



SLOVENSKI STANDARD
SIST ISO 8186:1996

01-avgust-1996

Zunanji zrak - Določevanje masne koncentracije ogljikovega monoksida - Metoda plinske kromatografije

Ambient air - Determination of the mass concentration of carbon monoxide - Gas chromatographic method

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Air ambient - Détermination de la concentration en masse du monoxyde de carbone - Méthode par chromatographie en phase gazeuse

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Ta slovenski standard je istoveten z: ISO 8186:1989

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13.040.20 Kakovost okoljskega zraka Ambient atmospheres

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en

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INTERNATIONAL STANDARD

**ISO
8186**

First edition
1989-07-01

Ambient air — Determination of the mass concentration of carbon monoxide — Gas chromatographic method

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*Air ambiant — Détermination de la concentration en masse du monoxyde de
carbone — Méthode par chromatographie en phase gazeuse*
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Reference number
ISO 8186 : 1989 (E)

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.

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 8186 was prepared by Technical Committee ISO/TC 146, *Air quality*.

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Annexes A and B form an integral part of this International Standard. Annex C is for information only.

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International Organization for Standardization

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Introduction

This International Standard originated from international demands for air pollution research and control.

The method in general can be used for the manual or automatic measurement of carbon monoxide over a wide range of concentrations found in air samples from a variety of sources. This International Standard, utilizing gas chromatography, is applicable to the measurement of low mass concentrations of carbon monoxide in ambient air and for situations where more accuracy, i.e. 5 % or better of the full scale in the ranges of 0 to 1 mg/m³ and 0 to 25 mg/m³ is required.

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Ambient air — Determination of the mass concentration of carbon monoxide — Gas chromatographic method

1 Scope

This International Standard specifies a gas chromatographic method for the determination of the mass concentration of carbon monoxide in ambient air from a variety of sources. The method is free from interference because the appropriate gas chromatographic conditions are used to separate the carbon monoxide from all the other constituents in the air sample, before the carbon monoxide is measured. It is intended for the measurement of carbon monoxide at concentrations less than 25 mg/m³ although it can be successfully applied to concentrations up to 1 000 mg/m³ respectively¹⁾ at 25 °C and 101,3 kPa. Measurements of carbon monoxide at concentrations of less than 1 mg/m³ can be obtained with careful operation. The automatic technique is continuous only insofar as several discrete air samples can be drawn and analysed each hour.

NOTE — Although this International Standard is written for two ranges, i.e. 0 to 1 mg/m³ and 0 to 25 mg/m³, an intermediate range, e.g. 0 to 10 mg/m³, could be utilized to advantage if all the concentrations fall within the expected range and more accuracy is desired. It is dependent on the error in reading the output trace.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 6143 : 1981, *Gas analysis — Determination of composition of calibration gas mixtures — Comparison methods.*

ISO 6144 : 1981, *Gas analysis — Preparation of calibration gas mixtures — Static volumetric methods.*

3 Principle

Passage of a fixed volume of the air sample through a chromatographic column for effective separation of carbon monoxide from other gaseous constituents. Reduction of the

separated carbon monoxide to methane by the hydrogen carrier gas by passage over a heated nickel catalyst (other catalysts and promoters may be used). Passage of the resulting methane through a flame ionization detector. The output signal is proportional to the amount of carbon monoxide present in the sample.

4 Materials

4.1 Gases

See ISO 6143 and ISO 6144. The gases required for the operation of the gas chromatograph shall not contain impurities that will give a signal equal to or more than the minimum detectable carbon monoxide concentration.

Their maximum moisture content shall be less than 10 mg/m³, total hydrocarbons expressed as methane shall be less than 0,1 mg/m³ and carbon monoxide shall be less than 0,1 mg/m³.

WARNING — When using compressed gases, the safety precautions recommended by the supplier shall be followed. In particular, handle methane and hydrogen with care because they present a fire hazard under certain conditions.

4.1.1 Hydrogen is used as the carrier gas in the gas chromatographic separation, for the catalytic reduction of carbon monoxide to methane and for the operation of the flame ionization detector.

4.1.2 Air is required for the operation of the flame ionization detector.

4.1.3 Nitrogen.

4.1.4 Helium.

For certain burners, nitrogen or helium or a mixture of both is added to the fuel gas to optimize sensitivity and stability.

1) At the temperature and pressure given, the following conversion factors apply :

1 mg/m³ ≅ 0,88 ppm (V/V)

1 ppm (V/V) ≅ 1,14 mg/m³

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4.2 Calibration gas mixtures

4.2.1 Methane in air

A certified standard mixture of methane in air is required for measuring the converter efficiency. Its concentration shall be known to within 1 % and be close to that of the highest carbon monoxide standard mixture to be used for calibration.

The converter efficiency is found by comparing the areas of peaks obtained from the known methane gas mixture and the known carbon monoxide gas mixture. The areas of the peaks are in the same ratio as the methane and carbon monoxide concentrations, if complete conversion of the carbon monoxide has been accomplished.

4.2.2 Carbon monoxide in air

Certified standard mixtures of carbon monoxide in air or nitrogen (certified accurate to ± 1 % of component) are used as calibration gases. At least four different concentrations covering the range of measurements being made shall be used, e.g. concentrations of 0, 20 %, 50 % and 80 % of the full scale.

