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Stationary source emissions — Manual determination of mass concentration of particulate matter

Émissions de sources fixes — Détermination manuelle de la concentration en masse de poussières

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 9096:2003), of which it constitutes a minor revision. It also incorporates the Technical Corrigendum ISO 9096:2003/Cor.1:2006. The changes compared to the previous edition are as follows:

- Table 3: in the row entitled “Isokinetic criteria (average measurement uncertainty)” the value “±10 %” has been replaced by “$^{+15}_{-\ 5}$ %” (according to ISO 9096:2003/Cor.1:2006).
- Formula (11): the percent symbol has been added twice.
- Formula (13): the percent symbol has been added twice.
- Figure A.2: < 0,2 has been corrected to > 0,2.
- Formula (B.6): the parentheses have been removed.
- Formula (B.7): the formula has been corrected.

Introduction

Close liaison and cooperation between ISO/TC 146/SC 1 and CEN/TC 264 has resulted in the preparation of this document, ISO 12141 and EN 13824-1. This document is similar to EN 13284-1 with additional emphasis given on the use of high-volume sampling techniques. A representative, integrated sample is extracted from the flue gas and the particulate matter entrained in the gas sample is separated by a filter. The pre-weighed filter is subsequently dried and weighed. A relative increase in the mass is attributed to the collection of particulate matter on the filter.

To meet the specifications of this document, the particulate sample is weighed to a specified level of accuracy. This level of accuracy is achieved by:

a) exercising extreme care in weighing, in accordance with the procedures of this document;

b) extending the sampling time at conventional sampling rates;

c) sampling at higher rates for conventional sampling times (high-volume sampling);

d) recovering all dust upstream of the filter.

Stationary source emissions — Manual determination of mass concentration of particulate matter

1 Scope

This document describes a reference method for the measurement of particulate matter (dust) concentration in waste gases of concentrations from 20 mg/m^3 to 1 000 mg/m^3 under standard conditions.

This document is applicable to the calibration of automated monitoring systems (AMS). If the emission gas contains unstable, reactive or semi-volatile substances, the measurement will depend on the filtration temperature. In-stack methods can be more applicable than out-stack methods for the calibration of automated monitoring systems.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 5725 (all parts), *Accuracy (trueness and precision) of measurement methods and results*

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

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
particulate matter
dust
particles, of any shape, structure or density, dispersed in the gas phase under the sampling conditions

Note 1 to entry: In the method described, all the compounds that may be collected by filtration under specified conditions after representative 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 can extend to condensibles or reaction products collected under specified conditions (e.g. temperatures lower than the flue gas temperature).

Note 2 to entry: This method restricts the definition of particulate matter to that material collected in the sampling system on and before a filter, under specified temperature conditions. Procedures for the measurement of secondary particulate matter (condensible materials) formed and collected after the filter are not within the scope of this document.

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

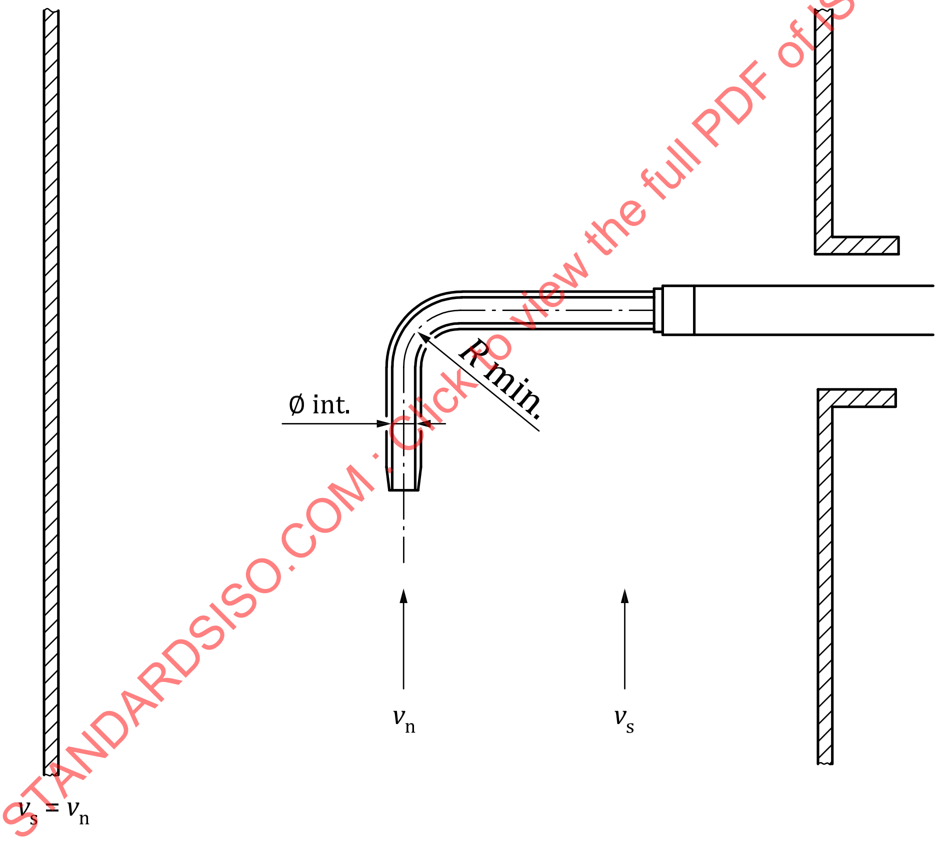
filtration outside of the duct with the filter in its heated filter holder placed downstream of the sampling nozzle and the suction tube (sampling probe)

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

Note 1 to entry: See Figure 1 and Annex D.

Note 2 to entry: The velocity ratio, v_n/v_s, expressed as a percentage characterizes the deviation from isokinetic sampling.



Key

v_s	stack gas velocity
v_n	velocity in the nozzle

Figure 1 — Isokinetic sampling

3.6

hydraulic diameter

d_h

characteristic dimension of a duct cross-section

$$d_\mathrm{h} = \frac{4 \times A_\mathrm{s}}{l_s} \tag{1}$$

where

A_s is the cross-sectional area of the sampling plane;

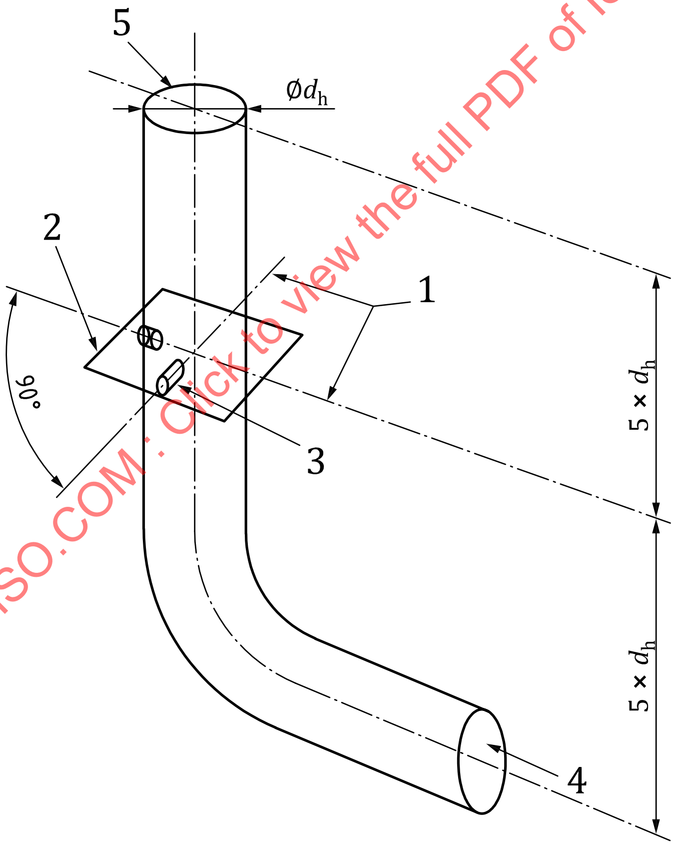
l_s is the length of the perimeter of the sampling plane.

3.7

sampling plane

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

Note 1 to entry: 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

3.8

sampling line

line in the *sampling plane* (3.7) along which *sampling points* (3.9) are located, bounded by the inner duct wall

Note 1 to entry: See Figure 2.

