
**Stationary source emissions — Sampling
for the automated determination of gas
emission concentrations for permanently-
installed monitoring systems**

*Émissions de sources fixes — Échantillonnage pour la détermination
automatisée des concentrations d'émission de gaz pour des systèmes
fixes de surveillance*

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ISO 10396:2007

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Reference number
ISO 10396:2007(E)

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Published in Switzerland

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 10396 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 1, *Stationary source emissions*.

This second edition cancels and replaces the first edition (ISO 10396:1993), which has been technically revised.

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Introduction

This International Standard describes the use of methodologies for the sampling of stack gases for the determination of pollutants by automated measuring systems (AMS). The methodology applies to the sampling of oxygen (O₂), carbon dioxide (CO₂), carbon monoxide (CO), sulfur dioxide (SO₂), nitrogen monoxide (NO) and nitrogen dioxide (NO₂), or the sum of NO and NO₂ as nitrogen oxides.

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Stationary source emissions — Sampling for the automated determination of gas emission concentrations for permanently-installed monitoring systems

1 Scope

1.1 General

This International Standard specifies procedures and equipment that will permit, within certain limits, representative sampling for the automated determination of gas concentrations of effluent gas streams. The application is limited to the determination of oxygen (O₂), carbon dioxide (CO₂), carbon monoxide (CO), sulfur dioxide (SO₂), nitrogen monoxide (NO) and nitrogen dioxide (NO₂), or the sum of NO and NO₂ as nitrogen oxides.

1.2 Limitations

It is recognized that there are some combustion processes and situations that may 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:

- a) corrosive or highly reactive components such as ammonia, hydrogen chloride and sulfuric acid;
- b) high vacuum, high pressure or high temperature gas streams;
- c) wet flue gases;
- d) fluctuations in velocity, temperature or concentration due to uncontrollable variation in the process;
- e) gas stratification due to the non-mixing of gas streams;
- f) measurements made using environmental control devices;
- g) relatively low levels of gas concentrations.

2 Normative references

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

ISO 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 document, the following terms and definitions apply.

3.1

condensate

material that is condensed in the sample conditioning equipment

3.2

corrosiveness

tendency of an enclosed gas stream to attack sampling equipment components or other exposed surfaces under sampling conditions

3.3

mass concentration

mass of a particular gas per unit volume of dry gas in the enclosed gas stream, unless otherwise stated

NOTE If expressed by volume concentration, these concentrations could be standardized by using a reference excess air level (for example, 3 % oxygen).

3.4

highly reactive component

contaminant in the gas stream that could react to form other chemical components

3.5

wet flue gas

gas stream that is at or below the saturation point and may contain droplets of water

3.6

sample integrity

maintenance of the representativeness of the sample by the elimination of leaks or physical and chemical reactions in the sample gas between the sample inlet and the measuring instrument

3.7

gas stratification

gas stream that forms a laminar layer without turbulence or a condition where the concentration at any point differs from the average concentration over the cross-section by more than 10 %

3.8

***in situ* sampling system**

non-extractive system that measures the gas concentration directly

NOTE *In situ* systems measure either across the stack or duct or at a point within the duct or stack.

3.9

calibration gas mixture

gas of known, reliable and stable composition that may be used to check the response of the AMS and should be used for the calibration of the AMS

4 Principle

4.1 General

This International Standard provides guidance for representative sampling of gases in a duct and includes both extractive and non-extractive methods. In extractive sampling, these gases are conditioned to remove aerosols, particulate matter and other interfering substances before being conveyed to the instruments. In non-extractive sampling, the measurements are made *in situ*; therefore, no sample conditioning other than filtering of filterable materials at the probe tip is required.

4.2 Extractive sampling

Extractive sampling includes extraction of the sample, removal of interfering materials and maintenance of a representative gas concentration throughout the sampling system for subsequent analysis by appropriate instrumentation (see Figure 1).

4.3 Non-extractive sampling

Non-extractive sampling does not involve removal of a sample, and sampling is confined to the gas stream in the duct (see Figures 2 to 4).

5 Representative sampling — Factors to be considered

5.1 Nature of the source

5.1.1 Important factors

To ensure the gaseous concentration in the sample gas stream is representative of that in the flue gas, several factors shall be considered:

- a) the heterogeneity of the process stream, such as variations in concentration, temperature, or velocity across the duct caused by moisture or gas stratification;
- b) gas leakage or air infiltration and continuous gas reactions;
- c) random errors due to the finite nature of the sample and the sampling procedure adopted to obtain a representative sample.

5.1.2 Problem areas in representative sampling

Obtaining a representative sample may be difficult to achieve for the following reasons:

- a) the operation of the source is not stable;
- b) the concentration level of the gas to be determined is either too low or too high;
- c) the size of the stack or duct is too large;
- d) the configuration of the duct network where samples are to be extracted is convoluted.

