

Workplace atmospheres — Short term detector tube measurement systems — Requirements and test methods

The European Standard EN 1231 : 1996 has the status of a
British Standard

ICS 13.040.30

Committees responsible for this British Standard

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Chemical Industries Association
Chief and Assistant Chief Fire Officers' Association
Health and Safety Executive
Ministry of Defence
Safety Equipment Association

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Contents

	Page
Committees responsible	Inside front cover
National foreword	ii
Foreword	2
Text of EN 1231	3

National foreword

This British Standard has been prepared by Technical Committee PH/10, and is the English language version of EN 1231 : 1996 *Workplace atmospheres — Short term detector tube measurement systems — Requirements and test methods*, published by the European Committee for Standardization (CEN). It supersedes BS 5343 : Part 1 : 1986 which is withdrawn.

Cross-references

Publication referred to	Corresponding British Standard
EN 482	BS EN 482 : 1994 <i>Workplace atmospheres — General requirements for the performance of procedures for the measurement of chemical agents</i>
EN 50014	BS EN 50014 : 1993 <i>Electrical apparatus for potentially explosive atmospheres — General requirements</i>

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 10, an inside back cover and a back cover.

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Descriptors: Air pollution, air, quality, atmospheres, workroom, accident prevention, measurements, concentration, chemical compounds, detectors, tubes, pumps, specifications, tests, testing conditions, mechanical strength, thermal stability, packing

English version

Workplace atmospheres — Short term detector tube measurement systems — Requirements and test methods

Air des lieux de travail — Systèmes de mesurage
par tube détecteur à court terme — Exigences et
méthodes d'essai

Arbeitsplatzatmosphäre — Kurzzeitprüfröhrchen —
Meßeinrichtungen — Anforderungen und
Prüfverfahren

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 137, Assessment of workplace exposure, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 1997, and conflicting national standards shall be withdrawn at the latest by June 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Contents

	Page
Foreword	2
0 Introduction	3
1 Scope	3
2 Normative references	3
3 Definitions	3
4 Requirements	4
5 General test conditions	5
6 Test methods	6
7 Test report	8
8 Marking	8
Annex A (informative) Examples for the determination of the stroke volume	9
Annex B (informative) Bibliography	10

0 Introduction

In connection with EN 482, a need has been identified to produce a standard for the detector tube method of determining the concentration of chemical agents in workplace air.

A short term detector tube measurement system consists of a detector tube connected to a compatible detector tube pump. When workplace air containing a particular chemical agent is drawn through the tube, a colour change, corresponding to the concentration, takes place.

Short term detector tube measurement systems have many applications. This European Standard refers to detector tubes used for workplace air monitoring. These tubes may be used for the measurement tasks laid down in EN 482 namely 'Measurement for comparison with limit value' and 'Periodic measurements', provided that the limit value of the chemical agent of interest is covered by the measuring range of the individual detector tube and the requirements for overall uncertainty are complied with for that measuring range. The other measurement tasks as described in EN 482, having different requirements, for overall uncertainty may be treated accordingly.

NOTE. This European Standard may be used to assess the performance of detector tube measurement systems used for other applications such as emission measurements and compressed air or technical gas analysis.

To cover the measuring range in accordance with EN 482, a combination of two detector tubes with restricted but complementary and overlapping measuring ranges may also be used.

It is the user's primary responsibility to choose appropriate procedures or devices that meet the requirements in this European Standard. One way of doing this is to obtain information or confirmation from the manufacturer.

Materials used in the construction of the detector tube pump should be such that it remains functional for a period of at least three years when used in accordance with the manufacturer's instructions.

This European Standard will enable manufacturers, test houses, certification bodies and users to adopt a consistent approach to the assessment of performance. It is the manufacturer's primary responsibility to ensure that detector tubes meet the performance requirements under specified laboratory conditions.

1 Scope

This European Standard specifies performance requirements and test methods under prescribed laboratory conditions for length of stain detector tubes and their associated pump (detector tube measurement system) used for short term measurements of the concentration of specified chemical agents in workplace air.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred to applies.

