Vlaamse Regering

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 concerning environmental policy, Article 5.6.2(6), inserted by the Decree of 25 April 2014, and Article 5.6.5(2), included by the Decree of 25 April 2014 and amended by the Decree of 8 December 2017;
- the VLAREL of 19 November 2010, Article 45(1), second paragraph, inserted by the Flemish Government Decree of 3 May 2019.

Procedural requirements

The following procedural requirements have been met:
- The Department of Area Development, Environmental Planning and Projects of the Environment Department made a proposal to amend the compendium for the sampling, measurement and analysis of[LUC) on [date].
- The draft was notified to the European Commission in notification No [xxx] on (date) in application of Article 5 of Directive (EU) 2015/1535 of the European Parliament and of the Council of 9 September 2015 laying down a procedure for the provision of information in the field of technical regulations and of rules on Information Society services.
- The draft ministerial decree was published on the website of the Environment Department and the participation portal for environmental policies from [date] to [date]. During that period, anyone could communicate their comments.

Justification

This Decree is based on the following grounds:
- As a result of international and Flemish developments and recent research, adjustments to a number of existing methods and the inclusion of a new method in the compendium for air sampling, measurement and analysis (LUC) are necessary.
- Article 45 of the Flemish Environmental Recognitions Regulation [VLAREL] 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. There exist cross-references between the compendia.
- For practical, organisational and legal reasons for the recognised laboratories, the government and clients, it is appropriate that all compendia enter into force on the same date. The joint date of entry into force shall be 15 January 2026.

- For operators, for example in the context of self-monitoring, the updated LUC shall enter into force ten days after the publication of this Ministerial Decree in the Belgian Official Gazette.
- Laboratories and other stakeholders were informed about and consulted on the amendments to the LUC. The updated compendium for air sampling, measurement and analysis (LUC) can be consulted online.

Legal context

This Decree is consistent with the following regulation:
- the Ministerial Decree of 29 April 2025 approving the compendium for air sampling, measurement and analysis (LUC) and amending the Ministerial Decree of 7 May 2024 approving the compendium for air sampling, measurement and analysis (LUC) and amending the Decree of the Flemish Government of 19 November 2010 adopting the Flemish regulation on environmental accreditations.

THE FLEMISH MINISTER FOR ENVIRONMENT AND AGRICULTURE DECREES:

Sole article. The compendium for air sampling, measurement and analysis, abbreviated to LUC, and the corresponding table of contents, as set out in the Annex to this Decree, are hereby approved.

With regards to laboratories for compliance with the conditions for approval referred to in Chapter 4 of the VLAREL of 19 November 2010, and compliance with the requirements for use referred to in Chapter 8 of the VLAREL of 19 November 2010, the approval referred to in the first paragraph shall take effect as of 15 January 2026. In all other respects, this approval shall take effect on the tenth day following the publication of this Decree in the Belgian Official Gazette.

Brussels, (date).

The Flemish Minister for Environment and Agriculture,

Jo BROUNS

Annex. Compendium of air sampling, measurement and analysis (LUC) as referred to in the sole article

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Measurement of gas velocity and volumetric flow rate in a gas channel	LUC/0/004	June 2023
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The compendium for air sampling, measurement and analysis (LUC)

The compendium mentioned in the sole article is available in full via the website https://emis.vito.be/nl/erkende-laboratoria/lucht-gop/compendium-luc with reference MB 2026.

To be annexed to the Ministerial Decree adopting the compendium for air sampling, measurement and analysis [LUC].

Brussels, (date).

The Flemish Minister for Environment and Agriculture,

Jo BROUNS

Determination of water in a ducted gas stream

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1 OBJECTIVE AND SCOPE

The water vapour content of a gas shall be measured for the determination of the gas velocity and volumetric flow rate, for the conversion of concentrations to standard conditions (dry gas) and for the isokinetic suction flow rate. This procedure applies to the determination of water vapour content in channelled gas streams or gas streams in flues.
For the determination of the water vapour content of a flue gas, the following norm applies:

- EN 14790: Stationary source emissions – Determination of the water vapour in ducts

This norm applies except for points where other requirements are explicitly imposed under Section 3. This compendium procedure explains the main aspects of this norm. Aside from the reference method, using the other methods indicated in the table is also permitted.

Reference method	Other permitted methods
• EN 14790 (Condensation/adsorption, or adsorption method)	Absorption in sulphuric acid by a method validated by VITO (subject to 30 minutes of sampling time) FTIR (subject to 30 minutes of sampling time)

The methods described only apply to unsaturated gas streams and gas streams in which no water droplets are present. The amount of water in the gas phase is determined.

If the water vapour content must be known for conversion from wet to dry, then in case of doubt about the presence of droplets in the gas stream and thus saturation of the gas stream , sampling must always be carried out according to the prescribed methods unless the water vapour content is less than 2 vol%. If the water content measured by the above methods exceeds the theoretically calculated or tabled water vapour content for saturated gases at the flue gas temperature, the gas stream shall be saturated and the measured value shall be invalid. In that case, the water vapour content for saturated gases can be reported at the temperature of the flue gas.
If the presence of droplets is to be demonstrated only for the selection of the sampling method of the VOC or to check whether isokinetic sampling of the water-soluble components is required, this can also be done by using psychrometry (see 10.1). If the method indicates that the gas stream is not saturated, sampling in accordance with EN 14790, absorption in sulphuric acid or continuous measurement with FTIR will still be carried out in order to be able to decide whether isokinetic sampling should be carried out, unless the water vapour content is less than 2 % vol.

2 PRINCIPLE

Standard EN 14790 prescribes an adsorption or a combined condensation/adsorption method as a reference method. The gas stream is dedusted and all parts for the water collection unit must be heated. The total mass of water collected shall be determined gravimetrically.

An equivalent alternative method is the absorption of water in concentrated sulphuric acid (96 %). A known amount of flue gas is drawn with a flow rate of 5 to 6 l/min through a pre-weighted wash bottle with concentrated sulphuric acid (Swaans et al, 1998). The weight increase of the wash bottle together with this of the suction probe is a measure of the water content of the gas. This method uses the pronounced hygroscopic character of concentrated sulphuric acid. The referenced method validated by VITO is valid for the determination of water in gas streams with water vapour content from 0.04 to 40 % (0.0004 to 0.536 kg/Nm^{3}dr). For water vapour content up to 15 %, a sample volume of 50 l at an intake rate of 5-6 l/min is required, corresponding to a sampling time of approximately 10 minutes. With even higher water vapour content, the suction volume and therefore the sampling time according to this method is further reduced (see '5 Sampling Procedure'). According to the European standard EN 14790, the minimum sampling time for water vapour content determination is 30 minutes. In order to comply with the validated VITO sulphuric acid method, consecutive sampling should be carried out until a total measuring period of 30 minutes is available (see '5 Sampling Procedure'). Alternatively, an increase in the volume of concentrated sulphuric acid is also permitted, provided that the conditions laid down in Chapter 5 are met.

Since the scope of this method is limited to unsaturated gas streams without droplets, sampling is carried out at a constant intake rate.

3 ADDITIONS TO OR DEVIATIONS FROM THE NORM

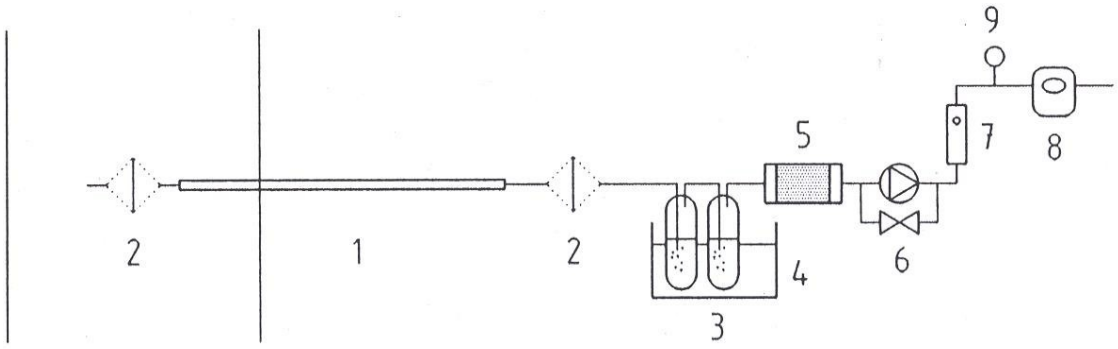
- Determine the water vapour content at least as a half-hour average;
- Determine the water vapour content at least by adsorption and weighing. Condensation alone is not sufficient unless the water vapour content is required only for speed measurement;
- The use of a non-heated probe and suction tube will also be permitted provided that it is included in the weighing before and after sampling;
- A balance must be available in the vicinity of the measurement site if the water vapour content is needed to conduct isokinetic sampling. Otherwise, weighing after the measurements is also permitted.
- The balance must feature a check weight and at least one set deviation criterion. If the value exceeds the criterion, appropriate action must be taken.
- The conditions for carrying out the leak test are described in the procedure 'Essential quality requirements for emission measurements' (LUC/0/005);
- The conditions under which gaseous components in unsaturated gas streams may be measured at one point are included in the procedure 'Essential quality requirements for emission measurements' (LUC/0/005). If this is not complied with, a grid measurement must be carried out.

4 EQUIPMENT

4.1 CONDENSATION/ADSORPTION METHOD

4.1.1 SETUP

Figure 1 displays a possible arrangement for the condensation/adsorption method according to EN 14790. A™ part of the flue gas is extracted from the stack and through a condensation and adsorption unit over the gas meter via a heated probe. EN 14790 (8.3) states that the wash bottles for condensation at the start of sampling must be partly filled with water (volume and then half the volume of the wash bottle).



1	heated probe	6	Pump
2	heated filter (in or outside the stack)	7	Gas flow rate meter
3	impingers or absorbers	8	Gas volume meter
4	cooling (optional)	9	temperature and pressure measurement
5	adsorption cartridge		

Figure 1: Condensation/adsorption method setup for water vapour content determination (source: EN 14790)

VITO has performed initial validation of such a method where the condensation stage consisted of an empty impinger and the adsorption stage filled from an impinger with newly purchased silica gel (Swaans et al, 2006). During validation of this method, both impingers were placed in an ice bath. More recent tests revealed that the partial water filling of the condensation wash bottles with regenerated silica gel resulted in an increase in the efficiency of water vapour content determination by around 4 % (see the following table: in the first empty wash bottle test followed by a wash bottle filled with silica gel, the efficiency is 95.4 % while in the other five wash bottle tests partly filled with water and wash bottle with silica gel, this was on average (99.3 ± 0.4) %). In order to avoid this drop in efficiency in the case of regenerated silica gel, it is prescribed to partially fill the wash bottle(s) for condensation with water at the start in accordance with EN 14790.

Additional validation tests of the condensation/adsorption method

Test	Water generation H_2O volume conc, in %	Measurement H_2O volume conc, in %	Setup	Measurement method efficiency (measured/generated) %
1	10.33	9.85	Empty WF + WF silica gel	95.4
2	10.33	10.27	WF with water +WF silica gel	99.4
3	10.33	10.23	WF with water +WF silica gel	99.0
4	10.33	10.22	WF with water +WF silica gel	98.9
5	10.33	10.31	WF with water +WF silica gel	99.8
6	10.33	10.24	WF with water +WF silica gel	99.1

WF: wash bottle

4.1.2 EQUIPMENT

All parts of the sampling equipment prior to water removal must be be heated and must consist of material that does not absorb water and does not react. Stainless steel, quartz or borosilicate glass, PTFE and titanium are allowed. By way of derogation from EN 14790, the use of a non-heated probe and suction tube is also permitted, provided that these are included in the weighing before and after sampling.

4.1.2.1 PROBE

The probe shall be fitted with a filter inside or outside the flue and a heated mantle set to at least 120 °C and 20 °C higher than the (acid) dew point of the gases. If the filter is located outside the stack, the filter housing will also be heated.

4.1.2.2 FILTER AND FILTER HOLDER

Dust particles ending up in the condensate bottles are included in the weighing. A filter can be used as standard, but is in principle only necessary for high dust content and high suction flow rates. Different types of filters can be used taking into account a minimum stay time of the gas.

When connecting the filter holder to the probe, cold zones should be avoided.

4.1.2.3 COLLECTION UNIT FOR WATER

The water collection unit for this method consists of:

a) condensation stage with an optional cooling system (e.g. ice cooling of the impingers or cryogenic system)
b) adsorption stage: cartridge, impinger or absorber filled with a suitable desiccant such as coloured silica gel

The temperature after the condensation stage should be as low as possible. The discolouration of the amount of silica gel in the last impinger or cartridge may not exceed 50 %.

4.1.2.4 SUCTION TUBE

Any type of high temperature resistant material that does not absorb water and does not react can be used as suction tube (e.g. PTFE). If the suction tube is not heated and therefore condensation can occur, it should be included in the weighing.

4.1.2.5 PUMP AND GAS METER

The pump must be leak-proof. A wet or dry gas meter can be used as a volume meter. The gas volume meter must be calibrated at the flow rate set during sampling. The maximum permitted relative extended measurement uncertainty is included in procedure LUC/0/005.
The gas meter is equipped with a thermometer specified under 4.1.2.6.

4.1.2.6 THERMOMETER

The thermometer should have low thermal inertia. For example, a Pt-100 resistance or a thermocouple can be used.

The maximum permitted relative expanded uncertainty on temperature measurement is set out in procedure LUC/0/005.

4.1.2.7 BAROMETER

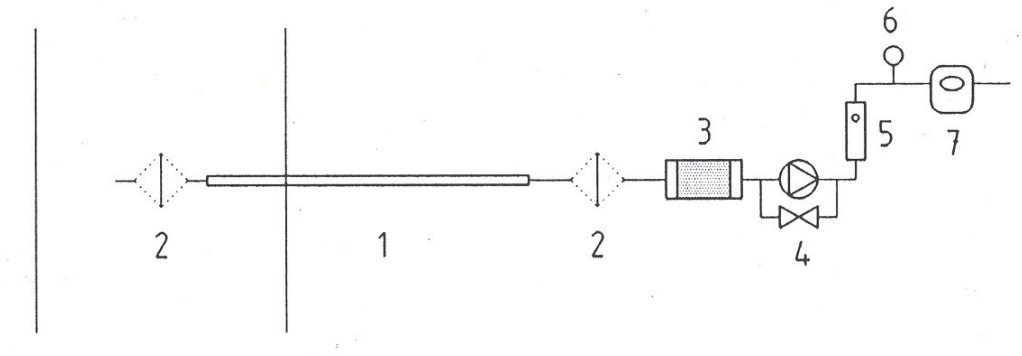
The barometric pressure must be known for converting the suction volume to standard conditions (273.15 K and 1013.25). The maximum permitted relative expanded uncertainty on absolute pressure measurement is included in procedure LUC/0/005.

4.1.2.8 BALANCE

The resolution of the balance must be equal or better than 0.1 g or 2.0 % of the weight of water to be measured (EN 14790 point 6.10).

4.2 ADSORPTION METHOD

4.2.1 SETUP



1	heated probe	5	Gas stream meter
2	heated filter (in-stack or out-stack)	6	temperature and pressure measurement
3	adsorption cartridge	7	Gas volume meter
4	pump		

Figure 2: Preparation of adsorption method for water vapour content determination (source: EN 14790)

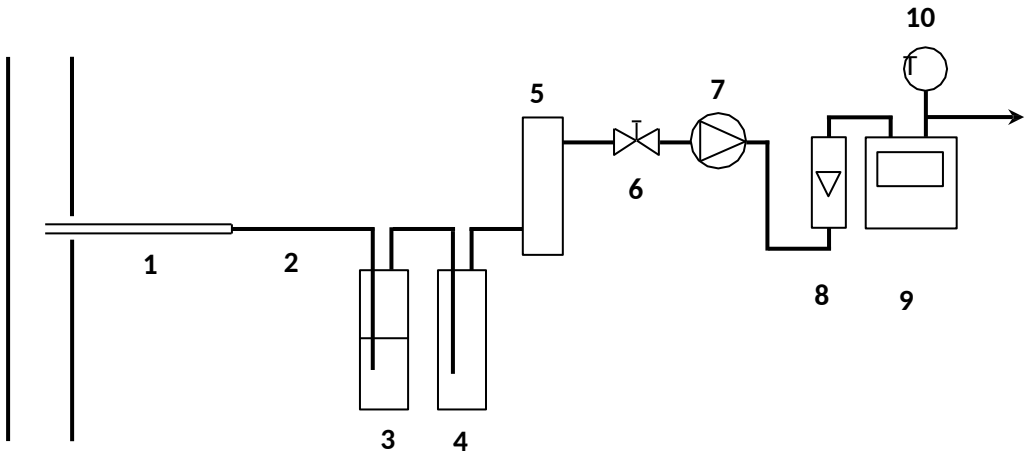
4.2.2 EQUIPMENT

The adsorption method equipment is similar to that of the condensation/adsorption method under 4.1.2, but the water collection unit here consists only of an adsorption stage (without prior condensation stage). The discolouration of the quantity of silica gel may not exceed 50 %. For water vapour content greater than 10 % or for hot flue gases or excessive outdoor temperatures, it is appropriate to apply a prior cooling stage (=impinger partially filled with water in an ice bath for prior condensation).

4.3 ABSORPTION IN SULPHURIC ACID

4.3.1 SETUP

Figure 3 displays the setup for determination of the water vapour content by absorption in sulphuric acid. This method was validated by VITO (Swaans et al, 1998).



1	suction probe (possibly with filter)	6	valve
		7	pump
2	suction tube	8	rotameter
3	impinger with H_2SO_4	9	gas meter
4	safety impinger	10	thermometer
5	silica gel cartridge		

4+ 5=optional for protecting the pump and gas meter

Figure 3: Determination of water vapour content via absorption in sulphuric acid

4.3.2 EQUIPMENT AND REAGENTS

4.3.2.1 PROBE

Analogous to 4.1.2.1 when using a heated probe. If the probe and the suction tube are not heated, it must be possible to weigh them before and after sampling.
A filter can be used as standard, but is in principle only necessary for high dust content, as the suction flow rate through the wash bottle is limited. It is then necessary to verify the dust content from which an interference with determination of the weight of the sulphuric acid occurs.

4.3.2.2 WASH BOTTLES

Absorption efficiency depends on the amount of sulphuric acid used, the concentration of the sulphuric acid and the type of wash bottle or impinger. Therefore, the method must be validated when using a particular type of wash bottle/impinger and the same type should always be used for the actual measurements.
VITO carried out validation of the method with a wash bottle (Egilabo) without frit or narrowing (see Figure 4). The weight of the filled wash bottle after sampling must be weighable on an analytical balance of 400 g (internal diameter of the insert is 5 mm). With this validated VITO method, the wash bottle is filled up to the mark (± 50 ml) with sulphuric acid (96 % by weight). Sulphuric acid can be used up to a concentration of 90 % by weight or up to a weight gain of approximately 6.5 g per 50 ml H_2SO_4.