4.3 Converter and gas chromatographic materials

The nickel nitrate hexahydrate used for preparation of the converter column shall be of reagent grade. The converter packing consists of 0,125 mm to 0,15 mm (100 to 120 mesh ASTM) diatomaceous earth (also known as kieselguhr firebrick Chromosorb P¹⁾; diatomaceous earth-pink; diatomaceous earth-firebrick).

The gas chromatographic column material is 0,18 mm to 0,25 mm (60 to 80 mesh ASTM) synthetic alkali metal aluminosilicate beads (may be known as molecular sieve 5A¹⁾ or Zeolox sieve 5A¹⁾).

If other mesh sizes are used, their equivalency shall be demonstrated.

5 Apparatus

A gas chromatographic system with a flame ionization detector (FID) shall be used. An automatic sampling and injection system provides a series of discrete samples and analyses.

The range of the instrument being used shall be compatible with the concentration of gas to be measured.

5.1 Column

A gas chromatographic column system shall be used to separate carbon monoxide from other components present in the sample. It is important that water is not permanently retained by the packing materials. Hence the column shall be back-flushed between sample injections.

The system recommended in this International Standard does not use a stripper column and works satisfactorily under the conditions recommended. The separated carbon monoxide is passed to the converter and the column is back-flushed in preparation for the next sample.

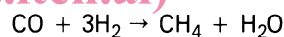
NOTES

1 Some systems utilize a stripper column that separates the fast-eluting components (methane and carbon monoxide) from the slower-eluting components (carbon dioxide, water and hydrocarbons). After elution of methane and carbon monoxide, the stripper column is switched and back-flushed to remove the slower eluting components.

2 Various packing materials and column dimensions may be used to achieve the required separation (columns are available commercially). A 3,2 mm outer diameter stainless steel tube (copper tube is not recommended) packed with 0,18 mm to 0,25 mm (60 to 80 mesh ASTM) molecular sieve 5A and operated at a temperature above 45 °C is recommended. (The tolerance in the isothermal temperatures recommended throughout this International Standard is ± 5 °C unless otherwise specified.) The column length must be determined by the carrier gas flow applicable to the gas chromatograph being used. For example, a carrier gas flow of 20 cm/min will require a column 2 m long, operated at 45 °C, to give adequate separation.

5.2 Converter

Carbon monoxide is converted to methane by reaction with hydrogen in the presence of a heated nickel catalyst. The reduction occurs according to the following equation :



NOTE — Added promoters, such as thorium oxide or ruthenium, have been used¹⁾ to extend the life of the nickel catalyst, especially when higher converter temperatures are used and when carbon dioxide is also being converted to methane. Experience with the procedures recommended in this International Standard has shown that the conversion efficiency of carbon monoxide to methane and the life of the nickel catalyst were not a problem, since small quantities of carbon monoxide were being converted to methane in the presence of pure hydrogen at a relatively low temperature, i.e. 260 °C.

5.3 Converter-oven assembly

Various designs for the heater are available, or it can be constructed in a machine-shop. A suitable arrangement may be constructed from an aluminium block as shown in figure 1. The 100 W cartridge heater with a stainless steel sheath and controlled by a rheostat will provide sufficient heat and stability for the system. The converter tube and the block assembly are wrapped in a suitable insulating material. A thermometer is inserted into the oven block and the desired temperature is reached by manual setting of the rheostat. A temperature of 260 °C has been found to be satisfactory for the conversion of carbon monoxide to methane and avoids problems of tailing and interference from oxygen^[2,3].

5.4 Sampling system

A sample injection valve with a sampling loop that has the desired capacity is used to introduce the sample into the gas

1) Chromosorb P, molecular sieve 5A, Zeolox sieve 5A are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

chromatographic system. A small pump capable of drawing approximately 100 cm³/min of air through the loop is used to collect the sample.

NOTE 1 — The sampling loop of the injection valve can be readily constructed from 3,2 mm outer diameter stainless steel tubing to give a capacity of approximately 4 cm³ ¹⁴. Condensation in the sampling loop can be avoided by holding the loop at a constant temperature slightly above sample temperature.

Use a suitable arrangement of sampling valves, such as shown in figure 2, to perform the following sequence of functions :

a) By-pass configuration. The carrier gas flow is split into two equal streams by means of needle valves. One portion flows through valve 4 and the converter to the detector, the other flows through valves 1, 2 and 3, back-flushing the column, and then through valve 4 to the detector. During this mode, the sampling pump is turned on to flush and fill the sample loop.

b) Inject configuration. In this mode the carrier gas flow previously passing through the converter is diverted directly to the detector by valve 4. The other stream of carrier gas is

diverted through the sampling loop by switching valve 1, and then passes directly through the column by switching valves 2 and 3. Valve 4 then allows the carrier gas stream to flow through the converter to the detector. Immediately after the converted methane has passed through the detector, the system is reverted to by-pass mode, allowing for back-flushing of the column and refilling of the sampling loop.

Use timers and valve actuators to control the sequence of operation and allow a series of discrete samples to be analysed. The recording device can also be timed to operate only during elution of the converted carbon monoxide. The time required for one cycle is dependent on the back-flushing needed to clean the column before injection of the next sample. With the system described, it should be possible to process up to 10 samples per hour.

NOTE 2 — Contamination of the gas chromatographic column by water vapour will change the retention time of the carbon monoxide and may require adjustment of the timing sequence. The line between the converter and detector should be as short as possible and held at a temperature above 100 °C to prevent condensation of water.

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