3.9

sampling point

specific position on a *sampling line* (3.8) at which a sample is extracted

3.10

standard conditions

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

Note 1 to entry: For the purposes of this document, 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 1 to entry: 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 procedures

quality control procedures utilized for detecting/correcting apparent mass variations due to climatic or environmental changes between pre- and post-sampling weighing series

Note 1 to entry: In these procedures, control parts are used (see 7.2) which are identical to those to be weighed for dust measurement and are pre-treated under the same conditions of temperature and humidity. The control parts are kept free from dust contamination.

3.13

measurement series

successive measurements carried out in the same *sampling plane* (3.7), and under the same process conditions

3.14

limit value

dust concentration that is permitted by authorities for the plant process (i.e. average limit value)

Note 1 to entry: For purposes other than regulatory uses, the measurement value is compared to a stated reference value.

4 Principle

4.1 General

A sample stream of the gas is extracted from the main gas stream at specified sampling points for a measured period of time, with an isokinetic, controlled flowrate. The volume of gas collected is measured, and a pre-weighed filter, which is then dried and reweighed, separates the particulate matter (dust) entrained in the gas sample. Deposits upstream of the filter in the sampling equipment are also recovered and weighed. The increase in mass of the filter and the mass deposited upstream of the filter is attributed to particulate matter collected from the sampled gas. The ratio of the mass of the particulate matter collected to the volume of gas collected allows the flue gas particulate concentration to be calculated.

Valid measurements can be achieved only when:

a) an adequate quantity of dust is collected during the sampling, which is at least 5 times the corresponding overall blank value;

b) the gas stream in the duct at the sampling location has a sufficiently steady and identified velocity, temperature and pressure, and a sufficiently homogeneous composition;

c) the flow of gas is parallel to the axis of the nozzle;

d) sampling is carried out without disturbance of the gas stream, using a sharp-edged nozzle facing into the stream;

e) isokinetic sampling conditions are maintained throughout the test;

f) samples are taken at a preselected number of stated positions in the sampling plane to obtain a representative sample for a non-uniform distribution of particulate matter in the duct or stack;

g) the sampling train is designed and operated to avoid condensation and to be leak-free;

h) calibration criteria are met;

i) sampling blank and leak-check criteria are met;

j) dust deposits upstream of the filter are recovered and/or taken into account;

k) the sampling and weighing procedures are adapted to the expected dust quantities as specified in this document.

4.2 Interferences

a) Positive interference

Gaseous species present in stack gases that are capable of reacting to form particulate matter within the sample train can result in positive interference. Examples include the potential reaction of sulfur dioxide (SO_2) to an insoluble sulfate compound in the high-humidity portion of the system, such as with limestone in flue gas following a wet flue-gas desulfurization system (FGDS) to form calcium sulfate ($CaSO_4$), or the reaction with ammonia gas (NH_3) to form ammonium sulfate (NH_4SO_4) [see 7.1 a)].

b) Negative interference

1) Certain acid gaseous species can erode the filter material, resulting in negative interference. For example, the reaction of hydrogen fluoride (HF) with glass components in the sample train (see 6.2.5).

2) Volatile matter existing in solid or liquid form in the stack gas may vaporize after collection on the sample train filtration material, due to continued exposure to the hot sample stream during the sampling period. This would result in a negative interference (see 8.1).

5 Sampling plane and sampling points

5.1 General

Representative sampling is possible when a suitable location is available, having a sufficiently homogeneous gas velocity at the sampling plane.

Sampling shall be carried out at a sufficient number of sampling points, usually located on several sampling lines. Convenient access ports and a working platform shall be available for the testing.

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. The sampling plane shall be as far downstream and upstream as possible from any obstruction that can cause a disturbance and produce a change in the direction of flow (disturbances caused by, for example, bends, fans or pollution abatement equipment).

5.3 Requirements for sampling points

Preliminary measurements at all the sampling points defined in 5.4 and Annex B shall prove that the gas stream in the sampling plane meets the following requirements:

a) the angle of gas flow is less than 15° with regard to the duct axis (a recommended method for estimation is indicated in ISO 10780:1994, Annex C);

b) no local negative flow is present;

c) the minimum velocity is higher than the detection limit of the method used for the flowrate measurement (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 uncertainty is higher than that specified by this document and the sampling location is not in compliance with this document (see 7.4.6).

The above requirements are generally met in sections of duct with at least five hydraulic diameters of straight duct upstream of the sampling plane and at least two hydraulic diameters downstream. (If the sampling plane is to be located near the stack exit, it should be no less than five hydraulic diameters from the exit.) Therefore, it is strongly recommended that sampling locations be selected accordingly.

5.4 Minimum number and location of sampling points

The dimensions of the sampling plane dictate the minimum number of sampling points. In general, 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 B).

Sampling points shall not be located within 3 % of the sampling line length (if $d > 1,5$ m) or 5 cm (if $d < 1,5$ m) from the inner duct wall. Choose the inner edge of the area when calculations result in sampling point positions within this area. 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 is sometimes possible to improve representative sampling by increasing the number of sampling points above those specified in Tables 1 and 2. See also 7.3.2 for sampling-point premeasurement procedures.

Table 1 — Minimum number of sampling points for circular ducts

Range of duct diameters m	Minimum number of sampling lines (diameters)	Minimum number of sampling points per line		Minimum number of sampling points per plane	
		including centre point	excluding centre point	including centre point	excluding centre point
< 0,35	—	1[a]	—	1[a]	—
0,35 to 0,70	2	3	2	5	4
0,70 to 1,00	2	5	4	9	8
1,00 to 2,00	2	7	6	13	12
> 2,00	2	9	8	17	16

[a] Using only one sampling point can give rise to errors greater than those specified in this document.

Table 2 — Minimum number of sampling points for rectangular ducts

Range of sampling plane areas m^2	Minimum number of side divisions[a]	Minimum number of sampling points per plane
< 0,09	—	1[b]
0,09 to 0,38	2	4
0,38 to 1,50	3	9
> 1,50	4	16

[a] Other side divisions can be necessary, for example if the longest duct side length is more than twice the length of the shortest side.

[b] Using only one sampling point can give rise to errors greater than those specified in this document.

5.5 Access ports

Ports shall be provided for access to the sampling points selected in accordance with Annex B.

The port dimensions shall provide space for the insertion and removal of the sampling equipment and associated devices, and allow for sealing once the sampling equipment is in place. A minimum diameter of 125 mm or a surface area of 100 mm × 250 mm are recommended, except for small ducts (less than 0,7 m diameter) for which the port size needs to be smaller (see Annex F for examples).

5.6 Sampling time

Assuming a volumetric flowrate characteristic of the sampling train to be used, a sampling time can be calculated that will lead to the collection of a desired or required mass of particulate matter if the approximate particulate concentration is known previously.

If the expected dust concentration, c_{exp} has been previously determined or assumed, and the mass of particulate matter, m, to be collected is required or set, then the necessary volume of the flue gas to be sampled is:

$$V_{\mathrm{n}} = \frac{m}{c_{\mathrm{exp}}} \tag{2}$$

However, the volume of the sample, V_{n} (litres), will be equal to the total sampling time, t (min), multiplied by the nozzle volumetric flowrate under actual conditions, Q_{a} (l/min), i.e. $V_{\mathrm{n}} = tQ_{\mathrm{a}}$.

The total sampling time in the sampling plane is thus estimated to be:

$$t = \frac{V_{\mathrm{n}}}{Q_{\mathrm{a}}} \quad \text{or} \quad t = \frac{m}{c_{\mathrm{exp}} \cdot Q_{\mathrm{a}}} \tag{3}$$

6 Apparatus and materials

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

Velocity measurements shall be carried out using standard Pitot tubes or other measurement devices, e.g. S-Type Pitot tubes, provided they are calibrated against standardized Pitot tubes in accordance with ISO 10780.

The temperature and the pressure in the duct shall be measured in order to calculate the actual density of the gas within ±0,05 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 or CO_2 concentration, humidity (moisture) and/or oxygen/CO_2 measurements shall be carried out in the vicinity of the sampling plane.

Specifications for this apparatus are given in Table 3.

