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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*

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Published in Switzerland

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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](http://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](http://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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 193, *Natural gas*, Subcommittee SC 1, *Analysis of natural gas*.

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## Introduction

Three methods for determination of sulfur compounds in natural gas already exist as International 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

**WARNING** — The use of this International Standard can involve hazardous material, operations, and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

## 1 Scope

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

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 6146, *Gas analysis — Preparation of calibration gas mixtures — Manometric method*

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 purity.

**4.3 Potassium iodide**, analytical purity.

**4.4 Oxygen**, minimum purity 99,99 % (by volume fraction), maximum volume fraction of sulfur containing gases 0,01 % (by volume fraction).

**4.5 Carrier gas**, argon, helium, or nitrogen with a minimum purity 99,99 % (by volume fraction), maximum volume fraction of sulfur containing gases 0,01 % (by volume fraction).

## 5 Apparatus

### 5.1 Converter

There are three independent heating sections in a converter: preheating section ( $800\text{ °C} \pm 20\text{ °C}$ ), combustion section ( $900\text{ °C} \pm 20\text{ °C}$ ), and exit section ( $800\text{ °C} \pm 20\text{ °C}$ ).

### 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 0,25 ml, 1 ml, 2 ml, and 5 ml. Syringe-delivered volumes should be calibrated by weighing pure water prior to initial use. Periodic calibration might be required thereafter.

### 5.7 Volumetric flask

One standard laboratory volumetric flask of nominal 25 ml 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 electrolyte shelf life after preparation is three months.

### 6.2 Reference sample

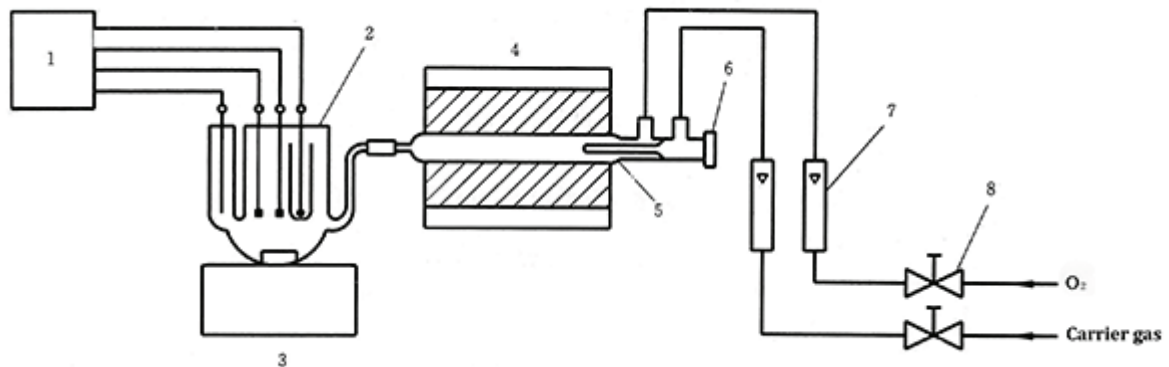
Use calibration gas mixtures of sulfur compounds prepared according to ISO 6142, ISO 6144, or ISO 6146. Sulfur concentration in the calibration gas mixtures shall be close to that in the tested sample.

It is recommended that sulfur compounds in the calibration gas mixture were hydrogen sulfide ( $\text{H}_2\text{S}$ ) in a methane matrix.



### 6.3 Apparatus installation

Install the apparatus following the instruction. Connect the carried gas tube and oxygen tube. An example of a typical microcoulometer set-up is given in [Figure 1](#).



#### Key

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

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**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 switch on the electromagnetic stirrer, adjust stirring speed to produce a slight vortex in the electrolyte

### 6.5 Check and adjustment of the instrument

Check and adjust all the operating parameters according to the instruction manual of the instrument.

### 6.6 Determination of recovery factor of sulfur

Install software of microcoulometer and display the sulfur recovery factor using computer software, typically installed on a person computer (PC), suited for the microcoulometer being used.

#### 6.6.1 Sampling and injection

Take samples after flushing the syringe with the gas standard sample 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 rate is about 0,15 ml/s to 0,2 ml/s and the injection volume is typically 0,25 ml to 5 ml.