

---

---

**Stationary source emissions —  
Determination of mass concentration of  
particulate matter (dust) at low  
concentrations — Manual gravimetric  
method**

*Émissions de sources fixes — Détermination d'une faible concentration en  
masse de matières particulaires (poussières) — Méthode gravimétrique  
manuelle*



Reference number  
ISO 12141:2002(E)

© ISO 2002

**PDF disclaimer**

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

© ISO 2002

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Case postale 56 • CH-1211 Geneva 20  
Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.ch](mailto:copyright@iso.ch)  
Web [www.iso.ch](http://www.iso.ch)

Printed in Switzerland

## Contents

	Page
1 Scope .....	1
2 Normative references .....	1
3 Terms and definitions .....	1
4 Principle .....	4
5 Sampling plane and sampling points .....	5
6 Apparatus and materials (see summary annex E) .....	7
7 Calculation of nozzle diameter to obtain weighable quantities of dust .....	13
8 Weighing procedure .....	14
9 Sampling procedure .....	16
10 Thermal behaviour of dusts .....	18
11 Validation of results .....	20
12 Calculations .....	21
13 Performance characteristics of the method .....	22
14 Test report .....	23

## Annexes

A Requirements related to the working platform .....	25
B Determination of flow direction with Pitot tubes .....	26
C Determination of positions of sampling points in circular and rectangular ducts .....	27
D Examples of suitable access ports for sampling equipment .....	31
E Proven entry nozzle designs .....	33
F Summary of apparatus requirements .....	35
G Sampling volume, flowrate and duration .....	36
H Examples of weighing bias .....	37
I Precision data .....	39
Bibliography .....	40

## 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 12141 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 1, *Stationary source emissions*.

Annexes A, C, E and F form a normative part of this International Standard. Annexes B, D, G, H and I of this International Standard are for information only.

## Introduction

This method was developed from close liaison and cooperation between ISO/TC 146/SC 1/WG 11 and CEN/TC 264/WG 5, resulting in the preparation of this International Standard and the European Standard EN 13284-1. This International Standard is similar to EN 13284-1 with additional emphasis given on the use of high-volume sampling techniques for the measurement of dust at low concentrations. It also gives procedures for extending the range of measurement of ISO 9096:1992 to lower concentrations. As in ISO 9096:1992, a representative, integrated sample is extracted from the flue gas and particulate matter entrained in the gas sample is separated by a filter. The pre-weighed filter is subsequently dried and weighed. Any increase in the mass is attributed to the collection of particulate matter on the filter.

To meet the specifications of this International Standard, the particulate sample must be weighed to a specified level of accuracy. At low dust concentrations, this level of accuracy may be achieved by:

- a) exercising extreme care in weighing, as per procedures of this standard,
- b) extending the sampling time at conventional sampling rates, or
- c) sampling at higher rates for conventional sampling times (high-volume sampling).

This International Standard in addition differs from ISO 9096:1992 by requiring the measurement of the mass of filter blanks, specifying weighing procedures.

This method may be used for calibration of automated monitoring systems (AMSs) (see ISO 10155). If the waste gas contains unstable, reactive or semivolatile substances, the measurement will depend on the filtration temperature, and in-stack methods may be more applicable than out-stack methods for the calibration of automated monitoring systems.

.....

# Stationary source emissions — Determination of mass concentration of particulate matter (dust) at low concentrations — Manual gravimetric method

## 1 Scope

This International Standard describes a reference method for the measurement of low dust content in ducted gaseous streams at concentrations below  $50 \text{ mg/m}^3$  under standard conditions. This method has been validated with special emphasis on the region around  $5 \text{ mg/m}^3$ .

This International Standard has been developed and validated for gaseous streams emitted by waste incinerators. More generally, it may be applied to emissions from other stationary sources, and to higher concentrations.

If the gases contain unstable, reactive or semi-volatile substances, the measurement will depend on the sampling and filter treatment conditions.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3966:1977, *Measurement of fluid flow in closed conduits — Velocity area method using Pitot static tubes*

ISO 5725 (all parts), *Accuracy (trueness and precision) of measurement methods and results*

ISO 9096:1992, *Stationary source emissions — Determination of concentration and mass flow rate of particulate material in gas-carrying ducts — Manual gravimetric method*

ISO 10780:1994, *Stationary source emissions — Measurement of velocity and volume flowrate of gas streams in ducts*

## 3 Terms and definitions

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

### 3.1

#### **particulate matter**

#### **dust**

particles, of any shape, structure or density, dispersed in the gas phase under the sampling conditions

**NOTE** In the method described, all the compounds that may be collected by filtration under specified conditions after sampling of the gas to be analysed, and which remain upstream of the filter and on the filter after drying under specified conditions, are considered to be dust (or particulate matter). However, for the purposes of some national standards, the definition of particulate matter may extend to condensibles or reaction products collected under specified conditions (e.g. temperatures lower than the flue gas temperature).

**3.2**

**filtration temperature**

temperature of the sampled gas immediately downstream of the filter

**3.3**

**in-stack filtration**

filtration in the duct with the filter in its filter holder placed immediately downstream of the sampling nozzle

**3.4**

**out-stack filtration**

filtration outside the duct with the filter in its heated filter holder placed downstream of the sampling nozzle and the suction tube (sampling probe)

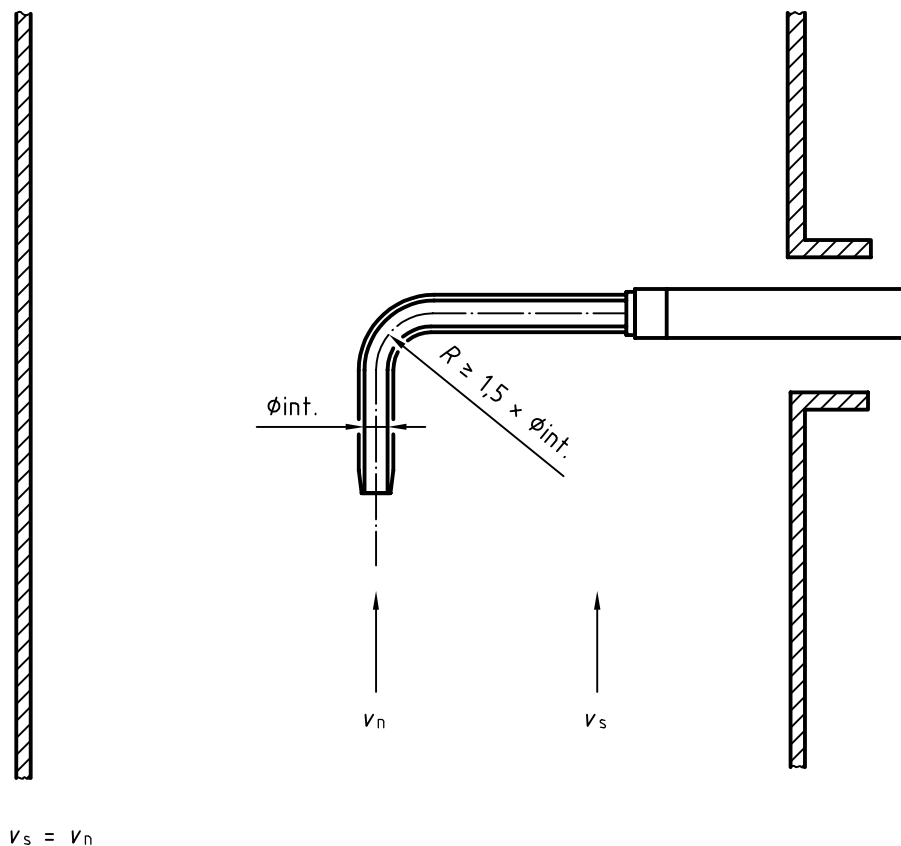
**3.5**

**isokinetic sampling**

sampling at a flowrate such that the velocity and direction of the gas entering the sampling nozzle ( $v_n$ ) are the same as that of the gas in the duct at the sampling points,  $v_s$

See Figure 1.

NOTE The velocity ratio  $v_n/v_s$  expressed as a percentage characterizes the deviation from isokinetic sampling.



**Figure 1 — Isokinetic sampling in duct**



**3.6**

**hydraulic diameter**

$d_h$

characteristic dimension of a duct cross-section

$$d_h = \frac{4 \times \text{area of sampling plane}}{\text{length of perimeter of sampling plane}} \quad (1)$$

**3.7**

**sampling plane**

plane normal to the centreline of the duct at the sampling position

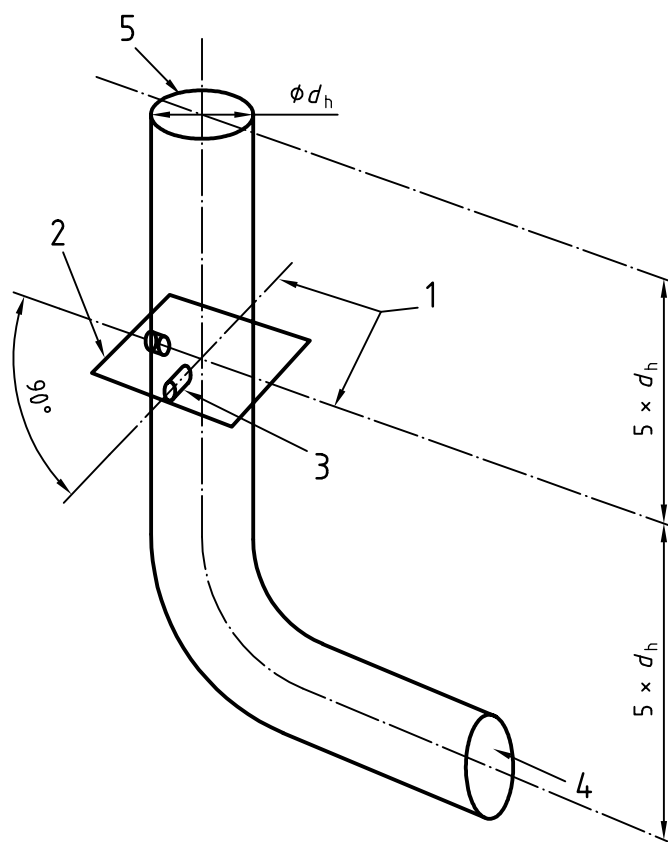
See Figure 2.

**3.8**

**sampling line**

line in the sampling plane along which sampling points are located, bounded by the inner duct wall

See Figure 2.



**Key**

- 1 Sampling lines
- 2 Sampling plane
- 3 Access port
- 4 Flow
- 5 Top of duct

**Figure 2 — Illustration of definitions in relation to a circular duct**

### 3.9

#### **sampling point**

the specific position on a sampling line at which a sample is extracted

### 3.10

#### **standard conditions**

gas pressure and temperature constants and conditions to which volumetric calculations are referred

NOTE For the purposes of this International Standard, standard conditions are 101,325 kPa rounded to 101,3 kPa; 273,15 K rounded to 273 K; dry gas.

### 3.11

#### **overall blank**

test sample taken at the plant site in an identical manner to the normal samples in the series, except that no gas is sampled during the test duration

NOTE The measured mass variation provides an estimation of the uncertainties. The overall blank value, divided by the average sampling volume of the measurement series, provides an estimation of the detection limit (milligrams per cubic metre) of the whole measurement process, as carried out by the operator. The overall blank includes possible deposits on the filter and on all parts upstream.

### 3.12

#### **weighing control**

procedure for the detection/correction of apparent variations in mass due to possible changes between pre- and post-sampling weighing conditions, by using parts, identical to those to be weighed for dust measurements, pretreated and weighed under the same conditions of temperature and humidity (e.g. desiccator) as pre- and post-sampling

NOTE The control parts are kept free from contamination.

