INTERNATIONAL STANDARD

ISO
16900-4

First edition
2011-09-01

Respiratory protective devices — Methods of test and test equipment —

Part 4:
Determination of gas filter capacity and migration, desorption and carbon monoxide dynamic testing

Appareils de protection respiratoire — Méthodes d'essai et équipement d'essai —

Partie 4: Détermination de la capacité d'un filtre à gaz et essais de migration, de désorption et dynamique au monoxyde de carbone

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 16900-4 was prepared by Technical Committee ISO/TC 94, *Personal safety — Protective clothing and equipment*, Subcommittee SC 15, *Respiratory protective devices*.

ISO 16900 consists of the following parts, under the general title *Respiratory protective devices — Methods of test and test equipment*:

— *Part 1: Determination of inward leakage*

— *Part 2: Determination of breathing resistance*

— *Part 3: Determination of particle filter penetration*

— *Part 4: Determination of gas filter capacity, migration, desorption and carbon monoxide dynamic testing*

Introduction

This part of ISO 16900 is intended as a supplement to the specific performance standards for respiratory protective devices. Test methods are specified for complete devices or parts of devices. If deviations from the test method given in this International Standard are necessary, these deviations will be specified in the relevant performance standard.

Respiratory protective devices — Methods of test and test equipment —

Part 4:
Determination of gas filter capacity and migration, desorption and carbon monoxide dynamic testing

1 Scope

This part of ISO 16900 specifies the test method for determining the gas capacity of separate or integral gas filters and combined filters for respiratory protective devices. It includes the validation test at specified flow rates, a desorption test to access the ability of the filter to retain the adsorbed or absorbed gas, and carbon monoxide dynamic testing.

NOTE These tests are conducted in laboratories using specified test agents under specified conditions and therefore do not indicate the performance of the device in actual use.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16972, *Respiratory protective devices — Terms, definitions, graphical symbols and units of measurement*

ISO/TS 21748, *Guidance for the use of repeatability, reproducibility and trueness estimates in measurement uncertainty estimation*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 16972 and the following apply.

3.1
sorption
process in which one substance (the filter medium) takes up or holds another (the test gas), either by adsorption or absorption

3.2
desorption
process in which one substance (the filter medium) releases an absorbed or adsorbed substance

3.3
breakthrough time
time taken from the start of the test until the test gas and specified reaction products are detected at the specified breakthrough concentration at the downstream side of the filter under test

3.4
gas filter capacity
mass or volume of a specific test agent that is removed or retained by a gas filter or combined filter under specified conditions of temperature, humidity, challenge test gas concentration and flow rate

NOTE   The mass or volume is determined by measuring the breakthrough time at a defined breakthrough concentration. The mathematical equation to calculate the gas capacity is:

$$C = V_{fl} \times c_{gas} \times t_{br} \times 10^{-6}$$

where

C is gas capacity (l);

V_{fl} is volume flow rate (l/min);

c_{gas} is gas concentration (ml/m³);

t_{br} is breakthrough time (min).

EXAMPLE $V_{fl} = 30$ l/min

$c_{gas} = 1\ 000$ ml/m³

$t_{br} = 30$ min

$C = 30$ l/min $\times 1\ 000$ ml/m³ $\times 30$ min $\times 10^{-6} = 0{,}9\ l$

3.5
gas filter validation test at specified flow rates
test to evaluate the ability of the filter to achieve a minimum performance level at its work rate classification

3.6
integral dose
volume of the test gas on the effluent side of the filter released during the testing period

NOTE   This is calculated as the integral of the instant effluent concentration (function of time) of the test gas during the testing time multiplied by the volume flow rate.

4   Prerequisites

The performance standard shall indicate the conditions of the test. This includes the following:

a)   number of specimens;

b)   sequence of preconditioning;

c)   type of test method (gas capacity test, gas filter validation test at specified flow rates, migration test A or migration test B, desorption test);

d)   test parameters for gas capacity test (test gas, concentration, reaction products if applicable, breakthrough concentration, breakthrough time, humidity, temperature, flow mode, flow rate, setting of breathing machine);

e)   test parameters for gas filter validation test at specified flow rates (test gas, concentration, reaction products if applicable, breakthrough concentration, breakthrough time, humidity, temperature, flow rate);

f)   test parameters for migration and desorption test (test duration, reaction products if applicable, termination, initial exposure prior to desorption).

5   General test requirements

Unless otherwise specified, the values stated in this part of ISO 16900 are expressed as nominal values. Except for temperature limits, values which are not stated as maxima or minima shall be subject to a tolerance of ±5 %. Unless otherwise specified, the ambient temperature for testing shall be between 16 °C and 32 °C and (50 ± 30) % RH. Any temperature limits specified shall be subject to an accuracy of ±1 °C.