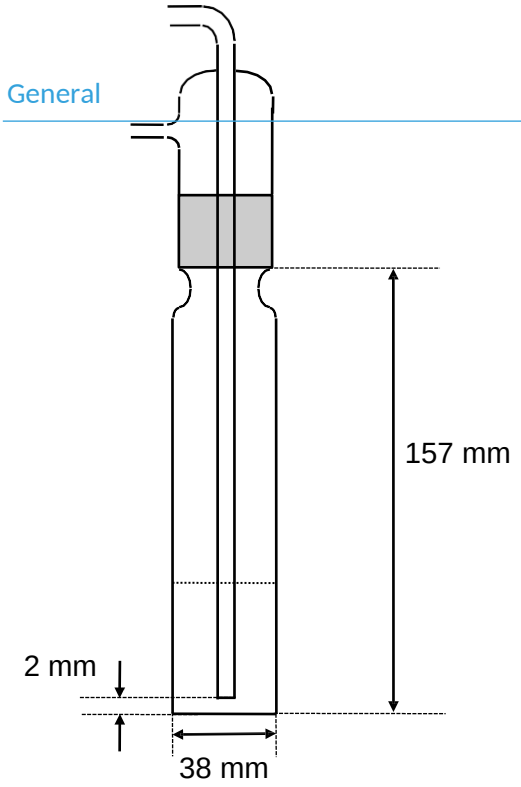


Figure 4: Dimensions validated wash bottle (Swaans et al, 1998)

4.3.2.3 *PUMP AND GAS METER*
Analogous to 4.1.2.5.

4.3.2.4 *THERMOMETER*
Analogous to 4.1.2.6.

4.3.2.5 *BAROMETER*
Analogous to 4.1.2.7.

4.3.2.6 *BALANCE*
Analogous to 4.1.2.8.

4.3.2.7 *ABSORPTION LIQUID*
As an absorbent liquid, concentrated sulphuric acid (concentration 96 %) is used in this method. Sulphuric acid can be used up to a concentration of 90 % by weight or up to a weight gain of approximately 6.5 g per 50 ml H_2SO_4.

5 SAMPLING PROCEDURE

- The water collection unit, including connectors, shall be mounted and pre-weighed. If probe and suction tube are not heated, these must be included in the weighing.
- If using a heated probe, allow probe and filter housing to reach temperature (at least 120 °C and 20 °C higher than the (acid) dew point of the gas stream).
- All parts of the setup are connected to each other in a way that is leakproof. A leak test shall then be carried out (e.g. by sealing the suction probe and then starting the pump); the leak flow rate shall not exceed 2 % of the rated suction flow rate.
- The temperature of the heating jacket and filter holder are checked.
- The initial position of the gas meter and the time shall be recorded;

- The suction probe shall be positioned sufficiently deep into the gas channel (at least 30 cm from the wall or in the centre of the gas channel) and the remaining aperture around the probe is carefully sealed, the pump is started and adjusted to the desired constant flow rate and a partial current is sampled. The minimum sampling time is 30 minutes, the minimum gas volume to be extracted is 50 l. In the case of the sulphuric acid method validated by VITO in 1998, the sampling time is the function of the expected water vapour content (Swaans et al, 1998): with high water content, dilution of the sulphuric acid may occur, reducing absorption efficiency. For water vapour content up to 15 %, the validated VITO method requires a sample volume of 50 l at an intake rate of 5-6 l/min. If the water vapour content is greater, a reduction in the suction volume is prescribed:
    - 0-15 % water: ± 50 l sampling
    - 15-20 % water: ± 30 l sampling
    - 20-30 % water: ± 20 l sampling
    - 30-40 % water: ± 10 l sampling

  If this validated sulphuric acid method is used and therefore it is not possible to sample for 30 minutes, consecutive sampling shall be carried out until a total measuring period of 30 minutes is available. Alternatively, an increase in the volume of concentrated sulphuric acid is also permitted, provided that the following conditions are met:
    - after the sulphuric acid impinger(s), an adsorption cartridge with a suitable desiccant will always be placed in the water collection unit as a last step; this adsorption cartridge must also be taken into account.
    - The quantity of discoloured desiccant will also be visually inspected and will not exceed 50 % of the total.
- The average temperature at the gas meter during sampling and the barometric pressure are recorded; standard EN 14790 states that the temperature and absolute pressure at the gas meter must be recorded at least five minutes after start and at the end of the sampling period.

- During sampling, it should be checked that the capacity of the water collection unit is not exceeded. The quantity of discoloured adsorption shall be inspected visually and may not exceed 50 % of the total. The sulphuric acid method was validated by VITO to a maximum of 6.5 g of water per 50 ml concentrated H_2SO_4 (96 %) and thus to an H_2SO_4 concentration of 90 %.
- After sampling, the water collection unit shall be re-weighed;
- The final volume of the gas meter and time shall be recorded;

6 CALCULATIONS

6.1 CONDENSATION/ADSORPTION OR ADSORPTION METHOD

The calculations for the condensation/adsorption or adsorption method are described in point 9 of the EN 14790 standard. The water vapour content is calculated from the weight of water collected in the condensation/adsorption unit and the volume extracted:

$$C_w = \frac{m_w}{V_{m(ref)}}$$

By which:

C_w: water vapour content on dry basis, in g/m^3
$V_{m\,(ref)}$: measured gas volume under normal conditions (273,15 K and 1013.25 hPa), dry in m^3
m_w: mass of water vapour collected with the water collection unit, in g

The gas volume under normal conditions, dry can be calculated as follows: Use of a

dry gas meter:

$$V\,m(ref) = (V_2 - V_1) \times \frac{T_{ref}}{Tm} \times \frac{P_m}{P_{ref}\,V} \times -V\,¿$$

Use of a wet gas meter:

$$V\,m(ref) = (V_2 - V_1) \times \frac{T_{ref}}{T} \times \frac{P_m - P_{s(Tm)}}{P_{ref}}$$

By which:

$V_{m(ref)}$: measured gas volume under normal conditions, dry in m^3

V_1: is the gas volume reading of the gas volume meter at the beginning of the sampling period, at actual conditions of temperature, pressure and humidity, in m^3;

V_2: is the gas volume reading of the gas volume meter at the end of the sampling period, at actual conditions of temperature, pressure and humidity, in m^3;

$V_2 - V_1$: measured gas volume, at temperature, pressure and humidity during the measurement, in m^3 T_m: average temperature of the sampled gas measured at the gas meter, in K T_{ref}: Normalised temperature (273.15 K)

P_m: absolute pressure at the gas meter, in hPa (this is the sum of the relative pressure at the gas meter + atmospheric pressure)

$P_{s(Tm)}$: saturation vapour pressure of water at the temperature at the gas meter, in hPa P_{ref}: Normalised pressure (1013.25 hPa)

The water vapour content in the wet gas in vol % is then equal to:

$$(\%)H_2O = (V_2 - V_1)\frac{\frac{m_w \times V_{mol}}{M_w}}{\frac{M_w \times V_{mol}}{M_w} + V_{m(ref)}} \times 100$$

where:

(%)H_2O: water vapour content in wet gas, in vol % (m^3 water vapour in m^3 wet gas)

$V_{m(ref)}$: measured gas volume under normal conditions, dry in m^3

m_w: mass of water collected with the water collection unit, in g

Mw: molecular weight of water, 18 g/mol

V_{mole}: molar volume at normal conditions ($22{,}4.10^{-3}$ m^3/mol at 273.15 K and 1013.25 hPa)

Normal conditions: normalised temperature (273.15 K) and pressure (1013.25 hPa)

6.2 ABSORPTION IN SULPHURIC ACID

The following data are required for the calculation of the water vapour content:

- the weight gain of probe and wash bottle (and possibly the desiccant adsorption cartridge) (m_w) in g
- the average temperature at the gas meter (T_m)
- absolute pressure to the gas meter, in hPa (this is the sum of the relative pressure at the gas meter + atmospheric pressure)
- the extracted volume ($V=V_2-V_1$) in m^3

The water vapour content is also calculated using the formulas under 6.1 where m_w is the weight gain of probe and wash bottle (and possibly the adsorption cartridge with desiccant).

In order to comply with the minimum sampling time of 30 minutes, this method shall be used to carry out consecutive sampling. An average water vapour content is calculated and reported. Alternatively, if an increase in the volume of sulphuric acid is applied, an adsorption cartridge with a suitable desiccant should always be placed in the water collection unit after the impinger(s) with sulphuric acid. This adsorption cartridge should also be considered.

6.3 SATURATED GAS STREAMS

The calculations under 6.1 and 6.2 are only valid for unsaturated gas streams. If the gas stream is saturated or oversaturated, the temperature of the gas corresponds to the dew point and the partial vapour pressure and volume mass of the saturated water vapour at the gas temperature is derived from tables (see Section 6.6 of EN 14790). The gas temperature is measured at a representative point in the gas channel.

To determine whether the gas stream is saturated or oversaturated, the maximum percentage of water that can be present at the temperature (up to 100 °C) and pressure in the gas channel can be calculated using the following formulas:

The maximum vapour pressure of water p_w at temperature t, expressed in hPa:

$$\log pw = 8.19621 - \frac{1730.63}{233.426 + t(°C)}$$

$$\%H_2O = \frac{pw(hPa)}{pchannel(hPa)} \times 100$$

Where pchannel= absolute pressure in the channel in hPa (= sum of atmospheric pressure + static pressure)

If the measured water vapour content exceeds this percentage, it must be assumed that the gases are oversaturated and there are droplets present.

7 FOCUS POINTS

7.1 CONDENSATION/ADSORPTION METHOD

If the condensation/adsorption method is combined with the dust measurement and thus higher suction flow rates are set, it must be ensured that the filter efficiency is sufficient. Otherwise, the material passed must also be weighed and considered condensate.

The method is not applicable to gas streams containing a high concentration of organic substances or other components that may cause a weight gain of the condensation/adsorption unit for water.

7.2 ABSORPTION IN SULPHURIC ACID

Very high water content (e.g. steam plumes) can lead to the boiling of the sulphuric acid, allowing it to escape from the wash bottle.
When the impinger is hot running, the vapour pressure of water increases, reducing the (maximum feasible) efficiency of the absorption. A pre-separation with an empty ice-cooled wash bottle may

be used to measure water vapour content greater than 40 %.

At high particulate levels, the result of the water vapour content is affected by the fact that the dust also causes a weight gain in the wash bottle. The procedure should specify the dust content from which precautions are applied to prevent dust from entering the wash bottle.

The method is not applicable to gas streams containing a high concentration of organic substances or other components that may cause an increase in sulphuric acid by weight.

When performing the leak test, caution should be exercised when reopening the seal so that the sulphuric acid would not be sucked into the silica gel cartridge and suction unit. To avoid this, an extra empty wash bottle can be placed after the wash bottle with sulphuric acid.

8 VALIDATION

For standard methods, validate the following parameters:

- Reproducibility/intra-reproducibility, such as from control charts;
- Correctness, e.g. from ring test data;
- Working range;
- Limit of demonstrability and determination;
- Measurement uncertainty;

Where possible, validate these performance characteristics for the combination of sampling and subsequent analysis. If not possible, validation should be carried out at least on the analysis method in accordance with the CMA/6/A procedure. This procedure also gives definitions for the various performance characteristics.

9 DETERMINATION OF MEASUREMENT UNCERTAINTY

According to the EN 14790 standard, the measurement uncertainty on the water vapour content must be less than 20 % of the measured value. The maximum permitted deviation during VITO ring tests shall be 10 % of the measured value.

The following factors should definitely be evaluated and quantified to determine the total measurement uncertainty on the water vapour content determination by condensation/adsorption and absorption in sulphuric acid, respectively:

- Uncertainty on the weight (or volume) determination of the amount of water collected:
  - ✓ Uncertainty associated with the balance sheet (typical uncertainty contributions include, for example, linearity of the balance sheet, resolution, reading, calibration);
  - ✓ Repeatability of weighing the water sampling unit;
- Uncertainty due to the permitted leakage rate (≤ 2% of the rated suction flow rate);
- Uncertainty on the extracted volume with the gas meter. Uncertainty contributions to be introduced here are:
  - ✓ Uncertainty of extracted volume (≤ 2.0%);
  - ✓ Uncertainty on temperature measurement at the gas meter (≤ 2.5K);
  - ✓ Uncertainty on the absolute pressure measurement at the gas meter (≤ 1% of absolute pressure);
- Uncertainty on sampling due to other substances that are adsorbed/absorbed and weighed (dust, organic substances).

In 2006, the validation of a condensation/adsorption method was carried out by VITO. Measurement uncertainty calculations for different water vapour levels are included in the validation report (Swaans et al, 2006 - https://emis.vito.be/nl/erkende-laboratoria/lucht-gop/onderzoeksrapporten).
The EN 14790 standard also provides an example for calculating the measurement uncertainty in the condensation/adsorption or adsorption method.

10 ALTERNATIVE METHODS

In Chapter 1, it was stated that, in addition to the standard reference method (SRM) according to standard EN 14790, the following alternative methods to the SRM are also permitted:

- Absorption in sulphuric acid by a method validated by VITO (subject to 30 minutes of sampling time);
- Measurement with FTIR in accordance with LUC/II/003 (subject to 30 minutes of sampling time);

If the water vapour content is required only for the velocity measurement (isokinetism setting), the methods under 10.1, 10.2 or 10.3 can be applied alternatively. These methods cannot be used for conversion from concentrations to dry gases for the test against emission limit values or for the conversion from wet volumetric flow rate to dry gas flow rate, in case of mass flow rates shall be calculated unless the water vapour content of the gas stream is less than 2 vol. %. If one of these methods shows that the water vapour content is below 2 % vol., no determination in accordance with EN 14790 or absorption in sulphuric acid method or through a FTIR measurement should be carried out. However, the actual measured value should be further calculated.

The alternative methods for the determination of water vapour content by psychrometry and condensation, respectively, are described in the standard NBN T 95-001.

The capacitive moisture probe (10.3) or psychrometry (10.1) can also be used to select the sampling method for VOC using the adsorption method.

With the help of psychrometry, it can also be checked whether the gas stream contains droplets and is therefore saturated and therefore an isokinetic sampling should be performed. If this method indicates that the gas stream is saturated, isokinetically sampled and no sampling in accordance with EN 14790 or absorption in sulphuric acid shall be carried out unless the water vapour content has to be further known for conversion from wet to dry gases. However, if the measurement indicates that the gas stream is not saturated, the measurement result is insufficient to decide that a non-isokinetic sampling may be carried out and a water determination according to EN 14790 or the absorption in sulphuric acid method will still be carried out, unless the absolute water vapour content is below 2 vol%.

For critical uses, the water vapour content shall be determined subsequently in accordance with EN 14790 or by absorption in sulphuric acid method or through a FTIR measurement when using the alternative methods, unless the water vapour content is less than 2 % vol.

Calculation of the water vapour content based on the measured oxygen content and knowledge of the chemical elemental fuel composition is also permitted for installations up to 10 MW falling under headings 31.1 (stationary engines and gas turbines) and 43 (combustion plants) of the classification list of Annex 1 of VLAREM II.

10.1 PSYCHROMETRY

This method uses two thermometers. One has a range from 0 to 150 °C, the other from 0 to 100 °C; both are readable to 0.2 °C. The bulb of the 0-100 °C thermometer is wrapped in a cloth that is kept moist with distilled water during the measurement.
The measurement shall be carried out in a pre-dedusted gas stream whose speed must be at least 3 m/s. The dry bulb thermometer is located upstream of the wet bulb thermometer in the gas stream.
It is necessary to heat all walls in contact with the extracted gas during sampling, from the suction aperture to the thermometers, to above the dew point. Partial water vapour pressure as a function of dry bulb temperature and the temperature difference between dry and wet bulb thermometers can be read from tables. With this partial water pressure, the water vapour content can be calculated.

10.2 Determination of water vapour content by condensation

Determination of the water vapour content by condensation is described in the standard NBN T 95-001 for volumetric flow rate. The extracted gas shall be cooled after dedusting in a cooler to a temperature below the dew point and not exceeding 20 °C. The amount of water vapour in the gas shall be determined as the sum of the amount of water vapour calculated from the amount of condensate collected and the water vapour content of the saturated gas at the outlet of the cooler. Determination of the amount of condensate is carried out volumetrically. However, at least 100 ml of condensate must be collected and measured at 1 ml.

10.3 Capacitive moisture probe

In a capacitive moisture probe, the effect of moisture on the dielectric constant of a polymer or metal oxide material is measured. When the water molecules are absorbed by the polymer, the capacitive value changes.

11 REFERENCES

EN 14790: 2017
Stationary source emissions-Determination of the water vapour in ducts – Standard reference method

NBN T 95-001: 1979 (withdrawn)
Determination of the volumetric flow rate of a gas in a tube by means of a Pitot tube

EN 15259: 2007
Air quality - Measurement of stationary source emissions - Requirements for measurement sections and sites and for the measurement objective, plan and report

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Essential quality requirements for emission measurements

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1 SCOPE

These quality requirements apply to measurements of emissions into the air taken by accredited 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 accredited laboratories. Their application is a precondition for accreditation and subject to verification at any time in accreditation 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:

2. Measurement site
3. Flow-rate measurement
4. Sampling
5. Method-specific requirements
    - Gas measurements with continuous monitors
    - Oxygen measurement
    - NO_2 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

2 MEASUREMENT SITE

2.1 MEASUREMENT SITE SUITABILITY

Verify the suitability of the measurement site using compendium procedure LUC/0/001 'Measurement site in the gas channel'.

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 stream < 15° with respect to the longitudinal axis of the gas channel;
- No local negative gas velocities;
- A minimum gas velocity depending on the measurement method for volumetric 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, particulate parameters and flow rate.

For installations where flow rate and dust/particulate parameters are not essential measurement quantities, 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).

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 measurement apertures;
- Site conditions;
- Protection from weather conditions;
- Any obstructions;
- Power supplies;

This preliminary inspection enables selection of the appropriate sampling equipment.

