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

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British Standards

National foreword

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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

Reference number
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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.

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 (O_2), carbon dioxide (CO_2), carbon monoxide (CO), sulfur dioxide (SO_2), nitrogen monoxide (NO) and nitrogen dioxide (NO_2), or the sum of NO and NO_2 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 (O_2), carbon dioxide (CO_2), carbon monoxide (CO), sulfur dioxide (SO_2), nitrogen monoxide (NO) and nitrogen dioxide (NO_2), or the sum of NO and NO_2 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.

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 ± 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 µm 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, O_2).

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.

6.2.5 Water vapour removal systems.

If a water removal procedure is employed, it shall be either a condensation/refrigeration or permeation drying method or a combination of both (see Figure 7). 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.

a) When the condensation/refrigeration method is employed, the removal shall take place outside the heated box.

b) 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 determine the acid dew-point. The amount of dry purge air used shall follow the manufacturer's recommendations.

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

NOTE It may be desirable to use multistage water reduction methods because the rapid condensation of water can disturb the sampling if the temperature is reduced abruptly. Some sulfur dioxide will be lost due to the dissolution into the condensed water.

6.2.6 Sampling pump.

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

The capacity of the pump shall be such that it can supply sufficient stack gas volumes and required gas flow rates to all analysers. 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 at lower flow rates.

6.2.7 Sampling line associated with the analyser.

The sampling line connected to the analyser shall be made of a suitable material and of appropriate 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 supplied 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 a pressurized blow-back system is used, clean the probe filter(s), suitable precautions shall be taken to protect the rest of the equipment from damage due to the pressure-pulse.

6.2.8 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.

6.2.9 Vacuum gauge (optional).

A vacuum gauge, 0 kPa to 100 kPa, may be installed on the suction of the sampling pump (6.2.6) 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 flow meter, with alarm-point monitoring mounted in the measuring gas line upstream of the analyser, can be used.

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.

6.2.10 Dilution system devices.

The dilution technique is an alternative to hot gas monitoring or to sample gas drying. The sample dilution technique is typically applied for (see Figure 8):

- 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 monitoring system;
- 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.

Moisture measurement is necessary when it is required to calculate the gas concentration on a dry gas basis.

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

- calibrated apertures (capillaries, sonic nozzles, needle valves, etc.);
- flow meters 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.

NOTE Dilution ratios are dependent upon changes in the flue gas density. Changes in the flue gas temperature, molecular weight and total stack pressure can affect the ratio and resultant concentration measurements.

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.

6.3 Components of non-extractive equipment

6.3.1 Point monitors.

Non-extractive point monitors sample the gas stream in the duct at the end of the probe (6.3.1.2). 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 6.3.1.1 to 6.3.1.7.

6.3.1.1 Instrument transceiver.

The component of the instrument that senses the response of the measurement cell (6.3.1.3) and generates an electrical signal that corresponds to the concentration of the gas being measured.

6.3.1.2 Probe.

A probe provides the support for the measurement cell extending from the instrument transceiver (6.3.1.1). Non-extractive point monitor probes shall meet the specifications of 6.2.2.1.

6.3.1.3 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 (6.3.1.2).

6.3.1.4 Probe filter.

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

6.3.1.5 Probe mounting.

A flange installed at the sampling port, used to support the transceiver (6.3.1.1) and probe (6.3.1.2) assembly.

6.3.1.6 Gas calibration line.

A tube used to inject calibration, reference, or zero gas into the measurement cell (6.3.1.3) for the purpose of calibrating the instrument.

6.3.1.7 Protective hood (optional).

A shroud that protects the transceiver from the ambient environment.

NOTE 1 The water concentration must be measured in order to calculate the equivalent dry gas concentrations.

NOTE 2 Some systems may not sample the entire cross-section of the duct and care should then be taken to ensure that the sample is representative, as, for example, with extractive sampling.

6.3.2 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 Figures 3 and 4). Path monitors may incorporate the components described in 6.3.2.1 to 6.3.2.5.

NOTE 1 The water concentration must be measured in order to calculate the equivalent dry gas concentrations.

NOTE 2 Some systems may not sample the entire cross-section of the duct and care should then be taken to ensure that the sample is representative, as for example with extractive sampling.

6.3.2.1 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 (6.3.2.2) located on the opposite side of the duct in the single path method. In the light reflection method, the light emission source and a detector are located on the same side of the duct (see Figures 3 and 4).

6.3.2.2 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 (6.3.2.1) and generates a signal that corresponds to the concentration of the gaseous component monitored.

In the light reflection method (see Figure 4), 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.

6.3.2.3 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.

6.3.2.4 Purge air blower.

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

6.3.2.5 Internal gas calibration cell (absorption).

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

NOTE 1 A pipe assembly can be used for support and/or calibration purposes. The monitor system is in a calibration mode when the pipe assembly is flushed with a calibration gas and closed to prevent entrance of flue gas. An anti-vibration system that isolates the transmitter (6.3.2.1) and receiver (6.3.2.2) assemblies from duct vibrations can also be used (not shown in Figures 3 and 4).

The problems of light axis alignment distortion, etc. caused by temperature increasing shall be considered in the case of the path monitoring systems.

NOTE 2 Calibration of path monitors is difficult.

7 System operation

7.1 Checking for leaks

Checking for leaks in extractive systems 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. The leak flow, e.g. measured by pressure variation after evacuation of the train at the maximal under pressure reached during sampling, shall be below 2 % of the normal flow rate. During sampling, a leak check can be monitored by measuring continuously the concentration of a relevant gas component (CO_2, O_2, etc.) directly in the duct and downstream of the sampling train: any detectable difference between those concentrations indicates a leak in the sampling equipment parts located out of the stack. This leak shall then be investigated and rectified.

