

**Flemish
Government**

**Ministerial Decree adopting the compendium for air sampling, measurement and analysis [LUC]**

**Legal bases**

This Decree is based on:

* the Decree of 5 April 1995 laying down general provisions on environmental policy, Article 5.4.1, inserted by the Decree of 25 April 2014, and Article 5.6.5, inserted by the Decree of 25 April 2014 and amended by the Decree of 8 December 2017;
* the Flemish Government Decree of 1 June 1995 laying down general and sector-specific provisions on environmental hygiene, Article 1.1.2, last amended by the Flemish Government Decree of 23 April 2021;
* the VLAREL of 19 November 2010, Article 45(§ 1)(2), inserted by the Flemish Government Decree of 3 May 2019.

**Procedural requirements**

The following procedural requirements have been met:

* the Recognitions team of the Area Development and Environmental Planning and Projects division of the Department of Environment proposed an amendment to the compendium for air sampling, measurement and analysis [LUC] on... (date);
* the Flemish Supervisory Committee for the Processing of Personal Data issued

Opinion No … on ... (date);

* the Council of State issued opinion No … on ... (date) pursuant to Article 84(§ 1)(1)(2°) of the Council of State Acts, coordinated on 12 January 1973.

**Background**

This Decree stems from the following aims:

* Following recent research and international and Flemish developments, adjustments are required to a number of existing methods in the compendium for air sampling, measurement and analysis [LUC].
* Article 45 of the Flemish Environmental Recognitions Regulation [VLAREL] of 19 November 2010 states that recognised laboratories shall apply the methods of the compendia for water [WAC], air [LUC], soil protection [BOC], the Manure Decree [BAM] and the Materials and Soil Decree [CMA] for the sampling, testing, measurements and analyses for which they are recognised. Cross-references exist between the compendia;
* for practical, organisational and legal reasons in the interest of laboratories, the government and the clients, the same date of

entry into force applies for all compendia. The proposed date is 1 September 2022. The laboratories have been informed of the new compendium for air sampling, measurement and analysis [LUC], which is available for viewing online.

THE FLEMISH MINISTER FOR JUSTICE AND ENFORCEMENT, ENVIRONMENT, ENERGY AND TOURISM HEREBY DECREES:

**Article 1.** The compendium for air sampling, measurement and analysis, abbreviated ‘LUC’ in Flemish, included in the Annex to this Decree, is hereby adopted.

**Article 2.** The Ministerial Decree of 22 January 2021 adopting the compendium for air sampling, measurement and analysis [LUC] is hereby repealed.

**Article 3.** This Decree shall come into force on 1 September 2022.

Brussels, (date)

The Flemish Minister for Justice and Enforcement, Environment, Energy and Tourism,

Zuhal DEMIR

Annex: Compendium for air sampling, measurement and analysis [LUC], as referred to in Article 1

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To be annexed to the Ministerial Decree of (date) approving the compendium for air sampling, measurement and analysis [LUC]

Brussels, (date)

The Flemish Minister for Justice and Enforcement, Environment, Energy and Tourism,

Zuhal DEMIR

Compendium for air sampling, measurement and analysis

# Determination of the content of PCDDs, PCDFs and dioxin-like and indicator PCBs

September 2021 version LUC/VI/002

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5. **SCOPE**

The following sampling and analysis standards apply for determination of the mass concentrations of PCDDs/PCDFs and dioxin-like PCBs in emissions within VLAREL packages L.9.1 and L.9.2:

* + NBN EN 1948-1: Stationary source emissions - Determination of the mass concentration of

PCDDs/PCDFs - Part 1: Sampling of PCDDs/PCDFs

* + NBN EN 1948-2: Stationary source emissions - Determination of the concentration of

PCDDs/PCDFs and dioxin-like PCBs - Part 2: Extraction and clean-up of PCDDs/PCDFs

* + NBN EN 1948-3: Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs - Part 3: Identification and quantification of PCDDs/PCDFs
	+ NBN EN 1948-4: Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs - Part 4: Sampling and analysis of dioxin-like PCBs

Standard NBN EN 1948-4 may also be used to measure PCB congeners other than the 12 non-ortho and mono-ortho PCBs such as indicator PCBs 28, 52, 101, 138, 153,

180. Additional PCBs may be included, provided a method validation is performed.

The above standards apply except where Section 2 of this compendium procedure expressly sets deviating or additional requirements.

## ADDITIONS TO OR DEVIATIONS FROM THE STANDARDS

* + Always take, analyse and report a field blank. The procedure ‘Essential quality requirements for emission measurements (LUC/0/005)’ gives the procedure and criterion for each standard.
	+ The first bullet in Section 5.6 of procedure LUC/0/005 indicates the maximum permissible relative expanded uncertainty of the gas volume meter and the temperature and pressure measurements at the gas meter.

## VALIDATION

In general, a measurement method under the Flemish Environmental Permitting Regulation [VLAREM] should be usable between 0.1 times and 3 times the emission limit. For standard methods, validate the following parameters:

* (Intra-)reproducibility
* Correctness, e.g. from ring test data
* Working range
* Limits of detection and quantification
* Measurement uncertainty

Where possible, validate these performance characteristics for the combination of sampling and subsequent analysis. If not possible, perform validation at least on the analysis method as per procedure WAC/VI/A/001. This procedure also gives definitions for the various performance characteristics.

## DETERMINATION OF MEASUREMENT UNCERTAINTY

Every recognised laboratory must have a measurement uncertainty assessment in place for the determination of PCDDs/PCDFs and dioxin-like PCBs in emissions, taking into account the contributions from the sampling on the one hand and the analysis on the other. The measurement uncertainty may be calculated using the ‘Guide to the expression of uncertainty in measurement’ or ‘GUM’, or the alternative top-down method detailed in procedure WAC/VI/A/002.

## REFERENCES

NBN EN 1948-1: 2006

Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs - Part 1:

Sampling of PCDDs/PCDFs

NBN EN 1948-2: 2006

Stationary source emissions - Determination of the concentration of PCDDs/PCDFs and

dioxin-like PCBs - Part 2: Extraction and clean-up of PCDDs/PCDFs

NBN EN 1948-3: 2006

Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs and

dioxin-like PCBs - Part 3: Identification and quantification of PCDDs/PCDFs

NBN EN 1948-4: 2010

Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs and

dioxin-like PCBs - Part 4: Sampling and analysis of dioxin-like PCBs

Compendium for air sampling, measurement and analysis [LUC] Essential quality requirements for emission measurements (LUC/0/005) <http://www.emis.vito.be/lne-erkenningen-lucht>

Method WAC/VI/A/001

Compendium for water sampling, measurement and analysis (WAC)

Performance characteristics https://emis.vito.be/nl/lne-erkenningen-water

Compendium for water sampling, measurement and analysis (WAC) Method WAC/VI/A/002

Measurement uncertainty

https://emis.vito.be/nl/lne-erkenningen-water

NBN ENV 13005: 2003

Guide to the expression of uncertainty in measurement

VLAREL

Flemish Government Decree adopting the Flemish Environmental Recognitions Regulation

<http://www.lne.be/themas/erkenningen/>

Compendium for air sampling, measurement and analysis

# Measurement of gas velocity and volume flow rate in a gas duct

September 2021 version LUC/0/004

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2. **SCOPE**

For the determination of gas velocity and flow rate in stacks and other gas ducts, the following standard applies unless this procedure indicates otherwise:

NBN EN ISO 16911-1

Stationary source emissions - Manual and automatic determination of velocity and volume flow rate in ducts - Part 1: Manual reference method

The standard applies to circular and rectangular flues with measurement sites set up as per the requirements of procedure LUC/0/001 and describes the measurement of the average velocity in a section by scanning or traversing, i.e. the measurement in a network of points located on different axes in a measuring plane. The axial gas velocity is measured at different points using a pitot tube (L-type, S-type or 2D and 3D-type pitot tube) or a vane anemometer. NBN EN ISO 16911-1 gives the selection criteria for use of the different types of pitot tubes and the vane anemometer. An S-type pitot tube is recommended for use in small measuring openings and for combined probes, in the presence of droplets and higher concentrations of dust. At gas velocities of less than 5 m/s or differential pressures of less than 5 Pa, use of a vane anemometer may result in a lower measurement uncertainty than use of a pitot tube. In cases of turbulence in the flue, a 3D-type pitot tube may be used.

Velocity measurement at different points is necessary due to an expected velocity gradient across the cross-section of the duct. Determine the gas flow rate in order to calculate the emission flow rate (in kg/h or g/h) of the pollutants present. Calculate the total volume flow rate as the product of the duct cross-section product (m²) and the average gas velocity (m/s), determined for a prescribed series of representative points. Moreover, a velocity measurement may be necessary based on isokinetic sampling in the case of dust components or droplets.

Some cases require velocity or flow rate determination due to an explicit request for this information.

The following alternative methods for determining the flow rate also appear in standard NBN EN ISO 16911-1 and are permitted for defined purposes:

* Calculation of the flow rate from energy consumption during a combustion process
* Tracer gas dilution method: determination of the flow rate from the dilution of a known concentration of an injected tracer
* Tracer transit time: determination of the volume flow rate based on the time it takes a tracer gas to travel a certain distance

This compendium procedure uses a pitot tube or anemometer to determine the average gas velocity and gas flow rate in a measurement cross-section.

The gas velocity is always expressed in meters per second; the waste gas flow rate is usually expressed in m³/h, under three different conditions:

* Waste gas flow rate in m³/h under duct conditions
* Waste gas flow rate in Nm³/h, under normal conditions (0 °C and 101.3 kPa)
* Dry waste gas flow rate under normal conditions

## PITOT TUBE MEASUREMENT PRINCIPLE

Determine the average velocity in the measurement section by using a pitot tube to take measurements for a specified period of time at a number of prescribed points in the duct. Calculate the waste gas flow rate from the average velocity at all measuring points multiplied by the area of the duct cross-section:

### Q = v  S  3600

where: Q = gas volume flow rate in m3/h, wet

v = average gas velocity in m/s

S = area of the measurement cross-section in m2

Pitot tube measurement is based on Bernoulli’s theorem:

Ptot = pC + pd (1)

ρ. v2

therefore:

pd  Δp 

2

v  K.

2 . Δp

ρ

(2)

(3)

where

Ptot total pressure (Pa) in the flue

PC absolute pressure in the flue = patm + pstat (avg) pd dynamic pressure (Pa)

p differential pressure measured across pitot tube = pd = Ptot – pC (Pa)

ρ density under duct conditions (kg/m³) v waste gas velocity (m/s)

K pitot tube calibration factor

The basic principle of a pitot tube is that one pressure opening is influenced by the flowing gas and one or more other pressure openings are exposed to the absolute pressure in the gas duct (duct pressure). Measure the dynamic pressure Δp as the difference between the total pressure and the absolute pressure, which is the differential pressure across the pitot tube connections. Use a micromanometer to take the reading.

Measure humidity, waste gas composition, absolute pressure and temperature to calculate the density of the waste gas.



*Figure 1. Principle for use of a pitot tube to measure gas velocity in a duct*

The method with an S-type pitot tube measures a higher pressure difference than pd with Bernoulli’s theorem. This requires insertion of a calibration factor K = 0.78 to 0.88.

The use of S-type pitot tubes is recommended for the following conditions where standard types cause problems:

* in gases containing dust and droplets, to avoid clogging the small holes of standard pitot tubes;
* at low flow velocities, where the higher response of the S-type pitot tube provides larger and thus more accurately measurable differential pressures;
* in ducts where only a small opening is available, in combination with thick walls that an L-type cannot pass through.

Standards ISO 10780 and EN 13284-1 state that an S-type pitot tube is more sensitive to misalignment with the gas flow direction. According to standard NBN EN ISO 16911 however, a laboratory evaluation shows that both L-type and S-type pitot tubes exhibit the same response to misalignment with the gas flow. Pitot tube misalignments that are limited to 15 to 20° do not lead to a significant (<1 %) change in the velocity reading.

To calculate the density of the waste gas, it is necessary to know or measure the absolute pressure in the flue, the temperature, the gas composition and the water content.

## EQUIPMENT

* 1. **PITOT TUBE**
		1. **STANDARD PITOT TUBE**



1. inner diameter of tube
2. outer diameter of tube
3. Eight openings with a diameter of 0.13*d*, not exceeding 1 mm, evenly distributed and free of burrs

*Figure 2. Standard or L-type pitot tube as per NBN EN ISO 16911-1 (AMCA type)*

The standard pitot tube consists of a cylindrical measuring head mounted perpendicular to a long lance, which may feature engraved distance markings. The tip is spherical, ellipsoidal or conical in shape with a central hole that measures the total pressure. At a prescribed distance from the tip, the device features various laterally drilled holes that measure the absolute pressure. The end of the lance is fitted with two nipples to connect the lines from the micromanometer. A direction indicator is mounted on the lance, parallel to the measuring head, to align the pitot tube with the tip pointed against the direction of flow.

The standard type of pitot tube is also sometimes called the Prandtl or L-type.

[Figure 2](#_bookmark10) is one example of a design with detailed dimensions as per NBN EN ISO 16911 (AMCA type). This standard covers other types that meet the standard requirements.

* + 1. **S-TYPE PITOT TUBE**

Figure 3 shows a diagram of an S-type pitot tube. Use of S- types is appropriate for gases containing droplets or dust, due to the larger openings, typically 4-10 mm. NBN EN ISO 16911 also permits other types of pitot tubes, such as 2D and 3D-types.