### 3.13

#### **measurement series**

successive measurements carried out in the same sampling plane, and under the same process conditions

### 3.14

#### **limit value**

##### **average limit value**

dust concentration that is permitted by authorities for the plant process

NOTE For purposes other than regulatory uses, the measurement value is compared to a stated reference value.

### 3.15

#### **high-volume sampling**

sampling at rates higher than typical in ISO 9096 by using larger-diameter nozzles and higher flowrates to maintain isokinetic sampling conditions

NOTE Nozzle diameters are typically 20 mm to 50 mm, with corresponding flowrates of 5 m<sup>3</sup>/h to 50 m<sup>3</sup>/h.

## 4 Principle

A sample stream of the gas is extracted from the main gas stream at representative sampling points for a measured period of time, with an isokinetically controlled flowrate and a measured volume. The dust entrained in the gas sample is separated by a pre-weighed plane filter, which is then dried and re-weighed. Deposits upstream of the filter in the sampling equipment are also recovered and weighed. The increase in mass of the filter and the deposited mass upstream of the filter are attributed to dust collected from the sampled gas, which allows the dust concentration to be calculated.

Two different configurations of the sampling equipment may be used (see 6.2.1) depending on the characteristics of the gases to be sampled.

Valid measurements can be achieved only when:

- a) the gas stream in the duct at the sampling location has a sufficiently steady velocity (see 5.2);
- b) sampling is carried out without disturbance of the gas stream, under isokinetic conditions, using a sharp-edged nozzle facing into the stream;
- c) samples are taken at a preselected number of stated positions in the sampling plane, to allow for a non-uniform distribution of dust in the duct or stack;
- d) the sampling train is designed and operated to avoid condensation and chemical reactions, to minimize dust deposits upstream of the filter, and to be leak-free;
- e) dust deposits upstream of the filter are taken into account;
- f) the overall blank value does not exceed 10 % of the daily limit set for the process;

NOTE High-volume sampling techniques or an extension of the sampling time may be employed to satisfy this requirement.

- g) the sampling and weighing procedures are adapted to the expected dust quantities.

A measurement series is validated only when the quantity of dust collected during the sampling is at least 5 times a corresponding positive difference in an overall blank. High-volume sampling techniques or an extension of the sampling time may be employed to satisfy this requirement.

Any emission value determined in the test series that is less than the blank value is not valid. However, when measured concentrations are below  $5 \text{ mg/m}^3$ , it may not be possible to fulfill this requirement. In such a case, the sampling time shall be extended or a larger sample nozzle and high-volume sampling techniques employed to collect sufficient particulate matter within the specified sampling period.

## 5 Sampling plane and sampling points

### 5.1 General

Sampling is only possible when a suitable location is available, with a sufficiently high and homogeneous gas velocity at the sampling plane.

The sampling plane shall be easily reached from convenient access ports and a safe working platform (see annex A).

Sampling shall be carried out at a sufficient number of sampling points, located on the sampling plane.

### 5.2 Sampling plane

The sampling plane shall be situated in a length of straight duct (preferably vertical) with a constant shape and cross-sectional area. Where possible, the sampling plane shall be as far downstream as possible and upstream from any disturbance that could produce a change in the direction of flow (disturbances can be caused by e.g. bends, fans or partially closed dampers).

Measurements at all the sampling points defined in 5.3 and annex C shall prove that the gas stream in the sampling plane meets the following requirements:

- a) the angle of gas flow is less than  $15^\circ$  with regard to the duct axis (a recommended method for estimation is indicated in annex B);
- b) no local negative flow is present;
- c) the gas velocity is at least the minimum for the flowrate-measuring method used (for Pitot tubes a differential pressure larger than 5 Pa);
- d) the ratio of the highest to lowest local gas velocities is less than 3:1.

If the above requirements cannot be met, the sampling location is not in compliance with this International Standard (see 11.2).

The above requirements are generally fulfilled in sections of duct with at least five hydraulic diameters of straight duct length upstream of the sampling plane and two hydraulic diameters downstream (five hydraulic diameters from the top of a stack). Therefore, it is strongly recommended that sampling locations be designed accordingly.

### 5.3 Minimum number and location of sampling points

The dimensions of the sampling plane dictate the minimum number of sampling points. This number increases as the duct dimensions increase.

Tables 1 and 2 give the minimum number of sampling points to be used for circular and rectangular ducts respectively. The sampling points shall be located at the centres of equal areas in the sampling plane (in accordance with annex C).

Sampling points shall not be located within 3 % of the sampling-line length or 5 cm, whichever is greater, from the inner duct wall. This may arise when selecting more than the minimum numbers of sampling points presented in Tables 1 and 2, for example in cases of unusual duct shape.

NOTE When the requirements for the sampling plane (see 5.2) cannot be met, it may be possible to improve representative sampling by increasing the number of sampling points above those specified in Tables 1 and 2. See also 9.3 for sampling-point premeasurement procedures.

**Table 1 — Minimum number of sampling points for circular ducts**

Range of sampling plane areas m <sup>2</sup>	Range of duct diameters (approx.) m	Minimum number of sampling lines	Minimum number of sampling points per plane
< 0,1	< 0,35	—	1 <sup>a</sup>
0,1 to 1,0	0,35 to 1,1	2	4
1,1 to 2,0	1,1 to 1,6	2	8
> 2,0	> 1,6	2	At least 12, and 4 per m <sup>2</sup> <sup>b</sup>

<sup>a</sup> Using only one sampling point may give rise to errors greater than those specified in this International Standard.

<sup>b</sup> For large ducts, 20 sampling points is generally sufficient.

**Table 2 — Minimum number of sampling points for rectangular ducts**

Range of sampling plane areas m <sup>2</sup>	Minimum number of side divisions <sup>a</sup>	Minimum number of sampling points
< 0,1	—	1 <sup>b</sup>
0,1 to 1,0	2	4
1,1 to 2,0	3	9
> 2,0	> 3	at least 12, and 4 per m <sup>2</sup> <sup>c</sup>

<sup>a</sup> Other side divisions may be necessary, for example if the longest duct side length is more than twice the length of the shortest side (see C.2).

<sup>b</sup> Using only one sampling point may give rise to errors greater than those specified in this International Standard.

<sup>c</sup> For large ducts, 20 sampling points is generally sufficient.

### 5.4 Access ports and working platform

Ports shall be provided for access to the sampling points selected in accordance with 5.3 and annex C.

The port dimensions shall allow sufficient space for the insertion and withdrawal of sampling equipment. A minimum diameter of 125 mm or a surface area of 100 mm × 250 mm is recommended, except for small ducts (less than 0,7 m diameter) for which the port size needs to be smaller.

Two examples of suitable access ports are given in annex D.

For safety and practical reasons, the working platform shall comply with the requirements of annex A.

## 6 Apparatus and materials (see summary annex E)

### 6.1 Gas velocity, temperature, pressure and gas composition measurement devices.

Velocity measurements should preferably be carried out using standard Pitot tubes, as described in annex A of ISO 3966:1977. Alternatively, other measurement devices (S-type Pitot tube, etc.) may also be used, provided that they are calibrated against standardized Pitot tubes (see ISO 10780).

The temperature and the pressure in the duct shall be measured in order to calculate the actual density of the gas within  $\pm 0,05 \text{ kg/m}^3$ , also taking the gas composition into account.

When expressing dust concentrations on a dry basis, and/or where the concentrations are to be expressed in relation to a reference oxygen concentration, humidity (moisture) and/or oxygen measurements shall be carried out in the vicinity of the sampling plane.

### 6.2 Sampling equipment.

The sampling train principally consists of the entry nozzle, filtration device, suction tube, gas pump, system for measurement of sampled gas volume at identified temperature and pressure, and system for controlling the sampling conditions so that they are isokinetic.

**6.2.1 Filtration device**, either located in the duct ("in-stack" filtration) or placed outside the duct ("out-stack" filtration).

- a) "In-stack" filtration devices (Figure 3): the part of the tubing between the nozzle and the filter shall be very short, thereby minimizing dust deposits upstream of the filter. Due to the dimensions of access ports available on ducts, the filter diameter is then typically limited to 50 mm, with a sample flowrate of approximately  $1 \text{ m}^3/\text{h}$  to  $3 \text{ m}^3/\text{h}$  (see annex G for further discussion of sample flowrates). Since the filtration temperature is generally identical to that of the gas in the duct, filter clogging may occur if the stack gas contains water droplets.

A rigid, leak-free tube of sufficient length (support tube) is used downstream of the filter housing for mechanical support of the nozzle and filter housing.

- b) "Out-stack" filtration devices (Figure 4): the part of tubing between the nozzle and the filter (suction tube) shall be of sufficient length to traverse the duct to the required sample points. The suction tube and the filter holder shall be temperature-controlled, to ensure evaporation of any water droplets and avoid filtration difficulties related to acid, high-dew-point gases (see also 9.4). For flat filters, diameters between 50 mm and 150 mm are generally used, with associated flowrates of  $1 \text{ m}^3/\text{h}$  to  $10 \text{ m}^3/\text{h}$ . Other filter sizes may be necessary for high-volume sampling applications.

**6.2.2 The sampling parts of the system**, made of corrosion-resistant and, if necessary, heat-resistant material, e.g. stainless steel, titanium, quartz or glass.

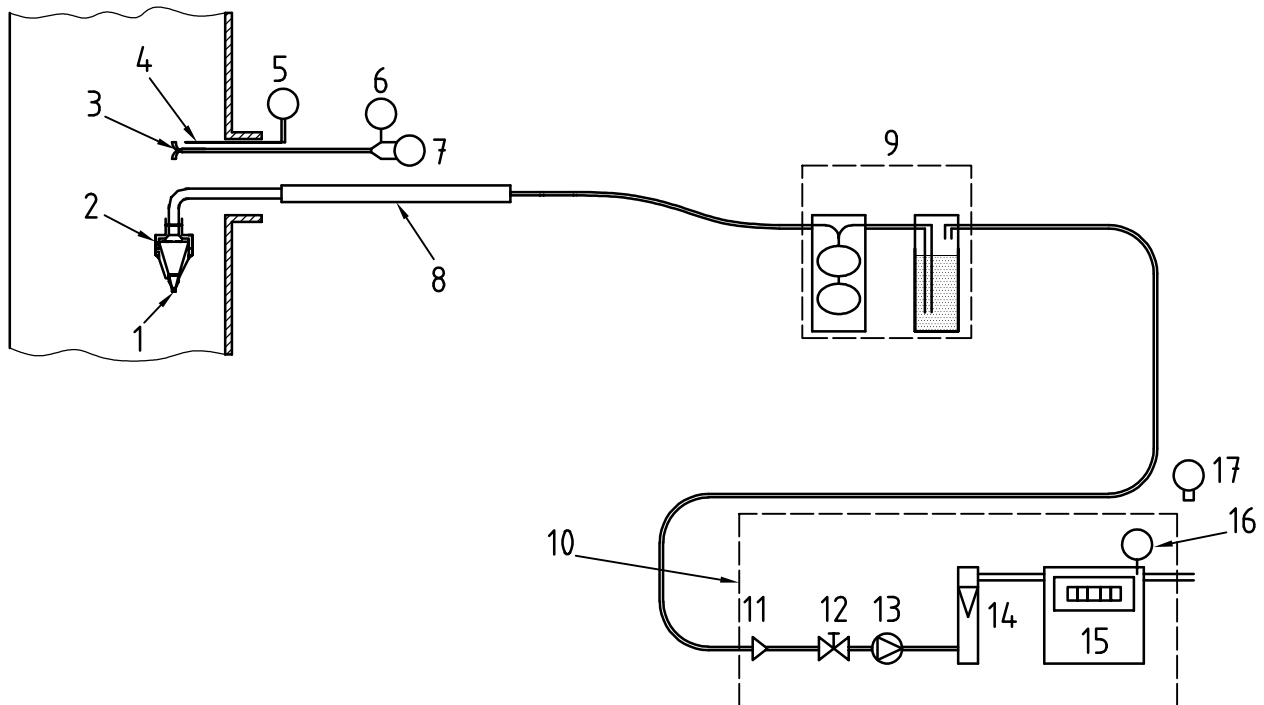
If further analysis of the collected dust is to be performed, materials in contact with the sample gas and the filter shall be fit for the purpose, in order to avoid contamination.