2.2 MEASUREMENT SITE EVALUATION IN THE REPORTING

2.2.1 General

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

- The results of the test against the 4 conditions of NBN EN 15259 for a homogeneous flow profile at each point in accordance with the standard, with the exception of the situations for which deviations are allowed under 2.2.2; in case of a non-homogeneous concentration profile in the measuring plane, flow-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 stream with respect to the channel axis for each measuring point; for angles under 15°, '< 15°' is sufficient.
  - Reporting the individual gas velocities for each measuring point and the vmax/vmin ratio
- The number of measurement apertures/axes/points present with respect to the requirements of NBN EN 15259. Under 2.2.2, the permitted deviations not giving rise to non-conformity are described.

With the exceptions of the deviations permitted under 2.2.2, 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 must be reported, with description of the deviations observed. For instance, in the event of a failure to meet the required number of measurement apertures/axes/points: not all required measurement apertures are present, a railing at the measurement apertures prevents full traversing at all points, measurement apertures 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 assumed 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 features and the verification of their conformity with the compendium (e.g. homogeneity determination).

2.2.2 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 measurement apertures/axes/points, which therefore do not result in a lack of compliance. The report must state which of these deviations from the norm 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 aperture instead of two measuring apertures available
    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 stream 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 streams with droplets.

- If only measuring gaseous parameters and flow rate in a homogeneous gas stream 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 streams with droplets.

- If only measuring gaseous parameters without a flow rate in a gas stream assumed to be homogeneous and without droplets, failure to meet the required number of measurement apertures/axes/points does not constitute a deviation. For non-water-soluble gaseous

parameters, this also applies to presumed homogeneous gas streams with droplets.

– If only measuring gaseous parameters and flow rate in a gas stream 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 measurement apertures/axes/points does not constitute a deviation. For non-water-soluble gaseous parameters, this also applies to presumed homogeneous gas streams with droplets.

2.3 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 has been demonstrated as per NBN EN 15259; a single-point measurement at each point in the section is then permitted;
- Or a single well-defined point demonstrated to be representative of the section as per NBN EN 15259;
- In the case of assumed homogeneity;
- For small gas-fired or gas oil-fired installations up to 5 MW, with the exception of atmospheric burners.

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

Assumed 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 $\leq 1 m^2$ (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 tube with a consistent shape and cross-section and a length of at least six times the hydraulic diameter D_h; it shall be chosen at two thirds of the length of the straight part measured from the starting point upstream.'

In the presence of droplets in the gas stream, assumed homogeneity never applies.

Suspected heterogeneity: If all the above conditions are not met, i.e. in cases of a less favourable section location, larger diameters (> 1.10 m) or multiple sources connected to the same channel, 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 channel, 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 O_2 (as a surrogate parameter).

For non-combustion processes where O_2 is not a suitable parameter for the homogeneity test and the gas stream 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

3 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.

3.1 PITOT TUBE CALIBRATION

Always calibrate all Pitot tubes before first using. All Pitot tubes used in the field must be calibrated regularly, at intervals depending on the frequency of use (one to five annually). 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 accredited for this purpose. 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.

3.2 PITOT TUBE INTEGRITY

A Pitot tube is an aerodynamic measuring instrument the proper response of which 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.

4 SAMPLING

4.1 GRID OR SINGLE-POINT MEASUREMENT?

4.1.1 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.

4.1.2 DUST-BORNE COMPONENTS OR MULTI-PHASE 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.
This therefore always applies to the sampling of dust, heavy metals, PAHs, dioxins and PCBs in emissions.

4.1.3 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 according to NBN EN 15259; in this case, any point can be chosen 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 assumed homogeneity (see point 2.3).
- 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, NH_3, 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); in this case, sampling is carried out on a time-proportional basis at the local rate at each point

4.2 LEAK TESTS

- The procedure must describe how the leak test is to be performed qualitatively; the duration of a leak test may be a crucial element;
- 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 each 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.

4.3 FIELD BLANKS

Table 1 displays the special requirements on field blanks. Because the absence of a field blank may result in challenges to the validity of a measurement, no exceptions to this table whatsoever are permitted. Always take and analyse a field blank and include it in the report.

4.3.1 FIELD BLANKS FOR MANUAL SAMPLING

Table 1: Requirements on field blanks

Norm	Frequency	Procedure[2]	Validation criterion?	Report
EN 1948-1 dioxins	See 4.3.2	As with sampling, ambient air for a minimum of 2 minutes throughout the complete sampling train	Yes, 10 % of emission limit	Yes, value of the field blank(s)
EN 13284-1 dust manual	Before or after each series[1] at least one/day	As with sampling, ambient air through the entire sampling train for at least 2minuten minutes	Yes, 10 % of the lowest emission limit for the process or 0.5 mg/Nm^3 (largest of both values)	Yes, value of the field blank(s)
EN 1911 Gaseous inorganic chlorides, expressed as HCl	for each set [1] at least one/day	As with sampling, ambient air for a minimum of 2 minutes throughout the complete sampling train	Yes, 10 % of emission limit	Yes, value of the field blank(s)
CEN/TS 13649 VOC through adsorption	At least 1/day For > 6stalen samples at least 2/day[3], For > 10stalen samples At least 3/day[3] Same batch as actual samples	As with sampling, ambient air for a minimum of 2 minutes throughout the complete sampling train	Yes, 10 % of emission limit	Yes, value of the field blank(s)
EN 13211 Hg in flue gas	1/campaign + each stack	As with sampling, ambient air for a minimum of 2 minutes throughout the complete sampling train	no	Yes, value of the field blank(s)
EN 14385 metals	1/campaign + each stack	As with sampling, ambient air for a minimum of 2 minutes throughout the complete sampling train	Yes, 10 % of emission limit	Yes, value of the field blank(s)

EN ISO 21877 NH_3	for each set [1] at least one/day	As with sampling, ambient air for a minimum of 2 minutes throughout the complete sampling train	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 (cartridge+desorption combination); the remaining
blanks can be filled in by analysing the backup section of actual samples.

4.3.2 FIELD BLANK FOR DIOXINS ACCORDING TO EN 1948-1, 2, 3

Due to the sampling complexity and high analysis cost for dioxin emissions, all accredited 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 three consecutive measurement days,
- on the measuring site itself (i.e. not at the bottom of the [...] stack if the sampling itself is carried out at height),
- 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.

5 METHOD-SPECIFIC REQUIREMENTS

5.1 GAS MEASUREMENTS WITH CONTINUOUS MONITORS

5.1.1 CALIBRATION AND INSPECTION OF GAS MONITORS

5.1.1.1 ON-SITE CALIBRATION AND RECORDING

- 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 on-site.
- The electronic data files must provide an audit trail for all calibrations (zero, span and control gases)

5.1.1.2 CALIBRATION GAS QUALITY

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

- A gas produced as certified reference material (CRM), with certificate issued under ISO 17034 accreditation (BELAC or equivalent);
- A gas with calibration certificate issued under an ISO 17025 accreditation (BELAC or equivalent) by a manufacturer accredited as a calibration body; or
- an ISO 17025 traceable calibration gas; in this case, the analysis by an accredited or recognised laboratory applies as the correct value. In the latter case, the requirements further described cf. 5.1.4 for self-certification of calibration gases should be followed
- 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 an accredited laboratory

Oxygen calibration gases

- For O_2, 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 % O_2 absolute). Accreditation 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 % O_2, the criterion in EN 14789 for the uncertainty of O_2 span gas of 2 % relative (k=2) is too high to meet the accreditation criterion (deviation $\leq$ 0.3 % absolute)

The permitted calibration uncertainty of the bottle for CO, CO_2, NO_x, SO_2 and propane may not exceed 2 %. This is in accordance with the EN standards, extending to SO_2 and CO_2.

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.

5.1.1.3 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 of another supplier.
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

5.1.1.4 MONITORING MAPS FOR FLUE GAS MEASUREMENTS WITH MONITORS

Make the control charts available on-site 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 six months.

When using set limits on control charts:

- The limits must be quantitatively justified.
- They must also undergo a periodic review (at least annually);

5.1.2 CONTROL OF LOSSES IN THE SAMPLING SYSTEM

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!)
- Supplying control gas through the entire sampling system. Specifically for SO_2 and NO_2, at a response time of more than 200 s during the provision of control gas, it is also permitted to offer the control gas directly to the monitor after calibration. Validation of losses throughout the sampling system and analyser should be carried out in this case for NO_2 according to EN 14792 10.1 and for SO_2 according to CEN/TS 17021 D.2.

Factor any sampling system losses into the total measurement uncertainty.

5.1.3 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 measured values	2 – 5%	2 – 5%
Rejection of the measurement	> 5%	> 5%

- Devices + data acquisition system must permit negative values (exception for electrochemical devices);

For oxygen, the criteria in EN 14789 Section 8.4.3 (identical with the table above) are far too broad and the following criteria apply:

	maximum drift, % O_2	
	Zero	**Span**
No correction	≤ 0.2%	≤ 0.2%
Correct	0.2 – 0.3 %	0.2 – 0.3 %
Reject	> 0.3%	> 0.3%

5.1.4 SELF CERTIFYING CALIBRATION GASES

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

Conditions for on-site 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 to be certified should be determined; how this is determined should also be set out in the procedure;
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 shall 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.

5.1.5 VALIDATION REQUIREMENTS OF MONITORS

5.1.5.1 VALIDATION PARAMETERS

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

- Reproducibility/intra-reproducibility, such as from control charts;
- Correctness, e.g. from ring test data;
- Working range;
- Limit of demonstrability and determination;
- Measurement uncertainty;

The European standards for continuous measurement of O_2, NO_x 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 flue gases and analysis of CO, CO_2, SO_2, NO_x, O_2

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.

5.1.5.2 MINIMUM REQUIREMENTS FOR EVALUATION OF THE LINEARITY OF MONITORS:

- Visually plotting the regression line so that deviations from the regression line (= residuals) become visible. The maximum permitted criterion for the linearity deviation of 2 % relative (all components except oxygen) should be applied to the maximum value of each measurement range in which in practice it is also measured effectively.
  For oxygen, the maximum permissible linearity deviation according to EN 14789 is 0.3 vol % oxygen.
- The linearity deviation is calculated as the difference between the measured value and the value obtained with the regression i.e. by entering the (generated) reference value into the regression equation.
- Minimum number of points: 5 + zero (after elimination of points, at least 5 points must remain);
- Do not force the regression line through zero;
- Measuring each concentration once will suffice;
- Linearity check frequency: see 5.1.5.3.

5.1.5.3 VALIDATION FREQUENCY

EN standards require an annual linearity test. For those participating in the LABs ring tests for flue gases, the Flemish Institute for Technological Research [VITO] permits a lower frequency of once every two 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 two years is sufficient.

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.

5.1.6 MEASUREMENT UNCERTAINTY ASSESSMENT

All accredited 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 proportional to the measuring range or constant (constant absolute error), while others are proportional to the measured concentration (constant relative error). The measurement uncertainty calculation must clearly distinguish these terms.

NB: The normative requirements apply. The non-normative 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 NO_x 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 NO_x and SO_2 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, the supplier must ask the supplier about the specifications of sensitivity factors according to EN standard (e.g. EN 15267-3 Table 1), which are necessary as a contribution to the measurement uncertainty. Preferably, use data determined by an independent institute.

5.1.7 MEASUREMENT RANGE AND MEASUREMENT UNCERTAINTY

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

5.2 OXYGEN MEASUREMENT

- Oxygen is subject to a stringent accuracy requirement of 0.3% absolute in Flanders because the oxygen measurement is used to convert measured 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 flue 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.

5.3 NO_2 MEASUREMENT

5.3.1 NO_2 MEASUREMENT BY CONVERTER AND NO MEASUREMENT:

- NO_2/NO converter efficiency:
  - Minimum of 95 % as per EN 14792 (NO_x measurement with the chemiluminescence monitor)
  - Criteria currently applicable as good practice:

	Converter efficiency
No correction	≥ 95%
Correction	80 – 95 %
Reject results	< 80%

- Converter efficiency: to be tested at the highest NO_2 concentrations that occur in practice. As a guideline for combustion process measurements, an NO_2 concentration of ±10 % of the highest NO_x measuring range or the emission limit will suffice for the test.
- Some converters appear to be less efficient at high NO_2 concentrations. When using a converter to measure processes with higher NO_2 concentrations, the efficiency must be demonstrated at the relevant NO_2 concentration.

5.3.2 Broader margins for drift NO_2

In cases of very small NO_2 contributions (up to the total NO_x), greater zero and span drifts may be permitted for NO_2 if the lab can demonstrate that this does not significantly increase the measurement uncertainty of NO_x, and that the measurement uncertainty remains below 10 % for measurements as per EN standards.

5.4 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 8 mm.
- Monitoring of the isokinetic sampling rate at each measuring point. For this purpose, note or log all required parameters every five 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 0.3 µm particles (99.5 % for 0.6 µm particles).
- 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).

5.5 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 balance 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 balance must feature a check weight and at least one set deviation criterion. If the value exceeds the criterion, appropriate action must be taken.
- 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 uses, in particular for conversion of concentrations to dry gases for emission limit testing or conversion of wet volumetric flow rates to dry gas flow rates, if mass flow rates must be calculated, unless the water content in the gas stream is less than 2 % by volume.

Calculation of the water content based on the measured oxygen content and knowledge of the chemical elemental fuel composition is also permitted for installations up to 10 MW falling under headings 31.1 (stationary engines and gas turbines) and 43 (combustion plants) of the classification list of Annex 1 of VLAREM II.

5.6 WET CHEMICAL SAMPLING OF INORGANIC GASEOUS CHLORIDES AND FLUORIDES, ETC.

- Relative expanded uncertainty of the gas volume meter:

Checks	Maximum permissible relative expanded uncertainty – when comparing the gas meter to be checked to the reference
Intake inspection = initial check on a 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 corrective actions taken with retroactive effect. 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 H_2O 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 SO_2 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 15 %.

- A continuous measuring device must be available for measuring SO_2. By participating in the ring tests, the reference laboratory ensures the equivalence of the device used in the ring tests with EN 14791 for SO_2 measurement and the approval of permanently installed measuring devices. For the latter however, the accuracy requirement of 15 % 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 inspection 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

5.7 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, with the maximum penetration being 5 % of the total amount. If the analytical concentration on the backup section is below the detection limit, it can also be said that there is no penetration.
- The selected extraction solvent must be of chromatographic quality, and must be free of interferences that co-eulate 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:
  - o Determine the desorption efficiencies once by validation. If the loading conditions change or another desorption fluid is used, a revalidation should be carried out; if the laboratory follows the compendium method, it should not validate this itself.
  - o 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 %.
  - o Further validation requirements when using compendium methods LUC/IV/001 to LUC/IV/012
    - Limits of detection and reporting
    - Trueness
    - 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)

6 REFERENCES

CEN/TS 13649: 2014
Stationary source emissions – Determination of the mass concentration of individual gaseous organic compounds – Sorptive sampling method followed by solvent extraction or thermal desorption

CEN/TS 17021: 2017
Stationary source emissions - Determination of the mass concentration of sulphur dioxide by instrumental techniques

Compendium for air sampling, measurement and analysis (LUC)
https://emis.vito.be/nl/erkende-laboratoria/lucht-gop/compendium-luc

Compendium for water sampling, measurement and analysis (WAC)
Method WAC/VI/A/001: Performance characteristics
https://emis.vito.be/nl/erkende-laboratoria/water-gop/compendium-wac

Compendium for the sampling, measurement and analysis of water (WAC)
Method WAC/VI/A/002: Measurement uncertainty
https://emis.vito.be/nl/erkende-laboratoria/water-gop/compendium-wac

EN 1911: 2010
Stationary source emissions – Determination of mass concentration of gaseous chlorides expressed as HCl – Standard reference method

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

EN 13211: 2001
Air quality – Stationary source emissions – Manual method of determination of the concentration of total mercury

EN 13284-1: 2017
Stationary source emissions – Determination of low range mass concentration of dust – Part 1: Manual gravimetric method

EN 14181: 2014
Stationary source emissions – Quality assurance of automated measuring systems

EN 14385: 2004
Stationary source emissions – Determination of the total emission of As, Cd, Cr, Co, Cu, Mn, Ni, Pb, Sb, Tl en V

EN 14789: 2017
Stationary source emissions – Determination of volume concentration of oxygen Standard Reference method – Paramagnetism

EN 14790: 2017
Stationary source emissions – Determination of the water vapour in ducts – Standard Reference method

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

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)

ISO 9096: 2017
Stationary source emissions – Manual determination of mass concentration of particulate matter

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

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

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

NBN EN ISO 21877: 2019
Stationary source emissions – Determination of the mass concentration of ammonia – Manual method (ISO 21877: 2019)

NF X 43-304: 2007
Emissions de sources fixes
Mesurage de la concentration en composés fluorés, exprimée en HF

ANNEX 1: PARAMETER PACKAGES ORGANIC COMPONENTS (EMISSION MEASUREMENTS) FOR APPLICATIONS FOR APPROVAL

Parameter package L.6 Emission measurements – sampling and analysis of volatile organic compounds (VOCs) – VOC basic package: aromatic hydrocarbons, paraffinic hydrocarbons, aliphatic halogenated hydrocarbons, esters, ketones, alcohols and ethers

aromatic hydrocarbons
Parameter
benzene
chlorobenzene
ethylbenzene
isopropenylbenzene
isopropylbenzene
styrene
toluene
trimethylbenzene (sum of 1,2,3-tmb, 1,2,4-tmb and 1,3,5-tmb)
xylenes (sum of o-xylene, m-xylene, p-xylene)

paraffinic hydrocarbons
Parameter
n-pentane
n-hexane
n-heptane
n-octane
n-nonane
n-decane

aliphatic halogenated hydrocarbons
Parameter
1,2-dibromoethane
1,1,2-trichloroethane
1,2-dichloroethane
tetrachloromethane
trichloromethane
1,1,1-trichloroethane
2-chloropropane
tetrachloroethylene
trichloroethylene
1,1-dichloroethylene
dichloromethane

esters
Parameter
ethyl acrylate
methyl acrylate
methyl acetate
vinyl acetate
butyl acetate (sum of iso-butyl acetate, n-butyl acetate, sec-butyl acetate, t-butyl acetate)
ethyl acetate

ketones
Parameter
2,6-dimethylheptane-4-one
cyclohexanone
methylcyclohexanone
2-butanone
4-methyl-2-pentanone
acetone

alcohols
Parameter
alkyl alcohol
ethanol
n-propanol
isopropanol
n-butanol
s-butanol
t-butanol
isobutanol
furfuryl alcohol

ethers
Parameter
1,4-dioxane
tetrahydrofuran
di-n-butyl ether
diethyl ether
di-isopropyl ether

Parameter package L.7 Emission measurements – sampling and analysis of organic compounds

L.7.1 olefinic hydrocarbons
Parameter
1-pentene
1-hexene
1-heptene
1-octene
1-nonene
1-decene

L.7.3 chlorobenzenes and chlorotoluenes
Parameter
chlorobenzene
1,2-dichlorobenzene
1,4-dichlorobenzene
α-chlorotoluene

Determination of the dust content in a ducted gas stream

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3 Validation 4

4 Determination of measurement uncertainty 4

5 References 5

1 SCOPE

For the determination of the dust content in a gas channel, the following standards apply:

- EN 13284-1: for dust content levels < 50 mg/Nm^3
- ISO 9096: for dust content levels of 20 mg/Nm^3 to 1000 mg/Nm^3

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

2 ADDITIONS TO AND FOCUS POINTS IN THE STANDARDS

- The measuring section and measuring plane must comply with LUC/0/001. This procedure also describes how the evaluation of the measuring site should be carried out.
- This must meet the requirements of standard EN 13284-1. EN 13284-1 takes precedence over ISO 9096 in terms of the number of measuring points, the monitoring of the isokinetic sampling rate and the requirement of a minimum nozzle diameter.
- Monitoring of the isokinetic sampling rate at each measuring point. For this purpose, note or log all required parameters every five 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 0.3 µm particles (99.9 % for 0.6 µm particles).
- The conditions for carrying out the leak test are described in the procedure 'Essential quality requirements for emission measurements' (LUC/0/005).
- The use of zero-pressure probes (equilibrated probes) is no longer compliant with EN 13284-1.
- The laboratory must always be able to condition the filter at 160° 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 value other than 160 °C (for dust types that exhibit specific thermal behaviour)
- The maximum permitted relative expanded uncertainty of the gas volume meter and of the temperature and pressure measurement at the level of the gas meter is included in procedure LUC/0/005.
- In the case of combination of dust with total metals according to NBN EN 14385, the filter must be placed outside the stack in heated filter housing at 160 °C if the flue gas temperature is below 160 °C and post-conditioning of the filter at 160 °C is applied for the determination of the dust content. This post-conditioning at 160 °C may cause volatilisation of metals that occur in dust form at the flue gas temperature. The temperature during the post-conditioning of the filter must never exceed the filtration temperature.
- EN 13284-1 states in Annex H that in case of the presence of volatile organic compounds (VOC), the temperature is conventionally taken to 160 °C so that there is volatilisation of the VOC and more reproducible results are obtained. Anything volatile at 160 °C shall not be measured as 'dust' according to EN 13284-1. If no specific conditions are imposed in the environmental permit and a lower conditioning and filter temperature (e.g. 105 °C for combination with dust-forming metals) is applied, the measurement is not legally valid in the event that the dust limit is exceeded at that lower temperature. In such a case, a new measurement for dust at 160 °C must be carried out.

