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ISO 12141

Second edition
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Stationary source emissions — Determination of low range mass concentration of dust — Manual gravimetric method

Émissions de sources fixes — Détermination de faibles concentrations en masse de poussières — Méthode gravimétrique manuelle

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Foreword

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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 document 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).

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For an explanation of 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 www.iso.org/iso/foreword.html.

This document was prepared by CEN (as EN 13284-1:2017) and drafted in accordance with its editorial rules. It was assigned to Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 1, *Stationary source emissions*, and adopted under the “fast-track procedure”.

This second edition cancels and replaces the first edition (ISO 12141:2002), which has been technically revised.

The main changes are as follows:

— all technical changes have been listed in Annex I;

— "this European Standard" has been changed to "this document";

— "section" has been changed to "Clause" or "subclause".

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The measurement method specified in this document has been developed in close liaison and cooperation between ISO/TC 146/SC 1 and CEN/TC 264, resulting in the preparation of the first editions of the International Standard ISO 12141:2002 and the European Standard EN 13284-1:2001.

In the meantime, CEN/TC 264 has revised EN 13284-1:2001 in order to adapt the content to the state of the art. The basic concept of the measurement method has not been changed. Against this background and to ensure comparability of measurement results at international level, ISO/TC 146/SC 1 has decided to adopt EN 13284-1:2017 without technical changes. However, some editorial adjustments have been made to take account of the international application of this document. For example, references to EN 15259:2007 in EN 13284-1:2017 have been replaced in this document by references to the technically identical ISO 15259:2023.

To meet the specifications of this document, a certain level of accuracy for weighing the particle sample is needed. At low dust concentrations, this level of accuracy can be achieved by:

a) exercising extreme care in weighing, as per procedures of this document;

b) extending the sampling time at conventional sampling rates; or

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

High-volume sampling is not part of this document since it was not part of the validation of the measurement method.

The measurement method specified in this document can be used for the calibration of automated measuring systems (AMS) (see ISO 10155). If the waste gas contains unstable, reactive or semivolatile substances, the measurement depends on the filtration temperature, and in-stack methods can be more applicable than out-stack methods for the calibration of AMS.

Stationary source emissions — Determination of low range mass concentration of dust — Manual gravimetric method

1 Scope

This document specifies the standard reference method (SRM) for the measurement of low dust concentration in ducted gaseous streams in the concentrations below 50 mg/m^3 at standard conditions.

This document is primarily developed and validated for gaseous streams emitted by waste incinerators. More generally, it can be applied to gases emitted from other stationary sources, and to higher concentrations.

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

This method has been validated in field tests with special emphasis to dust concentrations around 5 mg/m^3. The results of the field tests are presented in Annex A.

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 15259:2023, *Air quality — Measurement of stationary source emissions — Requirements for measurement sections and sites and for the measurement objective, plan and report*

ISO 16911-1, *Stationary source emissions — Manual and automatic determination of velocity and volume flow rate in ducts — Part 1: Manual reference method*

3 Terms and definitions

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

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

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

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

3.1
dust
particles, of any shape, structure or density, dispersed in the gas phase at the sampling point conditions which 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

3.2
filtration temperature
temperature of the sampled gas immediately downstream of the filter

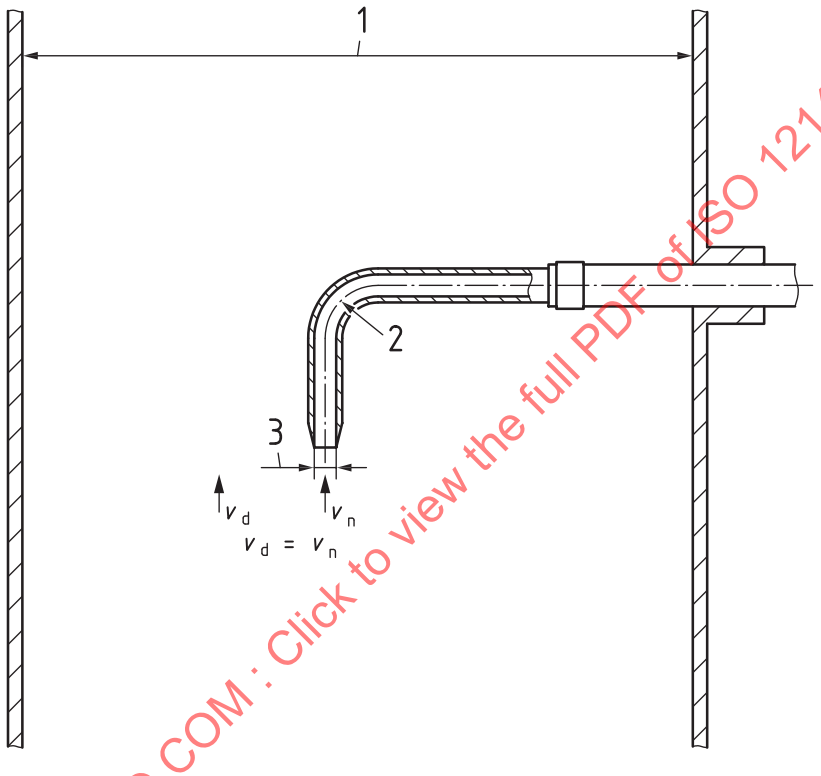
3.3
in-stack filtration
filtration in the duct with the filter in its filter housing placed immediately downstream of the sampling nozzle

3.4
out-stack filtration
filtration outside of the duct with the filter in its heated filter housing placed downstream of the sampling nozzle and the suction tube

3.5
isokinetic sampling
sampling at a flow rate such that the velocity v_n and direction of the gas entering the sampling nozzle are the same as the velocity v_d and direction of the gas in the duct at the *measurement point* (3.10)

Note 1 to entry: Figure 1 gives an illustration of isokinetic sampling.

Note 2 to entry: Annex B shows the influence of the *isokinetic rate* (3.6) on the representativeness of the collected particles.



Key

1 duct
2 radius of the bend (minimum 1,5 d_p)
3 internal diameter of the suction tube d_p

Figure 1 — Isokinetic sampling

3.6
isokinetic rate
velocity ratio v_n/v_d expressed in percentage as a characteristic of the deviation from *isokinetic sampling* (3.5)

3.7
hydraulic diameter
d_h
quotient of four times the area A and the perimeter P of the *measurement plane* (3.8)

$$d_h = \frac{4 \times A}{P} \quad (1)$$

[SOURCE: ISO 15259:2023, 3.14]

3.8
measurement plane
plane normal to the centreline of the duct at the sampling position

[SOURCE: ISO 15259:2023, 3.13]

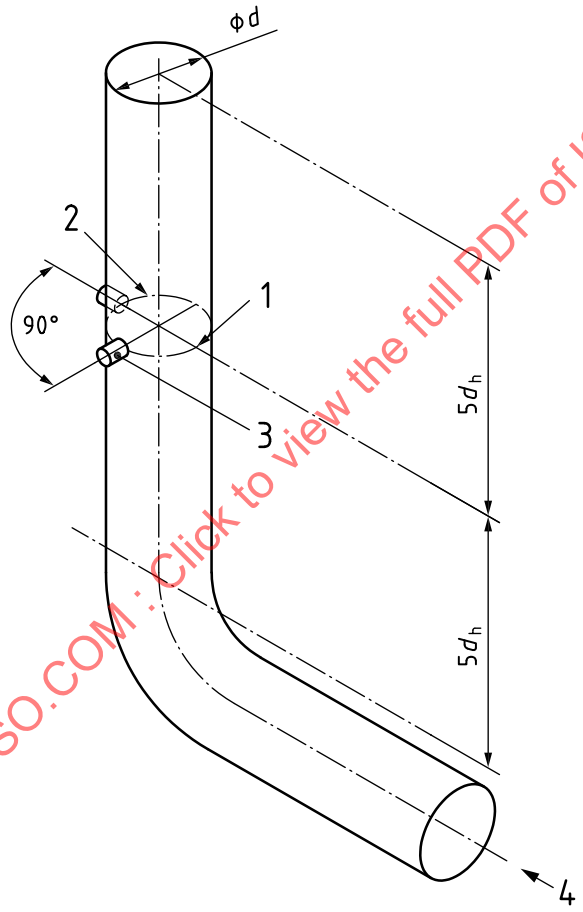
Note 1 to entry: Measurement plane is also known as sampling plane.

3.9
measurement line
line in the sampling plane along which the sampling points are located, bounded by the inner duct wall

[SOURCE: ISO 15259:2023, 3.15]

Note 1 to entry: Measurement line is also known as sampling line.

Note 2 to entry: Figure 2 gives an illustration of definitions in relation to a circular duct.



Key

1	measurement line	3	measurement port
2	measurement plane	4	flow direction

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

3.10
measurement point
position in the *measurement plane* (3.8) at which the sample stream is extracted or the measurement data are obtained directly

[SOURCE: ISO 15259:2023, 3.16]

Note 1 to entry: Measurement point is also known as sampling point.

3.11
measurement port
opening in the waste gas duct along the *measurement line* (3.9), through which access to the waste gas is gained

[SOURCE: ISO 15259:2023, 3.18]

Note 1 to entry: Measurement port is also known as sampling port or access port.

3.12
standard conditions
reference values for a dry gas at a pressure of 101,3 kPa and a temperature of 273,15 K

3.13
field blank
sample obtained according to the *field blank procedure* (3.14)

3.14
field blank procedure
procedure used to ensure that no significant contamination has occurred during all the steps of the measurement

Note 1 to entry: This includes for instance the equipment preparation in laboratory, its transport and installation in the field as well as the subsequent analytical work in the laboratory.

3.15
field blank value
result of a measurement performed according to the *field blank procedure* (3.14) at the plant site and in the laboratory

3.16
weighing control
procedure for the detection/correction of apparent weight variations due to possible changes between pre and post sampling weighing conditions

3.17
measurement series
successive measurements carried out at the same *measurement plane* (3.8) and at the same operating conditions of the industrial process

3.18
emission limit value
ELV
limit value given in regulations such as directives, ordinances, administrative regulations, permits, licences, authorizations or consents

Note 1 to entry: ELV can be stated as concentration limits expressed as half-hourly, hourly and daily averaged values, or mass flow limits expressed as hourly, daily, weekly, monthly or annually aggregated values.

Note 2 to entry: For purposes other than regulatory uses, the measurement value is compared to a stated reference value.

4 Symbols and abbreviations

4.1 Symbols

For the purposes of this document, the following symbols apply.

A area of the measurement plane

c dust concentration

d diameter of the duct

d_h hydraulic diameter

d_n internal diameter of the sampling nozzle

d_p internal diameter of the suction tube

f_c correction factor

h_a humidity of the gas in actual conditions, in percentage volume

h_m humidity of the gas in measurement conditions, in percentage volume

m total mass of dust collected upstream of the filter (rinsing) and on the filter

o_m oxygen concentration in percentage volume of dry gas measured in the duct

o_{ref} oxygen reference concentration in percentage volume of dry gas

P perimeter of the measurement plane

p_a absolute pressure of gases in actual conditions in the duct

p_m absolute pressure of the gas in measurement conditions at the volume meter

Q_a sampling volumetric flow rate, expressed in the actual conditions in the duct

Q_m measured sampling volumetric flow rate at gas meter conditions

T_a temperature of the gas in actual conditions in the duct, in Kelvin

T_m temperature of the gas in measurement conditions at the volume meter, in Kelvin

V sample volume

v_d velocity of the gas in the duct at the measurement point

v_n velocity of the gas entering the sampling nozzle

4.2 Abbreviations

For the purposes of this document, the following abbreviations apply.

ELV emission limit value

PTFE polytetrafluoroethylene

5 Principle

A sample stream of the gas is extracted from the main gas stream at representative measurement points for a measured period of time, with an isokinetically controlled flow rate 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 system are also recovered and weighed. The increase of mass of the filter and the deposited mass upstream 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 system may be used depending on the characteristics of gases to be sampled (see 7.2.2).

Valid measurements can be achieved only when:

a) the gas stream in the duct at the measurement site (sampling location) has a sufficiently steady velocity profile (see ISO 15259);

b) sampling is carried out without disturbance of the gas stream with a sharp edged nozzle facing into the stream under isokinetic conditions;

c) samples are taken at a pre-selected number of stated positions in the measurement plane, to allow for a non-uniform distribution of dust in the duct;

d) the sampling system is designed and operated to avoid condensation, chemical reactions and to minimize dust deposits upstream of the filter and to be leak free;

e) sampling is carried out at an appropriate filtration temperature, e.g. stack temperature or at least the recommended temperature of 160 °C (see Annex H);

f) dust deposits upstream of the filter are taken into account;

g) the field blank value does not exceed 10 % of the lowest emission limit value set for the process or 0,5 mg/m^3, whichever is greater;

h) the sampling and weighing procedures are adapted to the expected dust quantities;

i) the expanded uncertainty calculated by means of an uncertainty budget does not exceed the corresponding specification in the measurement objective. For regulatory purposes the expanded uncertainty shall not exceed 20 % of the emission limit value specified by the authorities unless specified otherwise by the competent authorities.