*Figure 3. S-type pitot tube and orientation in the gas flow*

It is not permissible to use an S-type pitot tube too close to a thermocouple or dust probe. This is because the thrust of the gas flow around these obstacles creates changes in pressure and direction that cause measurement errors. ISO 10780 gives the rules below (see [Figure 4](#_bookmark12)) on required distances from openings. Thus, when using a combined S-type pitot tube+thermocouple or sampling probe+S-type pitot tube+thermocouple, the thermocouple and/or sampling nozzle must remain an adequate distance from the pitot tube openings.



*Figure 4. Required distances between thermocouple and S-type pitot tube openings to prevent velocity measurement errors*

Flow rate measurements within a legal framework require the use of a pitot tube (possibly combined with thermocouple): it is not permitted to use a sampling probe fitted with a thermocouple and pitot tube.

* 1. **DIFFERENTIAL PRESSURE GAUGE**

It is necessary to measure differential pressure across a pitot tube in a gas flow with a micromanometer, such as a liquid-filled pressure gauge with adjustable slope, or with a calibrated electronic micromanometer. The minimum readable differential pressure must be 5 Pa.

Electronic differential pressure gauges are often more sensitive to fluctuations in waste gas velocity. Differential pressure fluctuations of over 10 % of the average reading require signal attenuation (NBN EN ISO 16911 A.2.2.3).

For pressure gauge liquid for the liquid-filled pressure gauge, it is permitted to use 95% ethanol with a red or blue dye, or better yet a mineral oil with known density (NB: pressure gauge oils from different brands have different densities; switching may result in serious velocity measurement errors).

Calibration and/or regular monitoring is required for both electronic and liquid-filled pressure gauges. It is not permitted for the expanded measurement uncertainty to exceed 1% of the reading or 4 Pa absolute (whichever is greater).

* 1. **TEMPERATURE GAUGE**

It is necessary to determine the gas temperature to calculate the density at each velocity measurement point. The temperature sensor used must be fine enough to ensure a quick response and limited influence from conductivity. The response time of the thermocouple must be less than 40 seconds. Under certain circumstances, shielding from radiant heat may be desirable.

The temperature gauge probe must be straight and rigid so it measures at exactly the same points as the gas velocity measurement. A combined pitot tube with permanently mounted thermocouple (without sampling probe) is recommended, also taking into account the distances as per ISO 10780.

Use thermocouples or Pt-100 sensors with a total measurement uncertainty of the end result, i.e. including those of all components and readout devices, of less than 1% of the absolute temperature.

* 1. **BAROMETER**

Use a calibrated barometer to measure atmospheric pressure. The measurement uncertainty must be better than 0.3 % of the measured air pressure or 300 Pa (ISO 10780).

* 1. **GAS ANALYSIS EQUIPMENT**

For the velocity measurement itself, gas analyses that result in an uncertainty of ≤ 0.05 kg/m3 for density in determining the density of waste gases will suffice.

In practice, a CO2 and oxygen measurement will suffice for this, along with measurement of the water content.

Airflow velocity measurement does not require gas analysis: humidity determination will suffice. The expanded measurement uncertainty in the moisture content determination must be less than ±20 % of the measurement value.

## MEASURING POINTS

Procedure LUC/0/001 applies for selection of the measurement section, measuring plane and number of measuring points. The measuring points are representative points for equal areas. For circular flues, NBN EN 15259 gives two diagrams for determination of equal areas: the general method and the tangential method. The gas velocity at the central point in the general method is not a measure of the average velocity in the central area, but rather gives a maximum value. Therefore, the tangential method is preferred for determining the flow rate. This method does not place a point at the centre of the flue.

## MEASUREMENT PROCEDURE

Measure the gas composition, water content and absolute pressure in the flue in advance to determine the waste gas density. For combustion and incineration plants, it may be possible to calculate the CO2 concentration if the O2 concentration is measured. Determine the temperature and differential pressure or velocity for each measuring point.

* For velocity measurements based on differential pressure measurements, measure the average differential pressure for at least 1 minute at each prescribed measuring point.
* The system logs the average reading electronically or the operator determines it visually. At least three instantaneous measurement values distributed over 1 minute are available for calculation of this average. In cases of wide fluctuations in differential pressures, visual reading is not permitted.
* Select a reference point to check the stability of the gas velocity and temperature:
	+ either set up a second pitot tube and temperature probe at the reference point, or
	+ if only one probe is available, repeat a measurement at the reference point at least once every 10 minutes;
	+ the average differential pressure, measured at least once every 10 minutes at the reference point, must not deviate by more than 10 % from the arithmetic mean of all the measurements taken there;
	+ the average gas temperature, measured at least once every 10 minutes at the reference point, must not deviate by more than 20 °C from the arithmetic mean of all the measurements taken there.
* Do not conduct the pre-sampling checks described in Section 9.3 of NBN EN ISO 16911-1, with the exception of the leak test on the pitot tubes (9.3.2). Do not perform this leak test with each measurement: it will suffice to check all pitot tubes for leaks twice a year in the lab (see also [9.2](#_bookmark24)).

## CALCULATIONS

Use formula (3) to determine the gas velocity vi at each individual measuring point. The density of the gas ρ for wet waste gases under duct conditions is determined as follows from the composition of the waste gas. Calculate the average molecular weight M of the waste gas from the mole fractions in the wet waste gases yj (equivalent to volume fraction) of all main components (yj > 1%), including water (j = N2, O2, CO2, H2O, etc.):

where

M = Σ (yj . Mj) (5)

yj = volume fraction or mole fraction of component j Mj = molecular mass of component j

M = average molecular mass of the gas

thus, ρN, the gas density under normal conditions (1013.25 hPa, 273.15 K), is:

ρN = M/22.4 (6)

Next, convert to duct conditions by correcting for temperature and pressure:

ρ = ρN . (Pk/1013.25) . (273.15/Tk) (7)

If the temperature differs by more than 5% at the measuring points, determine the density separately at each measuring point.

Obtain the average velocity of the n points measured with:

 v j

v  j

n

(8)

Next, obtain the volume flow rate under current temperature, pressure, water content and oxygen content conditions by multiplying the average velocity by the area of the duct cross-section A, in m²:

Q  v . A

(9)

Obtain the flow rate in normal cubic metres QN after correcting for temperature and pressure:

QN 

### Q. 273,15.

Tk

Pk 1013,25

(10)

where Pk= absolute pressure in the stack in mbar (= atmospheric pressure + static pressure) Tk= temperature of the gas in K

Obtain the flow rate in a normal cubic metre of dry gas QN,dr after correcting for the water content of the waste gas:

QN,dr

 QN . 100  %H2O

100

(11)

where %H2O: water content of the gas flow in per cent by volume

## PITOT TUBE CALIBRATION

Always calibrate all pitot tubes before commissioning. Calibrate all pitot tubes used in the field regularly, at intervals based on frequency of use (1 to 5 years). In addition to calibration of pitot tubes as per NBN EN ISO 16911-1 (e.g. in a wind tunnel to a laser Doppler anemometer as a reference instrument), calibration to a reference pitot tube in a wind tunnel as per ISO 10780 is also permitted. The latter method is further described below. Calibration is necessary at different speeds within the application range and must be metrologically traceable.

It is not permitted to use pitot calibration factors based solely on the design of the pitot tube.

#### Calibration by calibration bodies

It is preferable for pitot tubes to be calibrated by calibration bodies that are accredited for this. This enables traceability based on national standards or primary physical constants. The calibration certificate must include a reference to the accreditation of the body and a calibration uncertainty value.

#### Calibration in an in-house lab

To calibrate a pitot tube, an in-house laboratory must:

* have a detailed calibration procedure in place;
* have a wind tunnel that meets the standards (see below);
* evaluate the calibration uncertainty of each calibration.

#### Wind tunnel

The conditions on wind tunnels are given in documents such as ISO 10780. The wind tunnel used to calibrate pitot tubes must have a minimum diameter of 30 cm and an adequate cross-section so the pitot tubes inserted never exceed 3% of the area. The velocity variation in the gas flow will not exceed 1% at a gas velocity of 11 to 18 m/s during the time needed to read the standard pitot and the pitot tube being calibrated. The gas velocity must be adjustable up to at least 18 m/s.

#### Calibration procedure for calibration to a reference pitot tube (ISO 10780)

Use a standard pitot tube with known calibration factor as a reference. Perform calibration for at least two different velocities distributed across the measuring range (but not at velocities of less than 5 m/s, due to the greater measurement uncertainty), in the following steps:

1. Check connections for leaks and zero the micromanometer by connecting the two connections.
2. Start the fan and wait for the airflow to stabilise.
3. Insert reference pitot tube at a measuring point determined by pretesting to be free of gradients and rotating currents. This measuring point is preferably located in the centre of the tunnel, and in any case at least 10 cm from the wall. Seal measuring opening so air cannot leak in or out. Check the direction of the pitot tube. Note the dynamic pressure Δpref, expressed in pascals.
4. Remove reference pitot tube and install the pitot tube to be calibrated in the same place.

Seal and align the opening around the pitot tube and note the differential pressure Δpx in Pa.

1. Repeat steps 3 and 4 to obtain a set of three usable measurement pairs.
2. Calculate the calibration factor Kx for each measurement pair using the equation below and determine the average calibration factor. If any of the calibration factors determined deviates from the average by more than 0.02, repeat the calibration or replace the pitot tube. The same requirement applies for deviations between the factors determined at the different velocity levels.

Kx 

Kref

(12)

1. To calibrate an S-type pitot tube, determine the calibration factor with first one then the other opening pointed upstream. Only use S-type pitot tubes if the difference between the two calibration factors is less than or equal to 0.01.

Δpref Δpx

Use the average of the calibration factors obtained or – at higher numbers of test velocities – a value obtained by regression.

Three repetitions for each point will suffice for the ISO 10780 calibration procedure. Determination of calibration uncertainty also requires more data (at least six values) to calculate the standard uncertainty. This may be performed once, or it may be calculated from the collection of data at all velocities, e.g. by linear regression.

Deviations from the wind tunnel and the above procedures may be acceptable for laboratories that only conduct calibration for internal use. However, they must investigate the impact of these deviations and factor this into the calibration uncertainty.

For instance, the two pitot tubes enable working at a different point at the same time. In this case, also evaluate the difference in velocity between the two measuring points.

#### Calibration frequency

The calibration frequency for pitot tubes depends on the type and frequency of use.

* It is always necessary to calibrate all pitot tubes before commissioning. Barring any mechanical damage, repeated calibration is not necessary.
* Calibrate all types used in the field once every 1 to 5 years, depending on the frequency of use.

## DETERMINATION OF GAS FLOW DIRECTION

#### With standard pitot tube

A standard pitot tube provides acceptable gas velocity measurements as long as the orientation of the tube does not deviate from the direction of flow by more than 15°. At deviations of over 15°, the dynamic pressure reading declines sharply. At a horizontal orientation of 90°, the pressure reading will be negative. Rotate the pitot tube to find the orientation with the maximum differential pressure. This orientation corresponds to the direction of flow. This method can identify the presence of rotating currents or backflows.

#### With S-type pitot tube

S-type pitot tubes enable accurate measurement of the gas velocity, provided that the direction of the gas flow does not deviate from the pitot tube orientation by more than 15°. At a horizontal orientation of 90°, the pressure openings of the S-type pitot tube are parallel to the flow and the pressure reading on the micromanometer is zero. The gas flow direction may therefore be determined by turning the S-type pitot tube until the pressure gauge indicates zero. This means the direction of flow is at a 90° angle with respect to the pitot tube.

Standard NBN ISO 16911-1 states that if previous measurements, the geometry of the flue or the conditions in the stack show that rotating currents (swirls) are or may be present in the waste gas, it is necessary to determine the non-axial flow at each measuring point. If one of the tangential flow angles is greater than 15° with respect to the axial direction, the rotating currents will have a significant impact on the measurements. In such cases, it is necessary to measure the velocity at each point with devices that can give both the velocity and the flow angle at each point (3D, 2D and S-type pitot tubes). If the swirl is > 15°, then the velocity, corrected for direction of flow, vC, is equal to:

VC= cosmeasurement . vmeasurement where

COSmeasurement: cosine of the measured angle

Vmeasurement: measured velocity at angle θmeasurement

## POINTS FOR ATTENTION

* 1. **POINTS FOR ATTENTION IN VELOCITY MEASUREMENT**
* The pitot tube condition is critical for proper velocity measurement results. Regularly check for the straightness of the probe and tip, the right angle between tip and lance and any damage to the openings, and repair as needed.
* The pitot tube must be properly positioned straight in the opening to avoid pressure errors and to ensure that the tip is at exactly the intended measuring point. Preferably, use a permanent mounting with a flange so the pitot tube is secure and perpendicular to the two axes of the line. Regularly check the position in both planes.
* The measuring points near the wall are often less accurate due to the influence of the wall and measuring opening. ~~No closer than~~ ~~20 mm~~ At a distance of more than 3% of the length of the sampling line or more than 5 cm from the inner wall of the duct (whichever is greater), take the measurement and seal the measuring opening without parts protruding into the stack.
* The calibration factor of an S-type pitot tube is vital to measurement accuracy.
* Conduct temperature measurement at each measuring point. Allow sufficient time to reach the equilibrium temperature. Be mindful of the influence of outside air at the first measuring point (such as by taking the closest measuring point last).
* Variations over time in gas velocity, water content and gas composition may cause inaccurate results. Determine these parameters as well as the velocity multiple times under fluctuating conditions. A continuous measurement with a pitot tube at a single point provides a picture of the velocity fluctuations.
* Measures for unfavourable measurement cross-sections: if the position of the measurement section does not meet the standard requirements, it is possible to improve the accuracy by taking the speed measurement at more measuring points.
	1. **POINTS FOR ATTENTION IN FLOW RATE DETERMINATION**

In practice, waste gas flow rate measurements sometimes exhibit strong deviations between different measurements or between the measurement and the expected value.