3 VALIDATION

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

- Reproducibility/intra-reproducibility;
- Correctness, e.g. from ring test data;
- Working range;
- Limit of detection and determination
- 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.

For dust determination, a laboratory must have at least the reproducibility and accuracy of the filter weighing. The reproducibility and the limit of detection and determination of dust weighing must be determined for each type of filter by means of blank filters that run through the entire procedure from assembly/disassembly in the probe to conditioning and weighing.

4 DETERMINATION OF MEASUREMENT UNCERTAINTY

Each approved laboratory must have an evaluation of the measurement uncertainty, taking into account the contributions of sampling on the one hand and analysis (weighting here) 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.

5 REFERENCES

EN 13284-1: 2017
Stationary source emissions – Determination of low range mass concentration of dust – Part 1: Manual gravimetric method

ISO 9096: 2017
Stationary source emissions- Manual determination of mass concentration of particulate matter

EN 14385: 2004
Stationary source emissions – Determination of the total emission of As, Cd, Cr, Co, Cu, Mn, Ni, Pb, Sb, Tl and V

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

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

NBN ENV 13005: 2003
Guidance on the determination and indication of measurement uncertainty
Guide to the expression of uncertainty in measurement

Determination of the particulate fraction of metals in a ducted gas stream

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1 SCOPE

Some metals have an emission limit value in the Vlarem that applies to the 'dust form'. In this compendium method, 'particulate' metals are defined as the metals collected on the filter during filtration at a maximum temperature of 105 °C. This is based on the dust standard NBN X44-002, which was previously applicable when the Vlarem was established and which prescribed conditioning at (maximum) 105 °C. The procedure is valid for the following metals and their compounds: Sb, As, Cd, Cr, Co, Cu, Pb, Mn, Ni, Tl, V, Se, Sn and Hg.

2 PRINCIPLE

The principle of sampling consists of isokinetic aspiration of a subsample from a gas stream in a gas channel at a representative number of measuring points over a specified period of time, where the number of measuring points is determined by the dimensions of the gas channel. This number and the location of measuring points for circular and rectangular channels is specified in standard EN 13284-1.

The particulate metals are removed from the sampled gases by filtration in the stack at the flue gas temperature. However, if the flue gas temperature exceeds 105 °C, sampling must be carried out with an external filter at 105 °C. Drying of the filter is also carried out at a maximum temperature of 105 °C.

The particulate metals on the filter should be digested after sampling using the digestion method in accordance with standard EN 14385.

WAC/III/B includes the analysis methods for elements in water that may also be applied for the analysis of rendering fluids from filters/rinse from fluids.

The quality requirements for the analysis methods of WAC/VI/A/003 should be met. When analysing the rendering fluids of filters, a fly ash sample that undergoes the entire procedure including digestion should be included as inspection standard QC2. This can be a CRM, a real fly ash sample or a ring test sample.

3 EQUIPMENT

For sampling, the equipment should comply with the requirements of standards EN 14385 and EN 13211 (Hg). The use of impingers/wash bottles after the filter for sampling the gaseous fraction of metals is not applicable here.

4 QUALITY ASPECTS

- This must meet the requirements of standard EN 13284-1. EN 13284-1 takes precedence over ISO 9096 in terms of the number of measuring points, the monitoring of the isokinetic sampling rate and the requirement of a minimum nozzle diameter.
- Monitoring of the isokinetic sampling rate at each measuring point. For this purpose, note or log all required parameters every five 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 0.3 µm particles (99.9 % for 0.6 µm particles).
- The use of zero-pressure probes (equilibrated probes) is no longer compliant with EN 13284-1.
- The conditions for carrying out the leak test are described in the procedure 'Essential quality requirements for emission measurements' (LUC/0/005).
- A field blank must always be taken, analysed and reported. The procedure and the validation criterion for each standard are included in the procedure 'Essential quality requirements for emission measurements' (LUC/0/005).
- The maximum permitted relative expanded uncertainty of the gas volume meter and of the temperature and pressure measurement at the level of the gas meter is included in procedure LUC/0/005.

5 VALIDATION

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

- Reproducibility/intra-reproducibility, such as from control charts;
- Correctness, e.g. from ring test data;
- Working range;
- Limit of demonstrability and determination;
- 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.

6 DETERMINATION OF MEASUREMENT UNCERTAINTY

For the determination of metals in a gas channel, each approved laboratory must have an evaluation of the measurement uncertainty, taking into account the contributions of sampling on the one hand and 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.

7 REFERENCES

EN 14385: 2004
Stationary source emissions – Determination of the total emission of As, Cd, Cr, Co, Cu, Mn, Ni, Pb, Sb, Tl and V

EN 13211: 2001
Air quality – Stationary source emissions – Manual method of determination of the concentration of total mercury

EN 13284-1: 2017
Stationary source emissions – Determination of low range mass concentration of dust – Part 1: Manual gravimetric method

ISO 9096: 2017
Stationary source emissions- Manual determination of mass concentration of particulate matter

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/III/B
Methods for determining elements
https://emis.vito.be/nl/lne-erkenningen-water

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 water sampling, measurement and analysis (WAC)
Method WAC/VI/A/003
Quality requirements for analysis methods
https://emis.vito.be/nl/lne-erkenningen-water

ISO/IEC Guide 98-3: 2008 Uncertainty of measurement - part 3: Guide to the expression of uncertainty in measurement

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

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1 SCOPE

This procedure describes the sampling of O_2, CO, CO_2, SO_2, NOx and TOC in a gas stream 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.

2 SAMPLING

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

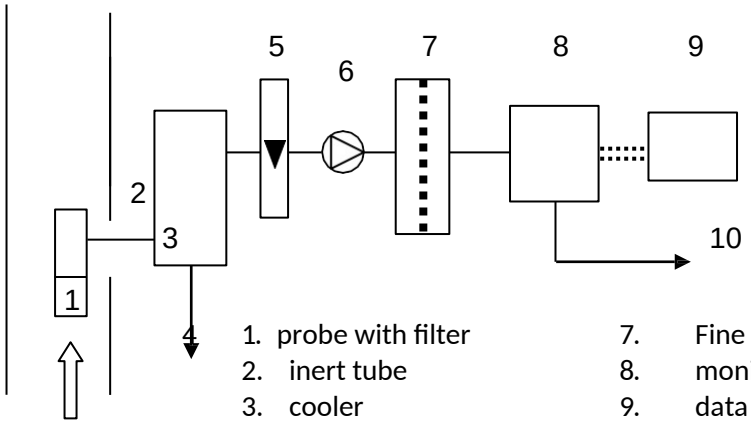
There are two options for a flue gas analysis with monitors: in the first case, the flue gas is measured using a portable setup in which the monitors are installed close to the measuring aperture (i.e. less than 3 m away); in the other case, the flue gas is preferably pumped via an overflow system to the monitors installed in a thermocouple car 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 channel, is possible under certain conditions. The procedure 'Essential quality requirements for emission measurements' (LUC/0/005) defines these conditions.

2.1 Sampling Setups

2.1.1 Monitors near the measuring point

Extract the flue 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 flue 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 measurement aperture, pump the cooled, dried and dust-free flue gas directly through the monitors via a rotameter and heated filter at a constant flow rate (see Figure 1).

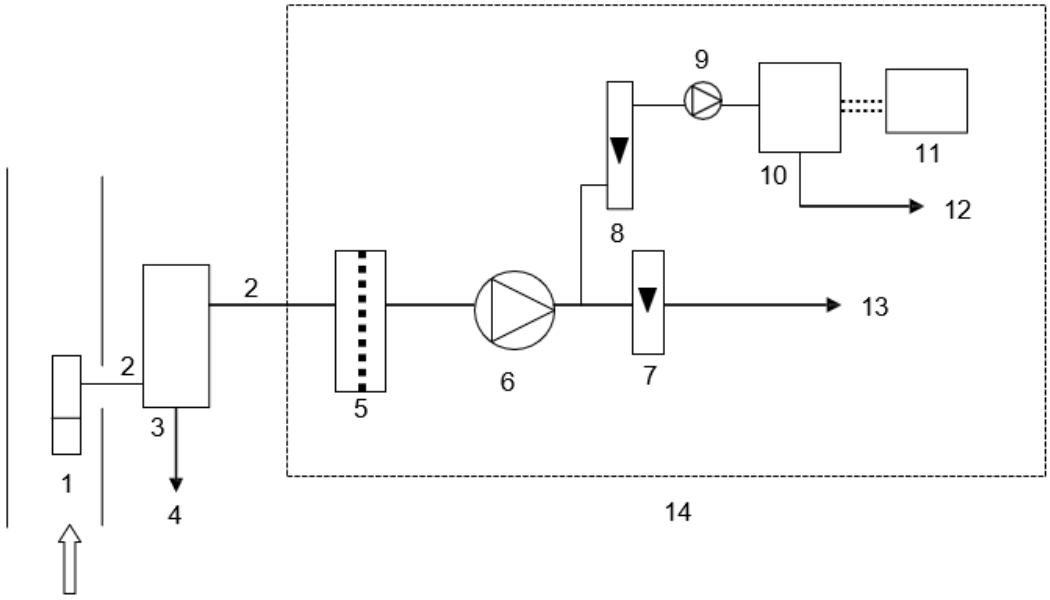


1. probe with filter
2. inert tube
3. cooler
4. condensate dissipation
5. rotameter
6. pump
7. Fine filter
8. monitor(s)
9. data logging
10. flue to ventilation

Figure 1: Flue gas sampling with monitors near the measuring point

2.1.2 MONITORS IN THERMOCOUPLE CAR

With a setup in a thermocouple car it is preferable to pump an excess of cooled and dried flue gas in an inert line through a rotameter to the measuring trolley. Next, use a separate pump to pump a partial stream of this excess flue gas at a constant flow rate through a heated filter and rotameter to the monitors (see Figure 2). 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.	inert tube	9.	pump
3.	cooler	10.	monitors
4.	condensate dissipation	11.	data logging
5.	fine filter	12 and 13	flue to ventilation
6.	pump	14	thermocouple car

Figure 2: Sampling of flue gases with monitors in a thermocouple car

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

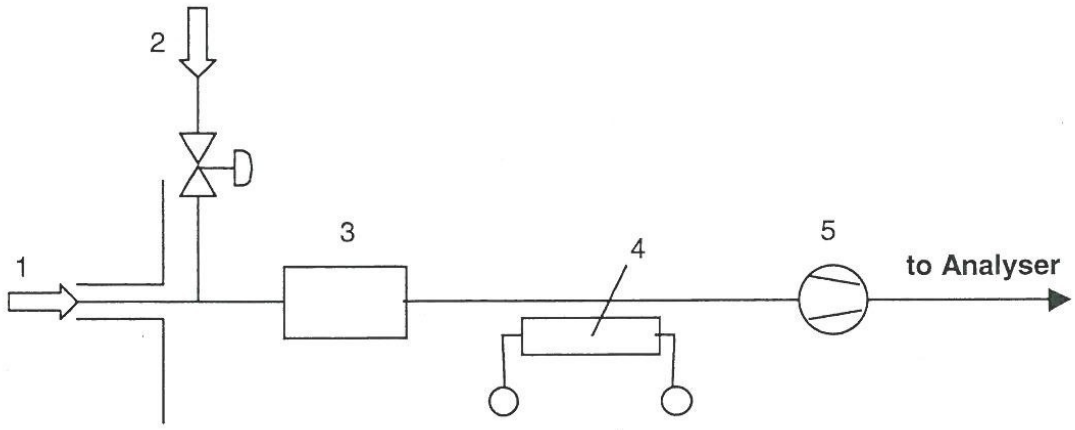
2.1.3 Special case of oxygen

Oxygen should be measured with high accuracy, due to its use as a reference for dilution in flue 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 SO_2 and NO_2.

2.1.4 Special case of KWS-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 duct, 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. A typical sampling setup with FID is displayed in Figure 3.



to Analyser to analyser

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

2.2 EQUIPMENT

The various components are described in Section 2.2.1 to Section 2.2.5. All connections between components must be made from inert materials and must be sufficiently leakproof.

2.2.1 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.

2.2.2 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 stream and preferably install it outside the stack, to enable better temperature inspection.

2.2.3 GAS CONDITIONING SYSTEM

For O_2 and CO, the EN standards prescribe a cooler or permeation dryer for the gas conditioning system (2.2.3.1 and 2.2.3.2). For NO_x, in addition to these two configurations, the standards also include a dilution system (2.2.3.3) and a heated line up to the heated monitor. If the NO_2/NOx ratio is more than 25 %, the configuration with cooler should be avoided unless it is demonstrated that the losses of NO_2 in the cooler are not significant.
For SO_2, 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.

2.2.3.1 COOLER

The cooler must be designed to minimise absorption of water-soluble components such as NO_2 or SO_2 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.

2.2.3.2 PERMEATION DRYER

Permeation dryers diffuse water through a semi-permeable membrane from the humid flue 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.

2.2.3.3 DILUTION SYSTEM

Dilution systems as described in ISO 10396 are used to cool the flue 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.

2.2.4 SECONDARY FILTER

The secondary filter or particulate filter purifies the flue 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.

2.2.5 PUMP AND FLOW WATER

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 flue 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.

2.3 IMPLEMENTATION

Install the cooler (if applicable) as close as possible to the measurement aperture. Install the probe, the filter, the intake line to the cooler and the duct 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. After checking the functioning of the cooler, insert the probe into the flue, seal it tight and start the measurement.

2.4 POINTS OF ATTENTION

2.4.1 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.

2.4.2 COOLER TEMPERATURE TEST

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.

2.4.3 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, over longer distances, the sample gas should only be

transported in heated tubes and in cold tubes 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 m), to reduce losses of heavier components in the duct. Replace any lines that exhibit visible signs of deposits.

2.4.4 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 two minutes.

2.4.5 STABLE EXTRACTION

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 %.

2.4.6 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 rinse from of the cooler and the filters to prevent corrosion, the main pump continues to draw ambient air for about 15 minutes after each measurement.

3 FLUE GAS ANALYSIS

The table below gives the reference and other permitted measurement methods for the various components: CO, NO_x, SO_2, O_2 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*)	FTIR
NOx	EN 14792 (NO: Chemiluminescence NO_2: Chemiluminescence using converter)	NO/NO_2 NDUV NO-NDIR+NO_2 to NO converter** FTIR
SO_2	EN 14791 (Wet chemical measurement method)	Measurement with monitors with the following measurement principles (included in CEN/TS 17021)*** NDUV NDIR

		UV fluorescence FTIR
O_2	EN 14789 (paramagnetism)	
TOC	EN 12619 (TVOC concentrations up to 1000 mg/m^3) Flame ionisation detection (FID)	

* Gas filter correlation photometer
** NO_2 NDIR is not permitted due to water interference
*** If it is necessary to measure SO_2 in a gas stream 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 SO_2 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 15 %.

A continuous measuring device must be available for measuring SO_2. By participating in the ring tests, the reference laboratory ensures the equivalence of the device used in the ring tests with EN 14791 for SO_2 measurement and the approval of permanently installed measuring devices. For the latter however, the accuracy requirement of 15 % applies (recognition condition).
For combustion plants of up to 10 MW, the use of electrochemical cells to measure CO, CO_2, NOx, SO_2 and O_2 is also permitted.

At a minimum, all analysers must have a certificate in accordance with EN 15267-3 (EN 15267-3:2007 or more recent) for AMS or a certificate in accordance with EN 15267-4 (2017 or more recent) for P-AMS (Portable Automated Measuring System) if these analysers are available and, ideally, certification in accordance with EN 15267-4 for P-AMS. Analysers purchased after this method came into effect must comply with these requirements.
A certification according to EN 15267-4 for P-AMS is always required for FTIR as the equivalence as AM with respect to the SRMs must be proven.

If an emission limit for SOx (as SO_2) applies for the installation being measured and the presence of SO_3 may also be assumed, then in addition to the continuous determination of SO_2, also measure SO_3 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 SO_2 and SO_3.
Carbon dioxide (CO_2) 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. CO_2 is measured under the same measurement principle as CO (NDIR), and both parameters are typically combined in a single device.

