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Work-place air — Determination of mass concentration of carbon monoxide — Method using detector tubes for short-term sampling with direct indication

iTeh STANDARD PREVIEW

Air des lieux de travail — Détermination de la concentration en masse du monoxyde de carbone — Méthode utilisant des tubes détecteurs pour échantillonnage rapide à lecture directe

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 8760 was prepared by Technical Committee ISO/TC 146, *Air quality*.

Annex A forms an integral part of this International Standard. Annex B is for information only.

Introduction

The determination of the mass concentration of carbon monoxide present in the air at work places (or work areas) by means of detector tubes for short-term sampling with direct indication, called detector tubes in the following text, is made with one of several suitable reagent systems. The most important of these are the reagent systems based on potassium palladosulfite and iodine pentoxide.

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Work-place air — Determination of mass concentration of carbon monoxide — Method using detector tubes for short-term sampling with direct indication

1 Scope

This International Standard specifies a method for the determination of the mass concentration of carbon monoxide present in the air at work places (or work areas) in concentrations greater than 10 mg/m³ using detector tubes.

Substances which, if contained in the air mass under investigation and thus in the air sample, are known to have an effect on the instrument reading, are indicated in clause 5. Information on performance characteristics is given in 9.2; in applications requiring better precision or freedom from interferences, the use of classical chemical or instrumental methods is recommended (see 8519[10]).

The method is suitable for personal, breathing-zone sampling as well as for the general area sampling.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 edition of the standard given below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 6879 : 1983, *Air quality — Performance characteristics and related concepts for air quality measuring methods.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 length-of-stain detector tube: A tube containing a reagent which reacts rapidly with carbon monoxide present in the air sample, producing a sharply defined interface between reacted and unreacted reagent system.

3.2 volume per stroke: The amount of air or other gas mixture drawn by the detector-tube pump during the opening time per stroke.

3.3 opening time per stroke: The time taken for one complete cycle of the detector-tube pump air chamber.

4 Principle

Reaction of carbon monoxide present in the air sample passing through a detector tube within a given period with reagents coated on a solid support contained in the tube, resulting in the formation of a coloured reaction product which provides a sharply defined stain in the tube.

Determination of the mass concentration of carbon monoxide by comparing the observed length of stain developed to lengths of stain prepared using calibration gas mixtures, taking into account the effect of interferents on the instrument reading and the correction factors for pressure, temperature and relative humidity.
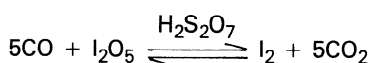
5 Reactions and interferents

Several colour-producing reactions by which carbon monoxide can be detected are known. The following are used in detector tubes.

5.1 Iodine pentoxide reaction

Tubes containing a reagent system based on iodine pentoxide are length-of-stain detector tubes; the change of intensity of colour is from white to green-brown.

5.1.1 Reaction equation

$$5CO + I_2O_5 \xrightarrow[]{H_2S_2O_7} I_2 + 5CO_2$$

5.1.2 Interferents

Acetylene, aliphatic hydrocarbons, and halogenated hydrocarbons are positive interferents. With the exception of acetylene, these can be removed by a pretreatment section.

5.2 Potassium palladosulfite reaction

Tubes containing a reagent system based on potassium palladosulfite are length-of-stain detector tubes; the change of intensity of colour is from yellow to brown.

5.2.1 Reaction equation

$$CO + K_2Pd(SO_3)_2 \rightarrow Pd + CO_2 + SO_2 + K_2SO_3$$

5.2.2 Interferents

Carbon disulfide, halogens, mercaptans, phosphine, and phosgene produce similar stains. Acetylene and hydrogen sulfide produce black stains. Sulfur dioxide represents a positive interferent, but does not give a stain by itself.

6 Apparatus

A complete measurement system consists of a compatible detector tube and detector-tube pump. These systems are normally calibrated and supplied by the same manufacturer.

6.1 Detector tubes, containing a reagent that is coated on a solid support and is suitable for a colour-producing reaction by which the mass concentration of carbon monoxide present in the air at work places (or work areas) can be determined in concentrations greater than 10 mg/m³, within the acceptable ranges of temperature, pressure and relative humidity stated by the manufacturer.

