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**Stationary source emissions — Sampling  
for the automated determination of gas  
concentrations**

**iTeh STANDARD PREVIEW**

*Émissions de sources fixes — Échantillonnage pour la détermination  
automatique des concentrations de gaz*

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## Foreword

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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.

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

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# Stationary source emissions — Sampling for the automated determination of gas concentrations

## 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 (O<sub>2</sub>), carbon dioxide (CO<sub>2</sub>), carbon monoxide (CO), sulfur dioxide (SO<sub>2</sub>), nitrogen monoxide (NO) and nitrogen dioxide (NO<sub>2</sub>). Although they are only mentioned briefly in this International Standard, detailed velocity measurements are required to determine the mass flow rates of gases.

### 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;
- 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) low levels of gas concentrations.

## 2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 7934:1989, *Stationary source emissions — Determination of the mass concentration of sulfur dioxide* — Hydrogen peroxide/barium perchlorate/Thorin method.

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

## 3 Definitions

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

**3.1 condensable material:** Moisture that is considered in the sample conditioning equipment.

**3.2 corrosiveness:** The tendency of an enclosed gas stream to attack sampling equipment components or other exposed surfaces under sampling conditions.

**3.3 gaseous concentrations:** The mass of a particular gas per unit volume of dry gas in the enclosed gas stream, unless otherwise stated.

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

**3.4 highly reactive source:** An enclosed gas stream that contains unstable components or devices for the removal of environmental contaminants that could react to form other chemical components as conditions change. The components may be formed either in the enclosed gas stream or in the sampling equipment and may cause inherent errors in sample analysis.

**3.5 wet flue gas:** A gas stream that is at or below the saturation point and may contain droplets of water.

**3.6 standard conditions:** 101,3 kPa (pressure) and 273 K (temperature).

**3.7 sample integrity:** The elimination of leaks or physical and chemical reactions in the sample gas between the sample inlet and the measuring instrument.

## 4 Principle

Representative sampling of gases in a duct, that 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 except filtering is required.

### 4.1 Extractive sampling

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

### 4.2 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 and 3).

## 5 Representativeness: Factors to be considered

### 5.1 Nature of source

**5.1.1** the representativeness of the determination of gaseous concentration in enclosed gas streams depends on several factors:

- 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** Representativeness may be difficult to achieve for the following reasons:

- nature of the source (e.g. cyclic, continuous or batch);
- concentration level of the gas to be determined;
- size of the source;
- configuration of the duct network where samples are to be extracted.

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** 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 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 leading to gas stratification;
- g) volumetric gas flow rates;
- h) expected gas composition and likely interfering substances.

NOTE 1 Caution must be exercised 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 site;
- d) available space for the sampling equipment and instrumentation and possible scaffolding requirements;
- e) availability of suitable electrical power, compressed air, water, steam, etc.;
- f) sampling port locations.

### SAFETY PRECAUTIONS

**The electrical equipment used shall be in accordance with the load 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

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 be not as stringent as those for particulate material. It is important that the sampling location be removed from any obstructions which will seriously disturb the gas flow in the duct or stack. The pollutant can have cross-sectional variation. The concentration at various points of the cross-section shall first be checked, in order to assess the homogeneity of the flow and to detect any infiltration of air or gas stratification, etc. If a preliminary analysis of cross-section at measurements taken indicates more than  $\pm 15\%$  variation in concentrations, and if an alternative acceptable location is not available, multipoint sampling is recommended.

This may be achieved either by moving the probe from point to point or having a probe with multiple access ports (see ISO 9096). 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 stack sampling site, positioned one-third of halfway in the stack or duct. When using nonextractive systems, a concentration as representative as possible is obtained, but care should also be

taken to ensure that the instrument location is representative.

### 5.3 Gas concentration, velocity and temperature profile

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. 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 sampling position is suitable and whether the conditions in the duct are satisfactory (see 5.1.2).

### 5.4 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 table A.1).

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 Equipment

Recommended construction materials are given in annex A.