3.1 IMPLEMENTATION

The complete procedure for continuous measurement with a monitor consists of the following steps:
- Conduct a leak test on the entire sampling system;
- calibration of the monitor with zerogas and span gas (in this order) with adjustment, the gases being presented directly to the monitor;
- first-line inspection with an independent control gas (see Section 0); with each measurement,

send the control gas through the entire sampling train;
- measurement;
- 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 O_2 measured value with a direct nitrogen supply to the monitor;
- the O_2 measured 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 O_2 concentration in the zero gas is less than 0.05 vol %.
For the span gas, use CO, CO_2, NO, SO_2 in N_2 or a mixture of these components in N_2. 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 flue 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.

3.1.1 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.

3.1.2 CHECKS

For each measurement, send the control gas through the entire sampling system. Specifically for SO_2 and NO_2, at a response time of more than 200 s during the provision of control gas, it is also permitted to offer the control gas directly to the monitor after calibration. Validation of losses throughout the sampling system and analyser should be carried out in this case for NO_2 according to EN 14792 10.1 and for SO_2 according to CEN/TS 17021 D.2.
Make the control charts available on-site 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 six months.

When using set limits on control charts:
- They must also undergo a periodic review (at least annually);
- The limits must be quantitatively justified.

3.1.3 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.

3.1.4 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. The criteria permitted for drift have been included under Section 3.4.1. The span drift is determined on the monitors themselves.

3.2 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
- an ISO 17025 traceable calibration gas; in this case, the analysis by an accredited or recognised laboratory applies as the correct value. In the latter case, the requirements described for self-certification of calibration gases are to be followed; these requirements are included in the compendium procedure 'Essential quality requirements for emission measurements' (LUC/0/005);
- 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, CO_2, NO_x, SO_2 and propane may not exceed 2 %. This is in accordance with the EN standards, extending to SO_2 and CO_2.

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

For O_2, 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 flue 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 % O_2 absolute). Accreditation 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 % O_2, the criterion in EN 14789 for the uncertainty of O_2 span gas of 2 % relative (k=2) is too high to meet the accreditation criterion (deviation ≤ 0.3 % absolute)

3.3 INDEPENDENT CONTROL GAS

After the calibration of the gas measuring devices, supply an independent control gas for all components, see Section 3.1.2. 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

3.4 CALCULATIONS

3.4.1 CORRECTIONS FOR DRIFT

The signal from the monitor may be subject to drift (see Figure 4). 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). Therefore, calibrate the monitor beforehand and recalibrate it afterwards (without adjustment).

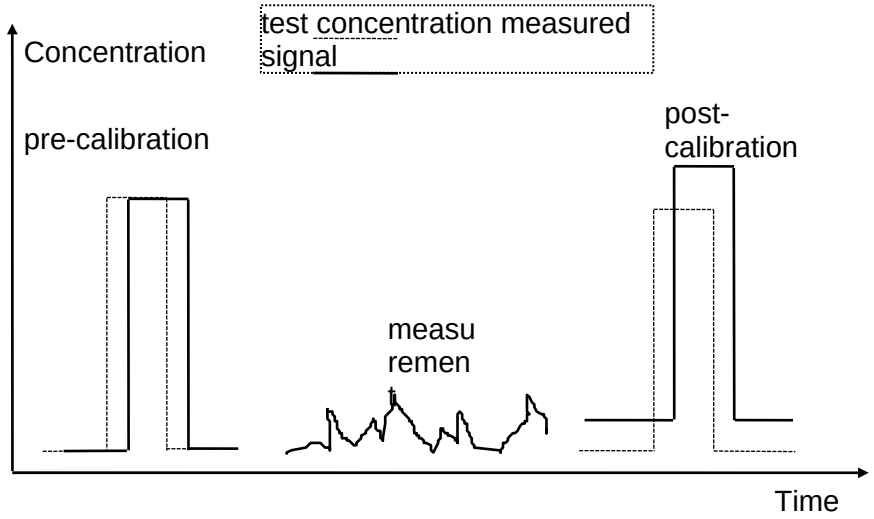


Figure 4: Drift of the monitor

Depending on the measured 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 measured values	2 – 5 %	2 – 5 %
Rejection of the measurement	> 5%	> 5%

For oxygen, the criteria in EN 14789 Section 8.4.3 (identical with the table above) are too broad and the following criteria apply:

	maximum drift, % O_2 absolute	
	Zero	Span
No correction	≤ 0.2%	≤ 0.2%
Correct	0.2 – 0.3 %	0.2 – 0.3 %
Reject	> 0.3%	> 0.3%

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

In cases of very small NO_2 contributions (up to the total NO_x), greater zero and span drifts may be permitted for NO_2 if the lab can demonstrate that this does not significantly increase the measurement uncertainty of NO_x, and that the measurement uncertainty remains below 10 % for measurements as per EN standards.

3.4.2 CONVERSIONS

The CO_2 measurement result is usually expressed in % on the monitor, and must also often be reported as such.

Typically, concentrations of CO, NO_x and SO_2 should be expressed for 273.15 K and 1013.25 hPa. The customary unit is mg/Nm³. The following formulas may be used to convert from ppm to mg/Nm³ for the different components:

$$CO\left(\frac{mg}{Nm^3}\right)=\frac{CO(ppm)\times 28}{22.4}$$

$$SO_2\left(\frac{mg}{Nm^3}\right)=\frac{SO_2(ppm)\times 64}{22.4}$$

$$NO\left(\frac{mg}{Nm^3}\right)=\frac{NO(ppm)\times 30}{22.4}$$

$$NO_2\left(\frac{mg}{Nm^3}\right)=\frac{NO_2(ppm)\times 46}{22.4}$$

$$No\ as\ NO_2\left(\frac{mg}{Nm^3}\right)=NO\left(\frac{mg}{Nm^3}\right)\times\frac{46}{30}$$

'NO_x' is defined as the sum of nitrogen oxide (NO) and nitrogen dioxide (NO_2), expressed as NO_2:

$$NO_x \text{ as } NO_2\left(\frac{mg}{Nm^3}\right) = NO \text{ if } NO_2\left(\frac{mg}{Nm^3}\right) + NO_2\left(\frac{mg}{Nm^3}\right)$$

with
22.4 l/mol: molar volume of the ideal gas at 273.15 K and 1013.25 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 1013.25 hPa. The customary unit is mg C/Nm^3 in propane equivalents:

$$\text{measured value}\left(mg\frac{C}{Nm^3}\right) = \frac{\text{measured value}(¿\,ppm) \times n_c \times MM_c}{V_m}$$

With
n_C: number of carbon atoms in the calibration gas (for propane, n_C=3) MM_C: molar mass of carbon (MM_C = 12 g/mol)
V_m: molar volume of the calibration gas at 273.15 K and 1013.25 hPa (V_m = 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 prevailing pressure there. This residual moisture content (± 0,8 vol %) can be calculated using the following formula:

$$\%H_2O = \frac{P_w(hPa) \times 100}{Pbaro(hPa)}$$

With % H_2O: residual moisture content in the gas after the cooler, in vol%
p_w: the vapour pressure of water at the temperature in the cooler,
P_{baro}: in hPa
the atmospheric pressure in hPa

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:

$$\log[P_w(hPa)] = 8.19621 - \frac{1730.63}{233.426 + t(℃)}$$

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

$$\text{Concentration}\left(\frac{mg}{Nm^3\,dr}\right) = \text{Concentration}\left(\frac{mg}{Nm^3\,dr}\right) \times \frac{100}{100 - \%H_2O}$$

where
Concentration (mg/Nm^{3}dr): concentration in per normal cubic metre of dry gas
Concentration (mg/Nm ^{3}): concentration in per normal cubic metre of wet gas
Normal cubic meter: volume of gas, reduced to normalised temperature (273,15 K) and pressure (1013.25 hPa)
%H_2O: the residual moisture content in the gas after the cooler, in vol%

If the water content determination shows that water was not present in the gas stream, do not apply this correction.
Also multiply the %CO_2 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 cocombustion 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:

$$Concentration_R = \frac{21 - ¿}{21 - OM} \times Concentration_M$$

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

with

= concentration at reference oxygen content in mg/Nm dr^3

= measured concentration in mg/Nm dr^3

OR = reference oxygen content in %

OM = 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 of attention

Avoid checking emission limits below 5% of the measuring range of a measuring device. Because a number of contributions to measurement uncertainty are proportional 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 measured 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 SO_2 in a gas stream that also contains methane, methane interference must not occur on the monitor.

4 VALIDATION

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

- Reproducibility/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 O_2, NO_x 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 flue gases, the Flemish Institute for Technological Research [VITO] permits a lower frequency of once every two 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 two years is sufficient.

The following minimum requirements apply to evaluation of monitor linearity:

- Visually plotting the regression line so that deviations from the regression line (residuals) become visible*;
- Minimum number of points: 5 + zero (after elimination of points, at least 5 points must remain);
- Do not force the regression line through zero;
- Measuring each concentration once will suffice;

*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. This regression line is represented graphically. 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 may not exceed 2 % of the maximum value of each measuring range in which in practice is also measured effectively. For oxygen, the maximum permissible linearity deviation according to EN 14789 is 0.3 vol % oxygen.

Thus:

$$\frac{y-y}{MB} = \times 100\,\% \leq 2\,\% \left(¿ \leq 0.3\, vol\,\% O_2 \right)$$

Where *y*= measured concentration

$\hat{}$= concentrations recalculated with the calibration curve (obtained by plugging the reference concentrations x into the calibration curve equation)
MB= each measuring range where in practice it is also effectively measured

Next, plot the residues $-\hat{}$ against the concentrations created. Visual inspection of the residues can recognise the inearity deviation from a clear pattern in the residues.

For the TOC measurement with an FID, also validate the relative response factors (RRFs) and oxygen synergism. 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 NO_2/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 – 95 %
Reject results	< 80%

Test the converter efficiency at the highest NO_2 concentrations that occur in practice. As a guideline for combustion process measurements, an NO_2 concentration of ±10 % of the highest NO_x measuring range or the emission limit will suffice for the test. Some converters appear to be less efficient at high NO_2 concentrations. When using a converter to measure processes with higher NO_2 concentrations, the efficiency must be demonstrated at the relevant NO_2 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.

5 DETERMINATION OF MEASUREMENT UNCERTAINTY

All accredited 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 proportional to the measuring range or constant (constant absolute error), while others are proportional 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 NO_x and EN 15058 for CO.

NB: The normative requirements apply. The non-normative 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 duct and conditioning system, loss/conversion at the filter;
- Residual water content (if not corrected);
- Uncorrected zero and span drifts;
- Inearity deviation ('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 NO_2 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, the supplier must ask the supplier about the specifications of sensitivity factors according to EN standard (e.g. EN 15267-3 Table 1), which are necessary as a contribution to the measurement uncertainty. Preferably, use data determined by an independent institute.

Also include these factors in the measurement uncertainty when using other monitors (e.g. SO_2 and FID) and in measurements on other installations, such as process plants, medium-sized combustion plants or post-combustion plants.

6 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: 2017
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Determination of mass concentrations of multiple gaseous components in a ducted gas stream using FTIR

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1 SCOPE

This procedure describes the sampling of CO, CO_2, SO_2, NOx, N_2O, NH_3, H_2O, CH_4 in a ducted gas stream with an FTIR analyser. The following technical specification applies except in cases where other or additional requirements are stipulated in this compendium procedure:

- CEN/TS 17337: 2019 Stationary source emissions - Determination of mass concentration of multiple gaseous species - Fourier transform infrared spectroscopy

Technical Specification CEN/TS 17337 describes a method for sampling and determining the concentration of gaseous emissions to the atmosphere from tubes and stacks by extractive FTIR spectroscopy. The specific components and requirements for the measurement system are described in the technical specification. A number of performance characteristics with corresponding minimum performance criteria are specified for the measurement system. These performance characteristics and the expanded uncertainty of the method shall comply with the specifications specified in the document.

This compendium procedure provides a non-exhaustive overview of a number of important aspects from the aforementioned technical specification.

The FTIR can be used for industrial processes that are representative of the scope of the standard reference method (SRM) or alternative method (AM). Application with other processes is possible if absence of interference is demonstrated.

If dust particles are present in the flue gas that release more gas at high temperatures than at the lower temperatures used in the reference measurement methods (e.g. max 105 °C for gaseous NH_3, 120 °C for formaldehyde, ...)[11], then FTIR should not be used as an alternative measurement method.

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.

2 ADDITIONS TO AND FOCUS POINTS REGARDING THE TECHNICAL SPECIFICATION

- The measuring section and measuring plane must comply with LUC/0/001. This procedure also describes how the evaluation of the measuring site should be carried out.
- The measurement strategy to be applied (e.g. 1-point measurement versus traversal and isokinetic or non-isokinetic sampling) is described in the procedure 'Essential quality requirements for emission measurements' (LUC/0/005).
- Purification of the FTIR interferometer with nitrogen during practical measurements is to be carried out in accordance with the instructions from the manufacturer of the device.

[1]This can be demonstrated by performing the following dust measurement:

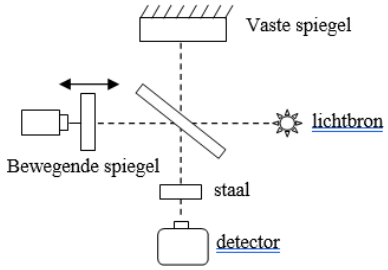
- The filter is first preconditioned at a temperature of 180 °C (i.e. the temperature at which an FTIR operates) and then pre-weighed;
- After sampling, the filter is conditioned at the temperature of the filter prescribed in the LUC method during sampling (e.g. 105 °C for gaseous NH_3 and 120 °C for gaseous formaldehyde) and then weighed;
- The filter is then reheated to 180 °C and weighed;
- If there is a significant difference (decrease) between the two weight measurements, the dust is likely to release the gas to be measured and the FTIR may not be used;

- It is important to periodically calibrate the FTIR for water vapour in accordance with the manufacturer instructions as this is the most common interference.
- For the inclusion of a spectrum, a binary mixture of the component in nitrogen (dry gas) is to be presented to the FTIR.

3 MEASUREMENT PRINCIPLE

FTIR is a technique in which IR electromagnetic radiation interacts with the test gas. Polychromatic light covering the IR area is passed through the sample cell. Each component present absorbs the radiation at a range of characteristic wavelengths that enable identification, while the degree of attenuation (absorption) of light provides concentration information as described by the Beer-Lambert law. FTIR is capable of measuring a wide range of components, with the exception of symmetrical molecules that do not show dipole change with vibration (e.g. oxygen and chlorine).

The output signal for fourier spectroscopy is an interferogram.



Vaste spiegel	Fixed mirror
Bewegende spiegel	Moving mirror
Lichtbron	Light source
Staal	Sample
detector	detector

Figure 1: Diagram of an interferometer

Light is emitted from an IR source and passes through a unit that splits the rays. One part of the light goes to the fixed mirror, the other goes to a moving mirror. The reflected light rays are recombined, resulting in constructive or destructive interference. The intensity measured by the detector as a function of the displacement of the moving mirror is called an interferogram. This signal contains information about the intensity of each IR wavelength, and this is converted into a spectrum using a mathematical transformation. A FTIR spectrum contains specific information on the chemical structure of organic and inorganic components based on the unique vibrational modes of the different chemical bonds. Each vibrational mode absorbs a specific wavelength of the IR radiation. Each bond in a molecule can have different vibrational modes so that the FTIR absorption spectrum serves as a fingerprint for a particular molecule and can be compared with reference spectra of known components.

4 REQUIREMENTS FOR USING AN FTIR AS AN ALTERNATIVE METHOD (AM) TO A STANDARD REFERENCE METHOD (SRM)

Table 1 provides an overview of the reference methods and other permitted measurement methods for a number of components within the Compendium Air.

When FTIR is used as an alternative method (AM) to the standard reference method (SRM), the performance characteristics from standard EN 15267-4 (see Chapter 6 in this procedure) applicable to P-AMS shall be determined in a general performance test according to the test procedures described in EN 15267-4. This includes the demonstration of equivalence to the SRM according to EN 14793 carried out by an independent testing laboratory accredited or recognised by the competent authorities for the implementation of test procedures of EN 15267-4 (e.g. TüV). The device must therefore have an EN 15267-4 certificate.

The present compendium procedure applies to measurements with an FTIR analyser.

Table 1: Reference and authorised alternative measurement methods for a number of parameters

Component	**Reference method (SRM) and measurement principle LUC method**	**Alternative method (AM) allowed within the LUC**
CO	EN 15058 (NDIR/GFC*) LUC/II/001	FTIR
NOx	EN 14792 (NO: Chemiluminescence NO_2: Chemiluminescence using converter) LUC/II/001	NO/NO_2 NDUV NO-NDIR+NO_2 to NO converter** FTIR
SO_2	EN 14791 (Wet chemical measurement method) LUC/III/008	Measurement with monitors with the following measurement principles (included in CEN/TS 17021)*** LUC/II/001 NDUV NDIR UV fluorescence FTIR
O_2	EN 14789 (paramagnetism) LUC/II/001	
TOC	EN 12619 (TVOC concentrations up to 1000 mg/m^3) Flame ionisation detection (FID)	
	LUC/II/001	
NH_3	EN ISO 21877 (wet chemical measurement method) LUC/III/003	FTIR
H_2O	EN 14790 (adsorption or condensation + adsorption) LUC/0/003	FTIR
N_2O	EN ISO 21258 NDIR	FTIR
Formaldehyde	(Wet chemical measurement method)	FTIR

	Based on EPA method 316 LUC/III/004	
Methane (CH_4)	EN ISO 25139 (sampling in gas bag/cylinder + analysis via GC-FID)	EN ISO 25140 (FID) FTIR

* Gas filter correlation photometer

** NO_2-NDIR is not permitted due to water interference

*** If it is necessary to measure SO_2 in a gas stream 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.

NB: The wet chemical SO_2 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 15 %.

A continuous measuring device must be available for measuring SO_2. By participating in the ring tests, the reference laboratory ensures the equivalence of the device used in the ring tests with EN 14791 for SO_2 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, CO_2, NOx, SO_2 and O_2 is also permitted.

At a minimum, all analysers must have a certificate in accordance with EN 15267-3 (EN 15267-3:2007 or more recent) for AMS or a certificate in accordance with EN 15267-4 (2017 or more recent) for P-AMS (Portable Automated Measuring System) if these analysers are available and, ideally, certification in accordance with EN 15267-4 for P-AMS. Analysers purchased after this method came into effect must comply with these requirements.

Certification according to EN 15267-4 for P-AMS is always required for FTIR
when equivalence as AM with respect to SRMs must be proven.

