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Natural gas — Determination of sulfur compounds — Determination of total sulfur by oxidative microcoulometry method

Gaz naturel — Détermination des composés soufrés — Détermination de la teneur totale en soufre par microcoulométrie oxydante

ICS 75.060

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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.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 16960 was prepared by Technical Committee ISO/TC 193, *Natural gas*, Subcommittee SC 1, *Analysis of natural gas*.

This second/third/... edition cancels and replaces the first/second/... edition (), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

Introduction

This international standard specifies a method for the determination of total sulfur in the range from 1 mg/m^3 to 200 mg/m^3 in pipeline natural gas by oxidative microcoulometry.

Three methods for determination of sulfur compounds in natural gas already exist as ISO standards:

ISO 6326-3 Natural gas -Determination of sulfur compounds - Part 3: Determination of hydrogen sulfide, mercaptan sulfur and carbonyl sulfide sulfur by potentiometry;

ISO 6326-5 Natural gas - Determination of sulfur compounds - Part 5: Lingener combustion method;

ISO 19739 Natural gas - Determination of sulfur compounds using gas chromatography.

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Natural gas — Determination of sulfur compounds — Determination of total sulfur by oxidative microcoulometry method

1 Scope

This international standard specifies a method for determination of total sulfur in the range from 1 mg/m^3 to 200 mg/m^3 in pipeline natural gas by oxidative microcoulometry. Natural gas with sulfur contents above 200 mg/m^3 can be analysed after dilution with a suitable sulfur-free solvent.

NOTE This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations before its application.

2 Normative references

The following referenced documents are indispensable for the application 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 3696, *Water for analytical laboratory use - Specification and test methods*

ISO 6142, *Gas analysis -- Preparation of calibration gas mixtures -- Gravimetric method*

ISO 6144, *Gas analysis -- Preparation of calibration gas mixtures -- Static volumetric method*

ISO 6145, *Gas analysis -- Preparation of calibration gas mixtures using dynamic volumetric methods*

ISO 10715, *Natural gas - Sampling guidelines*

3 Test principle

A gas sample containing sulfur is mixed with oxygen in a quartz furnace tube in order to convert the sulfur compounds to sulfur dioxide by oxidative pyrolysis. The obtained sulfur dioxide enters the titration cell along with carrier gas and reacts with iodine contained therein. The consumed iodine is complemented by the electrolysis of potassium iodide. In accordance with Faraday's law of electrolysis, the sulfur concentration in the gas sample can be calculated from the consumed electric quantity by electrolysis and corrected by comparison to a reference standard sample.

4 Reagents

4.1 Test water: conforming to the requirements of Grade 3 of ISO 3696;

4.2 Glacial acetic acid: analytical reagent;

4.3 Potassium iodide: analytical reagent;

4.4 Oxygen: minimum purity of 99,99 vol.-%;

4.5 Carrier gas: argon, helium or nitrogen with a minimum purity of 99,99 vol.-%.

5 Apparatus

5.1 Converter

There are three independent heating sections in a converter: preheating section (800 °C ± 20 °C), combustion section (900 °C ± 20 °C) and exit section (800 °C ± 20 °C).

5.2 Titration cell

Install a pair of electrolysis electrodes and a pair of indicator-reference electrodes in the cell.

5.3 Microcoulometer

Electrolysis commences automatically, when there is a reduction in the concentration of iodine caused by the presence of sulfur dioxide in the titration cell, to maintain the concentration of iodine at its original level. The microcoulometer can automatically record the electrolysis time and current and directly display the sulfur content.

5.4 Flow controller

Provides the specified flow rates at the outlet.

5.5 Electromagnetic agitator

Provides the specified rotational rates in the titration cell.

5.6 Medical syringe

Air tight syringes of volumes 0.25mL, 1mL, 2mL and 5mL. Syringe-delivered volumes should be calibrated by weighing pure water prior to initial use. Periodic calibration may be required thereafter.

5.7 Volumetric flask

One standard laboratory volumetric flask, of nominal 25mL volume.

6 Test preparation

6.1 Preparation of electrolyte

Weigh 0,5 g potassium iodide, dissolve it in 100 mL water, add in 5 mL glacial acetic acid, and then dilute the solvent to 1 L with water. The electrolyte should be stored in a brown reagent bottle. The valid period is three months.

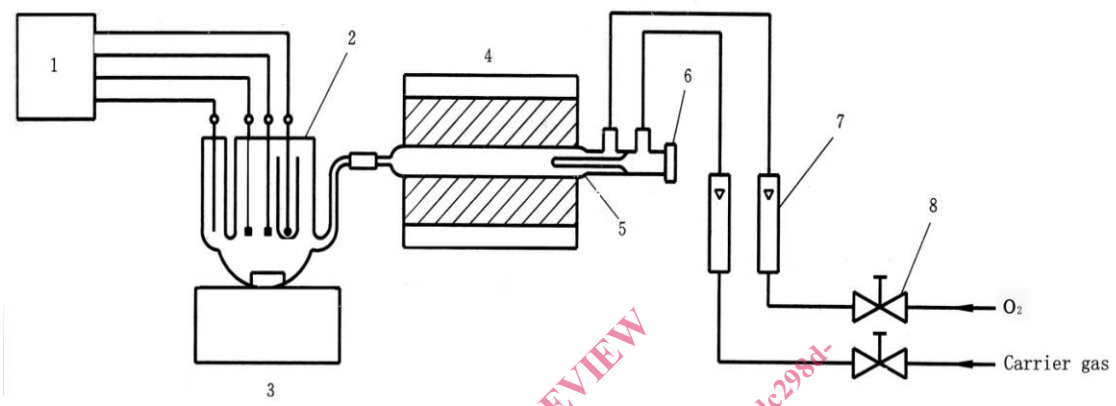
6.2 Reference sample

Use calibration gas mixtures of sulfur compounds prepared according to ISO 6142, ISO 6144, or ISO 6145. Sulfur concentration in the calibration gas mixtures shall be close to that in the tested sample.

It is recommended that sulfur compounds in the calibration gas mixture were carbonyl sulfide (COS) or hydrogen sulfide (H_2S).

6.3 Apparatus installation

Install the apparatus following the instruction. Connect the carried gas tube and oxygen tube. An example of a typical oxidative microcoulometer is given in Figure 1.



Key

1 microcoulombmeter
2 titration cell
3 electromagnetic stirrer
4 reforming furnace
5 quartz furnace tube
6 injection port
7 flow meter
8 needle valve

Figure 1 — Typical microcoulometric set-up

6.4 Preparation of instrument

Replace the silicone rubber plug in the injection port. Regulate the flow rate of carrier gas and oxygen to the values specified by the apparatus. Then open the electromagnetic stirrer, adjust stirring speed to produce a slight vortex in the electrolyte.

6.5 Check parameters

Adjust the potentiometer to the prescribed value. Check all the operating parameters according to the requirement by the instruction of the instrument.

6.6 Determination of conversion rate of sulfur

Install software of microcoulometer and display conversion rate automatically by PC.

6.6.1 Sampling and injection

Take samples after flushing the syringe with the gas standard sample for four to five times. The syringe plunger should be pushed to the required scale by the gas pressure in the bottle when sampling. Insert the syringe into the injection port, the injection speed is about 0.15 to 0.2 mL/s, the injection volume is generally 0.25 to 5 mL.