The surfaces of parts upstream of the filter shall be smooth and well polished, and the number of joints shall be kept to a minimum.

Any changes in bore diameter shall be smoothly tapered and not rigid or stepped.

The sampling equipment shall also be designed to facilitate the cleaning of internal parts upstream of the filter.

All parts of the equipment that will come in contact with the sample shall be protected from contamination during transportation and storage.



**Key**

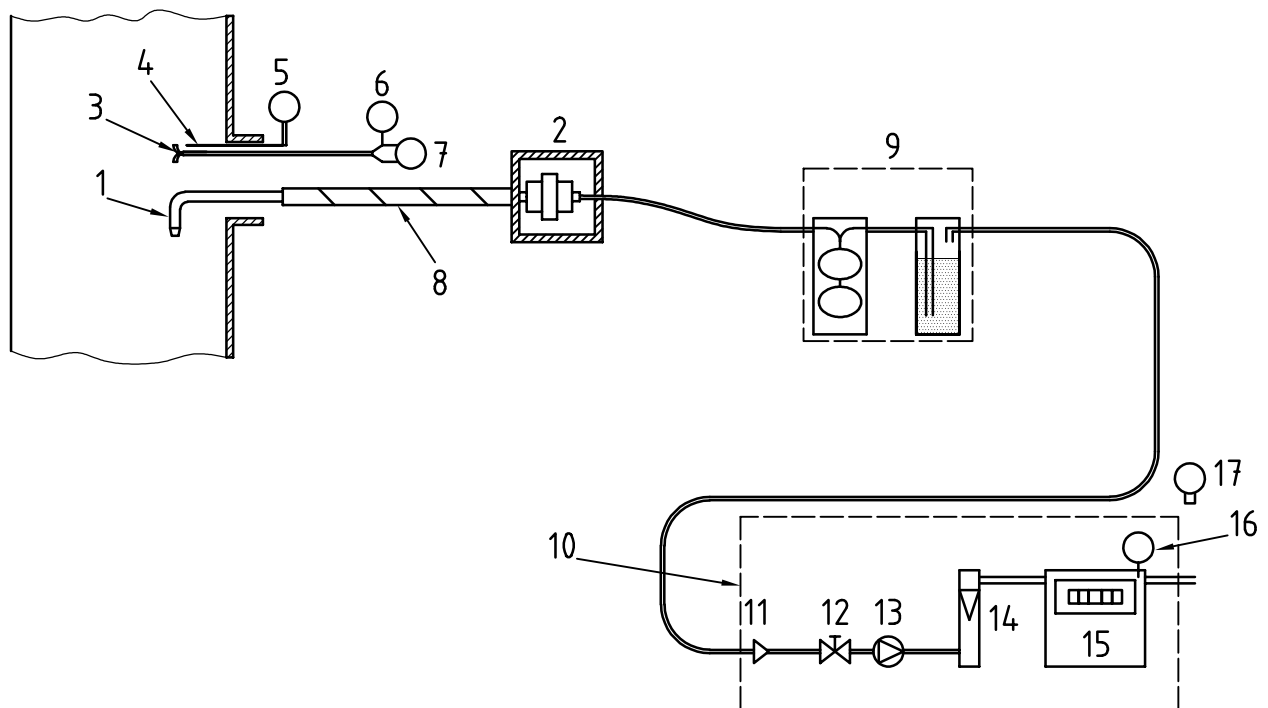
- 1 Entry nozzle
- 2 Filter holder
- 3 Pitot tube
- 4 Temperature probe
- 5 Temperature measurement
- 6 Static pressure measurement
- 7 Differential pressure measurement
- 8 Support tube (in-stack device)
- 9 Cooling and drying system
- 10 Suction unit and gas-metering device
- 11 Shut-off valve
- 12 Adjustment valve
- 13 Pump
- 14 Flowmeter
- 15 Dry gas volumeter
- 16 Temperature measurement
- 17 Barometer

**Figure 3 — Example of “in-stack” filter sampling system**

**6.2.3 Entry nozzle.**

The sample gas stream to be analysed enters the sampling equipment via the nozzle. The nozzle is connected either to the suction tube or to the filter holder.

In order to allow isokinetic sampling of gases flowing at a wide range of velocities (e.g. 3 m/s to 50 m/s) without causing major change in the sampled gas flowrate, the sampling equipment shall include a set of nozzles of different diameters.

**Key**

- 1 Entry nozzle
- 2 Filter holder
- 3 Pitot tube
- 4 Temperature probe
- 5 Temperature measurement
- 6 Static pressure measurement
- 7 Differential pressure measurement
- 8 Support tube (out-stack device)
- 9 Cooling and drying system
- 10 Suction unit and gas-metering device
- 11 Shut-off valve
- 12 Adjustment valve
- 13 Pump
- 14 Flowmeter
- 15 Dry gas volumeter
- 16 Temperature measurement
- 17 Barometer

**Figure 4 — Example of “out-stack” sampling system**

The entry nozzle shall be sharp in order not to disturb the main gas flow. Annex F details three proven designs. Other designs are allowed, provided it is demonstrated that they give equivalent results.

Because it is necessary, for mechanical reasons, for the nozzle bevel to have sufficient thickness, this leads to uncertainty in the effective sampling area. This uncertainty should be less than 10 % in order to fulfill isokinetic sampling criteria. For this reason, it is recommended that nozzles of inside diameter exceeding 8 mm be used.

## ISO 12141:2002(E)

In order to minimize disturbance of the gas flow near the nozzle tip, the following requirements shall also apply:

- a) the nozzle shall have a constant internal diameter for a minimum length of one internal diameter, or at least 10 mm, from the nozzle tip, whichever is the greater;
- b) any change in bore diameter shall be tapered and of conical angle less than 30°;
- c) bends are allowed only after a minimum straight length of 30 mm; their radius shall be at least 1,5 times the internal diameter;
- d) any change in the external diameter of the sampling equipment parts located at less than 50 mm from the nozzle tip shall be tapered and of conical angle less than 30°;
- e) obstacles related to the sampling equipment are
  - 1) prohibited upstream of the nozzle tip,
  - 2) allowed beside and downstream of the nozzle tip when situated at more than 50 mm, or at least one times the size of the obstacle, from the nozzle tip, whichever is the greater.

### 6.2.4 Suction tube (out-stack filtration systems)

The suction tube shall have a smooth and well-polished internal surface, and shall be designed to facilitate inspection and mechanical cleaning. The tube shall be temperature-controlled to maintain constant the planned conditions for the filtration of the gases (see also clause 9).

### 6.2.5 Filter holder

The filter holder is a casing in which the filter support and the filter are mounted. When the filter holder is placed "out-stack", it shall be temperature-controlled to maintain filtration conditions constant and to avoid condensation (see 9.4).

The parts to be weighed before and after sampling shall be:

- a) either the filter, or the filter support and the filter in which case the dust deposits at the inlet of the filter holder shall be recovered and weighed (see 11.5). The use of a filter holder with a conical inlet of angle less than 30° helps to minimize dust deposits;
- b) or the filter, the inlet part of the filter holder and the upstream parts (e.g. nozzle), in which case dust deposits upstream of the filter are taken directly into account. The outside surfaces shall be cleaned prior to weighing. It is necessary to check whether the parts to be weighed are compatible with the range of the balance (see also 8.2).

The filter holder and the filter support shall be designed in such a way that no gas turbulence will occur near the joints.

To reduce the filter pressure drop and to improve the distribution of dust on the filter, a coarse-grain filter support (e.g. fibre mesh) shall be used.

In high-volume sampling applications, the filter holder and filter shall be configured to avoid filter medium loss or filter damage at higher flowrates.

### 6.2.6 Filters.

The filters to be used shall comply with the following minimum requirements.

- a) The filter efficiency shall be better than 99,5 % on a test aerosol with a mean particle diameter of 0,3 µm, at the maximum flowrate anticipated (or 99,9 % on a test aerosol of 0,6 µm mean particle diameter). This efficiency shall be certified by the filter supplier. At higher flowrates, the filter shall have an area large enough to maintain a pressure drop sufficiently low to meet the filter efficiency specification.
- b) The filter material shall not react with or adsorb gaseous compounds contained in the gas sampled, and shall be thermally stable, taking into account the maximum anticipated temperature during conditioning, sampling, etc.



The choice of the filter shall also take into account the following considerations (see 9.4).

- The pressure drop across the filter, and the pressure increase due to the collection of dust while sampling. This depends on the kind of filter. As an example, the pressure drop can be between 3 kPa and 10 kPa for a gas velocity of about 0,5 m/s.
- When using filters with organic binders, care shall be taken to avoid mass variations due to their evaporation on heating.
- Glass-fibre filters may react with acidic compounds such as SO<sub>3</sub>, leading to an increase in mass. Their use is not recommended.
- Despite their low mechanical strength, quartz-fibre filters have been shown to be efficient in most cases.
- PTFE filters have also been shown to be efficient. However, the temperature of the gas passing through the filter shall not exceed 230 °C (refer to the filter supplier's documentation).

If it is planned to determine the composition of the dust collected, the choice of the filter material shall take into account the blank filter value for the relevant compounds.

When weighing filters, care shall be taken to avoid errors due to electrostatic charges.

NOTE 1 The overall blank value for the determination (see 11.6) depends to some extent on the choice of filter (mechanical properties, affinity for humidity, etc.).

NOTE 2 Fibre losses can occur from the filter during the initial sampling period, which might affect the overall result.

### 6.2.7 Suction unit and gas-metering devices.

The suction unit shall be gastight, corrosion-proof and capable of producing the maximum rated flowrate in the sampling conditions (pressure on the suction side down to e.g. 40 kPa). Course adjustments of the flowrate shall be controlled by a regulating valve and/or by-pass valve. A shut-off valve shall also be available to shut off the gas flow through the sampling train.