6.2 Sampling apparatus

6.2.1 Sampling train.

The sampling train consists principally of

a) a suction tube (sampling probe) with entry nozzle,

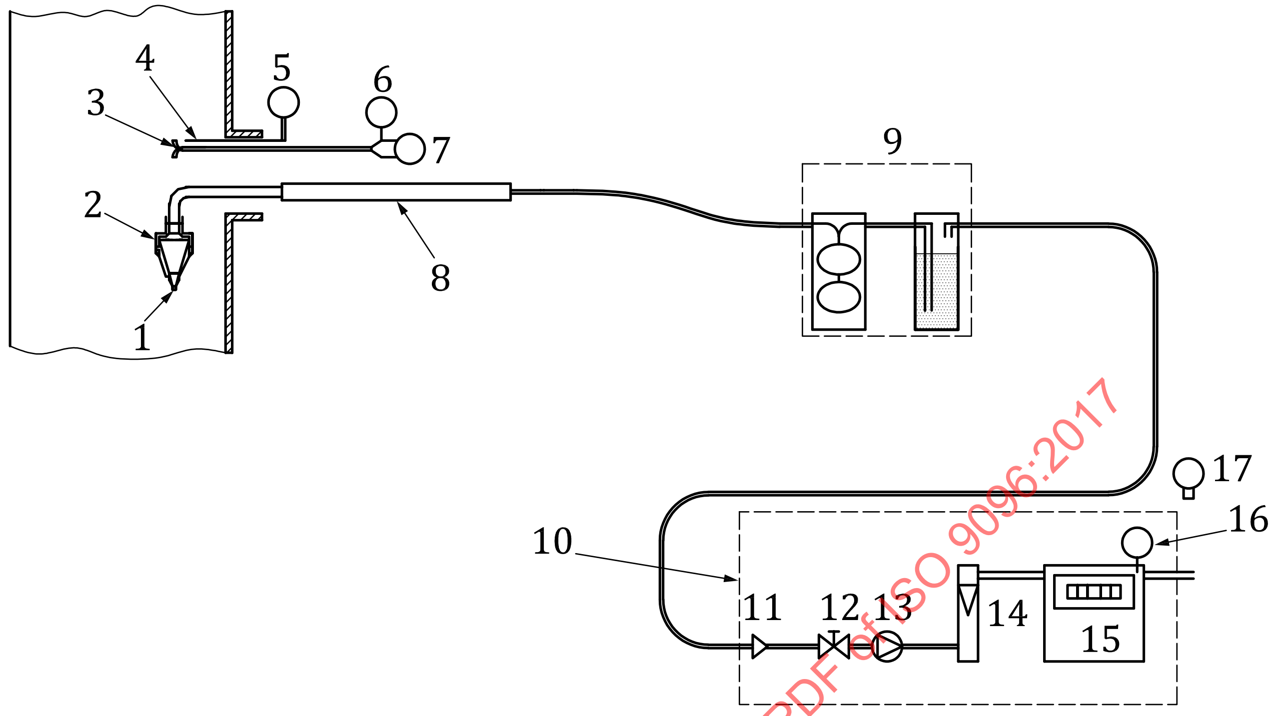
b) a filter housing, including a filter support and a filter, either located in the duct (in-stack filtration), or placed outside the duct (out-stack filtration), leading to slightly different kinds of sampling trains. In the presence of water droplets, out-stack filtration shall be used,

c) a suction unit, with a gas metering system.

6.2.2 Filtration device.

a) **In-stack filtration devices** (Figure 3): the part of the tubing between the nozzle and the filter shall be very short, to minimize dust deposits upstream of the filter. The tubing after the filter (suction tube) shall be of sufficient length to traverse the duct at the required sampling points. Since the filtration temperature is generally identical to that of the gas in the duct, filter clogging can occur if the gas contains water droplets.

   To allow traversing of the duct, a leak-free, rigid tube (support tube) of sufficient length is used downstream of the filter housing for mechanical support of the nozzle and filter housing;

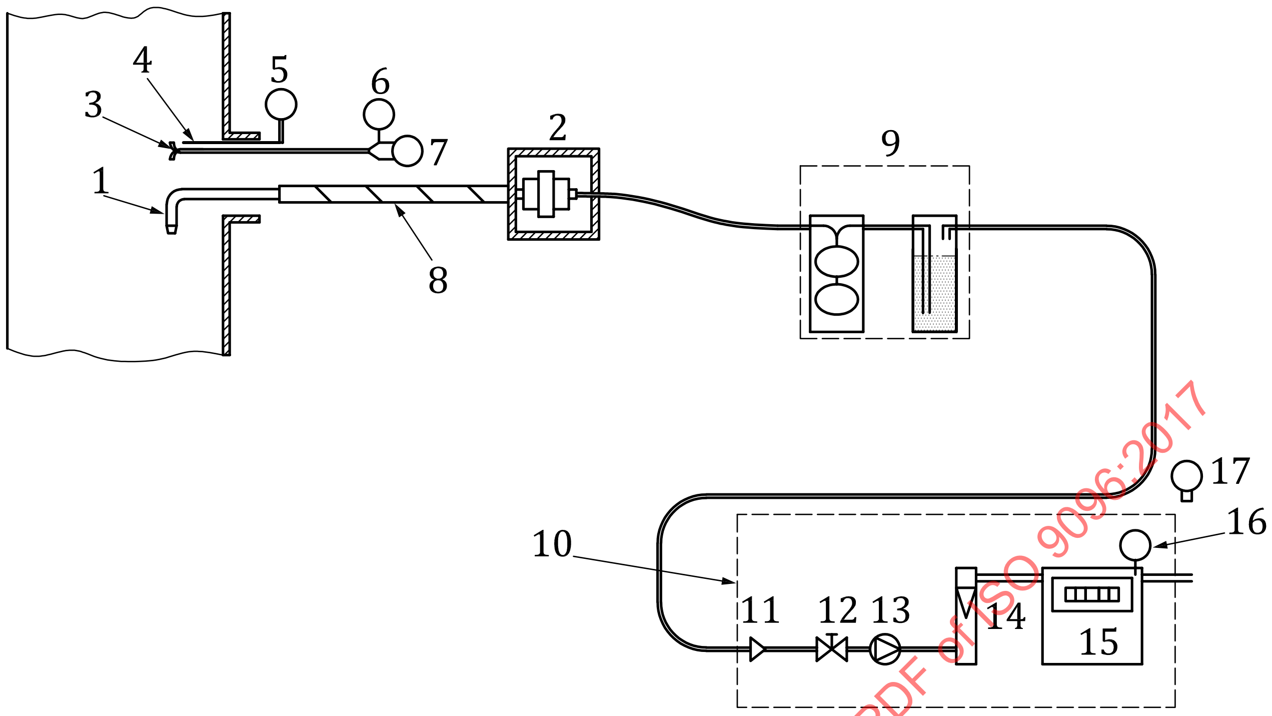


Key

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 volume meter
16	temperature measurement
17	barometer

Figure 3 — Example of sampling on a dry basis with an in-stack filter

b) **Out-stack filtration devices** (Figure 4): the part of the tubing between the nozzle and the filter (suction tube) has to be of sufficient length to traverse the duct at the required sample points. The suction tube and the filter housing is temperature-controlled, to provide vaporization of possible water droplets and avoid filtration difficulties related to acid gases having high dew points.



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 volume meter
16	temperature measurement
17	barometer

Figure 4 — Example of sampling on a dry basis with an out-stack filter

Water droplets are present in certain processes, e.g. after a wet-scrubbing abatement system. Knowledge of the process for low temperatures, below dew-point, is a requirement of this document. If there is a possibility that water droplets are present, then out-stack filtration should be used.

The sampling parts of the system shall be made of corrosion-proof and, if necessary, heat-proof material, for example stainless steel, titanium, quartz or glass. However, if further analysis of the collected dust is planned (for example for heavy metals), stainless steel should be avoided for the parts in contact with the sample gas.

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 tapered rather than stepped.

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

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

6.2.3 Set of entry nozzles of different diameters, sharp-edged, streamlined, and free enough of obstacles in order not to disturb the main gas flow.

The nozzle is connected either to the suction tube (sampling probe) or the filter housing. Annex A details three proven designs. Other designs are allowed, provided it can be validated that they give equivalent results.

In order to prevent disturbances of the gas flow near the nozzle tip, the following requirements also apply:

a) the nozzle shall have a constant internal diameter along a length equal to at least one internal diameter or at least 10 mm from the nozzle tip, whichever is greater. See 7.3.3 for calculation of the diameter;

b) any variation in bore diameter shall be tapered with a conical angle less than 30°;

c) bends shall be located at least 30 mm from the nozzle tip;

d) any variation in external diameter of the sampling equipment parts located 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 time the size of the obstacle, whichever is greater.

Since it is necessary, for mechanical reasons, for the nozzle bevel to have sufficient thickness, this leads to an uncertainty on the effective sampling area. This uncertainty should be less than 10 % in order to fulfil isokinetic sampling criteria. For this reason, it is recommended that nozzles of inside diameter exceeding 8 mm be used, and diameters less than 4 mm be avoided.