6   Principles

6.1   Sorption tests

6.1.1   Gas capacity test

Gas and combined filters are exposed to a defined test gas under given conditions to determine the time when breakthrough of the applied gas occurs at a specified concentration.

6.1.2   Gas filter validation test at specified flow rates

Gas and combined filters are exposed to a defined test gas under enhanced flow rates to evaluate whether breakthrough time exceeds a specified minimum.

6.2   Migration and desorption tests

6.2.1   Migration test A and migration test B

Gas and combined filters are exposed to a defined test gas under given conditions. After exposure, the filter is stored under defined conditions. After storage, clean air (migration test A) or test gas (migration test B) is passed through the filter to determine the ability of the filter to retain the test gas.

6.2.2   Desorption test

Gas and combined filters are exposed to a defined test gas under given conditions. After exposure, clean air is immediately passed through the filter to determine the ability of the filter to retain the test gas.

6.3   Carbon monoxide (CO) dynamic testing of type CO filter

The CO gas filter, or combined filter containing type CO, are exposed to CO under given conditions to determine the effluent CO concentration over time and the integral dose.

7   Apparatus

7.1   Apparatus for constant flow

The test apparatus consists of three modules:

a)   challenge gas generator;

b)   test chamber;

c)   detector.

A schematic drawing of an example for a test apparatus is shown in Figure 1.

7.2   Challenge gas generator

The challenge gas concentration can be generated using several methods. These include:

a)   use of a prediluted gas (in air);

b)   dilution of a gas by a carrier gas (air);

c)   evaporation of a chemical substance into carrier gas (air);

d)   *in situ* preparation by a chemical reaction where the product is taken up into carrier gas (air).

It shall be taken into account that the stabilization of the test gas concentration takes some time due to adsorption of test gas at the walls of the gas-generating system and the test chamber.

7.3 Test chamber

The filter test chamber shall be sufficiently large to accommodate the filter system under test and shall allow a homogeneous filter exposure.

The construction of the chamber shall be resistant to the test gas, shall be leak-tight and shall safely withstand any pressures, either positive or negative, that might be generated.

Specimen orientation shall be such that the gas flows horizontally and in line with the direction of minimum bed depth of the filter. A gas stream shall not directly impact on to the filter face.

7.4 Detector

The response time of the detection system, which includes sampling lines and connections, shall be known and taken into consideration.

The detector shall have sufficient sensitivity and resolution to accurately determine 10 % of the specified breakthrough concentration.

Key

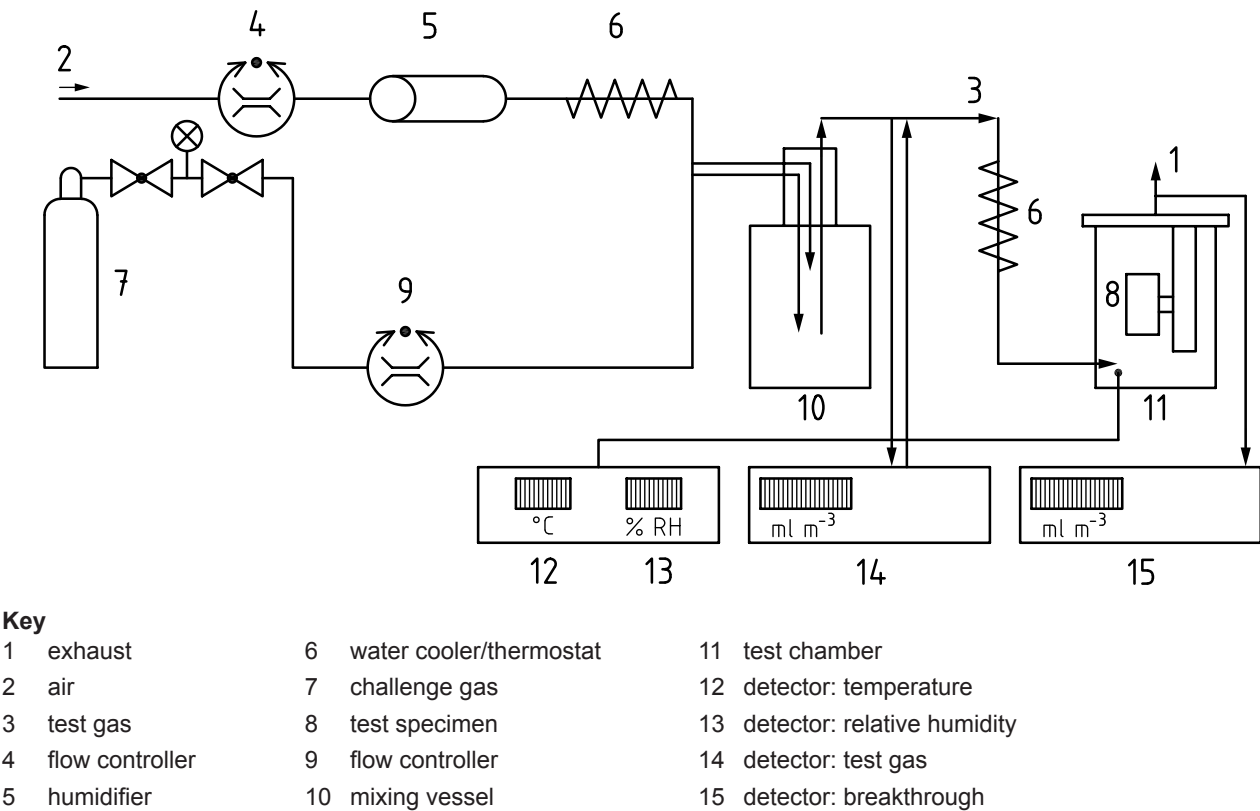|					
1	exhaust	6	water cooler/thermostat	11	test chamber
2	air	7	challenge gas	12	detector: temperature
3	test gas	8	test specimen	13	detector: relative humidity
4	flow controller	9	flow controller	14	detector: test gas
5	humidifier	10	mixing vessel	15	detector: breakthrough