7.2 Calibration, function and set-up

Extractive sampling calibration is necessary for both the analyser and the 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 analyser (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 (i.e. ISO 7934 and ISO 11564) or instrumented standard methods (i.e. ISO 7935, ISO 10849 and ISO 12039) 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 calibrate the monitoring system, zero gas and calibration gas (with a concentration of approximately 80 % of the full scale) are introduced, preferably as close to the probe as possible.

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) can be 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 or the calibration of the system can be checked by introducing calibration gas at a point as close to the probe as possible. For a dilution system, calibration gases shall be introduced prior to dilution. The setting of the analyser shall be checked regularly, for example, weekly (period of unattended operation). The calibration function shall be checked at longer intervals (for example, yearly) or after repairs to the analyser.

7.3 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.);
- scheduled maintenance of the sampling probe.

The maintenance of the sampling system shall be carried out frequently.

8 Documentation

The sampling report shall refer to this International Standard, and shall include the following information:

a) description of the purpose of sampling, identification of the site, date of sampling and environmental control devices;

b) description of the operating conditions of the plant process, and any variation during sampling;

c) identification of the sampling location

    1) duct dimensions,
    2) number and position of sampling points,
    3) evidence of determination of the sampling points;

d) identification of gas parameters in the duct

    1) stack pressure,
    2) velocity and gas stratification condition,
    3) temperature,
    4) gas volumes and fluctuations in velocity;

e) flue gas components in the duct

   1) sample gas concentrations of effluent gas streams,

   2) water contents,

   3) O_2 concentration,

   4) corrosive or highly reactive components,

   5) condensate components;

f) characteristics and applied temperature of the equipment

   1) probe characteristics (material, size, type) and applied temperature,

   2) filter characteristics (material, size, type) and applied temperature,

   3) sampling line, gas manifold characteristics (material, size, type) and applied temperature,

   4) removal method of water vapour and characteristics,

   5) dilution method and characteristics (if relevant);

g) quality assurance

   1) leak-tests results,

   2) physical and chemical reactions in the sample gas,

   3) is operation of the source stable?

   4) result of calibration;

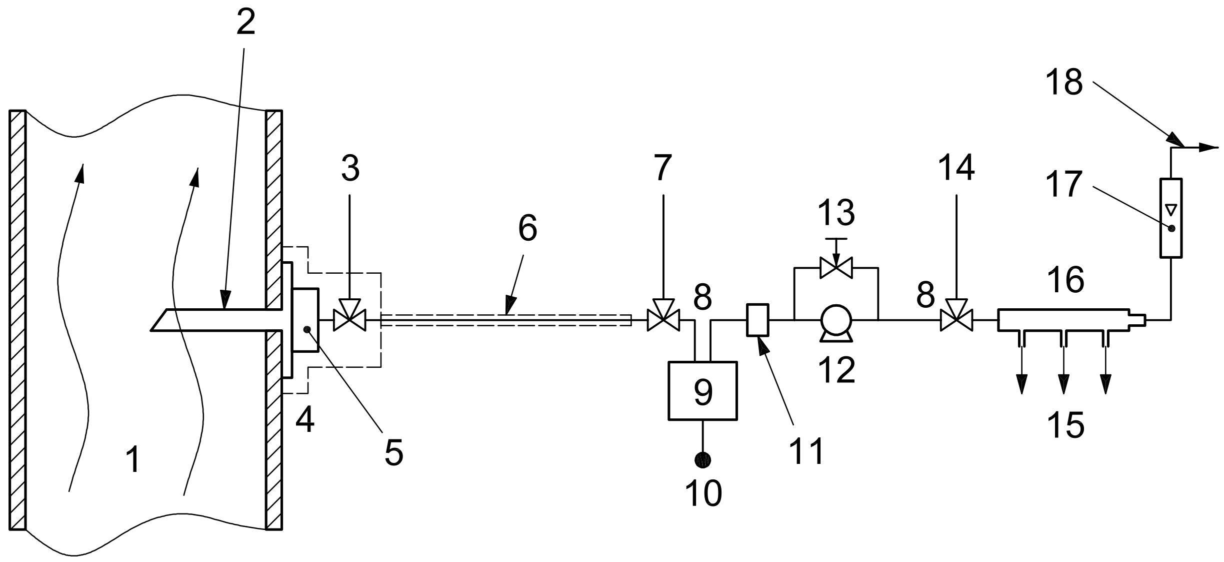
h) comments

   1) the sampling report shall also indicate any special circumstances that may have influenced the results, and any information concerning the uncertainty of the results,

   2) if it has been necessary to modify the method for any reason, then this modification shall be reported,

   3) if the representativeness may be difficult to achieve for the reasons of the nature of the source and the pertinent operating characteristics of the process, then the reasons and the modifications shall be reported.

These sheets shall be kept by the laboratory to demonstrate the traceability of the measurements.

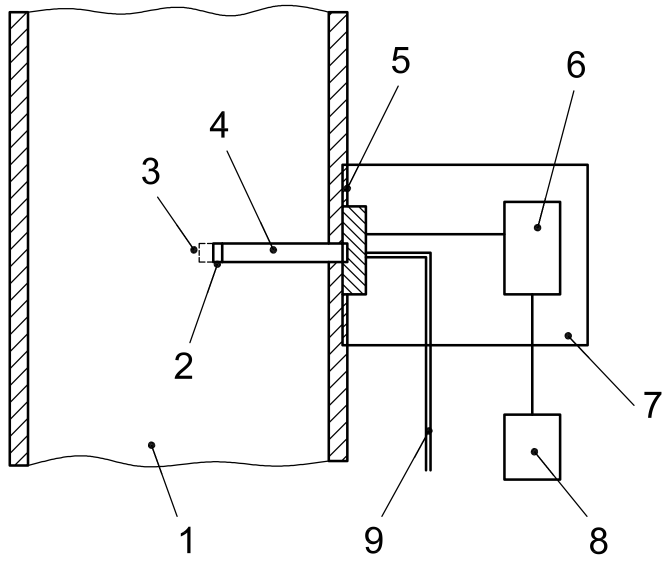


Key

1	flue gas
2	probe
3	calibration gas inlet (a)
4	heater
5	primary filter
6	heated sampling line
7	calibration gas inlet (b)
8	valve
9	dehumidifier unit
10	water discharge
11	secondary filter
12	sampling pump
13	bypass valve
14	calibration gas inlet (c)
15	to analyser(s)
16	manifold
17	flow meter
18	exhaust

The pump can be either placed before or after the dehumidifier depending on the system design.