6.2.4 Suction tube (sample probe) for out-stack filtration systems.

The suction tube shall have a smooth and well-polished internal surface, and shall be designed so that it can be cleaned easily using a brush or other mechanical means, a necessary condition before sampling (see 7.3.1).

The walls of the suction tube shall be heated and temperature-controlled (7.3.4) in order to minimize condensation or the formation of artifacts.

6.2.5 Filter housing.

When the filter housing is placed out-stack, it shall be heated and temperature-controlled (7.3.4) to avoid condensation.

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

To reduce the filter pressure drop and to improve the distribution of dust on the filter, a coarse-grain filter support is recommended.

6.2.6 Filters, of efficiency better than 99,0 % on a test aerosol with a mean particle diameter of 0,3 µm, at the maximum flowrate anticipated.

This efficiency shall be certified by the filter supplier.

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 (see 7.3.3).

The choice of the filter shall also take into account the following considerations.

a) Glass fibre filters can react with acidic compounds such as SO_3, leading to an increase in the filter mass. Their use is not recommended where this can occur;

b) Despite their weak mechanical strength, quartz fibre filters have been shown to be efficient in most cases;

c) Polytetrafluoroethylene (PTFE) filters have also proven to be efficient. However, the temperature of the gas passing through the filter shall not exceed the temperature specified by the supplier;

d) The size of filter shall be chosen in relation to the maximum allowable mass of particulate collected on the filter. This is to prevent loss of particulate matter because of overburdening the filter paper. The maximum amount that can be collected for a filter shall be certified by the filter manufacturer.

e) The pressure drop across the filter, and the pressure increase due to the collection of particulate matter while sampling. This depends on the kind of filter (e.g. a pressure drop of 3 kPa to 10 kPa at a filtration velocity in the range of 0,5 m/s is expected);

f) When using filters with organic binders, care shall be taken to avoid mass losses due to the evaporation of the binder material when heating;

g) The overall blank value of the measurement depends, to some extent, on the choice of the filter (mechanical properties, affinity for humidity, etc.);

h) If it is planned to determine the composition of the dust collected, a blank of the filter material shall be tested to determine the presence and mass levels of any relevant materials to be analysed;

i) When weighing some kinds of filter material (e.g. PTFE, etc.), take care to avoid errors due to electrostatic charges.

6.2.7 Combined particulate matter/gas sampling trains (optional design), to determine flue gas emissions.

When gaseous compounds are trapped downstream from the filter, any volume losses, temperature changes or pressure changes shall be accounted for when calculating the isokinetic sampling rate and when calculating the collected gas sample volume.

6.2.8 Suction unit and gas-metering devices, gas-tight, corrosion-proof and capable of maintaining a vacuum that will extract a flue gas sample at the calculated isokinetic sampling rate appropriate to the nozzle size and flue gas conditions.

The system shall incorporate a means of controlling the sample flowrate, for example, a pump by-pass valve or a regulating valve. A shut-off device shall also be incorporated into the system for stopping the gas flow through the sampling train.

Depending on the sampling measurement mode (on a wet or dry basis), three major kinds of arrangements may be used. Other types of arrangement are allowed, provided it is proven that they give the same accuracy as the systems described below.

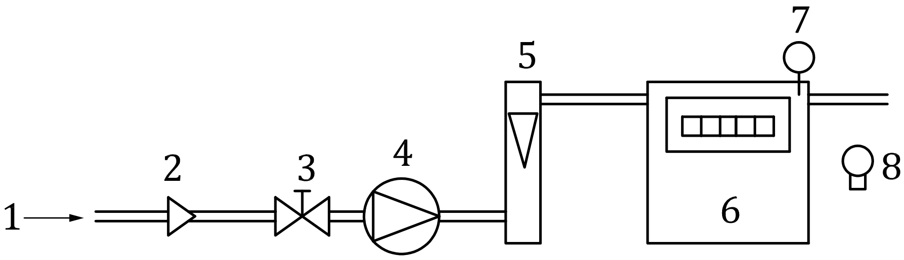
a) Sampling on a dry basis with an in-stack filter (see Figure 5), including:

   1) a **condenser** and/or **gas drying tower** providing a residual humidity less than 10 g/m^3 at the maximum flowrate;

   2) a **gas-tight pump** or **compressed air ejector**, acting as a suction device;

   3) a **flowmeter**, used to facilitate the flowrate adjustment, calibrated against the dry gas volume meter or measuring orifice plate;

4) a **dry gas volume meter** or **orifice plate**, accurate within 2 % at the anticipated flowrate, with associated absolute pressure and absolute temperature measurements accurate to within 1 %.



Key

1	dry gas sample flow
2	non-return valve
3	shut-off valve
4	pump
5	variable-area flowmeter
6	dry gas meter
7	temperature measurement
8	barometer

Figure 5 — Sampling control using flowmeter and dry gas meter

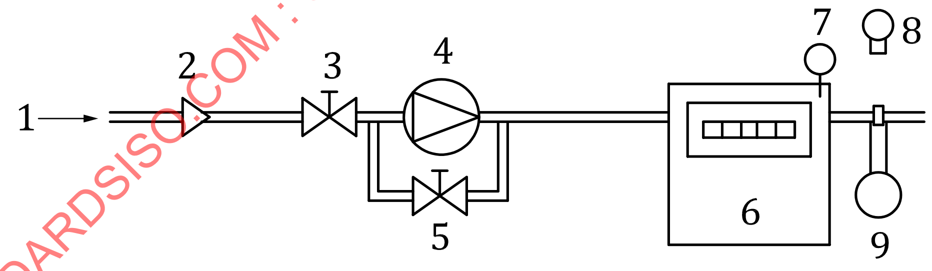
b) Sampling on a dry basis using an out-stack filter and condenser system (see Figure 6), including:

1) a **condenser** and/or **gas drying tower** providing a residual humidity of less than 10 g/m^3 at the maximum flowrate;

2) a **gas-tight** pump or **compressed air ejector**, acting as a suction device;

3) a **dry gas volume meter** accurate within 2 % at the anticipated flowrate, with associated absolute pressure and absolute temperature measurements accurate to within 1 %;

4) a **flowmeter** or **orifice plate**, used to facilitate the flowrate adjustment, calibrated against the dry gas volume meter or measuring orifice plate.



Key

1	dry gas sample flow
2	non-return valve
3	shut-off valve
4	pump
5	by-pass control valve
6	dry gas meter
7	temperature measurement
8	barometer
9	variable area flowmeter

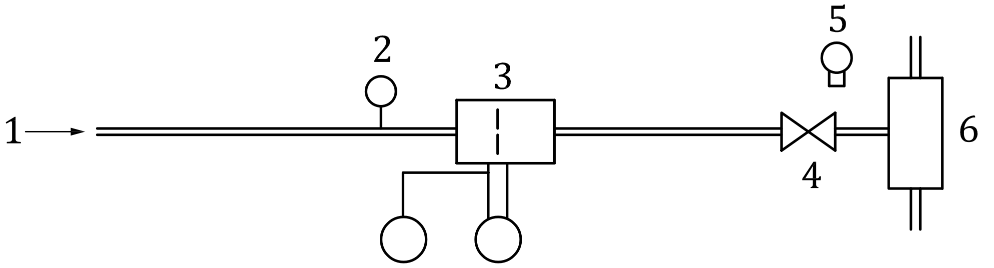
Figure 6 — Sampling control using dry gas meter and secondary orifice flowmeter

c) Sampling on a wet basis with an out-stack filter (see Figure 7), including:

1) a **insulated** or **heated flexible tubing**, used to prevent upstream condensation of humidity;

2) a **gas-tight pump** or **compressed air ejector**, acting as a suction device;

3) a **condensation-free orifice plate** or equivalent device serving as flowmeter. The temperature and pressure (absolute and differential) measurements at the orifice plate (flowmeter) shall be accurate to within 1 % and the orifice plate shall be calibrated within 2 % of the anticipated flowrate.



Key

1	wet gas sample flow
2	temperature measurement
3	heated orifice
4	shut-off valve
5	barometer
6	air injector

Figure 7 — Sampling control on a wet basis, mandatory out-stack filtration (see Figure 4)

6.3 Material for particulate matter recovery

6.3.1 Purified water, de-ionized and filtered.

6.3.2 Acetone, high grade with a residue of less than 10 mg/l.

6.3.3 Clean containers of appropriate size (e.g. 250 ml) for storage and transportation of the rinsing solution.

6.3.4 Plugs to close the suction tube. Plugs used shall not be a source of sample contamination.

6.4 Apparatus for conditioning and weighing

6.4.1 Weighing containers, for the drying procedure for the rinsing solutions, of mass appropriate for the balance used. Glass and ceramic have proven to be suitable materials for these containers; polymeric materials are not recommended.