Where there are difficulties due to the nature of the source as noted in 5.1.1, the concentration profile shall be established for each operating condition in order to determine the best sampling location.

Some sources may have more variability in process (i.e. cyclic variation) and, consequently, any time-dependent measurement may be less representative of the average concentration if a full cycle of variability is not sampled.

5.1.3 Source characteristics

Before any measurements are carried out, it is necessary to become familiar with the pertinent operating characteristics of the process from which emissions are to be sampled and determined. These operating characteristics shall include, but are not necessarily limited to, the following:

- a) mode of process operation (cyclic, batch charging, or continuous);
- b) process feed rates and composition;
- c) fuel feed rates and composition;

- d) normal operating gas temperatures and pressures;
- e) operating and removal efficiency of the pollution control equipment;
- f) configuration of the ducts to be sampled that might lead to gas stratification or flow disturbances;
- g) volumetric gas flow rates;
- h) expected gas composition and likely interfering substances.

CAUTION — Exercise caution if the duct to be sampled is under pressure or vacuum, or at a high temperature.

5.2 Location

5.2.1 Inspection parameters

An inspection of the physical characteristics of the test site shall be carried out in order to evaluate factors such as:

- a) safety of the personnel;
- b) location of the flow disturbances;
- c) accessibility of the sampling port;
- d) available space for the sampling equipment, analyser and possible sampling platform requirements;
- e) availability of suitable electrical power, compressed air, water, steam, etc.;
- f) sampling port locations.

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SAFETY PRECAUTIONS — The electrical equipment used shall be in accordance with the local safety requirements. Where a potentially explosive or hazardous atmosphere is suspected, particular attention is required and precautions shall be taken to ensure the safety of the operations.

5.2.2 Sampling site location

Before any sampling is undertaken, it is necessary to determine any spatial or temporal fluctuations in the gas concentrations, and to carry out a preliminary survey of the gas concentration, temperature and velocity. [When the mass flow rates of gases have to be determined, detailed velocity measurements (ISO 10780) are required.] Measure the concentration, temperature and velocity at the sampling points several times to obtain their spatial and temporal profiles. Conduct this survey when the plant is operating under conditions that will be adhered to during the test, in order to determine whether the selected sampling position is suitable and whether the conditions in the duct are satisfactory (see 5.1.2).

This survey may not be needed if the spatial or temporal fluctuations in the duct can be determined from the owner's investigation, a previous investigation or the process characteristics in advance of the survey. In this case, the information relating to previous procedures for the determination of the sampling point and the adoption of one point sampling shall be described in the report.

It is necessary to ensure that the gas concentrations measured are representative of the average conditions inside the duct or stack. The requirements for the extractive sampling of gas may not be as stringent as those for particulate material. It is important that the sampling point is not located near any obstructions that could seriously disturb the gas flow in the duct or stack. The pollutant may have cross-sectional variation. The concentration at various points of the cross-section shall first be checked in order to examine the presence of gas stratification or air infiltration indicating that the gas to be measured is stratified, and if an alternative acceptable location is not available, multipoint sampling is then required.

Conduct a stratification test in the following manner.

- With the unit(s) operating under steady-state conditions at normal load, use a traversing gas sampling probe to measure the pollutant and diluent (CO_2 or O_2) concentrations at a minimum of twelve (12) points located at sampling locations as specified in ISO 9096. Use automated analytical methods for the measurement of the gas concentrations. Measure for a minimum of 2 min at each traverse point. While traversing, measure the pollutant and diluent gas concentrations from the centre of the stack to determine if temporal, rather than spatial, variations in the flue gas concentrations are occurring.
- Calculate the average pollutant and diluent concentration at each of the individual traverse points. Then calculate the arithmetic average concentrations for the gas from all of the traverse points. The pollutant or diluent gas is said to be “nonstratified” if the concentration at each individual traverse point differs by no more than $\pm 10\%$ from the arithmetic average concentration for all of the traverse points.

Usually, the cross-sectional concentration of gaseous pollutants is uniform, because of the diffusion and turbulent mixing. In this case, it is only necessary to sample at one point within the stack or duct to determine the average concentration. A gas sample should be extracted near the centre of the sampling site, positioned one-third to halfway in the stack or duct. When using non-extractive systems, a representative location should be similarly selected.

5.3 Other factors

The principle of operation and the components of the instrument systems can significantly affect the degree to which a collected sample is representative of the measured gas in the source. For example, a point sampling extractive system requires more attention to sampling site location than an across-the-stack *in situ* sampling system. Furthermore, sampling lines should not be composed of materials that have gas adsorbing properties that can affect the response time of the measurement section (see Tables A.1 to A.4).