### 6.1 Components of extractive sampling equipment

#### 6.1.1 Primary filter

The filter medium shall be constructed of an appropriate alloy (such as a specific stainless steel cast alloy), quartz borosilicate, ceramics or another suitable material. A filter that retains particles greater than  $10\ \mu\text{m}$  is recommended. A secondary filter might be required as well (see 6.1.4). The filter medium may be located outside the duct or at the tip of the sample probe (6.1.2). If placed at the tip of the probe, a deflector plate may be added 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.

## 6.1.2 Probe

### 6.1.2.1 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 a condensation occurring in its interior; it shall also be cooled by an air or water jacket when sampling in very hot gases.

NOTE 2 When employed in an explosive atmosphere, metal probes should be carefully grounded.

### 6.1.2.2 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 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.

### 6.1.3 Heated sampling line connected to moisture removal assembly

The sampling line shall be made of stainless steel, or polytetrafluoroethylene (PTFE).

The tube diameter should be adequate to provide a flow rate that is sufficient to feed the monitors, bearing in mind the sampling line length and the pressure characteristics of the sampling pump (6.1.5) used.

The sampling line shall be kept at a temperature of at least 15 K above the water and acid dew-point temperature of the sampled gas. The temperature has to be monitored.

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 analytical units; 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.

### 6.1.4 Secondary filter

A secondary filter may be needed to remove the remaining particulate material, in order to protect the pump (6.1.5) and analyser. It shall follow the sampling line (6.1.3) immediately downstream of the probe. A filter that retains particles greater than 1  $\mu\text{m}$  is recommended. Acceptable materials are PTFE or quartz borosilicate. 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.

### 6.1.5 Sampling pump

A gas-tight pump is used to withdraw a continuous sample from the duct through the system. This may be a diaphragm pump, a metal bellows pump or another type of pump. The pump shall be constructed of corrosion-resistant material.

The capacity of the pump shall be such that it can supply all the analysers with their required flows, plus a 10 % excess flow margin. A bypass valve can be placed across the pump to control the flow rate. This valve will lengthen the life of the pump if used frequently at lower flow rates. It should be noted that some commercially available conditioning modules may be supplied with the pump upstream of the refrigeration/permeation drier. In such cases, when high acidity and moisture are present in the gas stream, the pump should be operated at a temperature of at least 15 K above the acid dew-point. In addition, increased pump maintenance may be anticipated in such conditions.

### 6.1.6 Removal of water vapour

If a water removal procedure is employed, it shall be either a condensation/refrigeration or permeation drying method or a combination of both. Use of a desiccant will be permissible if it does not affect the actual concentration of gases such as CO. Each method has its own merits and the following points shall be noted:

- When the condensation/refrigeration method is employed, the removal shall take place outside the heated box.
- Where permeation drying is employed, half of the dryer tube (wet end) shall be heated to a temperature of at least 15 K above the gas dew-point. When acid is present in the sample, it may be necessary to consider an acid dew-point. The manufacturer's recommendations on the quantity of purge air shall be followed.

- c) Dilution may be used to reduce the water concentration to a level where water removal may not be necessary.

Since there are many possible conditioning systems on the market, no specific recommendation for component placement is made in this International Standard.

### 6.1.7 Analyser sampling line

The part of the sampling line associated with the analyser shall be made of a suitable material and of sufficient size to accommodate the requirements of the instrumentation.

Depending on whether a drying technique or dilution is used, heat tracing is required where the monitor is fed with wet gases; in other cases, it may also be required to avoid condensation upstream of the condenser, where the system extracts hot gases. If back flushing is used to clean the in-stack filter, a pressure-regulated valve shall be installed upstream of the conditioner to protect the equipment from damage by high pressure air.

### 6.1.8 Gas manifold

The gas manifold shall be made of a suitable material with a separate take-off for each analyser. It shall be of sufficient size to accommodate the gas flow requirements of all the instruments, yet small enough to keep the gas residence time to a minimum. The exhaust gas must be safely vented.