Examples of two kinds of gas-metering system that may be used are as follows:

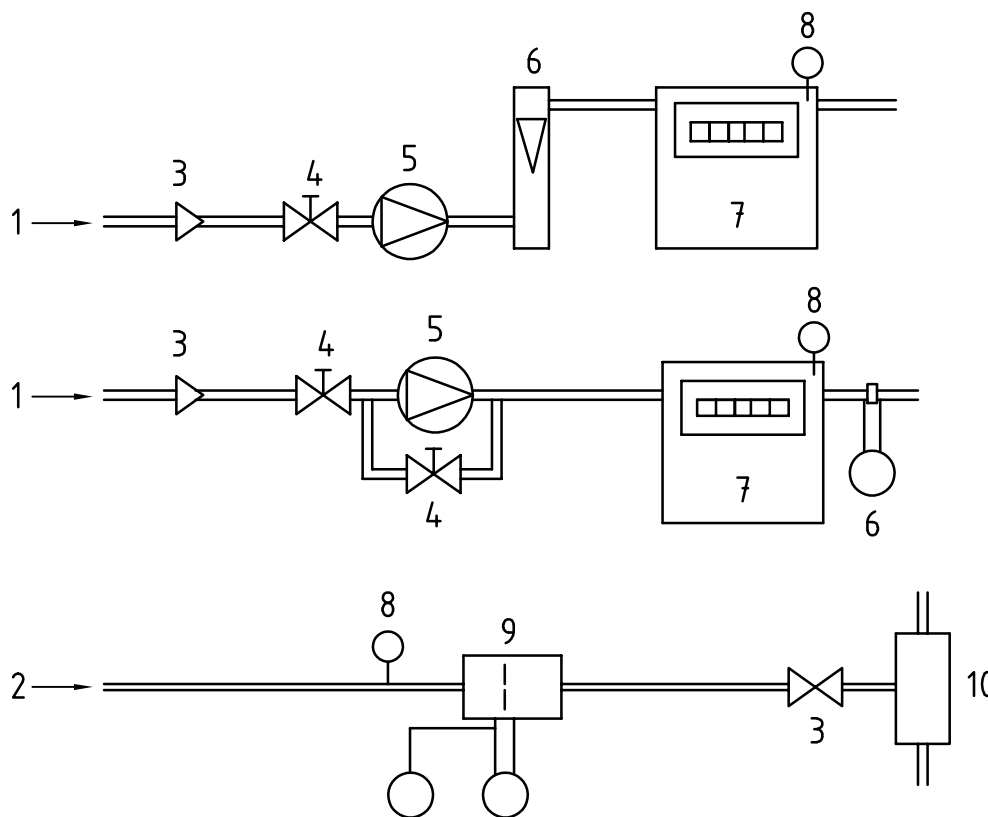
- a) flowrate measurements on dry gas (see Figure 5):
  - 1) condenser and/or gas drying tower providing a residual humidity less than 10 g/m<sup>3</sup> at the maximum flowrate;
  - 2) gastight pump;
  - 3) flowmeter, to facilitate the flowrate adjustment, calibrated against the volume meter;
  - 4) volume meter (precision better than 2 % at the anticipated flowrate) with associated absolute pressure and absolute temperature measurement equipment (better than 1 %);
- b) flowrate measurements on (see Figure 5):
  - 1) heated tubing;
  - 2) orifice plate or equivalent device (flow meter), calibrated within 2 % of the anticipated flowrate; the precision of temperature and pressure (absolute and differential) measurements shall be better than 1 %;
  - 3) compressed-air ejector acting as suction device;
  - 4) atmospheric pressure measuring device.

Other types of arrangement are allowed, provided the component parts meet the requirements of annex F.

If side-stream sampling is used, the side-stream flowrate is taken into account for the calculation of isokinetic sampling conditions and for the calculation of the total sampled gas volume.

## 6.3 Dust-deposit recovery materials.

### 6.3.1 Rinsing solutions: purified water (de-ionized and filtered) and acetone (dry residue less than 10 mg/l).



**Key**

- 1 Dry gas
- 2 Wet gas
- 3 Shut-off valve
- 4 By-pass control valve
- 5 Pump
- 6 Flowmeter
- 7 Volumeter
- 8 Temperature measurement
- 9 Heated orifice plate
- 10 Compressed air ejector

**Figure 5 — Examples of suction-unit and gas-metering devices**

**6.3.2 Clean containers** of appropriate size (e.g. 250 mg/l) for storage and transportation of the rinsing solutions.

**6.3.3 Plugs** (acetone-resistant) to close the suction tube.

**6.4 Apparatus for conditioning and weighing.**

**6.4.1 Weighing containers**, in which to dry the rinsing solutions. The mass shall be in accordance with the balance to be used. Glass and ceramic containers have proven to be suitable materials for these containers. Polymeric materials are not recommended.

**6.4.2 Desiccators**, located in the weighing room, with a desiccating agent (silica gel, calcium chloride, etc.).

**6.4.3 Laboratory drying oven**, controlled to maintain a specified temperature within  $\pm 5^\circ\text{C}$ .

**6.4.4 Balance**, with resolution from 0,01 mg to 0,1 mg, the range to be compatible with the mass of the parts to be weighed (see 6.2.6).

Depending on the balance room location, specific care may be required to avoid of instability reading related to vibrations, air draughts and temperature variations.

NOTE The weighing uncertainty is related not only to the balance characteristics, but to the whole procedure (see annex G).

**6.4.5 Thermometer and humidity meter**, located near the balance.

**6.4.6 Barometer**.

**6.4.7 Extraction hood and heating plate** for evaporation of the rinsing solutions. Their characteristics will depend on the evaporation procedure.

## 7 Calculation of nozzle diameter to obtain weighable quantities of dust

### 7.1 General

The nozzle size is estimated from either the volumetric flowrate characteristics for the particular sampling train to be used, or an initial assumption of the expected mass of particulate matter to be collected (obtained from process estimation or preliminary sampling).

### 7.2 Calculating the nozzle diameter from a characteristic sampling train flowrate

On the basis of the previously identified gas velocity ( $v_s$ ) at each sampling point, and the requirement that  $v_n = v_s$  (where  $v_n$  is the velocity of the gas through the nozzle), the area,  $A_n$  of the nozzle (and then the diameter,  $D_n$ ) is calculated simply as:

$$A_n \frac{\pi(D_n)^2}{4} = \frac{q_V}{v_n} \quad (2)$$

If the diameter of the nozzle ( $D_n$ ) is not suitable, a different volumetric flowrate  $q_V$  (or a different sampling train) is necessary.

### 7.3 Calculating the nozzle diameter when the sampling time is set and a required particulate mass is to be collected

In some standards, the sampling time is specified. Under such requirements, it may be necessary to increase the volumetric flowrate in order to collect a sufficient mass of particulate matter for weighing. The volumetric flowrate may be increased by increasing the size of the nozzle, however, changes in sample train design may be necessary to accommodate the increased flow requirements.

The volumetric flowrate through the nozzle ( $q_V$ ) is given by:

$$q_V = A_n v_n$$

where

$A_n$  is the area of the nozzle;

$v_n$  is the velocity of the gas through the nozzle

Under isokinetic conditions,  $v_n$  is equal to the gas velocity ( $v_s$ ), or  $v_n = v_s$ .

Under isokinetic sampling conditions, the volume of gas,  $V_n$ , through the nozzle, divided by the time,  $t$ , of sampling, gives the volumetric flowrate,  $q_V$ , under actual conditions, which is equivalent to  $A_n v_n$ :

$$q_V = \frac{V_n}{t} = A_n v_n = A_n v_s \quad (3)$$

and

$$A_n = \frac{V_n}{t v_s} \quad (4)$$

and if the expected dust concentration,  $c_{\text{exp}}$ , can be approximated from an estimated or desired mass,  $m$ , to be collected, then:

$$c_{\text{exp}} = \frac{m}{V_n} \quad (5)$$

therefore, from equation (4), the nozzle area can be estimated as:

$$A_n = \frac{m}{c_{\text{exp}} t v_s} \quad (6)$$

For high-volume sampling, characteristics of the entry nozzle shall be as specified in ISO 9096, allowing for nozzles of diameters from 1,25 cm to 3,43 cm to be used in order to achieve high isokinetic sampling rates.

## 8 Weighing procedure

### 8.1 General aspects

The filter shall be weighed with or without its support, and with or without all upstream parts from the filter, depending on the kind of sampling device to be used.

The rinsing solutions shall be evaporated and, depending on the procedure to be used, weighed in the same container or transferred to a smaller container for weighing.

### 8.2 Pre-sampling conditioning

Weighed parts shall be dried in a drying oven for at least 1 h, at a minimum of 180 °C (see clause 10).

Cool the filters and/or the weighing containers to ambient temperature in a desiccator located in the weighing room for at least 4 h. For larger parts, e.g. weighing containers, up to 12 h cooling may be necessary. If the humidity is controlled and dust is not hygroscopic, the filters and/or the weighing containers may be equilibrated in the weighing room.

### 8.3 Weighing

Since dust concentration is determined by difference between masses often obtained at one- or two-week intervals, special care is required in order to avoid weighing errors related to balance drift, to insufficient temperature equilibration of parts to be weighed, and to climatic changes (see examples in annex H). Therefore, before performing any measurement, the user shall validate the weighing procedure (see 8.6). It is strongly recommended to use the same balance for both pre-weighing and post-weighing.

Before each weighing series:

- a) check the balance against standard weights;
- b) carry out additional checks by weighing control parts, identical to the parts to be used in the measurement, pre-treated in the same temperature and humidity control conditions and kept free from contamination;
- c) record the climatic conditions in the room.

When weighing large-volume parts (e.g. beakers), the temperature and barometric pressure may influence the apparent mass. This may be detected using the reference mass of the control parts. In these conditions, weighing corrections shall be applied, based on the apparent mass modification of three identical control parts of each type (filter including support, container, etc.).

An increase or decrease in weighing result can be due to:

- a) electrostatic charges, which give erratic readings and which may have to be discharged/neutralized (metallic plate, ion gun);
- b) hygroscopic characteristics of the filter material and/or dust. Weighing shall be carried out within 3 min after removal from the desiccator. Three readings shall be taken at 1 min, 2 min and 3 min intervals. If a significant increase is detected, the sample shall be put back into the desiccator for at least 4 h. The weighing procedure is then repeated. The dry reference mass will then be calculated by extrapolation to zero time;
- c) small differences in temperature between the part to be weighed and the environment may disturb the balance.

#### 8.4 Post-sampling treatment of weighed parts

Dry all weighed parts in a drying oven for at least 1 h at 160 °C (see also clause 9 for specific cases). Afterwards equilibrate them to ambient temperature as described in 8.2.

When the presence of volatile or reactive compounds is suspected, dry at the sampling temperature, under a flow of dry nitrogen (see also 11.2).

#### 8.5 Post-sampling treatment of the rinsing solutions

All the solutions collected from rinsing all parts upstream of the filter with water and acetone as described in 9.5 are taken to the laboratory for further treatment. Care shall be taken that no contamination occurs.

Transfer the solutions carefully to the dried and pre-weighed containers (see 8.2). During the evaporation procedure, the solvent mixture shall not be boiled. As the volume of the solution is reduced through the evaporation process, progressively smaller containers may be used.

NOTE Proven methods for evaporation of rinsing solutions are:

- a) evaporation in an oven at 120 °C at ambient pressure. It is recommended to use air or nitrogen to dilute acetone vapour to a safe level;
- b) evaporation in a closed system (desiccator). The initial temperature is set at 90 °C and the pressure is reduced to 40 kPa (absolute). From time to time, the temperature is increased and the pressure is decreased. For the last period, they are kept at 140 °C and 20 kPa (absolute).

After the evaporation procedure, place the weighing containers in the drying oven for 1 h at 160 °C (see clause 10 for specific reasons), then cool to ambient temperature as described in 8.2.

Due to the relatively large mass and the volume of the weighing containers compared to the deposits under investigation, changes in the barometric pressure may influence the weighing results. Therefore, at least three empty weighing containers of equal size shall be weighed with each series. Any changes in mass may be used for correction.

Using the same volume of solvent, follow the same procedure to determine at least one blank value to be used for possible correction.

## 8.6 Improvement of the weighing procedure

Experience has shown that weighing uncertainties are related not only to the balance performance but to the whole procedure applied. Therefore, before performing any measurements, the user shall establish and validate his/her own procedure, taking into account the sampling equipment and filters to be used.

Repeated weighing of the same parts, spread over several weeks under various conditions (of e.g. external temperature, humidity, etc.), will provide, through the standard deviation, an estimation of the actual precision of weighing, including the uncertainties related to the manipulation of the filters, equilibrium time, etc.