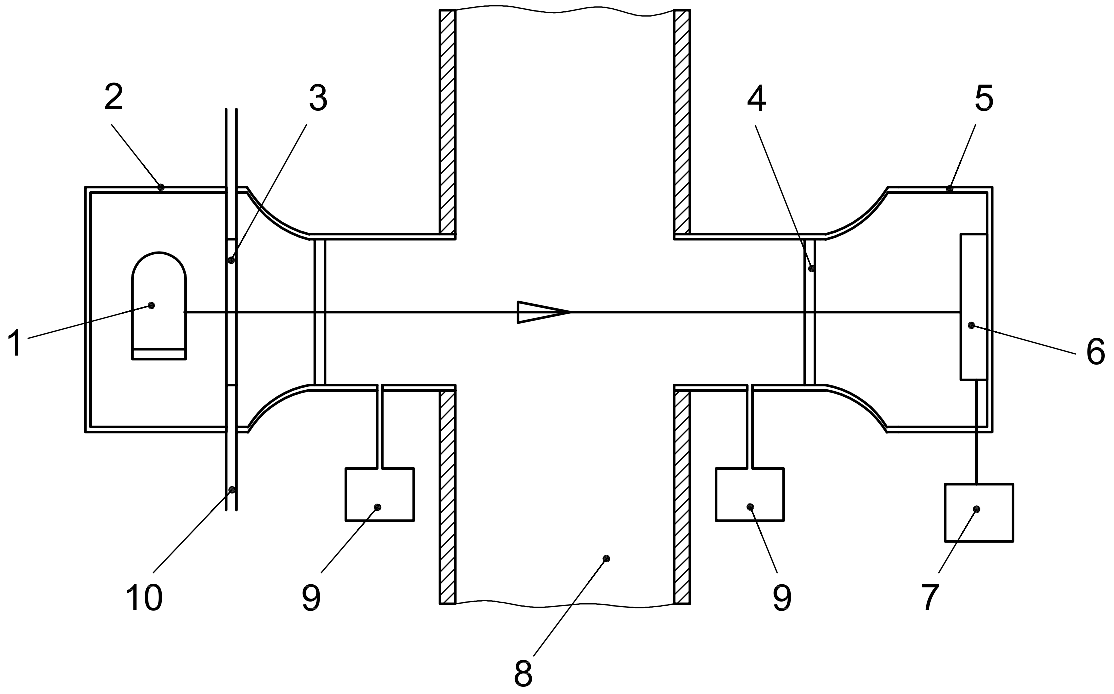
Figure 1 — Example of an extractive sampling and conditioning system



Key

1	duct or stack
2	measurement cell
3	probe filter
4	probe
5	probe mounting
6	instrument transceiver
7	protective hood
8	data recorder
9	gas calibration line

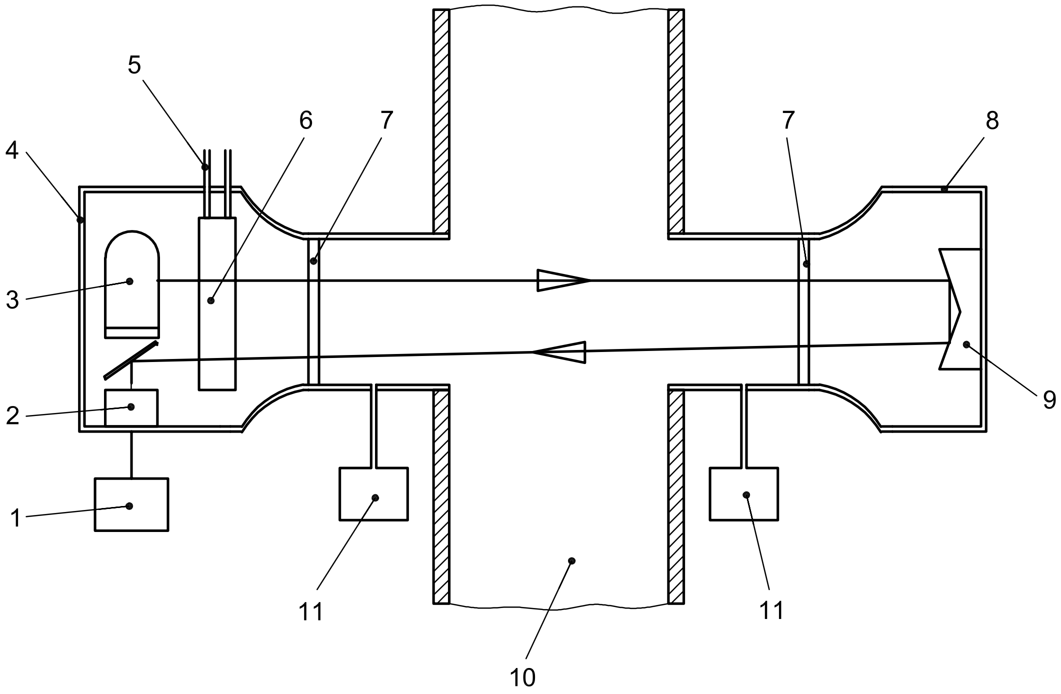
Figure 2 — Example of a non-extractive point monitor



