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**Stationary source emissions — Test  
method for determining PM<sub>2,5</sub> and  
PM<sub>10</sub> mass in stack gases using  
cyclone samplers and sample dilution**

*Émissions de sources fixes — Méthode d'essai pour la détermination  
de la concentration en masse de PM 2,5 et PM 10 dans les gaz émis à  
la cheminée en utilisant des échantillonneurs cyclone et une dilution  
d'échantillon*



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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](http://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](http://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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 146, *Air quality*, Subcommittee SC 1, *Stationary source emissions*.

This corrected version of ISO 25597:2013 incorporates the following corrections.

- Clause 4: Corrected the units for  $v_s$ , the average stack gas velocity. Deleted the row for “ $v_s$  velocity of the stack gas”.
- 6.3.2 (last paragraph): Changed 10:1 to 20:1.
- 9.4.4.5: Changed “See C.4” to “See [Annex D](#)”.
- 9.4.5: Changed “See C.5” to “See E.5”.
- 9.8 (last paragraph): Changed “sample container No. 5” to “sample container No. 4d”.
- 13.1: Corrected the reference from (ISO 25574:2013) to (ISO 25597:2013).
- C.1: In the first paragraph, changed “use Formula (B.1)” to “use Formula (C.1)”. In Formula (C.1), changed the value of the numerator (212,2 replaces 3,056).
- C.2.3: In the note, changed “ $\times 1,5$ ” to “ $\times R_{\max}$ ”.
- E.5.2: Deleted Formula (E.11) and renumbered accordingly.
- E.5.3: Removed “condensable” in the title and in the first line.
- E.5.3: Corrected Formulae (E.12) and (E.13) [formerly called Formulae (E.13) and (E.14)], and the explanations of  $N$  and  $D_F$  in the underlying where clause.
- E.5.4: Corrected Formulae (E.14) and (E.15) [formerly called Formulae (E.15) and (E.16)].

## Introduction

This International Standard provides conventions for the sampling and analysis of stack gas samples from stationary sources by the use of cyclone samplers to separate particulate matter with nominal aerodynamic diameters of 10  $\mu\text{m}$  ( $\text{PM}_{10}$ ) and 2,5  $\mu\text{m}$  ( $\text{PM}_{2,5}$ ).

This International Standard provides design guidelines for:

- the use of sampling cyclones, for the measurement of filterable particles;
- the measurement of filterable and condensable particles using the dilution sampling technique.

The dilution sampling technique allows for the capture and measurement of condensable, secondary particulate matter that is similar in characteristics to materials formed when a flue gas exhaust mixes with ambient air. The method is suited for obtaining particulate speciation data useful in local and regional source apportionment studies and health risk assessment studies.

This International Standard provides for the use of two types of sampling train:

- a) a basic sampling train to measure filterable particles using sampling cyclones that can distinguish between particle sizes in the range of 2,5  $\mu\text{m}$  and 10  $\mu\text{m}$ ;
- b) a dilution sampling train that uses in-stack sampling cyclones to measure filterable in particles in the same manner as the basic sampling train as in a), but measures also, condensable particles with additional  $\text{PM}_{2,5}$  and/or  $\text{PM}_{10}$  cyclones located after the dilution chamber in the sampling train.

The method using dilution sampling for the formation, collection, and analysis of condensable particulate matter allows for capture of secondary particulate matter that is similar in character to ambient particulate matter. The method is suitable for the collection of source emission data for local and regional source apportionment studies. Particulate speciation data may also be gathered using dilution sampling to provide data for health risk assessment studies.





# Stationary source emissions — Test method for determining PM<sub>2,5</sub> and PM<sub>10</sub> mass in stack gases using cyclone samplers and sample dilution

**WARNING** — This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

Collection of emission samples may require working on stack platforms and other elevated surfaces. Protective clothing (heavy long sleeves and pants, hard hats, and eye protection) should be worn at all times while working in the vicinity of hot surfaces. Insulated gloves should be worn when handling hot probes or accessing stack sampling locations with hot surfaces. Hearing protection should be worn if needed.

Equipment cleaning requires the use of acetone. This work should be performed in a well-ventilated area to minimize fire and worker exposure hazards.

## 1 Scope

### 1.1 General

This International Standard specifies procedures for the extraction and measurement of filterable particulate matter from stationary source flue gas samples by:

- the use of cyclone samplers;
- the measurement of condensed particulate matter using dilution sampling technique, which simulates the interaction of stack gas components with the atmosphere as they mix after the stack exit.

This International Standard provides for the use of two types of sampling train.

- Basic sampling train, a basic sampling train to measure filterable particles using sampling cyclones that can distinguish between particle sizes in the range of 2,5  $\mu\text{m}$  and 10  $\mu\text{m}$ . This method is especially suitable for measurements of particle mass concentrations above 50  $\text{mg}/\text{m}^3$  as a half-hourly average at standard conditions (293 K, 1 013 hPa, dry gas) and applies to primary particulate matter (PM) emissions equal to or less than an aerodynamic diameter of nominally 10  $\mu\text{m}$  (PM<sub>10</sub>) from stacks or ducts.
- Dilution sampling train, a dilution sampling train that utilizes a dilution chamber that mixes flue gas with conditioned dilution air to simulate the interaction of the stack gas components with ambient air. This simulation process may lead to the condensation of particulate matter that might not otherwise be produced in the basic sampling train. The dilution sampling train uses in-stack sampling cyclones to measure filterable particles in the same manner as the basic sampling train, but in addition, utilizes additional PM<sub>2,5</sub> and/or PM<sub>10</sub> cyclones in the sampling train to measure particles formed in the dilution chamber.

This method is intended for the measurement of mass concentrations of particles smaller than 2,5  $\mu\text{m}$  aerodynamic diameter (PM<sub>2,5</sub>) using weighing techniques. The method can be used to measure mass concentrations of particles with aerodynamic diameter smaller than 10  $\mu\text{m}$  aerodynamic diameter (PM<sub>10</sub>) or particles with aerodynamic diameters between 2,5  $\mu\text{m}$  and 10  $\mu\text{m}$ .

In this method, the dilution sampling train can be used in combination with the basic sampling train, using PM<sub>10</sub> and/or PM<sub>2,5</sub> depending upon the test objectives. The dilution sampling system is intended

for applications where measurement is required of particles similar in characteristics to materials formed when a flue gas exhaust mixes with ambient air.

Particulate matter filter samples collected using dilution sampling can be further analysed to provide chemical composition data that are applicable for developing PM<sub>2,5</sub> or PM<sub>10</sub> emission inventories, visibility impact assessments, health risk assessments, and source-receptor studies related to PM<sub>2,5</sub> and PM<sub>10</sub> emissions.

This method is not applicable to the determination of ultrafine particles with an aerodynamic diameter of less than 0,1 µm. This method has been applied to emission sources with low moisture and saturated moisture stack gases; however, it is not applicable to effluents where entrained water droplets are present.

**NOTE** Optionally, with additional equipment, as well as sampling and analytical procedures not described in the method, PM chemical speciation and particle size can be determined by applying ambient air sample collection and analysis methods to the diluted stack samples obtained by using this method. In addition, the concentration of gaseous precursors that can contribute to the formation of particulate matter, e.g. SO<sub>2</sub>, NO<sub>x</sub>, ammonia, SO<sub>3</sub>, HCl, volatile organic compounds (VOCs), can be determined using suitable analytical equipment to measure the diluted samples.

## 1.2 Limitations

### 1.2.1 General limitations

It is recognized that there are some combustion processes and situations that can limit the applicability of this International Standard. Where such conditions exist, caution and competent technical judgment are required, especially when dealing with any of the following:

- high-vacuum, high-pressure or high-temperature gas streams above 260 °C;
- fluctuations in velocity, temperature or concentration due to uncontrollable variation in the process;
- gas stratification due to the non-mixing of gas streams.

There are also limitations specific to each sampling technique.

### 1.2.2 Basic cyclone sampling technique

Stacks with entrained moisture droplets can have droplet sizes larger than the cut sizes for the cyclones. These water droplets normally contain particles and dissolved solids that become PM<sub>10</sub> and PM<sub>2,5</sub> following evaporation of the water.

### 1.2.3 Dilution sampling technique

For dilution sampling, a known limitation of this method concerns the presence of particles in the dilution air at very low concentrations, contributing to measurement background. This can be significant for certain very clean sources, e.g. gas-fired power plants. Dilution air system blanks are necessary when sampling sources with anticipated PM<sub>2,5</sub> or PM<sub>10</sub> mass concentrations less than or equal to about 1,0 mg/m<sup>3</sup>.

## 2 Normative references

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

ISO 7708:1995, *Air quality — Particle size fraction definitions for health-related sampling*

ISO 8178-1:2006, *Reciprocating internal combustion engines — Exhaust emission measurement — Part 1: Test-bed measurement of gaseous and particulate exhaust emissions*

ISO 9096, *Stationary source emissions — Manual determination of mass concentration of particulate matter*

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

ISO 12039, *Stationary source emissions — Determination of carbon monoxide, carbon dioxide and oxygen — Performance characteristics and calibration of automated measuring systems*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

### 3 Terms and definitions

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

#### 3.1

##### **aerodynamic diameter**

diameter of a sphere of density 1 g/cm<sup>3</sup> with the same terminal velocity due to gravitational force in calm air as the particle, under prevailing conditions of temperature, pressure and relative humidity

[SOURCE: ISO 23210:2009, [13](#) 3.1.1]

#### 3.2

##### **ambient air sample**

sample collected for quality assurance purposes by drawing unfiltered ambient air at the stationary source sampling location through the sampling media

#### 3.3

##### **bulk mean gas residence time**

actual sample volume flow rate taken at the actual bulk mean temperature of the sample gas flowing through the aging section

#### 3.4

##### **bulk mean temperature**

##### **actual bulk mean temperature**

mean of the diluted sample gas temperature measured at the entrance and exit of the aging chamber

#### 3.5

##### **condensable particulate matter**

particulate matter formed at temperatures below 30 °C due to physical and/or chemical processes

#### 3.6

##### **constant flow rate sampling**

withdrawal of a sample from the flue gas at a fixed flow rate through the sampling nozzle

Note 1 to entry: Withdrawal of a sample is chosen at a fixed flow rate to achieve the appropriate cut diameters for particles in the sampling cyclones and 80 % to 120 % isokinetic sampling conditions.

#### 3.7

##### **cut-off diameter**

aerodynamic diameter where the separation efficiency is 50 %

[SOURCE: ISO 23210:2009, [13](#) 3.1.2, modified — “of the impactor stage” deleted]

#### 3.8

##### **diluted stack gas**

combined stack gas sample and dilution air, after mixing

#### 3.9

##### **dilution air**

ambient air that is filtered to remove solid particulate and organic compounds before mixing with a stack gas sample in a dilution sampling system

**3.10**  
**dilution factor**  
**DF**

one plus dilution ratio

Note 1 to entry: The product of dilution factor and filter sample PM concentration is equal to the in-stack PM concentration.

**3.11**  
**dilution ratio**  
**DR**

ratio of the dilution air flow rate through a dilution system to the undiluted stack gas sample flow rate through the system

Note 1 to entry: Determined by direct flow measurement or tracer method (ISO 8178-1).

**3.12**  
**dilution sampling system**

sampling equipment that dilutes stationary source stack gases with filtered ambient air to simulate the mixing and cooling processes in a stack plume

**3.13**  
**dilution system blank**

quality assurance sample collected by assembling the dilution sampling equipment at the stationary source sampling location and then drawing only dilution air through the dilution system and sampling media

**3.14**  
**field blank**

sample collected for quality assurance purposes by assembling the dilution sampling equipment at the stationary source sampling location, performing a leak check, and then disassembling the equipment

Note 1 to entry: No stack gas sample passes through the equipment.

**3.15**  
**F-factor**

ratio of combustion gas volume to heat input determined from fuel composition using combustion calculations

**3.16**  
**filter holder assembly**

filter holder or cassette that contains the filter media for collecting particulate samples

**3.17**  
**filtration temperature**

temperature of the diluted sample gas immediately downstream of the filter

[SOURCE: ISO 12141:2002, 3.2, modified — “diluted” inserted]

**3.18**  
**flue gas sample**

flue gas, extracted from a duct or stack of a stationary source, before dilution, in the application of the method

**3.19**  
**isokinetic sampling**

sampling at a flow rate 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 point ( $v_s$ )

[SOURCE: ISO 12141:2002, 3.5]

**3.20****laboratory blank**

conditioned, unsampled filter used to determine any mass change between pre- and post-sampling weighings due to contamination occurring during the sampling

**3.21****PM**

particulate matter including PM<sub>2,5</sub>, PM<sub>10</sub>, and/or total suspended particulate matter

**3.22****PM<sub>2,5</sub>**

particles which pass through a size-selective inlet with a 50 % efficiency cut-off at 2,5 µm aerodynamic diameter

[SOURCE: ISO 23210:2009, [13](#) 3.1.4]

Note 1 to entry: PM<sub>2,5</sub> corresponds to the “thoracic convention” as defined in ISO 7708:1995, 6.

**3.23****PM<sub>10</sub>**

particles which pass through a size-selective inlet with a 50 % efficiency cut-off at 10 µm aerodynamic diameter

Note 1 to entry: PM<sub>10</sub> corresponds to the “thoracic convention” as defined in ISO 7708:1995, 7.1.

**3.24****coefficient of variation**

standard deviation divided by the mean

Note 1 to entry: The coefficient of variation is commonly reported as a percentage.

[SOURCE: ISO 3534-1:2006, [14](#) 2.38]

**3.25****sampling point**

specific position in a source vent at which a sample is extracted

**3.26****standard pressure**

absolute pressure at standard conditions

**3.27****standard temperature**

absolute temperature at standard conditions

**3.28****ultrafine particulate matter**

particulate matter with an aerodynamic diameter less than or equal to 0,1 µm

**3.29****volatile organic compound****VOC**

organic compound present with a vapour pressure below its saturated vapour pressure at ambient air pressure and temperature

**3.30****weighing control procedure**

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

## 4 Symbols and abbreviated terms

dscm	dry standard cubic metre
dscmm	dry standard cubic metre per minute
$A$	area of stack or duct at the sampling location ( $\text{m}^2$ )
$B_f$	average blockage factor
$B_{ws}$	moisture fraction of flue gas
$C$	Cunningham correction factor for particle diameter calculated using actual stack gas temperature
$C_p$	Pitot tube coefficient for tube attached to sampling apparatus
$C'_p$	Pitot tube coefficient for Pitot tube used in the preliminary traverse
$D$	inner diameter of sampling nozzle mounted on $\text{PM}_{10}$ cyclone (cm)
$D_{50}$	particle cut diameter ( $\mu\text{m}$ )
HEPA	high-efficiency particulate air
$K_p$	Pitot tube equation constant [ $(\text{m/s})/(\text{g/mol K})$ ]
$K_1$	unit conversion constant
$M_d$	molecular mass of dry gas ( $\text{g/mol}$ )
$M_s$	molecular mass of stack gas
$M_w$	molecular mass of wet gas ( $\text{g/mol}$ )
$N_{tp}$	number of iterative steps
$n$	number of sample pairs
$p_s$	absolute stack gas pressure (Pa)
$q_{Vs}$	sampling rate for $\text{PM}_{10}$ cyclone necessary to achieve specified $D_{50}$ (acmm)
$T_s$	absolute stack gas temperature (K)
$t$	Student factor for a 95 % confidence and the degrees of freedom $n - 1$
$t_n$	sampling time at point $n$ (min)
$t_r$	total projected run time (min)
$t_{tr}$	total run time (min)
$t_1$	sampling time at point 1 (min)
$V_c$	volume of water captured in impingers and silica gel (ml)
$V_{m,std}$	dry gas meter volume sampled, corrected to standard conditions (dscm)
$v_s$	average stack gas velocity in the same cross-sectional plane as the stack gas sample measurements ( $\text{m/s}$ )

$V_{w,std}$	volume of water vapour (scm)
$v_n$	sample gas velocity in the nozzle (m/s)
$\Delta H$	pressure drop across the orifice meter
$\Delta p$	Pitot tube velocity pressure differential
$\Delta p_m$	measured velocity pressure
$\Delta p_n$	velocity pressure differential point $n$ during the test run
$\Delta p_{s1}$	velocity pressure adjusted for combined cyclone Pitot tube
$\Delta p_{s2}$	velocity pressure corrected for blockage
$\Delta p_1$	velocity pressure differential measured at point 1
$\left(\sqrt{\Delta p}\right)_{avg}$	average of square roots of the velocity pressure differentials measured during the preliminary traverse
$\beta_1$	0,027 754 for metric units
$\eta$	gas viscosity ( $\mu\text{Pa}$ )
<sup>a</sup>	1 $\mu\text{P}$ = 0,1 $\mu\text{Pa s}$

## 5 Principle

### 5.1 Method using sampling cyclones

#### 5.1.1 Sampling cyclones

To determine filterable particulate matter size fractions,  $\text{PM}_{2,5}$  and  $\text{PM}_{10}$ , a sample is extracted from a stationary stack at a predetermined constant flow rate through an in-stack  $\text{PM}_{10}$  and a cyclone  $\text{PM}_{2,5}$ . The first cyclone separates particles with nominal aerodynamic diameters of 10  $\mu\text{m}$  and the following cyclone, particles with nominal diameters of 2,5  $\mu\text{m}$ . Sampling is conducted isokinetically, at sampling points on the sampling cross-section determined according to ISO 9096. After sampling, the  $\text{PM}_{10}$  and  $\text{PM}_{2,5}$  concentrations are determined by gravimetric analysis and the measured sampling volume. A summation of the two concentrations can provide a measure of the total suspended particulate in the flue gas. The basic sampling system is shown in [Figure 1](#).

NOTE In certain emission sources, e.g. gas-fired units, the use of the  $\text{PM}_{10}$  cyclone is optional.

#### 5.1.2 Theory of cyclones

Cyclones are devices designed to impart centrifugal force on the gas stream in a conical shaped chamber to use inertia for the removal of particles from the gas stream. The incoming gas stream is forced into a circular motion as it passes down the cyclone. At the bottom of the cyclone, the gas spirals up through the centre tube and out at the top. Particles in the gas stream are forced to the wall by the centrifugal force but retarded by the drag of the gas stream. Larger particles with larger inertia overcome the drag forces and deposition on the cyclone walls. Smaller particles follow the gas flow stream and exit the cyclone. The collection efficiency for specific particle size range is dependent upon the cyclone design, gas density, and the flow rate of the gas through the cyclone.

5.1.3 Isokinetic sampling

When PM<sub>10</sub> is being determined, the sample should be withdrawn from the stack gas isokinetically. Near-isokinetic sampling within 80 % to 120 % of the isokinetic sampling rate is allowed for PM<sub>2,5</sub> sampling. Sampling that is “isokinetic” within the range 90 % to 115 % is required for PM<sub>10</sub> sampling.

5.1.4 Method using sampling cyclones and sample dilution

To determine both filterable and condensable PM<sub>10</sub> and PM<sub>2,5</sub> the sample gas is rapidly mixed with dilution air (Figure 2). The dilution system approximately simulates the dilution and cooling processes that occur in the near field of a stack plume as it mixes with the atmosphere. These processes impact the homogeneous and heterogeneous nucleation of stack gas constituents. The diluted sample can then be analysed for particulate matter or gases, depending upon the requirements of the test.

5.2 Schematic of the apparatus

5.2.1 Cyclone sampling system

A schematic of the basic cyclone sampling system is shown in Figure 1. Other arrangements of components of the sampling system are acceptable as long as the criteria of 6.1 are met.