### 6.1.9 Vacuum gauge

A vacuum gauge, 0 to 100 kPa, may be installed on the suction of the sampling pump (6.1.5) to provide an indication of proper operation of the pump and to indicate clogging or leakage when a filter change or cleaning is required. Instead of a vacuum gauge, a flowmeter, with alarm-point monitoring mounted in the measuring gas line upstream of the analyser, can be used.

NOTE 3 It is necessary to ensure that an adequate calibration gas pressure be used to provide a sufficient flow rate for all the instruments. The calibration gas flow should be similar to that used during the continuous sample analysis. Care should be taken to avoid excessive pressurization of the analysers. The excess gaseous discharges from the manifold and instruments should be properly vented away from the working area.

### 6.1.10 Dilution

The dilution technique is an alternative to hot gas monitoring or to sample gas drying. Sample dilution is a technique for the following (see figure 4):

- reducing the concentration of the constituents of the gas to be measured to a level compatible with the range of the analytical unit;
- reducing the "water" dew point, by diluting the sample with a dry carrier gas so as to reduce the risk of condensation in the gas circuits;
- minimizing the interference of certain compounds present in the sample, the elimination of which would be particularly difficult, by reducing their relative variations in concentration (for example, dilution with dry air when the variations in oxygen content of the sample falsify the measurement of another constituent);
- slowing down the physico-chemical transformation of the sample because of the reduced reaction rates at low concentrations and low temperatures.

The dilution ratio shall be chosen according to the objectives of the measurement. It shall remain constant, because the calibration frequency of the measurement unit depends, among other things, on the stability of the dilution ratio.

NOTE 4 In the case of extractive sampling where water is removed, the measured concentrations are based on dry gas.

Many types of devices are suitable for use, such as:

- calibrated apertures (capillaries, sonic nozzles, needle valves, etc.);
- flowmeters by volume or mass;
- pressure and/or flow regulators;
- possibly, devices intended to correct the influence of the physical properties of the sample on the dilution ratio.

The nature and purity of the dilution gas shall be chosen as a function of each application. It is important that the gas being determined is not present in the dilution gas and that the dilution gas does not react with the constituent being analysed.

In addition, large dilution ratios may lead to very low concentrations of the constituent to be determined, as a consequence of which adsorption phenomena leading to significant errors in measurement may occur. In this case, appropriate materials should be chosen to eliminate these phenomena.

## 6.2 Components of non-extractive equipment

### 6.2.1 Point monitors

Non-extractive point monitors sample the gas stream in the duct at the tip of the probe (6.2.1.2). Gas is

monitored at one point or over a short path (less than 10 cm), depending upon the principle of measurement (see figure 2). Point monitors shall incorporate the components described in 6.2.1.1 to 6.2.1.7.

### 6.2.1.1 Instrument transceiver

The component of the instrument that senses the response of the measurement cell (6.2.1.3) and generates an electrical signal that corresponds to the concentration of the gas being measured.

### 6.2.1.2 Probe

A support for the measurement cell, extending from the instrument transceiver (6.2.1.1). Non-extractive point monitor probes shall meet the specifications of 6.1.2.1.

### 6.2.1.3 Measurement cell

A cell or cavity exposed to the gas stream, for the purpose of producing an electro-optical or chemical response to the gas concentration at the tip of the probe (6.2.1.2)

### 6.2.1.4 Probe filter

A porous ceramic or sintered metal tube or screen that minimizes the interference of particulate matter with the gas measurement.

### 6.2.1.5 Probe mounting

A flange installed at the sampling port, used to support the transceiver (6.2.1.1) and probe (6.2.1.2) assembly.

### 6.2.1.6 Gas calibration line

A tube used to inject calibration, reference, or zero gas into the measurement cell (6.2.1.3) for the purpose of calibrating the instrument.

### 6.2.1.7 Protective hood (optional)

A shroud that protects the transceiver from the ambient environment.

