
**Stationary source emissions — Sampling
and determination of gaseous fluoride
content**

*Émissions de sources fixes — Échantillonnage et détermination de la
teneur en fluorure gazeux*

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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 15713 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 1, *Stationary source emissions*.

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Introduction

This International Standard describes a method for the measurement of the concentration of gaseous fluoride compounds in flue gas passing through ducts or chimneys.

The effects of fluoride containing species are of concern as irritants to humans when inhaled at high concentration and because of the potential for adverse effects on vegetation.

For the purposes of this International Standard, the fluoride measured is quoted as hydrogen fluoride. The aim of the method is to measure hydrogen fluoride, but, in practice, the parameter assessed is operationally defined. The fluoride measured is from those compounds that pass through a filter and dissolve in dilute sodium hydroxide and which produce fluoride ions which remain present in solution when analysed.

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Stationary source emissions — Sampling and determination of gaseous fluoride content

1 Scope

This International Standard is applicable to the measurement of the gaseous fluorides that are entrained in gases carried in stacks or ducts. The gaseous fluoride content is expressed as a mass of hydrogen fluoride in the stack gas.

This International Standard is applicable to all stacks emitting gases with fluoride concentrations of below 200 mg/m³. It can be used for higher concentrations, but then the absorption efficiency of the bubblers should be checked before the results can be regarded as valid. The detection limit of the method is estimated as 0,1 mg m⁻³, based on a sample volume of 0,1 m³. All compounds that are volatile at the filtration temperature and produce soluble fluoride compounds upon reaction with water are measured by this method. The method does not measure fluorocarbons. The concentration of fluoride in the adsorbent solution is then measured using an ion selective electrode. The amount of fluoride measured is then expressed as hydrogen fluoride by convention, though this may not reflect the chemical nature of the compounds, which are measured.

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 9096:2003, *Stationary source emissions — Manual determination of mass concentration of particulate matter*

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

3 Terms and definitions

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

3.1

isokinetic sampling

sampling at a flowrate such that the velocity and direction of the gas entering the sampling nozzle is the same as that of the gas in the duct at the sampling point

[ISO 9096:2003]

3.2

sampling point

specific position on the sampling section at which a sample is extracted

NOTE Sampling points are spread out over the sampling section in order to be representative of subsections of equal areas.

3.3
STP

standard conditions for temperature, 273,15 K, and pressure, 101,325 kPa

4 Principle

As a result of the high reactivity and solubility of hydrogen fluoride, particular precautions are required to minimize irreversible loss of the species of interest and to take a representative sample. Firstly, if incorrect materials are selected for the sampling probe, impingers and connecting tubing they could react irreversibly with the species of interest. Secondly, if condensed liquid droplets are present in the probe before the impingers the species of interest may dissolve in them and so not be included in the measurement. Further, where droplets are present in the flue, if the sample is not taken isokinetically, then it may not be representative.

The flow conditions at a sampling plane are determined before sampling. If there are droplets present, then isokinetic sampling at a number of sampling points is necessary. The homogeneity of the spatial profiles of the flue gas velocity, temperature and oxygen concentration is investigated. If there is significant variability in any of these parameters, but no droplets, then sampling is carried out at a number of sampling points but at a constant flow rate. If these parameters exhibit homogeneity, then sampling is carried out at a single point at a constant flow rate.

To determine the gaseous fluoride content of the flue gas, a representative metered sample of that gas is drawn through a heated sampling probe and filter. Any droplets, which may contain dissolved gaseous fluoride compounds, are evaporated in the heated probe. Particulate bound fluoride species that may be present as solid materials are removed by filtration of particulates at a controlled temperature. Gaseous fluoride compounds or more precisely those water-soluble fluoride compounds that pass through the filter are absorbed using a sampling train made up of a series of impingers containing a sodium hydroxide solution.

The concentrations of dissolved fluoride ions in the collected solutions are measured using the ion selective electrode technique.

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5 Reagents

To carry out the method, the following reagents are required to be of a recognized analytical grade. If they have changed visibly, they should be discarded.