NOTE The Industrial Emissions Directive of the European Union (IED) e.g. specifies a maximum permissible uncertainty of 30 % of the daily emission limit value (ELV) for automated dust measuring systems. This requires that the expanded uncertainty of the SRM is lower for calibration purposes.

Annex D provides a summary of the requirements for the application of this measurement method.

6 Measurement planning and sampling strategy

6.1 Measurement planning

Emission measurements at a plant shall be carried out such that the results are representative of the emissions from this plant for operating conditions specified in the measurement objective and comparable with results obtained for other comparable plants. Therefore, dust measurements shall be planned in accordance with ISO 15259.

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 with the plant management.

Preliminary calculations shall be made on the basis of expected dust concentration in order to verify that expected sampled dust quantities are consistent with attainable field blank values, and that no overloading of the filter occurs (see Annex E).

For sampling duration limited to 30 min, required for certain trial or regulatory purposes, the uncertainty of measurement can increase due to the limited sample volume. Furthermore, completion of sampling along two diameters within 30 min, even for medium size ducts, can require simultaneous sampling with two or more sampling systems.

Where possible, the sampling duration can be extended, which decreases the quantification limit and improves the measurement uncertainty (see Annex E). The sampling duration should be selected, to minimize the effect of non-steady-state conditions of the stationary source.

Taking into account the objective of the measurements and the conditions of waste gases to be sampled, the user shall choose between an in-stack or an out-stack filtration device. If gas in the duct contains droplets out-stack filtration devices shall be used.

A field blank shall be taken (see 9.7).

If no suitable sampling location exists in the plant, and/or that measurements have been carried out during non-steady-state conditions of the plant, which leads to an increase of the uncertainty of the measurements, it shall be stated in the report.

6.2 Sampling strategy

6.2.1 General

Sampling requires a suitable measurement section and measurement plane.

The measurement plane shall be easily reached from convenient measurement ports and a safe working platform (see ISO 15259).

Sampling shall be carried out at a sufficient number of measurement points located on the measurement plane as specified by ISO 15259.

6.2.2 Measurement section and measurement plane

The measurement section and measurement plane shall meet the requirements of ISO 15259.

6.2.3 Minimum number and location of measurement points

The measurements shall be performed as grid measurements.

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

EN 15259 specifies the minimum number of measurement points to be used and the location in the measurement plane for circular and rectangular ducts. The number of measurement points and the location in the measurement plane shall be selected in accordance with ISO 15259.

6.2.4 Measurement ports and working platform

Measurement ports shall be provided for access to the measurement points selected in accordance with ISO 15259.

Examples of suitable measurement ports are given in ISO 15259.

For safety and practical reasons, the working platform shall comply with the requirements of ISO 15259.

7 Equipment and materials

7.1 Gas velocity, temperature, pressure and composition measurement devices

The equipment used for the point-related velocity measurements to establish isokinetic conditions shall meet the requirements of ISO 16911-1.

When expressing dust concentrations at standard conditions on a dry basis, and/or where the concentrations shall be expressed in relation to a reference oxygen concentration, the necessary measuring equipment shall meet the requirements of the applicable standards.

7.2 Sampling equipment

7.2.1 Sampling system

The sampling system principally consists of:

a) filtration device consisting of the filter housing and filter;

b) entry nozzle;

c) suction tube for out-stack filtration devices;

d) gas pump;

e) gas metering device including cooling and drying system and system for controlling isokinetic sampling conditions.

All parts of the sampling system which come in contact with the sampled gas shall be made of corrosion resistant and, if necessary, heat resistant material, e.g. stainless steel, titanium, quartz or glass.

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

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

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

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

All parts of the sampling system which come in contact with the sample gas shall be protected from contamination e.g. during handling and transportation.

7.2.2 Filtration device

7.2.2.1 General

The filtration device consists of the filter housing and the filter.

The filtration device is either located in the duct (in-stack filtration) or placed outside the duct (out-stack filtration):

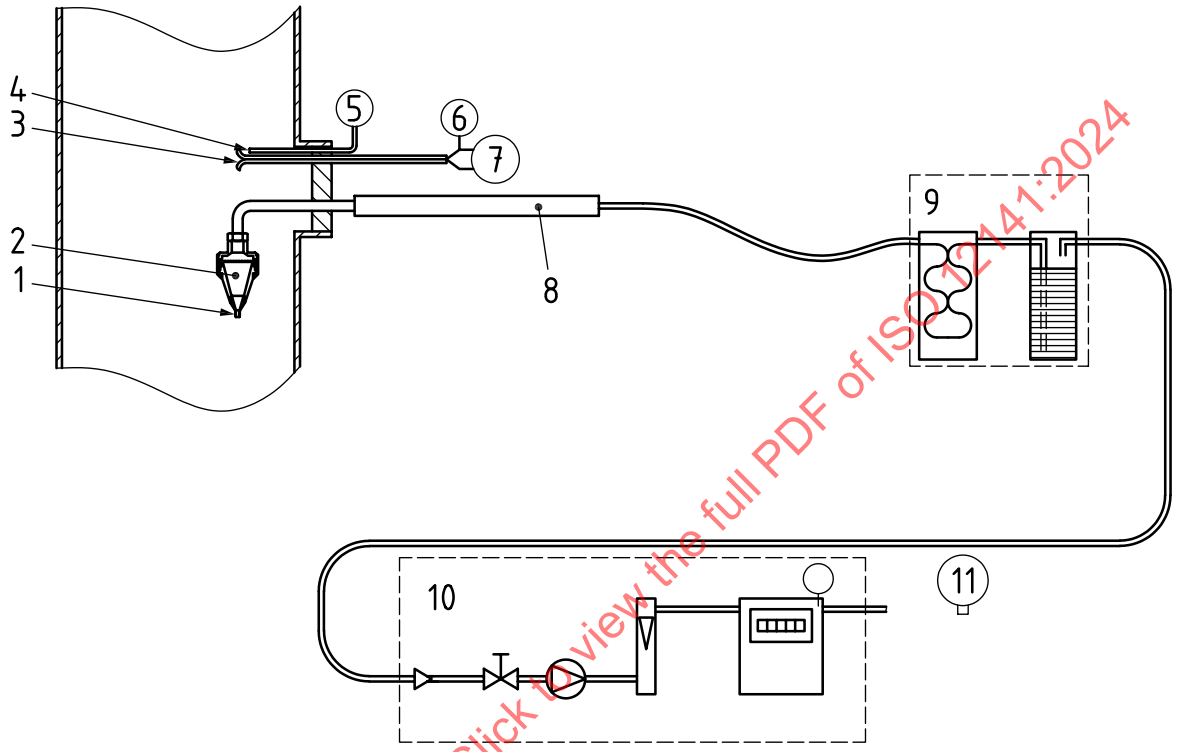
a) in-stack filtration devices (see Figure 3):

The part of the tubing between nozzle and filter should be very short, thereby minimizing dust deposits upstream of the filter. Due to available access port dimensions on ducts, the filter diameter is then typically limited to 50 mm, with a sample flow rate of approximately 1 m^3/h to 3 m^3/h. Since the filtration temperature is generally identical to that of the gas in the duct, filter clogging can occur if the stack gas contains water droplets.

To allow access to all measurement points in the duct, a leak free rigid tube of sufficient length (support tube) is used downstream of the filter housing for mechanical support of the filtration device.

b) out-stack filtration devices (see Figure 4):

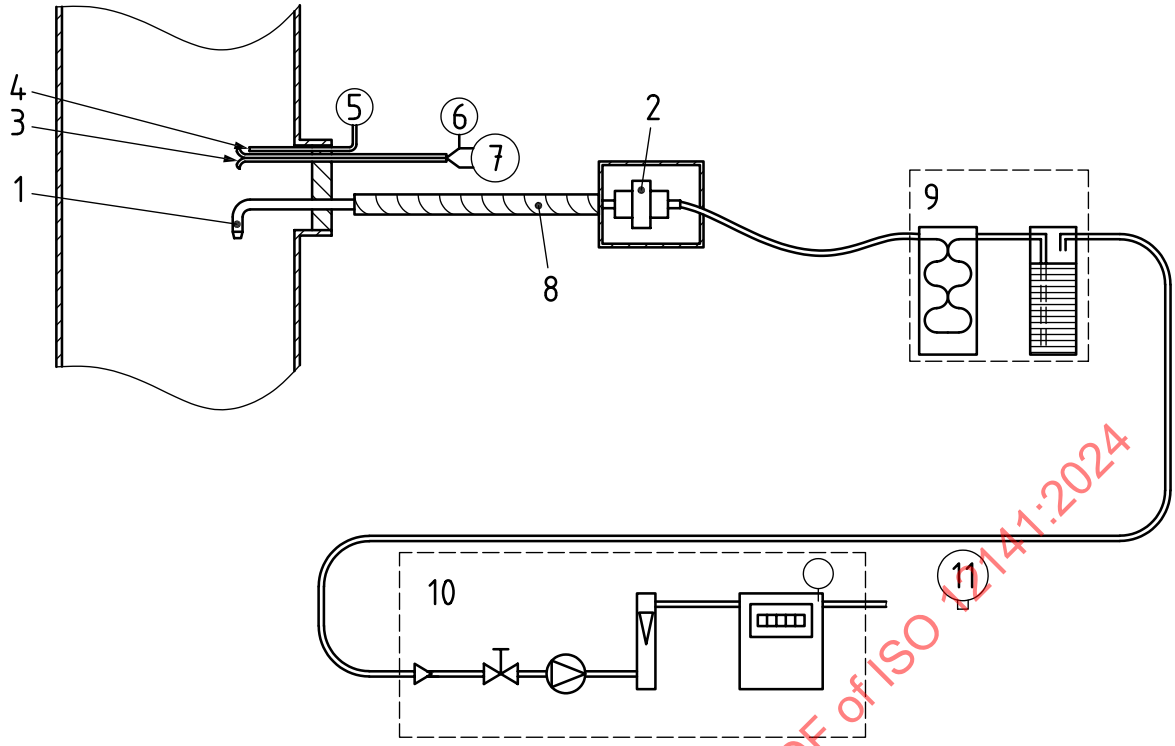
The part of tubing between the nozzle and the filter (suction tube) shall be of sufficient length to allow access to all measurement points in the duct. The suction tube and the filter housing shall be temperature controlled, which provides evaporation of possible water droplets or avoids filtration difficulties related to high acid dew point gases. Filter diameters between 50 mm and 150 mm are generally used, with associate flow rate of 1 m^3/h to 10 m^3/h.



Key

1 entry nozzle
2 filter housing
3 pitot tube
4 temperature sensor
5 temperature indicator
6 static pressure measurement
7 dynamic pressure measurement
8 support tube (in-stack device)
9 cooling and drying system
10 suction unit and gas metering device
11 pressure gauge

Figure 3 — Example of a sampling system for in-stack filtration



Key

1 entry nozzle
2 filter housing
3 pitot tube
4 temperature sensor
5 temperature indicator
6 static pressure measurement
7 dynamic pressure measurement
8 suction tube (out-stack device)
9 cooling and drying system
10 suction unit and gas metering device (see Figure 5)
11 pressure gauge

Figure 4 — Example of a sampling system for out-stack filtration

7.2.2.2 Filter housing

The filter housing is a casing in which the filter is mounted directly or by use of a filter holder. When the filter housing is placed out-stack, it shall be temperature controlled to maintain filtration conditions and to avoid condensation.

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

a) the filter, or the filter with the filter holder

In this case the dust deposits at the inlet of the filter housing shall be recovered and weighed (see 9.6). The use of a filter housing with a conical inlet of an angle less than 30° helps to minimize dust deposits.

b) the filter, inlet part of the filter housing and the upstream parts (e.g. nozzle)

In this case dust deposits upstream of the filter are taken directly into account. It is necessary to check whether the parts to be weighed are compatible with the range of the balance (see 8.3).

The filter housing and the filter holder shall be designed in such a way that no gas turbulence occurs near the joints.

7.2.2.3 Filters

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

— plane filter efficiency better than 99,5 % on a test aerosol with a mean particle diameter of 0,3 µm, at the maximum flow rate anticipated, or better than 99,9 % on a test aerosol of 0,6 µm mean particle diameter. This efficiency shall be certified by the filter supplier;

— the filter material shall not react with or adsorb gaseous compounds contained in the gas to be sampled, and shall be thermally stable, taking into account maximum temperature anticipated (e.g. for conditioning and sampling).