Use only detector tubes which

a) are uniformly packed, i.e. uniform packing tightness with no obvious segregation;

b) contain filling layers, the surfaces of which are perpendicular to the tube axis;

c) are free from leaks;

and, in addition, meet the following requirements:

d) length-of-stain detector tubes shall be designed to have an inaccuracy of less than ± 25 % and a length of stain of at least 15 mm at the applicable maximum permissible value, for example Threshold Limit Value (TLV) of the mass concentration of carbon monoxide.

Store the detector tubes in accordance with the manufacturer's instructions.

WARNING — Detector tubes not used within the stability period of the reagent system stated by the manufacturer shall be discarded (see 8.3).

6.2 Detector-tube pump, hand-operated or battery-powered.

Use only the detector-tube pump specified for use with the detector tube, which has a volume per stroke not differing by more than ± 5 % from the nominal volume per stroke, which is free from leaks, and which, when operated, gives the volume flow rate referred to in the calibration of the detector tube.

Handle the detector-tube pump carefully and maintain it according to the manufacturer's instructions. Test the detector-tube pump as specified in 8.1.

NOTE — The intensity-of-colour development in the detector tube is a function of volume flow rate as well as of the total volume of air or other gas mixture passed through the detector tube at a given mass concentration of carbon monoxide. The volume flow rate depends on the suction effect on the detector-tube pump and on the resistance to flow of the detector tube. The opening time per stroke of the detector-tube pump is affected by leaks of the detector-tube pump and particles deposited on the filter or on the wall of the flow channel of the detector-tube pump.

7 Sampling

Test the detector-tube pump (6.2) for leaks by inserting an unopened detector-tube (6.1) into the inlet port of the detector-tube pump and then operating the detector-tube pump. No air should flow in the system. If air-flow occurs, as indicated by, for example, a movement of the detector-tube pump air chamber, leaks may be present in the detector-tube pump, and the detector-tube pump shall be examined and repaired or replaced, as necessary.

Also test for flow blockage by operating the detector-tube pump without a detector tube. The detector-tube pump should operate freely. If not, examine and repair or replace the detector-tube pump.

Break off both ends of the detector tube and insert the opened detector tube, with the prescribed orientation if applicable, into the inlet port of the detector-tube pump. Note that the opened detector tube shall be tightly linked to the detector-tube pump.

Take, by operating the detector-tube pump at the previously determined measurement point, an amount of air the volume of which is prescribed by the number of detector-tube pump strokes to be made, in accordance with the calibration data of the detector tube.

At the end of the sampling period, remove the detector tube from the detector-tube pump. Flush the detector-tube pump with clean air to protect it from the corrosive action of reagents or reaction products which may be released from the detector tube.

NOTE — If a hand-operated dectector-tube pump is specified for use with the detector tube, variations in the air volume flow rates between the individual detector-tube pump strokes should be minimized by operating the detector-tube pump in a uniform manner, and, at the end of each detector-tube pump stroke, an adequate interval of time for equalization of pressure within the detector-tube pump should be allowed.

8 Procedure

8.1 Test of the detector-tube pump

Evaluate at stated intervals of time, or more often if there is any reason to suspect the pump performance:

a) the volume per stroke (3.2) of the detector-tube pump, with a representative detector tube (6.1) in place, using a soap-bubble flowmeter the capacity of which is at least 100 ml and which is equipped with a scale with scale intervals of 0,5 ml. For this test, connect the suction port of the

detector-tube pump hermetically to the exit port of the soap-bubble flowmeter;

b) (additionally, for hand-operated detector-tube pumps) the opening time per stroke (3.3) with a representative detector tube (6.1) attached to the detector-tube pump, in accordance with the manufacturer's instructions.

Compare the measured values obtained with the corresponding calibration data of the detector-tube pump and the detector tube. If these data do not agree within the limits stated in 6.2 the detector-tube pump shall be serviced or reconditioned.

NOTE — The resistance to flow of the detector tube and thus the opening time per stroke differs according to the type of detector tube used.

8.2 Determination

Immediately after completion of sampling, hold the used detector tube next to an unused detector tube (6.1) against a white background in adequate lighting, but not in direct sunlight, and proceed as specified in 8.2.1 and 8.2.2.