## 6.2.2 Path monitors

Non-extractive path monitors sample the gas stream in the duct on a line crossing the main part of the duct diameter at the sampling location (see figure 3). Path monitors shall incorporate the components described in 6.2.2.1 to 6.2.2.7.

### 6.2.2.1 Transmitter assembly

The component of the monitoring system that houses a light emission source and associated electro-optical components. The transmitter projects light through

the flue gas, to the receiver assembly (6.2.2.2) located on the opposite side of the duct.

### 6.2.2.2 Receiver assembly

The component of the instrument system that houses a detector and an associated electro-optical component. The detector senses the radiation projected from the transmitter assembly (6.2.2.1) and generates a signal that corresponds to the concentration of the gaseous component monitored.

In alternate designs, the receiver assembly is replaced by a retroreflector assembly. A retroreflector projects the light beam back to a transceiver, where a detector responds to the reflected light.

### 6.2.2.3 Protective windows

Windows or lenses at the interface between the flue gas and electro-optical assemblies, used to prevent flue gas from entering the assemblies.

### 6.2.2.4 Purge air blower

A device that blows clean air over the protective windows, to minimize particle deposition.

### 6.2.2.5 Alignment/calibration pipe (optional)

A pipe assembly used for support and/or calibration purposes. The monitor system is in a calibration mode when the pipe assembly is flushed with air and closed to prevent entrance of flue gas.

### 6.2.2.6 Anti-vibration system (optional not shown in figure 3)

A system that isolates the transmitter (6.2.2.1) and receiver (6.2.2.2) assemblies from duct vibrations.

### 6.2.2.7 Internal gas calibration cell (adsorption)

A cell designed to admit gases for the purpose of calibrating the instrument.

NOTE 5 Some systems may not sample the entire cross-section of the duct and care should then be taken to ensure representativeness, as for example with extractive sampling.

## 7 System operation

### 7.1 Checking for leaks

Checking for leaks shall be conducted by disconnecting the sampling line at the probe exit, plugging the line, and adjusting the vacuum to 50 kPa using the bypass valve; no leaks should be detected. When liquids or aerosols are present, routine checking should be performed using reference gases injected at the top of the sampling probe and the instrument inlet.



## 7.2 Calibration, function and set-up

Extractive sampling calibration is necessary for both the instrumentation and sampling line. In this case, an appropriate calibration gas insertion port is required. It is often necessary to provide two gas injection points for calibration, one of which is as close as possible to the point at which the sample is taken, and the other at the entry to the instrumentation (see figure 1).

Non-extractive sampling systems shall also be calibrated using calibration gases. A calibration cell may be required for path monitors.

Although costly to operate, it is possible to calibrate both extractive and non-extractive systems against extractive manual (wet chemical) standard methods at the discretion of the user.

The zero gas and the calibration gas are introduced as close as possible to the point at which the sample is taken without excessive pressure. Nitrogen may be used as zero gas.

In order to set the analyser, zero gas and, subsequently, calibration gas (with a concentration of approximately 70 % or 80 % of the full scale) are introduced. This operation is repeated once or twice if necessary.

In order to check the entire scale, for measuring equipment with a linear calibration function, four uniformly distributed calibration gas concentrations (approximately 20 %, 40 %, 60 % and 80 % of the full scale) are used. The calibration gas used for setting purposes can be diluted gradually. In the case of a non-linear calibration function, at least 10 measuring points are required.

The calibration function of the analysers can be checked by introducing calibration gases directly to the instruments. The setting of the analyser shall be checked regularly, for example weekly (period of unattended operation). The calibration function has to be checked at longer intervals (for example yearly) or after repairs to the analyser.

## 7.3 Integrity

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

## 7.4 Maintenance of sampling systems

The maintenance of the sampling system usually consists of performing the following operations:

- verification of the compliance with the safety regulations;
- verification of the operation of the safety devices;
- replacement of the consumed components of the treatment unit (filter, drier, etc.);
- adjustment of the operating parameter;
- checking of the utilities (water, power, calibration gases, etc.).

The sampling line maintenance shall be carried out frequently.