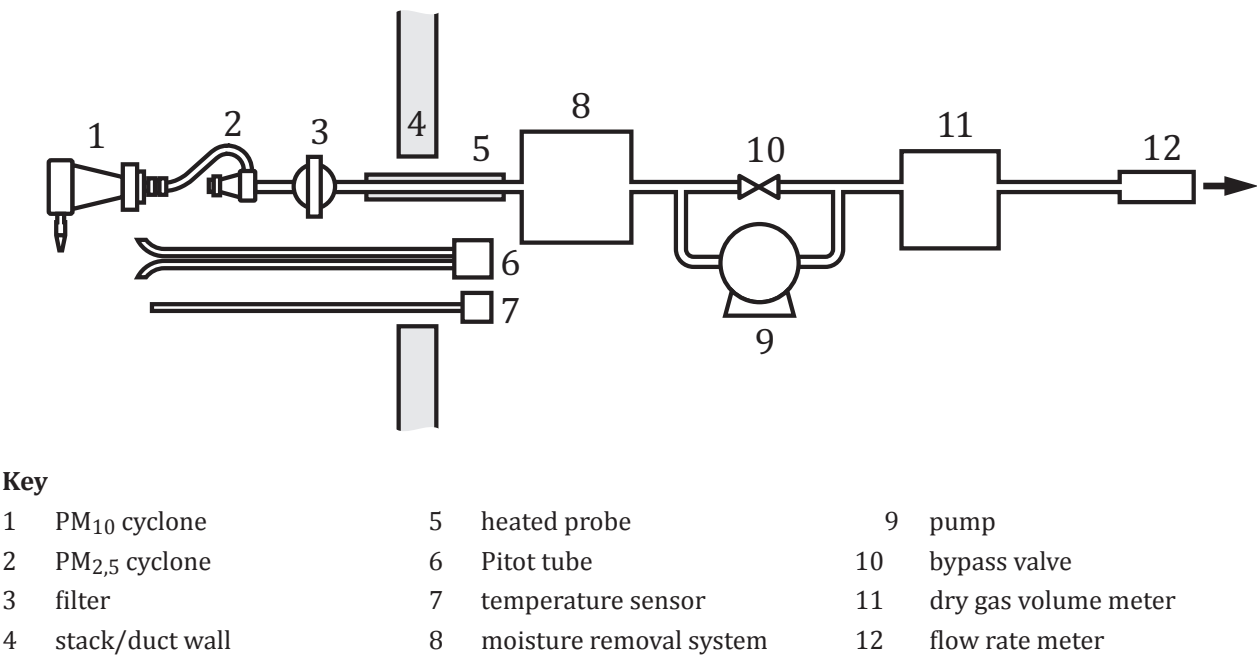


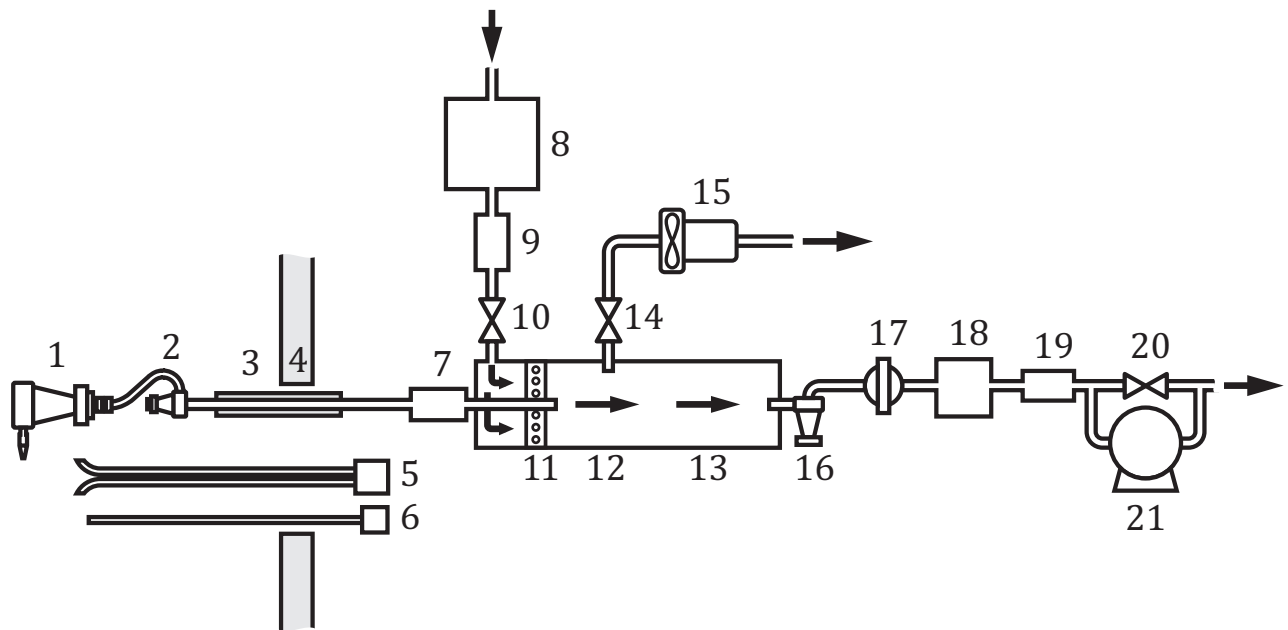
Figure 1 — In-stack combined PM<sub>10</sub> and PM<sub>2,5</sub> cyclone basic sampling train

5.2.2 Dilution sampling system

A conceptual schematic of a dilution sampling system is shown in Figure 2. Other arrangements of components of the sampling system are acceptable as long as the criteria of 6.2 are met.

In the dilution system, PM<sub>10</sub> and/or PM<sub>2,5</sub> cyclones are attached to the probe as in Figure 1. The sample then passes into a mixing section of a dilution chamber where it is diluted with dilution air. After dilution, the aerosols are aged in the aging zone for a minimum residence time of 10 s. The aged gas is then pulled through a secondary PM<sub>2,5</sub> cyclone a single stage PTFE membrane filter or equivalent and the particulate matter and condensable collected subsequently measured gravimetrically.





#### Key

1	PM <sub>10</sub> cyclone	8	dilution air-conditioning system	15	bypass pump or fan
2	PM <sub>2,5</sub> cyclone	9	flow rate meter	16	PM <sub>2,5</sub> cyclone
3	heated probe	10	valve	17	filter
4	stack/duct wall	11	distribution plane	18	other analytical monitoring equipment
5	Pitot tube	12	mixing zone	19	flow rate meter
6	temperature sensor	13	aerosol aging zone	20	valve
7	flow rate meter	14	bypass flow controller	21	pump

**Figure 2 — In-stack PM<sub>10</sub> and PM<sub>2,5</sub> cyclones with dilution sampling train**

Stack gas flow rates required for determination of mass emissions are measured by a Pitot tube traverse of the stack, fuel flow rates and *F*-factors, or equivalent method.

This International Standard applies only to the measurement of the collected particulate matter by filter mass measurement using a gravimetric balance. It is also possible to use paper tape sampling systems, inertial microbalances or light scattering methods to quantify particulate matter levels. However, in the application of these techniques, it is necessary to establish the relationship of the measured parameter to the gravimetrically determined particle mass concentration. Establishing such relationships does not come within the scope of this International Standard.

Using additional equipment and sampling or analytical procedures not described in this method, the collected particulate matter can be speciated for the determination of, for example, sulfates, nitrites, nitrates, as well as condensed hydrochloric and sulfuric acids. Also, the diluted stack sample can be analysed to determine concentrations of SO<sub>2</sub>, NO<sub>x</sub>, ammonia, SO<sub>3</sub>, HCl or VOCs by applying ambient air sample collection and analysis methods. It is necessary to use appropriate materials that do not contaminate the sample for the species of interest for all sampler surfaces that contact the sample.

An important source of PM mass measurement bias is from the handling of the filters. Extreme care should be taken during the loading and unloading of filters to ensure the filters do not fray or break. It is highly recommended that the loading and unloading of the filter housings be performed in a laboratory, rather than at the testing location. Personnel loading and unloading filter housings should wear gloves that are antistatic and powder-free when handling filters. Smooth, non-serrated forceps should be used when handling filters. The forceps should be used for this purpose only, cleaned with alcohol and lint-free wipes, and air dried prior to use.

Particle losses on surfaces include particle deposition on the sampling probe that tend to be the highest of all losses. Particles can deposit on surfaces of the sample probe of both the basic and dilution systems and thus may not be collected on the sampling media. Particle losses can also occur in the dilution sampler due to a combination of factors, e.g. thermophoretic losses in the sampling probe, and electrostatic, inertial, and diffusional losses in the sampler and tubing connecting sampling media to the sampler.

Improper design or use of a dilution sampler can cause the deposition of particulate matter on the surfaces in contact with the undiluted stack gas sample. Recover, analyse, and include in the total  $PM_{2,5}$  and  $PM_{10}$  results any deposits on these sampler components. Earthed electrically conductive surfaces can minimize electrostatic losses. Maintaining the sample probe at a temperature at or slightly above the stack gas temperature may minimize thermophoretic losses. Also, sharp changes in flow direction should be avoided to minimize inertial losses.

With proper sampler design, deposits of particulate matter on the surfaces of a dilution sampler in contact with the diluted stack gas sample, e.g. the mixing section and residence time section, have been determined to be 3 % or less of total  $PM_{2,5}$  and  $PM_{10}$  based on assessment results with monodisperse particles. Therefore, dilution samplers designed to minimize particle deposits on these components should be capable of achieving equivalent performance.

Liquid water content can impact ambient aerosol size distribution. At ambient relative humidity (RH) greater than 70 %, particle growth due to accumulation of moisture becomes significant. Particle size growth from moisture condensation can bias PM measurements if agglomerated particles grow to a size greater than the target size (2,5  $\mu m$  or 10  $\mu m$  aerodynamic diameter) and are removed by the dilution sampling system prior to the sampling media. Therefore, keep the humidity of the diluted sample humidity below 70 % to maintain particle growth conditions.

The sample can be altered during sample storage and shipping, if appropriate conditions are not maintained. For example, organic and nitrate compounds on filters may volatilize if the samples are stored at temperatures higher than the sample collection temperature contributing to negative bias in the filter mass.

## 6 Equipment and materials

### 6.1 Cyclone sampling system

**6.1.1 Details of sampling cyclones and sampling train.** [Figure 1](#) shows details of the combined sampling cyclones and sampling train used in the determination of filterable particulate matter. [Figure 2](#) shows the details of the dilution sampling train used for the determination of both filterable and condensable particulate matter.

The determination of  $PM_{10}$  and  $PM_{2,5}$  concentrations using the cyclone sampling system, divides the filterable particulate matter into three fractions:

- particles with aerodynamic diameter greater than 10  $\mu m$  (first cyclone);
- particles with aerodynamic diameters between 10  $\mu m$  and 2,5  $\mu m$  (second cyclone);
- particles with aerodynamic diameters smaller than 2,5  $\mu m$  (backup filter).

**NOTE** The cyclones can be used either in a cascade arrangement or separately. The use of the  $PM_{10}$  or  $PM_{2,5}$  cyclones is optional, if the size of the particles in the flue gas is sufficiently low.

**6.1.2 Nozzle.** Stainless steel [316 or equivalent or stainless steel coated with poly(tetrafluoroethylene) (PTFE)] to provide for near isokinetic sampling (80 % to 120 % isokinetic). With a sharp, tapered leading edge. Nozzles sizes are to be appropriate to achieve sampling rates between 80 % to 120 % isokinetic sampling at each sampling point.

**6.1.3 Sampling cyclones.** Use electropolished, stainless steel sampling cyclones with  $D_{50}$  values of 2,5  $\mu\text{m}$  and 10  $\mu\text{m}$ . [Annex A](#) provides examples of suitable designs. Other cyclone designs are acceptable, if they meet the criteria given in the efficiency curves of [Figures B.1](#) and [B.2](#).

NOTE The threads of the cyclones can gall or seize at temperatures exceeding 260 °C. Cyclones constructed of speciality metals, such as inconel, can be necessary at higher temperatures.

Inconel is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

Depending on the test objectives, PM<sub>10</sub> and or PM<sub>2,5</sub> cyclones may be used to withdraw the sample from the flue gas. The in-stack cyclones shall be constructed of electropolished stainless steel (316 or equivalent) and operated to achieve a 50 % cut-off diameter of 2,5  $\mu\text{m}$  and/or 10  $\mu\text{m}$ . Separation efficiencies are given in B.3.

**6.1.4 Filter holder (filterable PM sampling train).** An in-stack 316 stainless steel or PTFE-coated stainless steel filter holder to support either a 24 mm, 47 mm or 63 mm diameter filter. The filter holder is designed to contain a PTFE O-ring, a stainless steel filter support screen, and a final PTFE O-ring. The filter is attached to the outlet of the PM<sub>2,5</sub> cyclone.

**6.1.5 Sample transfer line options.** Use transfer lines with a diameter greater than or equal to the probe diameter, but no more than 13 mm. Avoid sharp changes in flow direction in order to minimize inertial losses. The probe extension should be glass-lined or PTFE, and be as short as possible. The sample transfer line is to be insulated and heated to a minimum wall temperature equal to the source temperature and the water dew point, but no greater than 315 °C.

NOTE PTFE is not suitable for sampling of coal fly ash due to electrical charge accumulated on particulate material causing attraction to the PTFE walls.

**6.1.6 Pitot tube.** An S-type Pitot tube made of heat-resistant tubing is attached to the probe in order to determine the value of the flue gas velocity,  $v_s$ , at each sampling point. This value is used to calculate the appropriate sampling rate for the test equipment at the sampling point. See ISO 10780 for design criteria.

**6.1.7 Temperature-sensing device.** Use a Pt 100 resistance thermometer or other temperature sensing device that has a maximum uncertainty of  $\pm 1$  °C.

## 6.2 Dilution sampling system

**6.2.1 Dilution ratios.** In the dilution sampling system, humidified air, filtered to remove fine particulate matter, is used to dilute the sample gas.

The dilution ratio shall be at least 20:1. Higher dilution ratios can be required to maintain the particle filter temperature at 42 °C or lower. The lowest dilution ratio commensurate with achieving the filter temperature limitation should be used under these circumstances. When the stack gas temperature is very high, it may be necessary to cool the dilution air and/or filter holder assembly to achieve the filter temperature limitation. When the ambient air temperature is very low, it may be necessary to heat the dilution air to maintain the filter temperature near 42 °C.

The diluted sample RH shall not to exceed 70 % because higher levels can significantly affect particle mass and size distribution within the dilution sampling system and thereby change the nucleation, condensation, and accumulation processes.

**6.2.2 Dilution air high-efficiency particulate air filter** (dilution sampling train), designed for air flows to give dilution ratios of at least 20:1. Use HEPA filters with a minimum filtering efficiency of 99,97 % when tested with an aerosol of 0,3  $\mu\text{m}$  diameter.

A known limitation of this method concerns the presence of particles in the dilution air at very low concentrations, contributing to measurement background. This may be significant for certain very clean

sources such as gas-fired power plants. Dilution air system blanks are necessary when sampling sources with anticipated PM<sub>2,5</sub> or PM<sub>10</sub> mass concentrations approximately less than or equal to 1,0 mg/m<sup>3</sup>.

A dilution air-activated carbon bed or filter, designed for air flows to give dilution ratios of at least 20:1, is recommended if samples for analysis of organic compounds are to be collected or if ambient air has high VOC concentrations.

A dilution air dehumidifier, designed for air flows to give dilution ratios of at least 20:1 and diluted gas humidity less than 70 % at temperatures equal to or less than 42 °C, may optionally be used.

A dilution air heater or cooler, designed for air flows to give dilution ratios of at least 20:1 and dilution gas temperatures equal to or less than 42 °C, is optional. The dilution air may have to be either cooled or heated, depending on the flue gas and ambient temperature, to maintain a temperature of 42 °C at the filter.

### **6.2.3 Specifications of the dilution sampling system mixer/residence time chamber (dilution sampling train).**

#### **6.2.3.1 Mixing point**, the inlet is the point of mixing of dilution air and the stack gas sample.

Mixing shall be rapid with completeness verified by measurement of the downstream CO<sub>2</sub> profile for combustion sources at a minimum of four points with a maximum difference of 10 % between any two traverse points. Other verification procedures such as VOC profiling may be used for non-combustion sources. Mixer designs can include jet entrainment, mixing orifice, venturi or equivalent.

#### **6.2.3.2 Dilution ratio**, the flow capacity of the dilution system shall be sufficient to provide a minimum 20:1 dilution ratio.

Larger dilution ratios may be required to completely eliminate water condensation in dilution and sampling systems and maintain temperature of the diluted stack at or below 42 °C, immediately upstream of the cyclone(s) of the residence time chamber.

#### **6.2.3.3 Residence time**, the minimum residence time shall be 10 s after mixing is complete.

#### **6.2.3.4 Construction materials**. The system shall be designed and constructed of non-reactive materials to minimize deposition or alteration of the particulates and electrically earthed to prevent electrostatic effects.

#### **6.2.3.5 Dilution air vacuum pump**, the vacuum pump shall have sufficient capacity to maintain specified dilution air flows.

A temperature control system, by insulating, cooling or heating, may optionally be required to maintain target temperature range.

### **6.2.4 Dilution sampling train — sampling system characteristics.**

#### **6.2.4.1 Aerosol aging zone — residence time**. The aging zone of the dilution sampler shall have a minimum bulk mean gas residence time 10 s after the raw stack gas sample and dilution air are fully mixed.

The gas is assumed to be fully mixed when the coefficient of variation of the sample gas concentration in the diluted sample at cross-sectional plane at the entrance to the aging section is less than 10 % of the mean concentration.

This may be determined by spiking the raw sample gas with a tracer gas such as CO<sub>2</sub> or methane, and measuring the tracer gas concentration at a minimum of 12 points, six points each on two orthogonal axes in the cross-sectional plane at the entrance to the aging section. The mean and coefficient of variation of the measurements are then calculated from the results.

The purpose of aging after dilution is to provide sufficient time for vapour phase species to diffuse to the surfaces of existing particles and condense. The minimum residence time required for this to occur, and therefore to achieve robust particulate mass results, is inversely proportional to the concentrations of particles and condensable vapours present in the sample. See ISO 80000-9[12] for changing mg/m<sup>3</sup> to ISO acceptable units.

- a) For sources with particle concentrations of 0,1 mg/m<sup>3</sup> and lower, the minimum residence time is 10 s (see Reference[4]). This includes sources such as gas-fired combustion sources (boilers, process heaters, steam generators). Such sources typically have extremely low concentrations of both particles and vapours.
- b) Excess residence time is not considered detrimental to method performance, therefore minimum residence time is 10 s, unless it is demonstrated that shorter residence times are sufficient. The minimum residence times for specific source types may deviate from those given above provided that the performance equivalent to 10 s residence time is demonstrated.

Equivalency is demonstrated by conducting tests with paired samplers and conducting nine test runs concurrently (total 18 samples). One sampler with a 10 s residence time is compared with another having a different residence time at which equivalency is desired. The sample probe tips should be 2,5 cm from the outside edges of each other. The samples from each sampler should be stored together under identical storage conditions until laboratory analysis. All of the samples shall be analysed within one week of sample collection. The bias is tested for statistical significance by calculating the paired *t*-test statistic. If the calculated *t*-value is less than the critical value at the 95 % confidence level, the bias is not statistically significant and the alternative residence time is acceptable.

**6.2.4.2 Cyclone.** After the sample is diluted and aged, use another PM<sub>2,5</sub> cyclone designed according to the dimensions in [Figure A.2](#) to collect a PM<sub>2,5</sub> sample from the residence time chamber. The flow rate through the cyclone(s) shall be sufficient to provide flow to the sample filter and other sample media. The cyclone used in the residence time chamber may be constructed of PTFE, for further characterization of particulate materials.

PM<sub>2,5</sub> cyclones may be used to withdraw the diluted and aged sample from the residence time chamber.

NOTE These cyclones are in addition to the in-stack sampling cyclones at the end of the probe. The PM<sub>2,5</sub> “condensables” cyclone is shown after the residence chamber in [Figure 2](#). PTFE cyclones can be used for further characterization of particulate materials.

**6.2.4.3 Flow rate** through the cyclone(s) shall be sufficient to provide flow to the sample filter and other sample media. The cyclone shall be constructed of electropolished stainless steel (316 or equivalent) and shall be operated to achieve a 50 % cut-off diameter of 10 µm and/or 2,5 µm ([Figures A.1](#) and [A.2](#)).