* A pitot tube leak test must be conducted in the laboratory twice a year:
	+ Seal the pitot tube openings and one opening of the pressure gauge.
	+ Bring the pitot tube to a pressure of at least the absolute pressure (patm + pstat) in the flue – this is possible by using a tap, for instance – and close the tap immediately.
	+ The pressure must not fall by more than 5% over a period of 1 minute.
* It is always advisable to conduct a plausibility check on the measured waste gas flow rate. This can be done as follows:
	+ Check the result against previous measurements and/or information from the operator (e.g. rated fan flow rate).
	+ Compare it to the calculated or rated flow rate, e.g. for combustion processes, where the waste gas flow rate may be calculated from the measured oxygen content and fuel consumption.
	+ Check the comparability of measured incoming and outgoing flows of the process.
	+ Add the measured total and individual flow rates together and compare them.
* An increase in the number of points applies an inversely proportional reduction to the measurement uncertainty of the average velocity, and thus also of the flow rate. In cases of doubt, it is always advisable to increase the number of measuring points.
* The measurement accuracy for the duct dimensions has a major impact upon the accuracy of the calculated flow rate (the area is proportional to the square or product of the linear dimensions). The maximum permissible deviation for the stack diameter is 2%.
* In the case of a strong rotating flow (swirl), it is possible to determine the flow angle as follows:
	+ Level and zero the pressure gauge.
	+ Connect to the S-type pitot tube and perform a leak test.
	+ Place the S-type pitot tube at each measuring point, one after the other, so the planes of the pitot tube openings are perpendicular to the measurement cross-section (this position is the 0° reference for the S-type pitot tube).
	+ Record the differential pressure at each point. If a differential pressure 0 is obtained at this 0° position, this means an acceptable flow rate condition exists at that point. If the differential pressure is not 0, rotate the pitot tube until the pressure gauge reading is 0. Then record the angle of rotation of the pitot tube.
	+ If the angle of rotation at each point is less than 15°, the flow rate condition in the gas duct is acceptable. If the angle with respect to the duct axis is not less than 15°, this constitutes a deviation from NBN EN 15259; for flow rate, however, a measurement that requires correction is in fact considered a compliant measurement for determining the mass flow rate.

If the direction of flow is not parallel to the axis of the line, the device may measure velocities that are higher than what corresponds to the actual axial flow. Section 8 describes a correction for this.

## MEASUREMENT UNCERTAINTY

In practice, flow rate determinations often exhibit wide deviations between different laboratories and between expected and measured values. In cases of systematic errors (bias), this is often attributable to non-conformity with conditions set out for the scope in Section 1, or to deviations in the measuring equipment or in the locations of the measuring points. The evaluation below assumes strict compliance with the standards, and does not take any major systematic deviations into account.

It is possible to determine the measurement uncertainty of the velocity with a pitot tube in three steps:

1. Uncertainty of the velocity measurement at a single point
2. Uncertainty of the average velocity
3. Uncertainty of the total flow rate, under actual and standard conditions

The GUM approach is used to evaluate the different uncertainty contributions to velocity and flow rate measurements. This method allows calculation of the uncertainty as a function of the gas velocity, and examination of the impact of the number of measuring points and the duct conditions.

The variables that contribute to measurement uncertainty according to the flow rate calculation are:

* Pitot factor K, uncertainty according to certificate or in-house calibration
* Δp
* ρ, determined in-house from gas composition, water content, temperature and pressure
* Stack dimensions or diameter D
* Temperature and pressure in the stack

Other factors not appearing in the formulas are:

* Variations as a function of time
* Variations in gas composition and water content
* Deviating pitot tube positions
* Repeatability

#### Uncertainty of local velocity measurement

With the ‘propagation of uncertainty’ rule for the combined measurement uncertainty according to the GUM:

2 *n*  *f* 2 2

*uc* *y*  

*x*

*i* 1

 *u* *xi* 

*i* 

(13)

Equation (3) gives the following expression for the measurement uncertainty in the velocity measurement at a single point based on the individual measurement uncertainties:

*u*2(*v*)

 *c*



*v*2

*u*2(*K*)

*K*2

* *u*2(Δ*p*)

4.Δ*p*2

 *u*2(*ρ*)

4. *ρ*2

(14)

#### Uncertainty of the average velocity

(8) gives the average velocity of the n measured points, making the measurement uncertainty of the average equal to:

*u*2 (*v* )  1  *u*2 (*v j* )

(15)

*n*2 *j*

Please note here that the standard uncertainty at the average gas velocity decreases as the number of measuring points increases.

#### Uncertainty of the volume flow rate

Calculate the volume flow rate from the average velocity by multiplying it by the area of the duct cross-section according to equation (9), so the measurement uncertainty of the average is equal to:

*u2 (Q)* 

*Q2*

*u2 (v) v 2*

 *u2 (A) A2*

(16)

For circular cross-sections with diameter D:

*u2 (A) A2*

 *4* 

*u2 (D)*

*D2*

Further evaluation of the measurement uncertainty of QN and QN,dr using formulas (10) and (11) is obvious and is not further discussed here.

## REFERENCES

NBN EN ISO 16911-1: 2013

Stationary source emissions - Manual and automatic determination of velocity and volume flow rate in ducts - Part 1: Manual reference method

NBN EN 15259: 2007

Air quality - Measurement of stationary source emissions - Limits for measurement sections and sites and for the measurement objective, plan and report

ISO 10780: 1994

Stationary source emissions - Measurement of velocity and volume flow rate of gas streams in ducts

ISO 14164: 1999

Stationary source emissions - Determination of the volume flow rate of gas streams in ducts - Automated method

EPA Method 1

Sample and velocity Traverses for stationary sources

EPA Method 2

Determination of stack gas velocity and volumetric flow rate (type S pitot tube)

ISO 3966: 2008

Measurement of fluid flow in closed conduits - Velocity area method using pitot static tubes

EN 13284-1: 2017

Stationary source emissions - Determination of low range mass concentration of dust - Part 1: Manual gravimetric method

Environment Agency

Method Implementation Document for EN 13284 BS EN 13284-1: 2002

Stationary source emissions - Determination of low range mass concentration of dust - Part 1: Manual gravimetric method

Compendium for air sampling, measurement and analysis

# Essential quality requirements for emission measurements

September 2021 version LUC/0/005

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1. **SCOPE**

These quality requirements apply to measurements of emissions into the air taken by recognised laboratories in Flanders under VLAREM and the environmental permit.

The summary, as a compendium method, is intended to group the main requirements together into a single, unambiguous reference document. Most of these requirements have already been communicated through messages, working groups and meetings of the recognised laboratories. Their application is a precondition for recognition and subject to verification at any time in recognition or field audits and in the measurement reports.

Some quality requirements already appear in other compendium procedures or Belgian, European or ISO standards. In this case, their repetition on this list means that deviation from the requirement is not permitted due to the impact on the quality of the results. Other quality requirements are stricter or more explicit than the standards. These correspond to the level of quality defined in Flemish law as ‘as accurate as possible’. At certain points, the text describes permissible deviations from the standards.

To ensure unambiguous references, the quality requirements are divided up as follows:

1. Measurement site
2. Flow rate measurement
3. Sampling
4. Method-specific requirements
	* Gas measurements with continuous monitors
	* Oxygen measurement
	* NO2 measurement
	* Gravimetric determination of dust content
	* Gravimetric determination of water content
	* Wet chemical sampling of inorganic gaseous chlorides expressed as HCl, gaseous fluorides expressed as HF, etc.
	* VOCs - organic components

## MEASUREMENT SITE

* 1. **MEASUREMENT SITE SUITABILITY**

Verify the suitability of the measurement site using compendium procedure LUC/0/001 ‘Measurement site in the gas duct’.

The measuring plane is located in a part of the flue (e.g. stack) where homogeneous flow conditions and concentrations may be assumed.

To verify measurement site suitability, every measuring point of the measuring plane must meet the following requirements of NBN EN 15259 for a homogeneous flow profile:

* + - Direction of the gas flow < 15° with respect to the longitudinal axis of the gas duct
		- No local negative gas velocities
		- A minimum gas velocity depending on the measurement method for volume flow rate:
			* When using pitot tubes, the differential pressure must be greater than 5 Pa.
			* When using vane anemometers, the velocity must be greater than 0.5 m/s.
		- Ratio of maximum/minimum measured gas velocity < 3

The four conditions mainly pertain to a homogeneous velocity profile and are therefore vital for the sampling of dust, dust parameters and flow rate.

For installations where flow rate and dust/dust parameters are not essential measurands, the homogeneity mainly matters for the gaseous parameters, not the velocity profile. In this case, the result of the homogeneity test for the gaseous parameters (presumed homogeneous, homogeneous or not homogeneous) determines the measurement strategy for the gaseous parameters (see [2.3](#_bookmark33)).

It is necessary to conduct a preliminary inspection of the installation and measuring plane on the following points:

* Access to the measurement location
* Number and size of the measuring openings
* Site conditions
* Protection from the elements
* Any obstructions
* Power supplies

This preliminary inspection enables selection of the appropriate sampling equipment.

* 1. **MEASUREMENT SITE EVALUATION IN THE REPORTING**
		1. General

Procedure LUC/0/006 applies to the reporting of sampling data and analysis findings by a recognised laboratory in the category of air. According to this procedure, the report must include a record of the characteristics of the measurement site and, where applicable, verification that these comply with the compendium or another required method as per Article 45 of VLAREL. For emission measurements, in addition to the

measurement site characteristics, the report must include at least the following information on the conformity of the measurement site:

* The results of the checks for the four conditions in NBN EN 15259 for a homogeneous flow profile at every point in accordance with the standard, except for situations where deviations are permitted under [2.2.2](#_bookmark32); in the case of a heterogeneous concentration profile in the measuring plane, flow rate-weighted sampling is required and the velocity profile must be known. In the latter case, always verify compliance with the conditions of NBN EN 15259. The report must further detail the checks for the four conditions by:
	+ for measured angles greater than 15°: reporting of the actual measured angles of the gas flow with respect to the duct axis for each measuring point; for angles less than 15°: ‘< 15°’ will suffice;
	+ reporting the individual gas velocities for each measuring point and the vmax/vmin ratio.
* The number of measuring openings/axes/points present with respect to the requirements of NBN EN 15259: [2.2.2](#_bookmark32) describes the permissible deviations that do not result in a lack of compliance.

With the exception of the deviations permitted under [2.2.2](#_bookmark32), report any failure to meet the four requirements for a homogeneous flow profile from NBN EN 15259 and any lack of sufficient measuring openings/axes/points as a non-conformity, with description of the deviations observed. For instance, in the event of a failure to meet the required number of measuring openings/axes/points: not all required measuring openings are present, a railing at the measuring openings prevents full traversing at all points, measuring openings are too small for the probes used, in particular for dust-borne components, measuring platform is too small, etc.

The measurement report must also give the following information on the measurement site, but this does not result in a non-compliant measurement site:

* The result of the homogeneity test with resulting strategy (single-point measurement or grid measurement) or compliance with the three conditions for presumed homogeneity (with a single-point measurement as the resulting strategy).

 ~~~~ Verification of the distance guidelines in NBN EN 15259 in the context of homogeneous flow conditions.

Record all data on the measurement site characteristics and the verification of their conformity with the compendium (e.g. homogeneity determination).

* + 1. Permissible deviations from standard NBN EN 15259

The section below details the situations where the LUC permits deviations from the four conditions of NBN EN 15259 for a homogeneous flow profile or from the number of measuring openings/axes/points, which therefore do not result in a lack of compliance. The report must state which of these deviations from the standard apply, with the indication that these are not deviations from the ‘Compendium for air sampling, measurement and analysis’ and the operation therefore complies with the LUC.

* For small gas-fired or gas oil-fired installations up to 5 MW, with the exception of atmospheric burners, the following deviations from NBN EN 15259 are permitted:
	+ - 1. Only one measuring opening available instead of two
			2. A measurement at a single point
			3. Deviations from one or more of the four conditions for a homogeneous flow profile
* If only measuring gaseous parameters without a flow rate in a homogeneous gas flow without droplets, it is not necessary to check the four conditions for a homogeneous velocity profile from NBN EN 15259, and failure to meet one or more of these conditions does not constitute a deviation. For non-water-soluble gaseous parameters, this also applies to homogeneous gas flows with droplets.
* If only measuring gaseous parameters and flow rate in a homogeneous gas flow without droplets, and an alternative method as per NBN EN ISO 16911-1 is available for determining the flow rate, it is not necessary to check the four conditions for a homogeneous velocity profile, and failure to meet one or more of these conditions does not constitute a deviation. For non-water-soluble gaseous parameters, this also applies to homogeneous gas flows with droplets.
* If only measuring gaseous parameters without a flow rate in a gas flow assumed to be homogeneous and without droplets, failure to meet the required number of measuring openings/axes/points does not constitute a deviation. For non-water-soluble gaseous parameters, this also applies to presumed homogeneous gas flows with droplets.
* If only measuring gaseous parameters and flow rate in a gas flow assumed to be homogeneous and without droplets, and an alternative method as per NBN EN ISO 16911-1 is available for determining the flow rate, failure to meet the required number of measuring openings/axes/points does not constitute a deviation. For non-water-soluble gaseous parameters, this also applies to presumed homogeneous gas flows with droplets.
	1. **DEMONSTRATION OF MEASUREMENT SECTION HOMOGENEITY FOR THE GASEOUS PARAMETERS**

The homogeneity of the measurement section for the gaseous parameters is relevant in determining the measurement strategy for these parameters.

Measurement of gaseous components at a single point, instead of measurement over the entire measuring plane (traversing, scanning), is only permitted:

* + - if the homogeneity of the measurement section was demonstrated as per NBN EN 15259. In this case, a single-point measurement at each point of the section is permitted; or
		- in a single well-defined point demonstrated to be representative of the section as per NBN EN 15259;

 ~~~~ in the case of presumed homogeneity;

* + - for small gas-fired or gas oil-fired installations up to 5 MW, with the exception of atmospheric burners.

