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Stationary source emissions — Test method for determining PM_{2,5} and PM₁₀ mass in stack gases using cyclone samplers and sample dilution

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National foreword

This British Standard is the UK implementation of ISO 25597:2013.

The ISO corrected text 1 February 2015 incorporates the following editorial 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)].

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A list of organizations represented on this subcommittee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Stationary source emissions — Test method for determining PM_{2,5} and PM₁₀ 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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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
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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).

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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*.

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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 μm (PM_{10}) and 2,5 μm ($\text{PM}_{2,5}$).

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 μm and 10 μm ;
- 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 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_{2,5} and PM₁₀ 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 µm and 10 µm. This method is especially suitable for measurements of particle mass concentrations above 50 mg/m³ 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 µm (PM₁₀) 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_{2,5} and/or PM₁₀ 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 µm aerodynamic diameter (PM_{2,5}) using weighing techniques. The method can be used to measure mass concentrations of particles with aerodynamic diameter smaller than 10 µm aerodynamic diameter (PM₁₀) or particles with aerodynamic diameters between 2,5 µm and 10 µm.

In this method, the dilution sampling train can be used in combination with the basic sampling train, using PM₁₀ and/or PM_{2,5} 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_{2,5} or PM₁₀ emission inventories, visibility impact assessments, health risk assessments, and source–receptor studies related to PM_{2,5} and PM₁₀ 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. SO₂, NO_x, ammonia, SO₃, 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 PM₁₀ and PM_{2,5} 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_{2,5} or PM₁₀ mass concentrations less than or equal to about 1,0 mg/m³.

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/cm³ 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_{2,5}, PM₁₀, and/or total suspended particulate matter

3.22

PM_{2,5}

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_{2,5} corresponds to the “thoracic convention” as defined in ISO 7708:1995, 6.

3.23

PM₁₀

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

Note 1 to entry: PM₁₀ 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 (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 PM_{10} cyclone (cm)
D_{50}	particle cut diameter (μm)
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 (g/mol)
M_s	molecular mass of stack gas
M_w	molecular mass of wet gas (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 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 (m/s)

$V_{w,std}$	volume of water vapour (scm)
v_n	sample gas velocity in the nozzle (m/s)
ΔH	pressure drop across the orifice meter
Δp	Pitot tube velocity pressure differential
Δp_m	measured velocity pressure
Δp_n	velocity pressure differential point n during the test run
Δp_{s1}	velocity pressure adjusted for combined cyclone Pitot tube
Δp_{s2}	velocity pressure corrected for blockage
Δp_1	velocity pressure differential measured at point 1
$(\sqrt{\Delta p})_{avg}$	average of square roots of the velocity pressure differentials measured during the preliminary traverse
β_1	0,027 754 for metric units
η	gas viscosity (μPa)
^a	1 μP = 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 PM_{10} , a sample is extracted from a stationary stack at a predetermined constant flow rate through an in-stack PM_{10} and a cyclone $\text{PM}_{2,5}$. The first cyclone separates particles with nominal aerodynamic diameters of 10 μm and the following cyclone, particles with nominal diameters of 2,5 μm . Sampling is conducted isokinetically, at sampling points on the sampling cross-section determined according to ISO 9096. After sampling, the 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 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_{10} 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 $PM_{2,5}$ sampling. Sampling that is “isokinetic” within the range 90 % to 115 % is required for PM_{10} sampling.

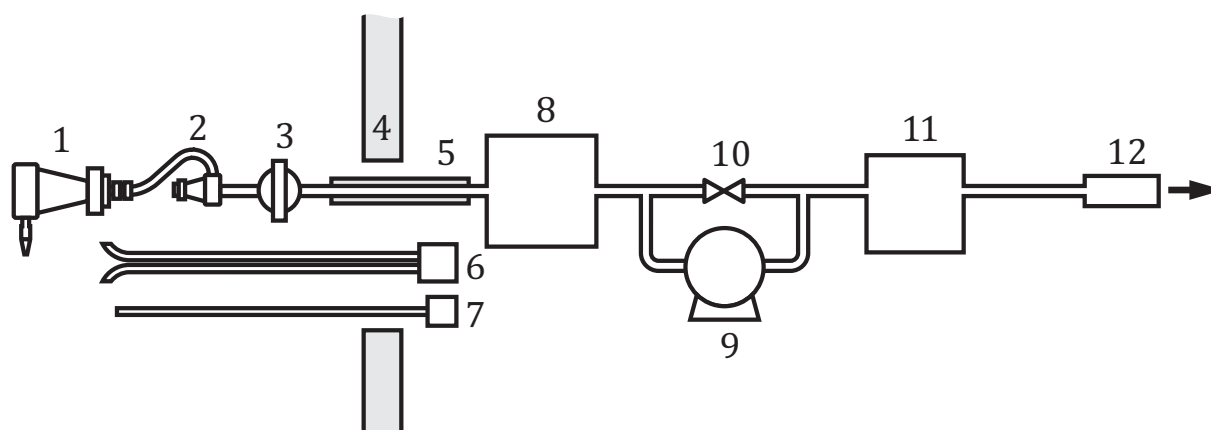
5.1.4 Method using sampling cyclones and sample dilution

To determine both filterable and condensable PM_{10} and $PM_{2,5}$ 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.