If an emission limit for SOx (as SO_2) applies for the installation being measured and the presence of SO_3 may also be assumed, then in addition to the continuous determination of SO_2, also measure SO_3 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 SO_2 and SO_3.

Carbon dioxide (CO_2) 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. CO_2 is measured under the same measurement principle as CO (NDIR), and both parameters are typically combined in a single device. CO_2 can also be measured with FTIR.

5 SAMPLING

The sampling principle consists of extracting a subsample from a gas stream in a gas channel. This gas stream may have a high temperature and may contain water and dust particles.

The sampling system shall consist of:

- Sampling probe
- Filter
- Sampling line

- Pump (can be part of the FTIR analyser)

Although the system is designed for use at elevated temperatures and is suitable for wet samples, this does not preclude the use of conditioning units. If a conditioning unit is used, the entire system, including the conditioning unit, must meet the performance requirements as described in CEN/TS 17337.

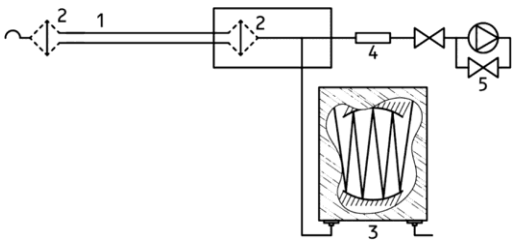
If a hot/wet system is used, the probe filter box and sample gas duct are generally heated to a similar temperature as the sample cell. In addition, the selected temperature must be at least 180 °C. If this is contradictory to the temperature used to demonstrate the conformity of the P-AMS with EN 15267-4, the temperature used in the test according to EN 15267-4 is to be used instead.

All components of the sampling system located in front of the FTIR analyser must be made of materials that do not react with or absorb the components.

The temperature and pressure in the FTIR sample cell are important parameters as they directly influence the measured concentrations. In addition, these parameters are important for preventing condensation for the sample in the gas cell. On the other hand, the flow rate through the gas cell does not affect the measured concentration if all other parameters remain unchanged.

Sampling using a side stream configuration may be necessary if the required suction flow rate exceeds the upper limits specified for the FTIR analyser. An example configuration for this kind of sampling is given in Figure 2. This is Figure A.1 from CEN/TS 17337, but with an integrated pump instead of a pump after the FTIR. The configuration with a pump after the FTIR, as shown in Figure A.1 of CEN/TS 17337, would cause incorrect measurements by creating a negative pressure in the cell.

If the FTIR does not have an integrated pump, a heated sampling system (pump + filter) should be provided between the probe and FTIR analyser; the tubes from the probe to the sampling system and from sampling system to FTIR analyser should also be heated.



1 heated sampling probe
2 heated particle filters (alternatives)
3 FTIR analyser with integrated pump
4 cartridge with desiccant (optional)
5 pump

Figure 2: Sampling with side stream configuration according to CEN/TS 17337, with FTIR with integrated pump

5.1 EQUIPMENT

The different components are described in 5.1.1 to 5.1.5. All connections between components must be made from inert materials and must be sufficiently leakproof.

5.1.1 PROBE

The probe must be chemically and physically inert for the components to be analysed and must not absorb any components.
Adapt the length and/or diameter of the probe to the size of the stack to enable sampling at all measuring points.

5.1.2 FILTER

A filter of inert material is to be placed after or as part of the sampling probe and must have a suitable pore size to prevent particles from entering the rest of the system. The filter must be replaced or cleaned periodically, depending on the dust load at the site. If the filter is located outside the stack, it must be heated to at least 180 °C. A pore size of 3 µm is sufficient for many locations.

5.1.3 SAMPLING LINE

In order to reduce the response time of the system, the sample gas duct should be as short as possible. The tube must be made of inert material that is resistant to absorption of the components. The tube must be heated to at least 180 °C.

5.1.4 PUMP

The sample pump must be capable of operating according to all specified stream requirements and pressure conditions required for the sample cell. The pump must be resistant to corrosion. An external pump must be consistent with the analyser to which it is connected.

5.1.5 OXYGEN SENSOR (OPTIONAL)

Some FTIR systems contain an oxygen sensor to enable measurements and automatic corrections to oxygen reference conditions. This kind of sensor must comply with standard EN 14789 and be operated in accordance with it. Oxygen should be measured with high accuracy, due to its use as a reference for dilution in flue gases. For combustion plants up to 10 MW, the use of electrochemical cells (e.g. zirconium sensor) for measuring O_2 is also permitted. Built-in oxygen sensors must also be certified.

6 PERFORMANCE CHARACTERISTICS OF THE METHOD

For measurements within a legal framework, the user must be able to demonstrate that the FTIR has a certification according to EN 15267-4. These tests are described in Sections 6.1 and 6.2.

6.1 MEASURED COMPONENTS THAT FALL UNDER AN SRM

Table 2 shows the performance characteristics applicable to P-AMS. If FTIR is used as an alternative method (AM) to a standard reference method (SRM), these performance characteristics must be

determined in a general performance test according to the test procedures described in EN 15267-4. These procedures must include the demonstration of equivalence to the SRM in accordance with EN 14793, carried out by an independent testing laboratory accredited or recognised by the competent authorities for the implementation of test procedures of EN 15267-4 (e.g. TüV).

The independent test laboratory is to check the conformity of the P-AMS with the performance criterion associated with each performance characteristic. The maximum permissible deviations as absolute values of the measured values are given as mass concentrations or as percentages of the upper limit of the range.

EN 15267-4 also requires performance tests on at least five different process types and cross-interference tests on at least the components listed in Annex B of EN 15267-4, as well as all other components known to be present in one of the five processes.

The user should also be aware that if the P-AMS (FTIR) is used outside its certification range, i.e. in a process other than the five different processes with different emission matrices included in the performance tests, there is a risk that the measurement results will have a bias due to uncharacterised cross-interference effects. The spectral residue test (see 7.3.5.2) may be used as an indication of the absence of uncharacterised cross-interference.

Table 2: EN 15267-4 Performance criteria for P-AMS during laboratory tests

Performance characteristic	**Performance criterion**		**Reference in EN 15267-4**
	Gases other than O_2	**O_2**	
Response time	≤ 400 s for Hg, NH_3, HCl and HF ≤ 200 s for other components	≤ 200 s	10.6
Repeatability standard deviation at zero	≤ 2.0 % [a]	≤ 0.20 % [b]	10.7
Repeatability standard deviation at the span point	≤ 2.0 % [a]	≤ 0.20 % [b]	10.8
Linearity deviation	≤ 2.0 % [a]	≤ 0.30 % [b]	10.9
Short-term zero-point drift	≤ 2.0 % [a]	≤ 0.20 % [b]	10.10
Short-term span drift	≤ 2.0 % [a]	≤ 0.20 % [b]	10.10
Effect of the change of ambient temperature from 5 °C to 25 °C and from 40 °C to 20 °C at zero	≤ 5.0 % [a]	≤ 0.50 % [b]	10.11
Effect of the change of ambient temperature from 5 °C to 25 °C and from 40 °C to 20 °C at the span point	≤ 5.0 % [a]	≤ 0.50 % [b]	10.11
Effect of the supply voltage, at -15% below and at +10% above the rated supply voltage[c]	≤ 2.0 % [a]	≤ 0.20 % [b]	10.12
Effect of vibrations	≤ 2.0 % [a]	≤ 0.20 % [b]	10.13
Effect of the pressure of the steel gas at the span point, for a pressure change Δp of 3 kPa	≤ 2.0 % [a]	≤ 0.20 % [b]	10.14

Effect of gas flow rate on extractive P-AMS for a given specification by manufacturer	≤ 2.0 % [a]	≤ 0.20 % [b]	10.15
Cross sensitivity CEN/TS 17337 see Table B.1 of EN 15267-4	≤ 4.0 % [a]	≤ 0.40 % [b]	10.16
Converter efficiency for P-AMS measurement of NOx	≥ 95.0%	-	10.17
Converter efficiency for P-AMS measurement of Hg	≥ 95.0%	-	10.18
a Percentage value as a percentage of the upper limit of the certification range. b Percentage value as oxygen volume concentration (volume fraction). c If the manufacturer specifies other limits for battery-powered P-AMS, these limits must be applied.			

Certification range (range over which the P-AMS is tested and certified for compliance with the relevant performance criteria)

The certification range over which the P-AMS are to be tested must include minimum and maximum values. The coverage must be suitable for the intended application of the P- AMS. See also EN 15267-4.

In addition to the certification ranges, manufacturers may also choose additional ranges that are larger than the certification range. The certification range and the performance criteria tested for each range must be indicated on the certificate.

For the field test, the testing laboratory should select several industrial installations covering a wide range of future uses. One of these industrial processes may be a test bench recognised by the competent authorities as capable of generating the appropriate gas matrices. In this case, the test bench must comply with the requirements of Annex B of EN 15267-4: 2023.

Additional ranges:

Additional ranges must be larger than the certification range. Limited additional tests are required for each additional range. These additional tests must include at least evaluations of the response time and linearity deviation. Cross-sensitivity must be tested for interferences that have proven relevant during tests in the certification range. The concentration of the relevant interferents must be proportionally higher than the values specified in Table C.1 of EN 15267-4, where the proportionality factor is given by the ratio of the considered additional range to the certification range. However, the concentrations for cross-sensitivity tests must be chosen with reference to the intended uses. Relevant interfering components are components that cause a deviation of more than 1.0 % from the upper limit of the certification range during cross-sensitivity testing, both at zero and at the span point of the certification range.

According to VLAREM II Article 4.4.4.2, the measurement range of a measurement method must cover at least the area between 0.1 times and 3 times the emission limit value (ELV). If the certification range is chosen based on the ELV for the sector which is lower than the general ELV, the requirement for the upper limit of at least 3 times ELV is not met for installations where the higher general ELV applies. In this case, the general VLAREM requirement concerning the measurement range must be covered via the additional ranges.

Although fewer performance characteristics are tested for the additional range than for the certification range, it is assumed that if all performance tests are met for the lowest certified range, this also applies to higher measurement ranges.

6.2 MEASURED COMPONENTS THAT DO NOT FALL UNDER AN SRM

For components that do not fall under an SRM, and where permitted by the national competent authority, a less burdensome set of performance characteristics tests may be performed. These tests must be carried out by a laboratory accredited or recognised for the implementation of these types of performance tests by the local competent authority. The minimum performance characteristics to be determined are:

- Zero (≤ 2.0 % of the measurement range);
- Response time (< 400 s for NH_3, HCl and HF, < 200 s for other components);
- Detection limit (≤ 2.0 % of the measuring range);
- Linearity deviation (≤ 2.0 % of the measurement range);
- Interferences (total ≤ 4.0% of the measuring range);

7 MEASUREMENT PROCEDURE

The following characteristics are to be considered before the measurement is carried out:

- Temperature and pressure of the flue gases;
- Moisture content of the flue gases;
- Dust load;
- Presence of droplets;
- Expected concentration ranges and applicable emission limit values:
- The expected concentration of potentially interfering dusts, including at least the components listed in EN 15267-4 (Annex B);

7.1 GENERAL

The complete measuring system must be connected in accordance with the manufacturer's instructions and stabilised at the required temperature.

The data recording and processing system used in combination with the measurement system must be checked to ensure it is functioning properly.

CEN/TS 17337 describes two work methods, the Check Gas Approach and the Span Approach:

- The **Check Gas Approach** includes validating the system before use by using certified reference materials of a subset of the components to be measured. This approach is based on annual calibration of the analyser (i.e. calibration in the field is not possible) and requires the selection of reference materials based on a prescriptive method. No calibration is performed with the Check Gas Approach. Test gases are offered to demonstrate that the FTIR remains within the specification (including calibration) within a prescribed tolerance.
- The **Span Approach** requires the system to be calibrated in the field with certified reference materials from each component to be measured.

In the following, the procedure only discusses the Check Gas Approach and the associated minimum annual checks to be performed on the FTIR. This approach is likely to be the most widely used in practice. Alternatively, laboratories may also opt to use the Span Approach.

7.2 SELECTION OF TEST GASES

7.2.1 ZEROGAS

The zero gas must be a gas that does not contain significant amounts of the components to be measured or known interferences. The zero gas can be any gas that is IR-inactive, usually nitrogen or synthetic air (only N_2+O_2) is used.

7.2.2 TEST GASES

The selected test gases are used to demonstrate that the FTIR is capable of producing valid and accurate spectra at the frequencies and absorption ranges used by the analytical model.

Test gases to be applied:

- A subset of the components to be measured, ideally in concentrations at the relevant ELV, but not higher than 3 times the ELV. Where there is no ELV, the concentration must approach the expected emission value.

It is recommended to use a combination of gases that cover the entire spectrum. The requirements of CEN/TS 17337 8.4.2.2 apply. Typically, 3-4 gases are sufficient. For example, CO, SO_2, NO and a hydrocarbon, such as methane or propane. See also Annex D of CEN/TS 17337.

- When using a test gas that is not a measured component (i.e. not emitted by the stack), its concentration should be chosen to approximately correspond to the certified range.

- The test gases are certified gases, i.e.:
  - A gas produced as certified reference material (CRM), with certificate issued under ISO 17034 accreditation (BELAC or equivalent);
  - A gas with calibration certificate issued under an ISO 17025 accreditation (BELAC or equivalent) by a manufacturer accredited as a calibration body; or
  - an ISO 17025 traceable test gas; in this case, the analysis by an accredited or recognised laboratory applies as the correct value. In the latter case, the requirements described for self-certification of gases are to be followed; these requirements are included in the compendium procedure 'Essential quality requirements for emission measurements' (LUC/0/005);
  - An operator conducting self-auditing measurements and whose methods have been approved as meeting ISO 17025 may certify gases for in-house use under the same conditions as a recognised laboratory.
  - The certified concentration must be traceable to SI units and the expanded uncertainty on the analytical certificate may not exceed 2.0 %.
  - If certification and compatibility so permit, multi-component cylinders may be used (e.g. CO/SO_2/NO/N_2).

If there are no commercially available products that meet these requirements, the best available product should be purchased. The best available reference material may be a certified liquid that is evaporated in the gas phase. The concentrations generated by the evaporation system must be traceable to SI units and the uncertainty must be determined. Where available, liquid reference materials may be used that have been certified by an organisation competent for this type of certification, where one method to demonstrate competence is ISO 17025 accreditation. The liquid generation system is excluded from the 2 % uncertainty. If FTIR is applied as AM for the SRM, then the total expanded measurement uncertainty should always meet the requirement for the

measurement uncertainty from the SRM.

CEN/TS 17337 Annex D provides sample test gases that can be used to perform the Check Gas Approach tests for some common components, for two types of FTIR analysers that are routinely used for periodic emission measurements.

7.2.3 TEST GASES FOR VALIDATION OF THE SAMPLE GAS DUCT AND RESPONSE TIME OF THE MEASUREMENT SYSTEM IN THE LABORATORY

The sample gas duct must be validated using a test gas of the most reactive component measured in practice.

Table 3 contains a list of common components arranged in order of reactivity. This order is based on components that are most likely to be subject to losses in sampling systems, and there is an approximate correlation with water solubility. Consequently, the reactivity of a component that is not included below must be ranked in terms of water solubility. Water vapour is excluded from Table 3, there is no requirement to route this component through the sampling system.

Table 3: Potential test gases ranked in order of reactivity

Grade	Component
1 (most reactive)	HCl
2	NH_3
3	SO_2
4	NO_2
5	CO_2
6	N_2O
7	NO
8 (least reactive)	CO

7.3 CHECKS BEFORE MEASUREMENT, ACTUAL MEASUREMENT AND CHECKS AFTER MEASUREMENT

The complete measuring system must be connected in accordance with the manufacturer's instructions and stabilised at the required temperature. The data recording and processing system used in combination with the measurement system must be checked to ensure it is functioning properly. Next, the different steps shown in Figure 3 are carried out.

Background recording with zero gas (zero calibration)

7.3.1

▼

Zerogas supplied directly to FTIR and measurement

7.3.2
Measurement value for each component of the certification range
10 baseline measurements to determine the detection limit (CEN/ TS 17337 Annex B)

▼

A subset of test gases supplied directly to FTIR on the one hand, and via the entire measuring system on

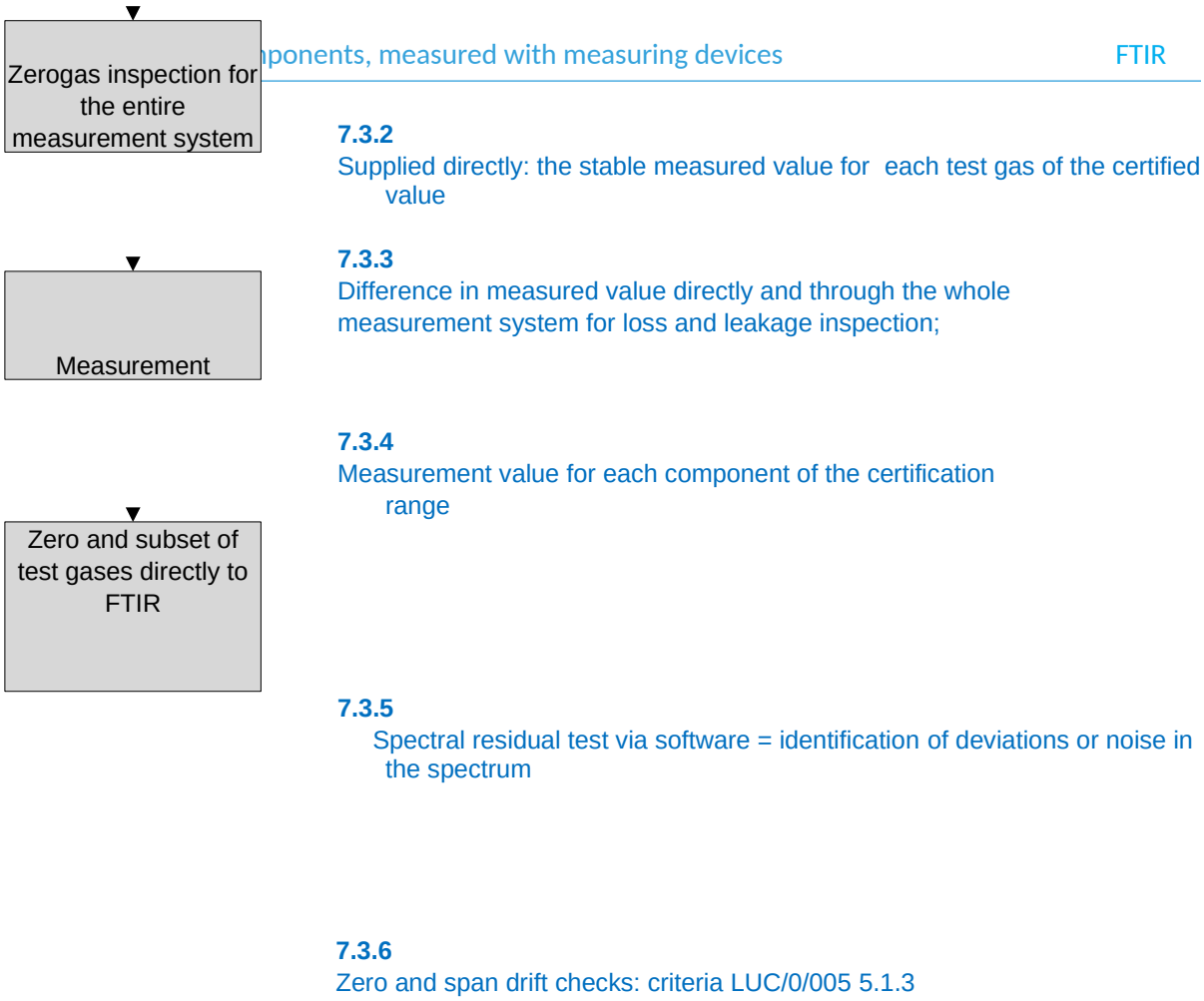


Figure 3: Different steps for FTIR checks and measurement

7.3.1 Background recording

Unless otherwise specified by the manufacturer, a background recording must be carried out using zero gas.