The choice of the filter should also take into account the following considerations:

— the pressure drop of the filter, and increase due to the collection of the dust while sampling. This depends on the kind of filter. As an example the pressure drop can be between from 3 kPa to 10 kPa for a filtration velocity in the range of 0,5 m/s;

— when using filters with organic binders, care shall be taken of possible weight variations due to binder losses by evaporation when heating;

— glass fibre filters can react with acidic compounds such as SO_3, which leads to an increase in weight; their use is not recommended;

— despite their weak mechanical properties quartz fibre filters are proven to be efficient in most cases;

— PTFE filters are also proven to be efficient, however the maximum allowable temperature of the gas passing through the filter is limited (see Table 1).

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

Table 1 gives an overview on filter material and their strengths and limitations.

Table 1 — Filter material and their strengths and limitations

Material	Strengths	Limitations
Cellulose nitrate		— cannot be exposed to temperatures above 125 °C — low filter capacity for dust
PTFE[a]	— proven efficiency — not prone to fibre loss	— cannot be exposed to temperatures above 230 °C — mechanically weak — prone to curling during conditioning in oven — electrostatic charges can affect weighing
Fibreglass	— strong mechanical properties	— reacts with acidic compounds, such as SO_3 — cannot be exposed to temperatures above 200 °C
Quartz fibre	— thermally stable — resistant to chemical reactions with waste gases, such as HF, HCl, SO_2, SO_3, H_2SO_4, NO and NO_2	— fibre loss may occur — weak mechanical properties — cannot be exposed to temperatures above 700 °C

[a] PTFE can be exposed to temperatures of up to 230 °C. However, some manufacturers have lower temperature limits, with some stating a limit of 120 °C.

Laser cut filters have a raised edge, due to the effect of the laser heating the filter material. For this reason laser cut filters can lose material, due to mechanical damage to the fused edge when the filter is clamped in a filter holder and removed from the holder for weighing.

7.2.3 Entry nozzle

The sample gas stream to be measured enters the sampling system via the nozzle. The nozzle is connected either to the suction tube or to the filter housing.

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 major change of the sample gas flow rate, the sampling equipment shall be supplied with a set of nozzles of different diameters.

The entry nozzle shall be sharp in order not to disturb the main gas flow. Annex C details four proven designs. Other designs are allowed, provided they are validated to give equivalent results.

The uncertainty of the area at the nozzle entry shall be less than 5 % in order to fulfil isokinetic sampling criteria. For this reason, it is recommended to use nozzles with an inside diameter exceeding 8 mm.

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

a) constant internal diameter of the nozzle 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 shall not exceed 30°;

c) bends are allowed only after a minimum straight length of 30 mm; their radius shall be at least 1,5 time the internal diameter;

d) any change in the external diameter of the sampling system parts located at less than 50 mm from the nozzle tip shall be tapered and of conical angle shall not exceed 30°;

e) obstacles related to the sampling system are:

   1) prohibited upstream the nozzle tip;

   2) allowed besides and downstream of the nozzle tip, when situated at a distance of at least 50 mm or one times the size of the obstacle, whichever is the greater.

7.2.4 Suction tube for out-stack filtration devices

The suction tube shall have a smooth internal surface, and shall be designed to facilitate inspection and mechanical cleaning. The tube shall be temperature controlled to maintain the planned conditions for the filtration of the gases.

7.2.5 Suction unit

The suction unit (gas pump) shall be tight, corrosion-proof and shall be capable of extracting the maximum rated flow rate in the sampling conditions (vacuum at the suction side down to e.g. 40 kPa). Wide adjustments of sampled flow rate shall be controlled by a regulating valve and/or by-pass. A shut off valve shall also be available to shut-off the gas flow through the sampling train.

7.2.6 Gas metering devices

The following two kinds of gas metering systems may be used:

a) flow rate measurements on a dry basis (see Figure 5):

   1) condenser and/or gas drying tower;

   2) gas-tight pump;

   3) flow meter, in order to facilitate the flow rate adjustment, calibrated against the dry gas volume meter;

4) dry gas volume meter (maximum expanded uncertainty of 5,0 % at the anticipated flow rate) with associated absolute pressure and temperature measurement (maximum expanded uncertainty of 2,0 % each);

b) flow rate measurements on a wet basis (see Figure 5):

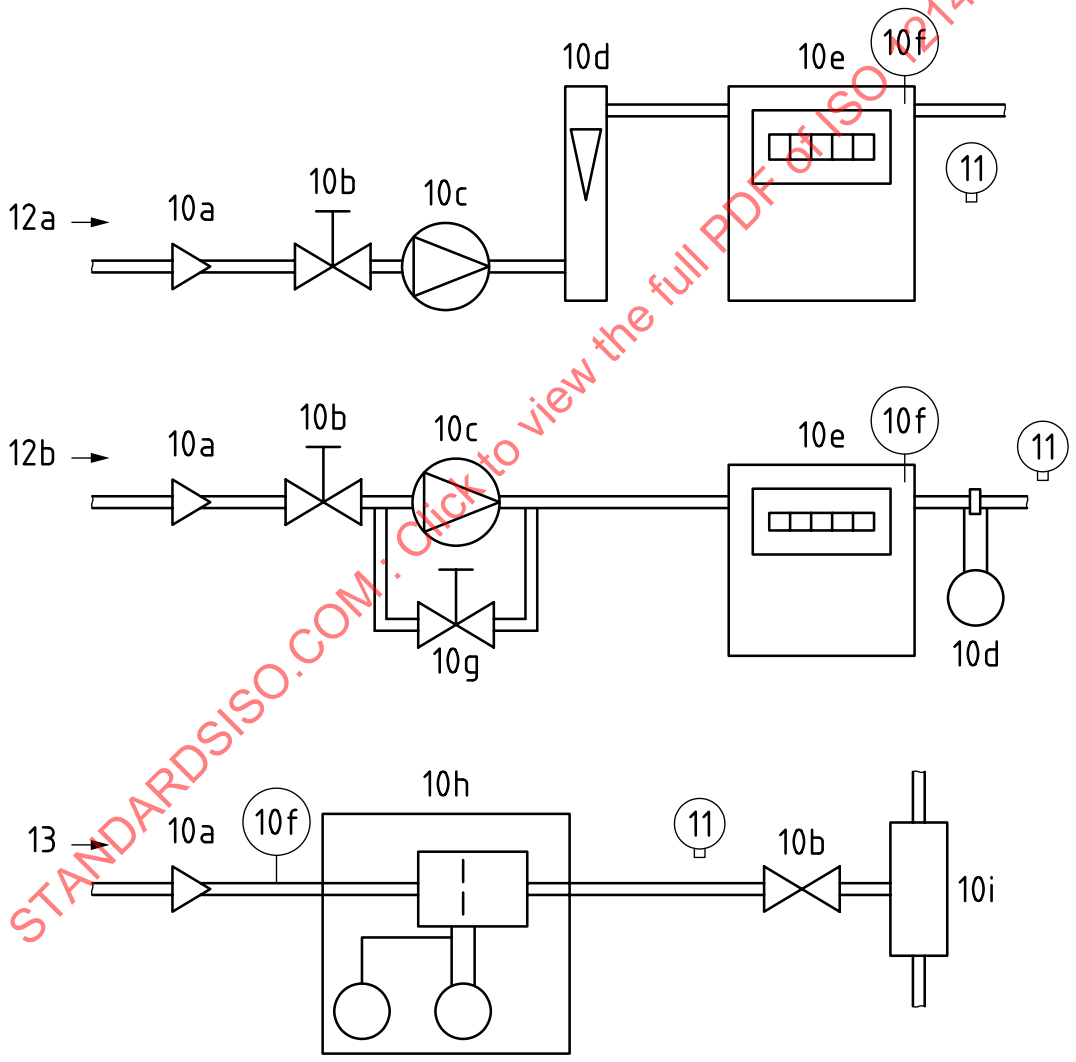
1) heated tubing, in order to prevent upstream condensation of the sample gas;

2) orifice plate or equivalent device (flow meter), maximum expanded uncertainty of 10,0 % of the anticipated flow rate; the expanded uncertainty of the measured values of temperature and pressure (absolute and differential) measurement shall not exceed 2,0 % each;

3) compressed air ejector acting as suction device;

4) atmospheric pressure measuring device.

Other types of systems are allowed, provided that the component parts meet the requirements specified in this subclause.



Key

10a shut off valve
10b adjustment valve
10c pump
10d flow meter
10g bypass control valve
10h heated orifice plate
10i compressed air ejector
11 pressure gauge

10e	dry gas volume meter	12a	dry gas
10f	temperature indicator for 12a	12b	dry gas
10f	temperature and pressure indicator for 12b and 13	13	wet gas

Figure 5 — Examples of suction unit and gas metering devices

7.3 Dust deposit recovery accessories

The following accessories may be used for the dust deposit recovery:

a) purified water (de-ionized and filtered having a dry residue of 1,0 mg/l or less)

b) acetone (pA grade having a dry residue of 1,0 mg/l or less);

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

d) plugs (acetone resistant) to close the suction tube.

7.4 Equipment for conditioning and weighing

The following equipment may be used for conditioning and weighing:

a) weighing containers for the drying procedure of the rinsing solutions. The mass shall be in accordance with the balance to be used. Glass and ceramic have proven to be suitable materials. Plastic materials are not recommended;

b) desiccators: located in the weighing room, with a desiccating agent (silica gel, calcium chloride);

c) drying oven: laboratory drying oven, thermally controlled within ± 5 °C;

d) balance: resolution from 0,01 mg to 0,1 mg, the range shall be compatible with the mass of parts to be weighed (see 7.2.2.2). Depending on the balance room location, specific care shall be taken to avoid reading instability related to vibrations, air draughts and temperature variations;

NOTE The weighing uncertainty is not only related to the balance characteristics but to the whole procedure (see Annex E).

e) thermometer and humidity meter near the balance;

f) pressure gauge;

g) depending on the evaporation procedure, an extraction hood and heating plate for the evaporation of rinsing solution shall be provided.

8 Weighing procedure

8.1 General

Depending on the kind of sampling system to be used, the parts to be weighed can be the filter with or without its filter holder, or can also include all upstream parts from the filter.

Depending on the procedure to be used the rinsing solutions can be evaporated and weighed in the same container or transferred to a smaller container for weighing.

8.2 Pre-sampling conditioning

If the filter housing is part of weighing parts the outside surfaces shall be cleaned prior to weighing.

Weighed parts shall be dried in a drying oven for at least 1 h before sampling at a temperature of at least 20 °C above the maximum temperature reached during sampling and post-sampling treatment (see Annex H).

The temperature used while conditioning before weighing shall be indicated in the measurement report.

The filters and/or the weighing containers are cooled down to ambient temperature in a desiccator located in the weighing room for at least 4 h. For larger parts, e.g. weighing containers, a duration of up to 12 h can be necessary.

8.3 Weighing

Since dust concentration is determined by difference between weights, special care is required in order to avoid weighing errors related to balance drift, to insufficient temperature equilibrium of parts to be weighed, and to climatic changes (see examples in Annex F). Therefore, before performing any measurement, the user shall validate their weighing procedure (see 8.6). It is strongly recommended to use the same balance for both pre-weighing and past weighing.

Before each weighing series:

a) the balance shall be checked against standard weights;

b) additional check shall be carried out 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) the climatic conditions in the room shall be recorded.

When weighing large volume parts (e.g. beakers), the temperature and barometric pressure can influence the apparent weight, this can be detected using the reference weight of the control parts. In these conditions, weighing corrections shall be applied, based on the apparent weight modification of the control part of each type (filter with filter holder, container etc.) or by calculating the influence of the change of barometric pressure on the basis of performance characteristics previously determined by each test laboratory.

Attention has also to be drawn on an increase or decrease in weighing due to:

— electrostatic charges, which give erratic readings and which may have to be discharged/neutralized (metallic plate, ion gun);

— hygroscopic characteristics of the filter material and/or dust;

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

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. If a significant increase is detected, the sample shall be put back into the desiccator for at least 4 h and then the weighing procedure shall be repeated. The dry reference weight shall then be calculated by extrapolation to zero time.

8.4 Post-sampling treatment of weighed parts

If the filter housing is part of weighing parts the outside surfaces shall be cleaned prior to weighing.

Weighed parts shall be dried after sampling in a drying oven for at least 1 h at 160 °C. Afterwards they shall be equilibrated to ambient temperature as described in 8.2. For specific reasons to be presented in the measurement report an alternative conventional temperature for post-sampling treatment may be selected (see Annex H).

The temperature used while conditioning shall be indicated in the measurement report.

8.5 Post-sampling treatment of the rinsing solutions

All the rinsing solutions (water and acetone) from all parts upstream of the filter as described in 9.6 shall be taken to the laboratory for the further treatment. Care shall be taken that no contamination occurs.