Care should be taken to preserve the integrity of the sample taken, by a good selection of equipment, and appropriate heating, drying and leak testing, etc. In addition, other factors such as corrosion, synergies, reaction with components, decomposition and adsorption might affect the integrity of a sample (see Clause 6).

6 Apparatus and equipment

6.1 Construction materials

Recommended construction materials are listed in Annex A.

6.2 Components of extractive sampling equipment

6.2.1 Primary filter.

The filter medium shall be constructed of an appropriate alloy (such as a specific stainless steel cast alloy), borosilicate glass, vitreous silica, ceramics or another suitable material. A filter that retains particles greater than $10\ \mu\text{m}$ is recommended. A secondary filter might also be required (see 6.2.4). The filter medium may be located outside the duct or at the tip of the sample probe (6.2.2). If placed at the tip of the probe, a deflector plate is recommended to prevent particle build-up on the leading edge of the filter. This will prevent blockage of the filter. Care should be taken to avoid contamination of the filter with particulate matter where condensate may react with gases to give an erroneous result. Blowback systems using high pressure air may be used to periodically blow particulate matter off of the filter. Care should be taken to use heated, clean, dry air in such procedures to avoid fouling of the probe or condensation within the extractive system.

6.2.2 Probe.

6.2.2.1 Metal probes.

Metal probes are widely used for gas sampling. The choice of the metal depends basically on the physical and chemical properties of the sample to be taken and on the nature of the gas to be determined.

Mild steel is subject to corrosion by oxidizing gases and may be porous to hydrogen. Thus, it is preferable to have stainless steel or chromium steels which can be used up to 1 175 K. Other special steels or alloys can be used above this temperature. The probe shall be heated if there is condensation occurring in its interior (see Figures 5 and 6); it shall also be cooled by an air or water jacket when sampling is done in very hot gases but it shall not be below the acid dew-point. The diameter of the probes shall conform to the requirements of the sampling line (6.2.3).

When employed in an explosive atmosphere, metal probes should be properly or safely grounded.

6.2.2.2 Refractory probes (see Annex A).

Refractory probes are generally made of vitreous silica, porcelain, mullite or recrystallized alumina. They are fragile and may warp at high temperatures with the exception of vitreous silica. They may also crack with thermal shock.

Borosilicate glass probes can withstand temperatures up to 775 K and vitreous silica probes up to 1 300 K. Other refractories of ceramic materials can withstand much higher temperatures.

6.2.2.3 Polymer probes.

For sampling saturated gas streams such as those found downstream of wet de-sulfurization systems, probes made from polypropylene, polytetrafluoroethylene (PTFE), PFA or other non-reactive polymers may be used. Probes of this type usually incorporate a quartz-wool plug or PTFE membrane to remove water droplets from the gas stream. Polymer probes are usable with gas temperatures up to 90 °C.

6.2.3 Heated sampling line connected to moisture removal assembly.

The sampling line shall be made of stainless steel, polytetrafluoroethylene (PTFE), perfluoroalkoxyalkane (PFA) or other suitably inert material.

The tube diameter should be appropriately sized to provide a flow rate that meets the requirements of the analyser under selected line length and the pressure characteristics of the sampling pump (6.2.6) used.

Heated sample lines are required to prevent loss of soluble gas components into water or acid condensate. Heating may be unnecessary if the water and acids have been removed at the sampling probe (e.g. by dilution, Nafion membrane, or gas cooler.)

Heating may be unnecessary if only insoluble gases are measured (e.g. CO, O₂).

Unheated sample lines should only be used when the ambient temperature will never be low enough to cause freezing.

Heated sample lines shall be kept at a temperature of at least 15 K above flue gas temperature or water and acid dew-point. This temperature shall be monitored and controlled.

In order to reduce the residence time in the sampling line and the risk of physico-chemical transformation of the sample, the gas flow can be greater than that required for the analyser; only part of the sample is then analysed and the excess flow discarded through a bypass valve (see Figure 1). It may be necessary to heat the transport line to avoid condensation.

6.2.4 Secondary filter.

A secondary filter may be needed to remove the remaining particulate material, in order to protect the pump (6.2.6) and the analyser. It shall follow the sampling line (6.2.3) immediately downstream of the probe. A filter that retains particles greater than 1 µm is recommended. Acceptable materials are PTFE or borosilicate glass. The size of the filter shall be determined from the required sample flow and the manufacturer's data on the flow rate per unit area.

The filter shall be heated to a temperature not less than 15 K above the water and acid dew-point of the sampled gas. The secondary filter may also be an unheated filter. In this case, it shall immediately follow the water vapour removal (cooler) device.