Key

1	lamp	5	receiver assembly	8	stack or duct
2	transmitter assembly	6	detector and electric module	9	purge air blower
3	internal gas calibration cell	7	data recorder	10	gas calibration line
4	protective window				

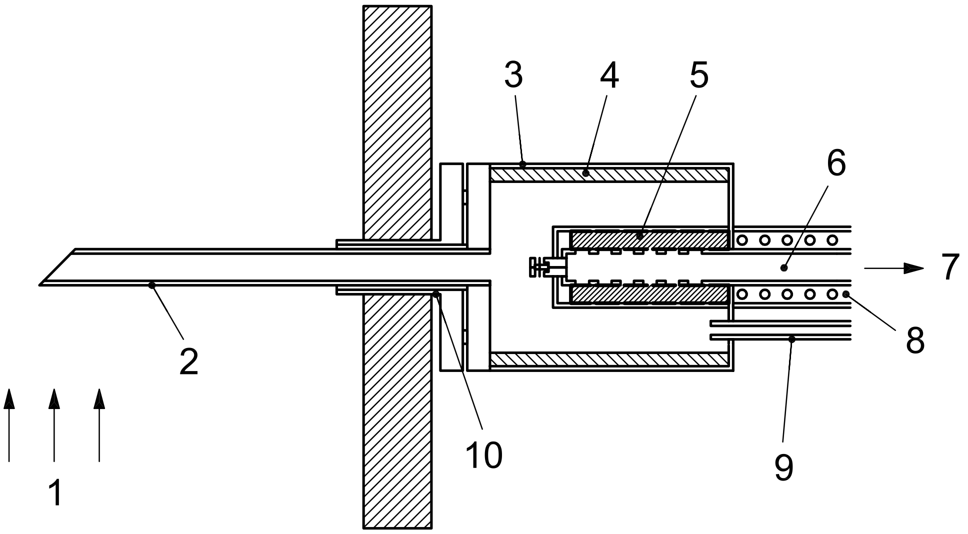
Figure 3 — Example of a non-extractive path monitor (single light path method)



Key

1 data recorder
2 detector and electric module
3 lamp
4 transmitter and receiver assembly
5 gas calibration line
6 internal gas calibration cell
7 protective window
8 reflector assembly
9 reflector
10 stack or duct
11 purge air blower

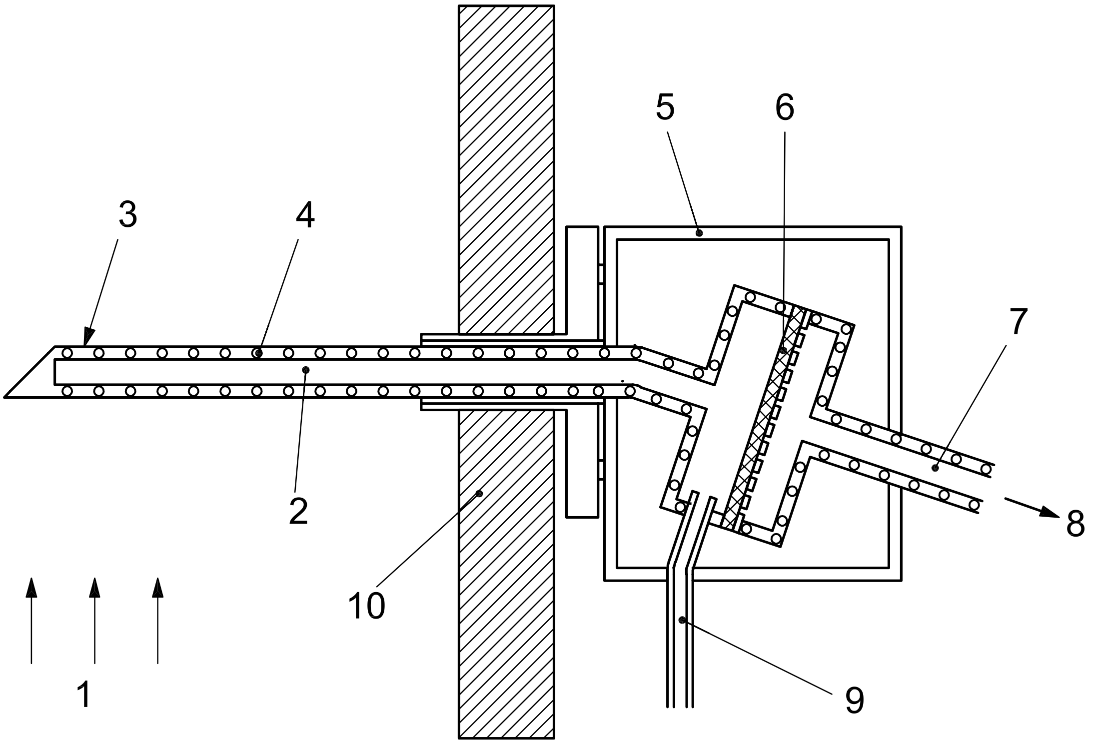
Figure 4 — Example of a non-extractive path monitor (light reflection method)



Key

1 flow of flue gas
2 probe
3 waterproof cover
4 heater
5 filter
6 sample line
7 gas
8 heater
9 calibration gas line
10 sampling port