The solutions shall be transferred carefully to the dried and pre-weighed containers (see 8.1). During the evaporation the solvent mixture shall not be boiled. As the volume of solution is reduced through the evaporation process, small vessels may be used before the final weighing container.

NOTE Proven methods for evaporation of rinsing solutions are:

a) evaporate in an oven at 120 °C at ambient pressure;

b) evaporate in a closed system (desiccator). The initial temperature is set to 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 evaporation the weighing containers shall be placed in the drying oven for at least 1 h at 160 °C, then cooled down to ambient temperature as described in 8.2. For specific reasons to be presented in the measurement report an alternative conventional temperature for post-sampling treatment may be selected (see Annex H).

From the solvents used at least one blank value from the same batch shall be determined, for possible correction.

8.6 Improvement of the weighing procedure

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

Repeated weighing of the same parts, spread over several weeks in various conditions, i.e. ambient temperature, atmospheric pressure and humidity, provide through the standard deviation an estimation of the actual precision of weighing, including the uncertainties related to i.e. the manipulation of the filters and equilibrium time.

The results are used as a first estimate of the field blank value and provide a means of calculation of the gas volume to be sampled, in order to get significant data, taking into account the anticipated range of dust concentrations (see Annex E).

If the actual precision of weighing is not sufficient, weighing can be improved by controlling the influence parameters e.g. in climate controlled environment.

9 Sampling procedure

9.1 Preparation

The equipment shall be cleaned, prepared and checked before moving to site. Care shall be taken not to reuse any part of a sampling system previously used for high dust concentration sampling without dismantling and thorough cleaning.

Depending on the measurement programme, filters and associated parts to be weighed shall be prepared for each measurement series.

Perform weighing of the parts in accordance with Clause 8.

All the weighed parts, the suction tube and the other parts of the equipment which will come in contact with the sample and will be rinsed later shall be protected from contamination during transportation and storage.

9.2 Filter handling

Filter handling can be a major source of uncertainty in the dust measurement, often leading to structural damage to the filters and the potential for loss of material from the filters. Therefore, handling of filters shall be reduced to a minimum.

Whenever possible, filters shall be installed into individual filter holders at the permanent premises of the test laboratory, prior to going to the measurement site. Each filter with its filter holder shall be placed in an individual storage box for transportation purposes.

At the measurement site, the filter in its filter holder is removed from the storage box and placed into the filter housing. After sampling the filter with its filter holder is removed from filter housing and placed back into the storage box for the transportation to the laboratory.

NOTE 1 The unit of a filter in its filter holder protects the filter from structural damage that can be caused by handling and from influences by surrounding conditions.

In case of in-stack filtration, the filter in its filter holder may be combined with the filter housing and the entry nozzle providing one filtration unit.

If one filter holder is used on site for several filters, measures shall be taken to prevent damage of the filters caused by handling and to prevent influences on the collected dust on the filters by surrounding conditions such as strong wind and rain.

NOTE 2 Filter holders that clamp the filter using a clamping ring can cause mechanical damage, which can lead to structural damage of the filter that can cause filter losses during sampling.

NOTE 3 Different approaches are used for handling and weighing filters after sampling. Filters in filter holders can be removed from the filter holder and weighed, taking care to ensure that any loose filter material attached to the filter holder is also removed and weighed. Alternatively, the filter holder and filter can be weighed as a combined unit. Filters in combined filtration units are weighed together.

NOTE 4 Experience has shown that filters can contain loose material. Filters can be preconditioned by pulling clean ambient air through the filter for a specified length of time and flow rate to ensure that no further loss of filter material occurs after conditioning. If pre-blowing of filters is carried out, the filters are then installed into filter holders in the same direction for sampling, as they were when they were pre-blown.

9.3 Pre-measurements

Dust measurements can be performed using simple probes without velocity device or with combined probes (waste gas sampling and velocity measurement simultaneous at each measurement point). The preferred method is to use combined probes with the isokinetic sampling flow calculated from the actual velocity measurement. For steady processes a simple probe may be used. The isokinetic sampling flow is then calculated from the velocity profile established previously. The selection of the nozzle diameter depends upon the capacity of the pump as well as the waste gas conditions (e.g. velocity, temperature and moisture).

Therefore, extra information about the duct cross-section, the waste gas and steadiness of the plant operation is needed for appropriate dust measurements.

The following steps shall be executed:

a) determine the waste gas composition, especially the concentration of oxygen, CO_2 and water-vapour;

b) calculate the density of the waste gas from the waste gas composition;

c) select the number and location of measurement points in accordance with ISO 15259;

d) if the dust measurement is not performed using a combined probe a velocity measurement device shall be installed at a relevant fixed point in the measurement plane. The velocity data from this fixed point is used to prove steadiness of the process;

e) measure the temperatures and velocities of the gas at the selected measurement points in the duct, check also for possible deviations of gas flow with regard to duct axis; verify that the requirements of ISO 15259:2023, 5.2 are fulfilled, otherwise, see 6.1;

f) taking into account the preliminary calculations (see Annex F) and the measured velocities, select a suitable entry nozzle diameter which meets the isokinetic conditions for all the measurement points;

g) unless steadiness of the stationary source is proved the actual data of the velocity during sampling is used for isokinetic sampling at each measurement point;

h) if a simple probe is used and if the variation in velocity measured with the fixed Pitot probe during the measurements under e) is less than 10 %, the process is assumed to be steady. In those cases the isokinetic flow during sampling can be calculated from the velocity profile of the pre measurements at every measurement point in the duct.

9.4 Leak test

Perform a leak test on the sampling system before each measurement. Check the sample gas line for leakage according to the following procedure or any other relevant procedure:

a) assemble the complete sampling system;

b) seal the nozzle inlet;

c) switch on the pump;

d) read the flow rate after reaching minimum pressure;

e) measure the leak flow rate, e.g. by a rotameter, which shall not exceed 2,0 % of the expected sample gas flow rate.

NOTE Good laboratory practice includes regular checks of the pump and of the equipment between the pump and the gas meter for leaks.

Perform the leak test at the operating temperature unless this conflicts with safety requirements.

Integrity of the sampling system can be also tested during sampling by continuously measuring the concentration of a suitable stack gas component (e.g. oxygen) directly in the stack and downstream the sample gas line. Any systematic difference between those concentrations indicates a leak in the system.

Leaks shall be investigated and rectified.

9.5 Sampling

Sampling shall be carried out according to the following procedure:

a) Preheat the relevant parts of the sampling train to the selected filtration temperature, e.g. stack temperature or at least the conventional temperature (see Annex H). The temperature used while sampling shall be indicated in the measurement report.

NOTE 1 For in-stack devices, the relevant parts of the sampling train can be preheated in the stack before sampling with the nozzle directed downstream.

Insert the sampling train into the duct with the nozzle, if possible, facing downstream avoiding contact with any parts of the duct; at very low pressures in the waste gas duct the pump shall run at low power before the probe is inserted to avoid that filter material is sucked back.

Seal up the opening of the measurement port in order to minimize air ingress in the duct or exposure of operators to harmful substances;

b) Turn the suction tube until the entry nozzle is facing upstream within ± 10°, open the shut off valve, start the suction device and adjust the flow rate in order to obtain isokinetic sampling within –5 % and +15 % (see Annex B). Use the same sampling duration of at least 3 min at each selected measurement point.

c) Use a total sampling duration of at least 30 min.

d) Record at the beginning of sampling the barometric pressure, temperature and volume of gas meter.

e) At each measurement point and at least every 5 min, record the parameters used for determining the stack gas velocity. Based on this value adjust the flow rate for isokinetic sampling within –5 % and +15 %.

NOTE 2 Good laboratory practice when using dry gas meter is to record at least every 5 min gas meter temperature and pressure and to use the results for calculating the final sampled volume.

f) Do not stop sampling when moving the sampling train to the following measurement point, and immediately adjust the flow rate for isokinetic conditions.

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

NOTE 3 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.

h) On completion of sampling at all measurement points:

1) Record at the end of sampling the temperature and volume of gas meter.

2) Remove the sampling system after closing the shut-off valve and suction device. At very low pressures in the waste gas duct the pump shall run at low power after the probe is removed to avoid that filter material is sucked back.

3) Dismantle the sampling system and check visually the filter and the filter housing for signs of breakage or condensation (sampling equipment operated below or too close the dew point). In such cases, the measurement is not valid.

i) Put the parts to be weighed in a closed electrostatic free container for transport to laboratory for weighing (see Clause 8).

9.6 Recovery of deposits upstream of the filter

9.6.1 General

Experimental work carried out when preparing this document 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 concentration around 5 mg/m^3.

These deposits depend probably 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 of the sampling equipment upstream of the filter shall be rinsed. The mass of dust on non-weighed parts upstream of the filter shall be indicated in the measurement report, besides 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 housing (see Annex C) on non-saturated gases, with a temperature well above the stack gas dew point, the upstream deposits do not have to be recovered provided that validation has been carried out at similar conditions as the process to prove that the deposits, expressed in the same units as the measurement results (e.g. in milligrams per cubic metre) do not exceed 10 % of the emission limit value set for the process.

NOTE The influence of deposits not recovered on the measurement uncertainty can be evaluated on the basis of the results of the validation.

9.6.2 Rinsing procedure

All the non-weighed parts upstream of the filter which are in contact with the gas sample shall be rinsed to recover the deposits unless the recovery of possible deposits is not required (see 9.6.1).

Special care shall be taken to avoid contamination if the rinsing is done on site. Rinsing shall be done in accordance with the following procedure:

a) Rinse the nozzle, elbow and the other parts upstream of the filter carefully with water into a storage container, taking care that nothing from the outside of the rinsed parts falls into the container. Depending on the properties of the dust the procedure shall be repeated with acetone into the same container.

b) To rinse the suction tube, seal one end and fill enough water to wet and clean the inner surface and then seal the other end. The tube shall be cleaned by rotating and tilting several times. Transfer the solution to the transport storage container. Depending on the properties of the dust the procedure shall be repeated with acetone into the same container.

No mechanical cleaning shall be applied to recover dust deposits upstream of the filter.

The upstream parts shall be rinsed at least after each measurement series on the same sampling plane and at least once a day. The recovered mass shall be attributed to individual tests in proportion to the mass collected on each filter.

9.7 Field blank

A field blank shall be taken at each measurement site either before or after each measurement series and at least once a day, by the following procedure as described in 9.4 and 9.6:

a) assemble the equipment;

b) disassemble the equipment;

c) rinse, if required.

This leads to an estimation of the dispersion of results for a near zero dust concentration i.e. contamination of filters and of rinsing solutions before and during handling on site, transport, storage, handling in the laboratory and weighing procedures.

The field blank value shall be calculated using the field blank mass divided by the average sample volume of the measurement series.

All field blank values shall be reported individually.

The field blank value shall not exceed 10 % of the emission limit value set for the process or 0,5 mg/m^3, whichever is greater.

10 Calculation

10.1 Sampling volumetric flow rate

In order to perform isokinetic sampling, calculate the required sampling volumetric flow rate, taking into account the velocity of the gas in the duct at the measurement point and the effective diameter of the sampling nozzle.

Because sampling flow rate is measured in conditions (temperature, pressure, humidity) which generally differ from the actual conditions of the gas in the duct. It shall be corrected as follows:

$$Q_m = Q_a \times \frac{100\,\% - h_a}{100\,\% - h_m} \times \frac{T_m}{T_a} \times \frac{p_a}{p_m} \quad (2)$$

where

Q_m is the measured volumetric sampling flow rate at gas meter conditions;

Q_a is the sampling volumetric flow rate, expressed in the actual conditions in the duct;

h_m is the humidity of the gas in measurement conditions, in percentage volume;

h_a is the humidity of the gas in actual conditions, in percentage volume;

T_m is the temperature of the gas in measurement conditions at the volume meter, in Kelvin;

T_a is the temperature of the gas in actual conditions in the duct, in Kelvin;

p_m is the absolute pressure of the gas in measurement conditions at the volume meter;

p_a is the absolute pressure of the gas in actual conditions in the duct.

Compare the Q_a target value to the Q_a performed during measurement, in order to check for isokinetic sampling compliance.

10.2 Dust concentration

For each test, calculate:

a) the sample volume *V*, specifying whether on a dry or wet basis and at standard conditions;

b) the total mass *m* of dust collected upstream of the filter (rinsing) and on the filter;

c) the dust concentration *c*:

$$c = \frac{m}{V} \tag{3}$$

It is sometimes necessary to express dust concentrations to a reference oxygen concentration to correct for dilution effect. Then the measured dust concentration on a dry basis and at standard conditions shall be multiplied by the correction factor f_c:

$$f_c = \frac{21\% - o_{ref}}{21\% - o_m} \tag{4}$$

where

o_{ref} is the oxygen reference concentration in percentage volume of dry gas;

o_m is the oxygen concentration in percentage volume of dry gas, measured in the duct.