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

6.4.3 Drying oven, classical laboratory type, thermally controlled within ±5 °C.

6.4.4 Balance, with resolution from 0,01 mg to 0,1 mg, with a range compatible with the mass of the parts to be weighed. Depending on the balance room location, specific care shall be taken to avoid reading instabilities related to vibrations, air draughts, temperature and humidity variations.

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

6.4.6 Barometer.

6.4.7 Depending on the evaporation procedure, an **extraction hood** and **heating plate** for the evaporation of the rinsing solutions.

7 Sampling and weighing procedures

7.1 General aspects

Before carrying out any measurements, the purpose of the sampling and the sampling procedures shall be discussed 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 is maintained during sampling.

Dates, starting times, duration of survey and sampling periods, as well as plant operating conditions during these periods shall be agreed upon with the plant management.

Preliminary calculations shall be made in order to determine the appropriate nozzle diameter and/or sampling time. Longer sampling times or sampling with the use of a larger nozzle and higher sample flowrates can be necessary to obtain sample filter masses sufficiently greater than blank filter masses.

Taking into account the objective of the measurements and the flue gas characteristics, the user shall

a) choose between an in-stack or an out-stack filtration device;

   If the flue gas is saturated with water or contains appreciable amounts of SO_3, out-stack filtration devices are recommended.

b) choose a relevant temperature for conditioning and drying the filter before and after sampling;

   When using an out-stack filtration device, set the out-stack filter temperature as specified by the regulatory agency or as determined for technical reasons.

c) take an overall blank sample after each measurement series and at least after each day of sampling, following the sampling procedure described in 7.3.5, either without starting the suction device or for a sample duration less than 1 min.

   This leads to estimation of the dispersion of results related to the whole procedure as carried out by the operators for a near-zero dust concentration, due to contamination of filters and of rinsing solutions during handling on-site, transport, storage, handling in the laboratory, and weighing procedures, etc.

While sampling, the velocity at the sampling plane shall be simultaneously monitored, to check for possible flowrate variations in the duct.

The temperature, pressure, moisture content, and molecular mass of the flue gas shall be determined at the sampling location in order to calculate isokinetic sampling conditions and correct emissions data from actual to standard conditions. These parameters shall be monitored at each sampling point if they vary spatially or temporally during the period of the test to the degree that the ±10 % isokinetic requirement (7.4.3) is exceeded.

When expressing particulate concentration on a dry basis, the flue gas moisture content shall be determined. If the particulate concentration is to be corrected by regulation to a specified oxygen or carbon dioxide concentration, oxygen or carbon dioxide shall also be measured.

7.2 Weighing procedure

7.2.1 Parts to be weighed

a) Depending on the internal arrangements, the parts of the filter system to be weighed before and after sampling may be:

  1) only the filter;

  2) the filter and filter support;

  3) the filter, the filter support, and the inlet part of the filter housing including the nozzle (depending upon the system design).

  In the first and second cases, dust deposits from the nozzle tip to the filter shall be recovered and weighed.

  In the third case, dust deposits upstream of the filter are accounted for in the weighing, but it is necessary to use a balance that will weigh the larger masses of these parts to within specified levels of accuracy. The outside surfaces shall be cleaned prior to weighing, using good laboratory practice.

b) Depending on the kind of sampling device used, the parts to be weighed can be the filter (with or without its support) or include all parts upstream from the filter. Options include:

  1) the filter and particulate matter collected upstream of the filter;

  2) the filter and its housing and particulate matter collected upstream of the housing;

  3) the nozzle, the filter and its housing, and any components connecting the nozzle to the housing.

  For the latter two designs above, all the relevant parts shall be pre- and post-treated in accordance with the procedure described in 8.3, and weighed together, without disassembling them.

c) Depending upon the system used, the solutions obtained from rinsing components can be evaporated and weighed in the same container or transferred to a smaller container for weighing following good laboratory practice.

7.2.2 Pre-sampling treatment of weighed parts

Weighed parts shall be dried in a drying oven for at least 1 h, at 160 °C (see also Clause 8 for specific cases).

After drying, the filters and/or the weighing containers shall be placed in a desiccator located in the weighing room for at least 8 h to ensure that the filter is conditioned in the same way throughout its use (i.e. preparation and final weighing). For larger parts and weighing containers, up to 12 h conditioning can be necessary.

7.2.3 Weighing

Weigh the filter on a suitable electronic balance to at least ±0,1 mg.

Since the sample masses are determined by calculating the difference between data, often obtained at one- or two-week intervals, special care is required to avoid weighing errors related to balance drift, to insufficient temperature equilibrium of parts to be weighed, and climatic changes (see examples in Annex C). Therefore, before performing any weighing, the analyst shall validate his or her weighing procedure.

Before each weighing series:

a) calibrate the balance against standard weights;

b) carried out additional checks shall be by weighing control parts, equivalent to the other parts and pre-treated in the same conditions, but 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 can influence the apparent mass. This can be detected using the reference mass of the control parts. In these conditions, weighing corrections shall be applied. Determine the weighing corrections based on the apparent mass modification of three equivalent control parts of each type (filter including support, container, etc.) (see Annex C).

Attention shall be given also to weighing artifacts related to:

— electrostatic charges, which it can be necessary to discharge or neutralize;

— hygroscopic and volatility characteristics of the filter material and/or dusts, which can lead to an increase or decrease in mass. For this reason, carry out weighing rapidly, within 1 min after removal from the desiccator. Take two additional readings at 5 s intervals after the initial reading. If there is a significant increase or decrease in the readings as a function of time, due to the nature of the material, special procedures such as extrapolating the reading to initial conditions can be necessary;

— small differences in temperature between the part to be weighed and the environment can disturb the balance.

7.2.4 Post-sampling treatment of weighed parts

Dry weighed parts in a drying oven for at least 1 h at 160 °C (see also 8 for specific cases).

After drying, equilibrate the parts to ambient temperature as described in 7.2.2.

When volatile or reactive compounds are suspected, carry out drying at the sampling temperature, using a flow of dry nitrogen.

7.2.5 Post-sampling treatment of the rinsing solutions

All the rinsing solutions (water and acetone) from all parts upstream of the filter as described in 7.3.5 are taken to the laboratory for further treatment. Care shall be taken that no contamination occurs.

Transfer the solutions quantitatively to the dried and pre-weighed containers. During the evaporation, do not boil the solvent mixture. As the volume of the solution is reduced through the evaporation process, smaller vessels may be used.

NOTE Two methods have been tested for evaporation: 1) evaporation in an oven at 120 °C at ambient pressure. An efficient ventilation system with clean air or nitrogen is necessary to dilute acetone vapour to a safe level; 2) evaporation in a closed system (desiccator). The initial temperature is set to 90 °C and the pressure is reduced to 40 kPa (absolute). Periodically, the temperature as well as the pressure is increased. For the last period, they are maintained at 140 °C and 20 kPa (absolute).

After the evaporation, place the weighing containers in the drying oven for 1 h at 160 °C (see Clause 8 for specific reasons). Transfer the weighing containers to the desiccator. After thermal equilibration in the weighing room as in 7.2.1, weigh the weighing containers, including the deposit remaining after evaporation.

Due to the relatively large mass and the volume of the weighing containers compared to the deposits under investigation, changes in barometric pressure can influence the weighing. Therefore, weigh at least three empty weighing containers of equal size with each series, so that any changes in mass can be used for correction.

Obtain at least one blank value for each solvent, using the same volume as used in the rinsing.

7.3 Sampling

7.3.1 Preparation

The equipment shall be cleaned (brushed and rinsed), calibrated and checked before moving to the test site. Care shall be taken not to reuse any part of a sampling train that has previously been used for high dust concentration sampling without dismantling and thorough cleaning.

Depending on the measurement program, the filter and associated parts to be weighed shall be prepared for each sample run. This includes parts for the overall blank tests and additional parts to accommodate process and equipment malfunctions.

Perform the weighing procedures according to 7.2.3.

All the weighed parts, including the suction tube and the other parts or equipment that come into contact with the sample (and will be rinsed later) shall be protected from contamination during transportation and storage.

7.3.2 Pre-measurements

Verify the dimensions of the duct to be sampled using a measuring rod, surveyor’s transit or other means. Select the number and location of sampling points in accordance with 5.4 and Annex B. Mark on the Pitot tube and the sampling tube the distance from the sampling points to the entry point of the access port.