- EN 482 *Workplace atmospheres — General requirements for the performance of procedures for the measurement of chemical agents*
- EN 50014 *Electrical apparatus for potentially explosive atmospheres — General requirements*
- ISO 7504 *Gas analysis — Vocabulary*

3 Definitions

For the purposes of this standard, the following definitions apply:

3.1 chemical agent, overall uncertainty, specified measuring range, true value

Definitions for these terms are as in EN 482.

3.2 length of stain detector tube (in the following called 'detector tube')

A glass tube containing chemical reagents in which a colour change may be produced when a sample of the atmosphere is drawn through it. The length of the stain produced, relative to a graduated scale, provides a measure of the concentration of a specified chemical agent in air.

3.3 short term detector tube

A detector tube that provides a means of obtaining a rapid measurement (typically up to 15 min) of the concentration of a specified chemical agent in air.

3.4 detector tube measurement system

A complete measurement system consisting of a detector tube (see 3.2) and a detector tube pump.

3.5 interferent

Any component of the air sample, excluding the analyte(s), affecting the detector tube reading.

3.6 calibration gas mixture

A gas mixture of known composition P, generally comprising one or more calibration components and a complementary gas (ISO 7504).

3.7 complementary gas

Generally the most abundant component (pure or gas mixture) which makes up the mixture intended for the calibration (ISO 7504).

4 Requirements

NOTE. A functional detector tube measurement system consists of a detector tube and an appropriate pump. Usually the detector tube measurement system is calibrated and supplied by the same manufacturer.

4.1 Detector tubes

4.1.1 Overall uncertainty

At the lower limit of the specified measuring range, the relative overall uncertainty shall be $\leq 50\%$; at 20% and above of the specified measuring range the relative overall uncertainty shall be $\leq 30\%$.

4.1.2 Specified measuring range

The manufacturer shall specify the measuring range in which the detector tube complies with the requirements for the overall uncertainty given in 4.1.1. The marking of the specified measuring range on the detector tube shall correspond with the range(s) indicated in the manufacturer's operating instruction. Where the specified measuring range can be extended this shall be clearly stated in the operating instructions.

4.1.3 Reusable tubes

For reusable detector tubes the manufacturer shall specify the time period during which the detector tube can be used for a subsequent measurement if the previous used gave zero reading. For this period of time the detector tube shall fulfil the requirements of 4.1.1 and 4.1.5.

4.1.4 Scale

The scale shall have a minimum of three lines perpendicular to the axis of the tube and shall be marked with concentration values or equivalent. The lines shall have a maximum width of 0,7 mm (with the exception of the line at the beginning of the indicating layer); the minimum length of a line shall be 3 mm and the size of printing a minimum of 1,5 mm.

The requisite number of pump strokes or sample volume for a particular scale shall be marked.

Detector tube scales shall be graduated either in volume per unit volume or mass per unit volume or shall be accompanied by a calibration graph in the same units.

4.1.5 Evaluation of the stain

The stain shall remain constant and clearly visible for a minimum period of 2 min after the measurement of the contaminated atmosphere.

At 20% of the measuring range and above, the maximum variation of stain length around the circumference of the tube at the interface between the stained and unstained indicating layer shall not exceed 25% of the stain length (measured in millimetres) when measured at its point of maximum length.

4.1.6 Shelf life

The shelf life of the detector tube, when stored in accordance with the manufacturer's instructions, shall be clearly indicated on the tube packet. At the end of the shelf life the detector tube shall comply with the requirements of 4.1.1 and 4.1.5.

4.1.7 Mechanical strength

Following the tests in 6.1.7 the detector tubes shall maintain their integrity and shall comply with the requirements of 4.1.1 and 4.1.5.

4.1.8 Temperature stability

After storage of the detector tubes at -5°C for 24 h and subsequently at $+60^{\circ}\text{C}$ for 24 h the tubes shall, after stabilizing to ambient temperature, meet the requirements of 4.1.1 and 4.1.5.

4.1.9 Packing of the detector tubes

If the box contains more than one tube it shall be re-closable.

If the manufacturer indicates that the tubes shall be protected from light, this shall be ensured by the box. Following the tests in 6.1.7 the box containing the detector tubes shall maintain its integrity.

4.1.10 Interferences

Information on the influence of typical interferences shall be provided by the manufacturer in the instructions for use (see 4.1.13c).

Information shall be given on interferences likely to suppress the full development of the stain.