Figure 1 — Schematic diagram of typical test apparatus for constant flow gas capacity testing

7.5 Apparatus for dynamic flow

Schematic diagrams of test arrangements that have been found suitable are represented by Figure 2. They mainly consist of:

a)   breathing machine equipped with control valves;

b)   humidifier;

c)   flow meters for air and test gas;

d)   test chamber equipped with sampling ports and exhaust;

e)   test gas analysers;

f)   means of measuring pressure, wet bulb and dry bulb temperature and moisture content;

g)   head form or suitable adapter;

h)   supply of test gas;

i)   adapter for replaceable inhalation/exhalation valve, if applicable.

Figure 2 — Schematic diagram of typical test equipment for cyclic CO capacity testing using filter adapter
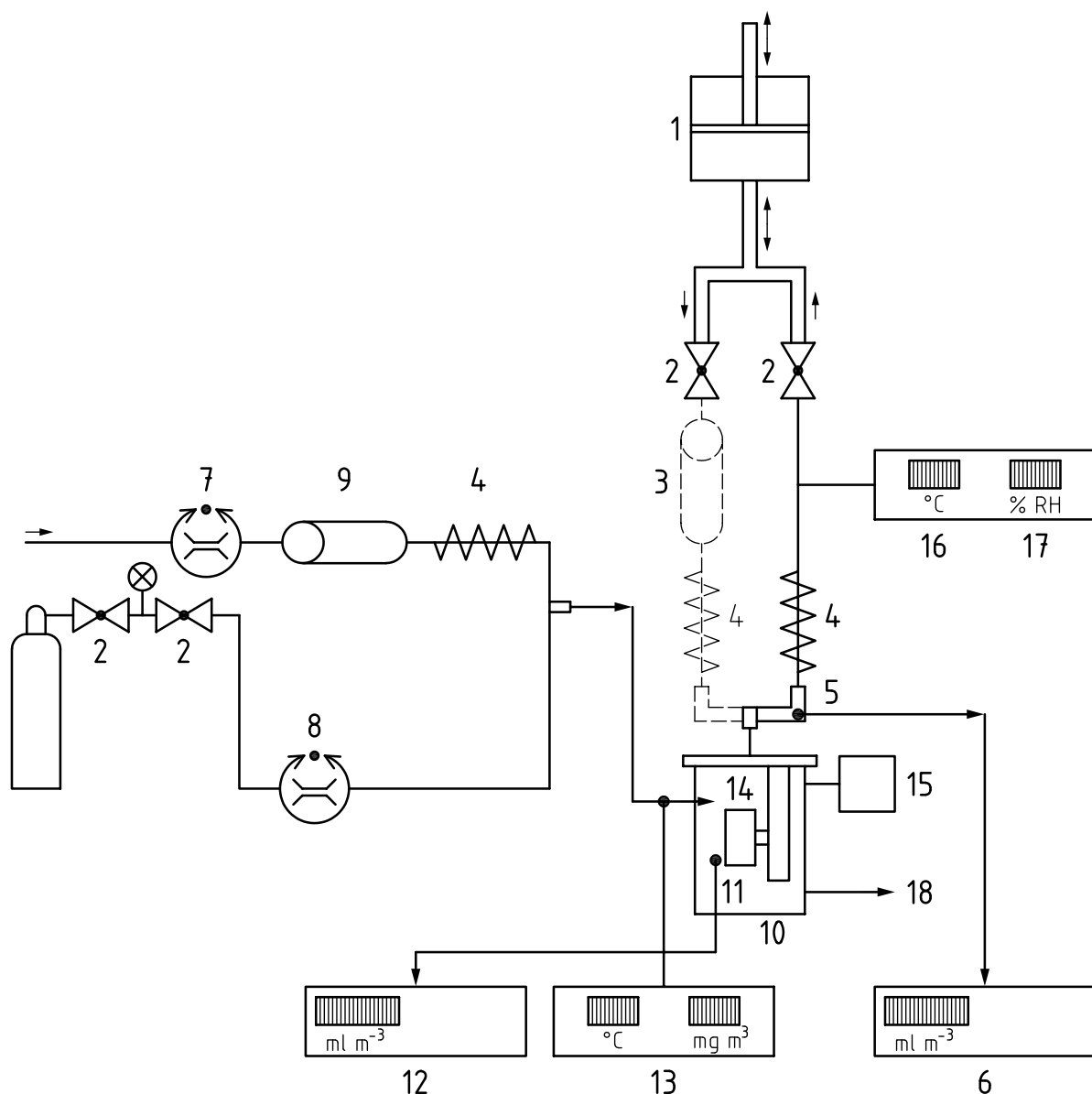
Key