Key

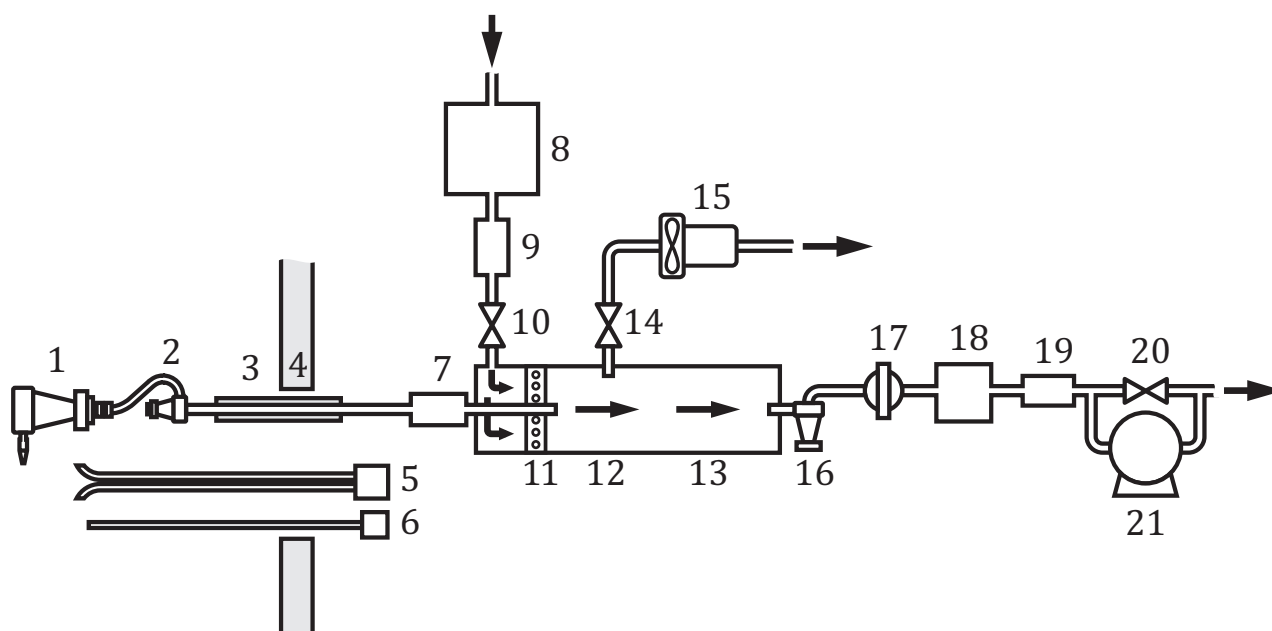
1	PM_{10} cyclone	5	heated probe	9	pump
2	$PM_{2,5}$ cyclone	6	Pitot tube	10	bypass valve
3	filter	7	temperature sensor	11	dry gas volume meter
4	stack/duct wall	8	moisture removal system	12	flow rate meter

Figure 1 — In-stack combined PM_{10} and $PM_{2,5}$ 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_{10} and/or $PM_{2,5}$ 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 $PM_{2,5}$ cyclone a single stage PTFE membrane filter or equivalent and the particulate matter and condensable collected subsequently measured gravimetrically.



Key

1	PM ₁₀ cyclone	8	dilution air-conditioning system	15	bypass pump or fan
2	PM _{2,5} cyclone	9	flow rate meter	16	PM _{2,5} 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 PM₁₀ and PM_{2,5} 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 SO₂, NO_x, ammonia, SO₃, 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 μm or 10 μm 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 μm (first cyclone);
- particles with aerodynamic diameters between 10 μm and 2,5 μm (second cyclone);
- particles with aerodynamic diameters smaller than 2,5 μm (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 μm and 10 μm . [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, PM₁₀ and or PM_{2,5} 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 μm and/or 10 μm . 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_{2,5} 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 ± 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 μm 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_{2,5} or PM₁₀ mass concentrations approximately less than or equal to 1,0 mg/m³.

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 CO₂ 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 CO₂ 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/m³ to ISO acceptable units.

- a) For sources with particle concentrations of 0,1 mg/m³ 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_{2,5} cyclone designed according to the dimensions in [Figure A.2](#) to collect a PM_{2,5} 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_{2,5} 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_{2,5} “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 ± 4 %.

6.3 Analytical balance

6.3.1 Precision. 95 % confidence uncertainty bounds: 5 μg for filter diameter less than or equal to 70 mm; 50 μg for filter diameter greater than 70 mm.

6.3.2 Resolution. Filter size <70 mm: 1 μg . Filter size ≥ 70 mm: 10 μg .

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 ± 2 °C over 24 h and the mean RH should be maintained between 45 % and 55 %, with a variability of not more than ± 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.