**6.2.4.4 Transfer line.** The transfer line(s) from the cyclone(s) shall be designed to minimize PM deposition (gradual bends if any), constructed of electrically conductive material (stainless steel tube is recommended) that does not react with gas constituents, and shall be electrically earthed. The transfer line may be heated to no more than 42 °C before stack gas is introduced to the chamber containing the filter packs and may be insulated. The transfer line is to be as short as possible, with a maximum length of 1,02 m.

NOTE PTFE is not suitable for sampling of coal fly ash due to electric charges accumulated on the particulate material.

**6.2.4.5 Filter cassette assembly** (gravimetric analysis method), constructed of 302, 303 or 304 stainless steel, polycarbonate or acrylonitrile–butadiene–styrene (ABS) resin with a minimum 47 mm stain area. A combination of these materials may also be used. Recommended loading of stain area: 0,5 mg



on 47 mm filter; 1,3 mg on 70 mm filter; 2,3 mg on 90 mm filter; 3,6 mg on 100 mm filter. The filter packs may be heated or insulated to maintain a maximum filter temperature of 42 °C.

The filter holder assembly design shall avoid sample losses that have a significant adverse effect on systematic and random test result variations.

- The entrance to the filter holder shall be designed using sound engineering judgment and leak-free construction, to an inside diameter no smaller than 0,85 cm, maintaining the 12,5° angle from the inlet of the top filter holder to the area near the sealing surface of the top of the filter cartridge assembly. Variations using sound engineering design are also acceptable provided that they provide even flow distribution across the filter media and a similar leak-free seal with the filter cartridge assembly.
- A bevel introduced on the inside diameter of the entrance to the filter cartridge, is also acceptable.
- Containers shall protect the cassette and prevent it from moving within the container during transport. Containers should be fabricated from materials that have been demonstrated not to cause a static charge buildup. To avoid losses of particulate matter, do not allow the surface of the filter to touch any interior surfaces of the protective container. Containers should be marked for identification purposes.
- An insulated shipping container should be used if the time between the end of the sampling and the conditioning of the filter is expected to be longer than 24 h or if the ambient temperature is higher than 40 °C. An insulated shipping container, capable of maintaining a temperature below 25 °C, is needed to transport the filter cassette containers between the field and laboratory after sampling. The sample cassette temperature should be monitored from the time of collection until the sample is equilibrated (i.e. during shipping and handling).

**6.2.4.6 Sample flow measurements.** Determine the sample gas flow with a flow measurement device accurate to at least  $\pm 4$  %.

### 6.3 Analytical balance

**6.3.1 Precision.** 95 % confidence uncertainty bounds: 5  $\mu\text{g}$  for filter diameter less than or equal to 70 mm; 50  $\mu\text{g}$  for filter diameter greater than 70 mm.

**6.3.2 Resolution.** Filter size <70 mm: 1  $\mu\text{g}$ . Filter size  $\geq 70$  mm: 10  $\mu\text{g}$ .

**6.3.3 Location** of the balance shall be as follows.

- Locate the microbalance on a sufficiently level, sturdy, vibration-free base (ideally, a level, stone weighing table), away from any equipment that produces vibrations, out of direct sunlight, and away from local heating or cooling sources such as open flames, hot plates, water baths, ventilation ducts, windows, and heat-producing lamps.
- Locate the balance in the same climate-controlled environment in which the filters are conditioned. Use temperature and humidity controls and meters to maintain and monitor the conditions within the filter conditioning and weighing room environment. The mean temperature of the weighing chamber should be maintained between 20 °C and 23 °C, with a variability of not more than  $\pm 2$  °C over 24 h and the mean RH should be maintained between 45 % and 55 %, with a variability of not more than  $\pm 5$  % over 24 h.

**6.3.4 Electrostatic effects** and/or static charge effects should be prevented as follows:

- electrically earth the analytical balance to prevent electrostatic effects;
- eliminate or minimize static charge effects by using a polonium-210 static charge neutralizer or device.

It may be necessary to place a polonium-210 neutralizer or other similar device in the weighing chamber to prevent electrostatic charge buildup. It may also be necessary to pass each filter near, but not touching, an antistatic strip before it is weighed. Charge neutralization times may need to be longer than 60 s for sampling situations in which:

- a) a high amount of charge has developed on collected particles due to their origin; or
- b) the particle loading on a filter is large.

Electrostatic charge buildup becomes greater as the air becomes drier. 60 s charge neutralization may be sufficient in ambient indoor air conditioned to 37 % RH and 23 °C but not in 20 % RH and 23 °C in arid environments. This latter environment may require that the filter sit for more time on the antistatic strip. The longer neutralization period may have to be performed inside the weighing chamber or in a second small chamber used only for charge neutralization. The effectiveness of charge neutralization shall be measured by making multiple weighings in conjunction with passing the filter over the charge neutralizer between successive readings until the weighing reading is stabilized.

**6.3.5 Calibration.** Calibrate the analytical balance at least annually using certified traceable mass reference standards. In addition, use working standards to verify the calibration of the analytical balance before each use. Verify the working standards every three months to six months against traceable laboratory primary standards.

Select mass reference standards so that they are in the expected mass range for clean, unexposed filters and exposed filters. They should be certified as being traceable to laboratory primary standards. Additionally, they should have an individual tolerance of no more than 0,025 mg.

## 7 Reagents and materials

Use only reagents of recognized analytical grade, unless otherwise specified.

**7.1 Water**, unless otherwise specified, use type III reagent water.

**7.2 Acetone**, with less than 0,001 % residue, in glass bottles. Acetone supplied in metal containers is unacceptable due to the prevalently high residue levels. Reject the acetone if blank residue mass is >0,001 % of the total acetone mass.

**7.3 Silica gel.** Filterable particulate sampling train ([Figure 1](#)). Use an indicating-type silica gel of 6 mesh to 16 mesh. Allow the silica gel to dry for 2 h at 175 °C if it is being reused.

**7.4 Tissue papers**, low-linting, non-abrasive.

**7.5 Towels.**

**7.6 Brushes**, to clean sample probes and lines.

**7.7 PTFE tape.**

**7.8 Wash bottles**, PTFE, or equivalent.

**7.9 Shipping containers.**

**7.10 Gloves**, insulated, heat-resistant.

**7.11 Forceps**, smooth, non-serrated, specifically dedicated to the task of handling filters, when handling the filters. Clean forceps using alcohol and lint-free wipes and allow to air dry before handling any filters.

## 7.12 Filter specifications.

**7.12.1 Size**, circular, 47 mm diameter or greater (with support ring).

**7.12.2 Medium**, borosilicate microfibres reinforced with woven glass cloth and bonded with PTFE.

**7.12.3 Support ring**, polymethylpentene (PMP) or equivalent inert material.

Check the support ring diameter for consistency. Variations in diameter affect the filter exposure area, which, in turn, affects any operations that involve use of a portion of the filter.

**7.12.4 Thickness**, 30 µm to 50 µm.

**7.12.5 Maximum pressure drop (clean filter)**, 2,94 kPa (30 cm) H<sub>2</sub>O column at 16,67 l/min clean air flow.

**7.12.6 Maximum moisture pickup**, no more than 10 µg mass increase after a 24 h exposure to air at 40 % RH, relative to the mass after a 24 h exposure to air at 35 % RH.

**7.12.7 Collection efficiency**, greater than 99,95 %, as measured by using dioctyl phthalate (DOP) spheres with 0,3 µm particles at the operating face velocity of the sampler.

The manufacturer's quality control test data are sufficient for validation of efficiency.

**7.12.8 Filter mass stability**, including test for loose, surface-particle contamination and test for temperature stability.

Filter mass loss <20 µg in either test.

**7.12.9 Alkalinity**, less than 25 micro equivalents per gram of filter.

**7.12.10 Irregularities**, check the filters for irregularities, flaws, or pinholes by holding them up against a light source.

Although not required for determination of PM<sub>2,5</sub> mass concentration in accordance with this International Standard, additional specifications for the filter should be developed by users who intend to subject archived PM<sub>2,5</sub> filter samples to subsequent chemical analysis. These supplemental specifications include background chemical contamination of the filter and any other filter parameters that may be required by the chemical analysis method.

**7.13 Personal protective equipment**, as needed.

## 8 Pre-sampling, filter conditioning, and weighing procedures

### 8.1 General aspects

#### 8.1.1 Purpose of sampling

Before carrying out any measurements, discuss the purpose of the sampling and the sampling procedures with the plant personnel concerned. The nature of the plant process, for example, steady-state or cyclic, can affect the sampling programme. If the process can be performed in a steady-state, it is important that this state be maintained during sampling.



### 8.1.2 Scheduling

Agree, with the plant management, the dates, starting times, duration of survey, and sampling periods as well as plant operating conditions during these periods.

### 8.1.3 Preliminary calculations

Make preliminary calculations on the basis of the expected particulate loading to determine the appropriate nozzle size, sample rate, and sampling conditions. Preliminary sampling may be necessary. Also determine whether the chosen nozzle size, sampling rate and sampling time result in sufficient matter collected to obtain a sample filter mass at least five times the dilution system blank filter mass.

If site-specific particulate concentration data are not available, use estimated values for total particulate, PM<sub>2,5</sub>, or PM<sub>10</sub> concentrations using published emission factor estimates for controlled or uncontrolled emission sources.

If possible, use system blank results from previous tests using the same sampling system. If no previous data are available, then select run times based on minimum detection limits, including background levels.

## 8.2 Gravimetric filter weighing procedures

### 8.2.1 Pre-sampling filter conditioning

Pre-sampling filter conditioning is as follows.

- Verify that the filter weighing chamber mean RH has been maintained between 45 % and 55 %, with a variability of not more than  $\pm 5$  % over 24 h. Verify that the mean temperature has been between 20 °C and 25 °C with a variability of not more than  $\pm 2$  °C over 24 h.
- Record the RH and temperature of the equilibration chamber on the laboratory data sheet.
- Condition the filters for a minimum period of 24 h and until mass changes between successive weighings are less than 6 µg for 47 mm filters.

### 8.2.2 Post-sampling filter equilibration and storage

Post-sampling filter equilibration and storage is as follows.

- Equilibrate filters for at least 24 h.
- Weighing should be completed within 240 h (10 d) after the end of sampling, unless the filter is maintained at 4 °C or below during the entire time between retrieval from the sampler and start of the conditioning, in which case the period shall not exceed 30 d.

If weighing was not completed within 240 h and the filters were to be maintained at 4 °C as required in the preceding during the time between retrieval from the sampler and start of the conditioning, verify that the container has been continuously maintained at 4 °C or below using a min/max thermometer or other suitable device placed in the container at the time of shipment or by inspecting the cool packs to determine if they are still frozen or cold. If the temperature is between 4 °C and 25 °C, but is below the maximum temperature that was recorded during the filter sampling period, assess the temperature and note other relevant information and evaluate the need to determine a shorter analysis deadline for the sample(s).

- Verify that the filter chamber mean RH has been maintained as specified in [8.2.1](#). Record the RH and temperature of the equilibration chamber on the laboratory data sheet.
- If the mean RH and/or temperature variation exceeds the specification in [8.2.1](#), repeat the equilibration period.

### 8.2.3 Gravimetric pre- and post-sampling filter weighing

#### 8.2.3.1 Gravimetric pre- and post-sampling filter weighing is as follows.

- Before each weighing series, verify the balance calibration using standard masses. The masses of the standards should bracket the masses of the filters being measured.
- Suitably mark the filter container and laboratory data form to identify each filter.
- Weigh the filter on a suitable balance. Both the pre- and post-sampling filter weighings should be performed on the same analytical balance. Use smooth, non-serrated forceps to handle filters (see [8.2.3.2](#) and [8.2.3.3](#)).
- Observe all weighing procedures. Perform all quality control (QC) checks.
- Repeat weighing in accordance with weighing procedures described in the preceding.
- Weigh at least two reference filters within 4 h of sample filter weighings. If the average mass of the reference filters changes between sample filter weighings by more than 5 % of the recommended minimum filter loading, then flag the data and report the deviation in reference filter masses.

**8.2.3.2** Because of the greater sensitivity needed for measuring microgram-range masses or mass differences, microbalances are vulnerable to relatively small changes in physical environmental conditions, such as vibration, electrostatic charge buildup, temperature, atmospheric pressure, or RH. Features to offset the effect of these variables on the measurements may be integrated into the design of the microbalances, or they may be offered as options. The following general guidelines should be followed to control environmental factors that may affect microbalance performance:

- a) Select a room that can ensure the RH is maintained between 45 % and 55 %, and the temperature is maintained between 20 °C and 25 °C.
- b) Locate the microbalance away from potential sources of draughts such as doors, windows, aisles with frequent traffic, ventilation ducts, and equipment with fans or moving parts.
- c) If these arrangements are not possible, isolate the microbalance from such equipment by placing a stabilizing slab under the microbalance and/or by placing composite vibration damping pads at three points under the legs of the microbalance or under the stabilizing slab. Placing the pads at only three points eliminates any possible rocking associated with pads placed at four points.
- d) Ensure that the base of the microbalance is sufficiently level to permit levelling of the microbalance according to the manufacturer's instructions. The filter weighing chamber shall be free of any ambient contaminants (such as dust) that could settle on filters during stabilization.

**8.2.3.3** Passive deposition of dust on sample filters prior to and following sampling can bias PM measurements. Minimized this effect by loading and unloading filter housings in the laboratory under controlled conditions and the use of the prescribed filter cassettes. Gases may be collected on sampling filters or on particles captured by the filter creating an artefact in the filter mass. Adsorption of organic gases on quartz filters and/or the sample is a known artefact that can contribute to significant positive bias in filter mass. The oxidation of acid gases (e.g. SO<sub>2</sub>, NO<sub>2</sub>) or retention of gaseous nitric acid on the surface of the filter also can contribute to positive bias in filter mass. Minimize these effects by the use of PTFE membrane filters that are less prone to gas adsorption than quartz fibre filters. Measurement artefacts due to filter handling can be reduced or eliminated by using an inertial mass measurement technique because filters are not handled during the mass measurement process.

## 9 Sampling procedures

### 9.1 Basic sampling method and dilution sampling method

Both the basic and dilution sampling systems incorporate the in-stack PM<sub>10</sub> and PM<sub>2,5</sub> sampling cyclones. Procedures associated with the use of these cyclones are common to both sampling trains.

Sampling procedures specific to the basic sampling train are given in 9.4. Sampling procedures specific to the dilution sampling system are given in 9.6.

### 9.2 Preparation

Preparation procedure is as follows.

- Clean (brush, rinse, and dry), calibrate, and check all the equipment before the first run of a new test. Exercise care not to reuse any part of a sampling train from a previous test programme without a thorough cleaning.
- Prepare the filters to be weighed for each sample run. This includes filters for the blank tests and additional filters to accommodate process and equipment malfunctions.
- If using filters for the gravimetric measurement of particulate mass prepare them according to 8.2.
- Perform the weighing procedures according to 8.2 and load the filters into the filter cassettes.
- Protect all the weighed filters and the other parts or equipment that come into contact with the sample from contamination during transportation and storage. Store and transport loaded filter cassettes in the filter cassette containers and insulated shipping containers.

### 9.3 Pre-sampling measurements and calculations

These pre-test calculations are necessary to select the appropriate sampling rate through the two in-stack cyclones.

- a) Determine the stack sampling location and traverse points as per criteria given in ISO 9096. Do not sample at locations where cyclonic flow is present.
- b) Calculate the probe/cyclone blockage. The cross-sectional diameter of the probe assembly should be 3 % or less of the cross-sectional area of the duct or stack at the sampling location. If not, choose another sampling location.
- c) Pre-sampling measurements. Determine the following:
  - stack gas temperature;
  - pressure;
  - moisture content;
  - CO<sub>2</sub> and O<sub>2</sub> content in accordance with ISO 12039.
- d) Average flue gas velocity:
  - use an S-type Pitot tube with an attached Pt 100 platinum resistance thermometer or other temperature sensor to conduct the traverse [ISO 10780];
  - conduct the preliminary traverse as close as possible to the anticipated testing time if the source exhibits hour-by-hour gas flow rate variations of  $\pm 20$  % or more and/or gas temperature variations of more than  $\pm 10$  °C;

- move the S-type Pitot tube to each traverse point and record the velocity pressures at each point;
- calculate the mean preliminary flue gas velocity using Formula (1)

$$v_s = K_P C_P \left( \sqrt{\Delta p} \right)_{\text{avg}} \left[ \sqrt{\frac{T_s}{p_s M_s}} \right] \quad (1)$$

where

- $v_s$  is the velocity of stack gas (m/s),
- $K_P$  is the Pitot tube equation constant [(m/s)/g/mol K],
- $C_P$  is the Pitot tube coefficient for the as attached to the cyclone probe,
- $\Delta p$  is the Pitot tube velocity pressure differential,
- $T_s$  is the absolute stack gas temperature (K),
- $p_s$  is the absolute stack gas pressure (Pa),
- $M_s$  is the molecular mass of stack gas;

- use this information to select the appropriate nozzle, Formula (2)

$$D = \left[ \frac{3,065 \times q_{Vs}}{v_s} \right]^{0,5} \quad (2)$$

where

- $D$  is the inner diameter of sampling nozzle mounted on PM<sub>10</sub> cyclone (cm),
- $q_{Vs}$  is the sampling rate for PM<sub>10</sub> cyclone necessary to achieve specified  $D_{50}$  (acmm),
- $v_s$  is the velocity of stack gas (m/s).

## 9.4 Basic sampling system — sampling procedures

### 9.4.1 Assembly

Assemble the basic sampling system in an area free of potential contaminants. A schematic of the sampling train is shown in [Figure 1](#).

- a) Attach the pre-selected nozzle on to the main body of the PM<sub>10</sub> cyclone using PTFE tape.

NOTE PTFE O-rings have a temperature limit of approximately 205 °C. Use cyclones with stainless steel sealing rings when stack temperatures exceed 205 °C.

- b) Do not contaminate the sampling train during preparation and assembly. Keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin. Keep the nozzle covered to protect it from nicks and scratches.
- c) Attach the pre-selected filter holder to the end of the combined cyclone sampling head, with the sampling filter placed in the holder. Condition and weigh the filter as specified in [8.2](#). Using forceps and clean surgical gloves, place and centre, a labelled pre-weighed filter into the filter holder. Properly place the filter holder gasket so that the sample gas stream does not circumvent the filter. Check the filter for tears after the assembly is completed. Then screw the filter housing together to seal the filter holder.

- d) Attach the S-type Pitot tube to the combined cyclones after the sampling head is fully attached to the end of the probe.

The Pitot tube tip shall be mounted slightly beyond the combined cyclone assembly and at least 25 mm from the cyclone nozzle. When calibrating the Pitot tube to obtain the  $C_p$  value, calibrate the tube as mounted on the sampling head.

- e) Place one dry modified Greenburg-Smith impinger, followed by two modified Greenburg-Smith impingers each containing 100 ml of distilled  $H_2O$ , followed by an impinger containing silica gel, in the sampling train impinger box ([Figure 1](#)).

## 9.4.2 Leak check

### 9.4.2.1 Sampling train, pretest the entire sampling train for leaks.

The pretest leak check shall have a leak rate of not more than 2 % of the average sample flow during the test run (whichever is less). Additionally, conduct the leak check at a vacuum equal to or greater than the vacuum anticipated during the test run.

### 9.4.2.2 Pitot tube assembly, after leak checking the sample train, leak check the Pitot tube assembly.

## 9.4.3 Sampling head preparation

### 9.4.3.1 Combined sampling head

Preheat the combined sampling head to the stack temperature of the gas stream at the test location ( $\pm 10$  °C) to prevent flue gas moisture from condensing in the head.

Record the site atmospheric pressure and stack pressure.

### 9.4.3.2 Unsaturated stacks

Complete a passive warm up before sampling, to avoid internal condensation of the probe head within the stack for 30 min to 40 min.

NOTE Unsaturated stacks do not have entrained droplets and operate at temperatures above the dew point of the stack gas.

### 9.4.3.3 Shortened warm-up for unsaturated stacks

The warm-up time can be shortened by thermostated heating of the filter outside of the stack (e.g. using a heat gun or oven).

After preheating the sample head outside of the stack, insert it into the stack and allow the temperature to equilibrate.

## 9.4.4 Sampling and sampling train operation

### 9.4.4.1 Portholes

Clean the portholes prior to the test run to minimize the chance of collecting non-representative flue gas particulate matter.