Measure the temperatures and velocities at the selected test points in the duct, checking also for possible deviations of gas flow with regard to the duct axis, and verify that the requirements of 5.3 are fulfilled. Otherwise, see 7.4.1.

Measure the molecular mass and moisture content of the flue gas.

In order to check for possible temporal flow variation in the duct while sampling, install a separate Pitot tube at a relevant fixed point of the sampling section to monitor the flue gas velocity over the sampling period. Monitoring the temperature and/or CO_2/O_2 concentration in the duct (or other relevant parameters) can also provide an indication of the steadiness of the stationary source operations.

Taking into account the preliminary calculation for the sampling time and the measured velocities, select a suitable nozzle in accordance with the requirements of 6.2.2 and 7.3.3.

7.3.3 Calculating the nozzle diameter

7.3.3.1 Nozzle description

In order to allow isokinetic sampling of gases flowing at a wide range of velocities, the sampling equipment shall include a set of nozzles of different diameters.

The nozzle diameter 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).

7.3.3.2 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:

$$A_\mathrm{n} = \frac{\pi \left(D_\mathrm{n} \right)^2}{4} = \frac{q_V}{v_\mathrm{n}} \tag{4}$$

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

7.3.3.3 Calculating the nozzle diameter for a fixed sampling time

In some circumstances, the sampling time is specified. Under such requirements, it can 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 sampling train design can be necessary to accommodate the increased flow requirements.

The volumetric flowrate through the nozzle, q_V, is $q_V = A_n v_n$, where A_n is the area of the nozzle and 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 $v_n = v_s$.

Therefore, from Clause 6 and under isokinetic sampling conditions,

$$q_V = \frac{V_\mathrm{n}}{t} = A_\mathrm{n} v_\mathrm{n} = A_\mathrm{n} v_\mathrm{s} \tag{5}$$

and

$$A_\mathrm{n} = \frac{V_\mathrm{n}}{t \cdot v_\mathrm{s}} \tag{6}$$

or, from Formula (6),

$$A_\mathrm{n} = \frac{m}{c_\mathrm{exp} \cdot t \cdot v_\mathrm{s}} \tag{7}$$

where

A_n is the area of the nozzle;

c_{exp} is the expected concentration of dust;

m is the mass of particulate matter;

t is the time;

q_V is the volume flowrate through the nozzle;

v_s is the velocity of the gas through the stack;

v_n is the velocity of the gas through the nozzle;

V_n is the volume of gas through the nozzle.

7.3.4 Overall blank

Take an overall blank sample after each measurement series or at least once a day, following the sampling procedure described in 7.3.5, without starting the suction device, and keeping the sampling nozzle in the duct for 15 min at 180° from the direction of flow. This leads to an estimation 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.

The overall blank value is reported in milligrams per cubic metre, and is calculated using the average sample run time achieved during the measurement series.

7.3.5 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. The leak flowrate measured, for example, by pressure variation after evacuation of the train at the maximal under pressure reached during sampling, shall be below 2 % of the normal flowrate. During sampling, a leak check can be monitored by continuously measuring the concentration of a relevant gas component (CO_2, O_2, 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 in order 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 in order to obtain isokinetic sampling within

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 Continuously monitor, or record at least every 5 min, the dynamic pressure measured by the Pitot tube or other suitable measurement system either installed at the fixed point or mounted on the sampling equipment (see 8.3);

   NOTE Good laboratory practice when using 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 following sampling point, and immediately adjust the flowrate for 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 the sampling line, close the shut-off valve and suction device, remove the sampling train from the duct and reposition it on the next sampling line;

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

   The filter load and the maximum gas velocity should not exceed the filter manufacturer's recommendation.

j) On completion of sampling run at all points:

   1) remove the sampling train after closing the shut-off valve and suction device;

   2) leak-check the equipment as under 7.4.2 if leakage has not been monitored during sampling;

   3) dismantle the sampling equipment and check visually 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 7).

7.3.6 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.

Take special care to avoid contamination of the sample if the rinsing procedure is performed on-site. Carry out rinsing according to the following procedure.

a) Rinse the internal surfaces of the nozzle, elbow and the front part of the filter housing carefully with water into a storage bottle. Allow no external dust to fall into the bottle. Rinse these same surfaces with acetone and collect the acetone into the same bottle.

b) To rinse the suction tube (sample probe), 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. Rotate the tube along its long axis and tilt the tube several times. Transfer the water to the transport storage container. Repeat the procedure with a second rinsing of water, followed by a rinsing with acetone.

Do not mechanically clean surfaces to recover deposits upstream of the filter after sampling. However, it is necessary to mechanically clean and rinse the equipment before each measuring series.

The overall blank shall include a blank rinse of a pre-cleaned sampling system. Obtain the blank rinse following the procedures given above, and evaporate and weigh following the same procedures as for the sample rinse.

7.4 Validation of results

7.4.1 Parameters depending on the stationary source

If testing was conducted at an unsuitable location, or was carried out under fluctuating plant operating conditions, the validity of the sample may be questioned and the measurement results uncertain. In such cases, the test report shall clearly indicate that the test was not conducted according to the specifications of this document.

Details of the flow characteristics at the sampling location and/or on the variations of the flowrate in the duct while sampling shall be included in the test report along with any explanations.

7.4.2 Leak check

Significant errors can be caused by leaks in the sampling train, especially in parts that are under vacuum.

Therefore, before and after each test, the sample train shall be checked for leaks by sealing the nozzle and starting the suction device. The flowrate shall be at most 2 % of the normal flowrate at the maximum vacuum reached during sampling. Otherwise, the measurement is not valid.

7.4.3 Isokinetic flowrate

If the actual flowrate through the nozzle differs by more than ±10 % from the theoretical isokinetic flowrate over the duration of the sampling, the flowrate measurement shall be repeated.

If this criterion is still not met due to variation of the flowrate in the duct, see 7.4.1.

7.4.4 Deposits of dust on non-weighed parts upstream of the filter

The mass of deposits on non-weighed parts upstream of the filter shall be added to the mass of deposits determined from the filter and weighed parts.

This requirement may be disregarded if an in-stack filter is used that has no bends between the nozzle and the filter, provided that it is used in an application having unsaturated gases, a temperature well above the stack gas dew-point, and if a validation has been carried out at conditions similar to the process to demonstrate that the deposits do not exceed 10 % of the daily average limit value set for the process.

7.4.5 Validation of sample collection

A measurement series is validated only when the quantity of dust collected during the sampling is at least five times the corresponding overall blank value.

7.4.6 Summary of the requirements of this document

A summary of the requirements of Clauses 4 and 5 of this document is given in Table 3.

8 Additional aspects

8.1 Thermal behaviour of particulate matter

Emitted dusts are generally thermally stable. However, in some processes the gases to be sampled contain unstable or semi-volatile compounds (i.e. in particulate form at low temperature, in gaseous form at higher temperature). In such cases, the measured concentration depends on the filtration temperature and/or on the drying temperature before final weighing.

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

- power plants equipped with desulfurization processes, because of the occurrence of hydrates;
- heavy fuel oil power plants or diesel engines, because of the presence of SO_3 and/or organic compounds;
- glass furnaces, because of the presence of semi-volatile boron compounds;
- waste incinerators with wet and semi-dry gas treatment processes.

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

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

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

a) conditioned at 180 °C before sampling,

b) set at any temperature equal to or less than 160 °C during sampling,

c) conditioned at 160 °C after sampling.

Table 3 — Summary of requirements — Apparatus and sampling conditions

Equipment for dust collection	**Value**
Nozzle internal diameter, *d*	> 4 mm
Nozzle area: measurement uncertainty	±10 %
Nozzle: length with constant internal diameter	> 10 mm
Nozzle: variation in diameter angle	< 30°
Elbow: radius of the bend	> 1,5*d*
Nozzle straight length before the first bend	> 30 mm
Nozzle tip: distance to obstacles	> 50 mm
Filter efficiency (test aerosol 0,3 µm)	> 99,5 %
Filter material (absorption of components)	No reaction and no absorption
Condenser, drying tower: residual gas moisture	< 10 g/m³
Gas meter volume measurement uncertainty	±2 %
Absolute pressure measurement uncertainty	±1 %
Absolute temperature measurement uncertainty	±1 %
Alignment of the nozzle	±10 %
Isokinetic criteria (average measurement uncertainty)	$^{+15}_{-5}$ %
Leak test	< 2 %
Balance resolution (mg)	0,01 mg to 0,1 mg
Weighing uncertainties	< 5 % of the LV[a] set for the process (see 7.4.4)
Thermal stability duration of probe-housing filter heating	> 8 h
Overall blank value	< 10 % of the LV[a] set for the process or 2 mg/m³, whichever is smaller
Sampling time measurement uncertainty	±5 s
Linear measurement uncertainty (duct diameter)	±1 %
(nozzle diameter)	±0,2 mm or ±5 %, whichever is greater
Sampling location	
Flow angle	< 15°
Negative flow	None
Pressure difference (Pitot tube)	> 5 Pa
Ratio of max. gas velocity to min. gas velocity	3:1
Straight length before the sampling plane	> 5 hydraulic diameters (recommended)
Straight length after the sampling plane	> 2 hydraulic diameters (recommended)
Straight length before emission point	> 5 hydraulic diameters (recommended)
Number of sampling points	See Tables 1 and 2
Equipment for flue gas characteristics	
Absolute temperature	±1 %
Flue gas density	±0,05 kg/m³

[a] LV = limit value.