4.1.11 Overloading

When the detector tube is tested at a concentration ten times the upper limit of the specified measuring range, the detector tube shall clearly indicate an overload, lasting at least 2 min.

4.1.12 Climatic influences

The manufacturer shall state the range of temperature and humidity for which the specified measuring range is valid. The temperature range shall be at least from 10°C to 30°C and the range of relative humidity shall be between at least 20% and 80%.

Outside these ranges the requirements of 4.1.1 and 4.1.5 may be fulfilled by the use of correction factors (see 6.1.12).

4.1.13 Instructions for use

The instruction for use supplied with each box of detector tubes shall be in the language(s) of the country where the detector tube is to be marketed. It shall contain at least the following information:

- a) directions for proper handling of a tube including opening and fitting it into the pump;
- b) a statement that the detector tube pump shall be tested for leakage before each use;
- c) general information on the reaction and colour change involved in the system and the levels at which other typical gases and vapours, including water, are likely to interfere to the extent of increasing the overall uncertainty above the level specified in this European Standard;
- d) a statement that additional information on possible interferences can be provided on request;
- e) if applicable, information about reagents and reactions that are hazardous;
- f) where the contents of tubes present a disposal hazard, a warning to that effect shall be given together with advice that national regulations for disposal of hazardous waste should be followed;
- g) a statement on the time required for the completion of one pump stroke;
- h) a statement about the limitations of reuse;
- i) information on the evaluation of the reading including calculation of results and correction factors for temperature and pressure, if applicable;
- j) reference to the operating instructions of the detector tube pump(s);
- k) specification of the detector tube pump(s);
- l) information on storage and transport.

4.2 Detector tube pump

4.2.1 Stroke volume

When tested in accordance with 6.2.1, the pump shall sample a volume of air within (100 ± 5) ml per stroke.

4.2.2 Leakage

The detector tube pump with the closed detector tube connection shall be tight, so that during the first minute of a pump stroke the leakage rate does not exceed 3 ml/min.

4.2.3 Time and end of stroke

The time of stroke of a detector tube pump, when used against a flow control, typical for the detector tube (see 5.5) shall be within $\pm 20\%$ of the mean value.

The end of stroke duration shall be clearly recognizable. The criteria shall be given in the instructions for use.

4.2.4 Mechanical strength

After the test given in 6.2.4, the pump shall meet the requirements in 4.2.1, 4.2.2 and 4.2.3.

4.2.5 Mechanical function

The pump shall meet the requirements of 4.2.1, 4.2.2 and 4.2.3 after the execution of 5000 strokes when fitted with the resistance in 5.5.

4.2.6 Explosion hazard (electrically driven pumps only)

If the pump is claimed by the manufacturer to be suitable for use in areas subject to explosion hazard, it shall fulfil the requirements of EN 50014.

4.2.7 Instructions for use

The instructions for use supplied with the pump shall be in the language(s) of the country where the pump is to be marketed. It shall contain at least the following information:

- a) operating instructions;
- b) instructions for testing for leakage before each use;
- c) maintenance instructions;
- d) reference to the operating instructions of the detector tube;
- e) stroke volume;
- f) the criterion for the end of one stroke.

5 General test conditions

NOTE. Parts of the detector tube measurement system which have already been tested to this European Standard are not required to be tested again.

5.1 Reagents

Calibration gas mixtures shall be prepared according to ISO Standards or other accepted methods (see annex B for corresponding ISO Standards).

5.2 Apparatus

5.2.1 Usual laboratory apparatus and resources.

5.2.2 An exposure chamber constructed of inert materials such as glass or PTFE, through which the generated atmosphere is passed.

5.2.3 Provisions for measuring, controlling and varying systematically the rate of air flow through the generating system and the composition, temperature and relative humidity of the calibration gas mixture (see annex B for corresponding ISO Standards).

5.3 Independent method

An independent validated method shall be used to verify the composition of the calibration gas mixture used.

The composition of the calibration gas mixture and the uncertainty shall be given in the test report.

5.4 Test conditions for detector tubes

Unless otherwise stated, the test procedures are run at the following climatic conditions:

- temperature: $(22 \pm 3) ^\circ\text{C}$;
- humidity: $(10 \pm 2) \text{ g/m}^3$;
- pressure: $(1013 \pm 30) \text{ hPa}$.

