

---

# International Standard 8173

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

---

## Light olefins for industrial use — Determination of molecular oxygen in gaseous phase — Electrochemical method with a membrane-covered cell

*Oléfines légères à usage industriel — Dosage de l'oxygène moléculaire en phase gazeuse — Méthode électrochimique avec une cellule à membrane*

First edition — 1986-11-15

[standards.iteh.ai](https://standards.iteh.ai)

ISO 8173:1986

<https://standards.iteh.ai/catalog/standards/sist/197c09ae-16c9-4ae7-aa39-ca4d56378ed0/iso-8173-1986>



---

UDC 661.725 : 543.5 : 546.21

Ref. No. ISO 8173-1986 (E)

**Descriptors** : industrial products, olefinic hydrocarbons, chemical analysis, determination of content, oxygen, apparatus, electrochemical methods.

Price based on 5 pages

## 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8173 was prepared by Technical Committee ISO/TC 47, *Chemistry*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

# Light olefins for industrial use — Determination of molecular oxygen in gaseous phase — Electrochemical method with a membrane-covered cell

## 0 Introduction

For the determination of traces of molecular oxygen in gaseous olefins, only an electrochemical method with aqueous electrolyte at ambient temperature may be used.

The method by measurement of the paramagnetic susceptibility of the gaseous phase cannot be used on account of lack of sensitivity in the range of contents near 1 ml/m<sup>3</sup>.

The method by measurement of the electromotive force of a concentration cell with a solid zirconia electrolyte must not be used because of the temperature requirements of 700 to 850 °C.

## 1 Scope and field of application

This International Standard specifies an electrochemical method for the determination of molecular oxygen in light olefins in the gaseous phase using a membrane-covered electrochemical cell containing a gelled aqueous electrolyte.

The method is applicable to light olefins having an oxygen content greater than 1 ml/m<sup>3</sup>.

## 2 References

ISO 6349, *Gas analysis — Preparation of calibration gas mixtures — Permeation method.*

ISO 6377, *Light olefins for industrial use — Determination of impurities by gas chromatography — General considerations.*

ISO 6711, *Gas analysis — Checking of calibration gas mixtures by a comparison method.*

ISO 7382, *Ethylene for industrial use — Sampling in the liquid and the gaseous phase.*

ISO 8563, *Propylene and butadiene for industrial use — Sampling in liquid phase.*

## 3 Principle

After sampling and preparation of the laboratory sample, passage of the gaseous test portion at a given rate through an electrochemical cell previously calibrated with air or with standard mixtures containing a known amount of oxygen, then measurement of the concentration of oxygen in the gaseous olefin.

NOTE — The measurement of the concentration of the oxygen is based on the following principle:

Specific electrochemical reduction of molecular oxygen after diffusion through a polymer membrane covering the porous cathode.

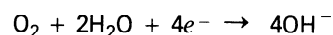
The overall electric current of the galvanic cell is a function of the diffusion process.

The electric current is proportional to the partial pressure of the oxygen in the adjacent gaseous phase, i.e. it is proportional to the oxygen concentration in the gaseous phase at constant total pressure.

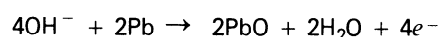
## 4 Reactions

The electrochemical reactions are as follows:

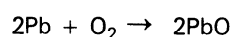
— Cathode reaction



— Anode reaction



— Overall reaction



## 5 Materials

### 5.1 Water, deoxygenated and deionized.

### 5.2 Nitrogen, having an oxygen content less than 5 ml/m<sup>3</sup>.

**5.3 Standard mixtures**, containing known amounts of oxygen in any inert gas such as argon or nitrogen.

These can be obtained commercially or can be prepared (see 8.3).

NOTE — The calibration is advisable in special cases for the exact determination of absolute values. Normally, a calibration with air is sufficient.

## 6 Apparatus

Ordinary laboratory apparatus and

**6.1 Oxygen meter**, for gaseous samples, comprising a detector cell without external polarization, an amplifier, a means of compensating for the temperature of the cell and a means of indicating or recording the electric current produced in the cell to an accuracy of 1 % of full scale over a range of at least 5<sup>10</sup>. The detection limit of commercial apparatus shall be less than 1 ml/m<sup>3</sup>.