Use the results as a first estimate of the overall blank value and to provide a means of calculating the volume of gas to be sampled in order to obtain significant data, taking into account the anticipated range of dust concentrations (see annex G).

## 9 Sampling procedure

### 9.1 General aspects

The following preliminary procedures shall be taken.

- a) Before carrying out any measurements, discuss the purpose of the sampling and the sampling procedures with the plant personnel concerned. The nature of the plant process, e.g. steady-state or cyclic, can affect the sampling programme. If the process can be performed in a steady state, it is important that this be maintained during sampling.
- b) Dates, starting times, duration of survey and sampling periods, as well as plant operating conditions during these periods shall be agreed with the plant management.
- c) Make preliminary calculations on the basis of expected dust concentration in order to verify that expected sampled dust quantities are consistent with attainable overall blank values, and that no overloading of the filter will occur (see annex G).

For sampling times limited to 0,5 h, required for certain trial or regulatory purposes, the uncertainty of measurement is in the range of  $2 \text{ mg/m}^3$  (see 12.2). Furthermore, completion of sampling along two diameters within 0,5 h will necessitate, even for medium-size ducts, simultaneous sampling with two teams, one on each sampling line.

Where possible, the sampling time can be extended, which decreases the detection limit to more practicable conditions (see annex G). The sampling time should be chosen taking into account the effect of non-steady state conditions of the stationary source and possible chemical reactions on collected dust on the filter.

- d) Taking into account the objective of the measurements and the conditions of waste gases to be sampled, the user shall
  - 1) choose between an in-stack or an out-stack filtration device. If gases in the duct are saturated (water,  $\text{SO}_3$ , etc.), out-stack filtration devices shall be used,
  - 2) in certain cases, choose a temperature for filtration and conditioning/drying of the filter before and after sampling (see clause 10).
- e) Take an overall blank sample after each measurement (see 9.6).

### 9.2 Preparation

Clean, prepare and check the equipment before moving to the test site. Do not reuse any part of a sampling train previously used for high dust concentration sampling without dismantling and thorough cleaning.

Filters and associated parts to be weighed shall be prepared for each measurement series as a function of the measurement programme. This includes the parts for the overall blank tests and spare parts (e.g. filter, filter holder, nozzles, etc.) to cope with process and equipment malfunctions.

Perform the weighing procedures in accordance with clause 8.

Protect all weighed parts, the suction tube and the other parts of the equipment that will come into contact with the sample (and will be rinsed later) from contamination during transportation and storage.

### 9.3 Premeasurements

Depending on the dimensions of the duct, which are to be verified, select the number and location of sampling points in accordance with 5.2 and annex C.

Measure the temperatures and velocities of the gas at the selected points in the duct, checking also for possible deviations of gas flow with respect to the duct axis; verify that the requirements of 5.2 are fulfilled. Otherwise, see 11.2.

In order to check for possible flowrate variations in the duct while sampling, install a separate Pitot tube or another suitable measurement device at a relevant fixed point on the sampling plane for the monitoring of the velocity.

Determine the composition of the exhaust, especially the contents of oxygen, CO<sub>2</sub> and water.

NOTE 1 Flow variations in the duct can also be checked using a Pitot tube mounted on the sampling equipment; this provides dynamic pressure measurement at each sampling point.

NOTE 2 The monitoring of the temperature in the duct and/or CO<sub>2</sub>/O<sub>2</sub> concentration (or other relevant parameter) may provide additional information on the steadiness of the stationary source.

Taking into account the preliminary calculations (see 7.2, and annex H) and the measured velocities, select a suitable entry nozzle diameter that will fulfill isokinetic conditions for all the sampling points.

### 9.4 Sampling procedure

The following steps shall be followed when sampling is carried out.

- a) Assemble the sampling equipment, and check for possible leaks by sealing the nozzle and starting the suction device. Care is required in this procedure because rapid changes in pressure and flow can damage the gas meter. The leak flowrate, e.g. measured by pressure variation after evacuation of the train at the maximum pressure reached during sampling, shall be below 2 % of the normal flowrate. During sampling, a leak check can be made by continuously measuring the concentrations of a relevant gas component (CO<sub>2</sub>, O<sub>2</sub>, etc.) both directly in the duct and downstream from the sampling train. Any detectable difference between those concentrations indicates a leak in the sampling equipment parts located out of the stack. This leak shall then be investigated and rectified.
- b) Preheat the relevant parts of the sampling train to the selected filtration temperature, e.g. stack temperature or recommended temperature of 160 °C ± 5 °C. Insert the sampling train into the duct with the nozzle, if possible, facing downstream, avoiding contact with any parts of the duct.  
Seal the opening of the access port to minimize air ingress or exposure of operators to toxic gases.
- c) Turn the sampling probe until the entry nozzle is facing upstream within ± 10°, open the shut-off valve, start the suction device and adjust the flowrate to obtain isokinetic sampling conditions within  $\pm \frac{15}{5}$  %.
- d) The sampling time at each selected point shall be identical.
- e) The total sampling time shall be at least 30 min.
- f) During sampling, check at least every 5 min and adjust the flowrate to maintain isokinetic sampling conditions within  $\pm \frac{15}{5}$  %. Continuously monitor or record, at least every 5 min, the dynamic pressure measured by the Pitot tube or another suitable measurement system either installed at the fixed point or mounted on the sampling equipment (see 9.3).

NOTE Good laboratory practice when using the dry-gas meter is to record at least every 5 min the gas meter temperature and pressure, and to use the results for calculating the final sampled volume.

- g) Do not stop sampling when moving the sampling train to the next sampling point, and immediately adjust the flowrate to obtain isokinetic conditions.
- h) Record the sampling time and sampled volume or flowrate at each sampling point.

- i) On completion of sampling at all the selected points of one sampling line, close the shut-off valve and the suction device, remove the sampling train from the duct and reposition it on the next sampling line.

For low dust concentration measurement, it is recommended to use only one filter for a complete measurement (cumulative sampling).

The filter load and the maximum gas velocity shall not exceed the filter manufacturer's recommendations.

- j) On completing the entire sampling run at all points:
- 1) remove the sample train after closing the shut-off valve and suction device;
  - 2) check the equipment for leaks as under 9.4 a) if leakage has not been monitored during sampling;
  - 3) dismantle the sampling equipment and visually check the filter and the filter holder for signs of breakage or stains due to pressure or to the concentration of moisture (sampling equipment operated below or too close to the dew point). If such signs are detected, the test is not valid. Check also for non-uniform distribution of dust on the filter.
- k) Measure and record the barometric pressure.
- l) Put the parts to be weighed in a closed electrostatic-free container for transport to the laboratory for weighing (see clause 8).

## 9.5 Recovery of deposits upstream of the filter

Rinse all the non-weighed parts that are in contact with the sample gas upstream of the filter to recover the deposits, unless the quantification of possible deposits is not required (see 11.5).

Take special care to avoid contamination if the rinsing is done on site. Carry out rinsing according to the following procedure.

- a) Rinse the nozzle, elbow and other parts upstream of the filter carefully with water into a storage container, taking care that nothing from the outside falls into the container. Repeat the procedure with a second rinsing of water followed by acetone into the same container.
- b) To rinse the suction tube, seal one end and fill with enough water to wet and clean the inner surface (1/3 to 1/2 of the volume of the suction tube) and then seal the other end. Clean the tube by rotating and tilting several times. Transfer the solution to the transport storage container. Repeat the procedure with a second rinsing of water followed by acetone into the same container.

No mechanical cleaning shall be applied to recover dust deposits upstream of the filter. However, it is necessary before each measuring series to mechanically clean and rinse the equipment.

Rinse the upstream parts at least after each measurement series on the same sampling plane, and at least once a day.

Attribute the recovered mass to individual tests in proportion to the mass collected on each filter.

## 9.6 Overall blank sample

Take an overall blank sample after each measurement series or at least once a day, following the sampling procedure described in 9.4 without starting the suction device. This provides an estimate of the dispersion of results related to the whole procedure as carried out by the operators for a near-zero dust concentration, i.e. contamination of filters and of rinsing solutions during handling on site, transport, storage, handling in the laboratory and weighing procedures. All overall blank values shall be reported individually.

## 10 Thermal behaviour of dusts

Emitted dusts are generally thermally stable. However, in some processes the gases to be sampled contain unstable or semi-volatile compounds (usually in particulate form at low temperature, in gaseous form at higher temperature).



In such cases, the measured dust concentration depends on the filtration temperature and/or on the drying temperature before final weighing.

Particulate matter may be defined in national standards in terms of a specified standard temperature (e.g. 125 °C, 15 °C). In such cases, the sampling system shall be designed so as to collect pollutant materials that exist as particulate matter [solid phase or liquid droplet (aerosol)] at the specified temperature. This can be done by installing the filter and filter assembly externally to the probe, into a temperature-controlled chamber.

If materials that are condensable in ambient air are included in the national standard definition for particulate matter, it may be necessary to include materials caught in a cooled impinger assembly added after the filter. Suitable extractive methods for organic or other materials should then be defined in the national standard. This International Standard does not address such procedures, however it is noted that care should be taken that any procedures applied do not result in the introduction of artifacts non-representative of actual emissions.

Such phenomena have been reported in various industries, e.g.:

- a) power plants equipped with desulfurization processes, because of the occurrence of hydrates;
- b) heavy fuel oil power plants or diesel engines, causing the formation of SO<sub>3</sub> and/or organic emissions;
- c) glass furnaces, because the occurrence of semi-volatile boron compounds has been experienced;
- d) waste incinerators with wet and semi-dry gas treatment processes.

Differences (up to a factor of 10) in the measured dust concentrations have been experienced and therefore in such cases, the measured results shall be associated with a stated temperature (i.e. the highest temperature sustained by the sampled dust before weighing). Because of the extreme variety of the situations that may be encountered, it is not possible to specify a conventional temperature that could be relevant in all cases.

However, since the complete trapping of volatile compounds would necessitate a very low filtration temperature and special care during sampling, more reproducible results may be achieved if these compounds are not trapped or are further evaporated when drying. Therefore a conventional temperature of 160 °C, which avoids the trapping of most volatile compounds and decomposes most hydrates, is generally convenient.

It is however possible to obtain reproducible results if unstable or semi-volatile compounds are either not trapped and/or are further evaporated when drying. In such cases, the measured results are only consistent when associated with a specified temperature, such as the highest temperature sustained by the sampled dust before weighing.

According to this convention, the parts of the sampling train to be weighed should therefore be

- conditioned at 180 °C before sampling,
- set at any temperature equal to or less than 160 °C during sampling,
- conditioned at 160 °C after sampling.

Depending on regulatory requirements and plant authorization, on special kinds of effluents, or on the specific objective of the measurement, other conventional temperature treatments can be adopted: e.g. if aerosols or condensable compounds are to be taken into account, the temperature shall be reduced during post-sampling treatment.

In any case:

- the weighed parts shall be conditioned before sampling (see 9.1) at a temperature at least 20 °C above the maximum temperature reached during sampling and post-sampling treatment;
- the temperature used while sampling and while conditioning before weighing shall be indicated in the test report.

## 11 Validation of results

### 11.1 General

Annex F provides a summary of the requirements mentioned in this International Standard.

### 11.2 Parameters depending on the stationary source

If no suitable sampling location (see 5.1) exists in the plant, and/or measurements have been carried out during non-steady state conditions of the plant, which leads to an increase in the uncertainty of the measurements, it shall be stated in the test report that the measurement is not in accordance with this International Standard.