8.2.1 Evaluation of detector tube indication

The evaluation of the detector tube indication should be made only by a person trained and experienced in detector tube reading.

Evaluate the length of stain obtained by comparison with lengths of stain associated with known concentrations of carbon monoxide, usually marked on the detector tube. Apply the following rules:

a) if the leading edge of the stain is not sharply defined, take the reading at the point where slight discoloration can just be distinguished from the colour of the unreacted reagent system;

b) if the leading edge of the stain is not perpendicular to the detector tube axis, take the mean resulting from the shortest and the longest length of stain, provided that the difference between the shortest and longest length of stain is not greater than 20 % of the mean. Discard the detector tube reading in cases where this requirement is not fulfilled.

Record the concentration of carbon monoxide associated with the length of stain obtained.

8.2.2 Evaluation of correction factors

The length of stain obtained and intensity of colour developed may be a function of temperature, pressure and relative humidity of air sample and may also be affected by substances other than carbon monoxide present in the air sample.

In some cases, the interfering effects shall be taken into account by applying correction factors. Therefore, evaluate, if necessary, correction factors appropriate for interpreting the detector tube reading, referring to, for example, correction factors supplied by the manufacturer of the detector tube.

8.3 Disposal of detector tubes

Dispose of detector tubes with due regard to the toxic or corrosive reagent or reaction products contained in the tubes, and in accordance with the manufacturer's disposal instruction and national requirements.

9 Expression of results

9.1 Calculation

The concentration of carbon monoxide in the air sample is given by the detector tube indication evaluated according to clause 8. This concentration shall be given as mass concentration $\varrho(\text{CO})$, expressed in milligrams per cubic metre.

NOTE — The mass concentration of carbon monoxide $\varrho(\text{CO})$, expressed in milligrams per cubic metre, is related to the volume concentration $\varphi(\text{CO})$, expressed in parts per million (1 ml per 10^6 ml), by the formula

$$\varrho(\text{CO}) = \frac{28,0 \times p \times 293,2}{24,05 \times (\theta + 273,2) \times 101,3} \times \varphi(\text{CO})$$

$$= \frac{3,37 \times p}{\theta + 273,2} \times \varrho(\text{CO})$$

where

p is the pressure, in kilopascals, of the air sample;

θ is the temperature, in degrees Celsius, of the air sample;

28,0 is the molar mass, in grams per mole, of carbon monoxide;

24,05 is the molar volume, in litres per mole, of an ideal gas at 293,2 K and 101,3 kPa.

At 293,2 K and 101,3 kPa the conversion factors for carbon monoxide are

1 ppm = 1,16 mg/m³

1 mg/m³ = 0,86 ppm

9.2 Precision

Relative errors in detector tube measurements are greater at the lower end of the measuring range than at the higher end. The maximum permissible random error, expressed as twice the relative standard deviation (coefficient of variation) of detector tube measurements specified in this International Standard is ± 25 % at the mass concentration level of carbon monoxide of 60 mg/m³. Some of the major sources of variation in detector tube measurements are:

a) variability of the internal diameter of the glass tubes; for example, a tolerance of ±0,1 mm is a ±4 % variation in a 5 mm internal diameter detector tube;

b) variability of the filling layer containing the reagent system for the colour-producing reaction; important parameters are, for example, purity and uniformity of distribution of the reagent system, uniformity of particle size and bulk density of the solid substrate, and alignment of the filling layer in the detector tube;

c) variability of the air volume and air volume flow rate due to the flow characteristics of the detector-tube pump.

10 Test report

The test report shall include at least the following information:

a) identification data for the air sample;

b) reference to this International Standard;

c) location in which the air sample was taken and whether personal or general area;

d) identification of the manufacturer of the detector tube and detector-tube pump used, the manufacturer's detector tube batch number, and the detector tube expiry date;

e) start time and end time of the sampling period;

f) number of detector-tube pump strokes or volume of the air sample;

g) temperature, pressure and relative humidity of the air sample;

h) interferents known or suspected to be present in the air sample;

i) mass concentration of carbon monoxide, expressed in milligrams per cubic metre, as determined by the measurement with the detector tube;

j) name of the person who performed the measurement.