At diameters of less than 0.35 m, it is only required to measure at a single point in the gas duct, not to demonstrate homogeneity. Between 0.35 m and 1.10 m, it is required to demonstrate homogeneity unless the conditions below for presumed homogeneity are met.

Presumed homogeneity: A measurement section may be considered homogeneous without further testing if all of the following conditions are met:

 ~~~~ The measurement section is located in a straight part of the flue with at least four hydraulic diameters upstream and two hydraulic diameters downstream (previous rules from NBN T 95-001\*);

* + - with a diameter of up to 1.10 m (circular flue) or with a cross-section

≤ 1 m2 (rectangular or square flue);

* + - and only one source is connected.

\* Standard NBN T 95-001 stated: ‘The measurement cross-section must be in a straight part of the line with constant shape and cross-section and a length of at least six times the hydraulic diameter Dh; it must be selected at two thirds of the length of the straight part measured from the starting point upstream.’

Suspected heterogeneity: If all the above conditions are not met, i.e. in cases of a less favourable section than prescribed by the standards for dust content determination or flow rate measurement, larger diameters (> 1.10 m) or multiple sources connected to the same duct, a suspicion of heterogeneity exists and homogeneity must be demonstrated.

Frequency of homogeneity demonstration: If performing a single-point measurement, homogeneity must already be demonstrated. For emission measurements repeated periodically in the same measuring plane, a one-time determination of homogeneity using the method in NBN EN 15259 will suffice, as long as the measuring point and the connected emitting processes do not change.

Minimum number of measuring points: Homogeneity must be demonstrated with a minimum of eight different measuring points for a round cross-section and a minimum of nine for a rectangular one. Given the excessive statistical uncertainty with lower numbers of measuring points – reflected in the high F and t values – tests with fewer measuring points are not very meaningful.

Permanently installed measuring devices: For permanently installed measuring devices, homogeneity must always be demonstrated as per the requirements of the Code of Good Practice or EN 14181.

Warning: For emissions that fluctuate widely over time, or multiple sources with different regimes connected to the same duct, it is not possible to demonstrate homogeneity with the prescribed methods. In principle, this means that a scan is always required, but this still does not guarantee that the measurement is representative of the emissions.

In combustion processes, it is possible to demonstrate homogeneity with O2 (as a surrogate parameter).

For non-combustion processes where O2 is not a suitable parameter for the homogeneity test and the gas flow does not offer any other continuously measurable parameters for the homogeneity test, the following conditions must be verified:

* Standard deviation of velocities is < 10 % of the average velocity.
* Local temperature differences vary by less than 10°C.

## FLOW RATE MEASUREMENT

Compendium procedure LUC/0/004 applies to measurement of the flow rate in a flue. This method follows NBN EN ISO 16911-1 unless the relevant method sets out other provisions.

* 1. **PITOT TUBE CALIBRATION**

Always calibrate all pitot tubes before commissioning. Calibrate all pitot tubes used in the field regularly, at intervals based on frequency of use (1 to 5 years). In addition to calibration of pitot tubes as per NBN EN ISO 16911-1 (e.g. in a wind tunnel to a laser Doppler anemometer as a reference instrument), calibration to a reference pitot tube in a wind tunnel as per ISO 10780 is also permitted. The latter method is further described in LUC/0/004. It is not permitted to use pitot calibration factors based solely on the design of the pitot tube.

It is preferable for pitot tubes to be calibrated by calibration bodies that are accredited for this. The conditions for calibration in an in-house lab are as follows:

* + - Have a detailed calibration procedure in place.
		- Have a wind tunnel that meets the standards (see procedure LUC/0/004).
		- Evaluate the calibration uncertainty of each calibration.

Always calibrate or verify the pitot tubes of combined probes in a wind tunnel. Only use combined probes (pitot tube+thermocouple connected to a sample probe for dust/inorganic chlorides, etc.) to measure the gas velocity to set the isokinetic sampling rate. It is not permitted to use flow rates measured with these combined probes to test emission limits.

* 1. **PITOT TUBE INTEGRITY**

A pitot tube is an aerodynamic measuring instrument whose proper response depends having the correct shape. It is not possible to ensure exact positioning at the required measuring points if the lance is not straight. Check for the correct geometric shape, tip integrity and lance straightness with each measurement and always assess these during audits.

It is not permitted to calibrate curved or deformed pitot tubes. Before each calibration, repair any deformation or damage.

## SAMPLING

* 1. **GRID OR SINGLE-POINT MEASUREMENT?**
		1. **FLOW RATE**

Flow rate measurement always requires a grid measurement. Procedure LUC/0/004 and standard NBN EN 15259 indicate the number and position of the measuring points.

* + 1. **DUST-BORNE COMPONENTS OR MULTIPHASE SAMPLING**

Sampling where the component(s) to be measured occur in the dust particles, or may occur in different phases such as dust, gas and droplets, always requires a grid measurement across the points and diameters specified in dust sampling method EN 13284-1 and isokinetic sampling.

Thus, this always applies to the sampling of dust, heavy metals, PAHs, dioxins and PCBs in

emissions.

* + 1. **GASEOUS COMPONENTS**

For gaseous components, a single-point measurement is permitted under any of the following conditions:

* The homogeneity of the entire measurement section was demonstrated as per NBN EN 15259. In this case, each point may be selected as a single measuring point.
* The selected measuring point was identified as a representative point for the emissions as per the methodology in NBN EN 15259 (also possible in a heterogeneous measuring plane).
* In cases of presumed homogeneity (see point [2.3](#_bookmark33)).
* For small gas-fired or gas oil-fired installations up to 5 MW, with the exception of atmospheric burners

For stack diameters of over 1.10 metres, always support the presumption of homogeneity with an experimental determination with a sufficient number of points.

If droplets are present, sample the water-soluble gaseous components (e.g. inorganic gaseous chlorides expressed as HCl, gaseous fluorides expressed as HF, NH3, water-soluble VOCs, etc.) using a grid measurement and isokinetic sampling.

If sampling gaseous parameters with a grid measurement, in the case of a heterogeneous measurement section and the absence of droplets in the flue, the following two cases can be distinguished:

* + - 1. The flow rate through the collection medium is adjusted to the local mass flow rate in the sub-area (mass flow rate-proportional sampling).
			2. The flow rate cannot be adjusted without reducing the collection efficiency of the system (manual methods, such as with wash bottles) or cannot be adjusted at all (automatic methods). This case involves time-proportional sampling at the local velocity at every point.
	1. **LEAK TESTS**
* The procedure must qualitatively describe how to perform the leak test. The duration of a leak test may be a crucial factor.
* Record the leak tests quantitatively (also recording times, gas meter readings, residual leakage rate!).
* Perform the leak test at least at the highest negative pressure that can occur during the test.
* The general criterion for the maximum permitted leakage rate in the standards is 2% of the nominal sampling flow rate. For PCDDs/PCDFs/PCBs/PAHs and VOCs by adsorption, the leakage rate must not exceed 5% of the sampling flow rate.
* The contribution factored into the measurement uncertainty must correspond to the leakage actually permitted (leaks and losses are separate items, even though various EN standards give a criterion of 2% for the sum).

Tips for different leak tests:

* + Qualitative or semi-quantitative leak tests (e.g. maximum of 10 air bubbles per minute, or rotameter float at bottom) are permitted provided that the operator can demonstrate the corresponding quantitative leakage rate, which must certainly remain below 2% of the sample flow rate (exception: for dioxins, PCBs and PAHs and VOCs by adsorption, a maximum of 5% is permitted).
	+ It is advisable to have stoppers for every type of probe used. For wet chemical sampling, these are preferably stoppers that can be released gradually so the liquid does not splash out of the wash bottles when removing the stopper.
	1. **FIELD BLANKS**

Table 1 shows the special requirements on field blanks. Because the absence of a field blank may result in challenges to the validity of a measurement, exceptions to this table are not permitted. Always take and analyse a field blank and include it in the report.

* + 1. **FIELD BLANKS FOR MANUAL SAMPLING**

*Table 1. Requirements on field blanks*

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Standard** | **Frequency** | **Procedure(**2) | **Validation****criterion?** | **Report** |
| EN 1948-1dioxins | See [4.3.2](#_bookmark45). | As with samplingAmbient air through the entire sampling train for at least 2 minutes | Yes, 10 % of emission limit | Yes, value of the field blank(s) |
| EN 13284-1dust, manual | Before or after each series(1)at least one/day | As with samplingAmbient air through the entire sampling train for at least 2 minutes | Yes, 10 % of the lowest emission limit for the process or 0.5 mg/Nm3 (whichever isgreater) | Yes, the value of thefield blank(s) |
| EN 1911Inorganic gaseous chlorides, expressed asHCl | For each series(1)at least one/day | As with samplingAmbient air through the entire sampling train for at least 2 minutes | Yes, 10 % of the emission limit | Yes, the value of thefield blank(s) |
| CEN/TS 13649VOCs by adsorption: | At least one/day For > 6 samples at least two/day(3)For > 10 samples, at least three/day(3)Same batch as actual samples | As with samplingAmbient air through the entire sampling train for at least 2 minutes | 10 % of emission limit | Yes, the value of thefield blank(s) |
| EN 13211Hg in waste gas | One/campaign + each stack | As with samplingAmbient air through the entire sampling train for at least 2 minutes | No | Yes, value of the field blank(s) |
| EN 14385metals | One/campaign + each stack | As with samplingAmbient air through the entire sampling train for at least2 minutes | Yes, 10 % of emission limit | Yes, value of the field blank(s) |

(1) The definition of ‘measurement series’ is taken from the standard for inorganic gaseous chlorides, EN 1911:2010, i.e. serial measurements taken in the same stack under the same process conditions. For different measurement series, it will suffice to take a single field blank if the measurements are performed with material prepared at the same time and with the same procedure and used in the same industrial process or in different lines of that industrial process.

(2) In the case of increased concentrations of the component to be measured in the environment, take the field blank according to the leak test procedure. In this case, also conduct a leak test on the sampling train itself.

(3) At least one field blank per day per measurement method (pattern+desorption combination); it may

be possible to fill in the other blanks by analysing the backup section of actual samples.

* + 1. **FIELD BLANK FOR DIOXINS AS PER EN 1948-1, 2, 3**

Due to the sampling complexity and high analysis costs for dioxin emissions, all recognised laboratories must meet the requirements below in a strictly identical manner.

Take a blank:

* + - * for each campaign, where a campaign may not be regarded as lasting more than 3 consecutive measurement days;
			* at the measurement site itself (i.e. not at the bottom of the stack if the actual sampling takes place at heights);
			* using the same equipment as for sampling, including all components with recovery of the same fractions as with the sample.
			* Always analyse this blank.
			* Always indicate the value of the field blank in the report.

The same requirements apply to field blanks for PCBs and PAHs.

## METHOD-SPECIFIC REQUIREMENTS

* 1. **GAS MEASUREMENTS WITH CONTINUOUS MONITORS**
		1. **GAS MONITOR CALIBRATION AND VERIFICATION**
			1. *ONSITE CALIBRATION AND LOGGING*
* Before measuring on the measurement site, calibrate all gas monitors with zero and span gases and check them with a control gas.
* After the measurement, check them again with zero and span gases onsite.
* The electronic data files must provide an audit trail for all calibrations (zero, span and control gases).
	+ - 1. *CALIBRATION GAS QUALITY*

The span gas must be a certified calibration gas, either:

* + - * + a gas with calibration certificate issued under an ISO 17025 accreditation (BELAC or equivalent) by a manufacturer accredited as a calibration body; or
				+ a calibration gas traceable to ISO 17025; here, the analysis by an accredited or recognised laboratory applies as the correct value. The latter case must meet the requirements further detailed in [5.1.4](#_bookmark51) for self-certification of calibration gases; or
				+ an operator conducting self-auditing measurements and whose methods have been approved as meeting ISO 17025 may certify calibration gases for in-house use under the same conditions as a recognised laboratory.

Oxygen calibration gases

* + - * + For O2, dried outside air is a good choice for a calibration gas. The oxygen content in dry air is 20.95%. When calibrating the oxygen monitor with outside air, either fully dry the gas (e.g. with a silica gel cartridge) or cool it with the flue gas cooler.
				+ European standard EN 14789 proposes using 20.9% with a relative uncertainty of 0.5% (= 0.1% O2 absolute). Recognition in Flanders is subject to stringent requirements on the accuracy of the oxygen measurement, and it is advisable to use the ‘more correct’ value of 20.95%. In this case, the uncertainty (< 0.05% relative) is negligible compared to other errors.

NB: At concentrations of over 15% O2, the criterion in EN 14789 for the uncertainty of O2 span gas of 2% relative (k=2) is too high to meet the criterion for recognition (deviation  0.3% absolute).

The permitted calibration uncertainty of the bottle for CO, CO2, NOx, SO2 and propane may not exceed 2%. This is in accordance with the EN standards, with expansion to SO2 and CO2.

The certified value of calibration gases must be based on or verified by means of an accurate verification analysis.

Calibration of the portable flame ionisation or photoionisation detector for the LDAR measurements does not require an ISO 17025 certificate for the calibration gas as with main emission measurements.

* + - 1. *CONTROL GAS*

After the calibration of the gas measuring devices, supply an independent control gas for all components. It is not necessary for the control gas to be from an accredited supplier or ISO 17025-certified.

Independent: the control gas must not be a diluted calibration gas.