1	breathing machine	8	flow meter for carbon monoxide	14	test specimen, head form or suitable adapter
2	control valves	9	humidifier	15	pressure sensor
3	heater and humidifier (for bidirectional method)	10	test chamber	16	detector: temperature sensor
4	gas cooler	11	sampling point for CO-content in test atmosphere	17	detector: humidity sensor
5	sampling point for CO analyser	12	detector: CO analyser	18	exhaust
6	detector: CO analyser	13	detector: humidity sensor		
7	flow meter for test atmosphere				

8 Methods

8.1 General

Prior to testing, the test specimen shall be stored in its smallest available commercial packaging for a minimum of 4 h at (21 ± 3) °C, unless otherwise specified in the performance standard. The test specimen shall be mounted in the test chamber and exposed to the appropriate gas challenge at the specified flow.

The specimen and its primary attachment shall be exposed to the challenge gas such that leakage at the primary attachment point can be detected. The connector shall be sealed within the chamber so that no leakage can occur where the connector is intended to be attached to the respiratory interface.

The above conditions can readily be achieved if the specimen is fitted with a standardized connector. If the specimen is equipped with a non-standardized connector, the required fitting can be obtained from the manufacturer or by disassembly of a suitable device. Where the gas filter(s) is (are) an integral part of the RPD, the whole device shall be effectively sealed to a mount within the test chamber.

Any experimental method may be employed to obtain the specified influent concentration and measure the effluent concentration, provided it conforms to the following limits:

a) influent concentration: ±10 % of specified challenge value;

b) effluent concentration: ±20 % of specified breakthrough value.

8.2 Test flow conditions

8.2.1 General

All tests shall be conducted so that the test gas flows horizontally and in line with the minimum bed depth of the filter. Care shall be taken that the test gas is not directed preferentially on to one part of the filter or filter system.

The concentration of the test gas shall be stabilized before the test is started.

8.2.2 Multiple filters

When one filter of a multiple filter device is tested separately, the air flow specified for a test shall be divided by the number of filters through which the air flow is proportioned. If, however, it is possible to use only one filter of a multiple filter device, then the full air flow shall be used for testing.

If the filters' resistances are in accordance with Equation (1), then the filter may be tested as a single filter with a proportioned flow. If the filters' breathing resistances are not in accordance with Equation (1), the filters shall be tested in a complete unit at the full flow rate.

$$\frac{R_{max} - R_{min}}{\bar{R}} \leq 0,2 \tag{1}$$

where

R_{max} is the maximum resistance;

R_{min} is the minimum resistance;

$\bar{R}$ is the mean resistance.

When testing one filter of a multiple filter device with the proportioned test air flow, the appropriate performance requirements of the performance standard shall be met.

8.3 Gas capacity test

8.3.1 General

Unless otherwise specified, the gas capacity test shall be performed using constant flow. The CO test shall be performed using dynamic flow.

8.3.2 Constant flow test

Test gases used shall be as specified in the performance standards.

The test gas shall be passed through the filter in continuous flow mode at the flow rate(s), humidity and temperature specified in the performance standard.