The cathode shall be constructed of a suitably unreactive metal such as silver, gold or platinum and the anode shall be constructed of lead or zinc.

The electrochemical cell (see figure 1) contains a gelled aqueous electrolyte which is maintained in a moist condition (see 8.4).

**6.2 Flow meter**, capable of measuring gas flows between 0,1 and 1 l/min at normal conditions.

**6.3 Coiled stainless steel, capillary tube**, for vaporization of liquid samples (see ISO 6377, sub-clause 4.2).

**6.4 Humidifier** (see figure 2): Vessel fitted with a plastics cylinder on which is coiled a silicone tube of length about 1 m, internal diameter 1 mm and external diameter 2 mm.

Before filling the assembled humidifier with the water (5.1), purge, for about 1 min, with a nitrogen stream through the open septum cap and purge also the silicone tube in the same manner.

Fill the humidifier completely with the water (5.1) (see figure 2) and screw the septum cap.

The water having been diffused through the silicon wall and evaporated shall be replaced at intervals of about 6 weeks by introducing the water (5.1) with an injection syringe through the septum.

**6.5 Extraction system** (see ISO 7382).

## 7 Sampling

See ISO 6377 clause 4, ISO 7382 and ISO 8563.

## 8 Procedure

**WARNING** — The operations specified shall be carried out under a well-ventilated hood and in a completely flame-free environment and the test samples shall be removed by the extraction system (6.5).

### 8.1 Assembly of the apparatus (see figure 3)

#### 8.1.1 Precautions to be taken to optimize the measurements

Prevent any leaks allowing atmospheric air to penetrate into the gas line. The connection tubing shall be made of stainless steel. At the outlet of the measuring device, a capillary tube shall be connected to prevent the introduction of traces of oxygen by diffusion from the atmospheric air.

#### 8.1.2 Cases of gaseous samples under pressure

Connect the oxygen meter (6.1) to the sampling bottle or sampling line by means of a stainless steel capillary and air-tight joints.

NOTE — If a pressure regulator is necessary, use a regulator with a welded metal membrane.

Place the flow meter (6.2) at the exit of the detector cell, and extend it with a stainless steel capillary tube of 50 cm of length to prevent the introduction of oxygen by diffusion of atmospheric air.

#### 8.1.3 Cases of liquid samples (propylene, butadiene) under pressure or cooled

In the case of liquid samples under pressure, carry out their vaporization by means of the coiled capillary tube (6.3) according to the indication given in clause 4 of ISO 6377 and produce a gaseous continuous sample flow.

In the case of liquid samples kept at low temperatures in a cooled trap, press these liquids into a capillary tube by means of a regulated nitrogen pressure of about 1 bar\*, which is not dangerous for the glass trap.

In both cases, connect the oxygen meter (6.1) to the outlet of the vaporization capillary (6.3) by means of sealed joints.

Place the flow meter (6.2) and the safe capillary as indicated in 8.1.2.

\* 1 bar = 10<sup>5</sup> Pa

## 8.2 Check of the apparatus

To test for possible leaks at the joints of the gas line, increase the flow rate of the gas sample to about twice that to be used for the determination as indicated by the flow meter (6.2). In the case of a possible variation in the initial value of the oxygen content, the air-tightness of the gas line is defective (there is introduction of oxygen from the atmospheric air).

NOTE — Since the permeation of oxygen through the membrane of the electrochemical cell depends on its partial pressure only, variations of flow rate will not influence the results.

## 8.3 Calibration

Calibrate with atmospheric air or with a commercial standard mixture for calibration (5.3).

Most current oxygen meters have a reliable linear range of  $6^{10}$ , so the calibration with atmospheric air is sufficient.

In cases of doubt or in special cases, it is advisable to use the calibration gas mixtures (5.3) with a comparison method (see ISO 6711).

The preparation of calibration gas flow with constant oxygen concentration is easily carried out by the permeation method (see ISO 6349).

The calibration shall be carried out at atmospheric pressure and at approximately the same flow rate as that used for the determination.

NOTE — Generally, the calibrations are necessary after a period of about 1 month.

Nevertheless, during the first time of use of a particular instrument, the calibration should be carried out more often to test its reliability.