The calibration and control gases preferably come from different gas suppliers. If they do not, the gas supplier must be able to demonstrate the independence of these gases, or the calibration gas must be certified by the laboratory with respect to a gas from another supplier. This requirement must be met by no later than 1 January 2018.

It is recommended to stagger purchases of calibration and control gases over time, in a controlled manner.

Control gas concentration: preferably around 50 % of measuring range

* + - 1. *CONTROL CHARTS FOR WASTE GAS MEASUREMENTS WITH MONITORS*

Make the control charts available onsite and fill them in immediately before starting the actual measurement.

It is not permitted to fill in the control chart afterwards, or for a quality manager who is not present on the measurement site to fill it out at another location, as this may result in propagation of measurements of uncontrolled quality.

The person taking the measurements must fully understand the control chart system and criteria, to enable proper and immediate action in the event of deviations.

Periodic processing of control charts is required unless, for instance, they feature less than 10 points over half a year.

When using set limits on control charts:

* + - * + The limits must be quantitatively justified.
				+ They must also undergo a periodic review (at least annually).
		1. **CHECK FOR LINE LOSSES**

Losses in the sampling system (intake line, gas conditioning, etc.) must be validated.

Check for these losses with each measurement as follows:

* + - * + Supply calibration gas directly to the monitors (as per EN standards!).
				+ Supply control gas through the entire sampling system.

Factor any sampling system losses into the total measurement uncertainty.

* + 1. **CRITERIA FOR ZERO AND SPAN DRIFT**
* Both before and after measurement on the measurement site, supply zero and calibration gases for the zero and span drift check.
* Zero and span drift criteria for measurement with monitors as per EN standards, which also apply to measurement with electrochemical devices:

|  |  |
| --- | --- |
|  | **Permitted deviation, as a % of concentration of the****calibration gas** |
|  | **Zero** | **Span** |
| **No correction needed** | ≤ 2% | ≤ 2% |
| **Correction of measurement values** | 2 to 5% | 2 to 5% |
| **Rejection of the measurement** | > 5% | > 5% |

* Devices + data acquisition system must permit negative values (exception for electrochemical devices).

For oxygen, the criteria in Section 8.4.3 of EN 14789 (identical to the above table) are far too broad. Instead, the following criteria apply:

|  |  |
| --- | --- |
|  | **maximum drift, % O2** |
|  | **Zero** | **Span** |
| **No correction** | ≤ 0.2% | ≤ 0.2% |
| **Correct** | 0.2 to 0.3 % | 0.2 to 0.3 % |
| **Reject** | > 0.3% | > 0.3% |

* + 1. **SELF-CERTIFICATION OF CALIBRATION GASES**

A recognised laboratory may calibrate its own calibration gases to another certified calibration gas or to primary variables of length, time and mass.

Conditions for onsite use of gases that the laboratory has self-certified to BELAC gases as per ISO 17025:

1. A gas calibration procedure must be available.
2. The calibration uncertainty of the bottle being certified must be determined. The procedure must also specify the method for determining this.
3. Permissible calibration uncertainty: maximum of 2%.

The calibration procedure must meet at least the following requirements:

* + Conduct at least two independent comparative measurements of the non-certified gas against the certified calibration gas or the primary standard.
	+ The average value of at least two measurements will be the certified value.
	+ The limitation to two comparative measurements only is permitted if the reproducibility of this measurement is sufficiently established in advance (with a minimum of six measurements).
	+ Factor this reproducibility into the measurement uncertainty of the average value, indicated on the certificate.
	+ Between these two measurements, take a zero and span measurement.
	+ Readjust the span and zero each time, or factor them into the final result.
		1. **VALIDATION REQUIREMENTS ON MONITORS**
			1. *VALIDATION PARAMETERS*

In general, a measurement method under the Flemish Environmental Permitting Regulation [VLAREM] should be usable between 0.1 times and 3 times the emission limit. For standard methods, validate at least the following parameters:

* + - * + (Intra-)reproducibility, such as from control charts
				+ Correctness, e.g. from ring test data
				+ Working range
				+ Limits of detection and quantification
				+ Measurement uncertainty

The European standards for continuous measurement of O2, NOx and CO at large combustion and waste incineration plants require the inclusion of several additional factors in the measurement uncertainty. Procedure LUC/II/001 (Sampling for waste gases and analysis of CO, CO2, SO2, NOx, O2 and TOC with monitors) lists all factors to be included in the measurement uncertainty. Determine these factors by validation, unless reliable data are available from another source. Factor these into the measurement uncertainty for all measurements with monitors, except for the electrochemical devices.

* + - 1. *MINIMUM REQUIREMENTS FOR EVALUATION OF MONITOR LINEARITY:*
				* Establish criterion for the correlation coefficient (e.g. R > 0.995).
				* Visually plot the calibration curve to show deviations from it (‘residuals’).
				* Minimum number of points: five + zero (after elimination of points, at least five points must remain).
				* Do not force calibration curve through the origin.
				* It will suffice to measure each concentration once.
				* Linearity check frequency: see [5.1.5.3](#_bookmark53).
			2. *VALIDATION FREQUENCY*

EN standards require an annual linearity test. For those participating in the LABs ring tests for waste gases, the Flemish Institute for Technological Research [VITO] permits a lower frequency of once every 2 years. All others are covered by the ring test. Devices not used in the ring test do however require an annual linearity test, except for the FIDs. For the latter, once every 2 years will suffice.

Revalidation is required in case of:

* + - * + An essential adaptation of the method
				+ A change of device type
				+ A statutory change to the measurement range

Validation by instrument type is permitted: an intake inspection will suffice when purchasing a device of a similar type (to be documented in a procedure, e.g. linearity check). Determine the limits of detection and quantification separately for each device.

* + 1. **EVALUATION OF MEASUREMENT UNCERTAINTY**

All recognised laboratories must have an evaluation of measurement uncertainty in place for measurements with monitors. If the applicable standards give a criterion for measurement

uncertainty, it must be met. For the oxygen measurement, the measurement uncertainty must fall within the recognition criterion of 0.3% absolute.

The measurement uncertainty must be determined around the emission limit, but calculation of this must be possible for each measured concentration. Some of the measurement uncertainty contributions are proportionate to the measuring range or constant (constant absolute error), while others are proportionate to the measured concentration (constant relative error). The measurement uncertainty calculation must clearly distinguish these terms.

NB: The requirements in the standards apply. The non-standard examples that these standards provide for illustration purposes may omit certain terms. This is no reason to leave these terms out of the calculation.

Perform the measurement uncertainty calculation for measurements with monitors at large combustion plants and waste incineration plants as per European standards EN 14789 for oxygen, EN 14792 for NOx and EN 15058 for CO. Here, for each emission limit tested, the results of the measurement uncertainty calculation must meet the maximum permissible measurement uncertainty of 10 % for NOx and SO2 and 6% for CO.

For the contributions from the sensitivity factors (mains voltage, pressure, sampling flow rate, temperature), the EN standards set relatively high specifications. If the exact value is not available for older devices, it is permitted to use half of the permissible specification. When purchasing new measuring devices, ask the supplier for the sensitivity factor specifications as per the EN standard (e.g. EN 15267-3, Table 1), which are needed as contributions to measurement uncertainty. Preferably, use data determined by an independent institute.

* + 1. **MEASURING RANGE AND MEASUREMENT UNCERTAINTY**

Avoid checking emission limits below 5% of the measuring range of a measuring device. Because the number of contributions to measurement uncertainty are proportionate to the measuring range, it is not possible to check an emission limit with sufficient accuracy at the bottom of the scale.

* 1. **OXYGEN MEASUREMENT**
		+ Oxygen is subject to a stringent accuracy requirement of 0.3% absolute in Flanders because the oxygen measurement is used to convert measurement values to the reference oxygen content. This strict requirement is justified based on the error propagation rules applied to the conversion formula for reference oxygen content. The requirement is most critical at high oxygen levels.
		+ When calibrating the oxygen monitor with outside air, either fully dry the gas or cool it with the flue gas cooler. If the residual water content in the outside air after the cooler is the same during oxygen monitor calibration and waste gas measurement, it is not necessary to factor this in. In this case, it is also permitted to use an oxygen concentration of 20.95 % to calibrate the monitor (such as for calibration with completely dry air). However, if the dew point of the outside air is lower than the temperature of the cooler, factor the water content difference after the cooler into calibration and measurement.
	2. **NO2 MEASUREMENT**
		1. **NO2 MEASUREMENT BY CONVERTER AND NO MEASUREMENT:**
		+ NO2/NO converter efficiency:
			- Minimum of 95% as per EN 14792 (NOx measurement with the chemiluminescence monitor)
			- Criteria currently applicable as good practice:

Converter efficiency

No correction ≥ 95%

Correction 80 to 95%

Reject results < 80 %

* + - Converter efficiency: to be tested at the highest NO2 concentrations that occur in practice. As a guideline for combustion process measurements, an NO2 concentration of ±10 % of the highest NOx measuring range or the emission limit will suffice for the test.
		- Some converters appear to be less efficient at high NO2 concentrations. When using a converter to measure processes with higher NO2 concentrations, the efficiency must be demonstrated at the relevant NO2 concentration.
		1. **WIDER MARGINS FOR NO2 DRIFT**

In cases of very small NO2 contributions (up to the total NOx), greater zero and span drifts may be permitted for NO2 if the lab can demonstrate that this does not significantly increase the measurement uncertainty of NOx, and that the measurement uncertainty remains below 10 % for measurements as per EN standards.

* 1. **GRAVIMETRIC DETERMINATION OF DUST CONTENT**
		+ This must meet the requirements of standard EN 13284-1. These requirements prevail over those of the other standards (ISO 9096, for instance), such as with regard to checks on the isokinetic sampling rate and material specifications, e.g. the minimum nozzle diameter of 6 mm.
		+ Check the isokinetic sampling rate at each measuring point. For this, note or log all required parameters every 5 minutes. Calculate the deviations from the isokinetic sampling rate for each point. The maximum deviation criteria (-5% to +15%) apply to each measuring point.
		+ The use of plug filters made from glass wool or quartz wool is not permitted because they cannot demonstrate the required efficiency of 99.5% for 0.3µm particles (99.5% for 0.6µm particles).
		+ EN 13284-1 no longer permits the use of zero-pressure probes (equilibrated probes).
		+ Permissible relative expanded uncertainty of the gas volume meter: see 5.6.
		+ The laboratory must always be able to condition the filter at 160 °C as per EN 13284-1, even if other temperatures are used in practice.
		+ It must be possible, on request, to set the filter conditioning to a temperature other than 160 °C (for dust types that exhibit specific thermal behaviour).
	2. **GRAVIMETRIC DETERMINATION OF WATER CONTENT**

This must meet the requirements of standard EN 14790. These requirements prevail over others.

* + - Determine the water content at least as a half-hour average.
		- Determine the water content at least by adsorption and weighing. Condensation alone will not suffice.
		- A scale must be available in the vicinity of the measurement site if the water content is needed to conduct isokinetic sampling. Otherwise, weighing after the measurements is also permitted.
		- The scale must feature a check weight and at least one set deviation criterion. If the value exceeds the criterion, take appropriate action.
		- Permissible relative expanded uncertainty of the gas volume meter: see 5.6.

LUC/0/003 describes a number of alternative measurement methods for water determination and their scope. It is not permitted to use these methods for critical applications, in particular for conversion of concentrations to dry gases for emission limit testing or conversion of wet volume flow rates to dry gas flow rates, if mass flow rates must be calculated, unless the water content in the gas flow is less than 2% by volume.

It is also permitted to calculate the water content based on the measured oxygen content and knowledge of the elemental composition of the fuel for installations up to 10 MW falling under VLAREM-I headings 43 and 31.1.

* 1. **WET CHEMICAL SAMPLING OF INORGANIC GASEOUS CHLORIDES AND FLUORIDES, ETC.**
* Relative expanded uncertainty of the gas volume meter:

|  |  |
| --- | --- |
| **Verification** | Maximum permissible relative expanded uncertainty – when comparing the gas meter to be checked to the referencegas meter |
| Intake inspection = initial check ona gas meter | 2.0 % of the volume under current conditions |
| For subsequent checks | 5.0 % of the volume under current conditions \* |

\* If the value exceeds the 5.0 % limit (under current conditions), a check must include retroactive corrective actions. It is recommended to correct the gas meter for the observed bias at a relative expanded measurement uncertainty of between 2.0 and 5.0 %.

The gas meter features a thermometer with a maximum relative expanded uncertainty of 2.0 % and an absolute pressure measurement with a maximum relative expanded uncertainty of 2.0 %.

* Determine the efficiency of the wash bottles/impingers at least for the selected sampling equipment and conditions (intake flow rate, time) by separate analysis of the absorption solution from the last wash bottle/impinger. The gas flow rate, liquid filling, tip shape and diameter, and distance from the bottom are critical parameters.
* Apply any absorption efficiency criteria provided in the relevant standards. If they do not provide any criteria, they are specified in the compendium method itself.
* Simultaneous sampling of inorganic gaseous chlorides and gaseous fluorides in NaOH solutions: this is for gaseous inorganic chlorides, expressed as HCl, not in accordance with EN 1911. The standards state that the inorganic gaseous chlorides must be sampled in two impingers with water and the gaseous fluorides in two impingers with 0.1 N NaOH.