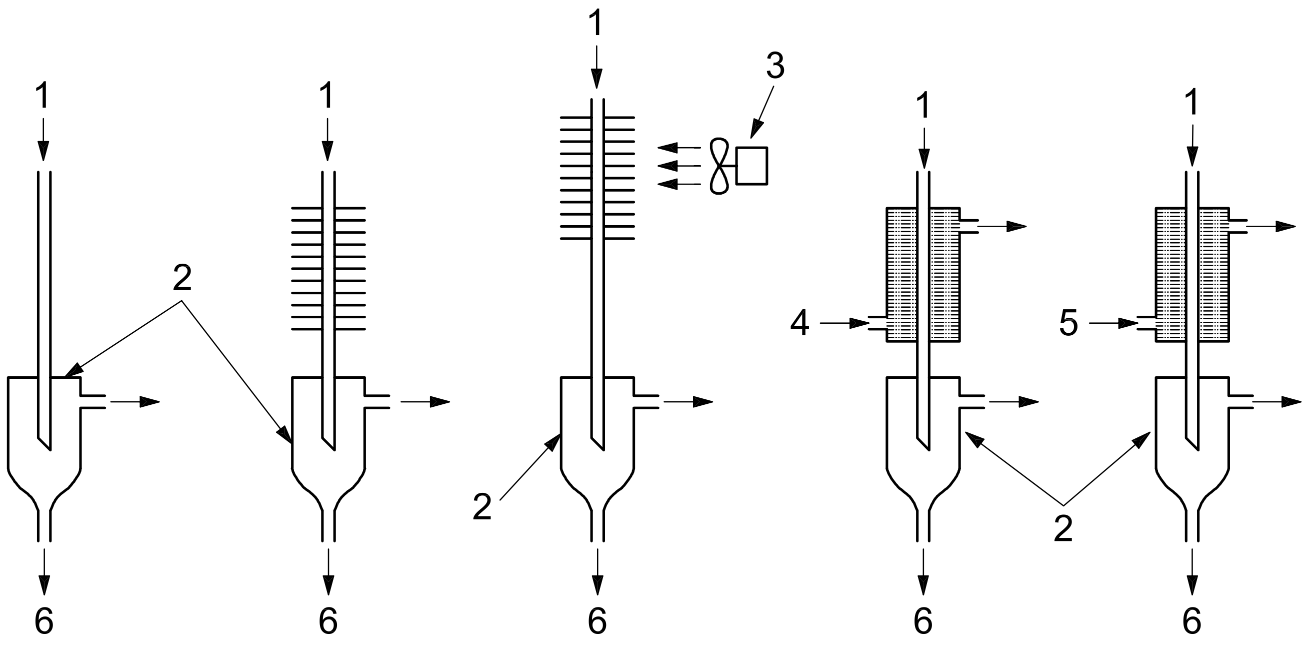
Figure 5 — Example of case where filtering material is attached at the rear stage of probe of extractive sampling system



Key

1	flow of flue gas	5	waterproof cover	8	gas
2	probe	6	filter	9	calibration gas line
3	protecting tube	7	sample line	10	duct wall
4	heater				

Figure 6 — Example of an electrically-heated-type probe with a protecting tube of probes of extractive sampling system