The concentration of the test gas and specified reaction products, if applicable, in the effluent air shall be recorded during the test until breakthrough occurs, or until the breakthrough time has been reached (including compensation for variations from the nominal value of influent concentration), whichever is sooner.

The measured breakthrough time, $t_{br\,(measured)}$, shall be adjusted to the corrected breakthrough time, $t_{br\,(corr)}$, by simple, linear proportion of the actual, $C_{(actual)}$, and the specified, $C_{(specified)}$, influent concentrations according to Equation (2):

$$t_{br\,(corr)} = t_{br\,(measured)} \times C_{(actual)} \big/ C_{(specified)} \tag{2}$$

EXAMPLE $C_{(specified)} = 5\,000$ ml/m^3

$C_{(actual)} = 4\,773$ ml/m^3

$t_{br\,(measured)} = 44$ min

$t_{br\,(corr)} = 44$ min $\times$ 4 773 ml/m^3 / 5 000 ml/m^3 = 42 min

8.3.3 Dynamic flow test

8.3.3.1 General

The specimen to be tested shall be mounted on a head form or on a suitable adapter when tested as a separate component. If tested as a separate component, the test shall be carried out with valves to simulate the relevant air flow conditions.

Details of head forms and airway openings will be given in a future part of this International Standard.

NOTE   It might be necessary to use a sealant to ensure an effective seal between the device and, respectively, the head form or adapter.

The delivery of test gas to the test chamber shall be greater than the average flow through the device, such that the maximum pressure difference of the test chamber with regard to ambient during testing shall not exceed ±50 Pa.

The following conditions in the test chamber shall be established using the breathing machine set to the relevant number of cycles per minute and volume per stroke as given by the prerequisites.

— humidity (moisture content of test atmosphere): (50 ± 30) % RH;

— temperature of test atmosphere: 16 °C to 32 °C;

— temperature of exhaled air: (37 ± 1) °C;

— humidity of exhaled air: 95 % to 100 %.

The temperature and humidity of the test atmosphere and exhalation air shall be controlled using suitable conditioners.

The test gas and water vapour concentrations, as well as the differential pressure in the test chamber, shall be monitored and recorded continuously during the test. The effect of humidity of the challenge atmosphere by exhaled air returning to the test chamber shall be taken into account by adequate placing of the humidity measuring point.

The effect of differential pressure and of the dilution of the challenge atmosphere by exhaled air returning to the test chamber shall be taken into account and the concentration of the test gas and moisture shall be adjusted accordingly.

Care shall be taken that the influence of the exhaled air to the challenge gas concentration at the point of the air inlet to the test specimen is minimized. The dry bulb temperature and the concentration of the challenge gas shall be measured 10 mm to 20 mm in front of the air inlet of the test specimen.

The dry bulb temperature of the inhaled air shall be measured using a fast response thermocouple (e.g. NiCr-Ni 0,2 mm diameter).

8.3.3.2 Bidirectional method

In the bidirectional method, the test gas flows through the filter during the inhalation and the exhalation phase. This method requires a test set as shown in Figure 2 where the humidifier (key 3) and the gas cooler (key 4) shall be used.

8.3.3.3 Unidirectional method

In the unidirectional method, the test gas flows through the filter only during the inhalation phase. This method requires a test set as shown in Figure 2 where the humidifier (key 3) and the gas cooler (key 4) shall not be used.

8.4 Gas filter validation test at specified flow rates

Test gases used shall be as specified in the performance standards.

The test gas shall be passed through the filter in continuous flow mode at the flow rate(s), humidity and temperature specified in the performance standard.

The concentration of the test gas and specified reaction products, if applicable, in the effluent air shall be recorded during the test until breakthrough occurs, or until the minimum specified time has been reached, (including compensation for variations from the nominal value of influent concentration), whichever is sooner.

An alternative method for conducting the validation test at specified flow rates greater than 110 l/min for testing non-catalytic filter types OV, AC, BC and OG, or their combination(s), is given in Annex B.

8.5 Migration test A and Migration test B

8.5.1 Migration test A

The filters shall be exposed to the test gas as specified in the performance standard.

After exposure, the filters shall be stored for (66 ± 6) h according to the information supplied by the manufacturer for between-use storage. After storage, clean air shall be passed through the filter at the flow rate, humidity and temperature given in the performance standard.

The duration of the migration test and the conditions for terminating the test shall be specified in the performance standard. The concentration of the test gas and specified reaction products, if applicable, in the effluent air shall be continuously recorded during this test.

8.5.2 Migration test B

The filters shall be exposed to the test gas as specified in the performance standard.

