas can be fully absorbed by the solution and then titrated. In the version with a diaphragm, the cell is divided into a large anode and a small cathode compartment, each filled with different reagents. Spatial separation is achieved by means of the diaphragm. In both compartments platinum electrodes are installed, via which a working current is passed through the titration cell. Due to the applied current, at the anode iodine is formed, which immediately reacts with the absorbed water from the gas sample. When all the water has been consumed by the reaction, an excess of iodine is formed that will be detected voltametrically, ending the titration. The amount of electricity consumed can be used to directly calculate, using Faraday’s law, the quantity of water.

$$m_{\mathrm{H_2O}} = \frac{M_{\mathrm{H_2O}} \cdot Q}{z \cdot F}$$

where

z is the number of exchanged electrons;

$M_{\mathrm{H_2O}}$ the molecular weight of water;

F the Faraday constant (96 485 C/mol);

Q the charge which has flowed in C.

In the KF titration cell variant without a diaphragm there is no separation between the anode and cathode chambers. Thus, for the filling of the cell only one reagent is needed and used. In order to prevent direct reduction of iodine at the cathode, the cathode and anode are spatially separated from each other by a large distance. The use of the cell without a diaphragm has the advantage that the titration cell is easier to clean and only one reagent is consumed, whose replacement can be completely automated. In addition, unlike in cells with a diaphragm, during longer downtimes no moisture can accumulate in the diaphragm, making the titration cell faster to become operational. For the measurement of extremely low water contents (few ppm of water), the leading KF equipment manufacturers recommend, despite these advantages, use of a KF coulometer with diaphragm. For practical implementation, however, this adds possible sources of error, complication and prolongation of the measurement times.

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