Details on the characteristics of the flow at the sampling location and/or on the variations of the flowrate in the duct while sampling shall be stated in the test report.

### 11.3 Leak check

Errors are caused by the occurrence of leaks in the sampling train. Leakage shall be less than 2 % of the minimal flowrate or, when using monitors, any detectable difference shall be investigated and rectified or a leak test carried out as described in 9.4 a).

Therefore, the sampling train shall be checked for leaks.

### 11.4 Isokinetic flowrate

If the mean actual isokinetic flowrate during the sampling at the sampling plane varies by more than  $-5\%$  to  $+15\%$ , the measurement is not valid.

If this criterion is not fulfilled due to frequent variation of the flowrate in the duct, see 11.2.

### 11.5 Deposits of dust on non-weighed parts upstream of the filter

Experimental work carried out when preparing EN 13824-1 proved that dust deposits upstream of the filter are often in the range of 10 % to 30 % of the total dust when sampling gases from waste incinerators at particulate concentrations around  $5\text{ mg/m}^3$ .

These deposits probably depend on the design of the sampling equipment, and on the kind of dust to be sampled, but no efficient means was found to keep them at a negligible level. For this reason, all non-weighed parts upstream of the filter shall be rinsed. The mass of the dust on non-weighed parts upstream of the filter shall be indicated in the test report, in addition to the mass on the filters used during the same measurement series.

When sampling with in-stack filtration devices with no bends between the nozzle and filter holder (see annex E) on non-saturated gases, with a temperature well above the stack gas dew-point, the upstream deposits do not have to be quantified provided that validation has been carried out under conditions similar to the process to prove that the deposit concentration, expressed in the same units as the measurement results (e.g. milligrams per cubic metre), does not exceed 10 % of the daily average limit value set for the process.

### 11.6 Overall blank

The overall blank shall not exceed 10 % of the daily limit value set for the dust concentration.

No result below the overall blank value is valid.

Weighing uncertainties (see annex I) contribute to the overall blank, therefore the weighing uncertainties shall be less than 5 % of the limit value.

## 12 Calculations

### 12.1 Sampling volumetric flowrate

In order to perform isokinetic sampling, calculate the required sampling flowrate, taking into account the velocity of the gas in the duct at the sampling point (see annex B), and the effective diameter of the sampling nozzle. For isokinetic sampling, the velocity of the gas at the nozzle,  $v_n$ , is equal to the flue gas velocity at the sampling point,  $v_s$ , or:

$$v_n = v_s \quad (7)$$

The volumetric flowrate at the nozzle,  $q_V$ , expressed in the actual conditions at the nozzle is:

$$q_V = A_n \cdot v_n \quad (8)$$

According to equation (7), isokinetic sampling is conducted when:

$$q_V = A_n \cdot v_s \quad (9)$$

where  $A_n$  is the area of the nozzle.

Because the sampling flowrate is measured under conditions (temperature, pressure, humidity) that usually differ from the actual conditions of the gas in the duct,  $q_V$  shall be corrected as follows:

$$q_{V,m} = q_{V,a} \frac{100 - H_a(T_m \cdot p_a)}{100 - H_m(T_a \cdot p_m)} \quad (10)$$

where

$q_{V,m}$  is the sampling flowrate at the gas meter;

$q_{V,a}$  is the sampling volumetric flowrate through the nozzle in the duct (actual conditions);

$H_m$  is the moisture, expressed as a volume fraction (percent), of gases at the gas meter;

$H_a$  is the moisture, expressed as a volume fraction (percent), of gases in the duct (actual conditions);

$T_m$  is the temperature of gases at the gas meter, in kelvin;

$T_a$  is the temperature of gases in the duct (actual conditions), in kelvin;

$p_m$  is the absolute pressure of gases at the gas meter measurement;

$p_a$  is the absolute pressure of gases in the duct (actual conditions).

Compare the  $q_V$  target value to the  $q_{V,a}$  obtained during the test, in order to check for isokinetic sampling compliance.

### 12.2 Dust concentration

For each test, calculate,

- a) the sample volume  $\bar{V}$ , specifying whether it is expressed on a dry or wet basis and under normal conditions;
- b) the total mass of dust collected,  $m$ , upstream of the filter (rinsings) and on the filter;

c) the dust concentration,  $c$ :

$$c = \frac{m}{V} \quad (11)$$

It is sometimes necessary to express dust concentrations relative to a reference  $O_2$  concentration, to correct for dilution effects.

The measured dust concentration shall be multiplied by the correction factor  $f_c$ :

$$f_c = \frac{21 - \varphi_{O_2,ref}}{21 - \varphi_{O_2,m}} \quad (12)$$

where

$\varphi_{O_2,ref}$  is the volume fraction, in percent, of dry  $O_2$  under reference conditions;

$\varphi_{O_2,m}$  is the volume fraction, in percent, of dry  $O_2$  measured in the duct;

21 is the percent of oxygen in air.

### 13 Performance characteristics of the method

Because waste gas composition varies with time, it is not possible to determine the repeatability and reproducibility of the method in accordance with ISO 5725 (all parts). See annex I for precision data.

However, performance of successive parallel sampling tests with two identical systems allows a statistical comparison between paired values  $x_1$  and  $x_2$  to be calculated.

The standard deviation,  $s$ , of the paired values is calculated as follows:

$$s = \frac{\sqrt{\sum_{i=1}^n (x_{i,1} - x_{i,2})^2}}{2n} \quad (13)$$

where  $n$  is the number of sample pairs  $x_1$  and  $x_2$ .

The standard deviation may be used for the calculation of:

a) the internal uncertainty  $u$  (or internal confidence interval) linked to an individual measurement carried out by a team:

$$u = t_{0,95; n-1} \cdot s$$

where  $t_{0,95; n-1}$  is the Student factor for a 95 % confidence and  $n - 1$  degrees of freedom;

b) the repeatability,  $r$ , in accordance with ISO 5725, i.e. the maximum difference between two measurements by the same team, for a 95 %, confidence level:

$$r = \sqrt{2} t_{0,95; n-1} \cdot s \quad (14)$$

These data are to be considered as tools for measuring institutes in the framework of quality assurance.

When data are provided by several independent teams operating together, similar calculations can be conducted and provided as an estimation of:

- the external uncertainty linked to an individual measurement carried out by any team in compliance with the requirements of this International Standard. This uncertainty shall be taken into account when comparing the measured values to the emission limit value;
- the reproducibility,  $R$ , i.e. the maximum difference, that can be expected, at a 95 % confidence level, between two measurements carried out by different teams working in accordance with this International Standard at the process conditions.

When carrying out measurements at low level concentrations, the detection limit may be estimated

- by parallel measurements and calculation of the uncertainty,
- by successive measurements at near zero concentration. The detection limit is assumed to be three times the standard deviation.

## 14 Test report

The test report shall refer to this International Standard, and shall include the following information:

- a) identification of the customer and of the persons responsible for and involved in the measurements;
- b) description of the purpose of tests, identification of the site, date of sampling;
- c) description of the operating conditions of the plant process, and any variation during measurements;
- d) identification of the sampling location, and gas parameters in the duct:
  - 1) duct dimensions, number and position of sampling lines and sampling points,
  - 2) velocity and temperature profile,
  - 3) O<sub>2</sub>/CO<sub>2</sub>, humidity concentrations, volumetric mass of gases,
  - 4) compliance with requirements of 5.2;
- e) measurement procedures:
  - 1) velocity measurement (calibration of velocity measurement devices other than standardized Pitot-tubes, etc.);
  - 2) characteristics of sampling equipment:
    - i) nozzle diameter,
    - ii) filter (material, sizes, etc.),
    - iii) calibration of volume or flowrate measurement devices,
    - iv) filtration temperature;
  - 3) weighing procedures:
    - i) conditioning temperature,
    - ii) correction of apparent masses;

## ISO 12141:2002(E)

f) test results:

- 1) number of tests;
- 2) for each test:
  - i) date, time and duration,
  - ii) sampled volume and average flowrate,
  - iii) dust mass on the filter and in the rinse solutions,
  - iv) raw results (dust on filter, dust in rinses) and corrected results (standard conditions),
  - v) any special circumstance or incidents.

Raw data, e.g. gross mass, average temperature during sampling, average dust pressure during sampling, etc., shall be included in the report or shall be available for inspection.

g) quality assurance:

- 1) leak test results,
- 2) overall blank value,
- 3) compliance with isokinetic criterion;

h) comments:

- 1) value of the ratio of the overall blank value to the measured values in the same unit,
- 2) any special circumstances that might have influenced the results, and any information concerning the uncertainty of the results (see annex, etc.),
- 3) any modifications to the method, for any reason.

## Annex A (normative)

### Requirements related to the working platform

For safety reasons, the permanent and temporary working platform shall be provided with

- a) an adequate working area, normally not less than 5 m<sup>2</sup>;
- b) capacity to bear at least 400 kg point load;
- c) handrails (approximately 0,5 m and 1 m high) and vertical baseboards (approximately 0,25 m), handrails shall have removable chains across the top of the ladders or self-closing gates;
- d) waterproof electrical sockets, plugs and equipment if they are to be exposed to weather.

For practical and quality assurance reasons, the working platform

- shall be positioned relative to the access ports in such a way that the handrail is clear of the apparatus to be used and free from obstructions that would hamper insertion and removal of the sampling equipment (the length of which may exceed 4 m for large ducts);
- shall have a minimum length and breadth across each access port of from 2 m to more than 3 m (the length of the probe plus 1 m).

The measuring site shall have artificial lighting and be ventilated. Provisions shall be made for requisite electric power, and upon request, water and compressed air, etc., shall be supplied. Hoists for raising and lowering of equipment may be needed.

Suitable protection shall also be considered for personnel and equipment if the platform is exposed to weather.

.....

## Annex B (informative)

### Determination of flow direction with Pitot tubes

#### B.1 Type L Pitot tube

The type L Pitot tube provides an accurate measure of gas velocity when the Pitot tube is aligned (yawed) within  $15^\circ$ , of the direction of the gas flow. However, the pressure difference between the two pressure-sensing orifices decreases sharply when misalignment exceeds  $15^\circ$ , until a negative response occurs when the head is at  $90^\circ$  to the gas flow. This provides a simple method for estimating gas flow direction and may be used to test for the presence of swirling flow within the duct.

#### B.2 Type S Pitot tube

The type S Pitot tube accurately measures the gas velocity to within 4 % when the Pitot tube is aligned (yawed) within  $15^\circ$  of the direction of the gas flow. However, when the planes of the Pitot tube pressure-sensing orifices are parallel to the stack flow, a null (zero) reading is obtained. Thus, the pressure of swirling flow can be determined by rotating the type S Pitot tube (with the manometer properly zeroed and levelled) until a null reading is obtained. The direction of flow will be parallel to the pressure-sensing orifice planes.



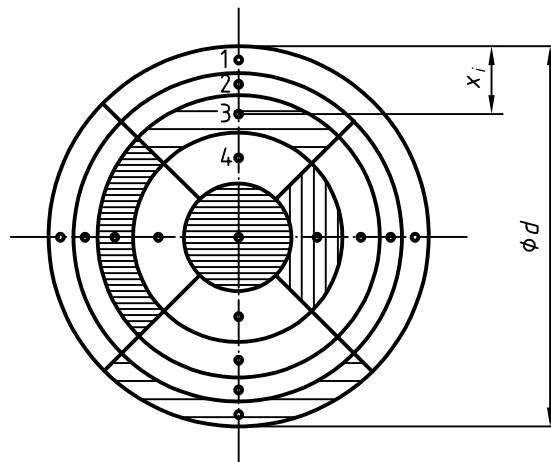
## Annex C (normative)

### Determination of positions of sampling points in circular and rectangular ducts

#### C.1 Requirements for circular ducts

##### C.1.1 General

For circular ducts, the sampling plane shall be divided into equal areas. The sampling points, one at the centre of each area, shall be located on two or more diameters (sampling lines), and one sampling point at the centre of the duct (see Figure C.1).