5.1 Absorber solution, 0,1 mol/l NaOH solution.

5.2 Sample gas drying agent, self-indicating coarse grade silica gel.

5.3 Total ionic strength adjustment buffer (TISAB).

Sodium chloride

Sodium acetate trihydrate

Trisodium citrate monohydrate

Glacial acetic acid

Deionized or distilled water

5,0 mol/l NaOH

5.4 Calibration solutions.

Deionized or distilled water

Sodium fluoride

6 Apparatus

6.1 Introduction

A schematic diagram of the equipment for the sampling of gaseous fluorides is given in Figure 1. The apparatus consists of a sampling probe and filter assembly that may be heated if required, an impinger train containing sodium hydroxide solution to capture gaseous fluorides, a pressure gauge, a suction control valve, a suction pump, a gas meter, and a sample gas volume flow rate measurement system. A thermometer and manometer shall be included in the sample train to allow the temperature and relative pressure of the metered gas to be determined. A barometer shall be used to measure atmospheric pressure during the test in order that the volume of the gas sampled can be normalized to standard conditions of 273,15 K and 101,325 kPa.

6.2 Probe

The probe shall be a length of rigid tubing and shall be capable of withstanding the temperature within the duct. It shall be resistant to chemical attack from the various pollutants in the duct. In particular, the probe shall be resistant to fluoride attack to avoid sample loss. Suitable materials for fluoride sampling are silica or Monel[®] type alloys¹⁾.

The probe shall have a heating system capable of maintaining a gas temperature at its exit of at least 423 K or $> 20 \text{ K} \pm 5 \text{ K}$ above the dew point temperature, whichever is the higher.

The internal surfaces of the sample probe shall be cleaned thoroughly before each sample run by rinsing it with deionized water. Between samples, it will first be necessary to allow the probe to cool. The probe rinse shall be repeated until the rinse water shows no evidence of particulate matter.

6.3 Filter and filter housing

A filter shall be used to capture particulate material to prevent dissolution of any soluble particulate fluoride. Filters can be placed in the duct between the nozzle and the probe only if there are no droplets present, or out of the duct before the first impinger. If a filter is used outside the duct, it shall be heated to a temperature of at least 423 K or $> 20 \text{ K} \pm 5 \text{ K}$ above the dew point temperature, whichever is the higher, to avoid condensation. If the amount of particulate fluoride within the sample is below 10 % of the total, then the filter can be omitted.

Filters and filter holders shall be made of material resistant to attack by fluorides; for example, glass frits will remove gaseous fluoride and therefore cannot be used as filter supports. Filter holders shall have an airtight seal against leakage from outside or around the filter.

The filter shall be capable of withstanding prolonged exposures up to 40 K above the temperature setting and have at least 99,5 % collection efficiency for 0,3 μm diameter particles.

The filter housing shall be cleaned thoroughly prior to use and before each sample run using deionized distilled water until no particulate matter is present on the inner surfaces of the filter holder.

1) Monel[®] type alloys is an example of a suitable product(s) available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

6.4 Sampling train

The impingers shall be connected to the sampling probe using HF resistant materials. Suitable materials are polypropylene, polyethylene or Viton® tubing²⁾. The sampling train consists of a series of four impingers through which the sample gases are passed and the fluorides are removed into solution. The impingers can be made of quartz, polypropylene or polyethylene. Convenient sizes for impingers are 125 ml to 250 ml.

The first two impingers shall contain between 50 ml and 100 ml of 0,1 mol/l NaOH solution of analytical grade.

The third impinger shall be left empty to catch any carryover of the absorption solution.

The fourth impinger shall be used a drying unit. Its materials of construction need not be resistant to HF. It shall be filled with silica gel to dry the sample gas prior to the suction unit, gas meter and volume flow meter. Prior to use, the impingers shall all be rinsed and cleaned with distilled or deionized water and a bottlebrush.

During sampling, the gases shall enter the first impinger at its base and bubble up through the sodium hydroxide solution before entering the second impinger at its base.