Depending on eventual regulatory requirements and plant authorization, on special kinds of effluent 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 (7.2.1) 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.

8.2 Particulate deposits upstream of the filter

Experimental work has shown that particulate deposits upstream of the filter cannot be neglected when sampling dust concentrations. It has also been shown that significant errors can be introduced in the measurements if these deposited materials are not recovered carefully.

Particulate deposits are dependent upon the design of the sampling equipment, and on the characteristics of the particulate matter to be sampled. Therefore, all non-weighed parts shall be checked for the deposition of particulate matter and, if present, the particulate matter shall be collected.

Deposits may be collected using the rinsing procedures of 7.3.6, either after each test of the measurement series, or at least after each measurement series on the same sampling plane, and at least once a day. If the material is collected at the end of the measurement series, the recovered mass is then attributed to the individual tests in proportion to the mass collected on each filter.

8.3 Improvement of the weighing procedure

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

Repeated weighing of the same parts, spread over several weeks under varying conditions, e.g. of external temperature, humidity, etc., provides an estimation of the actual precision of weighing, including the uncertainties related to the manipulation of the filters, equilibrium time, etc.

The results shall be used as a first estimate of the overall blank value, and can provide a means of calculation of the gas volume to be sampled in order to obtain significant data, taking into account the anticipated range of particulate concentrations.

9 Calculations

9.1 Isokinetic flowrate

To perform isokinetic sampling, the sampling system flowrate shall be calculated so that the velocity of sample gas entering the nozzle, v_n, is equal to the flue gas velocity at the sampling point, v_s, or:

$$v_n = v_s \tag{8}$$

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

$$q_V = A_n \cdot v_n \tag{9}$$

According to Formula (5), isokinetic sampling is conducted when:

$$q_V = A_n \cdot v_s \tag{10}$$

where A_n is the area of the nozzle.

Because the sampling flowrate is measured under conditions (temperature, pressure, and moisture content) that differ generally from the actual conditions of the gas in the duct, q_V, must be corrected as follows:

$$q_m = q_{V,a} \frac{(100\,\% - H_a)T_m \cdot p_a}{(100\,\% - H_m)T_a \cdot p_m} \tag{11}$$

where

q_m is the measured sampling volumetric flowrate;

$q_{V,a}$ is the sampling volumetric flowrate, expressed in the actual conditions at the sampling point;

H_m, H_a are the moisture contents (humidity), as percentage volume, of gases in the measured (m) and actual (a) conditions;

T_m, T_a are the temperatures (measured and actual) of the gas, in kelvin;

p_m, p_a are the absolute pressures (measured and actual) of the gas.

Compare the calculated target value of q_V to the $q_{V,a}$ obtained during the test to determine that they agree within ±10 %, the isokinetic sampling criteria of this document.

9.2 Dust concentration

9.2.1 General

For each test, calculate:

a) the sampled volume, V, specifying whether on a dry or wet basis and under normal condition;

b) the total mass of dust collected, m, on the filter and upstream of the filter from the filter rinses;

c) the dust concentration, c:

$$c = \frac{m}{V} \tag{12}$$

It can be necessary to express the particulate concentration relative to a reference O_2 content to correct for dilution effects and/or moisture.

This can be achieved by multiplying by the oxygen and carbon dioxide correction factors, $f_{c,O2}$ and $f_{c,CO2}$, (9.2.2 and 9.2.3 respectively).

9.2.2 Oxygen correction factor

$$f_{c,O_2} = \frac{21\% - \varphi_{O_2,ref}}{21\% - \varphi_{O_2,m}} \tag{13}$$

where

$\varphi_{O2,ref}$ is the volume fraction, in percent, of O_2 under reference conditions;

$\varphi_{O2,m}$ is the volume fraction, in percent, of O_2 measured;

21 % is the percent of oxygen in air.

9.2.3 Carbon dioxide correction factor

$$f_{c,CO_2} = \frac{\varphi_{CO_2,ref}}{\varphi_{CO_2,m}} \tag{14}$$

where

$\varphi_{CO2,ref}$ is the volume fraction, in percent, of CO_2 under reference conditions;

$\varphi_{CO2,m}$ is the volume fraction, in percent, of CO_2 measured.

10 Performance characteristics

10.1 General aspects

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

However, if one team performs successive parallel sampling tests with two identical sampling systems, such a procedure allows a statistical comparison between paired values x_1 and x_2 to be calculated.

The standard deviation, s, of the paired values is:

$$s = \frac{\sqrt{\sum_{i=1}^{n} \left(x_{i,1} - x_{i,2}\right)^2}}{2n} \tag{15}$$

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 that 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 (all parts), i.e. the maximum difference between two measurements by the same team, for a 95 % confidence level:

$$r = \sqrt{2} t_{0,95;n-1} \cdot s \tag{16}$$

These data shall 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 may be conducted and provided as an estimation of:

c) the external uncertainty linked to an individual measurement carried out by any team fulfilling the requirements of the standard. This uncertainty is to be taken into account when comparing the measured values to the emission limit value;

d) the reproducibility [according to ISO 5725 (all parts)], R, i.e. the maximum difference that can be expected, at a 95 % confidence, between two measurements by different teams working according to the standard at the process conditions.

When doing measurements at low concentrations, the detection limit may be estimated:

e) by parallel measurements and calculation of the uncertainty;

f) by successive measurements at near zero concentration. The detection limit is assumed to be three times the standard deviation.

10.2 Experimental data for sampling

See Annex E for information regarding validation of the sampling method.

11 Test report

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

a) description of the purpose of tests, identification of the site, date of sampling;

b) description of the operating conditions of the plant process, and any variation during measurements;

c) identification of the sampling location, and gas parameters in the duct;

   1) duct dimensions, number and position of sampling lines and sampling points,

   2) stack pressure,

   3) velocity and temperature profile,

   4) O_2/CO_2 (if relevant), humidity concentrations, gas volumes.

d) measurement procedures;

   1) velocity measurement (calibration of S-Pitot tubes, etc.),

   2) characteristics of sampling equipment,

   3) make (manufacturer of the sampling train),

   4) nozzle diameter, characteristic of filter (material, size, type),

    5) calibration of flowrate measurement devices,
    6) filtration temperature,
    7) weighing procedures,
    8) conditioning temperature,
    9) correction of apparent masses.

e) test results, for each test;

    1) test number and date,
    2) mass of particulate matter collected,
    3) temperature and pressure of the gas in the sampling train,
    4) gas composition (including moisture content),
    5) sampling time,
    6) sampled volume and flowrate,
    7) any special circumstances or incidents,
    8) corrected results (standard conditions),
    9) any intermediate and final calculation results.

f) quality assurance;

    1) leak tests results,
    2) overall blank value,
    3) isokinetic criteria,
    4) deposits of dust upstream of the filter.

g) comments.

    1) Indicate in the test results the ratio of the measured values (mg of dust) to the overall blank value (mg of dust).
    2) The test report shall also indicate any special circumstances that potentially influenced the results, and any information concerning the uncertainty of the results.
    3) If it has been necessary to modify the method for any reason, then this modification shall be reported.

Annex A
(normative)

Proven design of the entry nozzle

One of the designs shown in Figures A.1, A.2 and A.3 shall be used for the entry nozzle.