11 Measurement report

The measurement report shall provide a comprehensive account of the measurements, a description of the measurement objective and the measurement plan. It shall provide sufficient detail to enable the results to be traced back through the calculations to the collected basic data and process operating conditions.

The measurement report shall include the items specified in ISO 15259 and at least the information on the following items:

a) Identification of the sampling location and gas parameters in the duct:

1) duct dimensions, number and position of measurement lines and measurement points;

2) velocity and temperature at each measurement point;

3) O_2, CO_2, water-vapour content;

4) compliance with the requirements on the gas stream specified in ISO 15259.

b) Measurement procedures:

1) dust measurement:

i) reference to this document (i.e. ISO 12141 :2024);

ii) any modification of the measurement method and corresponding validation data;

2) characteristics of sampling equipment:

i) type of filtration device;

ii) nozzle diameter;

iii) characteristics of the filter (material, sizes, etc.);

iv) filter conditioning and filtration temperature;

c) Test results:

1) number of tests;

2) for each test:

i) date, time and duration;

ii) sampled volume;

iii) dust mass on the filter and dust mass recovered from the rinse solutions;

iv) dust concentration at the conditions specified in the measurement objective.

d) Quality assurance:

1) leak test results;

2) field blank value;

3) compliance with the isokinetic criterion.

Results below the field blank value shall be reported as less than the field blank value.

Annex A
(informative)

Performance characteristics of the method obtained in the method validation

A.1 General

Because waste gas composition varies in time, it is not possible to determine the repeatability and reproducibility of the method in accordance with ISO 5725-2.

However, if one team performs successive parallel sampling tests with two identical sampling systems, such a procedure allows statistical comparison between paired values $x_{i,1}$ and $x_{i,2}$ to be calculated.

The standard deviations s of the paired values are:

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

where n is the number of the paired values

The standard deviation can be used for the calculation of:

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

$$u = t_{0,95;n-1}\, s \tag{A.2}$$

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

— the repeatability r (in accordance with ISO 5725-2), i.e. the maximum difference between i.e. two measurements by the same team, for a 95 % confidence level:

$$r = \sqrt{2}\, t_{0,95;n-1} s \tag{A.3}$$

These data can be used by the test laboratories as tools in the framework of quality assurance.

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

— the expanded uncertainty linked to an individual measurement carried out by any team fulfilling the requirements of this standard. This uncertainty shall be taken into account when comparing the measured values to the emission limit value;

— the reproducibility R (in accordance with ISO 5725-2), i.e. the maximum difference R, which can be expected at a 95 % confidence, between two measurements by different teams working in accordance with this standard, at the process conditions.

When doing measurements at low level concentrations, the detection limit can 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.

A.2 Experimental data

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

— Plant A: semi dry process with a fabric filter, stack gas temperature: 140 °C;

— 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 A.1.

Table A.1 — Results of validation test

	Plant		
	A		B
Number of teams in parallel	4		3
Number of out-stack/in-stack devices	1/3		3/0
Number of successive tests	32		16
	Dust on filter only mg/m³	**Total dust including rinsing** mg/m³	**Total dust** mg/m³
Dust concentration			
mean:	4,7	6,4	2,5
range covered:	2 to 17	3 to 19	0,3 to 6,8
Repeatability	1,7	2,1	1,9
Expanded uncertainty	2,4	4,0	1,8
Reproducibility	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/m³ (dust on filter only);

— for water saturated gases: approximately 2 mg/m³ (total dust).

A.3 Comments

During the above tests, some high field blank values were reported (±1 mg/m³ or higher) due to weighing uncertainties of rinses dry extracts (e.g. use of vessels of improper material).

Further investigation showed that these uncertainties can be reduced to less than 0,5 mg/m³, leading to an improvement of repeatability and reproducibility.

Increased sampling time to 60 min or to 90 min would improve significantly the reproducibility of measurements.

Annex B
(informative)

Influence of the isokinetic rate on the representativeness of the collected particles

Figure B.1 shows the influence of the isokinetic rate v_n/v_d on the representativeness of the collected particles for the following cases:

a) If the isokinetic rate is equal to one, then the gas flow in the nozzle and the gas flow in the duct are identical as shown in the left part of Figure B.1. In this case fine and coarse particles can evenly follow the gas flow and the dust concentration can be correctly measured.

b) If the isokinetic rate is smaller than one, then the gas flow in the nozzle is smaller than the gas flow in the duct, which leads to a disturbed gas flow as shown in the middle of Figure B.1 and a lower sample gas volume. Fine particles can follow the gas flow, whereas coarse particles follow the original flow direction due to the mass inertia. This leads to a higher dust mass on the filter, which is primarily caused by the coarse particles in front the nozzle plane, which do not follow the gas flow out of the nozzle plane. In this case the measured dust concentration is too high.

c) If the isokinetic rate is greater than one, then the gas flow in the nozzle is greater than the gas flow in the duct, which leads to a disturbed gas flow as shown in the right part of Figure B.1 and a higher sample gas volume. As in the previous case, the fine particles can follow the gas flow, whereas the coarse particles follow the original flow direction due to the mass inertia. This leads to a lower dust mass on the filter, which is primarily caused by the coarse particles outside the nozzle plane, which do not follow the gas flow into the nozzle plane. In this case the measured dust concentration is too low.

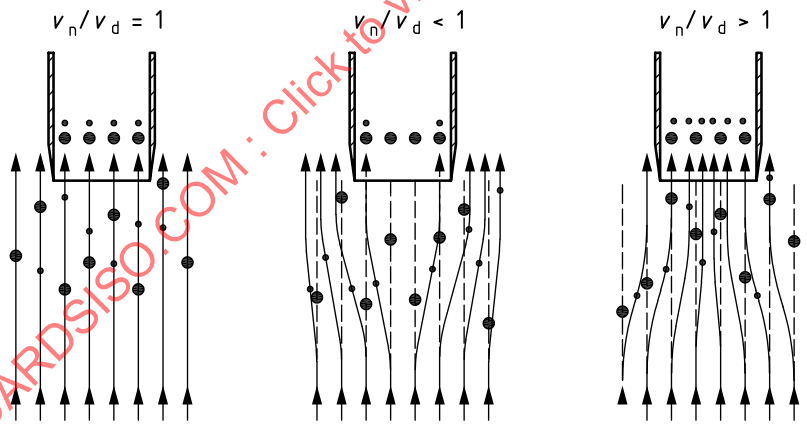


Figure B.1 — Schematic showing the effect of the isokinetic rate v_n/v_d on the representativeness of the collected particles

The collection efficiency of a sampling system for particles can be calculated from the isokinetic rate (see References [1], [2] and [3]). Figure B.2 to Figure B.6 show examples of collection efficiencies as a function of the ratio of the gas velocity in the entry nozzle to the gas velocity in the duct for varying the particle diameter (Figure B.2), the waste gas flow (Figure B.3), the nozzle diameter (Figure B.4), the particle density (Figure B.5) and the waste gas temperature (Figure B.6).

NOTE The collection efficiency is the ratio of the sampled concentration sampled at a specific isokinetic rate to the concentration at isokinetic sampling with an isokinetic rate of 1,0.

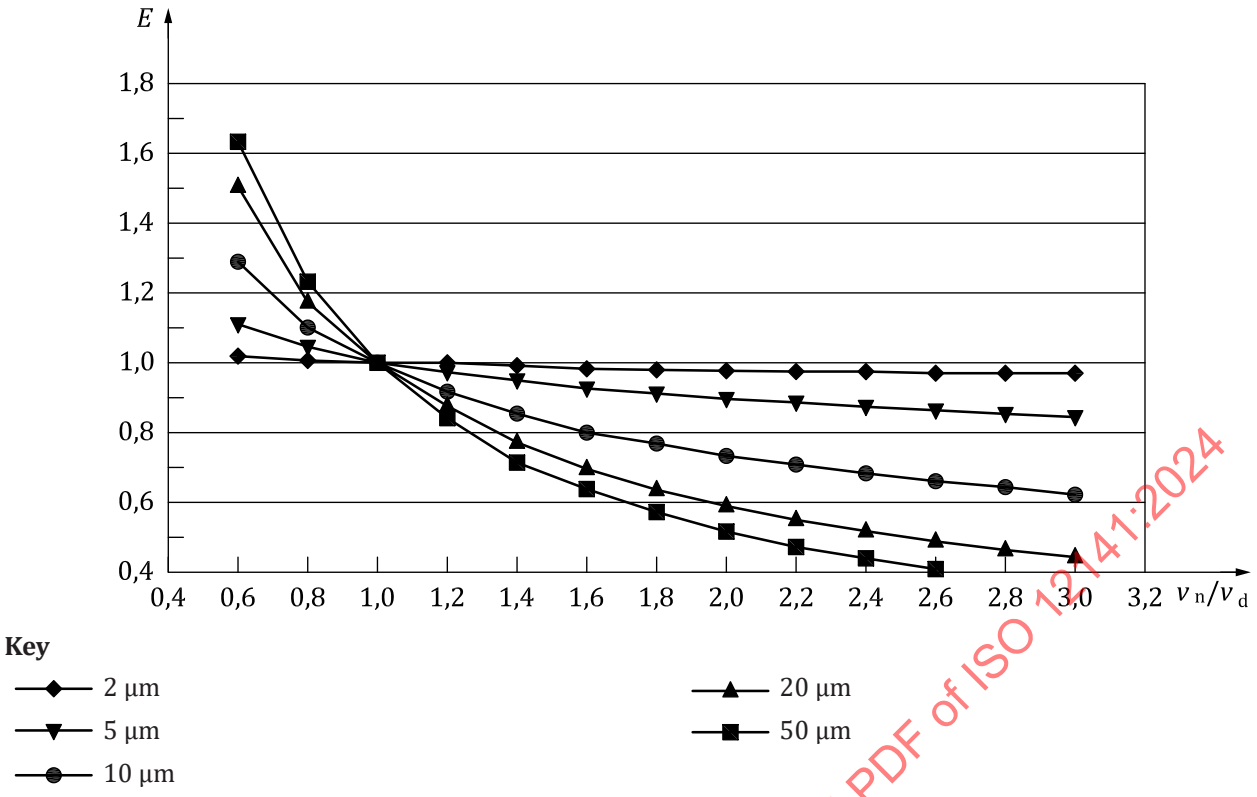


Figure B.2 — Theoretical dependence of the collection efficiency E on the ratio of the gas velocity in the entry nozzle v_n to the gas velocity v_d in the duct for different particle diameters and a waste gas velocity of 10 m/s, a nozzle diameter of 10 mm, a particle density of 1 000 kg/m^3 and a waste gas temperature of 0 °C

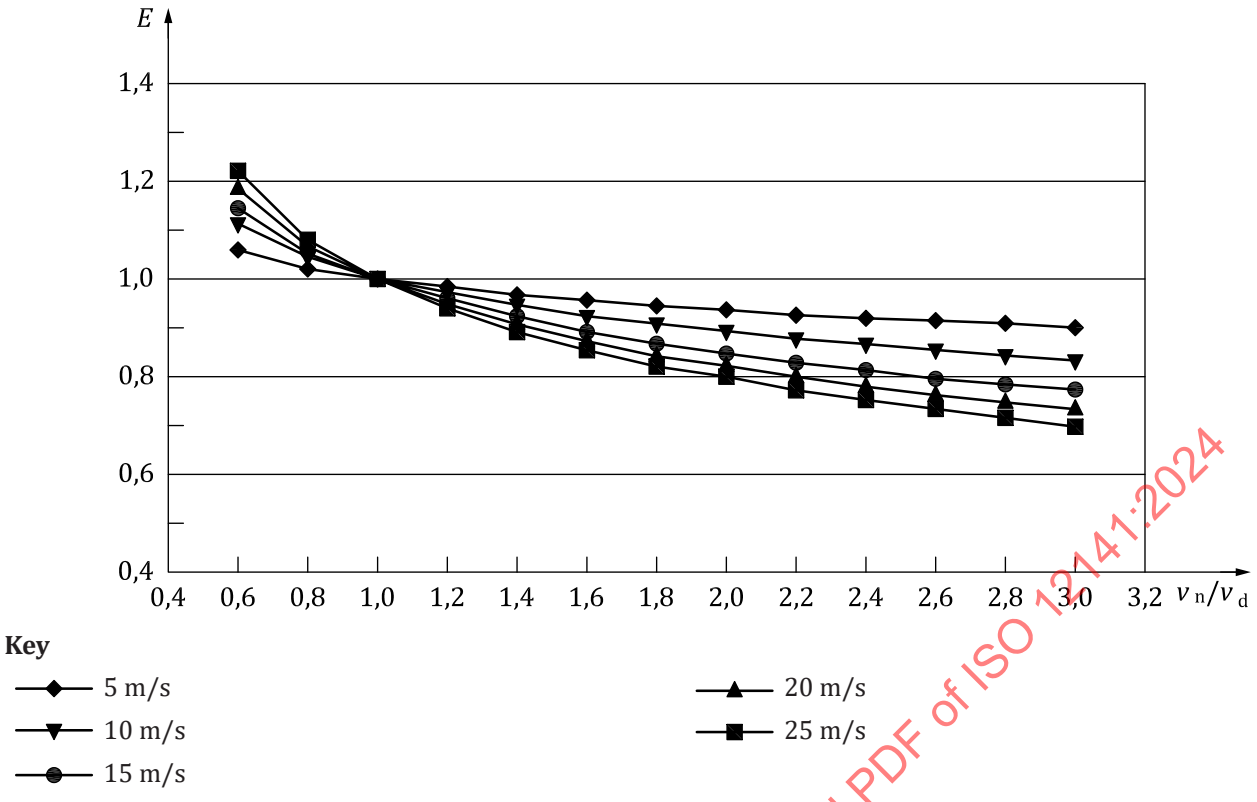


Figure B.3 — Theoretical dependence of the collection efficiency E on the ratio of the gas velocity in the entry nozzle v_n to the gas velocity v_d in the duct for different waste gas velocities and a particle diameter of 5 µm, a nozzle diameter of 10 mm, a particle density of 1 000 kg/m^3 and a waste gas temperature of 0 °C