### 9.4.4.2 Traverse points

Determine the minimum number of traverse points as per ISO 9096.

If it can be shown that the mean particle size is less than 5  $\mu m$ , single point sampling is allowed.

### 9.4.4.3 Traverse point sampling time

Determine the minimum total projected sampling time ( $t_r$ ) necessary to collect sufficient particulate matter to achieve the data quality objectives.

#### 9.4.4.4 Sampling time

Calculate the sampling time ( $t_n$ ) at each sampling point to ensure that a velocity weighted average is obtained over all of the traverse points.

Vary the sampling time at each traverse point proportionately with the velocity at each traverse point. Using the data from the preliminary traverse, calculate the sampling time at the first sampling point.

$$t_1 = \left[ \frac{\sqrt{\Delta p_1}}{(\sqrt{\Delta p})_{\text{avg}}} \right] \left( \frac{t_r}{N_{\text{tp}}} \right) \quad (3)$$

where

$t_1$  is the sampling time at point 1 (min);

$\Delta p_1$  is the velocity pressure differential at point 1;

$(\sqrt{\Delta p})_{\text{avg}}$  is the average of square roots of the velocity pressure differentials measured during the preliminary traverse;

$t_r$  is the total projected run time (min);

$N_{\text{tp}}$  is the number of iterative steps.

After obtaining the value for  $t_1$ , for each succeeding traverse point, use the actual  $\Delta p$  value at that point to obtain the subsequent sampling time at point  $n$  as follows:

$$t_n = t_1 \left( \frac{\sqrt{\Delta p_n}}{\sqrt{\Delta p_1}} \right) \quad (4)$$

where

$t_n$  is the sampling time at point  $n$  (min);

$t_1$  is the sampling time at point 1 (min);

$\Delta p_n$  is the velocity pressure differential at point  $n$ ;

$\Delta p_1$  is the velocity pressure differential at point 1.

Adjust the preliminary velocity data for any differences in Pitot tube coefficients ( $C_p$ ) between the Pitot tube used for the preliminary traverse and the Pitot tube which is part of the cyclone sampling assembly. Use Formula (5) for the adjustment:

$$\Delta p_{s1} = \Delta p_m \left( \frac{C_p}{C'_p} \right)^2 \quad (5)$$

where

$\Delta p_{s1}$  is the velocity pressure adjusted for combined cyclone Pitot tube;

$\Delta p_m$  is the measured velocity pressure;

$C_p$  is the Pitot tube coefficient for tube attached to sampling apparatus;

$C'_p$  is the Pitot tube coefficient for Pitot tube used in preliminary traverse.

Ducts or stacks with diameters between 0,5 m and 0,6 m have blockage effects for the typical cyclone sampling probe. When conducting this method on small ducts, the observed velocity pressures shall be adjusted using an estimated blockage factor whenever the sampling apparatus blocks more than 3 % of the stack or duct cross-section.

To correct for blockage, compute the average probe blockage factor.

$$B_f = \frac{12}{A} \quad (6)$$

where

$B_f$  is the average blocking factor;

$A$  is the area of stack or duct at the sampling location (cm<sup>2</sup>).

The velocity pressure  $\Delta p$  values are then corrected as follows:

$$\Delta p_{s2} = \Delta p_{s1} \left[ \frac{1}{(1 - B_f)} \right]^2 \quad (7)$$

where

$\Delta p_{s2}$  is the velocity pressure corrected for blockage;

$\Delta p_{s1}$  is the velocity pressure adjusted for combined cyclone Pitot tube;

$B_f$  is the average blocking factor.

#### 9.4.4.5 Flow rate

Setting the sampling system flow rate. See [Annex D](#).

#### 9.4.4.6 Pre-check

Verify that the filter and probe heating systems are at the proper temperature, remove the nozzle cap and verify that the Pitot tube and probe are properly positioned.

#### 9.4.4.7 First traverse point

Position the nozzle at the first traverse point with the tip pointing directly into the gas stream.



Immediately start the sample pump and adjust the flow to the isokinetic sampling rate previously calculated ( $q_{VS}$ ). Maintain the flow rate throughout the run, provided that the stack temperature is within  $\pm 28^\circ\text{C}$  of the temperature used to calculate the sampling rate. If the stack temperature varies by more than  $\pm 28^\circ\text{C}$ , recalculate the sampling rate and adjust accordingly. Sample for the period calculated in [9.4.4.4](#).

**NOTE** If the stack static pressure is more negative than 1,2 kPa (12 cmH<sub>2</sub>O), adjust the sample pump vacuum to the calculated sample flow rate after performing the leak check and just prior to insertion to prevent back pressure from rupturing the sample filter.

#### **9.4.4.8 Stack traverse**

Traverse the stack cross-section at points specified by ISO 9096.

Do not bump the cyclone nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes. Do not leak check the sampling system during port changes.

#### **9.4.4.9 Recording data**

Record the initial dry gas meter reading and then take dry gas meter readings at the beginning and end of sampling at each traverse point, and when sampling is halted.

#### **9.4.4.10 Box pressure drop across the orifice meter**

Keep the box pressure drop across the orifice meter ( $\Delta H$ ) at the value calculated for the stack temperature that is observed during the test.

Record at the point-by-point data and other source test parameters.

#### **9.4.4.11 Ending the sampling run**

Maintain the flow through the sampling system at the last sampling point.

Remove the sampling train from the stack while it is still operating (running stop). Then stop the pump and record the final dry gas meter reading. Remove the combined cyclone and filter assembly from the port. Keep the sampling head level to avoid tipping particulate matter from the cyclone cups into the filter assembly or downcomer tubing. After cooling, and when the probe can be safely handled, wipe off all external surfaces near the cyclone nozzle and cap the inlet to the cyclone.

#### **9.4.4.12 Cyclone and filter sampling head**

Remove the combined cyclone and filter sampling head from the probe.

Cap the outlet of the filter housing to prevent particulate matter from entering the assembly

#### **9.4.4.13 Post-leak check**

After the sample head (combined cyclone and filter assembly) is removed from the stack, perform a post-test leak check of the remainder of the sample train.

Conduct the leak check at a vacuum greater than or equal to the maximum vacuum achieved during the test run. If the leak rate of the sampling train (without the combined sampling head) exceeds 0,000 57 m<sup>3</sup>/min or 4 % of the average sampling rate during the test run (whichever is less), the run is invalid.

### **9.4.5 Total sample volume**

Calculate and correct to standard conditions, the dry gas volume of the flue gas sample measured by the dry gas meter of the sampling train. See E.5.



#### 9.4.6 Impinger recovery

Measure the liquid in the first impingers to within 1 ml using a clean graduated cylinder or by weighing it to within 0,5 g using a balance. Record the volume of the liquid or mass of the liquid collected to determine the moisture content of the flue gas.

If the water from the cold impinger used for moisture collection is weighed in the field, it can be discarded after weighing. Otherwise, transfer the liquid into a clean sample bottle and mark the liquid level on the bottle. Because the gain in mass is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If the silica gel has been weighed in the field to measure water content, it can be discarded.

Transfer the silica gel to its original container and seal. Weigh the silica gel to within 0,5 g. If analysing the impinger contents for condensable materials, leave the silica gel in the impinger for recovery later.

#### 9.4.7 Particulate matter recovery

Particulate matter is recovered quantitatively from the sampling cyclones, filter, and sample lines. Use a nylon or PTFE brush and acetone to recover particles from the sampling probe. Have available five separate containers and perform the following procedures for each container.

- Container No. 1 — Filterable particulate matter  $\leq 2,5 \mu\text{m}$ . Quantitatively remove the filter from the filter holder into a Petri dish identified as container No. 1. Using a dry nylon bristle brush and/or a sharp-edged blade, carefully transfer to the Petri dish any particulate matter and/or filter fibres that adhere to the filter holder gasket or filter support screen. This container holds particles  $\leq 2,5 \mu\text{m}$  that were caught on the in-stack filter.
- Container No. 2 — Filterable particulate matter  $> 10 \mu\text{m}$ . Quantitatively recover the particulate matter from the  $\text{PM}_{10}$  cyclone cup. Brush and rinse with acetone, the internal surface of the nozzle, the internal surface of the cyclone, and the outside surface of the nozzle downcomer. Keep any dust found on the outside of the cyclone and cyclone nozzle external surfaces, out of the sample. Rinse the particulate matter into container No. 2. Seal the container and mark the liquid level on the outside of the container. This container holds particulate matter  $> 10 \mu\text{m}$  in diameter.
- Container No. 3 — Filterable particulate matter  $> 2,5 \mu\text{m}$  and  $\leq 10 \mu\text{m}$  in diameter. Quantitatively recover particulate matter from the  $\text{PM}_{2,5}$  cyclone cup using a brush and acetone. Brush and rinse with acetone the cyclone cup, the inside of the downcomer, and interior surfaces. Rinse the particulate matter into container No. 3. Seal the container and mark the liquid level on the outside. This container holds particulate matter  $> 2,5 \mu\text{m}$  and  $\leq 10 \mu\text{m}$  in diameter.
- Container No. 4 — Filterable particulate matter  $\leq 2,5 \mu\text{m}$  from the exit tube of the  $\text{PM}_{2,5}$  cyclone and front half of the filter holder. Quantitatively brush and rinse with acetone, the exit tube of the  $\text{PM}_{2,5}$  cyclone and the front half of the filter holder in to container No. 4. This container holds particulate matter that is  $\leq 2,5 \mu\text{m}$  in diameter.
- Container No. 5 — Acetone rinse blank. Take 100 ml of the acetone directly from the wash bottle used and place it in container No. 5, labelled acetone rinse blank.

### 9.5 Basic sampling system — analytical procedures

Determine the dry mass of particulate matter from the samples collected.

- Container No. 1 — Filterable particulate matter  $\leq 2,5 \mu\text{m}$ . Transfer the filter and any loose particulate matter from the sample container to a tared glass weighing dish. Condition the filter and sample following the procedures given in [8.2](#). Weigh to a constant mass, and report the results to the nearest 0,1 mg. For the purposes of this subclause, the term “constant mass” means a difference of no more than 0,5 mg or 1 % of the total mass less the tare mass, whichever is greater, between two consecutive weighings. Consecutive weighings should be no less than 6 h apart.

- Container No. 2 — Filterable particulate matter >10 µm. Separately treat the sample in this container in a similar fashion to that in container No. 1.
- Container No. 3 — Filterable particulate matter >2,5 µm and ≤10 µm in diameter. Separately treat the sample in this container in a similar fashion to that in container No. 1.
- Container No. 4 — Filterable particulate matter ≤2,5 µm from the exit tube of the PM<sub>2,5</sub> cyclone and front half of the filter holder. Note the level of liquid in the container, and confirm whether leakage occurred during transport. If leakage occurred, either void the sample or correct the final results. Quantitatively transfer the contents of the container to a tared 250 ml beaker and evaporate to dryness at ambient temperature and pressure. Condition the sample as per procedures given in 8.2 and weigh to a constant mass.
- Container No. 5 — Acetone rinse blank. Transfer 100 ml of the acetone blank to a clean 250 ml beaker. Evaporate the acetone at room temperature and pressure to approximately 10 ml. Quantitatively transfer the beaker contents to a 50 ml preweighed tin, and evaporate to dryness at room temperature and pressure. Condition the sample as per procedures given in 8.2 and weigh to a constant mass.
- Sample train impinger water. If the amount of water in the impingers has not been determined in the field, note the level of liquid in the container and determine if leakage occurred during transport. If leakage occurred, void the sample. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0,5 g.

The volume of water vapour corrected to standard conditions is:

$$V_{w,std} = 0,407 V_c \quad (8)$$

where

$V_{w,std}$  is the volume of water vapour (standard m<sup>3</sup>, scm);

$V_c$  is the volume of water captured in impingers and silica gel (ml).

and the moisture content of the flue gas is:

$$B_{ws} = \left[ \frac{V_{w,std}}{V_{m,std} + V_{w,std}} \right] \quad (9)$$

where

$B_{ws}$  is the moisture fraction of flue gas;

$V_{w,std}$  is the volume of water vapour (scm);

$V_{m,std}$  dry gas meter volume sampled, corrected to standard conditions (dry standard m<sup>3</sup>, dscm).

- Silica gel absorbent. If the mass of the silica gel has not been determined in the field, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0,5 g.

## 9.6 Dilution sampling system — sampling procedures

### 9.6.1 Dilution sampling system

The dilution sampling system is typically fitted with a PM<sub>2,5</sub> cyclone, with backup filter (Figure 2) located after the residence chamber. Other particle-measuring devices, not a part of this International Standard, may be used either in place of or parallel to the cyclone for alternative measurements.

## 9.6.2 Assembly leak check

Assemble the dilution sampling system ([Figure 2](#)) in an area free of potential contaminants. Immediately before and after each test run, check for leaks in the in-stack cyclone, sample probe, and sample flowmeter assembly and in the entire dilution sampler assembly. If using tracer gas procedures to determine the dilution ratio then only that portion of the sampling system upstream of the first tracer sensor location shall be leak checked.

### 9.6.2.1 Pre-test leak check, for components that normally operate under negative pressure.

It is important to avoid any leakage of ambient air or other sources of contamination into the sampling system. Most systems withdraw stack gas sample by inducing flow with a pump, in which case the stack gas sample in the components upstream of the pump is under negative pressure. All components that normally operate under negative pressure shall be leak checked. For systems in which the entire dilution system operates under negative pressure, this leak check shall be performed in two steps: first, the probe and components that are in contact with the undiluted sample alone, and second the entire system.

### 9.6.2.2 Pre-test leak check, undiluted sample components.

Temporarily seal the in-stack cyclone sample nozzle and draw a vacuum on the cyclone, sample probe, and sample flowmeter assembly equal to the maximum vacuum experienced during a run and measure the sample flow (leak) rate for a period of approximately 5 min. If the pre-run leak rate is greater than 2 % of the target sample flow rate determined in D.4, identify the source of the leak and eliminate or reduce it until the leak rate is equal to or less than 2 % of the target sample flow rate. If the leak rate after the test run is greater than 2 % of the actual average sample flow rate during the run, the results of that run may not be valid and shall be flagged in the report.

### 9.6.2.3 Pre-test leak check, entire system.

Attach the in-stack cyclone, sample probe, and sample flowmeter assembly to the dilution sampler. Temporarily seal the cyclone sample nozzle, dilution air inlet, and diluted sample bypass. Then draw a vacuum on the entire assembly equal to the maximum vacuum experienced during a run and measure the sample flow (leak) rate for a period of approximately 5 min. If the leak rate before the test run is greater than 2 % of the total flow through the dilution chamber during a run, then the source(s) of the leak(s) shall be identified and eliminated until the leak rate is equal to or less than 2 % of the total flow through the dilution chamber. If the leak rate after the test run is greater than 2 % of the total flow through the dilution chamber during the run, the results of that run may not be valid and shall be flagged in the report.

### 9.6.2.4 Pitot tube traverse

Conduct a stack gas Pitot tube traverse per ISO 10780 for determination of stack gas velocity and flow rate.

If sampling is being performed at a single point for PM<sub>2,5</sub> in the stack gas, conduct Pitot tube traverses immediately before and after the test run and measure the velocity head with the Pitot tube at the single sampling point continually during the test run. Calculate the ratio of the average velocity over the full traverse to the velocity at the single sampling point based on the pre- and post-run Pitot tube traverses. Calculate the average stack gas velocity at the single sampling point during the test run based on the velocity head measurements during sampling, and multiply this by the average-to-single point velocity ratio determined from the pre- and post-run traverses. Use the resulting corrected velocity to calculate average stack gas flow rate during the test run.

If sampling is being performed at multiple traverse points for PM<sub>10</sub> in the stack gas, conduct the Pitot tube traverse during sampling.

Alternatively, for combustion sources, the stack gas flow rate may be measured by stack gas oxygen, fuel flow and fuel composition measurement according to ISO 8178-1:2006, Annex A, or other equivalent method.

### 9.6.3 Sampling

Perform the sampling procedure as follows.

- Place the sample probe nozzle at the first sampling point.
- Preheat the stack gas sample probe to the specified temperature. Avoid contacting the nozzle with any deposits on surfaces of the access port or stack walls.
- Seal the opening of the access port to minimize air in-leakage or exposure of operators to toxic gases.
- Turn the pump on to start the dilution air flow.
- After the minimum probe temperature has been achieved, start the stack gas sample flow. Adjust the dilution air flow rate to achieve a dilution ratio of at least 20:1. Higher dilution ratios should be used if required to completely eliminate water condensation or to lower the temperature of the diluted stack gas to less than or equal to 42 °C at the cyclone(s) upstream of the filter holders. Adjust the dilution ratio to achieve the maximum humidity and maximum temperature criteria. Adjust the temperature of dilution air if needed. Adjust the humidity of the dilution air, if necessary, to ensure that the RH is not higher than 70 %.
- Verify the dilution ratio. Document the method chosen to verify the dilution ratio in the test report and show the calculations used to determine the ratio. Methods may include the following:
  - 1) direct measurement of flows (stack gas sample, dilution air, total diluted stack gas sample; at least 2 of the 3 needed), or
  - 2) measurement of CO<sub>2</sub>, NO<sub>x</sub>, VOC, or other appropriate tracer in the stack gas sample, dilution air, total diluted stack gas sample and calculation of dilution ratio by mass balance, or
  - 3) from carbon balance by measuring CO<sub>2</sub>, fuel composition, and flow according to ISO 8178-1:2006, Annex A.
- After the dilution tunnel temperature has stabilized to  $\pm 2$  °C for 3 min, turn on the filter sample system pump and adjust the flow to the target rate. The filter face velocity should not exceed 100 cm/s and the pressure differential across the filter should not exceed 7,4 kPa (75 cmH<sub>2</sub>O).
- Continue to collect the sample for the target duration. Target sampling durations vary depending on process conditions, the concentration of particulate matter in the stack gas, and the test objectives for minimum detection and quantification limits. Sampling durations of 30 min to 60 min may be sufficient for most continuous processes. Batch processes may require sampling for one or more process cycles. Record dilution tunnel and sample system operating conditions (temperature, flow rates, humidity, dilution ratio, pressure differential across the sample filter) a minimum of 12 times during a test run, or more often if needed, to capture expected process variations. The data collection should be conducted at equally spaced intervals during each test run.
- At the end of the run, record the final dilution system and sample filter operating data. Turn off the sampling pump and record the final sample volumes. Shut down the dilution sampling system. Perform the post-run leak checks.
- Recover the sample filter cassette with filter intact, place the filter cassette in the filter cassette holder and seal tightly, and affix a sample label with a unique number to each sample. Record sample information on a suitable sample tracking or chain of custody form. Pack the samples in the insulated shipping and storage container for shipment or transfer to the analytical laboratory. Store and transport samples in accordance with [6.2.4.5](#).

- Inspect the sample lines for condensed material, record any observations.

### 9.7 Dilution sampling train — recovery of deposits from the in-stack cyclones

Follow the procedures given in [9.4.7](#) above for the basic sampling train.

### 9.8 Dilution sampling train — recovery of deposits upstream of the filter

These procedures are conducted for the PM<sub>2,5</sub> cyclone located after the dilution chamber, in the residence time chamber shown in [Figure 2](#).

Depending on the test objectives and whether PM<sub>10</sub> and/or PM<sub>2,5</sub> in-stack cyclones are used, recovery may involve the quantitative transfer of particles in the following size ranges:

- larger than 10 µm;
- smaller than or equal to 10 µm but larger than 2,5 µm;
- smaller than or equal to 2,5 µm.

Use a nylon brush and an ultrapure acetone rinse to recover particles from the cyclone sampling head, sample probe, and sample gas flowmeter. Keep any dust found on the external surfaces out of the samples. Minimize the volume of acetone used for recovery to minimize the influence of acetone blanks.

