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

*Air des lieux de travail — Détermination de la concentration en masse
du dioxyde d'azote — Méthode utilisant des tubes détecteurs pour
échantillonnage rapide à lecture directe*



Reference number
ISO 8761:1989(E)

Foreword

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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 8761 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.

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Introduction

The determination of the mass concentration of nitrogen dioxide present in the air at work places (work areas or stationary posts) 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 *N,N'*-diphenyl-benzidine, *N*-(1-naphthyl)-ethylenediamine dihydrochloride, and *o*-tolidine.

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Work-place air — Determination of mass concentration of nitrogen dioxide — 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 nitrogen dioxide present in the air at work places (work areas or stationary posts) within the range of approximately, 1 mg/m³ to 50 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.

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 indicated 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 nitrogen dioxide 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 nitrogen dioxide 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 nitrogen dioxide by comparing the observed length of stain developed to lengths of stain prepared using calibration gas mixtures, taking into account the effect of interferences on the instrument reading and the correction factors for pressure, temperature and relative humidity.

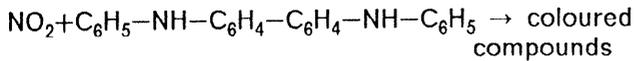
5 Reactions and interferences

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

5.1 *N,N'*-diphenylbenzidine reaction

Tubes containing a reagent system based on *N,N'*-diphenylbenzidine are length-of-stain detector tubes; the change of intensity of colour is from grey to blue-grey.

5.1.1 Reaction equation



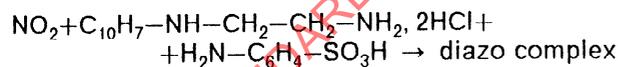
5.1.2 Interferents

Chlorine and ozone produce stains similar to those produced by nitrogen dioxide, but with lower sensitivity. For example, at the mass concentration of chlorine of 3 mg/m³, a length of stain corresponding to the length of stain at the mass concentration of nitrogen dioxide of 0,8 mg/m³ is produced; at the mass concentration of chlorine of 6 mg/m³, a length of stain corresponding to the length of stain at the mass concentration of nitrogen dioxide of 1,6 mg/m³ is produced; and at the mass concentration of ozone of 2 mg/m³, a length of stain corresponding to the length of stain at the mass concentration of nitrogen dioxide of 0,4 mg/m³ is produced.

5.2 *N*-(1-naphthyl)-ethylenediamine dihydrochloride reaction

Tubes containing a reagent system based on *N*-(1-naphthyl)-ethylenediamine dihydrochloride are length-of-stain detector tubes; the change of intensity of colour is from white to red.

5.2.1 Reaction equation



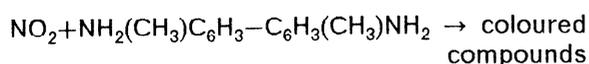
5.2.2 Interferents

Chlorine and ozone are positive interferents.

5.3 *o*-Tolidine reaction

Tubes containing a reagent system based on *o*-tolidine are length-of-stain detector tubes; the change of intensity of colour is from white to yellow-orange.

5.3.1 Reaction equation



5.3.2 Interferents

Halogens and chlorine dioxide and most other oxidizing agents produce stains similar to that produced by nitrogen dioxide. Sulfur dioxide at mass concentrations greater than 100 mg/m³ bleach the stain produced by nitrogen dioxide.

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 nitrogen dioxide present in the air at work places (work areas or stationary posts) can be determined, with measuring range approximately 1 mg/m³ to greater than 50 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 requirement:

- d) length-of-stain detector tubes shall be designed to have an inaccuracy of less than $\pm 25\%$ 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 nitrogen dioxide.

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 $\pm 5\%$ 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 1 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 nitrogen dioxide. 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 2 If a hand-operated detector-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 performances:

- a) the volume per stroke (see 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 3 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 nitrogen dioxide, 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 nitrogen dioxide 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 nitrogen dioxide 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 nitrogen dioxide in the air sample is given by the detector tube indication evaluated according to clause 8. This concentration shall be given as mass concentration ρ (NO₂), expressed in milligrams per cubic metre.

NOTE 4 The mass concentration of nitrogen dioxide ρ (NO₂), expressed in milligrams per cubic metre, is related to the volume concentration φ (NO₂), expressed in parts per million (1 ml per 10⁶ ml), by the formula

$$\rho(\text{NO}_2) = \frac{46,0 \times p \times 293,2}{24,05 \times (\theta + 273,2) \times 101,3} \times \varphi(\text{NO}_2)$$

$$= \frac{5,536 \times p}{\theta + 273,2} \times \varphi(\text{NO}_2)$$

where

- p is the pressure, in kilopascals, of the air sample;
- θ is the temperature, in degrees Celsius, of the air sample;
- 46,0 is the molar mass, in grams per mole, of nitrogen dioxide;
- 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 nitrogen dioxide are

$$1 \text{ ppm} = 1,91 \text{ mg/m}^3$$

$$1 \text{ mg/m}^3 = 0,52 \text{ ppm}$$

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 nitrogen dioxide of 6 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 $\pm 0,1$ 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) interferences known or suspected to be present in the air sample;
- i) mass concentration of nitrogen dioxide, 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 nitrogen dioxide, expressed in milligrams per cubic metre, as determined by a method other than measurements with detector tubes.

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