After exposure, the filters shall be stored for (66 ± 6) h, according to the information supplied by the manufacturer for between-use storage. After storage, test gas shall be passed through the filter at the flow rate, humidity and temperature given in the performance standard.

The duration of the migration test and the conditions for terminating the test shall be specified in the performance standard. The concentration of the test gas and specified reaction products, if applicable, in the effluent air shall be continuously recorded during this test.

8.6 Desorption test

The filters shall be exposed to the test gas as specified in the performance standard.

Immediately after exposure, clean air shall be passed through the filter at the flow rate, humidity and temperature used in the gas filter capacity test. The duration of the desorption test and the conditions for terminating the test shall be specified in the performance standard. The concentration of the preadsorbed agent and specified reaction products, if applicable, in the effluent air shall be continuously recorded during this test.

9 Test report

9.1 General

The test report shall include information regarding those parameters specified in Clause 4 together with the information given in 9.2 to 9.6.

9.2 Gas capacity test

Report the corrected breakthrough time or the test time, or the fact that breakthrough did not occur.

9.3 Gas filter validation test at specified flow rates

Report whether or not breakthrough occurred before the minimum specified time and report the actual breakthrough time in cases where the application of the alternative method (Annex B) is to be used.

9.4 Migration test

Report the maximum concentration of the test gas and the reaction product(s), if applicable, in the effluent air and the corresponding time.

9.5 Desorption test

Report the maximum concentration of the test gas and the reaction product(s), if applicable, in the effluent air and the corresponding time.

9.6 CO dynamic test

Report the effluent CO concentration over time and the integral dose.

10 Uncertainty of measurement

An estimate of the uncertainty of measurement associated with this test method shall be established in accordance with ISO/TS 21748. The value of this estimate shall not exceed ± 10 %.

NOTE   The use of transfer standards might assist in establishing common uncertainties of measurement between laboratories.

Annex A
(normative)

Application of uncertainty of measurement

A.1   Determination of compliance

In order to determine compliance or otherwise of the measurement made in accordance with this test method, when compared to the specification limits given in the protective device standard, the following protocol shall be applied.
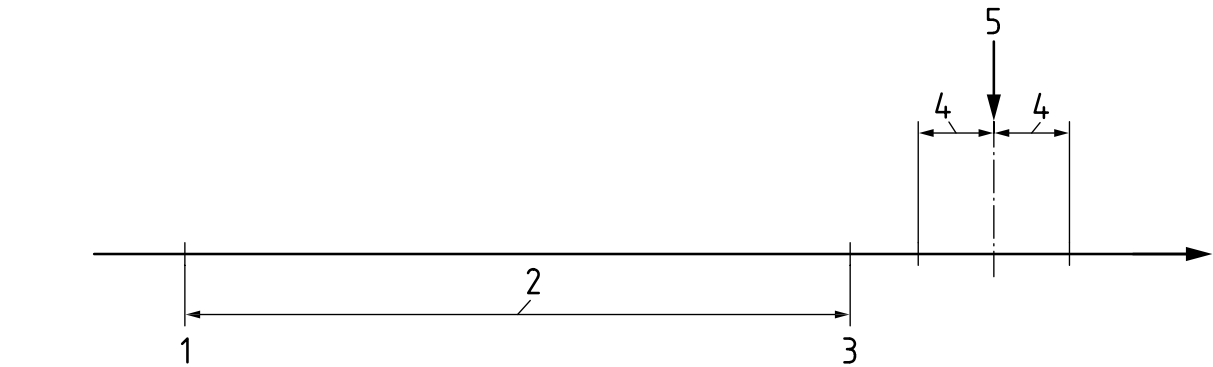
If the test result $\pm$ the uncertainty of measurement, U, falls completely inside or outside the specification zone for the particular test given in the protective device standard, then the result shall be deemed to be a straightforward pass or fail (see Figures A.1 and A.2).
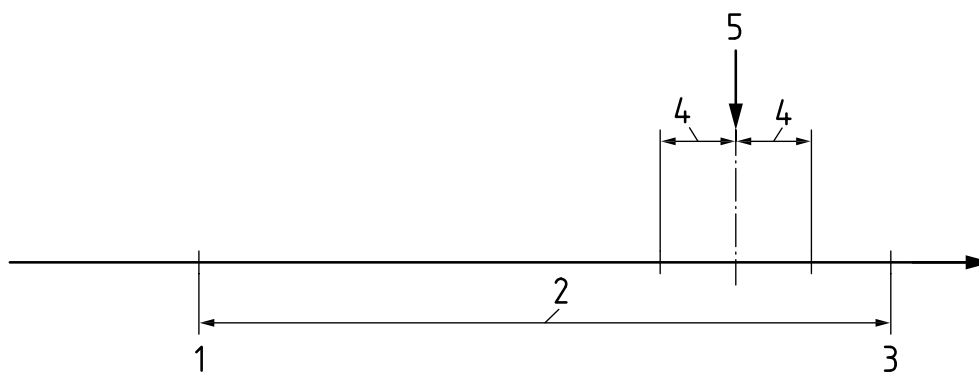
Key