- Container No. 1d. Quantitatively recover the: a) particulate matter from the PM<sub>10</sub> cyclone cup and acetone rinses (and brush cleaning) of the cyclone cup; b) internal surface of the nozzle; and c) container and mark the liquid level on the outside of the container. This container holds particulate matter larger than 10 µm.
- Container No. 2d. Place the solids from the PM<sub>2,5</sub> cyclone cup and the acetone (and brush cleaning) rinses of the PM<sub>2,5</sub> cyclone turnaround cup (above inner downcomer tube), inside of the downcomer tube, and interior surfaces of the PM<sub>2,5</sub> cyclone into the container. Seal the container and mark the liquid level on the outside. This container holds particulate matter smaller than 10 µm and larger than 2,5 µm.
- Container No. 3d. Retrieve the acetone rinses (and brush cleaning) of the exit tube of the PM<sub>2,5</sub> cyclone, the sample probe, the sample gas flowmeter, and all surfaces downstream of the cyclones in contact with the undiluted sample gas into container No. 3. Seal the container and mark the liquid level on the outside of the container. This container holds particulate matter that is smaller than 2,5 µm.

Acetone is the recommended rinse solvent, however, other solvents such as water or methylene chloride may be substituted.

- Container No. 4d. Collect 250 ml of acetone or the same volume as the highest volume used in the rinses of the various fractions, whichever is higher, from the wash bottle used to perform sample recovery rinses into sample container No. 4d. This is the acetone field reagent blank.

### 9.9 Dilution sampling system — analytical procedures

Determine the dry mass of particulate matter from the samples collected, as follows.

- Container No. 1d. Transfer the filter and any loose particulate matter from the sample container to a tared glass weighing dish. Condition the filter and sample following procedures given in [8.2](#). Weigh to a constant mass, and report the results to the nearest 0,1 mg. For the purposes of this subclause, the term “constant mass” means a difference of no more than 0,5 mg or 1 % of the total mass less the tare mass, whichever is greater, between two consecutive weighings. Consecutive weighings should be no less than 6 h apart.
- Container No. 2d. Separately treat the sample in this container the same as specified in [9.5](#).

- Container No. 3d. Separately treat the sample in this container the same as specified in [9.5](#).
- Container No. 4d. Transfer 100 ml of the acetone blank to a clean 250 ml beaker. Evaporate the acetone at room temperature and pressure to approximately 10 ml. Quantitatively transfer the beaker contents to a 50 ml preweighed tin, and evaporate to dryness at room temperature and pressure. Condition the sample as per procedures given in [8.2](#) and weigh to a constant mass.
- Sample train impinger water. If the amount of water in the impingers has not been determined in the field, note the level of liquid in the container and determine if leakage occurred during transport. If leakage occurred, void the sample. Measure the liquid in this container either volumetrically to  $\pm 1$  ml or gravimetrically to  $\pm 0,5$  g.
- Silica gel absorbent. If the mass of the silica gel has not been determined in the field, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0,5 g.

## 9.10 Dilution sampling system — validation of results

Validate the results as follows:

- Testing should not be conducted at an unsuitable location or carried out under fluctuating plant operating conditions. Under such conditions, the representativeness of the sample(s) may be questioned and the uncertainty of the measurement results unknown.
- Include in the test report details of the flow characteristics at the sampling location or on the variations of the flow rate in the stack while sampling, or both.

## 10 Calibration and QA/QC activities

### 10.1 Calibrations

#### 10.1.1 Velocity measurement apparatus

Calibrate the velocity measurement apparatus according to ISO 10780.

#### 10.1.2 Gas flow rate meters

Calibrate stack gas flow rate meters (undiluted stack gas, dilution air, and diluted stack gas) at least every six months or whenever a system repair or change is made that could influence calibration.

Calibrate sampling system flow rate meters at least every three months or whenever a system repair or change is made that could influence calibration. Calibrate the gas flow rate meters with a traceable standard or equivalent. For the sample gas flow meters (both undiluted and diluted), any calibrations exceeding the  $\pm 4$  % limit invalidate all results of tests conducted between calibrations.

NOTE More frequent calibration checks are not required; however, periodic calibration checks such as post test calibrations checks can help ensure valid measurements between the required full calibrations.

#### 10.1.3 Analytical balance

Verify the analytical balance calibration at the beginning of each weighing session.

Conduct calibration verifications using traceable mass standards.

#### 10.1.4 Temperature sensors

Verify the calibration of the temperature sensing devices at least every three months or whenever a system repair or change is made that could influence calibration.



Temperature sensing devices should be calibrated with a traceable standard and agreement should be within  $\pm 2$  °C.

#### **10.1.5 Relative humidity meters**

Verify the calibration of RH meters at least every month or whenever a system repair or change is made that could influence calibration.

Meters should be calibrated with a traceable standard and agreement should be within  $\pm 3$  % of reading.

#### **10.1.6 Pressure sensors**

Calibrate pressure sensors at least every three months or whenever a system repair or change is made that could influence calibration.

The sensors should be calibrated with a traceable standard or equivalent and agreement should be within  $\pm 2$  % of reading.

### **10.2 QA/QC activities — dilution sampling system**

#### **10.2.1 Pre-test dilution sampling system cleaning (before each test condition)**

Wash and rinse all components with acetone and allow to dry completely. Wipe down large surfaces.

#### **10.2.2 Dilution system blank**

Take at least one dilution system blank per test and sampling location. For each test programme, collect at least one dilution system blank by collecting a sample with all openings except for the dilution air inlet plugged so that only dilution air is drawn through the dilution sampler. Analyse the sample in the same manner as the stack gas samples. This provides an indication of total measurement background due to dilution air background and contamination of filters and rinsing solutions during sample handling, transport, and analysis. Calculate the equivalent in-stack concentration using mean values of the dilution ratio and sample volume for the stack samples. The level of significance varies depending on test objectives. Report these results along with the stack sample results.

Optional, but recommended — take at least one ambient air sample per test. Collect the ambient air sample at the sampling location by directly pulling unfiltered ambient air through a PM<sub>10</sub> or PM<sub>2.5</sub> cyclone head (or equivalent) and through the sampling media only. Conduct sampling for the same duration as stack samples or longer depending on the test objectives. These results may be used in conjunction with the dilution system blank to evaluate the dilution air filtration system performance.

#### **10.2.3 Field blank**

Field blanks should be transported to the sampling site, momentarily installed in the sampler, removed, and stored in their protective containers, inside the case of the sampler at the sampling site until the exposed filters are retrieved for post sampling weighing. Field blanks should be collected at the start of the test series. At least one field blank should be collected for each test series.

#### **10.2.4 Pre-run and post-run leak checks**

Conduct leak checks as specified in this International Standard.

#### **10.2.5 Dilution ratio check**

If the dilution ratio is determined by tracer methods, this check is not required. If the dilution ratio is determined by direct flow rate measurements, this check is recommended to verify the calibration and proper operation of the system. Perform a dilution ratio check before the start of each run using an appropriate method, such as by measuring CO<sub>2</sub>, NO<sub>x</sub> or other tracer concentrations in the raw stack

gas sample and diluted stack gas sample in accordance with ISO 8178-1. The measured dilution ratio determined by each of the two methods should agree within 10 % relative percentage difference. If the pre-test dilution ratio check is not within 10 % of the measured value, correct the error before starting the test. Include the results of the dilution ratio checks in the final report.

#### **10.2.6 Mixing**

Verify that the gases in the dilution sampling system are uniformly mixed under the test conditions to within 10 % of the mean concentration at all points. Mixing may be verified by measuring the CO<sub>2</sub> or other tracer gas profile upstream of the residence time section, or any sample collection media, at a minimum of four points across the dilution sampler cross-sectional diameter. Alternately, the degree of mixing may be warranted by the equipment manufacturer or vendor using these procedures.

#### **10.2.7 Gravimetric weighing QA/QC**

After approximately every 10th filter, re-zero the microbalance and reweigh at least one working standard. Weigh approximately 10 % laboratory blanks per weighing session. Reweigh one replicate filter at the end of the weighing session. Weigh approximately 10 % of the field blanks.

#### **10.2.8 Working standard measurements**

The working standard measurements should agree within 3 µg of the verified values. If the working standard measurements do not agree within this limit, recalibrate the balance, or use an alternate calibrated balance, and reweigh all filters since the last successful working standard check.

#### **10.2.9 Laboratory blank and replicate measurements**

Laboratory blank and replicate measurements should agree within 15 µg. If laboratory blanks or replicate measurements do not agree within this limit, determine and correct the source of the discrepancy and reweigh all filters since the last successful laboratory blank and replicate check.

#### **10.2.10 Gravimetric filter handling**

Careful handling of the filter during sampling, conditioning, and weighing is necessary to avoid measurement errors due to damaged filters or a gain or loss of collected particles on the filters.

- Whenever filters are handled, the analyst should wear antistatic, powder-free gloves; these gloves act as an effective contamination barrier. Gloves that are packed in a box can carry an electrostatic charge. It is a good practice to discharge them by touching a good electrical earth after putting them on.
- The filters should be handled carefully by the support ring, rather than by the filter material, with smooth, non-serrated forceps that are used only for that purpose. Mark these forceps to distinguish them from the forceps used to handle mass reference standards. Forceps should be cleaned with alcohol and lint-free wipes before handling filters and then allowed to air dry. These precautions reduce the potential effect from body moisture or oils contacting the filters and subsequently affecting the measured masses.
- If filter loadings are speciated chemically, the possibility of contamination from gloves should be considered. Some gloves have been found to be contaminated with sulfate. If antistatic gloves are used, ensure that ammonium chloride is not a component of the antistatic reagent. Powder-free gloves that are certified to be free of chloride, nitrate, and sulfate are available.
- In the laboratory, each filter should be transferred from its sealed manufacturer's packaging to a clean filter-handling container, such as a glass or polycarbonate Petri dish or Petri slide, to reduce the risk of contamination. The filter should remain in this container, except for weighing, until it is loaded into a filter cassette prior to sampling. Each filter should have a unique identification number.



### 10.2.11 Laboratory blanks

Laboratory blanks should be kept inside the conditioning chamber except during weighing sessions. Weigh enough laboratory blanks during a presampling weighing session to provide at least one single-use laboratory blank during each subsequent post-sampling weighing session. The pre- and post-sampling masses should be recorded in the QC notebook and the laboratory data form or an equivalent database. If the mass change exceeds 15 µg, contamination in the conditioning chamber may be occurring. Take appropriate troubleshooting and corrective actions.

## 10.3 Personnel qualifications

Persons using this test method shall have adequate training and experience in preparing, maintaining and operating the sampling equipment, conducting the test procedures and performing tests at similar sampling locations. It is strongly recommended, but not required, that the testing organization meet the criteria specified in ISO/IEC 17025.

## 11 Additional aspects discussion

### 11.1 Sampling duration and detection limit

In-stack minimum detection limits are dependent upon the greater of analytical uncertainty or dilution system blank levels and upon dilution ratio, sampling duration, sample flow rate, sample filter flow rate, filter face velocity, filter pressure drop, and potentially other factors. Based on the pre-test estimated analytical uncertainty and dilution system blank results, select the sample flow rate, dilution ratio, sample filter flow rate, and sampling duration to achieve in-stack minimum detection limits commensurate with the test objectives.

### 11.2 Particulate deposits upstream of the filter

#### 11.2.1 Errors

Errors can be introduced into the measurements if particulate deposits on the surfaces of the sampling system in contact with the sample upstream of the filter are not recovered quantitatively. Such errors are more significant in the portions of the dilution sampler in contact with the undiluted stack gas sample.

#### 11.2.2 Particulate deposits

Particulate deposits are dependent upon the design of the sampling equipment and the characteristics of the particulate matter to be sampled. Therefore, all parts of the sampler in contact with the undiluted and diluted sample are to be quantitatively recovered to collect the particulate matter by using the rinsing procedures described in [9.4.7](#) and [9.8](#).

#### 11.2.3 Residue

The residue contained in reagent grade acetone (typically less than 1 mg/kg) and other solvents may be significant for some sources with very low particulate matter concentration in the stack gas, such as some natural gas-fired combustion equipment (see F.2 for additional discussion). The analytical uncertainties introduced by the solvent residue have been shown to be much greater than the analytical uncertainty associated with analysing the filters by gravimetric methods. For such sources where the deposits on the internal surfaces downstream of the cyclone(s) are greater than 25 % of the total mass collected and the correction for the residue contained in the solvent rinse is more than half of the deposits on the internal surfaces, flag the data and note the increased uncertainties introduced by the solvent residue.

## 12 Determination of precision and bias

### 12.1 General

A user has the obligation and responsibility to validate any method it uses for a specific application and to provide an estimate of the bias and precision associated with the application. It is recommended that paired sampling tests be conducted to provide data for calculations such as those described as follows.

### 12.2 Parallel sampling

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.

### 12.3 Standard deviation

The standard deviation of the differences can be calculated as follows.

$$s_D = \left[ \frac{\sum (x_1 - x_2)^2}{2n} \right]^{0,5} \quad (10)$$

where

$s_D$  is the standard deviation;

$n$  is the number of sample pairs.

### 12.4 Repeatability

The repeatability,  $r$  (according to ISO 5725[11]), that is the maximum difference between two measurements by the same team, for a 95 % confidence level is:

$$r = t s_D \times 2^{0,5} \quad (11)$$

where

$r$  is the repeatability;

$t$  is the student factor for a 95 % confidence and the degrees of freedom  $n - 1$ ;

$s_D$  is the standard deviation.

### 12.5 Independent tests

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

- the external uncertainty linked to an individual measurement carried out by any team fulfilling the requirements of this International Standard; and
- the reproducibility (according to ISO 5725[11]), that is the maximum difference,  $R$ , that can be expected at a 95 % confidence between two measurements by different teams working according to the standard at the process conditions.

## 12.6 Low concentration measurements

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

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

## 13 Test report

### 13.1 General

Refer to this International Standard (ISO 25597:2013) in the test report and include the information required in [13.2](#) to [13.8](#).

### 13.2 Test purpose

Provide a description of the purpose of the test, identification of the site, date of sampling, and participants in the test programme.

### 13.3 Operating conditions

Provide a description of the operating conditions of the plant process and any variation in operating conditions that may have occurred during the test programme.

### 13.4 Sampling locations

Identify the sampling location(s) and gas parameters in the stack.

- Stack dimensions, number and position of velocity measurement points, and the sampling points.
- Velocity and temperature profiles.
- O<sub>2</sub> and CO<sub>2</sub> (if relevant), humidity concentrations, gas volumes.

### 13.5 Measurement procedures

Give details of the measurement procedures performed, as follows.

- Velocity measurements, if taken, or other stack gas flow rate determinations (calibration of S Pitot tubes, and so forth).
- Characteristics of sampling equipment:
  - 1) make (type, manufacturer) and detailed description of the sampling system,
  - 2) nozzle diameter, characteristic of filter (material, size, type),
  - 3) calibration of flow rate measurement devices,
  - 4) filtration temperature,
  - 5) dilution ratio, and
  - 6) response time.
- Weighing procedures: conditioning temperature and humidity.

### **13.6 Test results**

List the number, date, duration of runs for each test run:

- a) mass of PM collected;
- b) temperature and relative humidity of the fully diluted stack gas sample in the dilution sampling system;
- c) dilution air temperature, relative humidity, and dilution ratio;
- d) sampling duration;
- e) undiluted and diluted stack gas sample volume and flow rate;
- f) initial and final pressure drop across the filter pack;
- g) filter face velocity;
- h) any special circumstances or incidents;
- i) all intermediate and final calculation results; and
- j) raw results and corrected results (standard conditions).

### **13.7 Quality assurance**

Quality assurance applied to the following:

- leak tests results;
- blank values;
- calibration documentation;
- dilution ratio verification.

### **13.8 Comments**

Record the following subjects in the test report:

- the value of the ratio of the measured values to the blanks values;
- any special circumstances that may have influenced the results and any information concerning the uncertainty of the results;
- a modification or deviation, if it was necessary to modify or deviate from this International Standard for any reason.

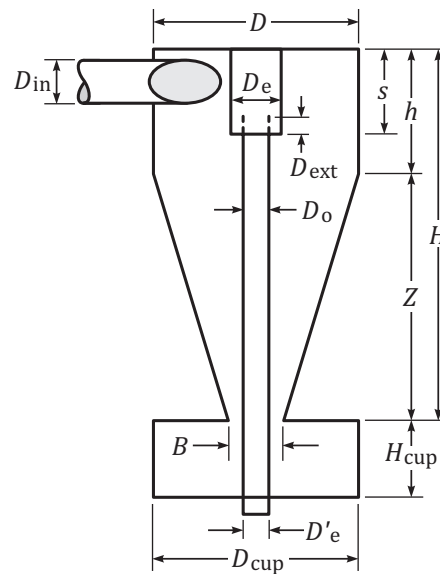
## Annex A (informative)

### Design of the 10 µm and 2,5 µm cyclones

#### A.1 Example design for the 10 µm sampling cyclone

See [Figure A.1](#).

Dimensions in centimetres



#### Key

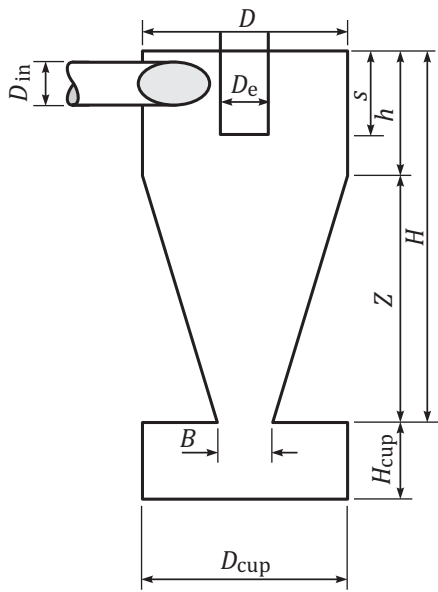
$B$	$1,88 \pm 0,02$	$D_{\text{ext}}$	$0,025 \pm 0,02$	$H_{\text{cup}}$	$2,25 \pm 0,02$
$D$	$4,47 \pm 0,02$	$D_{\text{in}}$	$1,27 \pm 0,02$	$h$	$2,24 \pm 0,02$
$D_{\text{cup}}$	$4,45 \pm 0,02$	$D_o$	$1,24 \pm 0,02$	$s$	$1,57 \pm 0,02$
$D_e$	$1,50 \pm 0,02$	$H$	$6,95 \pm 0,02$	$Z$	$4,71 \pm 0,02$
$D'_e$	$1,02 \pm 0,02$				

**Figure A.1 — Example design specifications for the interior dimensions of a 10 µm sampling cyclone**

#### A.2 Example design for the 2.5 µm sampling cyclone

See [Figure A.2](#).

Dimensions in centimetres



Key

$B$	$1,09 \pm 0,02$	$H$	$2,68 \pm 0,02$
$D$	$2,54 \pm 0,02$	$H_{cup}$	$2,22 \pm 0,02$
$D_{cup}$	$2,62 \pm 0,02$	$h$	$1,03 \pm 0,02$
$D_e$	$0,59 \pm 0,02$	$s$	$0,58 \pm 0,02$
$D_n$	$0,51 \pm 0,02$	$Z$	$1,65 \pm 0,02$

Figure A.2 — Example design specifications for the interior dimensions of a 2,5 µm sampling cyclone

## Annex B (normative)

### Calculation of the $D_{50}$ for the 10 $\mu\text{m}$ and 2,5 $\mu\text{m}$ cyclones

#### B.1 Calculating $D_{50}$ for a 10 $\mu\text{m}$ cyclone

Calculate the actual  $D_{50}$  of the 10  $\mu\text{m}$  cyclone for the given conditions as follows:

$$D_{50} = \beta_1 \left( \frac{T_s}{M_w p_s} \right)^{0,2091} \left( \frac{\eta_s}{q_{Vs}} \right)^{0,7091} \quad (\text{B.1})$$

where

$D_{50}$  is the particle cut diameter ( $\mu\text{m}$ );

$\beta_1$  is 0,027 754 for metric units;

$T_s$  is the absolute stack gas temperature (K);

$M_w$  is the molecular mass of wet gas (g/mol);

$p_s$  is the absolute stack gas pressure (Pa);

$\eta_s$  is the stack gas viscosity ( $\mu\text{P}$ );

$q_{Vs}$  is the sampling rate for PM<sub>10</sub> cyclone necessary to achieve the specified  $D_{50}$  (acmm).

$$M_w = M_d (1 - B_{ws}) + 18,0 (B_{ws}) \quad (\text{B.2})$$

where

$M_d$  is the molecular mass of dry gas (g/mol);

$B_{ws}$  moisture fraction of flue gas

18,0 is the molecular mass of water.

$$q_{Vs} = \frac{T_s}{K_1 p_s} \left( q_{Vs, \text{std}} + \frac{V_{w, \text{std}}}{t_{\text{tr}}} \right) \quad (\text{B.3})$$

where

$q_{Vs}$  is the sampling rate for PM<sub>10</sub> cyclone necessary to achieve the specified  $D_{50}$  (acmm);

$K_1$  is a unit conversion constant;

$q_{Vs, \text{std}}$  is the dry gas sampling rate through the sampling assembly (dscmm);

$V_{w, \text{std}}$  is the volume of water vapour (scm);

$t_{\text{tr}}$  is the total run time (min).