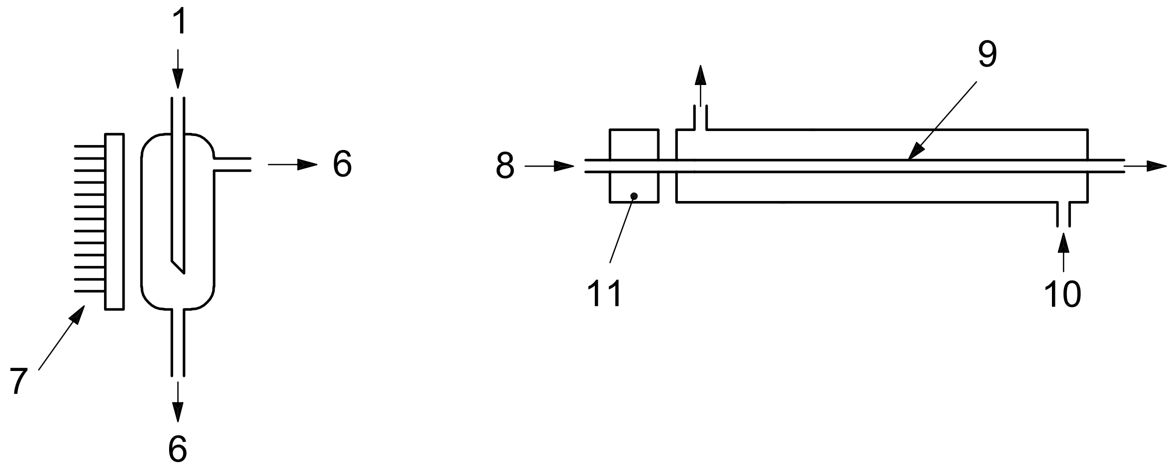
a) Room temperature cooling type

b) Room temperature cooling type

c) Forced air cooling type

d) Double tube water type

e) Refrigerated cooling type



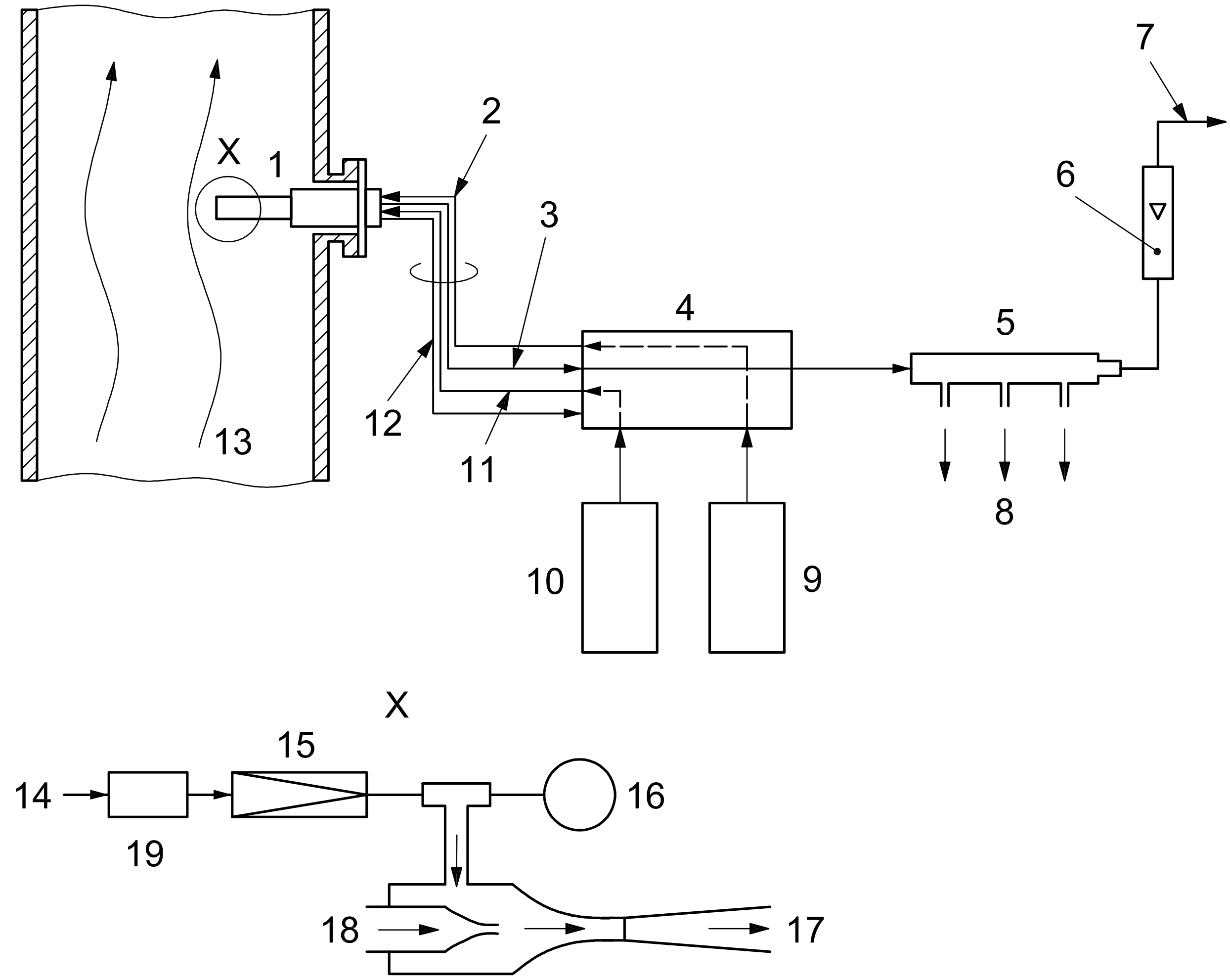
f) Electrically cooling type

g) Semipermeable material, Teflon or equivalent type

Key

1 sample gas
2 gas-liquid separator
3 blower for cooling
4 water
5 cooling with ethylene glycol coolant or other
6 condensed water
7 cooling module (Peltier element)
8 sample gas (or dried air)
9 permeation tube
10 dried air (or sample gas)
11 heater at least 15 K above the gas dew-point

Figure 7 — Examples of moisture removal systems



Key

X	Details of dilution probe
1	dilution probe
2	dilution air line
3	diluted sample line
4	control unit
5	manifold
6	flow meter
7	exhaust
8	to analyser(s)
9	pressurized air (zero gas)
10	calibration gas
11	calibration gas line
12	vacuum gauge line
13	flue gas
14	sample gas (Q2)
15	critical orifice
16	vacuum gauge
17	diluted sample (Q1+Q2)
18	dilution air (Q1)
19	filter

Flow rate (dilution air) = Q1.

Flow rate (sample gas) = Q2.

$$\text{Dilution ratio} = \frac{Q1 + Q2}{Q2}.$$

$$\text{Original source concentration} = \frac{Q1 + Q2}{Q2} \times \text{Measured concentration.}$$

Figure 8 — Example of a dilution probe

Annex A
(informative)

Recommended construction materials

A.1 Sample components

The choice of proper construction materials is a very important part of designing a sampling interface. Acceptable construction materials shall meet three criteria:

a) the materials shall have sufficient chemical resistance to withstand the corrosive constituents of the sample;

b) the materials shall not exhibit excessive interaction (reaction, absorption, and adsorption) with the sample gases;

c) materials used in or near the stack shall be heat resistant.

For a sampling system of minimum design, the above criteria shall be examined in the light of material costs.

A.2 Chemical resistance

The corrosive constituents encountered in monitoring sources are alkaline gas or acid gas such as nitrogen dioxide, sulfur dioxide, dilute nitric acid, dilute sulfurous acid, diluted or concentrated sulfuric acid [wet sulfur trioxide (SO_3) or acid mist], ammonia, and hydrogen chloride. The chemical resistances of various materials to these constituents have been collected from a number (8 to 12) of sources and are summarized in Table A.1 and Table A.2. All the materials are evaluated at room temperature and may be considerably less resistant at higher temperatures.

Of the metals, Carpenter 20 SS stainless steel and Hastelloys are the most resistant, followed by 316 SS, 304 SS, and finally aluminium. Borosilicate glass and PTFE (Teflon®) 1) are quite resistant to all components of the sample. Polyvinyl chloride (PVC) is somewhat less resistant to all constituents except concentrated nitric acid which is not anticipated to be present in the sampling interface. PVC is resistant to corrosiveness but may react with corrosive constituents. For measurements of nitrogen dioxide and sulfur dioxide at low concentrations in wet samples, PTFE, borosilicate glass and vitreous silica are recommended. Polyethylene and polypropylene have very nearly the same chemical resistance and are acceptable for use except where concentrated nitric acid is encountered (acid mist from sulfuric acid plant). Nylon is a material of apparent limited usefulness in sampling systems; while sources differ on the performance of Viton® 1) during resistance tests.