1   lower specification limit
2   specification zone
3   upper specification limit
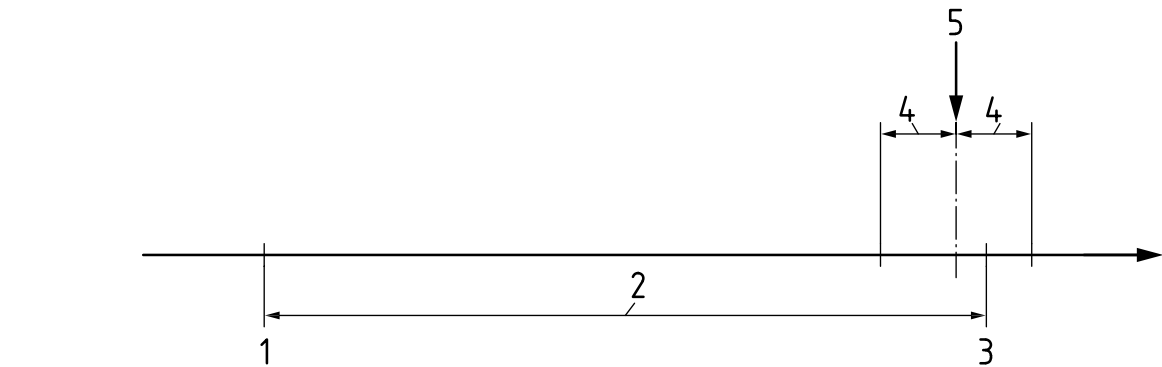4   uncertainty of measurement, U
5   measured value

Figure A.1 — Result pass

Key

1   lower specification limit
2   specification zone
3   upper specification limit
4   uncertainty of measurement, U
5   measured value

Figure A.2 — Result fail

If the test result ± the uncertainty of measurement, U, overlaps a specification limit value (upper or lower) for the particular test given in the protective device standard, then the assessment of pass or fail shall be determined on the basis of safety for the wearer of the device; that is, the result shall be deemed to be a fail (see Figure A.3).

Key

1   lower specification limit
2   specification zone
3   upper specification limit
4   uncertainty of measurement, U
5   measured value

Figure A.3 — Result fail

Annex B
(informative)

Alternative method for the gas filter validation test at specified flow rates

B.1　Principle

Use of a mathematical extrapolation method for predicting the breakthrough time at the specified flow rate from measurements taken at lower flow rates.

B.2　Theoretical breakthrough time

B.2.1　The Wheeler-Jonas equation

The theoretical breakthrough time can be calculated using the predictive Wheeler-Jonas equation[1][2]:

$$t_b = \frac{W_e}{C_0 Q} \left\{ W - \frac{\rho_B Q\, l_n \left[(C_0 - C_x)/C_x \right]}{k_v} \right\}$$ (B.1)

where

t_b　is the breakthrough time;

W_e　is the maximum mass of adsorbate per gram of adsorbent that can be adsorbed at the applied concentration ("equilibrium uptake");

C_0　is the applied gas concentration;

C_x　is the effluent concentration at which the breakthrough time is recorded;

Q　is the volume flow rate;

W　is the mass of adsorbent adsorbed at breakthrough time, t_b;

ρ_B　is the bulk density of the adsorbent;

k_v　is a pseudo-first-order rate constant for the adsorption process;

l_n　is the natural logarithm.

B.2.2　Defining constants

$$A = W_e W$$

$$B = \frac{W_e\,\rho_B}{k_v}$$

and

$$t_b = \frac{A}{C_0} \times \frac{1}{Q} - \frac{B\,l_n\left[(C_0 - C_x)/C_x\right]}{C_0}$$ (B.2)

The breakthrough time at the specified flow rate is derived from the plot of tb versus $\frac{1}{Q}$ or by using Equation (B.2).

B.3   Applicability

Limited to non-catalytic filter types OV, AC, BC and OG, or their combination(s).

B.4   Procedure

Breakthrough time shall be measured at a minimum of three flow rates, preferably evenly spaced on $\frac{1}{Q}$ axis.

Two of the flow rates shall be the gas capacity test flow rate and the lower flow rate taken for the validation test. The third flow rate shall be (40 ± 5) % of the specified flow rate.