The test report shall also contain, if available, the mass concentration of carbon monoxide, expressed in milligrams per cubic metre, as determined by a method other than measurements with detector tubes.

Annex A

(normative)

Calibration of detector tubes

Detector tubes suitable for the determination of the concentration of carbon monoxide present in the air at work places (or work areas) as commercially available and specified in 6.1 have been calibrated by the manufacturer of the detector tubes using calibration gas mixtures. These shall be mixtures of carbon monoxide and a complimentary gas (e.g. purified air or nitrogen). The composition of these mixtures shall be known within specified limits. At the request of the user, the manufacturer of the detector tubes shall supply the calibration data for the detector tubes. Therefore, recalibration of detector tubes is required only in exceptional cases.

Recalibration of detector tubes can be carried out by the user of the detector tubes provided that staff trained and experienced in preparing calibration gas mixtures are available. Some remarks on carrying out the calibration of detector tubes specified in 6.1 are given below.

Prepare calibration gas mixtures following one of the static or dynamic methods listed in annex B or, preferably, by the use of standardized compressed gas cylinders containing, for example, 0,1 % (V/V) carbon monoxide (CO) in nitrogen (N_2) in a dynamic gas flow mixing system. Prepare a zero gas and several gas mixtures at different concentration levels of carbon monoxide within the accuracy of ±5 % covering the working range, for example 10 mg/m³, 20 mg/m³, 40 mg/m³, 60 mg/m³, 100 mg/m³ and 200 mg/m³.

NOTE — The concentration of carbon monoxide in the gas mixture generated should be independently verified by, for example, an electrochemical method (see ISO 8519[10]).

At each of the selected concentration levels, take at least five samples as follows:

Test the detector-tube pump (6.2) for leaks by inserting an unopened detector tube (6.1) into the inlet port of the detector-tube pump and then operating the detector-tube pump. No air should flow in the system. If air-flow occurs, as indicated by, for example, a movement of the detector-tube pump air chamber, leaks may be present in the detector-tube pump, and the detector-tube pump shall be examined and repaired or replaced, as necessary. Also test for flow blockage by operating the detector-tube pump without a detector tube. The detector-tube pump should operate freely. If not, examine and repair or replace the detector-tube pump. Break off both ends of the detector tube and connect the opened detector tube, with the prescribed orientation if applicable, to the device used for generating the calibration gas mixture by a butt-to-butt glass joint with, for example, a polyvinyl chloride connection. Insert the free end of the detector tube into the inlet port of the detector-tube pump. Note that the detector tube shall be tightly linked to the detector-tube pump and proceed as specified in clause 7.

Immediately after completion of sampling, hold the detector tube next to an unused detector tube (6.1) against a white background in adequate lighting, but not in direct sunlight, and proceed as specified in 8.2.1.

For length-of-stain detector tubes, prepare a calibration graph by plotting the length of stain observed in the tube versus the mass concentration φ(CO), expressed in milligrams per cubic metre, or volume concentration ϱ(CO), expressed in parts per million, of carbon monoxide present in the calibration gas mixtures.

Record the volume of gas mixture passed through the detector tube, as well as the sampling period.

Evaluate the range of values of temperature and the range of values of relative humidity for which the calibration graph (see ISO 6879) is valid and, in addition, evaluate calibration graphs for further values of temperature and relative humidity. Proceed as specified above.

NOTE — The calibration graphs should be valid for gas mixtures having a temperarure of between 18 °C and 30 °C and a relative humidity of approximately 50 %.

Evaluate the repeatibility within the laboratory of the detector tube measurements at, for example, mass concentrations of carbon monoxide of 10 mg/m³, 20 mg/m³, 40 mg/m³, 60 mg/m³, 100 mg/m³ and 200 mg/m³, using the results of at least five detector tube determinations at each of the selected concentration levels.

Determine substances which are potential interferents in the use of detector tubes (see 5.1.2 and 5.2.2). Evaluate their effects on the detector tube reading as specified above and record them.

NOTE — In cases where the effects of interferents can be reduced or eliminated by connecting a pretreatment tube containing a suitable reagent coated on a solid support in series with the detector tube, corresponding calibration graphs for the detector tube and the pretreatment tube shall be prepared.