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1) Teflon® and Viton® are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

Table A.1 — Chemical resistance of various materials (sampling lines, sampling probe)

Material	Dry	Dry	Diluted	Diluted	Diluted	Concentrated [b]	Concentrated	Diluted							Max. temp.
	SO_2[a]	NO_2[a]	HNO_3	H_2SO_3	H_2SO_4	HNO_3	H_2SO_4	H_2S	HCN	NH_3	Cl	HCl	HF	Mercaptan	K
Stainless steel, type 304 SS	S (Some pitting observed)	S	S	Q	U	S	U	S	S	S	U	S	S	S	1 075
Stainless steel, type 316 SS [d]	S	S	S (< 0,051)[c]	S	S or Q (< 0,508)	S (< 0,508)	U (> 1,27)	S	S	S	U	S	S	S	1 075
Stainless steel, type Carpenter 20 SS [d]	S	S	S	S	S or Q	S	S	S	S	S	U	S	S	S	1 075
Hastelloy®	S	S	S	S	S	S	S	S	S	S	S	S	S	S	
Aluminium	S	N/A	S (0,127 to 0,508)	S (0,127 to 0,508)	Q (0,508 to 1,27)	U (> 1,27)	U (> 1,27)	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
Borosilicate glass	S	S	S (< 0,127)	N/A	S (< 0,127)	S (> 0,127)	S (> 0,127)	S	S	S	S	S	S	S	675
Vitreous silica	S	S	S (< 0,127)	N/A	S (< 0,127)	S (> 0,127)	S (> 0,127)	S	S	S	S	S	U	S	1 275
Polytetra-fluoroethylene	S	S	S	S	S	S	S	S	S	S	U	S	S	S	475
Unplasticized polyvinyl chloride	S	S	S	S	Q	U	S or Q	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
polyvinyl chloride	S	S	S or Q	S	S	Q or U	S or Q	S	S	U	S	S	S	U	345
Polyethylene	S	S	S or Q	S	S	U	Q or U	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
Polypropylene	S	S	S	S	S	U	Q or U	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
Nylon	–	S	S	U	U	U	U	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
Viton	S or U	S	S	S	S	S or Q	S or Q	N/A	N/A	N/A	N/A	N/A	N/A	N/A	

S = Satisfactory, Q = Questionable, U = Unsatisfactory, N/A = No data available

[a] Recommendation: If corrosivity plays a role, polytetrafluoroethylene (Teflon®) can be used for dry SO_2 and dry NO_2.

[b] High concentrations of HNO_3 are not anticipated in the sampling interface.

[c] Quantities in parentheses indicate corrosion rates, in millimetres per year.

[d] Stainless steel containing Mo introduces a positive error when NH_3 is present in the measurement of NO_2 because of the catalytic action of Mo contained in stainless steel on ammonia.

Table A.2 — Chemical resistance of various materials (connecting line, filter)

Parts	Material	Dry	Dry	Diluted							Max. temp.
		SO_2 [a]	NO_2 [a]	H_2S	HCN	NH_3	Cl_2	HCl	HF	Mercaptan	K
Filter	Sintered borosilicate glass	S	S	S	S	S	S	S	U	S	675
	Stainless steel net [b]	S	S	S	S	S	U	U	U	U	975
	Sintered stainless steel [b]	S	S	S	S	S	U	U	U	U	975
	Hastelloy	S	S	S	S	S	S	S	S	S	
	Porous ceramics	S	S	S	S	S	U	S	U	U	1 275
	Polytetra-fluoroethylene	S	S	S	S	S	U	S	S	U	475
Connecting line	Fluororubber	S	S	S	S	S	S	S	S	S	455
	Silicone rubber	S	S	S	S	S	U	S	S	S	425
	Chloroprene rubber	S	S	U	U	U	U	U	U	U	355

S = Satisfactory, Q = Questionable, U = Unsatisfactory

[a] Recommendation: If corrosivity plays a role, polytetrafluoroethylene (Teflon®) can be used for dry SO_2 and dry NO_2.

[b] Stainless steel containing Mo introduces a positive error when NH_3 is present in the measurement of NO_2 because of the catalytic action of Mo contained in stainless steel on ammonia.

A.3 Table A.2 — Additional information

a) SUS 316 and SUS 316L introduce a positive error when NH_3 is present in the measurement of NO_2 because of the catalytic action of Mo contained in stainless steel on ammonia.

b) In the case of a short application time, ethylene tetra-fluoride resin can withstand temperatures up to 535 K.

c) Because there are several kinds of materials among the stainless steel, such as 304 SS, 316 SS, Carpenter 20 SS, etc., they should be chosen in accordance with the kind of gas and its coexistences.

d) The reason to use borosilicate glass, but not to use vitreous silica for probe for hydrogen fluoride is that, though both materials are collected in absorbent as silicon tetra-fluoride after corrosion, vitreous silica is more corrosive than borosilicate glass and very expensive. Furthermore, the reason to use stainless steel for the probe, but not to use sintered stainless steel for filter materials is because the material with a hole diameter of 0,8 µm shall be used as filtering material for fluorine compounds.

e) The reason to use titanium for hydrogen chloride, but not to use for chlorine is because, though titanium resists wet chlorine, it reacts explosively with dry chlorine gas.

f) The maximum service temperature of stainless steel and borosilicate glass is prescribed as 100 K lower than the general service temperature that is 1 175 K and 775 K respectively. The reason is because stainless steel tends to increase the number of pin holes at high temperature and becomes more brittle under condition of 1 175 K and over. Because 775 K is the boundary temperature of the liquid phase and solid phase for borosilicate glass, the probe inserted at the sampling point tends to bend and/or to incur blockage depending on the flue gas velocity. These prescriptions are for safety purposes. In either event, the temperature of 1 175 K and 775 K is that of combustion processes and it is not a reasonable temperature at which to conduct flue gas sampling.