## B.2 Calculating $D_{50}$ for a 2,5 $\mu\text{m}$ cyclone

### B.2.1 Reynolds number <3,162

Calculate the actual  $D_{50}$  of the 2,5  $\mu\text{m}$  cyclone for the given conditions as follows.

$$D_{50} = 0,002\,430\,2 \left( \frac{\eta_s}{q_{Vs}} \right)^{1,1791} \left( \frac{1}{C} \right)^{0,5} \left( \frac{T_s}{p_s M_w} \right)^{0,6790} \quad (\text{B.4})$$

where

- $D_{50}$  is the particle cut diameter ( $\mu\text{m}$ );
- $\eta_s$  is the stack gas viscosity ( $\mu\text{P}$ );
- $q_{Vs}$  is the sampling rate for PM<sub>10</sub> cyclone necessary to achieve specified  $D_{50}$  (acmm);
- $C$  is the Cunningham correction factor for particle diameter calculated using actual stack gas temperature;
- $T_s$  is the absolute stack gas temperature (K);
- $p_s$  is the absolute stack gas pressure (Pa);
- $M_w$  is the molecular mass of wet gas (g/mol).

### B.2.2 Reynolds number >3,162

Calculate the actual  $D_{50}$  of the 2,5  $\mu\text{m}$  cyclone for the given conditions as follows.

$$D_{50} = 0,019\,723 \left( \frac{\eta_s}{q_{Vs}} \right)^{0,8058} \left( \frac{1}{C} \right)^{0,5} \left( \frac{T_s}{p_s M_w} \right)^{0,3058} \quad (\text{B.5})$$

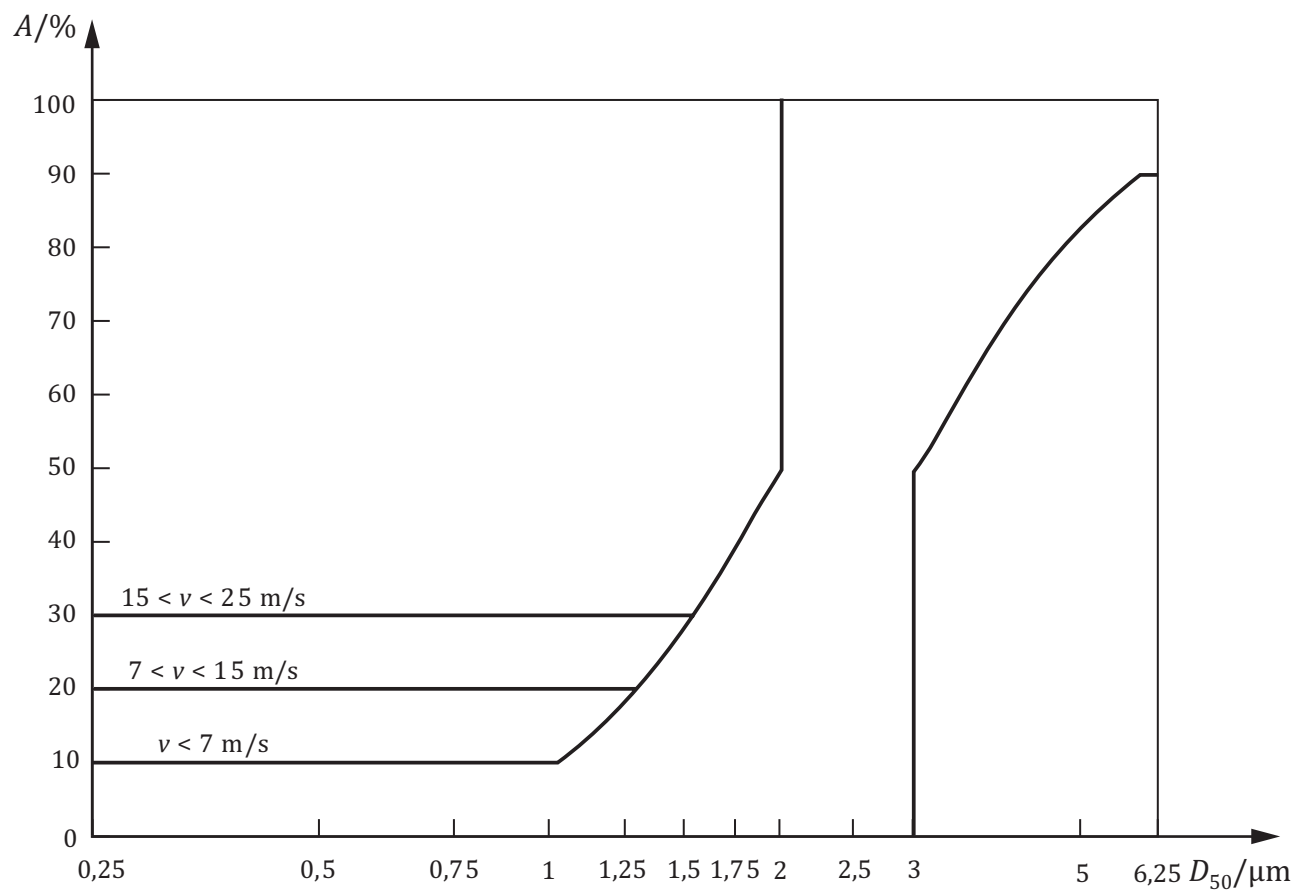
where

- $D_{50}$  is the particle cut diameter ( $\mu\text{m}$ );
- $\eta_s$  is the stack gas viscosity ( $\mu\text{P}$ );
- $q_{Vs}$  is the sampling rate for PM<sub>10</sub> cyclone necessary to achieve specified  $D_{50}$  (acmm);
- $C$  is the Cunningham correction factor for particle diameter calculated using actual stack gas temperature;
- $T_s$  is the absolute stack gas temperature (K);
- $p_s$  is the absolute stack gas pressure (Pa);
- $M_w$  is the molecular mass of wet gas (g/mol).

## B.3 Cyclone separation efficiency

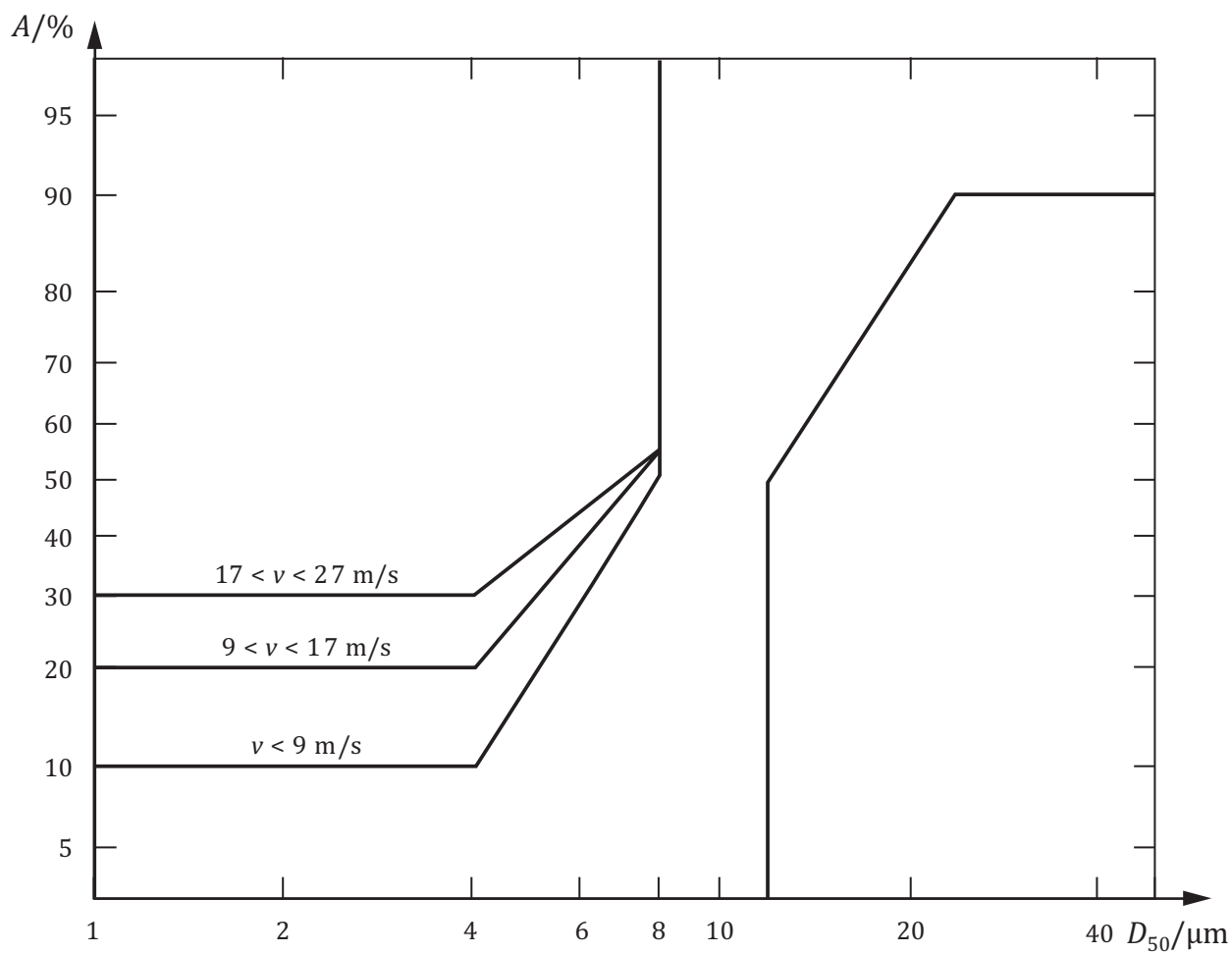
The cyclones for the measurement of PM<sub>10</sub> and PM<sub>2,5</sub> shall be designed such that the separation curves of PM<sub>10</sub> and PM<sub>2,5</sub> have a similar shape separation efficiency of 50 % at 10  $\mu\text{m}$  and 2,5  $\mu\text{m}$  as specified in ISO 7708.

The separation curve for PM<sub>2,5</sub> is shown in [Figure B.1](#) as a plot of the separation efficiency versus the cut diameter. The separation curve for PM<sub>10</sub> is given in [Figure B.2](#).

**Key**

$A$	separation efficiency
$D_{50}$	cut diameter
$v$	velocity

**Figure B.1 — Efficiency envelope for PM<sub>2,5</sub> cyclone samplers**



**Key**

$A$  separation efficiency  
 $D_{50}$  cut diameter  
 $v$  velocity

**Figure B.2 — Efficiency envelope for PM<sub>10</sub> cyclone samplers**

## Annex C (informative)

### Entry nozzle

#### C.1 Choosing the nozzle size —nozzle size calculation

Determine the nozzle size necessary to achieve the target in-stack cyclone particle size cut point(s) and a nozzle sample gas velocity that is within 80 % to 120 % of the average isokinetic rate. Based on the preliminary calculations, the measured stack gas velocity profile and other stack parameters, use Formula (C.1):

$$D = \left[ \frac{212,2q_{Vs}}{v_s} \right]^{0,5} \quad (C.1)$$

where

$D$  is the inner diameter of sampling nozzle mounted on PM<sub>10</sub> cyclone (cm);

$v_s$  is the velocity of the stack gas (m/s);

$q_{Vs}$  is the sampling rate for PM<sub>10</sub> cyclone necessary to achieve specified  $D_{50}$  (acmm).

If measurements are intended for determination of PM<sub>2,5</sub> only, isokinetic sampling is not required. Calculate the resulting velocity of the flue gas through the nozzle:

$$v_n = \frac{q_{Vs}}{60A_n} \quad (C.2)$$

where

$v_n$  is the sample gas velocity in the nozzle (m/s);

$q_{Vs}$  is the sampling rate for PM<sub>10</sub> cyclone necessary to achieve specified  $D_{50}$  (acmm);

$A_n$  is the area of the nozzle (cm<sup>2</sup>).

#### C.2 Choosing the optimum sampling nozzle

##### C.2.1 General

The nozzle selected from B.1 is appropriate if all of the observed velocity pressures during the preliminary velocity traverse fall within the range of  $\Delta p_{\min}$  and  $\Delta p_{\max}$ , as calculated in [C.2.2](#).

### C.2.2 Minimum nozzle, stack velocity ratio parameter and minimum velocity pressure

Calculate the minimum nozzle to stack velocity ratio,  $R_{\min}$

$$R_{\min} = \left\{ 0,245\,7 + \left[ 0,307\,2 - \frac{0,260\,3\eta_s (q_{Vs})^{0,5}}{v_n^{1,5}} \right]^{0,5} \right\} \quad (C.3)$$

where

$R_{\min}$  is the nozzle to stack velocity ratio parameter;

$\eta_s$  is the stack gas viscosity ( $\mu\text{P}$ );

$q_{Vs}$  is the sampling rate for  $\text{PM}_{10}$  cyclone necessary to achieve specified  $D_{50}$  (acmm);

$v_n$  is the sample gas velocity in the nozzle (m/s).

$$\eta = C_1 + C_2\sqrt{T_s} + C_3T_s^{-2} + C_4\varphi(\text{O}_2)_w - C_5B_{ws} + C_6B_{ws}T_s^2 \quad (C.4)$$

where

$T_s$  is the absolute stack temperature (K);

$B_{ws}$  is the moisture fraction of flue gas;

$\varphi(\text{O}_2)_w$  oxygen volume fraction in wet flue gas (%)

$C_1$  is 150,316 2 ( $\mu\text{P}$ );

$C_2$  is 18,061 4 ( $\mu\text{P}/\text{K}^{0,5}$ );

$C_3$  is  $1,191\,83 \times 10^6$  ( $\mu\text{P}/\text{K}^2$ );

$C_4$  is 0,591 123 ( $\mu\text{P}$ );

$C_5$  is 91,972 3 ( $\mu\text{P}$ );

$C_6$  is  $4,917\,05 \times 10^{-5}$  ( $\mu\text{P}/\text{K}^2$ );

NOTE For  $R_{\min} < 0,5$ , take  $v_{\min} = v_n \times 0,5$ . For  $R_{\min} > 0,5$ , take  $v_{\min} = v_n \times R_{\min}$

Calculate the minimum velocity pressure,  $\Delta p_{\min}$

$$\Delta p_{\min} = 1,368\,6 \times 10^{-4} \left[ \frac{p_s M_w}{T_s} \right] \left[ \frac{v_{\min}}{C_p} \right]^2 \quad (C.5)$$

where

$\Delta p_{\min}$  is the minimum velocity pressure;

$p_s$  is the absolute stack gas pressure (Pa);

$M_w$  is the molecular mass of wet gas (g/mol);

$T_s$  is the absolute stack gas temperature (K);

$v_{\min}$  is the minimum gas velocity (m/s);

$C_p$  is the calibration coefficient for the Pitot tube attached to the sampling apparatus.

### C.2.3 Maximum nozzle, stack velocity ratio parameter and maximum velocity pressure

Calculate the maximum nozzle to stack velocity ratio,  $R_{\max}$

$$R_{\max} = \left\{ 0,4457 + \left[ 0,5690 - \frac{0,2603\eta_s(q_{Vs})^{0,5}}{v_n^{1,5}} \right]^{0,5} \right\} \quad (C.6)$$

where

$R_{\max}$  is the nozzle to stack velocity ratio parameter;

$\eta_s$  is the stack gas viscosity ( $\mu\text{P}$ );

$q_{Vs}$  is the sampling rate for PM<sub>10</sub> cyclone necessary to achieve specified  $D_{50}$  (acmm);

$v_n$  is the sample gas velocity in the nozzle (m/s).

NOTE For  $R_{\max} < 1,5$ , take  $v_{\max} = v_n \times R_{\max}$ . For  $R_{\max} > 1,5$ , take  $v_{\max} = v_n(1,5)$ .

Calculate the maximum velocity pressure,  $\Delta p_{\max}$

$$\Delta p_{\max} = 1,3686 \times 10^{-4} \left( \frac{p_s M_w}{T_s} \right) \left( \frac{v_{\max}}{C_p} \right)^2 \quad (C.7)$$

where

$\Delta p_{\max}$  is the maximum velocity pressure;

$M_w$  is the molecular mass of wet gas (g/mol);

$T_s$  is the absolute stack gas temperature (K);

$p_s$  is the absolute stack gas pressure (Pa);

$v_{\max}$  is the maximum gas velocity (m/s);

$C_p$  is the Pitot tube coefficient for the tube attached to the sampling apparatus.

### C.3 Optimum nozzles

Compare the minimum and maximum velocity pressures with the observed velocity pressures at all traverse points during the preliminary velocity traverse. The nozzle selected is appropriate if all the observed velocity pressures during the preliminary test fall within the range between  $\Delta p_{\min}$  and  $\Delta p_{\max}$ .

## Annex D (informative)

### Calculating the sampling flow rate

#### D.1 Calculations

##### D.1.1 General

Use the data obtained in the pre-sampling measurements to perform the calculations in [D.1.2](#) to [D.1.7](#).

##### D.1.2 Dry molecular mass

$$M_d = 0,44\varphi(\text{CO}_2) + 0,32\varphi(\text{O}_2) + 0,28 \times [100 - \varphi(\text{O}_2) - \varphi(\text{CO}_2)] \quad (\text{D.1})$$

where

- $M_d$  is the molecular mass of dry gas (g/mol);
- $\varphi(\text{CO}_2)$  carbon dioxide volume fraction in flue gas (%);
- $\varphi(\text{O}_2)$  oxygen volume fraction in flue gas (%).

##### D.1.3 Wet molecular mass

$$M_w = M_d(1 - B_{ws}) + 18(B_{ws}) \quad (\text{D.2})$$

where

- $M_w$  is the molecular mass of wet gas (g/mol);
- $M_d$  is the molecular mass of dry gas (g/mol).
- $B_{ws}$  is the moisture fraction of flue gas.



#### D.1.4 Gas viscosity

The gas viscosity is used to calculate the Cunningham correction factor.

$$\eta = C_1 + C_2 \sqrt{T_s} + C_3 T_s^{-2} + C_4 \varphi(O_2)_w - C_5 B_{ws} + C_6 B_{ws} T_s^2 \quad (D.3)$$

where

- $\eta$  is the gas viscosity ( $\mu\text{P}$ );
- $T_s$  is the absolute stack temperature (K);
- $\varphi(O_2)_w$  oxygen volume fraction in wet flue gas (%)
- $B_{ws}$  is the moisture fraction of flue gas;
- $C_1$  is  $-150,316 \text{ }^2$  ( $\mu\text{P}$ );
- $C_2$  is  $8,061 \text{ }^4$  ( $\mu\text{P}/\text{K}^{0,5}$ );
- $C_3$  is  $1,191 \text{ }^83 \times 10^6$  ( $\mu\text{P}/\text{K}^2$ );
- $C_4$  is  $0,591 \text{ }^3$  ( $\mu\text{P}$ );
- $C_5$  is  $91,972 \text{ }^3$  ( $\mu\text{P}$ );
- $C_6$  is  $4,917 \text{ }^5$  ( $\mu\text{P}/\text{K}^2$ ).