## 8.4 Determination

The apparatus being assembled as in 8.1, introduce the gas sample at a given flow rate (within the range recommended by the manufacturer of the instrument) as long as necessary to obtain a constant concentration value during a period of at least 2 min.

Before and after the determination, the oxygen meter (6.1) shall be kept in good running order, by passing a current of nitrogen (5.1) moistened by the humidifier (6.4) through the electrochemical cell of the detector at a flow rate between 1 and 2 l/h to remoisten the gelled aqueous electrolyte.

NOTE — At a flow rate of 1,5 l/h at 23 °C, the moisture content of the gas flow is about 1 % (V/V).

## 9 Expression of results

### 9.1 Method of expression

Express the oxygen content in millilitres per cubic metre.

### 9.2 Repeatability

The results obtained for identical test samples, using the same apparatus, shall not differ by more than 2 % of the mean value.

## 10 Test report

The test report shall include the following information:

- a) all information necessary for the complete identification of the sample (lot, date, time and duration of each sampling, etc.);
- b) a reference to this International Standard;
- c) a statement of any experimental conditions which are regarded as optional, such as a description of the electrochemical cell;
- d) the results and the method of expression used;
- e) details of any unusual features noted during the determination;
- f) details of any operations not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

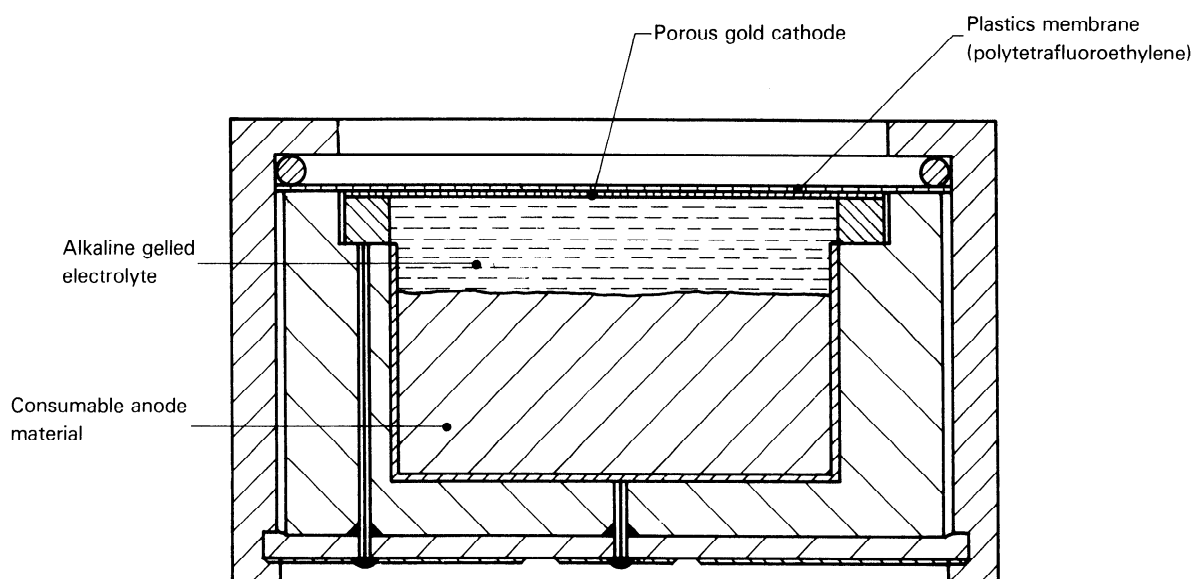


Figure 1 — Cross-section of a typical electrochemical cell

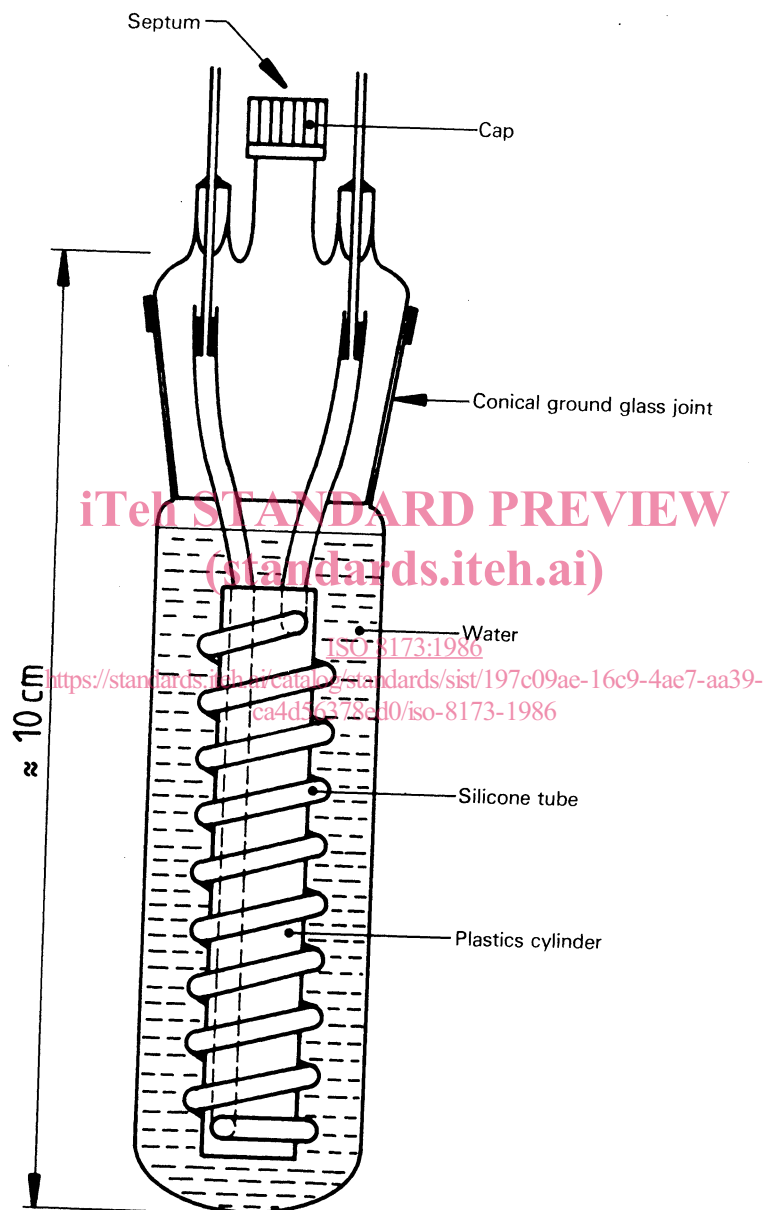


Figure 2 – Humidifier

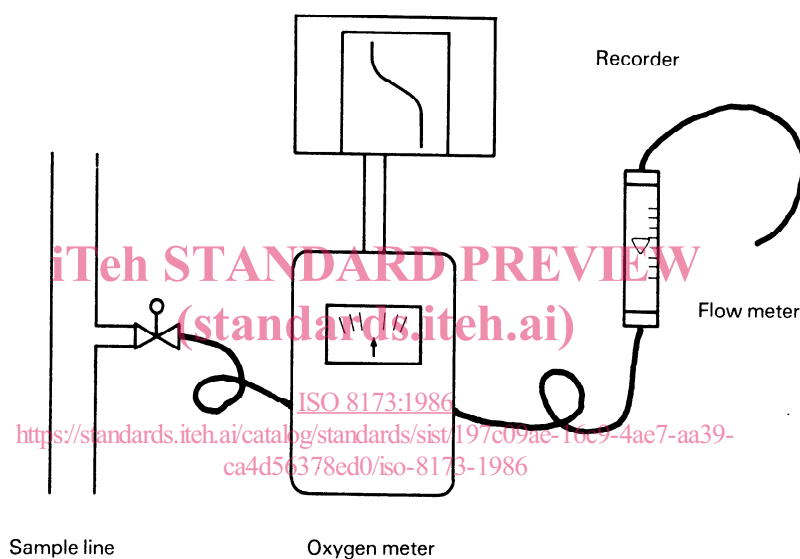


Figure 3 — Diagram of assembly of oxygen meter

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

This page intentionally left blank

[ISO 8173:1986](#)

<https://standards.iteh.ai/catalog/standards/sist/197c09ae-16c9-4ae7-aa39-ca4d56378ed0/iso-8173-1986>