At a pressure outside of the stated range, the measured values have to be corrected to a pressure of 1013 hPa.

5.5 Test conditions for pumps

One pump shall be tested. Tests according to 6.2.1, 6.2.3 and 6.2.5 are carried out with a resistance, typical for detector tubes when connected to the pump, with a pressure drop of either:

- a) $(140 \pm 10) \text{ hPa}$, flow rate 500 ml/min; or
- b) $(430 \pm 30) \text{ hPa}$, flow rate 100 ml/min.

6 Test methods

6.1 Detector tubes

6.1.1 Overall uncertainty

Prepare three test concentrations of the appropriate calibration gas mixture at the lower and upper limit and at 20 % of the specified measuring range. Calculate the relative overall uncertainty, expressed as a percentage, using the following formula (see EN 482):

$$\frac{|\bar{x} - x_{\text{ref}}| + 2s}{x_{\text{ref}}} \times 100$$

where

- \bar{x} is the mean value of results of a number n of repeated measurements;
- x_{ref} is the true or accepted reference value of concentration;
- s is the standard deviation of n measurements.

For detector tubes with two specified measuring ranges, the test procedure is performed for each range.

NOTE. The uncertainty of the calibration gas mixture concentration should be considered when assessing the overall uncertainty of the detector tube system using appropriate statistical calculations.

6.1.2 Specified measuring range

Determine whether the measuring range specified in the instruction for use and on the detector tube are complete and identical.

6.1.3 Reusable tubes

Test ten detector tubes with complementary gas up to the maximum volume stated in the user's instruction. Then determine the overall uncertainty according to 6.1.1 at a concentration between 20 % and 30 % of the specified measuring range. For detector tubes with several measuring ranges perform the test for the lowest one.

6.1.4 Scale

Measurement and visual check.

6.1.5 Evaluation of the stain

In each series of measurements in 6.1.1 observe the coloured zones of two tubes for 2 min.

If any of the detector tubes used for the measurement according to 6.1.1 shows a significant distortion of the colour zone, the biggest (l_{max}) and the smallest (l_{min}) length of the colour zone is measured in millimetres and the distortion determined as a percentage using the following formula:

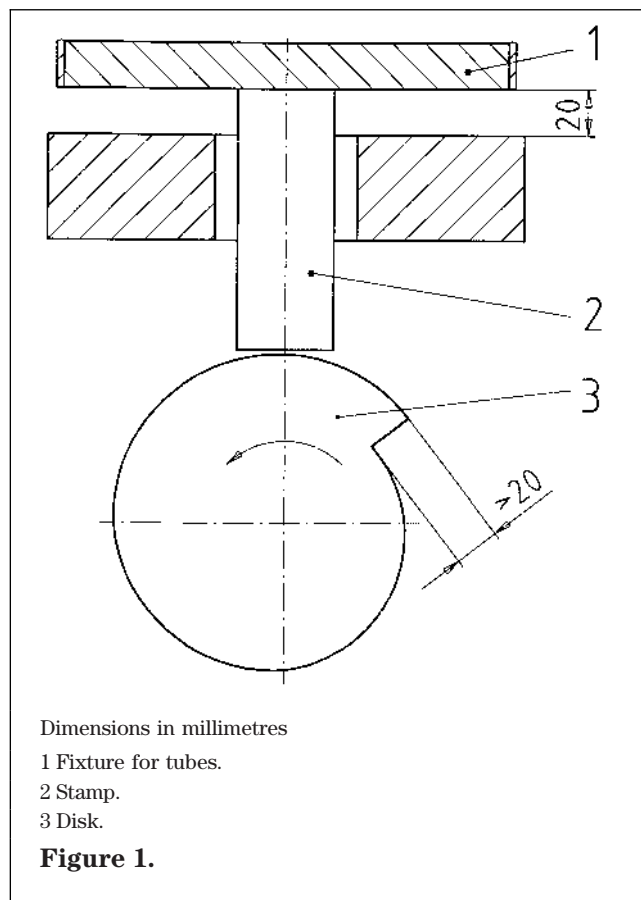
$$\frac{l_{\text{max}} - l_{\text{min}}}{l_{\text{max}}} \times 100$$

6.1.6 Shelf life

Within the last three months of the shelf life specified by the manufacturer testing according to 6.1.1 and 6.1.5 shall be carried out. Test with ten detector tubes and at a concentration between 20 % and 30 % of the specified measuring range.

