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

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

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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.

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

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

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

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

Introduction

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

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

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

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

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

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

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

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

1 Scope

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

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

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

2 Normative references

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

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

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

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

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

3 Terms and definitions

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

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

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

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

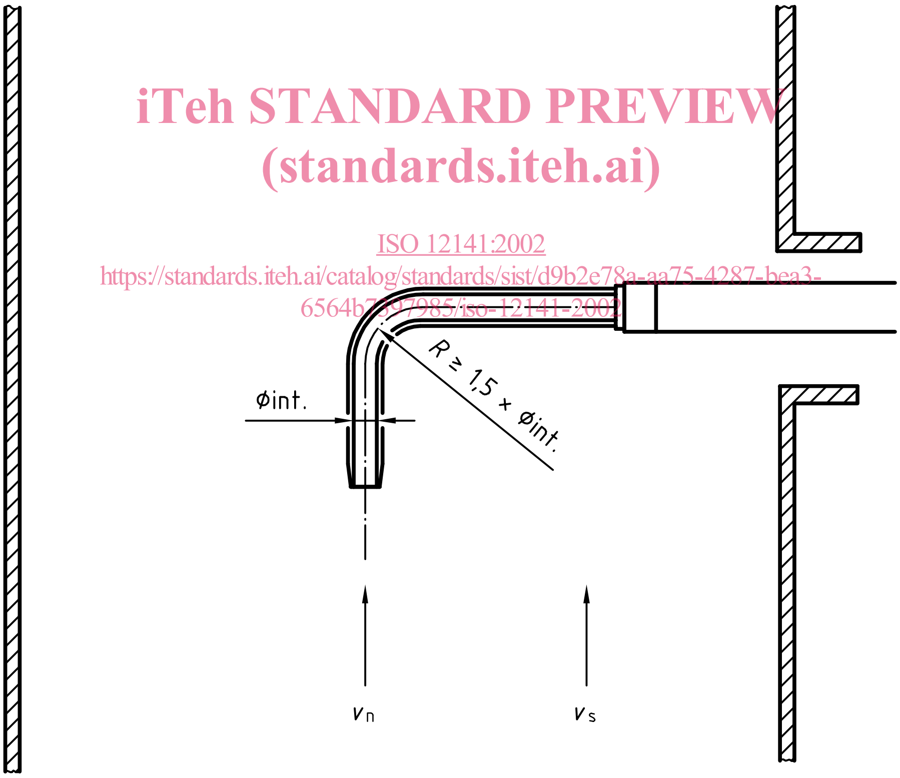
3.3
in-stack filtration
filtration in the duct with the filter in its filter holder placed immediately downstream of the sampling nozzle

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

3.5
isokinetic sampling
sampling at a flowrate such that the velocity and direction of the gas entering the sampling nozzle (v_n) are the same as that of the gas in the duct at the sampling points, v_s

See Figure 1.