* Possible deviation (based on NF X 43-304): a combination of one wash bottle with H2O and two wash bottles with 0.1 N NaOH is permissible for combined sampling of the inorganic gaseous chlorides and fluorides. The concentrations of fluoride and chloride are determined separately in each impinger. For sampling the inorganic gaseous chlorides and fluorides, demonstrate compliance with the absorption efficiency requirements. Alternatively, it is also permitted to use two wash bottles with water followed by two wash bottles with 0.1 N NaOH, with analysis of chloride only in the first two wash bottles and fluoride in all wash bottles.
* The wet chemical SO2 determination as per EN 14791 has a limited accuracy (20 %) and slow response. It is only permitted to use EN 14791 for the approval of permanently installed measuring devices if validation or ring testing has shown that they can meet the stronger accuracy requirement of 10 %.
* For measurement of SO2, a continuous measuring device must be available. By participating in the ring tests, the reference laboratory ensures the equivalence of the device used in the ring tests with EN 14791 for SO2 measurement and the approval of permanently installed measuring devices. For the latter however, the accuracy requirement of 10 % applies (recognition condition).
* Analysis:
* Always produce the calibration standards in the analysis in the same medium where the samples are measured, unless the medium can be demonstrated to have no influence. In any case, for each analysis series, always include a control standard in the medium of the samples. The result may be plotted in water on the control chart of the standard.
* Procedure WAC/VI/A/003 sets out the general quality requirements for the analysis methods.
	1. **VOC SAMPLING BY ADSORPTION**
* Mandatory logging of the total volume of gas sampled on cartridges, e.g. with gas meter
* Permissible relative expanded uncertainty of the gas volume meter: see 5.6.
* Always analyse the backup section. The maximum penetration is 5% of the total amount.
* The selected extraction solvent must be of chromatographic quality, and must be free of interferents that coeulate with the components being determined.
* The minimum recovery (desorption efficiency) is 80 %. Contrary to standard CEN/TS 13649, a desorption efficiency of up to 75% is permitted, provided that this value is repeatable. This means a spread of up to 5%. The target value is a desorption efficiency of 90 %. Apply a correction for recovery in the calculation of the result.
* Prepare the calibration solutions in the same solvent used for desorption. The concentrations of the samples to be analysed must fall within the concentration range of the calibration solutions. For calibration, use a calibration solution in at least five different concentration levels. Validation requirements:
* Determine the desorption efficiencies once by validation. In the case of a change in loading conditions or use of a different desorption solvent, conduct a new validation. If the lab follows the compendium method, it should not validate this itself.
* The stability of the components being analysed on the absorbent used should be known from validation. The corresponding maximum retention period of the samples must be known for each component and must correspond to a maximum loss of 10 %.
* Further validation requirements when using compendium methods LUC/IV/001 to LUC/IV/012:
	+ Limits of detection and reporting
	+ Accuracy
	+ Reproducibility
		- Measurement uncertainty for the combination of sampling and analysis (WAC/VI/A/002)
	+ Linearity
* Further validation requirements when using methods other than LUC/IV/001 to LUC/IV/012:
	+ The minimum requirements in procedure WAC/VI/A/001 of the Compendium for water sampling, measurement and analysis
	+ Measurement uncertainty for the combination of sampling and analysis (WAC/VI/A/002)

## REFERENCES

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NBN EN 15259: 2007

Air quality - Measurement of stationary source emissions - Limits for measurement sections and sites and for the measurement objective, plan and report

EN 13284-1: 2017

Stationary source emissions - Determination of low range mass concentration of dust - Part 1: Manual gravimetric method

EN 14792: 2017

Stationary source emissions - Determination of mass concentration of nitrogen oxides -

Standard reference method: chemiluminescence

EN 14181: 2014

Stationary source emissions - Quality assurance of automated measuring systems

ISO 10780: 1994

Stationary source emissions - Measurement of velocity and volume flowrate of gas streams in ducts

ISO/IEC 17025:2005

General requirements for the competence of testing and calibration laboratories

EN ISO/IEC 17025:2017

General requirements for the competence of testing and calibration laboratories

EPA Method 2: 2000

Determination of stack gas velocity and volumetric flow rate (type S pitot tube)

EN 1948-1: 2006

Stationary source emissions - Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs - Part 1: Sampling of PCDDs/PCDFs

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EN 13211: 2001

Air quality - Stationary source emissions - Manual method of determination of the concentration of total mercury

EN 14385: 2004

Stationary source emissions - Determination of the total emission of As, Cd, Cr, Co, Cu, Mn, Ni, Pb, Sb, TI and V

EN 14789: 2017

Stationary source emissions - Determination of volume concentration of oxygen - Standard reference method: Paramagnetism

EN 15058: 2017

Stationary source emissions - Determination of the mass concentration of carbon monoxide - Standard reference method: non-dispersive infrared spectrometry

EN 15267-3: 2007

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ISO 9096: 2017

Stationary source emissions - Manual determination of mass concentration of particulate matter

EN 14790: 2017

Stationary source emissions - Determination of the water vapour in ducts

NF X 43-304: 2007

Stationary source emissions -

measurement of the concentration of fluorinated compounds, expressed in hydrofluoric acid (HF)

EN 14791: 2017

Stationary source emissions - Determination of mass concentration of sulphur oxides - Standard reference method

NBN EN ISO 16911-1

Stationary source emissions - Manual and automatic determination of velocity and volume flow rate in ducts - Part 1: Manual reference method (ISO 16911- 1:2013)

Compendium for water sampling, measurement and analysis (WAC) Method WAC/VI/A/001

Performance characteristics <https://emis.vito.be/nl/lne-erkenningen-water>

Compendium for water sampling, measurement and analysis (WAC) Method WAC/VI/A/002

Measurement uncertainty

<https://emis.vito.be/nl/lne-erkenningen-water>

Compendium for air sampling, measurement and analysis

# Sampling for waste gases and analysis of CO, CO2, SO2, NOx, O2 and TOC with monitors

September 2021 version LUC/II/001

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3. **SCOPE**

This procedure describes the sampling of O2, CO, CO2, SO2, NOx and TOC in a gas flow using a continuous sampling system and subsequent analysis of these components with monitors.

Follow the European standards applicable to the various components, except where this compendium procedure stipulates different or additional requirements.

Measurements with permanently installed devices must meet the requirements in the Code of Good Practice for approval of permanently installed emission measuring devices. Measurement principles for permanently installed devices do not fall under the scope of this procedure.

## SAMPLING

The sampling principle consists of extracting a subsample from a gas flow in a gas duct. This gas flow may have a high temperature and may contain water and dust particles. The system described ensures a continuous flow of cooled, dried and dust-free gas for the monitors.

Two scenarios are possible for waste gas analysis with monitors: the first involves measuring the waste gas using a portable setup with the monitors installed near the measuring opening (i.e. less than 3 m away); the second involves pumping the waste gas, preferably through an overflow system, to the monitors set up in a measuring trolley or container. The choice between these two options depends on the application and the measurement site.

For gaseous components, a single-point measurement, such as in the centre of the gas duct, is possible under certain conditions. The procedure ‘Essential quality requirements for emission measurements’ (LUC/0/005) defines these conditions.

* 1. **SAMPLING SETUPS**
		1. **MONITORS NEAR THE MEASURING POINT**

Extract the waste gas using an inert probe. This probe features a filter to catch the dust. Use an inert, heated line that is as short as possible to transport the waste gas to a cooler, which cools it adequately and condenses the moisture (up to a dew point of (4 ± 3)

°C) so it can supply the gas to the analysers.

If the monitors are set up near the measuring opening, pump the cooled, dried and dust-free waste gas directly through the monitors through a rotameter and heated filter at a constant flow rate (see [Figure 1](#_bookmark69)).

5 7 8 9

6

2

3 10

1

4 1. probe with filter 7. Fine filter

1. inert line 8. monitor(s)
2. cooler 9. data logging
3. condensate drain 10. flue to ventilation
4. rotameter
5. pump

*Figure 1. Waste gas sampling with monitors near the measuring point*

* + 1. **MONITORS IN THERMOCOUPLE CAR**

With a setup in a thermocouple car it is preferable to pump an excess of cooled and dried waste gas in an inert line through a rotameter to the measuring trolley. Next, use a separate pump to pump a substream of this excess waste gas at a constant flow rate through a heated filter and rotameter to the monitors (see [Figure 2](#_bookmark71)). The response time of the overall sampling setup must be less than the performance criterion of 200 s from the EN standards.

* + - 1. probe with filter 8. rotameter

2

2

3

1

4

14

5

7

6

13

12

8

11

10

9

* + - 1. inert line 9. pump
			2. cooler 10. monitors
			3. condensate drain 11. data logging
			4. fine filter 12. and 13. flue to ventilation
			5. pump 14 thermocouple car

*Figure 2. Sampling of waste gases with monitors in a thermocouple car*

Another option for waste gas preparation when using a thermocouple car consists in using a heated line after the probe (1) to transport the waste gas hot, and without water separation, to the cooler and condenser in the thermocouple car.

* + 1. **SPECIAL CASE: OXYGEN**

Oxygen should be measured with high accuracy, due to its use as a reference for dilution in waste gases. When calibrating the oxygen monitor with outside air, either fully dry the gas or cool it with the flue gas cooler. It is preferably however to fully dry the sample gas before supplying it to the oxygen monitor. This rules out measurement uncertainty due to incomplete drying. With oxygen, absolute drying, such as with silica gel, does not risk changing the concentration, unlike with gases such as SO2 and NO2.

* + 1. **SPECIAL CASE: HC TOC WITH FID**

The setups shown here cannot measure hydrocarbons as Total Organic Carbon (TOC) measured with an FID. Standard EN 12619:2013 prohibits the use of condensation or other forms of drying in this process. Heat the sampling line to at least 180 °C.

As a general rule, to further reduce losses of heavier components in the line, set up the FID as close as possible to the measuring point (preferably 1 to 2 metres away). Shield the device from wind and rain as much as possible. [Figure 3](#_bookmark74) shows a typical sampling setup with an FID.



1. Probe
2. Zero and span gas supply
3. Heated particulate filter (inside or outside the stack)
4. Heated sheath/line
5. Heated pump

*Figure 3. Sampling setup for TOC measurement with FID*

* 1. **EQUIPMENT**

Sections [2.2.1](#_bookmark76)  to [2.2.5](#_bookmark83) describe the various components. All connections between components must be made from inert materials and must be sufficiently leakproof.

* + 1. **PROBE**

The probe must be chemically and physically inert for the components to be analysed and must feature a device to install a filter. For the inorganic components, it may be made from glass, stainless steel, titanium, polytetrafluoroethylene (PTFE) or any other inert material. Suitable materials for TOC are stainless steel, PTFE and polypropylene fluoride. Do not use PTFE at temperatures of over 200 °C. Materials such as quartz, titanium and ceramics are suitable at these temperatures. Minimise the gas retention time in the probe.

Adapt the length and/or diameter of the probe to the size of the stack to enable sampling at all measuring points.

* + 1. **FILTER**

The filter must be made from an inert material. The filters used may be flat filters, thimble filters or filter plugs made from quartz or fibreglass or a ceramic/sintered metal filter with a suitable pore size.

For temperatures of over 400 °C, use quartz wool or quartz fibre in any case.

Heat the filter to above the dew point of the gas flow and preferably install it outside the stack, to enable better temperature control.

* + 1. **GAS CONDITIONING SYSTEM**

For O2 and CO, the EN standards prescribe a cooler or permeation dryer for the gas conditioning system ([2.2.3.1](#_bookmark79) and [2.2.3.2](#_bookmark80)). For NOX, in addition to these two configurations, the standards also include a dilution system ([2.2.3.3](#_bookmark81)) and a heated line up to the heated monitor. If the NO2/NOx ratio exceeds 10 %, do not use the configuration with a cooler.

For SO2, it is permitted to use a flue gas cooler, permeation dryer or dilution system. EN 12619:2013 prescribes a heated line up to the monitor.

* + - 1. **COOLER**

The cooler must be designed to minimise absorption of water-soluble components such as NO2 or SO2 in the condensate. The loss of water-soluble components increases with overpressure in the cooling system. Therefore, install the pump between the cooling system and the monitor. At the cooler outlet, the dew point must be (4 ± 3) °C.

Check the cooler for proper functioning at least at the start and end of each measurement.

* + - 1. **PERMEATION DRYER**

Permeation dryers diffuse water through a semi-permeable membrane from the humid waste gas to a dry gas on the other side. Check permeation dryers for efficiency and selectivity.

At the permeation dryer outlet, the dew point must be (4 ± 3) °C.

* + - 1. **DILUTION SYSTEM**

Dilution systems as described in ISO 10396 are used to cool the waste gases and to avoid condensation of water. Dilution systems may be useful in certain situations, but are not considered the most accurate method for emission limit testing (within the meaning of Article 4.4.4.2 of VLAREM) due to the additional measurement uncertainty of the dilution factor.

Standard EN 14792 for NOx prescribes this as a possible configuration to avoid condensation in the measuring system.

* + 1. **SECONDARY FILTER**

The secondary filter or particulate filter purifies the waste gas of the finest dust with a diameter of 1 to 2 µm and serves to protect the analyser. This filter is usually present in the monitors themselves.

* + 1. **PUMP AND FLOW METER**

Monitors that lack an internal pump require an external one.

For setups with direct measurement on the measuring platform, the inert pump must be able to maintain a constant flow rate (suitable for the monitors).