7.3.2 Check Gas Approach for testing the analyser

Zerogas is guided directly into the FTIR sample cell, a measurement is recorded for each component channel and each channel may not exceed 2.0 % of the respective certified range.

A total of 10 baseline measurements are to be recorded and the detection limit for each component is to be calculated as described in Annex B of CEN/TS 17337.

The subset of test gases selected in 7.2.2 to validate the matrix of components is to be guided directly to the FTIR sample cell until stable measurements are achieved. The stable value for each test gas may not deviate from the certified value by more than 5.0 %. If one component channel does not comply, the FTIR analyser is not suitable for use. The manufacturer must take corrective measures and the annual testing requirements must be complied with before the system is re-deployed for measurement.

7.3.3 Checking the sample gas duct for losses and leakage, and checking the response time in the field

After direct supply of the subset of test gases, this subset must then be passed through the entire sampling system at the same point as the zero gas. The difference between the reading obtained by passing the subset of test gases directly through the analyser and the reading obtained by passing the subset of test gases through the sampling system may not exceed 2.0%.
If the criterion is not met, one possible method to determine whether this is due to a leak (more oxygen at the bottom of the duct) or loss (oxygen remains unchanged) is by measuring oxygen at the inlet and outlet of the sample gas duct.

The response time should be shorter than 400 s for NH_3, HCl and HF, and shorter than 200 s for all other measured components, if applicable.

Vacuum leak testing is not recommended because in many FTIR systems a sub-atmospheric pressure can cause damage to internal optical components.

7.3.4 ZEROGAS INSPECTION FOR THE ENTIRE MEASUREMENT SYSTEM

Zerogas should be passed through the measurement system at the sampling probe, as close as possible to the end of the nozzle (before the filter if possible). A stable measurement should be recorded for each component channel, and the measured value should not exceed 2.0 % of the respective certification range.

7.3.5 ACTUAL EMISSION MEASUREMENT

7.3.5.1 General

The sampling of the stack must be carried out in accordance with EN 15259. A new background recording is performed at the beginning of each day.
A zero gas inspection of the entire system is to be carried out at least once a day, meeting the requirements set out in 7.3.4.

7.3.5.2 Spectral residue test

The spectral residue test in FTIR is used to assess the quality of the spectra obtained. This test helps to identify deviations or noise in the spectrum that may arise from various factors such as instrumental errors, environmental influences or incorrect sample preparation. By analysing the residues, which are the differences between the measured spectrum and a reference spectrum, it can be ascertained whether the spectrum is reliable.

For further information and interpretation, see CEN/TS 17337.

The spectral residues check can be done via the FTIR software according to criteria defined by the manufacturer and can also be used as a measure of the presence of unknown interferences.

However, there are several causes that can trigger an alarm:
-poor background
-poor water calibration
-hardware issues
-high concentrations outside the measurement range
-interferences

If an alarm occurs during a measurement in the spectral residue test, the cause of this must be further investigated. The possible presence of unknown interferences can be investigated by sampling a gas bag in the field, which is then analysed in the laboratory.

7.3.6 DETERMINATION OF DRIFT OVER THE MEASURING PERIOD

Zero- and span drifts are determined based on the difference between the measured values when zero gas is supplied directly and the subset of test gases at the end and beginning of the measuring period at the FTIR. The criteria are set out in LUC/0/005.

8 EXPRESSION OF RESULTS

The results are expressed in milligrams per cubic metre at 273.15 K and 1013 013.25 hPa. Corrections for water and oxygen may also be required.

When the results are given on a wet basis, the concentrations on a dry basis are calculated according to:

$$C_{dry} = C_{wet} \times \frac{100\,\%}{100\,\% - \%\,H_2O}$$

where

C_{dry}: the measured concentration on a dry basis;

C_{wet}: the measured concentration on a wet basis;

(%H_2O: water vapour content in wet gas, in vol % (m^3 water vapour in m^3 wet gas);

Also multiply the %CO_2 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 the installation type, for example

- waste incineration and cocombustion plants: 11%
- Solid fuel combustion plants (e.g. coal): 6%
- liquid and gaseous fuel combustion plants: 3%
- gas turbines: 15%
- brick plants: 18%

This conversion is based on the following formula:

$$Concentration_R = \frac{21 - ¿}{21 - OM} \times Concentration_M$$

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

with

= concentration at reference oxygen content

= measured concentration

OR = reference oxygen content in vol % in the dry flue gas OM = measured oxygen content in vol % in the dry flue gas

9 REPORTING AND STORING THE MEASUREMENT CAMPAIGN DATA

For reporting, procedure LUC/0/006 applies.
All spectra and/or interferograms recorded during the measuring period shall be stored with sufficient details of the acquisition parameters used so that it is later possible to re-analyse the spectra/interferograms.

10 VALIDATION

In general, a measurement method under the Flemish Environmental Permits Regulation VLAREM should be usable between 0.1 times and three times the emission limit. The following parameters should be validated at a minimum:

- Working range;
- Limit of detection (CEN/TS 17337 Annex B) and limit of determination;
- Reproducibility/intra-reproducibility;
- Trueness;
- Linearity;
- Validation of the sample gas duct and response time of the measurement system in the laboratory (see 7.2.3);
- Measurement uncertainty;

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.

Periodic quality inspection is described in Section 11.

11 QUALITY INSPECTION

Table 4 shows the minimum required frequency of continuous QA/QC controls.

Table 4: Frequency of checks

Checks	Frequency	Action criterion
Cleaning or replacement of the dust filter[1]	As required	
Regular maintenance of the measuring system	As required by the manufacturer	As required

Response time (CEN/TS 17337 Annex B)	At least once a year for each measured component and after a change in the sampling system or after a repair of the measurement system	Comply with the performance criterion in Table 2
Linearity	At least annually for each measured component	Comply with the performance criterion in Table 2
Calibration for water	Record and store new spectra in the device every 6 months or annually[2]	
Conditioning system[3]	Annually and after a repair	Dew point temperature at the outlet < 4 °C at a water vapour content > 10 %

1 The particulate filter must be replaced periodically, depending on the dust load at the sampling site. During this filter change, the filter housing must also be cleaned. Overloading the particulate filter may increase the pressure drop in the sample gas duct.

2 For water vapour, it is recommended to repeat the calibration every six months. However, for some measurement systems, an annual repetition is sufficient.

3 Only applicable to systems that operate under dry conditions.

12 DETERMINATION OF MEASUREMENT UNCERTAINTY

All accredited laboratories must have an evaluation of measurement uncertainty in place for measurements with FTIR. 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 vol% absolute.

The measurement uncertainty must be determined around the emission limit, but calculation of this must be possible for each measured concentration.

A measurement uncertainty calculation can be found in the information in Annex C of CEN/TS 17337.

When purchasing new measuring devices, the supplier must be asked about the specifications of sensitivity factors (mains voltage, pressure, sampling flow rate, temperature, vibrations) required as a contribution to the measurement uncertainty. Preferably, use data determined by an independent institute.

13 REFERENCES

CEN/TS 17337: 2019
Stationary source emissions – Determination of mass concentration of multiple gaseous species – Fourier transform infrared spectroscopy

EN 14789: 2017
Stationary source emissions-Determination of volume concentration of oxygen– Standard Reference method-Paramagnetism

EN 14790: 2017
Stationary source emissions - Determination of the water vapour in ducts - Standard reference method

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 (NBN EN 15267-3: 2008)
Air quality - Certification of automated measuring systems – Part 3: Performance criteria and test procedures for automated measuring systems for monitoring emissions from stationary sources

(NBN) EN 15267-3: 2023
Air quality - Assessment of air quality monitoring equipment -Part 3: Performance criteria and test procedures for stationary automated measuring systems for continuous monitoring of emissions from stationary sources

(NBN) EN 15267-4: 2017
Air quality - Certification of automated measuring systems – Part 4: Performance criteria and test procedures for automated measuring systems for periodic measurements of emissions from stationary sources

(NBN) EN 15267-4: 2023
Air quality - Assessment of air quality monitoring equipment - Part 4: Performance criteria and test procedures for portable automated measuring systems for periodic measurements of emissions from stationary sources

CEN/TS 17021: 2017
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)
https://emis.vito.be/nl/erkende-laboratoria/lucht-gop/compendium-luc

Determination of the content of PCDDs, PCDFs and dioxin-like and marker PCBs in a ducted gas stream

CONTENTS

1 Scope 3

2 Additions to or deviations from the standards 3

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5 References 4

1 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. It must be possible to measure these marker PCBs within VLAREL package 'L.9.3 PCBs'. 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.

2 ADDITIONS TO OR DEVIATIONS FROM THE STANDARDS

- A field blank must always be taken, analysed and reported. The procedure 'Essential quality requirements for emission measurements (LUC/0/005)' gives the procedure and criterion for each standard.
- The maximum permitted relative expanded uncertainty of the gas volume meter and of the temperature and pressure measurement at the level of the gas meter is included in procedure LUC/0/005 under the first bullet in Section 5.6.

3 VALIDATION

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

- Reproducibility/intra-reproducibility;
- Correctness, e.g. from ring test data;
- Working range;
- Limit of demonstrability and determination;
- 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.

4 DETERMINATION OF MEASUREMENT UNCERTAINTY

Every approved laboratory must have a measurement uncertainty assessment in place for the determination of PCDDs/PCDFs, dioxin-like and marker PCBs in emissions, taking into account the contributions of sampling on the one hand and 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.

5 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)
https://emis.vito.be/nl/erkende-laboratoria/lucht-gop/compendium-luc

Method WAC/VI/A/001
Compendium for water sampling, measurement and analysis (WAC)
Performance characteristics
https://emis.vito.be/nl/erkende-laboratoria/water-gop/compendium-wac

Compendium for water sampling, measurement and analysis (WAC)
Method WAC/VI/A/002
Measurement uncertainty
https://emis.vito.be/nl/erkende-laboratoria/water-gop/compendium-wac

ISO/IEC Guide 98-3:2008 Uncertainty of measurement – part 3: Guide to the expression of uncertainty in measurement (GUM:1995)

VLAREL
Flemish Government Decree adopting the Flemish Environmental Recognitions Regulation

Determination of the concentration of perfluoroalkyl and polyfluoroalkyl compounds (PFAS) in a ducted gas stream

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1 OBJECTIVE AND SCOPE

This procedure describes a method for the quantitative determination of perfluoroalkyl and polyfluoroalkyl compounds (PFAS) in a flue gas channel. It includes the concentration determination in mass per unit gas volume (ng/Nm3) of both (semi-)volatile and particle-bound PFAS (boiling point > 100 °C, > C4) via LC-MS/MS analysis of the following samples taken from a sampling train (see diagram in Figure 1):

- Rinse from fluid of the probe and the filter holder
- Filter
- Rinse from fluid from the condenser
- XAD2 cartridges: 1 at the start (primary) and 1 at end (secondary/breakthrough) of the train
- Condensate from condensate bottle
- Absorption fluid (water) from impingers
- Rinse from fluid from impingers

The procedure is primarily aimed at the quantitative analysis of 28 PFAS components that were the subject of a validation test based on 4 repeated measurements (+ field blank) with spiked sampling trains (Section 2.1) during an inter-laboratory test (ILC) in February 2024 (Hofman et al., 2025a). The sampling trains were equipped with media (filter/XAD2 cartridge/impinger water) that were spiked at a representative level. The associated measurement uncertainty (U, *k=2*) according to WAC/VI/A/002 and total train recovery (REC) for each of these components is shown in Table 1. For quantification analysis, a U of <50 % and total train recovery of 70-120 % for both sampling (LUC/VI/003) and analysis (WAC/IV/A/025) is set. The limit of quantification (LOQ) was calculated per analysis fraction (filter, XAD2 and water) as 10x times the standard deviation based on 5 repeated measurements (n=5) on both low (+/- 0.05 ng/fraction) and high (+/- 3 ng/fraction) spiked fractions (Table 1).

For most of the perfluorinated compounds only the linear form occurs. From a number of perfluorinated compounds (PFOA, PFHxS, PFOS, PFOSA, MePFOSA, EtPFOSA, MePFOSAA and EtPFOSAA) multiple branched isomers (same molecular formula, different structural formula) may occur. In this procedure, L-PFOA, L-PFHxS, L-PFOSA, L-PFOSA, L-MePFOSA, L-EtPFOSA, L-MePFOSAA and L-EtPFOSAA refer to the linear form. The total concentration (sum of linear and branched forms) is reported separately as respectively. T-PFOA, T-PFHxS, T-PFOS, T-PFOSA, T-MePFOSA, T-EtPFOSA, T-MePFOSAA and T-EtPFOSAA.

Table 1 Measurement uncertainty (U), native spike recovery (%) and limit of quantification (LOQ) of the quantitative PFAS components as determined by a validation test (Hofman et al., 2025a).

COMPONENT	CAS	REC (%)	U (k=2)	LOQ*		
		%	%	XAD2	Filter	OW
				ng	ng	ng
PFBA	375-22-4	120%	34%	ND	0.9	0.01
PFPeA	2706-90-3	100%	2%	3	0.8	0.01
PFHxA	307-24-4	98%	10%	1	1.3	0.01
PFHpA	375-85-9	93%	14%	0.9	0.9	0.01
L-PFOA	335-67-1	91%	19%	5	0.1	0.01
PFNA	375-95-1	92%	20%	0.8	0.01	0.01
PFDA	335-76-2	99%	15%	2	0.05	0.01
PFUnDA	2058-94-8	104%	28%	0.2	0.04	0.01
PFDoDA	307-55-1	107%	33%	0.5	0.04	0.01
PFTeDA	376-06-7	96%	50%	0.3	0.01	0.01
PFBS	375-73-5	96%	7%	0.4	0.8	

PFPeS	2706-91-4	91%	13%	0.03	0.03	0.01
L-PFHxS	355-46-4	92%	15%	0.05	0.05	0.01
PFHpS	375-92-8	86%	26%	0.07	0.04	0.01
L-PFOS	1763-23-1	101%	8%	3	0.8	0.01
PFNS	68259-12-1	90%	21%	0.08	0.04	0.01
PFDS	335-77-3	85%	28%	0.1	1.2	0.01
4:2FTS	757124-72-4	98%	3%	0.02	0.4	0.01
6:2FTS	27619-97-2	93%	11%	2.7	0.8	0.01
8:2FTS	39108-34-4	93%	22%	0.1	0.5	0.01
PFBSA	30334-69-1	100%	5%	6	1.8	0.01
MePFBSAA	159381-10-9	103%	5%	0.8	0.1	0.01
PFHxSA	41997-13-1	97%	14%	0.4	0.8	0.01
L-MePFOSAA	2355-31-9	94%	21%	0.05	0.01	0.01
L-EtPFOSAA	2991-50-6	97%	22%	0.05	0.7	0.01
HFPO-DA	13252-13-6	102%	8%	3.5		0.01
DONA	919005-14-4	89%	17%	0.1	0.02	0.01
PFECHS	646-83-3	88%	22%	0.1	0.04	

**The limit of quantification (LOQ) was calculated per analysis fraction (filter, XAD2 and water) as 10x times the standard deviation based on five repeated measurements (n=5) on both low (+/-0.05 ng/fraction) and high (+/-3 ng/fraction) spiked fractions*

At the same time, the following connections (14) can be indicatively determined (**Error! Invalid bookmark reference.**). These components showed a higher measurement uncertainty (>50 %) or anomalous total train recovery (<70 % or >120 %) during the inter-laboratory test (Hofman et al., 2025a), or are listed as indicative in WAC/IV/A/025.

Table 2 Measurement uncertainty (U), total spike recovery (%) and limit of quantification (LOQ) of the indicative PFAS components as determined by a validation test (Hofman et al., 2025a).