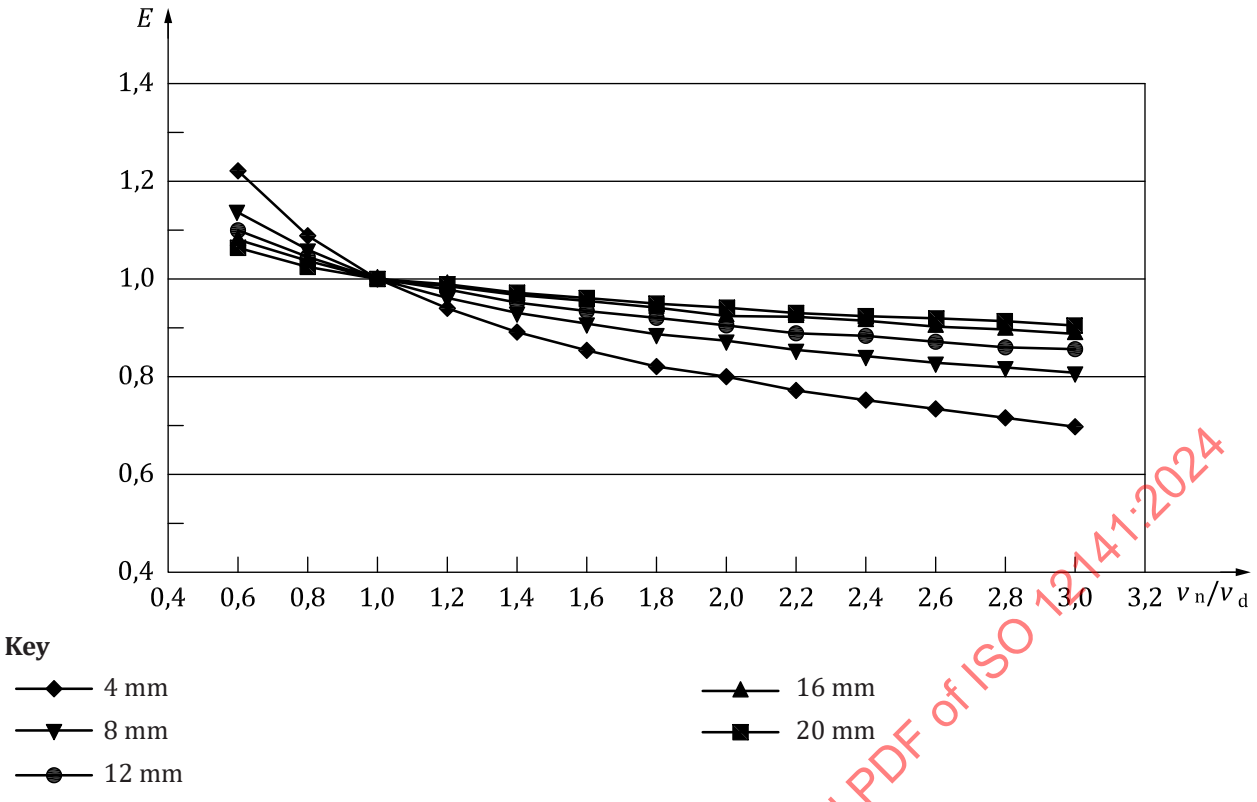


Figure B.4 — Theoretical dependence of the collection efficiency E on the ratio of the gas velocity in the entry nozzle v_n to the gas velocity v_d in the duct for different nozzle diameters and a particle diameter of 5 µm, a waste gas velocity of 10 m/s, a particle density of 1 000 kg/m^3 and a waste gas temperature of 0 °C

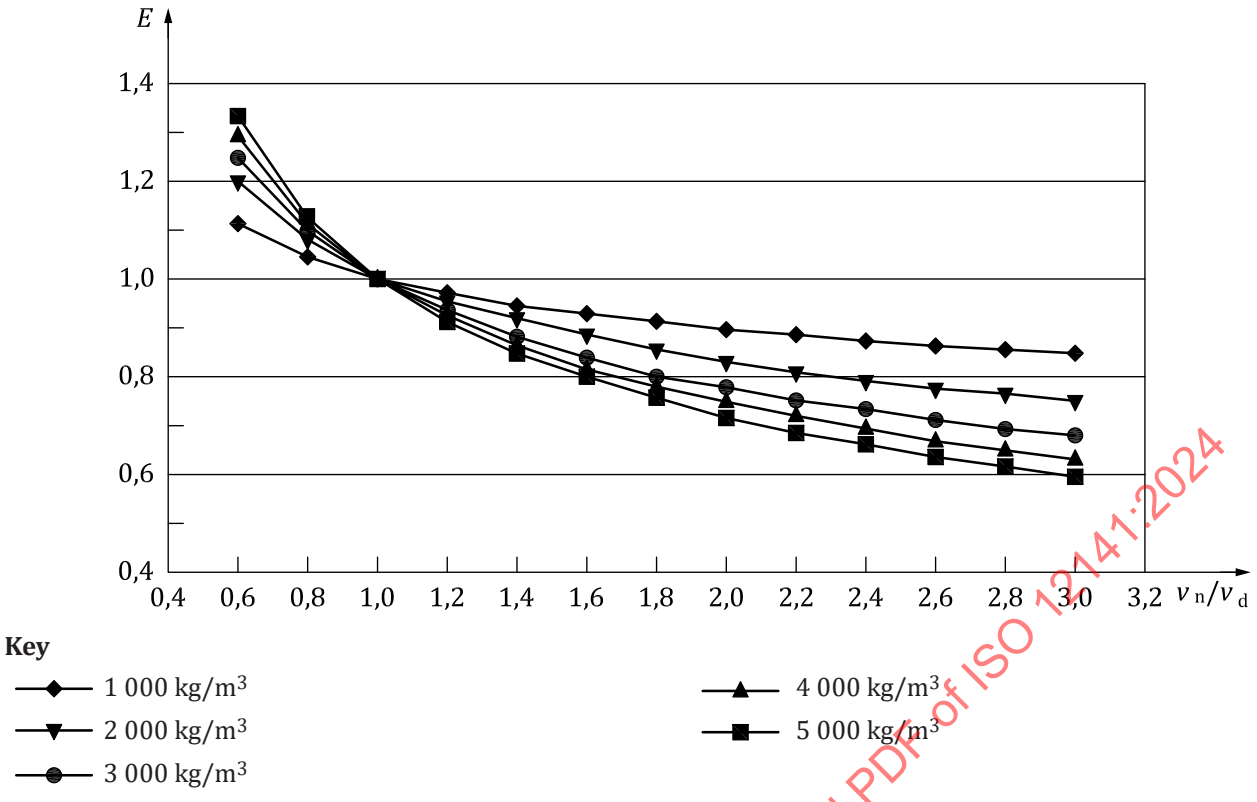


Figure B.5 — Theoretical dependence of the collection efficiency E on the ratio of the gas velocity in the entry nozzle v_n to the gas velocity v_d in the duct for different particle densities and a particle diameter of 5 µm, a waste gas velocity of 10 m/s, a nozzle diameter of 10 mm and a waste gas temperature of 0 °C

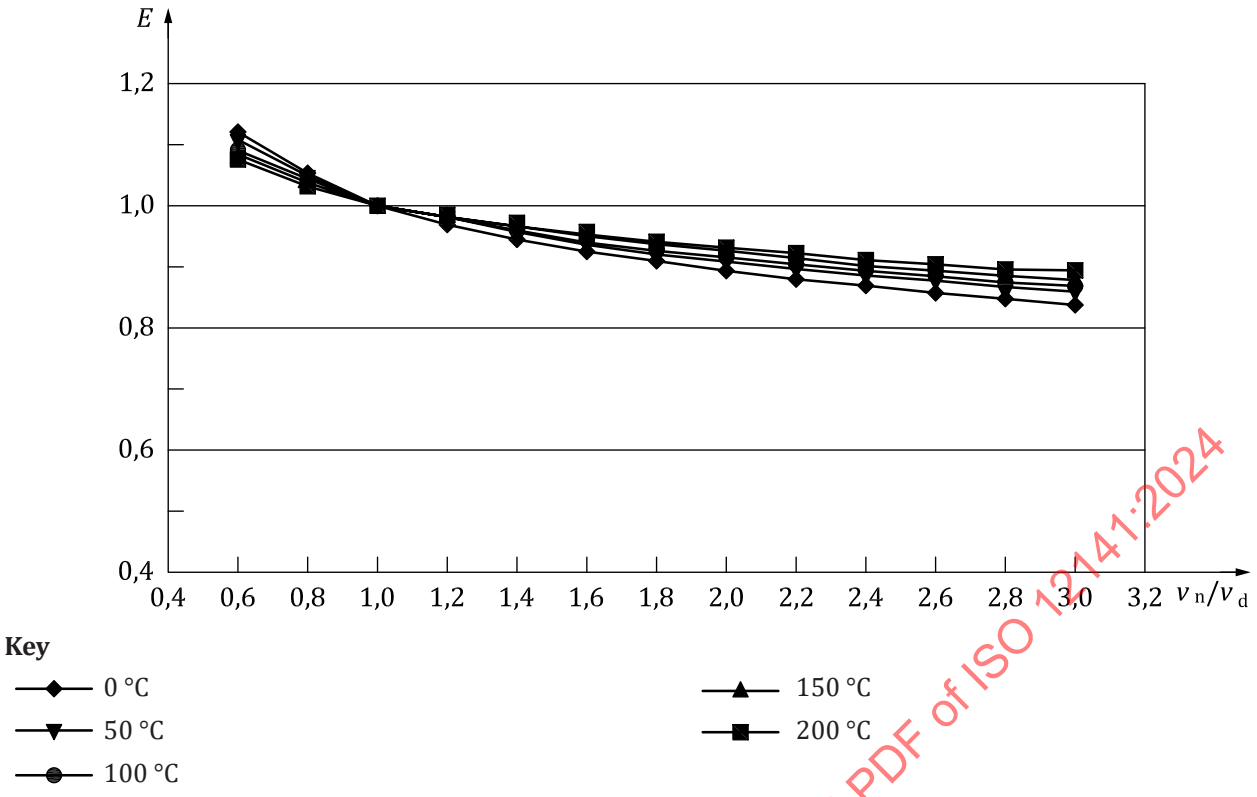


Figure B.6 — Theoretical dependence of the collection efficiency E on the ratio of the gas velocity in the entry nozzle v_n to the gas velocity v_d in the duct for different waste gas temperatures and a particle diameter of 5 µm, a waste gas velocity of 10 m/s, a nozzle diameter of 10 mm and a particle density of 1 000 kg/m^3

Annex C
(informative)

Proven design of the entry nozzles

Figures C.1, C.2 and C.3 show three designs of the entry nozzles, where:

e is the thickness of the side of the nozzle;

d_n is the internal diameter of the entry nozzle;

R is the radius of curvature of the nozzle entry edge;

L is the length with constant internal diameter.

a) Figure C.1:

1) $e < d_n/12$ but at least 0,8 mm;

2) $L \geq 10$ mm;

3) $R \leq 0{,}2$ mm.