For setups with a measuring trolley, the main pump must be inert and offer adequate capacity to limit the response time of the sampling setup. A second (smaller) inert pump with a constant flow rate and upstream particulate filter handles waste gas extraction for the monitors.

The flow rate of the main pump must be at least twice the flow rate supplied to the monitors.

A rotameter controls and displays the flow rate through the monitors and any main flow rate.

* 1. **PROCEDURE**

Install the cooler (if applicable) as close as possible to the measuring opening. Install the probe, the filter, the intake line to the cooler and the line between the cooler and the pump. Set the gas conditioning system, probe, filter and monitor to the required temperature and condition/heat them. Start the pump and use the rotameter to check that the pump is drawing properly. Once the monitors are sufficiently warmed up and the intake flow rate is constant, it is permitted to start calibration and verification as per Section [3.1](#_bookmark93). After checking the functioning of the cooler, the push the probe into the flue, seal it tight and start the measurement.

* 1. **POINTS FOR ATTENTION**
		1. **LEAK TEST**

Once the sampling system is set up, conduct a leak test: close the gas preparation inlet. The leakage flow rate should not exceed 2% of the normal intake flow rate. If it does, find and seal the leak.

During the leak test, make certain that the monitors cannot be damaged by excessive under or overpressure. Two methods are available for the leak test:

1. After sealing the probe, check whether the rotameter goes to zero or measures the residual flow rate. If using a semi-quantitative criterion for the leak test (e.g. rotameter float goes to zero), the operator must demonstrate that this corresponds to the maximum leakage of 2%.
2. Supply nitrogen at the probe inlet and check whether the oxygen measurement goes to zero. One condition for valid leak testing with this method is that the system must come to the (maximum) under-pressure that occurs during the measurements.

Log leak tests quantitatively, i.e. record the times, gas meter readings and residual leakage flow rate.

* + 1. **COOLER TEMPERATURE CHECK**

The residual water contents of gases that are dried in a cooler still remain significantly higher (around 1%) than the concentrations of the pollutants being measured. Given that many measuring devices work using IR absorption, they are sensitive to changes in water content. The high degree of dependence on residual water content after the cooler makes it necessary to keep the temperature of the cooler constant within narrow limits. Install a control system for cooler temperature during measurement. […] At the cooler outlet, the dew point must be (4 ± 3) °C.

* + 1. **LINE LENGTH**

In any case, keep intake line lengths as short as possible, to ensure adequate response times. Other negative effects of long lines are losses from adsorption, chemical reactions, diffusion, leaks and memory effects. Avoid using lengths of over 50 metres. Condensate or dust particles may cause a sharp increase in line losses. Therefore, only transport sampling gas over longer larger distances ~~(> 2 meter)~~ in heated lines, and in cold lines only after drying in a cooler/condenser. For the TOC measurement with an FID, always heat the sampling line to at least 180 °C. As a general rule, set up the FID as close as possible to the measuring point (preferably within 1 to 2 metres), to reduce losses of heavier components in the line. Replace any lines that exhibit visible signs of deposits.

* + 1. **DEAD VOLUMES**

Unnecessary dead volumes in sampling gas preparation may result in errors due to inadequate response times (especially during calibration) and due to cross-contamination between zero gas, calibration gases and sampling gas. Dead volumes sometimes occur in coolers/condensers and switching systems in measuring trolleys. Just as with line lengths, it is necessary to minimise dead volumes. It is generally possible to detect excessive dead volumes by measuring the response time T90 with increasing and decreasing concentrations. The value should not exceed 2 minutes.

* + 1. **STABLE INTAKE**

With each measurement, regularly check the rotameter reading(s), including at the start and end of the measurement. If deviations from the normal course (clogged line or filters) are detected during the measurement, take immediate action. Keep the flow rate through the sampling system and monitors constant at ±10 %.

* + 1. **END OF MEASUREMENT**

At the end of the measurement, remove the probe from the stack and disconnect it. The probe, coolers and lines may be rinsed with water and possibly also soap. Next, rinse thoroughly with water and dry with oil-free compressed air. If the probe and/or lines are too contaminated, it is advisable to use new ones. Avoid cleaning with organic solvents because their residues may damage the cuvettes and their windows.

For adequate rinsing of the cooler and the filters to prevent corrosion, the main pump continues to draw ambient air for about 15 minutes after each measurement.

## WASTE GAS ANALYSIS

The table below gives the reference and other permitted measurement methods for the various components: CO, NOx, SO2, O2 and TOC. This compendium procedure only applies for continuous measurement with monitors.

|  |  |  |
| --- | --- | --- |
| Component | Reference method and measurement principle | Other permitted methods |
| CO | EN 15058(NDIR-GFC\*) |  |
| NOx | EN 14792(NO: ChemiluminescenceNO2: Chemiluminescence using converter) | NO/NO2 NDUVNO NDIR+NO2 to NO converter\*\* |
| SO2 | EN 14791(Wet chemical measurement method) | Measurement with monitors with the following measurement principles (included in CEN/TS 17021)\*\*\*NDUV NDIRUV fluorescence |
| O2 | EN 14789(paramagnetism) |  |
| TOC | EN 12619 (TVOC concentrations up to 1 000 mg/m3)Flame ionisation detection (FID) |  |

\* Gas filter correlation photometer

\*\* NO2 NDIR is not permitted due to water interference.

\*\*\* If it is necessary to measure SO2 in a gas flow that also contains methane, methane interference must not occur on the monitor.

The EN methods apply in cases such as installations falling under the EU Industrial Emissions Directive and calibration of permanently installed continuous emission measuring equipment. Aside from the reference method, it is also permitted to use the other methods indicated in the table. The wet chemical SO2 determination as per EN 14791 has a limited accuracy (20 %) and slow response. It is only permitted to use EN 14791 for the approval of permanently installed measuring devices if validation or ring testing has shown that they can meet the stronger accuracy requirement of 10 %.

For measurement of SO2, a continuous measuring device must be available. By participating in the ring tests, the reference laboratory ensures the equivalence of the device used in the ring tests with EN 14791 for SO2 measurement and the approval of permanently installed measuring devices. For the latter however, the accuracy requirement of 10 % applies (recognition condition).

For combustion plants of up to 10 MW, the use of electrochemical cells to measure CO, CO2, NOx, SO2 and O2 is also permitted.

If an emission limit for SOx (as SO2) applies for the installation being measured and the presence of SO3 may also be assumed, then in addition to the continuous determination of SO2, also measure SO3 as per compendium method LUC/III/008 (wet chemical). In this case, the same compendium method may also be used for wet chemical determination of the total of SO2 and SO3.

Carbon dioxide (CO2) is not a pollutant regulated under the VLAREM, but it may be measured, for instance, as an alternative reference gas to oxygen, to determine the dilution factor. CO2 is measured under the same measurement principle as CO (NDIR), and both parameters are typically combined in a single device.

* 1. **PROCEDURE**

The complete procedure for continuous measurement with a monitor consists of the following steps:

* + Conduct a leak test on the entire sampling system.
	+ Calibrate the monitor with zero and span gases (in that order) with adjustment. For this, supply the gases directly to the monitor.
	+ Conduct a first-line check with an independent control gas (see Section [3.3](#_bookmark99)). With each measurement, send the control gas through the entire sampling train.
	+ Measure.
	+ Supply zero and span gases again without adjustment (drift determination).

Perform these operations on the measurement site itself. The electronic data files must provide an audit trail for all calibrations (zero, span and control gases).

When conducting a leakage test with nitrogen before monitor calibration, check the leak test criterion for the difference between:

* + - the O2 measurement value with a direct nitrogen supply to the monitor; and
		- the O2 measurement value when sending nitrogen through the entire system.

Alternatively, it is also permitted to conduct the nitrogen leak test after monitor calibration. If the value exceeds the leak test criterion, recalibration with zero and span gases is in fact required.

The zero gas must not contain significant amounts of the components being sampled (e.g. nitrogen or compressed air). For oxygen, the O2 concentration in the zero gas is less than 0.05% by volume.

For the span gas, use CO, CO2, NO, SO2 in N2 or a mixture of these components in N2. For the FID measurement, use nitrogen or air as the zero gas and propane in nitrogen or air as the calibration gas. In this process, the oxygen content of these gases should be as close as possible to the oxygen content of the waste gases.

The span gas concentration must meet the requirements set out in the European standards. Preferably, the control gas concentration should be around 50 % of the span gas concentration.

* + 1. **CALIBRATION BEFORE MEASUREMENT**

Before each measurement, calibrate the monitor with a zero gas and a span gas (in that order, with adjustment). Supply both calibration gases to the monitor directly and under the same pressure and flow rate conditions as during the measurement. The rotameter reading during calibration must be the same as during the measurement.

After obtaining a stable measurement signal, adjust the signal to the correct value. Next, collect the calibration data for at least 5 minutes, both for the zero gas and for the span gas.

* + 1. **VERIFICATION**

For each measurement, send the control gas through the entire sampling system. Make the control charts available onsite and fill them in immediately before starting the actual measurement. It is not permitted to fill in the control chart afterwards, or for a quality manager who is not present on the measurement site to fill it out at another location, as this may result in propagation of measurements of uncontrolled quality. The person taking the measurements must fully understand the control chart system and criteria, to enable proper and immediate action in the event of deviations. Periodic processing of control charts is required unless, for instance, they feature less than 10 points over half a year.

When using set limits on control charts:

* + They must also undergo a periodic review (at least annually).
	+ The limits must be quantitatively justified.
		1. **MEASUREMENT**

After supplying the independent control gas, it is permitted to start the measurement. Log the measurement results from the monitors on a regular basis (at least minute-average values). The devices and the data acquisition system must allow negative values. An exception is granted here for electrochemical devices, as they cannot record negative values.

For each measurement, log the performance times of the leak test, calibration, verification, measurement and recalibration (without adjustment), as well as the identification numbers of the gases used.

* + 1. **DRIFT CHECK AFTER MEASUREMENT**

To verify the drift of the monitor, supply zero and span gases directly back to the monitor after the end of the measurement, but without adjusting the monitor. [3.4.1](#_bookmark101) gives the permissible criteria for drift. Contrary to the EN standards, determine the span drift on the monitors themselves.

* 1. **CALIBRATION GAS QUALITY**

The span gas must be a certified calibration gas, either:

* + - a gas with calibration certificate issued under an ISO 17025 accreditation (BELAC or equivalent) by a manufacturer accredited as a calibration body; or
		- a calibration gas traceable to ISO 17025; here, the analysis by an accredited or recognised laboratory applies as the correct value. The latter case must meet the requirements set out for self-certification of calibration gases; The compendium procedure ‘Essential quality requirements for emission measurements’ (LUC/0/005) provides these requirements.
		- An operator conducting self-auditing measurements and whose methods have been approved as meeting ISO 17025 may certify calibration gases for in-house use under the same conditions as a recognised laboratory.

The permitted calibration uncertainty of the bottle for CO, CO2, NOx, SO2 and propane may not exceed 2%. This is in accordance with the EN standards, with expansion to SO2 and CO2.

The certified value of calibration gases must be based on or verified by means of an accurate verification analysis.

For O2, dried outside air is a good choice for a calibration gas. The oxygen content in dry air is 20.95%. When calibrating the oxygen monitor with outside air, either fully dry the gas (e.g. with a silica gel cartridge) or cool it with the flue gas cooler. If the residual water content in the outside air after the cooler is the same during oxygen monitor calibration and waste gas measurement, it is not necessary to factor this in. In this case, it is also permitted to use an oxygen concentration of 20.95 % to calibrate the monitor (such as for calibration with completely dry air). However, if the dew point of the outside air is lower than the temperature of the cooler, factor the water content difference after the cooler into calibration and measurement.

European standard EN 14789 proposes using 20.9% with a relative uncertainty of 0.5% (= 0.1% O2 absolute). Recognition in Flanders is subject to stringent requirements on the accuracy of the oxygen measurement, and it is advisable to use the ‘more correct’ value of 20.95%. In this case, the uncertainty (< 0.05% relative) is negligible compared to other errors.

NB: At concentrations of over 15% O2, the criterion in EN 14789 for the uncertainty of O2 span gas of 2% relative (k=2) is too high to meet the criterion for recognition (deviation  0.3% absolute).

* 1. **INDEPENDENT CONTROL GAS**

After the calibration of the gas measuring devices, supply an independent control gas for all components; see also Section [3.1.2](#_bookmark95). It is not necessary for the control gas to be from an accredited supplier or ISO 17025-certified.

Independent: the control gas must not be a diluted calibration gas.

The calibration and control gases preferably come from different gas suppliers. If they do not, the gas supplier must be able to demonstrate the independence of these gases, or the calibration gas must be certified by the laboratory with respect to a gas of another supplier. This requirement must be met by no later than 1 January 2018.