#### D.1.5 Cunningham correction factor

The Cunningham correction factor is used to calculate the Reynolds number for particles  $< 2,25 \text{ } \mu\text{m}$  diameter.

$$C = 0,0057193 \left( \frac{\eta}{p_s D_p} \right) \left( \frac{T_s}{M_w} \right)^{0,5} \quad (D.4)$$

where

- $C$  is the Cunningham correction factor for particle diameter calculated using actual stack gas temperature;
- $\eta$  is the stack gas viscosity ( $\mu\text{P}$ );
- $p_s$  is the absolute stack gas pressure (Pa);
- $D_p$  is the particle size ( $\mu\text{m}$ );
- $T_s$  is the absolute stack gas temperature (K);
- $M_w$  is the molecular mass of wet gas (g/mol).

#### D.1.6 Reynolds number

$$Re = 8,64 \times 10^5 \left( \frac{p_s M_w}{T_s} \right) \left( \frac{q_{Vs}}{\eta} \right) \quad (D.5)$$

where

$Re$  is the Reynolds number;

$p_s$  is the absolute stack gas pressure (Pa);

$M_w$  is the molecular mass of wet gas (g/mol);

$q_{Vs}$  is the sampling rate for PM<sub>10</sub> cyclone necessary to achieve specified  $D_{50}$  (acmm);

$T_s$  is the absolute stack gas temperature (K);

$\eta$  is the gas viscosity (μP).

#### D.1.7 $D_{50LL}$

Preliminary lower limit cut diameter for  $Re < 3,162$  cut diameter — PM<sub>10</sub> cyclone

$$D_{50LL} = 9,507C^{0,3007} \left( \frac{p_s M_w}{T_s} \right)^{0,1993} \quad (D.6)$$

where

$D_{50LL}$  is the cut diameter for PM<sub>10</sub> cyclone corresponding to the 2,25 μm cut diameter for the PM<sub>2,5</sub> cyclone (μm);

$C$  is the Cunningham correction factor;

$p_s$  is the absolute stack gas pressure (Pa);

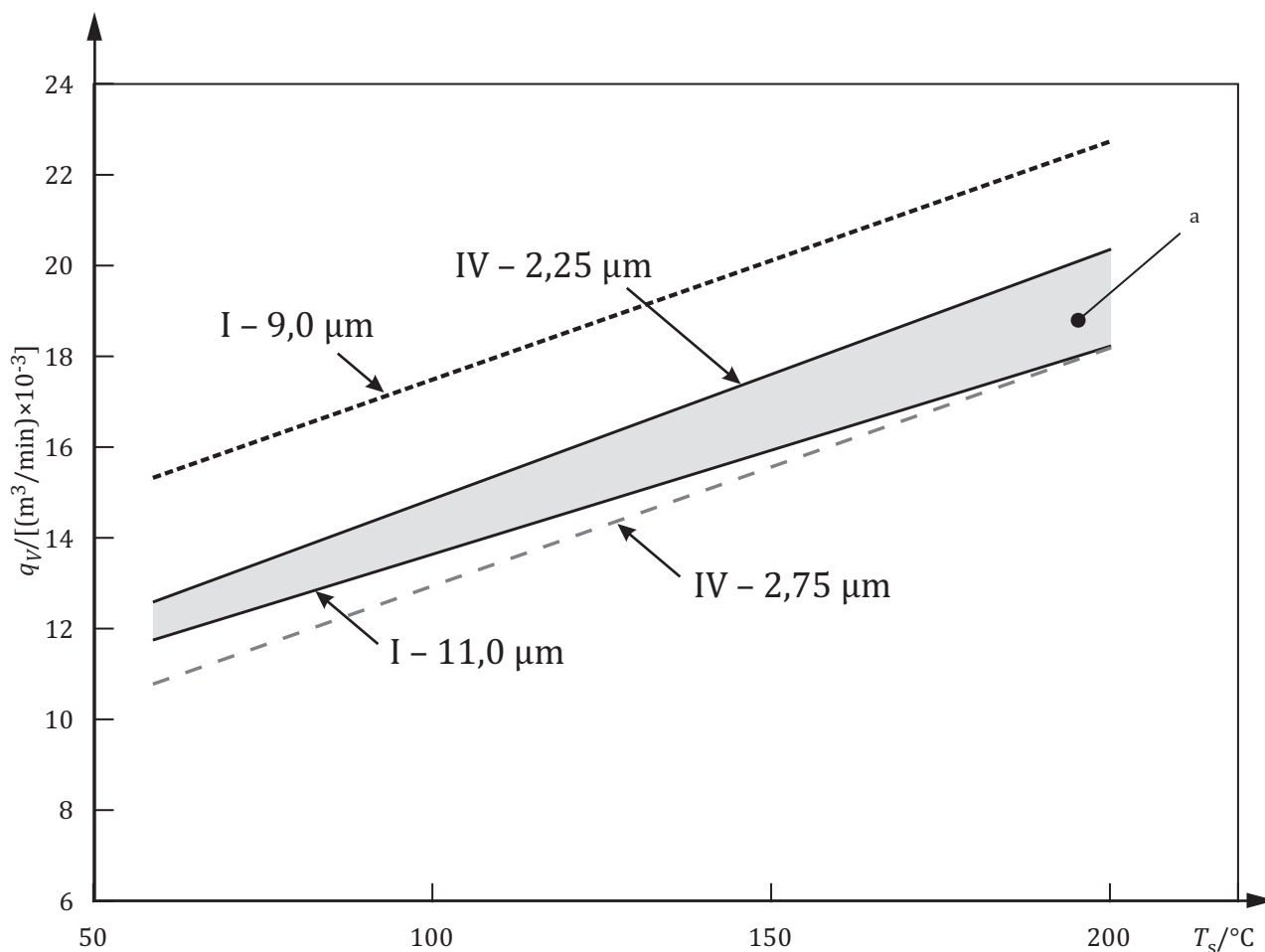
$M_w$  is the molecular mass of wet gas (g/mol);

$T_s$  is the absolute stack gas temperature (K).

## D.2 Constant sampling flow rate

Determine the constant sample flowrate. The gas sampling rate is defined by the performance curves for both cyclones as illustrated in [Figure D.1](#).

The optimum gas sampling rate is the overlap zone defined as the range below the 2,25 μm curve down to the 11,0 μm curve (area between the two solid lines of [Figure D.1](#)). Choose a gas sample rate in the middle of the overlap zone to maximize the acceptable tolerance for slight variations in flow characteristics at the sampling location.



### Key

$q_V$	flowrate (acmm)	I — 9,0 $\mu\text{m}$	cyclone I, 9,0 $\mu\text{m}$ cut size
$T_s$	stack temperature	I — 11,0 $\mu\text{m}$	cyclone I, 11,0 $\mu\text{m}$ cut size
		IV — 2,25 $\mu\text{m}$	cyclone IV, 2,25 $\mu\text{m}$ cut size
a	Flow rate range for PM <sub>10</sub> and PM <sub>2,5</sub> .	IV — 2,75 $\mu\text{m}$	cyclone IV, 2,75 $\mu\text{m}$ cut size

**Figure D.1 — Sampling rates for combined cyclone sampling heads, based on  $\varphi(\text{O}_2) = 8\%$ ,  $\varphi(\text{CO}_2) = 10\%$ ,  $\varphi(\text{H}_2\text{O}) = 2\%$**

**NOTE** The overlap zone is also a weak function of the gas composition. The acceptable range is limited, especially for gas streams with temperatures less than 40 °C. At lower temperatures, it may be necessary to apply the PM<sub>10</sub> and PM<sub>2,5</sub> cyclones separately in order to achieve actual  $D_{50}$  cut sizes from 9  $\mu\text{m}$  to 11  $\mu\text{m}$  and 2,25  $\mu\text{m}$  to 2,75  $\mu\text{m}$ , respectively.

Verify the assumed Reynolds number first calculating the sampling rate for the PM<sub>10</sub> cyclone:

$$q_{Vs} = 0,7296\eta \left( \frac{T_s}{p_s M_w} \right) 0,2949 \left( \frac{1}{D_{50T}} \right)^{1,4102} \quad (D.7)$$

where

$q_{Vs}$  is the sampling rate for PM<sub>10</sub> cyclone necessary to achieve specified  $D_{50}$  (acmm);

$\eta$  is the gas viscosity (μP);

$T_s$  is the absolute stack gas temperature (K);

$p_s$  is the absolute stack gas pressure (Pa);

$M_w$  is the molecular mass of wet gas (g/mol);

$D_{50T}$  is the PM<sub>10</sub> cyclone cut diameter corresponding to the middle of the overlap.

$$D_{50T} = \left( \frac{11 + D_{50LL}}{2} \right) \quad (D.8)$$

where

$D_{50T}$  is the PM<sub>10</sub> cyclone cut diameter corresponding to the middle of the overlap;

$D_{50LL}$  is the cut diameter for PM<sub>10</sub> cyclone corresponding to the 2,25 μm cut diameter for the PM<sub>2,5</sub> cyclone (μm).

### D.3 Basic sampling system orifice meter pressure drop

If the Reynolds number is <3,162, calculate the pressure drop across the orifice meter ( $\Delta H$ ) for the basic sampling system (which, when set, establishes the sampling rate through the nozzle).

$$\Delta H = \left[ \frac{q_{Vs} (1 - B_{ws}) p_s}{T_s} \right] \left( \frac{1,083 T_m M_d \Delta H_2}{p_{atm}} \right) \quad (D.9)$$

where

$\Delta H$  is the pressure drop across the orifice meter;

$q_{Vs}$  is the sampling rate for PM<sub>10</sub> cyclone necessary to achieve specified  $D_{50}$  (acmm);

$B_{ws}$  is the moisture fraction of flue gas;

$p_s$  is the absolute stack gas pressure (Pa);

$T_s$  is the absolute stack gas temperature (K);

$T_m$  is the meter box and orifice gas temperature (K);

$M_d$  is the molecular mass of dry gas (g/mol);

$\Delta H_2$  is the orifice pressure differential for a flow rate of 0.021 2 m<sup>3</sup>/min at standard conditions (cmH<sub>2</sub>O).

$p_{atm}$  is the atmospheric pressure (Pa).

If the Reynolds number is  $>3,162$ , recalculate  $D_{50LL}$  using the Formula (D.10):

$$D_{50LL} = 10,0959 C^{0,4400} \left[ \frac{M_w p_s}{T_s} \right]^{0,0600} \quad (D.10)$$

where

$D_{50LL}$  is the cut diameter for PM<sub>10</sub> cyclone corresponding to the 2,25 µm cut diameter for the PM<sub>2,5</sub> cyclone (µm);

$M_w$  is the molecular mass of wet gas (g/mol);

$p_s$  is the absolute stack gas pressure (Pa);

$T_s$  is the absolute stack gas temperature (K).

Substitute this value into the equation for  $D_{50T}$  and recalculate  $D_{50T}$  to recalculate  $q_{Vs}$  and the sampling train orifice meter pressure drop,  $\Delta H$ .

NOTE The stack gas temperature can vary during the test, potentially affecting the sampling rate. If the stack temperature varies by more than  $\pm 30$  °C, recalculate  $\Delta H$  using the new temperature.

#### D.4 Setting the sampling system flow rate

For isokinetic sampling, calculate the parameters necessary for the sampling system so that the sampling rate,  $q_{Vs}$  is achieved.  $q_{Vs}$  is the sampling rate for the PM<sub>10</sub> cyclone necessary to achieve the specified  $D_{50}$  (acmm).

#### D.5 Total sample volume

Calculate and correct to standard conditions, the dry gas volume of the flue gas sample measured by the dry gas meter of the sampling train. The sampling flow rate at standard conditions is described in Formula (D.11).

$$q_{Vs, std} = \frac{V_m}{t_{tr}} \quad (D.11)$$

where

$q_{Vs, std}$  is the dry gas sampling rate through the sampling assembly (dscm);

$V_m$  is the dry gas meter volume sampled (ACM);

$t_{tr}$  is the total run time (min).

And the actual sampling rate over the run was:

$$q_{Vs} = \frac{29,92}{528} q_{Vs, std} \left[ \frac{1}{(1 - B_{ws})} \right] \left[ \frac{T_s}{p_s} \right] \quad (D.12)$$

where

$q_{Vs}$  is the sampling rate for PM<sub>10</sub> cyclone necessary to achieve specified  $D_{50}$  (acmm);

$q_{Vs, std}$  is the dry gas sampling rate through the sampling assembly (dscmm);

$B_{ws}$  is the moisture fraction of flue gas;

$T_s$  is the absolute stack gas temperature (K);

$p_s$  is the absolute stack gas pressure (Pa).

## Annex E (informative)

### Method calculations

#### E.1 Stack gas flow rate

For each run calculate the stack gas flow rate,  $q_{Vs}$ , in standard  $\text{m}^3/\text{min}$ :

$$q_{Vs} = A_s v_s \left( \frac{T_{\text{std}}}{T_s} \right) \left( \frac{p_s}{p_{\text{std}}} \right) \quad (\text{E.1})$$

where

- $q_{Vs}$  sampling rate for  $\text{PM}_{10}$  cyclone necessary to achieve specified  $D_{50}$  (acmm);
- $A_s$  is the stack area in the same cross-sectional plane as the stack gas sample measurements ( $\text{m}^2$ );
- $v_s$  is the average stack gas velocity in the same cross-sectional plane as the stack gas sample measurements (m/min);
- $T_s$  is the absolute stack gas temperature (K);
- $p_s$  is the absolute stack gas pressure (kPa);
- $T_{\text{std}}$  is the absolute temperature at standard conditions (K);
- $p_{\text{std}}$  is the absolute pressure at standard conditions (hPa).

#### E.2 Dilution ratio

For each run, calculate the dilution ratio, DR, from tracer gas measurements (ISO 8178-1) or from direct flow measurements with Formula (E.2).

$$D_R = q_{Vda}/q_{Vuds} \quad (\text{E.2})$$

where

- $D_R$  is the dilution ratio;
- $q_{Vuds}$  is the flow rate of the undiluted stack gas sample at the flow meter, at standard pressure and temperature;
- $q_{Vda}$  is the flow rate of the dilution air at standard temperature and pressure.



$$q_{Vda} = q_{Vmda} \left( \frac{T_{std}}{T_{mda}} \right) \left( \frac{p_{mda}}{p_{std}} \right) \quad (E.3)$$

where

$q_{Vmda}$  is the measured dilution air flow rate at actual temperature and pressure (m<sup>3</sup>/min);

$T_{std}$  is the absolute temperature at standard conditions (K);

$T_{mda}$  is the measured dilution air temperature at the flow meter (K);

$p_{mda}$  is the measured dilution air pressure at the flow meter (hPa);

$p_{std}$  is the absolute pressure at standard conditions (hPa).

The flow rate of the undiluted stack gas sample at the flow meter, at standard pressure and temperature,  $q_{Vuds}$ , is calculated with Formula (E.4) (m<sup>3</sup>/min).

$$q_{Vuds} = q_{Vmuds} \left( \frac{T_{std}}{T_{muds}} \right) \left( \frac{p_{muds}}{p_{std}} \right) \quad (E.4)$$

where

$q_{Vuds}$  is the flow rate of the undiluted stack gas sample at the flow meter, at standard pressure and temperature;

$q_{Vmuds}$  is the measured undiluted stack gas sample flow rate at the flow meter, at actual temperature and pressure (m<sup>3</sup>/min);

$T_{muds}$  is the measured undiluted stack gas sample temperature at the flow meter (K);

$p_{muds}$  is the measured undiluted stack gas sample pressure at the flow meter (hPa);

$T_{std}$  is the absolute temperature at standard conditions (K);

$p_{std}$  is the absolute pressure at standard conditions (hPa).

### E.3 Percentage isokinetic

Calculate the percentage isokinetic for the sampling run, as follows:

$$\%I = \left[ \frac{T_s V_{m,std} 29,92}{60 v_s t_{tr} A_n (1 - B_{ws})} \right] 100 \quad (E.5)$$

where

$\%I$  is the percent isokinetic;

$T_s$  is the absolute stack gas temperature (K);

$V_{m,std}$  is the dry gas meter volume sampled, corrected to standard conditions (dscm);

$v_s$  is the velocity of stack gas (m/s);

$t_{tr}$  is the total run time (min);

$A_n$  is the area of nozzle (m<sup>2</sup>);

$B_{ws}$  is the moisture fraction of flue gas.

## E.4 In-stack sampling cyclones PM<sub>2,5</sub> and/or PM<sub>10</sub> concentration

### E.4.1 General

For each run, calculate the concentration of the total filterable particulate matter, filterable PM<sub>10</sub> particulate matter, and filterable PM<sub>2,5</sub> particulate matter obtained from the in-stack cyclone and filter sampling system at standard temperature and pressure in mg/m<sup>3</sup>.

### E.4.2 Total filterable particulate matter

Calculate the total filterable particulate matter concentration as follows:

$$\gamma_{\text{tf}} = \left( \frac{7\,000}{453,592} \right) \left( \frac{m_1 + m_2 + m_3 + m_4}{V_{\text{m,std}}} \right) \quad (\text{E.6})$$

where

- $\gamma_{\text{tf}}$  is the concentration of the total filterable particulate matter (mg/m<sup>3</sup>);
- $m_1$  is the mg of particulate matter ( $\leq 2,5 \mu\text{m}$ ) collected on the filter of the basic sampling train;
- $m_2$  is the mg of particulate matter ( $> 10 \mu\text{m}$ ) recovered from container No. 2 (acetone blank corrected);
- $m_3$  is the mg of particulate matter ( $\leq 10$  and  $> 2,5 \mu\text{m}$ ) recovered from container No. 3 (acetone blank corrected);
- $m_4$  is the mg of particulate matter ( $\leq 2,5 \mu\text{m}$ ) recovered from container No. 4 (acetone blank corrected);
- $V_{\text{m,std}}$  is the dry gas meter volume sampled, corrected to standard conditions (dscm).

### E.4.3 Filterable PM<sub>10</sub> particulate matter

Calculate the concentration of filterable PM<sub>10</sub> particulate matter.

$$\gamma_{\text{fPM}_{10}} = \left( \frac{7\,000}{453,592} \right) \left( \frac{m_1 + m_3 + m_4}{V_{\text{m,std}}} \right) \quad (\text{E.7})$$

where

- $\gamma_{\text{fPM}_{10}}$  is the concentration of filterable PM<sub>10</sub> particulate matter (mg/m<sup>3</sup>);
- $m_1$  is the mg of particulate matter ( $\leq 2,5 \mu\text{m}$ ) collected on the filter of the basic sampling train;
- $m_3$  is the mg of particulate matter ( $\leq 10$  and  $> 2,5 \mu\text{m}$ ) recovered from container No. 3 (acetone blank corrected);
- $m_4$  is the mg of particulate matter ( $\leq 2,5 \mu\text{m}$ ) recovered from container No. 4 (acetone blank corrected);
- $V_{\text{m,std}}$  is the dry gas meter volume sampled, corrected to standard conditions (dscm).

#### E.4.4 Filterable PM<sub>2,5</sub> particulate matter

Calculate the concentration of filterable PM<sub>2,5</sub> particulate matter

$$\rho_{fPM_{2,5}} = \left( \frac{7\,000}{453,592} \right) \left( \frac{m_1 + m_4}{V_{m,std}} \right) \quad (E.8)$$

where

- $\rho_{fPM_{2,5}}$  is the concentration of filterable PM<sub>2,5</sub> particulate matter (mg/m<sup>3</sup>);
- $m_1$  is the mg of particulate matter ( $\leq 2,5 \mu\text{m}$ ) collected on the filter of the basic sampling train;
- $m_4$  is the mg of particulate matter ( $\leq 2,5 \mu\text{m}$ ) recovered from container No. 4 (acetone blank corrected);
- $V_{m,std}$  is the dry gas meter volume sampled, corrected to standard conditions (dscm).

### E.5 Dilution system cyclones PM<sub>2,5</sub> and/or PM<sub>10</sub> condensable concentration

#### E.5.1 General

For each run, calculate the PM<sub>2,5</sub> and/or the PM<sub>10</sub> concentration(s) in the undiluted stack gas sample at standard temperature and pressure in mg/m<sup>3</sup>, using the following data.