Effective diameter $d_{eff} = d_n + 2R$

Dimensions in millimetres

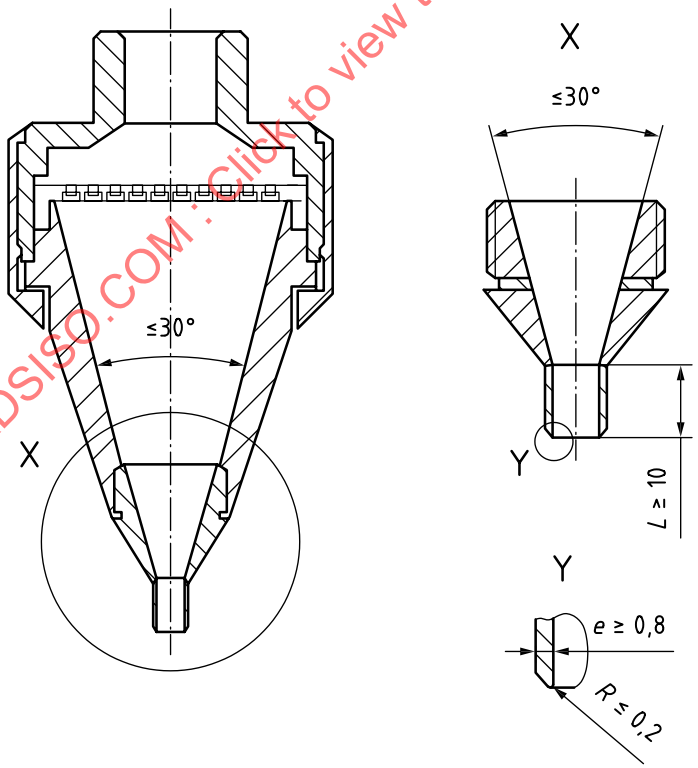


Figure C.1 — Example of in-stack filter housing with proven integral nozzle

b) Figure C.2:

$e \leq 0{,}2$ mm.

Effective diameter $d_{eff} = d_n + e$

Dimensions in millimetres

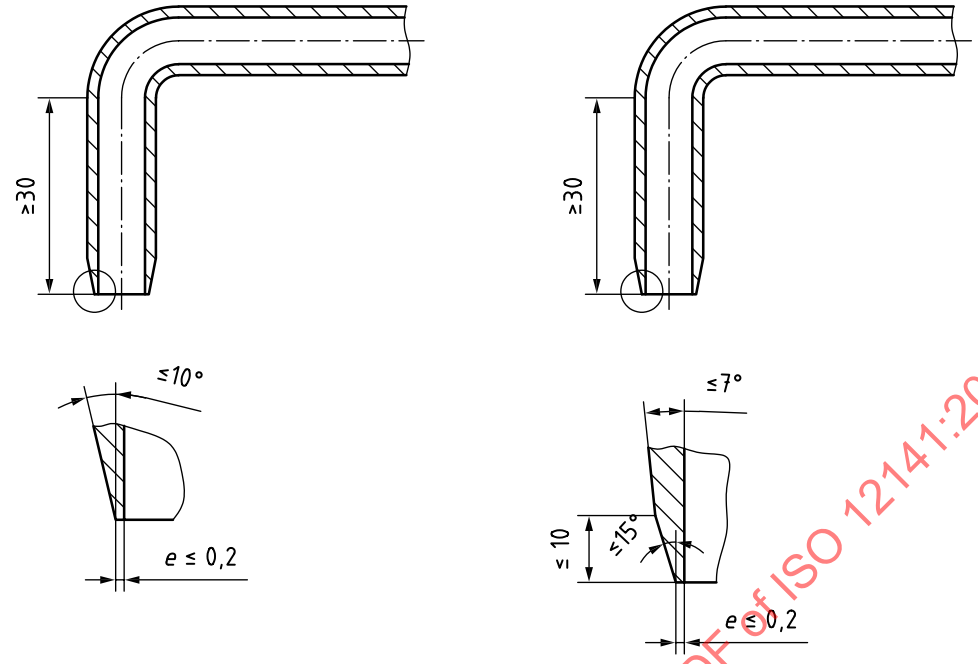
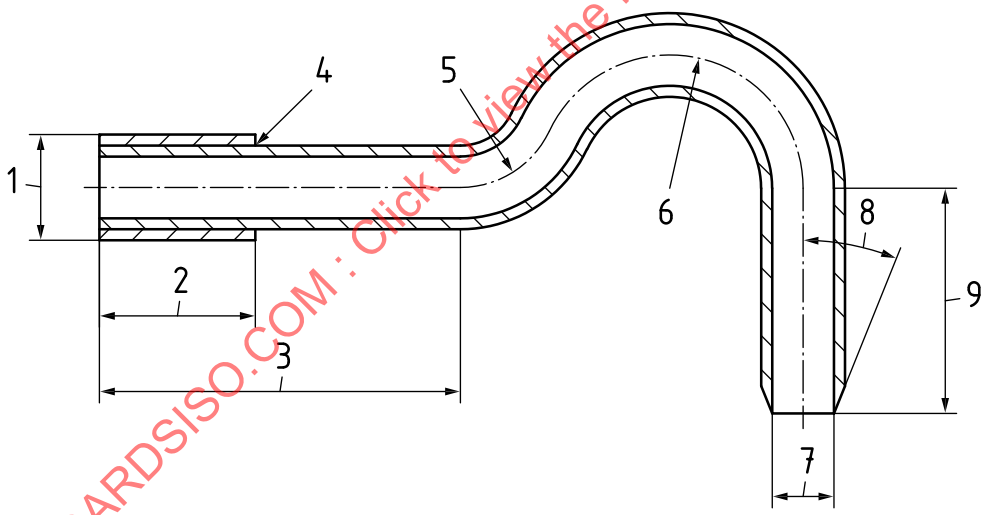


Figure C.2 — Example of proven entry nozzles



Key

1 16 mm
2 25 mm
3 60 mm
4 size to suit nozzle tube
5 15 mm radius
6 20 mm radius
7 nozzle diameter
8 20°
9 35 mm

Figure C.3 — Example of proven goose neck nozzle

Annex D
(informative)

Summary of the requirements

Equipment	Value	Specified in
Nozzle:		
— uncertainty of area at nozzle entry	≤ 5 %	7.2.3
— length with constant internal diameter	≥ 10 mm	7.2.3
— change in diameter angle	≤ 30°	7.2.3
— radius of the bend	≥ 1,5 times internal diameter of the bend	7.2.3
— straight length before the first bend	≥ 30 mm	7.2.3
— distance to obstacles	≥ 50 mm	7.2.3
Filter:		
— efficiency on test aerosol of 0,3 µm	> 99,5 %	7.2.2.3
— efficiency on test aerosol of 0,6 µm	> 99,9 %	7.2.2.3
— material	no reaction and no absorption of the components	7.2.2.3
Sample gas:		
— maximum expanded uncertainty of gas volume	≤ 5,0 % of measured value	7.2.6
— maximum expanded uncertainty of absolute pressure	≤ 2,0 % of measured value	7.2.6
— maximum expanded uncertainty of absolute temperature	≤ 2,0 % of measured value	7.2.6
Angle of the nozzle with regard to gas flow	≤ 10°	9.5
Isokinetic rate	95 % to 115 %	9.5
Leak rate	≤ 2,0 %	9.4
Balance: resolution	0,01 mg to 0,1 mg	7.4
Pre- and post-sampling treatment of weighed parts: temperature equilibrium duration	4 h to 12 h	8.2, 8.4
Field blank value	≤ 10 % of the ELV[a] or 0,5 mg/m^3, whichever is greatest	9.7
Expanded uncertainty	≤ 20 % of the ELV[a]	Clause 5
Sampling location	**Value**	**Specified in**
Duct gas flow:		
— angle with regard to duct axis	< 15°	ISO 15259
— negative velocity	not permitted	ISO 15259
— differential pressure at Pitot tube	> 5 Pa	ISO 15259
— ratio of maximum to minimum velocity	< 3:1	ISO 15259
Straight length before the measurement plane	> 5 hydraulic diameters (recommended)	ISO 15259
Straight length after the measurement plane	> 2 hydraulic diameters (recommended)	ISO 15259
Straight length before emission point	> 5 hydraulic diameters (recommended)	ISO 15259
Number of sampling points	according to ISO 15259	ISO 15259
Waste gas characteristics	**Value**	**Specified in**
Waste gas density: uncertainty	≤ 0,05 kg/m^3	ISO 16911-1

[a] ELV set for the process.

Annex E
(normative)

Sampling volume, flow rate and duration

E.1 General

The minimum gas volume to be sampled shall be derived from the uncertainties in dust weighing and the emission limit value set for the process.

E.2 Weighing uncertainties

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

E.3 Sampling volume

For an emission limit value E the dust mass m shall be at least 10 times the expanded uncertainty U_w of weighing (see 9.7).

The necessary minimum sampling volume V_{min} is then determined by Formula (E.1):

$$V_{min} = \frac{m}{E} = \frac{10 U_w}{E} \quad \text{(E.1)}$$

E.4 Sampling flow rate and duration

When the sampling duration t_s is limited (e.g. 30 min), the minimum sampling flow rate Q_{min} is determined by Formula (E.2):

$$Q_{min} = \frac{V_{min}}{t_s} \quad \text{(E.2)}$$

The sampling flow rate Q_{min} is compared to the practical flow rate which can be achieved by the used sampling equipment (e.g. filter pressure drop, pump characteristics).

Annex F
(informative)

Examples of weighing bias

F.1 General

Weighing bias related to insufficient temperature equilibrium, and to climatic changes between pre- and post-sampling weighing, 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 (density 8 g/ml). Density of glass 2 g/ml, of air 1,2 mg/ml.

F.2 Effect of insufficient temperature equilibrium

Because of too low equilibrium time after drying, the inside air of the Petri box is assumed to have a temperature 2 K higher than that of the balance room (300 K). The difference of air temperature leads to an apparent mass variation of:

$$40\,\text{ml} \times 1{,}2\,\frac{\text{mg}}{\text{ml}} \times \frac{2\,\text{K}}{300\,\text{K}} = 0{,}3\,\text{mg} \qquad \text{(F.1)}$$

F.3 Effect of temperature variations

The balance room temperature is 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 of (10 K) compared to the temperature of the balance room (300 K) this air volume leads to an apparent weight modification of:

$$9{,}4\,\text{ml} \times 1{,}2\,\frac{\text{mg}}{\text{ml}} \times \frac{10\,\text{K}}{300\,\text{K}} = 0{,}4\,\text{mg} \qquad \text{(F.2)}$$

F.4 Effect of barometric pressure variations

The barometric pressure is assumed to be:

a) when weighing before sampling 98,5 kPa;

b) when weighing after sampling 104 kPa.

Therefore, a relative pressure variation of 5,5 % is observed.

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

$$9{,}4\,\text{ml} \times 1{,}2\,\frac{\text{mg}}{\text{ml}} \times 0{,}055 = 0{,}6\,\text{mg} \qquad \text{(F.3)}$$

F.5 Conclusions

When weighing parts with large internal volume, it is mandatory, that the temperature equilibrium has been reached before weighing.

There is no need for correction of temperature effects if the room where the balance is situated is thermally controlled. But it remains necessary that the effect of barometric pressure variations will be taken into account, especially if the density 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 8.3.

Annex G
(informative)

Determination of the measurement uncertainty

G.1 General

This annex provides an example on how to determine the measurement uncertainty of dust emission measurements by analysis of the individual steps of the measurement method, consisting of three parts:

— principle of the determination of the uncertainty contributions of the individual measurands (see Clause G.2);

— combination of the uncertainty contributions of the individual measurands (see Clause G.3);

— calculation of the effective number of degrees of freedom (see Clause G.4);

G.2 Principle of the determination of the uncertainty contributions of measurands

G.2.1 General

In the following, the principle of the calculation of the uncertainties of individual measurands is explained. Uncertainty contributions of measuring devices, as caused by e.g. calibration, signal drift or resolution, and uncertainty contributions to be determined by experiments are taken into account. In G.2.2 the mass m is considered to be the measurand.