The geometry of the impingers and quantity of absorbing solution employed shall be such that a gaseous fluoride absorbance efficiency of not less than 95 % is achieved at the chosen sampling flow rate and in the concentration range examined. Evidence that this criteria is met shall be demonstrated on at least one occasion at the maximum flow rate used with that design of equipment.

6.5 Suction unit

The pump is used to draw the sample through the sampling train. It shall be an airtight pump capable of maintaining the selected sampling flow rate throughout the sampling period and shall be adjusted using a flow regulator.

6.6 Thermometer

An airtight thermometer shall be fitted into the sample line after the drying unit and before the gas meter. The thermometer shall be capable of measuring absolute temperature to within 1 % of the absolute temperature.

6.7 Differential pressure gauge

The differential pressure gauge shall be used to measure the difference in pressure between the gas entering the volume meter and atmospheric pressure. It shall be capable of measuring pressure difference to within 1% of the differential pressure.

6.8 Gas volume meter

The volume of the dried sample gas shall be measured using a calibrated gas meter. The gas meter shall be capable of measuring the sampled gas volume to within 2 % of the actual volume.

6.9 Sample gas flow rate meter

A flow rate meter shall be used to ensure that the sample flow rate stays within the limits specified in section 6.4 and to perform the actions described in 7.5, 7.7 and 7.8. This meter shall be capable of measuring the flow rate to within ± 10 % of the flow.

6.10 Barometer

A barometer shall be used to measure the local atmospheric pressure in kilopascals (kPa) to within 1 % of the absolute pressure.

2) Viton® tubing is an example of a suitable product(s) available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

6.11 Working platform

A safe working platform shall be provided at the sampling position so that all the sampling points can be reached with safety.

7 Sampling

7.1 Sampling position and sampling points

The sampling position shall comprise of suitable access port(s) through which the sampling probe can be passed into the duct. The port(s) shall be capable of being sealed when not in use. Sampling can be performed either isokinetically (if droplets are present) or non-isokinetically at points according to the relevant requirements of ISO 9096. Multipoint sampling at a constant flow rate shall be carried out according to the relevant requirements of ISO 9096. Sampling will normally be carried out on at least two duct diameters and at a number of sampling points on each line.

Where this is not possible, due to constraints of either duct design or safety considerations, the sampling plane shall be situated in a length of straight duct, preferably vertical with a constant shape and constant cross-sectional area. It shall be as far as practicable downstream from any obstruction that may cause a disturbance or produce a change in the flow (for example, a bend, a fan or a damper).

The position at which a representative sample of the gas is to be taken is an important part of the sampling procedure. Representative sampling requires the gas to be taken from a homogenous flow in the duct. To achieve this, the velocity, temperature, and the oxygen concentration, shall first be determined at representative sampling points across the chosen sampling plane. The object of this procedure is to confirm that the velocity profile conforms to the requirement of ISO 9096. The temperature and oxygen shall not vary across the duct by more than 5 % from the mean value to avoid stratification affecting the measured concentration. If no stratification is detected, then a representative location shall be chosen for sampling.

7.2 Minimum sampling duration and minimum sample volume

The minimum sampling period and number of samples taken will depend on the nature of the process that is producing the emissions. The sampling duration shall be at least 30 min. If emissions from a cyclical process are to be measured, the total sampling period shall cover at least one cycle of the process operation.

The minimum sampling time also needs to take into account the detection limit of the sampling and analytical method. If the process is operating under steady state conditions, the minimum sampling time and volume can be calculated prior to sampling, by using the expected emitted gas concentration or a tenth of the emission limit value if appropriate and the sample train detection limits given in 10.1. If multiple point sampling is employed, the minimum sampling time at any one point shall not be less than 3 min.

7.3 Number and location of sampling points

An appropriate representative location shall be chosen for sampling. The number and location of the sampling points on the sampling plane shall conform to ISO 9096, unless this is not practicable.

Sampling at a single point shall only be acceptable if the flue gas velocity, temperature, and oxygen concentrations meet the requirements stated in 7.1.

If sampling from a number of points is required, then the sampling time chosen shall be the same at each sample point.