For each flow rate at least three samples shall be tested.

For greater accuracy the test may be conducted at additional flow rates.
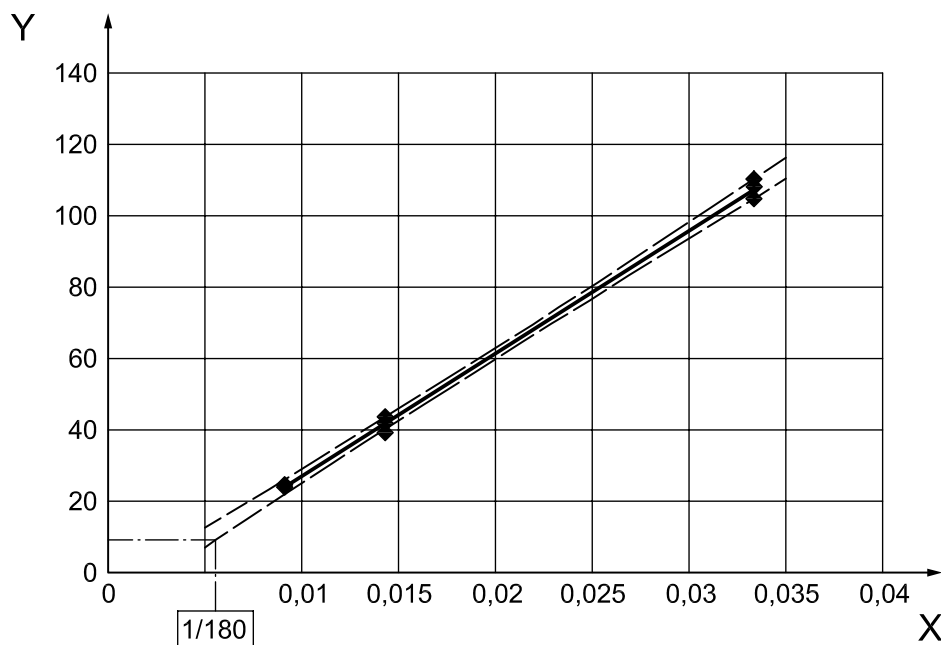
Key

X	reciprocal flow (min/l)
Y	breakthrough time (min)

Equation for regression analysis: $Y = 3\,442,9\,X - 7,272\,6$; $R^2 = 0,997\,7$

◆	measured breakthrough time
------	95 % confidence interval
—.—.—	lower confidence level of predicted breakthrough time at 180 l/min

Figure B.1 — Example for regression analysis and prediction of breakthrough time

B.5   Data analysis

Perform a linear regression analysis of the breakthrough time as a function of reciprocal flow as illustrated in Figure B.1. The slope calculated by the regression analysis is equal to the term $\dfrac{A}{C_0}$ in Equation (B.2). The intercept calculated by regression analysis is equal to the second term in Equation (B.2).

B.6   Determination of breakthrough time

The breakthrough time shall be calculated as the lower 95 % confidence limit of the breakthrough time predicted for a flow of 180 l/min. The formula for this calculation is:

$$y_{LCL} = \hat{y}_{180} - t_{\alpha/2} \times \sqrt{MS_{\text{Res}}} \times \sqrt{\frac{1}{n} + \frac{(1/180 - \overline{1/Q})^2}{\sum_i (1/Q_i - \overline{1/Q})^2}} \qquad (B.3)$$

where

$\hat{y}_{180}$ is the breakthrough time predicted by Equation (B.2) at a flow of 180 l/min;

$t_{\alpha/2}$ is the Student's t statistic for the $(1-\alpha)$ confidence interval based on n-2 degrees of freedom;

n is the number of data points used in the regression analysis (a minimum of 9);

$\overline{1/Q}$ is the average of the reciprocal flows in the data set.

The term MS_{Res} is the estimated variance of the predicted breakthrough time:

$$MS_{\text{Res}} = \frac{\sum_{i=1}^{n}(y_i - \hat{y}_i)^2}{n-2} \qquad (B.4)$$

where

y_i is the measured breakthrough time for sample number i;

$\hat{y}_i$ is the breakthrough time predicted by Equation (B.2) at the same flow.

The term MS_{Res} is also known as the standard error of the y estimate, designated as SE[y].

Commercially available statistical analysis software can simplify the calculation of the values described in this annex.

Bibliography

[1]   WHEELER, A., *Performance of Fixed-Bed Catalytic Reactors with poison in the feed*, J. Catal. 1969; 13: 299

[2]   JONAS, L.A. and REHRMANN, J.A., *Predictive Equations in Gas Adsorption Kinetics*, Carbon 1973; 11: 59