**Figure C.1 — Sampling-point positions in circular ducts — General rule for ducts over 2 m diameter —**  
(hatched positions are of equal area)

The locations of the sampling points depend on the number of sampling points chosen. For circular ducts, two duct diameters are sufficient. The distance,  $x$ , of each sampling point,  $i$ , from the duct wall may be expressed as:

$$x_i = k_i \cdot d \quad (\text{C.1})$$

where

$k_i$  is a factor, expressed as a percentage, in accordance with Table C.1;

$d$  is the length of the diameter of the duct.

Table C.1 gives values of  $k_i$  as a percentage, where  $n_d$  is the number of sampling points per diameter and  $i$  is the position number (index) of the individual sampling point along the diameter.

**Table C.1 — Value of  $k_i$  as a percentage — General rule for circular ducts**

Sampling point index $i$	$k_i$			
	$n_d = 3$	$n_d = 5$	$n_d = 7$	$n_d = 8$
1	11,3	5,9	4,0	3,0
2	50,0	21,1	13,3	9,8
3	88,7	50,0	26,0	17,8
4		78,9	50,0	29,0
5		94,1	74,0	50,0
6			86,7	71,0
7			96,0	82,2
8				90,2
9				97,0

For circular ducts where it is necessary to increase the number of sampling lines (diameters) or the number of sampling points (because of adverse flow conditions, for instance), the general formulae (C.1) for calculating the distance of the sampling point, from the duct wall along the diameter become:

$$x_i = \frac{d}{2} \left[ 1 - \sqrt{\frac{n_d(n_i - 2i) + 1}{n_d(n_i - 1) + 1}} \right] \quad \text{for } i < \frac{n_i + 1}{2} \tag{C.2}$$

$$x_i = \frac{d}{2} \quad \text{for } i = \frac{n_i + 1}{2} \tag{C.3}$$

$$x_i = \frac{d}{2} \left[ 1 - \sqrt{\frac{n_d(2i - 2 - n_i) + 1}{n_d(n_i - 1) + 1}} \right] \quad \text{for } i > \frac{n_i + 1}{2} \tag{C.4}$$

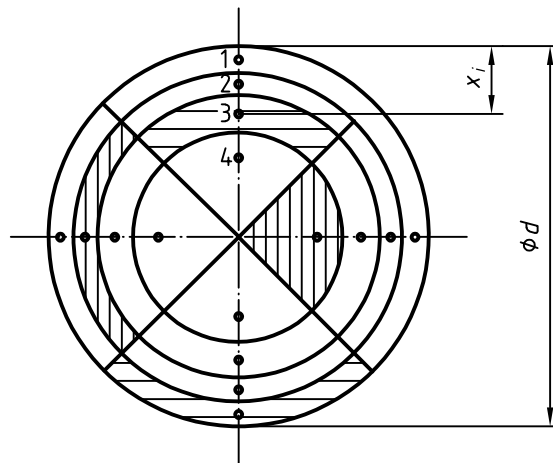
where

- $x_i$  is the distance of point  $i$  from the duct wall;
- $d$  is the length of the diameter of the duct;
- $i$  is the index of sampling point along the diameter;
- $n_i$  is the number of sampling points along each sampling line (including the centre);
- $n_d$  is the number of diameters of sampling.

### C.1.2 Tangential rule

In the “tangential rule” applicable to circular ducts, the sampling plane is divided into equal areas. The sampling points, one at the centre of each area, are located on two or more diameter (sampling lines), there being no sampling point at the centre of the duct (see Figure C.2).

The locations of the sampling points on each diameter depend on the number of sampling points on each diameter, but are independent of the number of sampling diameters.



**Figure C.2 — Sampling-point positions in circular ducts — Tangential rule for ducts over 2 m diameter**

For circular ducts where two sampling lines (diameters) are sufficient, the distance of each sampling point from the duct wall may conveniently be expressed as in clause C.1:

$$x_i = k_i \cdot d \tag{C.5}$$

where

$k_i$  is a factor, expressed as a percentage, in accordance with Table C.2.

Table C.2 give values of  $k_i$  as a percentage, where  $n_d$  is the number of sampling points per sampling line (diameter), and  $i$  is the position number (index) of the individual sampling point along the diameter.

**Table C.2 — Values of  $k_i$  as a percentage — Tangential rule for circular ducts**

Sampling point index $i$	$k_i$			
	$n_d = 2$	$n_d = 4$	$n_d = 6$	$n_d = 8$
1	14,6	6,7	4,4	3,3
2	85,4	25,0	14,6	10,5
3		75,0	26,0	17,8
4		93,3	70,4	32,3
5			85,4	67,7
6			95,6	80,6
7			96,0	82,2
8				89,5
9				96,7

For circular ducts where it is necessary to increase the number of sampling lines (diameters) or the number of sampling points, the tangential formulae (C.5) for calculating the distance from the duct wall along the diameter become:

$$x_i = \frac{d}{2} \left[ 1 - \sqrt{1 - \frac{2i - 1}{n_i}} \right] \text{ for } i \leq \frac{n}{2} \tag{C.6}$$

$$x_i = \frac{d}{2} \left[ 1 + \sqrt{1 - \frac{2i - 1}{n_i}} \right] \text{ for } i > \frac{n}{2} \tag{C.7}$$

where the symbols have the same meaning as in formulae (C.2) to (C.4).

This method is particularly useful for large ducts where it is difficult to reach the centre of the duct.

### C.2 Requirements for rectangular ducts

For rectangular ducts, the sampling plane shall be divided into equal areas by lines parallel to the sides of the duct, and a sampling point located at the centre of each area. (See Figure C.3.)

In general, the two perpendicular sides of the rectangular duct are divided into an equal number of parts, giving areas that have the same shape as the duct. The number of partial areas is thus the square of 1, 2, 3, etc. depending on the number of divisions per side. [See Figure C.3 a).]

If the lengths of the sampling plane sides  $l_1$  and  $l_2$  have a ratio  $l_1/l_2 > 2$ , side  $l_1$  must be divided by a number greater than  $l_2$ , so that each of the smaller areas meets the criterion that the longer side wall shall not be more than twice the length of the shorter side.

If the lengths of the sides of the sampling plane  $l_1$  and  $l_2$  are divided into  $n_1$  and  $n_2$  parts respectively, the number of sampling points will be  $n_1 \cdot n_2$  and the smallest distance from a wall of the duct will be  $l_1/2n_1$  and  $l_2/2n_2$ .

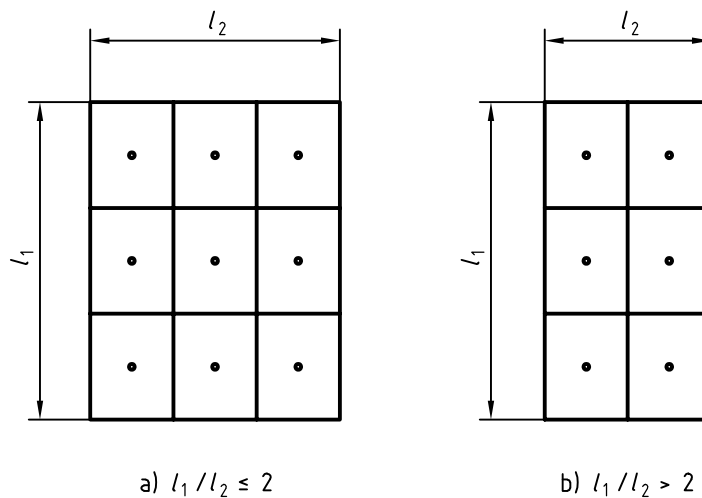


Figure C.3 — Illustrations of sampling point positions in rectangular ducts

## Annex D (informative)

### Examples of suitable access ports for sampling equipment

Dimensions in millimetres

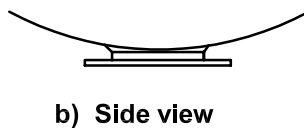
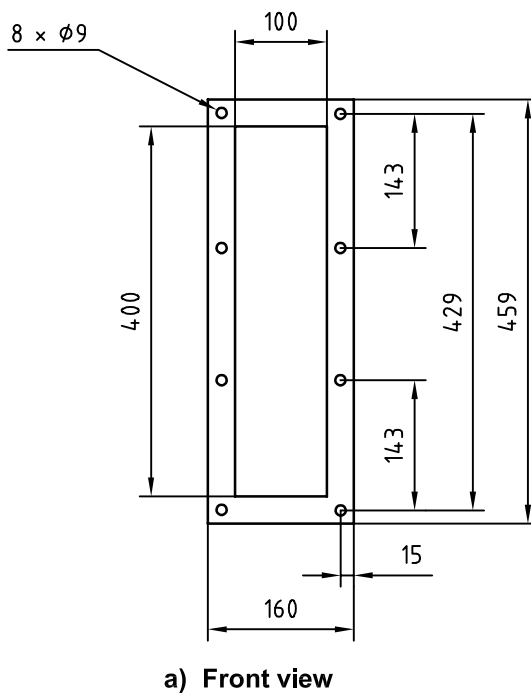
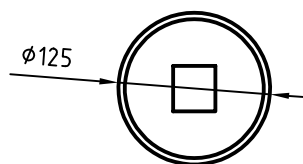
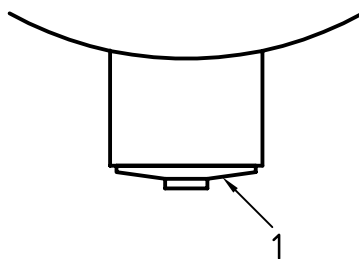


Figure D.1 — Rectangular access port



a) Front view



b) Side view

**Key**

1 Screw-in plug

**Figure D.2 — Circular access port**

## Annex E (normative)

### Proven entry nozzle designs

Figures E.1 to E.3 show three designs for an entry nozzle, which are:

a) Case A (see Figure E.1)

For  $e < \frac{d_i}{12}$ , but for mechanical reasons,  $e = 0,8 \text{ mm}$  may be accepted

$l_2 \geq 10 \text{ mm}$ ;

$l_1 \leq 0,2 \text{ mm}$ .

Effective diameter  $D_{\text{eff}} = d_i + 2l_1$

where

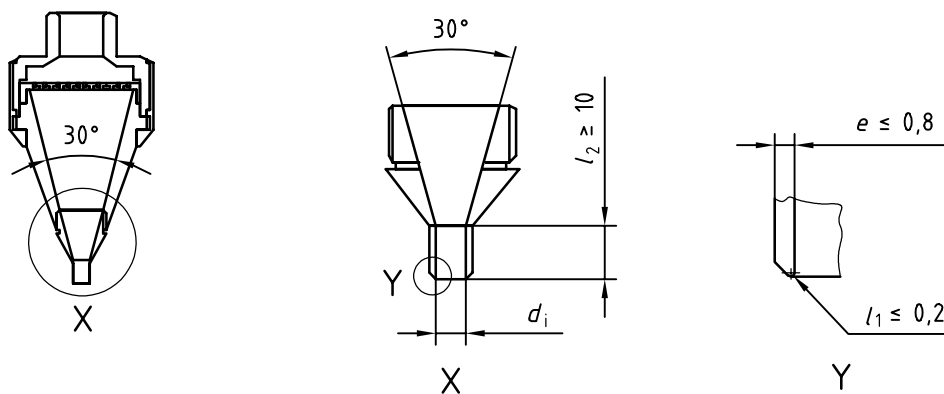
$e$  is the thickness of the side of the entry nozzle;

$d_i$  is the internal diameter of the entry nozzle;

$l_1$  is the length of the bevel;

$l_2$  is the length with constant internal diameter.