6.1.7 Mechanical strength

6.1.7.1 Equipment



6.1.7.2 Procedure

Insert the detector tubes, still in the unopened detector tube package as they are delivered, into the test equipment horizontally and vertically and fix tightly. Gross test mass of the sample holding device with stamp is $3 \text{ kg} \pm 0,5 \text{ kg}$.

2000 strokes per test are generated. The frequency is chosen, in a way that the detector tube packages come to a standstill between two strokes, e.g. 2000 strokes in 15 min. After visual examination of filling layers and holding elements, examine ten detector tubes from each of the two tests, regarding reading and measurement deviation according to 6.1.1 and 6.1.5. Carry out the tests at a concentration between 20 % and 30 % of the specified measuring range. For detector tubes with different measuring ranges the testing is performed for the lowest one.

6.1.8 Temperature stability

Store the detector tubes for successive 24 h periods at -5°C and $+60^\circ\text{C}$. After adjusting to room temperature test ten detector tubes regarding reading and measurement deviation according to 6.1.1 and 6.1.5. Carry out the tests at a concentration between 20 % and 30 % of the specified measuring range. For detector tubes with different measuring ranges the testing is performed for the lowest one.

6.1.9 Packing of the detector tubes

Visual check.

6.1.10 Interferences

Check if this information is included in the instructions for use (see 4.1.13c).

NOTE. When information on interferences likely to suppress the full development of the stain is included in the instruction for use, potential effects and the test requirements should be considered.

6.1.11 Overloading

Three detector tubes are tested at concentration 10 times the upper limit of the specified measuring range. Testing is performed with the undiluted measuring component if the upper limit of the specified measuring range corresponds to a volume concentration of 10 % or more.

For detector tubes with more than one scale, the test is performed by using the scale with the highest scale end value at the appropriate stroke number.

6.1.12 Climatic influences

The reading and the measurement deviation are tested according to 6.1.1 and 6.1.5 with ten detector tubes at each of the following climatic conditions:

- a) temperature: $(10 \pm 2)^\circ\text{C}$;
pressure: $(1013 \pm 30) \text{ hPa}$;
humidity: $(2 \pm 0,5) \text{ g/m}^3$;
- b) temperature: $(30 \pm 2)^\circ\text{C}$;
pressure: $(1013 \pm 30) \text{ hPa}$;
humidity: $(25 \pm 2) \text{ g/m}^3$.

Test at a concentration between 20 % and 30 % of the specified measuring range. For detector tubes with several specified measuring ranges, the test is performed for each range.

If the manufacturer states a wider range of climatic conditions in the instruction for use (see 4.1.12), the wider range shall be applied for the test instead of the indicated figures.

6.1.13 Instructions for use

Check the information for completeness and correctness.

6.2 Detector tube pumps

6.2.1 Stroke volume

Test with a volume measuring device with a range of at least 90 ml to 110 ml and an uncertainty of at most $\pm 1 \text{ ml}$. The stroke volume is measured at the end of a pump stroke as indicated in the instructions for use, a second value is measured 30 s later. Both values shall be in a range of $(100 \pm 5) \text{ ml}$. Test each detector tube pump six times.

NOTE. Examples of suitable methods are given in annex A.

6.2.2 Leakage

6.2.2.1 Pressure test

Connect the detector tube pump tightly to a manometer. Then start a pump stroke. Read the pressure drop immediately. After one minute read the pressure drop again.

6.2.2.2 Volume test

Test with a volume measuring device according to 6.2.1. The detector tube pump is connected to the instrument via a cut-off valve. A pump stroke is started with closed cut-off valve. After one minute the cut-off valve is opened. Volume is measured at the visible end of the pump stroke. The mean value of six repeated measurements shall not differ by more than 3 ml from the mean volume determined in 6.2.1.

6.2.3 Time and end of stroke

Time is measured from the start till the end of a pump stroke as indicated in the instructions for use. Test each detector tube pump six times.

The end of stroke is determined by visual test according to the manufacturer's instructions for use.

6.2.4 Mechanical strength

Test with equipment according to 6.1.7 using 4000 strokes in 40 min. Then the pump is tested according to 6.2.1, 6.2.2 and 6.2.3.