G.2.2 Uncertainty contribution of calibration

Calibration means the comparison of a measured value obtained by measuring a certified reference with the nominal value of this reference. During weighing, for example, the value measured when putting a reference weight on the balance is compared with the nominal value according to its gauging certificate. If a sufficient number of individual results of repeated calibration measurements are available, the uncertainty of a measurement (e.g. mass determination) due to the calibration is calculated according to Formula (G.1):

$$u_{\mathrm{cal}}^2(m) = \frac{\sum_{i=1}^{n}(m_i - m_{\mathrm{ref}})^2}{n} + u^2(m_{\mathrm{ref}}) = s^2(m)\cdot\left(1-\frac{1}{n}\right) + u_{\mathrm{b}}^2 + u^2(m_{\mathrm{ref}}) \qquad \text{(G.1)}$$

where

$u_{\mathrm{cal}}(m)$ is the experimentally determined uncertainty of the measurand m due to calibration, e.g. by weighing a reference weight;

m_i is the ith measured value with i = 1 to n;

m_{ref} is the nominal value of the reference weight;

$s(m)$ is the repeatability standard deviation of the measured values m_i;

u_{b} is the bias (deviation between the mean value of the measurement series and the nominal value of the reference, e.g. determined during weighing on the basis of the readings of the balance for repeated placing of the reference weight and the nominal value of the reference weight);

$u(m_{ref})$ is the uncertainty of the nominal value of the reference, e.g. uncertainty of the reference weight according to the gauging certificate;

n is the number of repeated measurements, e.g. weighings;

with

$$s^2(m) = \frac{\sum_{i=1}^{n}(m_i - \bar{m})^2}{n-1} \quad \text{(G.2)}$$

where $\bar{m}$ is the mean value of the measured values m_i.

The repeatability standard deviation can be estimated by repeating the measurement sufficiently often ($n > 5$).

NOTE 1 Formula (G.1), for example, allows for the bias between the experimentally obtained value for the mass of the reference weight and the value obtained during gauging, as well as for the uncertainty of the reference weight.

If the individual measured values m_i are not available to the user, but only information on the measurement uncertainty U_{cal} of the calibrated measuring system, this information may be considered as expanded uncertainty on the basis of a confidence interval of 95 %. In this case, the uncertainty $u_{cal}(m)$ shall be estimated approximately as follows:

$$u_{cal}(m) = \frac{U_{cal}}{2} \quad \text{(G.3)}$$

NOTE 2 This relates to a conservative estimation. Assuming a confidence interval of 99 % instead of 95 % would result in smaller variance and uncertainty.

G.2.3 Uncertainty contribution of the drift

In general, measurement signals are not long term stable, but slightly change with time. This systematic deviation is also called drift. It can be assumed to be uniformly distributed and thus be estimated on the basis of a rectangular distribution according to Formula (G.4):

$$u_{drift}(m) = \frac{a_{drift}}{\sqrt{3}} \quad \text{(G.4)}$$

Parameter a_{drift} is the drift specified for the measuring device or the determined maximum drift between two adjustments.

G.2.4 Uncertainty contribution of the display resolution

Analogue and digital displays contribute to measurement uncertainty. The resolution of analogue displays is limited by the graduation. Digital displays are rounded or truncated from a certain digit. If the space between two graduation lines of an analogue display or the smallest possible step of a digital display is designated as a_{res}, the true value is evenly distributed in the interval $\pm\, a_{res}/2$ around the displayed value. Thus, according to Formula (G.5), the uncertainty contribution due to the resolution is:

$$u_{res}(m) = \frac{a_{res}}{2\sqrt{3}} \quad \text{(G.5)}$$

If the measured value is the difference between an initial value and a final value, or the difference between a zero value and a final value, the uncertainty contribution of the resolution is considered twice in the calculation.

G.3 Combination of the uncertainty contributions of the individual measurands

G.3.1 Procedure

In the following, the application of the indirect approach to determine the uncertainty of the results of a dust measurement method is explained. This approach makes it necessary to include each single step that affects the measurement result in the evaluation of uncertainty, i.e. the measurement and analysis procedure shall be thoroughly analysed.

The calculation of the measurement uncertainties is based on variances. In the simplest case, the variance of a result is calculated by adding the variances of the different influence quantities taking into account the sensitivity coefficients of these influence quantities. They can be derived from the method model equation which describes the dependence of the measurement result on the input quantities (e.g. mass, pressure, volume, temperature).

The relevance of the sensitivity coefficients becomes clear when expressing e.g. the gas volume of a sample, measured at a higher temperature, at the standard temperature of 0 °C. This is common for the results of emission measurements and can be expressed by Formula (G.6):

$$V_0 = V_m \cdot \frac{273\,\mathrm{K}}{273\,\mathrm{K} + t_m} \tag{G.6}$$

where

V_0 is the sample volume at 0 °C;

V_m is the sample volume measured at temperature t_m;

t_m is the temperature at the gas meter, in °C.

The measurement uncertainty of volume V_m has a much greater effect on the normalized volume V_0 than the measurement uncertainty of temperature t_m. As can be seen in Formula (G.6) the absolute temperature with the constant additional factor 273 K, the measurement uncertainty of which may be neglected in this context and which is high compared to the measured temperature t_m including its measurement uncertainty, is used for the calculation of the normalized volume V_0. For a volume V_M = 100 ℓ and a temperature t_m = 30 °C, the calculation shows that the variance of V_0 is affected by V_M with a factor of about 0,8. The factor of the temperature effect is only about 0,09. This example illustrates the necessity for determining and considering sensitivity coefficients.

The sensitivity coefficients are calculated by partial differentiation of the model equation. Thus, factors are obtained, either directly or by inserting the numerical measurement results into the mathematical terms, which result from the partial differentiation. These are considered quadratically in the calculation of the variance of the measurement result. The variance equation for model Formula (G.6) is given by Formula (G.7):

$$\mathrm{var}(V_0) = e^2(V_m) \cdot \mathrm{var}(V_m) + e^2(t_m) \cdot \mathrm{var}(t_m) \tag{G.7}$$

The following example of the determination of the uncertainty of the measurement of low dust contents at stationary sources involves different steps in accordance with the previous explanations. At first, the variances of the individual influence quantities are calculated. These are:

— dust mass;

— gas volume;

— temperature at the gas meter;

— pressure at the gas meter;

— atmospheric pressure;

— oxygen content of the exhaust gas for conversion with respect to the reference oxygen content.

The tolerances are assumed to originate from different sources, e.g. from calibration, from the experimentally determined drift, or from the manufacturer's specifications. To calculate the standard deviations and variances from these tolerances, Type B evaluations are also used here. A rectangular distribution of the values is assumed which serves as basis for the calculation of the respective variances. In the example, the uncertainties of the following influence quantities are taken into account:

— calibration;

— drift;

— display resolution.

From the variances of these influence quantities, the total variance of the individual parameters is calculated by addition, such as the variance of the measured sample volume, the temperature and the pressure at the gas meter, and of the atmospheric pressure.

The calculation of the dust concentration in dependence on the measurands sample volume at the gas meter, temperature at the gas meter, pressure at the gas meter, atmospheric pressure, weighted dust mass and reference oxygen content for normalization can be described with *one* equation and can therefore also be mathematically treated in one step. In order to elucidate the calculation process, the following single steps are dealt with separately in this example:

— calculation of the standard volume;

— calculation of the dust mass difference of pre- and post-weighing;

— calculation of the dust concentration;

— calculation of the dust concentration for oxygen reference conditions.

Each of these calculations follows the same scheme. In the first step, the model equation is set up. In the second step, the partial differentials are formed for all measurands contained in this model equation and the sensitivity coefficients are determined.

Using the so obtained sensitivity coefficients and the variances of the individual measurands, the variance of the measurement result is calculated in the third step.

G.3.2 Specification of the method model equation

Measurand of the dust measurement is the mass concentration c_{s,O_2} of the dust at standard conditions and for the reference oxygen content. The method model equation considers K = 7 input quantities:

$$c_{s,O_2} = f(m_1, m_2, V_m, p_{atm}, \Delta p, t_m, o_m) \quad \text{(G.8)}$$

where

m_1 is the mass of the empty filter (before sampling);

m_2 is the mass of the dust loaded filter (after sampling);

p_{atm} is the atmospheric pressure (ambient pressure);

Δp is the relative pressure (usually low pressure), measured at the gas meter;

V_m is the sample volume;

t_m is the temperature at the gas meter, in °C;

o_m is the oxygen content by volume.

The method equation for the measurement result as a function of the identified input quantities is:

$$c_{s,O_2} = \frac{1013\,\text{hPa}}{273\,\text{K}} \cdot \frac{(273\,\text{K} + t_m)}{(p_{atm} + \Delta p)} \cdot \frac{(21\% - o_{ref})}{(21\% - o_m)} \cdot \frac{(m_2 - m_1)}{V_m} \quad \text{(G.9)}$$

where o_{ref} is the reference oxygen content by volume.

In Formula (G.9), the reference values for pressure (1 013 hPa) and temperature (273 K) are rounded as usual.

The temperature t_m obtained at the gas meter can be converted into the absolute temperature T_m = 273 K + t_m. The measurement uncertainty of the constant 273 K is considered negligible in this example. The measurement uncertainty of the measured temperature t_m is then equal to the measurement uncertainty of the absolute temperature T_m.

G.3.3 Stepwise calculation of the individual uncertainty contributions

G.3.3.1 Determination of variances for the individual measurands

In the following, the variances of the individual measurands (sample volume, temperature at the gas meter, pressure at the gas meter, atmospheric pressure) are estimated. Mainly instrument specifications are used that are known or determined as tolerances or measured values for the different uncertainty contributions. The following uncertainty contributions are concerned:

— calibration;

— drift;

— display resolution.

Variances can also be calculated if it is only known that the measured value lies within a known range about the true value (desired value). This is e.g. the case when gauged systems with given gauging error limits are used. The variance is then calculated on the assumption of an equal distribution (rectangular distribution). The assumption of such a rectangular distribution means a conservative uncertainty estimate.

G.3.3.2 Determination of variance of weighing with a test weight as reference

For weighing, gauged mass standards are used as reference. For these, the responsible gauging authority specifies, during gauging, the deviation from the nominal mass as well as a standard deviation as measurement uncertainty. It is assumed that this is a standard deviation on the basis of a normal distribution. From this data and several repeated weighings the uncertainty of the weighing operation can be calculated according to Formula (G.10):

$$u^2(m_1) = s^2(m) \cdot \left(1 + \frac{1}{n}\right) + u_b^2 + u^2(m_{ref}) \quad \text{(G.10)}$$

where

$u(m_1)$ is the uncertainty of the weighing operation;

$s(m)$ is the standard deviation of the measured values m_i of the comparison weighings using the reference mass standard;

u_b is the bias (deviation between the mean value of the measurement series, determined on the basis of the readings of the balance in the case of repeated placing of the reference weight, and the nominal value of the reference weight);

$u(m_{ref})$ is the uncertainty of the reference weight;

n is the number of weighings.

Since the uncertainty contributions of the bias and of the reference weight are generally small compared to the total uncertainty $u(m_1)$, the number of degrees of freedom during weighing equals $n - 1$.

G.3.3.3 Determination of the variance of the dust loading

The model equation for determining the dust mass by weighing includes the two steps pre-weighing and post-weighing. Both steps involve measurement uncertainty:

$$m = m_2 - m_1 \tag{G.11}$$

where

m is the mass of the collected dust;

m_1 is the mass of the empty filter (pre-weighing);

m_2 mass of the loaded filter (post-weighing).

The variance of m is calculated according to Formula (G.12):

$$\mathrm{var}(m) = e^2(m_2) \cdot \mathrm{var}(m_2) + e^2(m_1) \cdot \mathrm{var}(m_1) \tag{G.12}$$

where

$e(m_1)$ is the sensitivity coefficient of pre-weighing;

$e(m_2)$ is the sensitivity coefficient of post-weighing;

$\mathrm{var}(m_1)$ is the variance of pre-weighing;

$\mathrm{var}(m_2)$ is the variance of post-weighing.

The sensitivity coefficients are derived from Formula (G.13) and Formula (G.14):

$$e(m_2) = \frac{\partial m}{\partial m_2} = \frac{\partial}{\partial m_2}(m_2 - m_1) = 1 \tag{G.13}$$

$$e(m_1) = \frac{\partial m}{\partial m_1} = \frac{\partial}{\partial m_1}(m_2 - m_1) = -1 \tag{G.14}$$

Inserting the sensitivity coefficients into Formula (G.12) provides Formula (G.15):

$$\mathrm{var}(m) = e^2(m_2) \cdot \mathrm{var}(m_2) + e^2(m_1) \cdot \mathrm{var}(m_1) = 1^2 \cdot \mathrm{var}(m_2) + (-1)^2 \cdot \mathrm{var}(m_1) = \mathrm{var}(m_2) + \mathrm{var}(m_1) \tag{G.15}$$

If the mass m of collected dust is small compared to the filter mass m_1 (which is usually the case when weighing the filter holder as well), the measurement uncertainties for pre- and post-weighing can be considered to be equal:

$$\mathrm{var}(m_2) = \mathrm{var}(m_1) \tag{G.16}$$

The absolute measurement uncertainty $u(m)$ and the relative measurement uncertainty $w(m)$ of the collected dust are calculated as follows:

$$u(m) = \sqrt{\mathrm{var}(m)} = \sqrt{2 \cdot \mathrm{var}(m_1)} \tag{G.17}$$