It is recommended to stagger purchases of calibration and control gases over time, in a controlled manner.

Control gas concentration: preferably around 50 % of span gas

* 1. **CALCULATIONS**
		1. **CORRECTIONS FOR DRIFT**

The signal from the monitor may be subject to drift (see [Figure 4](#_bookmark102)). This is a gradual change in the signal over a certain period of time. Therefore, calibrate the monitor beforehand and recalibrate it afterwards (without adjustment). If these values differ, the signal for the zero and/or span concentration has drifted.

Concentration

pre-calibration

post-calibration

measurement

test concentration measured signal

Time

*Figure 4. Drift of the monitor*

Depending on the measurement values for the zero and span gases before and after the measurement, it may be necessary to apply corrections for drift to the recorded signals. If the zero or span drifts exceed certain values (see tables below), the measurement must be rejected.

The following zero and span drift criteria apply for measurement with monitors as per EN standards, as well as for measurement with electrochemical devices:

|  |  |
| --- | --- |
|  | Permitted deviation, as a % of concentration of the calibration gas |
|  | Zero | Span |
| No correction needed | ≤ 2% | ≤ 2% |
| Correction of measurement values | 2 to 5% | 2 to 5% |
| Rejection of the measurement | > 5% | > 5% |

For oxygen, the criteria in Section 8.4.3 of EN 14789 (identical to the above table) are too broad. Instead, the following criteria apply:

|  |  |
| --- | --- |
|  | maximum drift, % O2 absolute |
|  | Zero | Span |
| No correction | ≤ 0.2% | ≤ 0.2% |
| Correct | 0.2 to 0.3 % | 0.2 to 0.3 % |
| Reject | > 0.3% | > 0.3% |

For the calculation, distribute the drift (first for zero, then for span) linearly over the entire measurement period and apply it to the measurement values.

In cases of very small NO2 contributions (up to the total NOx), greater zero and span drifts may be permitted for NO2 if the lab can demonstrate that this does not significantly increase the measurement uncertainty of NOx, and that the measurement uncertainty remains below 10 % for measurements as per EN standards.

* + 1. **CONVERSIONS**

The CO2 measurement result is usually expressed in % on the monitor, and must also often be reported as such.

Typically, concentrations of CO, NOx and SO2 should be expressed for 273.15 K and 1 013.25 hPa. The customary unit is mg/Nm3. The following formulas may be used to convert from ppm to mg/Nm³ for the different components:

 3 

COmg/Nm  

 

CO ppm 28

22,4

SO2 mg/Nm3  SO2 ppm 64

22,4

NO mg/Nm3  NO ppm 30

22,4

NO2 mg/Nm3  NO2 ppm 46

22,4

### NO alsNO2 (mg/Nm3)  NO(mg/Nm3)  46

30

‘NOX’ is defined as the sum of nitrogen oxide (NO) and nitrogen dioxide (NO2), expressed as NO2:

NOX als NO2 (mg/Nm3) = NO als NO2 (mg/Nm3) + NO2 (mg/Nm3)

where

22.4 l/mol: molar volume of the ideal gas at 273.15 K and 1 013.25 hPa 28 g/mol: molar mass of carbon monoxide

64 g/mol: molar mass of sulphur dioxide

30 g/mol: molar mass of nitric oxide 46 g/mol: molar mass of nitrogen dioxide

The total concentration of hydrocarbons (TOC) should also be expressed for 273.15 K and 1 013.25 hPa. The customary unit is mg C/Nm3 in propane equivalents:

measurement value (mgC/Nm3)  measurement value(in ppm) nC  MMC

Vm

where

nC: number of carbon atoms in the calibration gas (for propane, nC = 3) MMC: molar mass of carbon (MMC = 12 g/mol)

Vm: molar volume of the calibration gas at 273.15 K and 1 013.25 hPa (Vm = 22.4 l/mol)

In most cases, it is also necessary to convert the measurement results to the concentrations in the dry gas. The residual moisture content of the measured gas is the water content of saturated gas at the temperature to which the coolers are set, normally 4 °C, and at the pressure that exists there. The following formula may be used to calculate this residual moisture content (±0.8%):

pw (mbar)100

%H2O 

pbaro(mbar)

|  |  |  |
| --- | --- | --- |
| where | % H2O: | residual moisture content of the gas after the cooler |
|  | pw: | the vapour pressure of water at the temperature in the cooler, in mbar |
|  | Pbaro: | the atmospheric pressure in mbar |

The vapour pressure of water at temperature t(°C) may be read from vapour pressure tables or a Mollier diagram. The following equation (Antoine equation) provides a good approximation:

log[pw

(mbar)] = 8,19621 

1730,63

233,426  t (C)

Use the following formula to factor in the residual moisture content:

### Concentration mg/Nm3 dr  concentration mg/Nm3  100



100 %H2O

If the water content determination shows that water was not present in the gas flow, do not apply this correction.

Also multiply the %CO2 by this latter factor, to obtain the concentration in the dry gases.

To account for potential dilution effects, the measurement results are often expressed for a reference percentage of oxygen, in the dry gas under normal conditions. The legislation defines these oxygen levels, with the percentage determined by installation type:

* Waste incineration and coincineration plants: 11%
* Solid fuel combustion plants (e.g. coal): 6%
* Liquid and gaseous fuel combustion plants: 3%
* Thermal post-combustion plants: 18%
* Gas turbines: 15%
* Brick plants: 18%

This conversion is based on the following formula:

concentrationR  21  O2R concentrationM

 

21  O2 M

(NB: If the oxygen monitor is calibrated to 20.95%, apply 20.95 instead of 21% oxygen in the above formula as well.)

3

 where concentration R 

concentration at reference oxygen content in mg/Nm dr

concentrationM = measured concentration in mg/Nm3 r

d

O2R = reference oxygen content in %

O2M  measured oxygen content in %

The percentage of carbon dioxide is expressed at the measured oxygen concentration and is not converted to the reference value.

If the concentration must be expressed at a reference oxygen content, conduct an oxygen measurement at the same time as this measurement.

**3.5 POINTS FOR ATTENTION**

Avoid checking emission limits below 5% of the measuring range of a measuring device. Because the number of contributions to measurement uncertainty are proportionate to the measuring range, it is not possible to check an emission limit with sufficient accuracy at the bottom of the scale.

In principle, every optical measurement technique is non-linear and exhibits a curved calibration curve. This effect typically grows worse as the measurement value gets further from the calibration points.

CO is a parameter that often occurs with peaks. A sufficiently high measuring range is desirable for this. Do not blindly apply the general rule (VLAREM) for a measuring range between 0.1 and 3 x the emission limit to the measuring ranges of the devices, as this would cut off the peaks. It is advisable to use devices with a dual measuring range (two optical cells) or two devices with different measuring ranges.

The European standard for NOx also states that it must be possible to measure peak emissions.

If it is necessary to measure SO2 in a gas flow that also contains methane, methane interference must not occur on the monitor.

## VALIDATION

In general, a measurement method under the Flemish Environmental Permitting Regulation [VLAREM] should be usable between 0.1 times and 3 times the emission limit. For standard methods, validate the following parameters:

* + (Intra-)reproducibility, such as from control charts
	+ Correctness, e.g. from ring test data
	+ Working range
	+ Limits of detection and quantification, if relevant
	+ Measurement uncertainty

The European standards for continuous measurement of O2, NOx and CO at large combustion and waste incineration plants require the inclusion of several additional factors in the measurement uncertainty. Section 5 lists all factors to be included in the measurement uncertainty. Also determine these factors by validation, unless reliable data are available from another source. Factor these into the measurement uncertainty for all measurements with monitors, except for the electrochemical devices.

EN standards require an annual linearity test. For those participating in the LABs ring tests for waste gases, the Flemish Institute for Technological Research [VITO] permits a lower frequency of once every 2 years. All others are covered by the ring test. Devices not used in the ring test do however require an annual linearity test, except for the FIDs. For the latter, once every 2 years will suffice.

The following minimum requirements apply to evaluation of monitor linearity:

* Establish criterion for the correlation coefficient (R ≥ 0.995).
* Visually plot the calibration curve to show deviations from it (‘residuals’).\*
* Minimum number of points: five + zero (after elimination of points, at least five points must remain).
* Do not force calibration curve through the origin.
* It will suffice to measure each concentration once.

\* To verify device linearity, fit a straight line through all points, with the measured concentration as the y coordinate and the reference concentration as the x coordinate. Display this regression line along with the equation and the correlation coefficient. This enables visual detection of systematic deviations.

Residuals:

Use the regression line equation to recalculate the reference concentrations (x values), to obtain ‘recalculated measured concentrations (̂ values)’.

The % deviation of these recalculated concentrations from the measured concentrations must not exceed 2% of the full measuring range.

Thus:

𝑦 − 𝑦̂

𝑀𝐵

× 100% ≤ 2%

where *y*= measured concentration

̂= concentrations recalculated with the calibration curve (obtained by plugging the reference concentrations x into the calibration curve equation)

MB= full measuring range

Next, plot the residues − ̂ against the concentrations created. Visual inspection of the residues can recognise the linearity error from a clear pattern in the residues.

For the TOC measurement with an FID, also validate the relative response factors (RRFs) and oxygen synergism, possibly by regular participation in the ring test. Validation of these parameters for each device type will suffice.

The performance criteria set out in the relevant standard apply.

According to EN 14792, the NO2/NO converter efficiency when measuring NOx with the chemiluminescence monitor must be at least 95%. This compendium method designates the following criteria as good practices:

|  |  |
| --- | --- |
|  | Converter efficiency |
| No correction | ≥ 95% |
| Correction | 80 to 95 % |
| Reject results | < 80 % |

Test the converter efficiency at the highest NO2 concentrations that occur in practice. As a guideline for combustion process measurements, an NO2 concentration of ±10 % of the highest NOx measuring range or the emission limit will suffice for the test. Some converters appear to be less efficient at high NO2 concentrations. When using a converter to measure processes with higher NO2 concentrations, the efficiency must be demonstrated at the relevant NO2 concentration.

Revalidation is required in case of:

* An essential adaptation of the method
* A change of device type
* A statutory change to the measurement range

Validation by instrument type is permitted: an intake inspection will suffice when purchasing a device of a similar type (to be documented in a procedure, e.g. linearity check). Determine the limits of detection and quantification separately for each device.

## DETERMINATION OF MEASUREMENT UNCERTAINTY

All recognised laboratories must have an evaluation of measurement uncertainty in place for measurements with monitors. If the applicable standards give a criterion for measurement uncertainty, it must be met. For the oxygen measurement, the measurement uncertainty must fall within the recognition criterion of 0.3% absolute.

The measurement uncertainty must be determined around the emission limit, but calculation of this must be possible for each measured concentration. Some of the measurement uncertainty contributions are proportionate to the measuring range or constant (constant absolute error), while others are proportionate to the measured concentration (constant relative error). The measurement uncertainty calculation must clearly distinguish these terms.

Perform the measurement uncertainty calculation for measurements with monitors at large combustion plants and waste incineration plants as per European standards EN 14789 for oxygen, EN 14792 for NOx and EN 15058 for CO.

NB: The requirements in the standards apply. The non-standard examples that these standards provide for illustration purposes may omit certain terms. This is no reason to leave these terms out of the calculation. Be certain to include the following parameters in the evaluation of the measurement uncertainty for monitors:

* + Leakage rate
	+ Losses in the line and conditioning system, loss/conversion at the filter
	+ Residual water content (if not corrected)
	+ Uncorrected zero and span drifts
	+ Linearity error (‘lack of fit’)
	+ Repeatability at span level
	+ Interferences
	+ Uncertainty of the calibration gas
	+ Contributions from the following sensitivity factors: mains voltage, pressure, sampling flow rate, temperature
	+ Converter efficiency for NO2 if applicable

For the contributions from the sensitivity factors (mains voltage, pressure, sampling flow rate, temperature), the EN standards set relatively high specifications. If the exact value is not available for older devices, it is permitted to use half of the permissible specification. When purchasing new measuring devices, ask the supplier for the sensitivity factor specifications as per the EN standard (e.g. EN 15267-3, Table 1), which are needed as contributions to measurement uncertainty. Preferably, use data determined by an independent institute.

Also include these factors in the measurement uncertainty when using other monitors (e.g. SO2 and FID) and in measurements on other installations, such as process plants, medium-sized combustion plants or post-combustion plants.

## REFERENCES

ISO 10396: 1993

Stationary source emissions - Sampling for the automated determination of gas concentrations

EN 14789: 2017

Stationary source emissions - Determination of volume concentration of oxygen - Standard reference method: Paramagnetism

EN 14791: 2017

Stationary source emissions - Determination of mass concentration of sulphur oxides - Standard reference method

EN 14792: 2017

Stationary source emissions - Determination of mass concentration of nitrogen oxides - Standard reference method: chemiluminescence

EN 15058: 2017

Stationary source emissions - Determination of the mass concentration of carbon monoxide - Standard reference method: non-dispersive infrared spectrometry

EN 15267-3: 2007

Air quality - Certification of automated measuring systems - Part 3: Performance criteria and test procedures for automated measuring systems for monitoring emissions from stationary sources

NEN EN 12619: 2013

Stationary source emissions - Determination of the mass concentration of total gaseous organic carbon - Continuous flame ionisation detector method

CEN/TS 17021

Stationary source emissions - Determination of the mass concentration of sulphur dioxide by instrumental techniques

Compendium for air sampling, measurement and analysis [LUC] Essential quality requirements for emission measurements (LUC/0/005) <http://www.emis.vito.be/lne-erkenningen-lucht>

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