Dimensions in millimetres



**Figure E.1 — Entry nozzle with combined filter holder**

b) Cases B and C (see Figures E.2 and E.3)

For  $e < 0,2 \text{ mm}$ .

Effective diameter  $D_{\text{eff}} = d_i + e$

where

$e$  is the thickness of the side of the entry nozzle;

$d_i$  is the internal diameter of the entry nozzle;

$l_1$  is the length of the level;

$l_2$  is the length with constant internal diameter.

Dimensions in millimetres

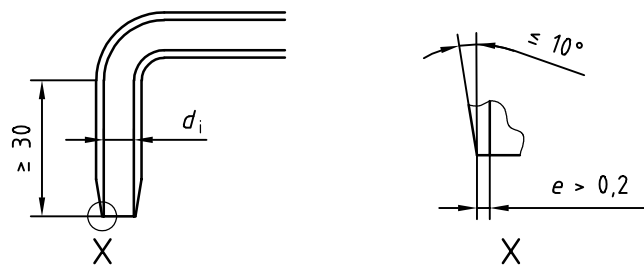


Figure E.2 — Entry nozzle, single angle

Dimensions in millimetres

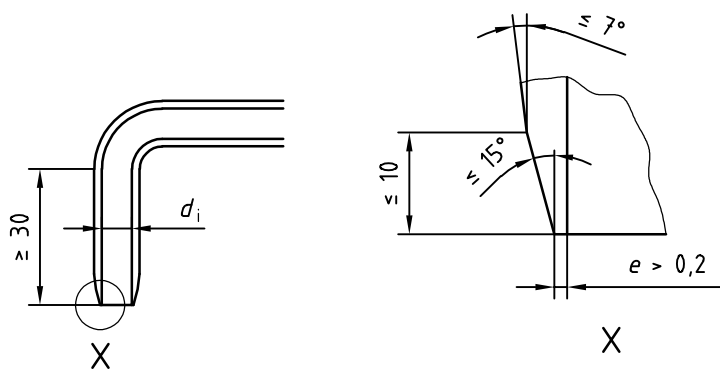


Figure E.3 — Entry nozzle, double angle



## Annex F (normative)

### Summary of apparatus requirements

Apparatus: dust	Value	Subclause
Nozzle internal diameter	> 6 mm	6.2.3
Nozzle: uncertainty of area at nozzle entry	$\pm 10\%$	6.2.3
Nozzle: length with constant internal diameter	> 10 mm	6.2.3
Nozzle: change in diameter angle	$< 30^\circ$	6.2.3
Nozzle: radius of the bend	> 1,5 times internal diameter	6.2.3
Nozzle straight length before the first bend	> 30 mm	6.2.3
Filter holder distance to obstacles	> 50 mm	6.2.3
Filter: — efficiency on test aerosol of 0,3 $\mu\text{m}$ — efficiency on test aerosol of 0,6 $\mu\text{m}$	$> 99,5\%$ $> 99,5\%$	6.2.6 6.2.6
Filter material	no reaction and no absorption of the components	6.2.6
Condenser, drying tower: residual gas moisture	$< 10 \text{ g/m}^3$	6.2.7
Gas meter: — uncertainty of gas volume — uncertainty of absolute pressure — uncertainty of absolute temperature	$\pm 2\%$ $\pm 1\%$ $\pm 1\%$	6.2.7 6.2.7 6.2.7
Angle of alignment of the nozzle	$\pm 10\%$	8.4
Isokinetic criteria (average within)	95 % to 115 % related to gas velocity	8.4
Leak rate	$< 2\%$	11.2
Balance: resolution	0,01 mg to 0,1 mg	6.4
Weighing uncertainties	$< 5\%$ of the LV <sup>a</sup>	11.6
Temperature equilibrium duration (weighed parts)	4 h to 12 h	8.2
Overall blank value	$< 10\%$ of the LV <sup>a</sup>	11.6
Sampling location		
Duct gas flow	$< 15^\circ\text{C}$	5.2
Duct gas flow: negative velocity	not permitted	5.2
Duct gas flow: differential pressure at Pitot tube	$< 5 \text{ Pa}$	5.2
Duct gas flow: Ratio of max. to min. velocity	$< 3 : 1$	5.2
Straight length before sampling plane	5 hydraulic diameters (recommended)	5.2
Straight length after sampling plane	2 hydraulic diameters (recommended)	5.2
Straight length before emission point	5 hydraulic diameters (recommended)	5.2
Number of sampling points	See 5.3	5.3
Apparatus: flue gas characteristics		
Flue gas density: uncertainty	$\pm 0,05 \text{ kg/m}^3$	

<sup>a</sup> LV: Limit Value. A typical limit value (LV) for which this International Standard is applicable, is 20 mg/m<sup>3</sup> or less. Process operations should be such that at the time of the test, measured results will be in the range of the limit value. At higher values, other standard test methods may be applicable.

## Annex G (informative)

### Sampling volume, flowrate and duration

#### G.1 General

The minimum gas volume to be sampled is derived from the uncertainties in dust weighing and the reference dust concentration (Limit Value set for the process,  $LV$ ).

#### G.2 Weighing uncertainties

These uncertainties are not only related to the balance performances, but to the whole weighing procedure. According to 8.6, they are to be determined by repeated weighing of filters and weighing containers.

The mass of dust  $\Delta m$  to be collected when sampling at  $LV$  concentration is at least 20 times the weighing uncertainties (see 11.6).

#### G.3 Sampling volume

The necessary minimum sampling volume  $V_{\min}$  is then determined by the following equation:

$$V_{\min} = \frac{\Delta m}{LV} \quad (\text{G.1})$$

#### G.4 Sampling flowrate and duration

When the sampling duration  $t_s$  is limited (e.g. 30 min), the minimum sampling flowrate  $q_{\min}$  is:

$$q_{\min} = \frac{V_{\min}}{t_s} \quad (\text{G.2})$$

where  $q_{\min}$  should be compared to the practical flowrate that can be achieved by the sampling equipment used (filter pressure drop, pump characteristics, etc.)

## Annex H (informative)

### Examples of weighing bias

#### H.1 General

Weighing biases related to insufficient temperature equilibrium, and to climatic changes between pre- and post-sampling weighings, are illustrated in the following example.

In this example, the filter is placed in a closed glass Petri box, mass 25 g , inside air volume 40 ml. The balance is calibrated against a standard mass 25 g (volumetric mass 8 g/ml). Volumetric mass of glass 2 g/ml, of air, 1,2 mg/l.

#### H.2 Effect of insufficient temperature equilibrium

Because of insufficient time to equilibrate the temperature after drying, the air inside the Petri box is assumed to have a temperature 2 K higher than that of the room balance (300 K). Due to the change in air buoyancy, this difference of air temperature leads to an apparent mass variation of:

$$40 \text{ ml} \times 1,2 \text{ mg/l} \times \frac{2 \text{ K}}{300 \text{ K}} = 0,3 \text{ mg} \quad (\text{H.1})$$

#### H.3 Effect of temperature variations

The room balance temperature is assumed to be 15 °C when weighing before sampling, and 25 °C when weighing after sampling.

The difference between the volume of air displaced by the standard mass (25 g, volume 3,1 ml) and by the Petri box (25 g, volume 12,5 ml) is 9,4 ml.

Due to the temperature change (10 K) this air volume leads to an apparent mass modification of:

$$9,4 \text{ ml} \times 1,2 \text{ mg/l} \times \frac{10 \text{ K}}{300 \text{ K}} = 0,4 \text{ mg} \quad (\text{H.2})$$

#### H.4 Effect of barometric pressure variations

The barometric pressure is assumed to be:

- a) when weighing before sampling: 98,5 kPa (740 mmHg);
- b) when weighing after sampling: 104 kPa (780 mmHg).

Therefore a relative variation of 5,5 % exists.

Due to this relative variation, the 9,4 ml air volume leads to an apparent mass modification of:

$$9,4 \times 1,2 \times 0,055 = 0,6 \text{ mg.} \quad (\text{H.3})$$

## H.5 Conclusions

- a) When weighing parts of large internal volume, it is mandatory to wait for ambient and internal temperatures to equilibrate before weighing;
- b) There is no need to correct for temperature effects if the room where the balance is situated is thermally controlled. But it remains necessary to take into account the effect of barometric pressure variations, especially if the volumetric mass of parts to be weighed is very different from those of standard masses used for calibration. The required correction may be done by weighing the “control parts”, as indicated in 8.2.

## Annex I (informative)

### Precision data

#### I.1 Data obtained from CEN collaborative study

##### I.1.1 Experimental data

Validation tests were performed in two municipal waste incinerators equipped with different types of gas treatment:

- a) plant A: semi-dry process, with a fabric filter, stack gas temperature 140 °C;
- b) plant B: electrostatic precipitator with a scrubber, stack gas temperature 60 °C, water-saturated.

Sampling duration was limited to 30 min.

The results are given in Table I.1.

**Table I.1 — Results of validation test for 30 min sampling duration**

	Plant A		Plant B
Teams in parallel	4		3
Out-stack/in-stack devices	1/3		3/0
Number of successive tests	32		16
	Dust on filter only	Total dust, filter + rinse	Total dust
Mean concentration, mg/m <sup>3</sup>	4,7	6,4 (2 to 18)	2,5 (0,3 to 6,8)
Repeatability (1 team using 2 sampling trains)	2 to 17	3 to 19	0,3 to 6,8
External uncertainty	2,4	4,0	1,8
Reproducibility (2 teams)	3,4	5,7	2,6

The detection limit was estimated from results by one team to be:

- for dry gases: approximately 0,3 mg/m<sup>3</sup> (dust on filter only);
- for water-saturated gases: approximately 2 mg/m<sup>3</sup> (total dust).

##### I.1.2 Comments on the CEN experimental data

During the above tests, some high overall blank values were reported (+ 1 mg/m<sup>3</sup> or higher) due to weighing uncertainties of the dry extracts from the rinses, use of vessels of improper materials, etc.

Further investigation showed that these uncertainties can be reduced to less than 0,5 mg/m<sup>3</sup>, leading to an improvement in repeatability and reproducibility.

Increasing the sampling time to 60 min or 90 min will improve significantly the reproducibility of the measurements.

#### I.2 Precision of the high volume method

The percent coefficient of variation of a high-volume test method (ASTM D-4536-91) was found to be about 8 %. This value was determined through a collaborative test in which five laboratories each sampled the stack of their choice using two sampling trains with nozzles positioned very close to each other in the stack. Each laboratory collected six to eight sets of paired-train samples from their stack, for a total of twelve to sixteen samples per laboratory. Stack dust concentrations of 5,6 mg/m<sup>3</sup> were encountered in these evaluations.

## Bibliography

- [1] ISO 6879:1995, *Air quality — Performance characteristics and related concepts for air quality measuring methods*
- [2] ISO 10155:1995, *Stationary source emissions — Automated monitoring of mass concentrations of particles — Performance characteristics, test methods and specifications*
- [3] ASTM-D4536-1991, *Standard Test Method for High-Volume Sampling for Solid Particulate Matter and Determination of Particulate Emissions*
- [4] EN 13284-1:—<sup>1)</sup>, *Stationary source emissions — Determination of low range mass concentration of dust — Part 1: Manual gravimetric method*

---

1) To be published.