6.2.5 Mechanical function

Connect the detector tube pump and standard flow control to each other followed by 5000 pump strokes according to the instructions for use. Then test the pump according to 6.2.1, 6.2.2 and 6.2.3.

NOTE. Batteries used in electrically operating pumps may need to be recharged or replaced during this test.

6.2.6 *Explosion hazard (electrical driven pumps only)*

Verify by means of a test certificate.

6.2.7 *Instructions for use*

Check the information supplied for completeness and correctness.

7 Test report

7.1 Detector tubes

The test report shall include at least the following information:

- a) a reference to this European Standard;
- b) the type of detector tube;
- c) the type of detector tube pump used;
- d) the specification and uncertainty of the calibration gas mixtures;
- e) the independent method;
- f) the test results;
- g) if the acceptance criteria were met;
- h) any unusual features noted during the determinations;
- i) any operation not included in this European Standard that may have influence on the results.

7.2 Detector tube pumps

The test report shall include at least the following information:

- a) a reference to this European Standard;
- b) the type of detector tube pump;
- c) the test results;
- d) if the acceptance criteria were met;
- e) any unusual features noted during the determinations;
- f) any operation not included in this European Standard that may have influence on the results.

8 Marking

8.1 Boxes

Each box of detector tubes shall be marked with the following information:

- a) the manufacturer's name or trademark and a type identifying mark;
- b) the number of this European Standard;
- c) an indication of the gas(es) or vapour(s) for which the tubes may be used and the measuring ranges;
- d) the manufacturer's batch number;
- e) the expiry date;
- f) recommended storage conditions;
- g) number of tubes;
- h) reference to the instructions for use.

8.2 Detector tubes

Each tube shall be marked with the following information:

- a) the manufacturer's name or trademark and a type identifying mark;
- b) an indication of the gas(es) or vapour(s) for which the tube is designed to be used;
- c) an indication of the direction of gas flow;
- d) scales and measuring units;
- e) the number of pump strokes or sampled volume.

8.3 Pumps

Each pump shall be marked with at least the following information:

- a) the manufacturer's name or trademark and a type identifying mark;
- b) identification number and/or month and year of manufacture;
- c) number of the European Standard.

On the box:

- d) reference to the instructions for use.

Annex A (informative)

Examples for the determination of the stroke volume

A.1 Syringe method of calibration

A.1.1 Principle

The sampled volume when the detector tube pump is operated is measured against the calibrations of a hypodermic syringe.

A.1.2 Apparatus

100 ml graduated hypodermic syringe, additionally graduated at 110 ml, with a Luer fitting, sufficiently accurate to assess compliance with the requirements of 4.2.1.

NOTE. 100 ml hypodermic syringes generally have sufficient barrel length to enable an extra graduation at 110 ml to be added. Normal hypodermic syringes are preferable to gas tight ones, since the former are, in fact, sufficiently gas tight for the purpose of the test and their plungers have a much lower resistance to movement.

A.1.3 Procedure

Withdraw the plunger of the hypodermic syringe to the 110 ml graduation mark. Attach the detector tube pump to a freshly opened detector tube, and attach the tube to the hypodermic syringe, clamped in the horizontal position. Operate the detector tube pump in accordance with the manufacturer's instructions.

Read off the sampled volume from the hypodermic syringe and determine if the pump meets the requirements of 4.2.1.

A.2 Soap bubble flow meter test

A.2.1 Principle

When the detector tube pump is operated, a soap bubble moves up a graduated burette and indicates the volume at its resting point.

A.2.2 Apparatus

- 120 ml graduated burette;
- 50 ml beaker, containing about 25 ml soap solution;
- connecting tube, 6 mm internal diameter.

A.2.3 Procedure

Break off the tips of a detector tube and insert into the detector tube pump with the arrow indicating the direction of flow. Connect the inlet end of the detector tube to the outlet end of the burette by means of the connecting tube. Briefly dip the bottom opening of the burette into the soap solution and operate the pump.

Note the graduation mark on the burette when the bubble ceases to move.

Annex B (informative)

Bibliography

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- [2] ISO 6142 *Gas analysis — Preparation of calibration gas mixtures — Weighing methods*
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List of references

See national foreword.

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