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



$v_s = v_n$

Figure 1 — Isokinetic sampling in duct

3.6
hydraulic diameter
d_{h}
characteristic dimension of a duct cross-section

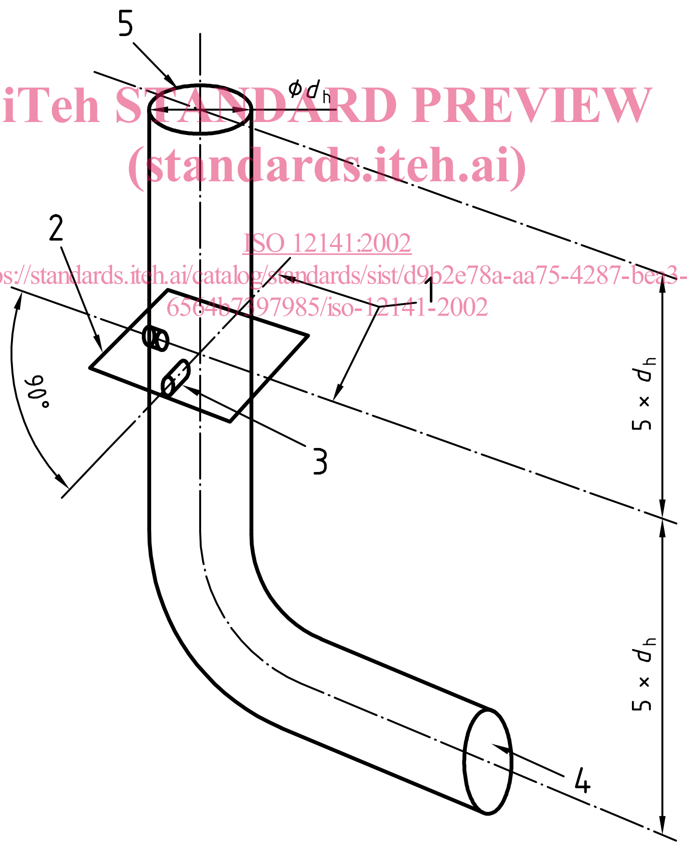
$$d_{\mathrm{h}} = \frac{4 \times \text{area of sampling plane}}{\text{length of perimeter of sampling plane}} \quad (1)$$

3.7
sampling plane
plane normal to the centreline of the duct at the sampling position

See Figure 2.

3.8
sampling line
line in the sampling plane along which sampling points are located, bounded by the inner duct wall

See Figure 2.



Key

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

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

3.9
sampling point
the specific position on a sampling line at which a sample is extracted

3.10
standard conditions
gas pressure and temperature constants and conditions to which volumetric calculations are referred

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

3.11
overall blank
test sample taken at the plant site in an identical manner to the normal samples in the series, except that no gas is sampled during the test duration

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

3.12
weighing control
procedure for the detection/correction of apparent variations in mass due to possible changes between pre- and post-sampling weighing conditions, by using parts, identical to those to be weighed for dust measurements, pretreated and weighed under the same conditions of temperature and humidity (e.g. desiccator) as pre- and post-sampling

NOTE The control parts are kept free from contamination.

3.13
measurement series
successive measurements carried out in the same sampling plane, and under the same process conditions

3.14
limit value
average limit value
dust concentration that is permitted by authorities for the plant process

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

3.15
high-volume sampling
sampling at rates higher than typical in ISO 9096 by using larger-diameter nozzles and higher flowrates to maintain isokinetic sampling conditions

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

4 Principle

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

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

Valid measurements can be achieved only when:

a) the gas stream in the duct at the sampling location has a sufficiently steady velocity (see 5.2);

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

c) samples are taken at a preselected number of stated positions in the sampling plane, to allow for a non-uniform distribution of dust in the duct or stack;

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

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

f) the overall blank value does not exceed 10 % of the daily limit set for the process;

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

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

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

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

5 Sampling plane and sampling points

5.1 General

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

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

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

5.2 Sampling plane

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

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

a) the angle of gas flow is less than 15° with regard to the duct axis (a recommended method for estimation is indicated in annex B);

b) no local negative flow is present;

c) the gas velocity is at least the minimum for the flowrate-measuring method used (for Pitot tubes a differential pressure larger than 5 Pa);

d) the ratio of the highest to lowest local gas velocities is less than 3:1.

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

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

5.3 Minimum number and location of sampling points

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

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

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

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

Table 1 — Minimum number of sampling points for circular ducts

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

[a] Using only one sampling point may give rise to errors greater than those specified in this International Standard.

[b] For large ducts, 20 sampling points is generally sufficient.

Table 2 — Minimum number of sampling points for rectangular ducts

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

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

[b] Using only one sampling point may give rise to errors greater than those specified in this International Standard.

[c] For large ducts, 20 sampling points is generally sufficient.

5.4 Access ports and working platform

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

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

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

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

6 Apparatus and materials (see summary annex E)

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

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

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

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

6.2 Sampling equipment.

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

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

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

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

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

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

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

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

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

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

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