Dimensions in millimetres

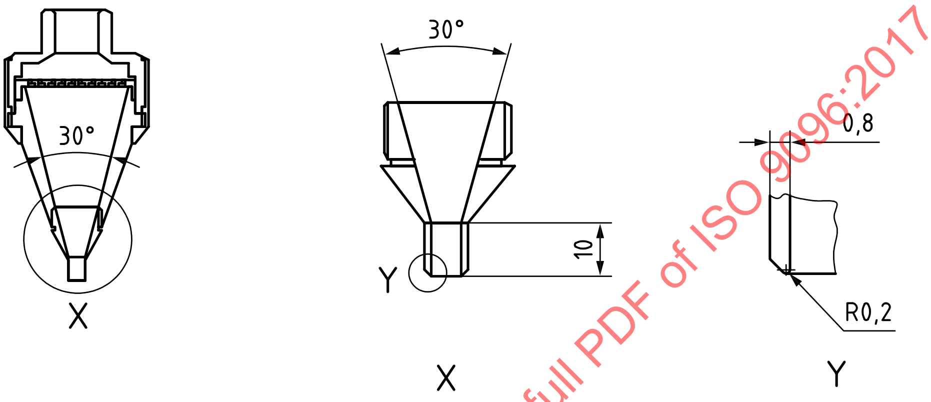


Figure A.1 — Entry nozzle, with combined filter holder

Dimensions in millimetres

Figure A.2 — Entry nozzle, single-angle

Dimensions in millimetres

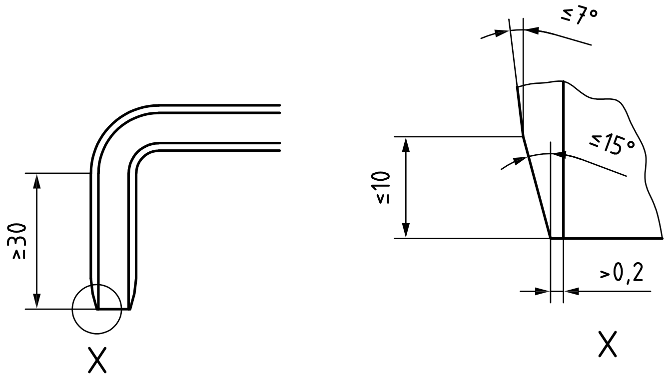


Figure A.3 — Entry nozzle, double-angle

Annex B
(normative)

Determination of positions of sampling points in circular and rectangular ducts

B.1 Requirements for circular ducts

B.1.1 General rule for circular ducts

In the general rule applicable to circular ducts, the sampling plane is 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 point at the centre of the duct (see Figure B.1).

The locations of the sampling points are dependent on the number of sampling points chosen.

For circular ducts, two sampling lines (diameters) are sufficient, the distance x of each sampling point i from the duct wall can be expressed as:

$$x_i = K_i \cdot d \quad \text{(B.1)}$$

where

K_i is a factor, expressed as a percentage, in accordance with Table B.1;

d is the length of the diameter of the duct.

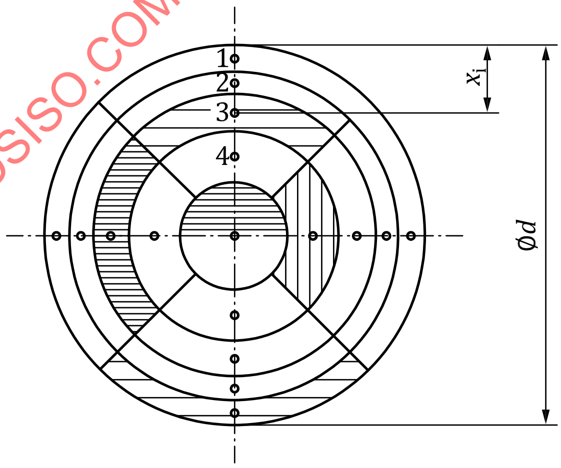


Figure B.1 — Sampling point positions in circular ducts — General rule for ducts over 2 m diameter (hatched portions are of equal area)

Table B.1 gives 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 B.1 — Values of K_i as a percentage — General rule for circular ducts

Sampling point index i	K_i			
	$n = 3$	$n = 5$	$n = 7$	$n = 9$
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 Formula (B.1) for calculating the distance of the sampling point from the duct wall, along the diameter, becomes:

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

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

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

where

i is the index of sampling point along the diameter;

x_i is the distance of point i from the duct wall;

n_i is the number of sampling points along each sampling line (including the centre);

n_d is the number of sampling lines (diameters);

d is the diameter of the duct.

B.1.2 Tangential rule for circular ducts

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 diameters (sampling lines), there being no sampling point at the centre of the duct (see Figure B.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.

For circular ducts where two sampling lines (diameters) are sufficient, the distance of each sampling point from the duct wall can conveniently be expressed as

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

where k_i is a factor, expressed as a percentage, in accordance with Table B.2.

Table B.2 gives 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.

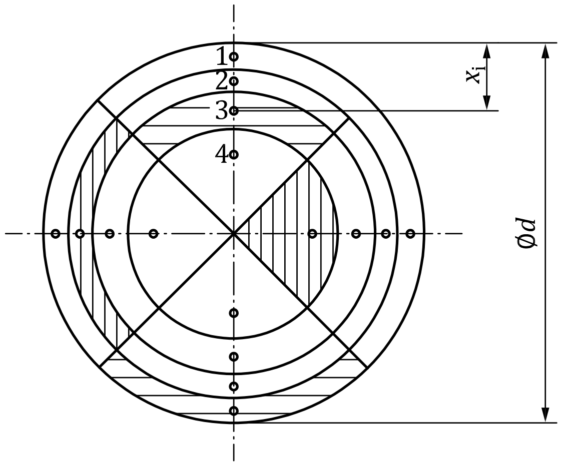


Figure B.2 — Sampling-point positions in circular ducts — Tangential rule for ducts over 2 m in diameter (hatched portions are of equal area)

Table B.2 — Values for k_i as a percentage — Tangential rule for circular ducts

Sampling point index i	k_i			
	$n = 2$	$n = 4$	$n = 6$	$n = 8$
1	14,6	6,7	4,4	3,3
2	85,4	25,0	14,6	10,5
3		75,0	29,6	19,4
4		93,3	70,4	32,3
5			85,4	67,7
6			95,6	80,6
7				89,5
8				96,7

For circular duct where it is necessary to increase the number of sampling lines (diameter) or the number of sampling points, the tangential Formulae (B.6) and (B.7) for calculating the distance, from the duct wall along the diameter are:

$$x_i = \frac{d}{2}\left[1-\sqrt{1-\frac{2i-1}{n_i}}\right] \qquad \text{for } i \le \frac{n_i}{2} \tag{B.6}$$

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

where the symbols have the same meaning as in Formula (B.2), but n_i does not include the centre.

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

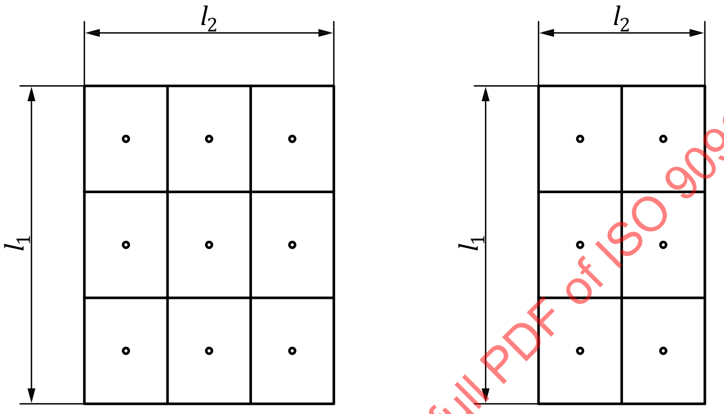
B.2 Requirements for rectangular (and square) ducts

In the rule applicable to rectangular ducts, including square 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 B.3.

In general, the two perpendicular sides of the rectangular duct are divided into an equal numbers 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 B.3 a).

If the lengths of the sampling plane sides, l_1 and l_2, where l_1 is greater than l_2, have a ratio $l_1 / l_2 > 2$, side l_1 shall 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. See Figure B.3 b).

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



a) $l_1 / l_2 \leq 2$

b) $l_1 / l_2 > 2$

Figure B.3 — Illustration of sampling point positions in rectangular (and square) ducts

Annex C
(informative)

Examples of weighing bias

C.1 General

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

In these examples, 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/ml.

C.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 at the room balance (300 K). Due to the change in air buoyancy, this difference of air temperature leads to an apparent mass variation of:

$(40 \times 1,2 \times 2)/300 = 0,3$ mg.

C.3 Effect of temperature variations

The room balance temperature is determined 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:

$9,4 \times 1,2 \times 10/300 = 0,4$ mg.

C.4 Effect of barometric pressure variations

The barometric pressure is determined 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:

$9,4 \times 1,2 \times 0,055 = 0,6$ mg.