Table A.3 — General composition and characteristics of stainless steel

Stainless steel	General composition	Characteristics and use
SUS 304	18Cr-8Ni	Most widely used as heat resistant steel for food processing and general chemical facilities
SUS 316	18Cr-12Ni-2,5Mo	More non-corrosive and non-pinhole properties than SUS 304 with seawater and other media
SUS 316J1	18Cr-12Ni-2Mo-2Cu	Sulfuric acid resistant material, more non-corrosive and non-pinhole properties than SUS 316
SUS 317	18Cr-12Ni-3,5Mo	More non-pinhole properties than SUS 316J1, use for dyeing processing material
SUS 317J1	18Cr-12Ni-5Mo	Used for heat exchanger, nitric acid plant, phosphoric acid plant, bleaching process, which use solution containing chlorine ion
SUS 329J1	25Cr-4,5Ni-2Mo	Two phases structure material, which is highly acid and pinhole resistant and high strength, used as sea water resistant material
SUS 329J2L	25Cr-6Ni-3,5Mo-0,2N	Highly non-corrosive against dense chlorides and environment such as seawater, etc., strength and other characteristics are same or better than SUS 329J1
Carpenter 20 SS	20Cr-29Ni-2Mo-3Cu	Highly sulfuric acid resistant
Hastelloy A	57Ni-20Mo-20Fe	High mechanical properties under the high temperature and highly resistant against oxidation
Hastelloy B	64Ni-28Mo-6Fe	
Hastelloy C	57Ni-17Mo-6Fe-15Cr-4W	
As Mo added acts as catalyst in the case of measurement of low content, i.e. lower than 50 ppm, of nitrogen dioxide in flue gas, the negative errors occur in the measurement value.		

Table A.4 — Characteristics of fluoride resin

Chemical name			**Polytetrafluoroethylene**	**Tetrafluoroethylene-Perfluoroalkylvinylether Co-polymer**	**Tetrafluoroethylene-Hexafluoro propylene Co-polymer**	**Polychlorotrifluoro-ethylene**	**Polyvinylidene fluoride**	**Tetrafluoroethylene-Ethylene Co-polymer**	**Tetrafluoroethylene-Ehtylene Co-polymer: vitreous silica contained**	**Chlorotrifluoroethylene-Ethylene Co-polymer**
Trade name and others [a]			Teflon® Polyflon® Aflor®	Teflon®	Teflon® Neoflon®	Dyflon®	Kainer KF polymer	Tefzer afron COP	Teflon®: vitreous silica contained	Teflon®
Acronym			PTFE	PFA	FEP	PCTFE	PVDF	ETFE	ETFE	ECTFE
Characteristics	**Unit**	**ASTM**								
Physical										
Melting point	K	-	600	583	548	493	444	553	543	518
Specific gravity		D 792	2,14 – 2,20	2,12 – 2,17	2,12 – 2,17	2,1 – 2,2	1,75 – 1,78	1,70	1,80	1,68 – 1,69
Thermal										
Heat conductivity	10^{-4} cal-m-sec-K	C 177	6,0	6,0	6,5	4,7 – 5,3	3,0	5,7	5,7	5,8
Specific heat	cal/K/g	N/A	0,25	0,25	0,28	0,22	0,33	0,46 – 0,47	N/A	N/A
Coefficient of linear expansion	10^{-5}/K	D 696	10	12	8,3 – 10,5	4,5 – 7,0	8,5	9 – 9,3	1,7 – 3,2	8
Thermal deformation point		D 648								
18,5 kg/cm^2	K		328	320	323	N/A	363	347	483	350
4,6 kg/cm^2	K		394	347	345	399	405 – 422	377	538	389
Max. service temperature	K	(No load)	533	533	473	450 – 473	423	423 – 453	473	438 – 453
Durability, etc.										
Water absorption (24 h)	%	D 570	0,00	0,03	< 0,01	0,00	0,04	0,029	0,022	0,001
Combustibility [1/8-in thick]	-	(UL-94)	V-0	V-0	V-0	V-0	N/A	V-0	V-0	V-0
Oxygen index	-	D 2863	> 95	> 95	> 95	> 95	N/A	30	N/A	60
Sunlight effect	-	-	non	none	none	none	none	none	N/A	none
Weak acid effect		D 543	none	none	none	none	none	none	N/A	none
Strong acid effect	-	D 543	none	none	none	none	Degradated with fume sulfuric acid	none	N/A	none
Weak alkali effect	-	D 543	none	none	none	none	none	none	N/A	none
Strong alkali effect	-	D 543	none	none	none	none	none	none	N/A	none
Solvent effect	-	D 543	none	none	none	A little expansion with halogen compounds	Resistant against almost all solvents	none	N/A	well durable

Sources: the Modern Plastics Encyclopedia 76/77, with additional data from Du Pont Corp.

The heat resistance of ethylene tetrafluoride resin differs from the maximum service temperature in the case of continuous measurement. Note that of Teflon PFA is below 473 K, that of Neoflon FEP is below 423 K, and that of Diflon CTFE is below 393 K. The heating temperature shall be chosen based on the materials used.

[a] Teflon®, Polyflon®, Aflor®, Neoflon® and Dyflon® are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

Bibliography

[1] ISO 7934, *Stationary source emissions — Determination of the mass concentration of sulfur dioxide — Hydrogen peroxide/barium perchlorate/Thorin method*

[2] ISO 7935, *Stationary source emissions — Determination of the mass concentration of sulfur dioxide — Performance characteristics of automated measuring methods*

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

[4] ISO 10849, *Stationary source emissions — Determination of the mass concentration of nitrogen oxides — Performance characteristics of automated measuring systems*

[5] ISO 11564, *Stationary source emissions — Determination of the mass concentration of nitrogen oxides — Naphthylethylenediamine photometric method*

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

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