#### E.5.2 Volume calculations

Calculate  $V_{uds}$ , the volume of the undiluted stack gas sample, corrected to standard temperature and pressure with Formula (E.9).

$$V_{uds} = \frac{V_{bp,std} + V_{f,std}}{D_F} \quad (E.9)$$

where

- $V_{uds}$  is the volume of the undiluted stack gas sample, corrected to standard temperature and pressure;
- $D_F$  is the dilution factor;
- $V_{bp,std}$  is the volume of the diluted stack gas, m<sup>3</sup> of the bypass flow, corrected to standard temperature and pressure;
- $V_{f,std}$  is the volume of the diluted stack gas, m<sup>3</sup>, which passed through the filter corrected to standard temperature and pressure.

$$V_{f,std} = V_f \left( \frac{T_{std}}{T_{mf}} \right) \left( \frac{p_{mf}}{p_{std}} \right) \quad (E.10)$$

where

- $V_{f,std}$  is the volume of the diluted stack gas, m<sup>3</sup>, which passed through the filter corrected to standard temperature and pressure;
- $p_{std}$  is the absolute pressure at standard conditions (hPa);

- $T_{\text{std}}$  is the absolute temperature at standard conditions (K);
- $V_f$  is the measured volume of diluted stack gas that passed through the filter (m<sup>3</sup>);
- $T_{\text{mf}}$  is the measured temperature of the diluted stack gas sample at the filter flow meter (K);
- $p_{\text{mf}}$  is the measured pressure of the diluted stack gas sample at the filter flow meter;
- $V_{\text{bp,std}}$  is the volume of the diluted stack gas, m<sup>3</sup> of the bypass flow, corrected to standard temperature and pressure;
- $V_{\text{uds}}$  is the volume of the undiluted stack gas sample, corrected to standard temperature and pressure;
- $D_R$  is the dilution ratio.
- $$V_{N,\text{uds}} = \sum_i V_{\text{uds}} \quad (\text{E.11})$$

where

- $V_{N,\text{uds}}$  is the total volume of the undiluted stack gas sample obtained during the  $N$  test runs, corrected to standard temperature and pressure;
- $i$  is 1, 2 ...  $N$ th test prior to recovery;
- $N$  is the total number of tests conducted in the test series prior to recovery.

### E.5.3 PM<sub>2,5</sub> particulate matter

Calculate the concentration of PM<sub>2,5</sub> particulate matter

$$\gamma_{\text{PM}_{2,5}} = (1 + D_R) \frac{(m_{\text{fp}} - m_{\text{ft}})}{V_{\text{f,std}}} + \frac{(m_{\text{r3}} - m_{\text{r4}})/N}{V_{N,\text{uds}}} \quad (\text{E.12})$$

$$\gamma_{\text{PM}_{2,5}} = D_F \frac{(m_{\text{fp}} - m_{\text{ft}})}{V_{\text{f,std}}} + \frac{(m_{\text{r3}} - m_{\text{r4}})/N}{V_{N,\text{uds}}} \quad (\text{E.13})$$

where

- $\gamma_{\text{PM}_{2,5}}$  is the concentration of condensable PM<sub>2,5</sub> matter;
- $D_R$  is the dilution ratio;
- $V_{\text{f,std}}$  is the volume of the diluted stack gas, m<sup>3</sup>, which passed through the filter corrected to standard temperature and pressure;
- $N$  is the total number of test runs conducted in the test series prior to recovery;
- $D_F$  is the dilution factor, which is equal to  $(1 + D_R)$ ;
- $m_{\text{fp}}$  is the mass of the particulate laden filter(s) (mg);
- $m_{\text{ft}}$  is the tare mass of the filter(s) (mg);
- $m_{\text{r2}}$  is the PM mass recovered in container No. 2d (mg);
- $m_{\text{r3}}$  is the PM mass recovered in container No. 3d (mg);

$m_{r4}$  is the PM mass recovered in container No. 4d (mg).

#### E.5.4 Condensable PM<sub>10</sub> particulate matter (PM<sub>10</sub> cyclone)

Calculate the concentration of condensable PM<sub>10</sub> particulate matter.

$$\gamma_{PM_{10}} = (1 + D_R) \frac{(m_{fp} - m_{ft})}{V_{f,std}} + \frac{(m_{r2} + m_{r3} - m_{r4})/N}{V_{N,uds}} \quad (E.14)$$

$$\gamma_{PM_{10}} = D_F \frac{(m_{fp} - m_{ft})}{V_{f,std}} + \frac{(m_{r2} + m_{r3} - m_{r4})/N}{V_{N,uds}} \quad (E.15)$$

where  $\gamma_{PM_{10}}$  is the concentration of condensable PM<sub>10</sub> matter.

#### E.6 Condensable mass emission rate

For each run, calculate the PM<sub>2,5</sub> and/or the PM<sub>10</sub> mass emission rate (wet basis),  $q_{mPM}$ , kg/h.

$$q_{mPM} = \gamma_{PM} q_{Vs} \times 60 \times 10^{-6} \quad (E.16)$$

where

$q_{mPM}$  is the PM<sub>2,5</sub> and/or PM<sub>10</sub> mass emission rate (wet basis) (kg/h);

$\gamma_{PM}$  is the concentration of condensable particulate matter (mg/m<sup>3</sup>);

$q_{Vs}$  is the sampling rate for PM<sub>10</sub> cyclone necessary to achieve specified  $D_{50}$  (acmm).

## Annex F (informative)

### Results of method validation

#### F.1 Field experimental data for diesel engine tests and 120 min sampling duration

##### F.1.1 Comparison tests

Comparison tests were conducted on a diesel engine equipped with a catalytic particulate filter with two dilution samplers employing similar design principles but different configurations. Sampler A is a very similar derivative of previously validated reference design. Sampler B is a compact design (N17). The measured PM<sub>2,5</sub> concentration is approximately 4 mg/m<sup>3</sup> under standard conditions.

##### F.1.2 Accuracy

Results for six co-located samples for two different engine loads showed an average relative difference in PM<sub>2,5</sub> concentration of 2,9 % for sampler B relative to sampler A. The difference is not significant at the 95 % confidence level based on a paired *t*-test.

##### F.1.3 Precision

The average coefficients of variation of three replicate samples at each of two different engine loads were 2 % and 9 % for sampler A and sampler B, respectively. The difference in precision for sampler A and sampler B is not significant at the 95 % confidence level based on *F*-tests.

##### F.1.4 Confidence interval

The average 95 % confidence interval (two standard deviations) was 3 % to 5 % for sampler A and 14 % to 24 % for sampler B for three replicate samples at each of two different engine loads.

##### F.1.5 Uncertainty

The uncertainty of average emission rates in pounds per million British thermal units (1 lb/mmBtu = 430 ng/J), calculated including estimates of error in all underlying measurements was approximately 15 % to 16 %, at the 95 % confidence level.

#### F.2 Experimental data for natural gas-fired sources and 360 min sampling duration

##### F.2.1 Comparison tests

Comparison tests with sampler A and sampler B were conducted on a natural gas-fired combined cycle power plant (with duct burners, CO oxidation catalyst and selective catalytic NO<sub>x</sub> reduction emission controls). Tests with sampler B were conducted at a gas-fired cogeneration plant (with duct burners, CO oxidation catalyst and selective catalytic NO<sub>x</sub> reduction emission controls). The average PM<sub>2,5</sub> concentration was approximately 0,1 mg to 0,2 mg/m<sup>3</sup>. Dilution ratio was approximately 20,47 mm. PTFE filters were used, and the total sample volume through each filter was approximately 27 m<sup>3</sup>. The acetone rinses were excluded from these results because they could not be distinguished from blanks at the 95 % confidence level.

### F.2.2 Relative accuracy

Results for seven co-located samples under two loads at the power plant showed an average relative difference of approximately 6 % for sampler B relative to sampler A. The difference was not significant at the 95 % confidence level.

### F.2.3 Bias and minimum detection limit

Background PM<sub>2,5</sub> concentration in the filtered dilution air during the power plant site tests was estimated to be approximately 0,04 mg/m<sup>3</sup> to 0,09 mg/m<sup>3</sup> (10th to 90th percentile), corrected to in-stack concentration at a dilution ratio of approximately 20. This was significant relative to the measured stack sample results. Based on bias due to background levels in the dilution air, the minimum in-stack detection limit (three times standard deviation of system blanks) in these tests was estimated to be 0,06 mg/m<sup>3</sup> for a 6 h test. Extrapolating this result, the minimum in-stack detection limit for a 1 h test is estimated to be 0,4 mg/m<sup>3</sup>. If background PM<sub>2,5</sub> in the dilution air is neglected (i.e. if additional measures are taken to reduce background to negligible levels) and only the analytical detection limit is used, the minimum in-stack detection limit for a 1 h test is estimated to be 0,04 mg/m<sup>3</sup>.

### F.2.4 Precision

The PM<sub>2,5</sub> coefficient of variation at the power plant for three to four replicate samples at two different process loads was 15 % to 29 % for sampler A and 30 % to 46 % for sampler B. The difference in precision for sampler A and sampler B was not significant at the 95 % confidence level based on *F*-tests. The coefficient of variation of three replicate test runs at a gas-fired cogeneration plant was 23 % at an average PM<sub>2,5</sub> concentration of approximately 0,2 mg/m<sup>3</sup> under dry standard conditions.

### F.2.5 Confidence interval

The average 95 % confidence interval (two standard deviations) for a four run test at the power plant was approximately  $\pm 0,05$  mg/m<sup>3</sup> (or  $\pm 52$  % of 0,1 mg/m<sup>3</sup>). It should be noted that these values represent conservative estimates of measurement precision since they include the combined variability in the measurements, the process, and external factors. Measurement variability is believed to be the dominant source of variation.

### F.2.6 Uncertainty

The uncertainty of average emission rates in pounds per million British thermal units (lb/mmBtu), calculated including estimates of error in all underlying measurements, ranged from 37 % to 41 % at the power plant and 60 % at the cogeneration plant, at the 95 % confidence level.

## F.3 Experimental data for three dilution samplers employed at coal-fired utility boilers with a 180 min sampling duration

### F.3.1 Comparison tests

Comparative tests with dilution samplers of type A, B, and C were conducted on a 160 MW coal-fired boiler equipped with an electrostatic precipitator. The average PM<sub>2,5</sub> concentration was approximately 4,8 mg/m<sup>3</sup> to 5,4 mg/m<sup>3</sup>. Dilution ratios were 1:33, 1:56, and 1:30 for samplers A, B, and C, respectively. Residence times from the samplers were 1 s, 20 s, and 30 s for samplers A, B, and C, respectively. Sampler A used 142 mm PTFE filters, whereas samplers B and C use 47 mm PTFE filters. Filters were pre-conditioned and weighed at 23 °C and at 40 % RH.

### F.3.2 Relative accuracy

Results for seven co-located samples under similar loads at the coal-fired power plant showed an average relative difference of samplers A and B, relative to C, of 9,8 %. The average relative difference between samplers A and C was 17,9 %. The average relative difference between samplers B and C was 13,9 %.



### F.3.3 Precision

The PM<sub>2,5</sub> coefficient of variation determined on an oil-fired pilot scale boiler, for six replicate samples at a similar process load were 9,4 %, 6,7 %, and 9,6 % for samplers, A, B, and C, respectively at a particulate loading of 46,5 mg/m<sup>3</sup>.

### F.3.4 Confidence interval

The average 95 % confidence interval (two standard deviations) for the mean difference of samplers A and B were -2,178 and 0,921 mg/m<sup>3</sup>. The 95 % confidence interval for the mean difference between samplers A and C were 2,937 8 and 1,21 mg/m<sup>3</sup> and for samplers B and C were 0,819 and 3,181 mg/m<sup>3</sup> at the oil-fired pilot boiler.

## Annex G (informative)

### Alternative analytical techniques

#### G.1 Analytical flexibility

After dilution in a dilution sampling system, the flue gas sample is assumed to have the characteristics of an ambient air sample in terms of its temperature, humidity and particle concentration. Analytical techniques used in ambient air, or indoor air and workplace monitoring could then be applied for measurements at the relatively lower particle concentration levels. Techniques used for ambient air pollutant gas analysis can also be used to further characterize the diluted sample.

Although this International Standard provides the procedures for the gravimetric weighing of particulate matter collected either in the cyclones or filters of the dilution system, other measurement techniques can also be applied to the diluted sample. The gravimetric technique of this International Standard provides a short-term sample of the particulate mass concentration. Other analytical techniques using inertial oscillators, beta-radiation or optical scattering instruments can provide either semi-continuous or continuous measurements, which may be an advantage in some applications. Other measurement techniques using impactors (see ISO 23210[13]) or optical scattering instruments can provide particle size distributions over a wider range than the 2,5 µm and 10 µm cut-sizes of this International Standard.

These techniques can be used in conjunction with the dilution system described in this International Standard, either in place of or parallel to the PM<sub>2,5</sub> cyclone and filter located after the residence time chamber. However, the flow rates shall be suitably balanced so as to obtain representative samples in each analytical system while maintaining proper flow rates through the sampling system nozzle. A number of the alternative methods are indirect mass measurement methods which require that a number of assumptions be satisfied in order to provide measurements comparable to the direct gravimetric mass measurement technique of this International Standard. By using suitable calibration standards, comparability to the gravimetric methods of this International Standard may be achieved, however, the level of comparability shall be determined.

#### G.2 Mass measurement using inertial oscillators

##### G.2.1 General

Inertial oscillators, such as a quartz crystal microbalance, oscillating filters, or piezo-electric micro-oscillators can be used to measure the mass collected on the oscillator surface. An inertial microbalance used in ambient air monitoring, follows the relation:

$$\Delta m = K \left( \frac{1}{f_b^2} - \frac{1}{f_a^2} \right) \quad (G.1)$$

where

$K$  is a calibration factor;

$\Delta m$  is the change of mass on the oscillator;

$f_a^2$  is the oscillation frequency after the initial oscillation frequency;

$f_b^2$  is the oscillation frequency after the mass of the oscillator has changed.

The oscillator is first calibrated using a known mass, after which it can read the mass collected on the filter directly without having to make any assumptions or reference method correlations.

### G.2.2 Application

The inertial microbalance has been standardized under ASTM D6831[1] as a manual in-stack source test method. It is capable of measuring down to 0,2 mg/m<sup>3</sup>. Although measuring continuously, measurements can only be made over the period from when the oscillator is clean and when it becomes loaded, or overly damped. After the oscillator becomes loaded, it shall either be cleaned or replaced.

## G.3 Mass measurement using beta-gauges

### G.3.1 General

The beta-radiation attenuation method measures the attenuation of beta-rays passing through a spot of particulate matter collected on a movable tape. The intensity of the transmitted radiation follows the approximate exponential relation,

$$I = I_0 \exp(-\alpha \rho_A) \quad (\text{G.2})$$

where

$I$  is the intensity of the transmitted radiation through the sample spot;

$I_0$  is the intensity of the initial radiation (or that passed through a blank section of filter tape);

$\alpha$  is the mass absorption coefficient (g/cm<sup>2</sup>);

$\rho_A$  is surface density of the sample (g/cm<sup>2</sup>).

The paper tape can run continuously, indefinitely, so a continuous mass measurement can be made. Standards of known composition and mass can also be placed on the tape for calibration.

### G.3.2 Application

The mass absorption coefficient is dependent upon the ratio of the atomic number to the atomic mass of the atoms in the sample and the energy of the beta ray. When low velocity electrons are used, the atomic number dependence is relatively small, and is relatively constant for materials making up particulate matter in combustion processes. Absorption coefficients also average out with the mixture of compounds collected on the tape. Errors can be minimized by using standards having a mass absorption coefficient equivalent to that of the measured material.

Beta-gauges have been designed for both ambient and source monitoring applications.

### G.3.3 General

An impactor could be used instead of a cyclone after the residence time chamber, to determine the particle mass concentrations at 10 µm, 2,5 µm or other sizes. An impactor separates particles according to their specific aerodynamic diameter by accelerating them through a nozzle and deflecting them by 90°. Particles with aerodynamic diameter not able to follow the gas flow due to their inertia are collected on plate. An impactor may have several plates, each with a different particle cut-off diameter. Impactor measurement procedures for source measurements are given in ISO 23210.[13]

Virtual impactors operate in a similar fashion, without impaction of the particles on a plate, but where the particles are collected on filters after each separation stage.

#### **G.3.4 Application**

Impactors provide a test measurement and are not typically continuous. The procedures given in ISO 23210,<sup>[13]</sup> would have to be suitably modified in application to the dilution sample to accommodate the essentially, ambient conditions of measurement.

### **G.4 Mass measurement using optical scattering instruments**

#### **G.4.1 General**

Optical scattering instruments are closed optical cell instruments that typically contain a low volume optical measurement chamber, where particles pass one by one through the sensing zone. Particles are illuminated by a focused light beam of specified characteristics and scatter this light according to the properties of the particles.

The scattered light is sensed by a photocell, where the pulse height is correlated to the effective particle diameter and the number of electronic pulses per volume is related to the particle number concentration.

#### **G.4.2 Application**

Optical scattering instruments are suitable for reporting results in terms of number density; the number of particles per volume. Conversion to mass concentration requires the determination of both the refractive index and density of each particle.

#### **G.4.3 Sampling for gases**

Depending upon the test requirements, samples can also be extracted from the residence chamber for the analysis of gaseous precursors that may contribute to the formation of particulate matter (e.g. SO<sub>2</sub>, NO<sub>x</sub>, ammonia, SO<sub>3</sub>, HCl, VOCs), using suitable analytical equipment.

## Bibliography

- [1] ASTM D6831, *Standard test method for sampling and determining particulate matter in stack gases using an in-stack, inertial microbalance*
- [2] DAWES S.D. Application guide for measurement of PM<sub>2.5</sub> at stationary sources. Washington, DC: US Environmental Protection Agency, 1990. (Report No.: EPA/600/3-90/057)
- [3] ENGLAND G.C., WIEM S., CHANG M.C Characterization of stationary source PM<sub>2,5</sub> emissions. Presentation No. 11, CEM 2002, 5th International conference on emissions monitoring, 2002-09-11/13, Odense, Denmark
- [4] ENGLAND G.C., WATSON J.G., CHOW J.C., ZIELINSKA B., CHANG M.C., LOOS K.R., Hidy G.M Dilution-based emissions sampling from stationary sources — Part 1: Compact sampler methodology and performance. J. Air Waste Manage. Assoc. 2007, **57** pp. 65–78
- [5] FARTHING W.E., & DAWES S.S. Application guide for source PM<sub>10</sub> measurement with constant sampling rate. Washington, DC: US Environmental Protection Agency, 1988. (Report: EPA/600/3-88-057)
- [6] LEE S.W., HERAGE T., YOUNG B. Development of a simplified fine particulate measurement system for electric utility boilers and industrial combustors. Ottawa: National Resources Canada, 2006 [Report CDETC-O-CEPG-05-040(CF)]
- [7] SMITH W.B., & WILSON R.R. Development and laboratory evaluation of a five-stage cyclone system. Washington, DC: US Environmental Protection Agency, 1978. (Report: EPA/600/7-78-008)
- [8] LEE S.W., HERAGE T., DUREAU R., IDRIS A. Parallel evaluation of CETC Prototypes and the USEPA proposed equipment for measuring PM<sub>2.5</sub> emissions from oil- and coal-fired boilers. Ottawa: National Resources Canada, 2007 [Report: CETC-CEPG-07-007(CF)]
- [9] LEE S.W. Fine particulate matter measurement and international standardization for air quality and emissions from stationary sources. Fuel. 2010, **89** pp. 874–882
- [10] US EPA Method 201A, *Determination of PM<sub>10</sub> and PM<sub>2.5</sub> emissions (Constant sampling rate procedure)*. Available (viewed 2013-01-31) at: <http://www.epa.gov/ttn/emc/promgate/m-201a.pdf>
- [11] ISO 5725 (all parts), *Accuracy (trueness and precision) of measurement methods and results*
- [12] ISO 80000-9, *Quantities and units — Part 9: Physical chemistry and molecular physics*
- [13] ISO 23210, *Stationary source emissions — Determination of PM<sub>10</sub>/PM<sub>2,5</sub> mass concentration in flue gas — Measurement at low concentrations by use of impactors*
- [14] ISO 3534-1, *Statistics — Vocabulary and symbols — Part 1: General statistical terms and terms used in probability*