COMPONENT	CAS	REC (%)	U (k=2)	LOQ		
		%	%	XAD2	Filter	OW
				ng	ng	ng
PFTrDA	72629-94-8	109%	34%	0.1	0.01	
PFHxDA	67905-19-5	80%	59%	0.5	0.03	0.01
PFODA	16517-11-6	51%	117%	ND	0.7	
PFUnDS	749786-16-1	81%	34%	0.09	0.03	
PFDoDS	79780-39-5	79%	36%	0.03	0.7	
PFTrDS	791563-89-8	72%	51%	0.02	0.02	
10:2FTS	120226-60-0	108%	29%	0.1	0.7	0.05
MePFBSA	68298-12-4	55%	140%	ND	1.4	0.01
L-PFOSA	754-91-6	56%	85%	0.08	0.8	0.01
L-MePFOSA	31506-32-8	1%	273%	0.2	1.4	0.01
L-EtPFOSA	4151-50-2	0%	289%	ND	0.7	0.01
6:2diPAP	57677-95-9	92%	47%	0.09	0.005	
6:2/8:2diPAP	943913-15-3	81%	63%	0.1	0.05	0.05
8:2diPAP	678-41-1	67%	72%	0.7	0.08	0.01

**The limit of quantification (LOQ) was calculated per analysis fraction (filter, XAD2 and water) as 10x times the standard deviation based on five repeated measurements (n=5) on both low (+/-0.05 ng/fraction) and high (+/-3 ng/fraction) spiked fractions*

2 PRINCIPLE & SETUP

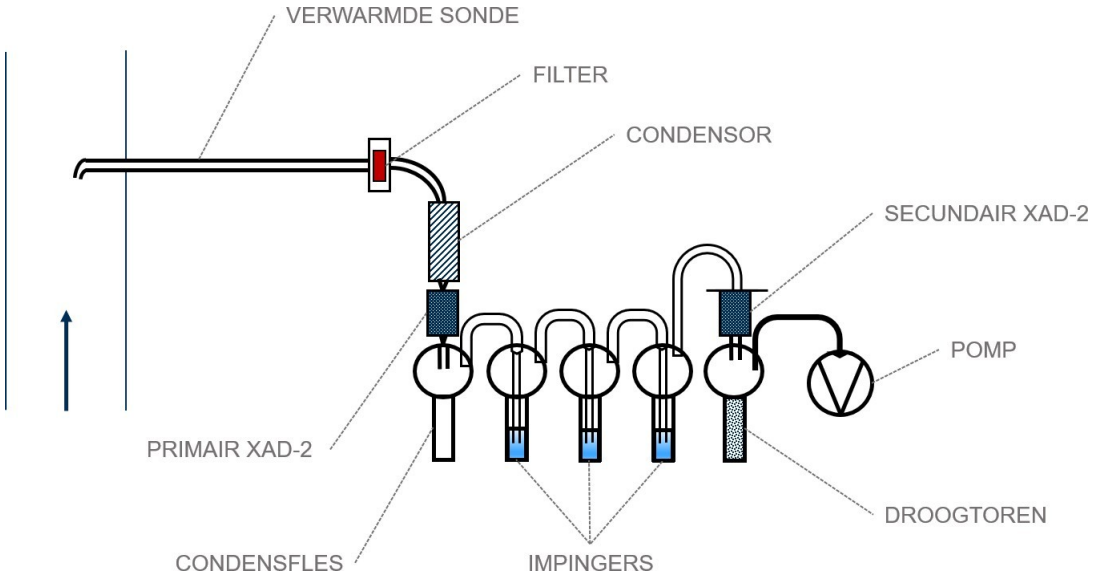
For the measurement of PFAS (>C4, boiling point > 100 °C) in ducted emissions, a sampling train shall be used based on the OTM-45 method[1] from EPA (published 13/1/2021) and as shown in Figure 1. This OTM-45 method was published by EPA to promote consistency in the sampling and analysis of PFAS in ducted emissions and is considered best available technique. Gaseous and particle-bound PFAS (>C4, boiling point > 100 °C) shall be isokinetically (same size and direction of gas velocity) sampled from the stack using a heated probe. Particle-bound PFAS are collected on a heated quartz fibre filter (120 °C or at least 10 °C above stack temperature), after which gaseous PFAS are captured using a primary adsorbent (XAD-2) cartridge, a condensate bottle, a series of

impingers with absorption reagent (ultrapure water) and then a secondary adsorbent (XAD-2; breakthrough) cartridge.

2.1 OTM-45 SETUP

The OTM-45 setup is displayed in Figure 1 and consists of:

- a heated probe with quartz filter
- a condenser
- a primary adsorbent (XAD-2) cartridge
- an empty condensate bottle to absorb the condensate
- a series of three impingers filled with ultrapure water
- a secondary adsorbent (XAD-2) cartridge (breakthrough)
- a drying tower (silica or equivalent desiccant) with pump



VERWARMDE SONDE	HEATED PROBE
FILTER	FILTER
CONDENSOR	CONDENSER
SECUNDAIR XAD-2	SECONDARY XAD-2
POMP	PUMP
DROOGTOREN	DRYING TOWER
IMPINGERS	IMPINGERS
CONDENSFLES	CONDENSATE BOTTLE
PRIMAIR XAD-2	PRIMARY XAD-2

Figure 1 Schematic representation of OTM-45 sampling train

[1] https://www.epa.gov/sites/default/files/2021-01/documents/otm_45_semivolatile_pfas_1-13-21.pdf

The sampling train consists of borosilicate or quartz glass (probe, filter holder, condenser, condensate bottle, impingers). Although Teflon (PTFE) should be avoided as much as possible (risk of contamination), it may be used in connectors, O-rings and/or coatings. Although Teflon (PTFE) should be avoided as much as possible (risk of contamination), it may be used in connectors, O-rings

and/or coatings. Any contamination in the media or sampling train used can be identified in the respective medium and field blanks.

Further specifications for the different parts of the sampling train include:

- The quartz fibre filter has an efficiency of at least 99.95 % (< 0.05 % penetration) for 0.3 µm particles and is kept at a temperature of 120 ± 12 °C during the stack measurement to avoid condensation/moisture on the filter. This temperature should be measured in the filter housing.
- Tubing, condenser and XAD-2 cartridges should be vertically oriented in the sampling train to promote maximum flow and prevent moisture accumulation (only required for OTM-45 method).
- Adsorbent cartridges consist of glass cartridges with water jacket and contain 20 to 40 g of solid adsorbent (XAD-2).
- The temperature of the input gas stream at the XAD-2 cartridges should be kept below 20 °C. This (incoming) temperature is measured at the level of the primary XAD-2 cartridge. An ice bath can be used to circulate cooled water to the condenser and adsorbent cartridges with a pump. If the ambient temperature is > 20 °C, the XAD-2 cartridges must be cooled.
- Impingers are of the standard type.
- The adsorbent (XAD-2) is stored and transported in glass cartridges or clean HDPE jars with wide opening.
- During sampling, the XAD-2 cartridges are wrapped with aluminium foil to protect the adsorbent from radiant heat and sunlight
- The impingers are filled with about 100ml ml of ultrapure water

Recovery of the samples takes place in existing cartridges (e.g. XAD-2 cartridges) or polyethylene (HDPE) bottles and results in 19 different samples (Appendix 1; including medium, field, and post-rinse blanks) that are further reprocessed into 6 analytical fractions (Table 3).

Since the condensate in the sampling train described above is passed through the primary XAD-2, the adsorbent cartridge can become very wet for stacks with a high moisture content. This could potentially have an effect on the capturing efficiency of the adsorbent cartridge or the required negative pressure for the suction of the sampling train. Two alternative sampling variants were therefore defined in which the condensate water is collected in a condensate bottle before being passed through the primary XAD-2 cartridge, namely the OTM-45 variant (Section 8.2.1) and the cooled lance method (Section 8.2.2). The equivalence of these sampling variants was demonstrated for 3 PFAS components (PFBA, T-PFOA and PFHxA; C4-C8) by means of simultaneous stack measurements and for the entire WAC/IV/A/025 scope during an inter-laboratory test (February, 2024).

3 SAMPLING PROCEDURE

3.1 PREPARATION

All glassware must be uniquely marked in advance, so that traceability to possible contamination is possible. Depending on the use/re-use of the glassware, there are two different cleaning procedures. Glass liners (probe) and filter house are renewed and rinsed with methanol at each measurement.

For new glassware the following shall apply:

- The glassware (excluding liner) shall be rinsed with warm water for a maximum of two

weeks before sampling, then rinsed with methanol and dried at 150 °C. The glassware shall be stored in a sealed container pending sampling.
- Glass liner (probe) is rinsed with methanol

For existing glassware that is re-used, the following applies:
- The glassware (excluding liner) is cleaned with warm water up to two weeks before sampling, and then rinsed successively with methanol and cyclohexane.
- After cleaning with methanol and cylchohexane, the glassware is fired at a min. of 450 °C for a min. of 3 hours. The glassware is now ready for sampling. Pending sampling, the glassware must be stored in a sealed container.
- Glass liners (probe) and filter house are renewed and rinsed with methanol at each measurement

The various parts of the sampling train are cleaned in advance with methanol so that they are free from PFAS.
- Adsorbent: The adsorbent (XAD-2) is extracted at night (16 hours) with methanol by means of Soxhlet extraction. Following this, the XAD-2 cartridges are dried at 40 °C in an oven and (packaged in aluminium foil) stored in a sealed glass jar. The **cleaning of the XAD-2 is carried out up to one week before sampling**. The cartridges are filled with 20-40 g purified XAD-2, with a minimum height-width ratio of 2. The sampling standards (SS) are doped for sampling in the primary XAD-2 cartridge (13C3 PFBA, 13C8 PFOA, 13C8 PFOS; Hamilton pipette) and on the filter (13C2-PFUnDA, 13C2-PFDoDA). For the cooled lance method, 13C2-PFUnDA and 13C2-PFDoDA are not doped on filters, but in the condensate bottle. Typically, doping includes 10 ng of each standard. The XAD-2 is secured in the cartridge by means of purified glass wool.
- Glass wool: Glass wool is purified with methanol and dried at room temperature with N_2.

3.2 SAMPLING

The sampling procedure depends on the sampling train used (OTM-45 (Section 2.1), OTM-45 variant (Section 8.2.1) or cooled lance variant (Section 8.2.2)) and consists successively of:
- Building up sampling train, performing leak test and collecting field blank sample (6, Appendix 1)
- Heating (or cooling) of probe and filter housing;
- Installing the probe in the gas duct;
- Recording gas meter readings;
- Starting the pump and setting required isokinetic suction flow rates;
- Sampling of the flue gas duct for a minimum of three hours (~2 Nm^3);
- Carrying out a new leak test after sampling to ensure that no leaks were created during the measurement.

Due attention should be paid to preventing the contents of the impingers being sucked back as a result of an underpressure in the gas duct or as a result of pressure variations in the sampling line at the start-up of the pumps.

- Check the suction flow rates regularly and adjust them if necessary. Record the pressure, temperature, flow rates and gas meter readings. If sampling is to be carried out at different points, move the sampling probe from point to point without stopping the pump, and adjust the sampling parameters depending on an isokinetic sampling. In the event of a change of measurement opening, the pump must be switched off.
- The pump is subsequently stopped. Re-record the gas meter readings. Remove the probe from the gas channel.
- Then proceed to the recovery of the field samples (7; Section 3.5)
- Re-rinse the glassware and recover the re-rinse from blank samples (2; Section 6.1.1)

3.3 CONTROLS

During a stack measurement, the following checks are carried out:

- check the gas speeds at the sampling points and calculate the sampling parameters to be set at each point (flow rate, sampling time) if necessary;
- check the sampling train for leak density before each sampling (see also procedure essential quality requirements LUC/0/005);
- for each measurement series and a minimum of once a day, a blank of the equipment must be taken (field blank);
- Install the probe in the gas channel and condition the sampling system for at least 10 minutes, so that the entire sampling train reaches operating conditions, and check the proper operation of the probe;
- during the stack measurement, check the temperature of:
  - Filter: the temperature at the filter level (filter housing or directly into the outgoing gas stream) shall be 120 ± 12 °C. Monitor the temperature of the filter during the stack measurement;
  - XAD-2 cartridges: the temperature of the XAD-2 cartridges must be below 20 °C for efficient caption of the TARGET components.
- Recover the field samples immediately after removing the probe from the stack;

3.4 OTHER PARAMETERS TO RECORD

In order to convert the quantified mass (ng/analysis fraction) to normalised stack emissions (ng/Nm^3), it is necessary to measure additional stack parameters during the sampling period (LUC/0/005):

- volumetric flow rate (m^3) and temperature (°C) of the emitted gases, together with a continuous measurement of these parameters at a reference point if the flow rate is not constant.
- water content (%)
- O_2 concentration (%)

3.5 RECOVERY AND AGGREGATION OF FIELD SAMPLES

19 samples are collected for each emission measurement (Appendix 1), including medium blank samples (5), field blank samples (5) and re-rinse from blank samples (2). Treatment of the field samples (S11-17 in Appendix 1) is specifically explained below:

Rinse from probe + front part of filter holder (S12 in Appendix 1): The glassware in front of the filter (the front part of the filter holder, probe and nozzle) are rinsed 3x times with methanol/1 % NH_4OH after sampling (in the field) and this rinse from fluid is delivered to the lab in an HDPE container (S11).

Filter (S14 in Appendix 1): The loaded filter is removed from the setup and delivered to the laboratory in an aluminium-wrapped petri dish.

Water fraction (S17 in Appendix 1): The condensate from the condensate bottle and the water from the impingers are transferred to a cleaned HDPE container and delivered to the laboratory as a composite sample.

MeOH rinse from fraction (S15 in Appendix 1): The glassware after the filter (the rear part of the filter holder, condenser, condensate bottle and impingers) are rinsed 3x times with methanol/1 % NH_4OH after sampling (in the field) and delivered to the laboratory as a composite sample in an HDPE container (S15).

Cyclohexane rinse from fraction (S13 in Appendix 1): From the probe to the condensate bottle (probe, filter holder, condenser and condensate bottle), after sampling and after MeOH rinse from (S15), the pieces are rinsed again 3x times (in the field) with cyclohexane and delivered to the laboratory as a composite sample in a separate HDPE container (S13).

XAD-2 (S16+ S18 in Appendix 1): The XAD-2 cartridges are removed from the set-up and delivered to the laboratory in their glass holder (two cut glass caps are placed on the ends) or in an HDPE jar as separate samples (S16 and S18 in Appendix 1). As more data are collected through this method on the occurrence of breakthroughs, the analysis requirements of the XAD-2 cartridges as separate fractions may be relaxed.

If the cooled lance variant set-up (Section 8.2.2) was applied, any deposition of particulate PFAS in the condensate shall be taken into account by performing an additional filtration of the condensate after which the residue is added to the filter sample (S14*) and filtrate to the water fraction (S17*).

All field samples collected are stored and transported in the dark (shielded against UV light) and on ice (~4 °C). In order to avoid contamination of the media and field samples, particular attention should be paid to hygiene during transport, field handling, sampling, recovery and laboratory analysis, as well as during the preparation of the XAD-2 cartridges. Each sampling results in 20 samples (Appendix 1) that are aggregated to six analytical fractions that are further processed and analysed (Table 3).

Table 3 Aggregation of field samples to analytical fractions (F1-F6) for the three variant setups (OTM-45, OTM-45 variant and cooled lance). The references of the field samples refer to the described treatment of the field samples above (Section 3.5).

Fraction	OTM-45	OTM-45 variant	Cooled lance
F1	Filter (S14) + rinse from probe (S12)	Filter (S14) + rinse from probe (S12)	Filter (S14) + residue filtration (S14*)
F2	Rinse from condenser + rinse from condensate bottle + rinse from impingers (S15)	Rinse from condenser + rinse from condensate bottle + rinse from impingers (S15)	Rinse from probe (S12) + rinse from condensate bottles + impingers (S15)
F3	Primary XAD-2 (S16)	Primary XAD-2 (S16)	Primary XAD-2 (S16)
F4	Condensate + water impingers 1-2-3 (S17)	Condensate + water impingers 1-2-3 (S17)	Condensate (+ filtrate filtration) + water impingers (S17)
F5	Secondary XAD-2 (S18)	Secondary XAD-2 (S18)	Secondary XAD-2 (S18)
F6	Rinse from probe + condenser (Cyclohex) (S13)	Rinse from probe + condenser (Cyclohex) (S13)	Rinse from probe cyclohex (S13)

3.6 Transport & Storage

All field samples are transported on ice (4 °C; except for the filter) and in the dark (shielded from UV light). In the laboratory, the analytical fractions (F1-F6) are stored in the dark and cooled (< 6 °C) and extracted within 28 days of sampling. The extracts can then be cooled for up to one year (< 4 °C) and stored in the dark.

Notes:

- Contact with teflon or other fluorinated polymers should be avoided.
- The concentration of >C10 PFAS in water samples may decrease with increasing storage time, due to absorption to recipient wall or precipitation (WAC/IV/A/025).

4 ANALYSIS PROCEDURE

In the laboratory, the aggregated analytical fractions (Table 3) are further processed (Sections 4.2-4.6).

Quantitative analysis of the fractions via LC-MS/MS (Section 4.7) is carried out in accordance with WAC/IV/A/025. The content of the different PFAS is calculated using the internal standard method. Perfluoroalkyl and polyfluoroalkyl compounds and internal standards shall be identified on the basis of the criteria for retention times and ion ratios as specified in WAC/VI/A/003.

If a high content of PFAS is expected, the samples can be pre-screened e.g. by means of a non-target analysis. For this purpose, a small portion (max. 2 %) is taken for analysis.

4.1 MATERIALS AND REAGENTS

For the materials, reagents, standards and solutions used, reference is made to WAC/IV/A/025.

4.2 EXTRACTION OF FILTER (FRACTION F1)

- Transfer the filter to a (PP) tube.
- Add an appropriate amount of internal standards (IS) so that the theoretical concentration of the IS in the measurement extract is equal to that in the calibration standards.
- Add 10 mL of the rinse from fluid from the probe and filter holder, ensuring the filter is fully submerged. Add extra MeOH/1 % NH_4OH if needed. Evaporate the remaining rinse from fluid to a few mL.
- Vortex the whole thing
- Sonicate for 1 hour
- Decant the liquid into another (PP) tube.
- evaporate extract to approximately 2 ml
- Centrifuge and transfer the MeOH fraction to a new PP tube without transferring the filter residue with it
- Wash the filter residue with MeOH, vortex, centrifuge, and transfer MeOH to the same PP tube
- The above washing step is repeated
- Add any excess of the rinse from fluid (a few mL)
- Evaporate extract to 500 µL (MeOH fraction)
- Vortex regularly to prevent the components from depositing on the wall of the test tube
- +500 µl MeOH and vortex well
- Transfer to a measuring vial

Of the extract, 10 µl is typically injected into the LC-MS.

The shelf life of preparations shall be one month when stored in the refrigerator. Preparations that have been in the refrigerator are vortexed before placing them in the injection machine.

4.3 EXTRACTION OF MEOH RINSE FROM FRACTION (FRACTION 2)

- If desired, the sample can be divided into two equal parts and one part can be stored for re-analysis.
- Add an appropriate amount of internal standards so that the theoretical concentration of the internal standards in the measurement extract is equal to that in the calibration standards.
- Evaporate to about 2 ml
- Transfer the sample to a 15 ml PP tube and rinse the container 4x times with MeOH
- Transfer the sample to a 500 ml PP tube and rinse the container 4 times with MeOH
- Vortex regularly to prevent the components from depositing on the wall of the test tube
- +500 µl MeOH and vortex well
- Transfer to a measuring vial.

Of these, 10 µl is injected into the LC-MS.

The shelf life of preparations shall be one month when stored in the refrigerator. Preparations that have been in the refrigerator are best vortexed before placing them in the injection machine.

4.4 EXTRACTION OF THE XAD-2 CARTRIDGES (FRACTION F3 AND F5)

- For extraction, the primary XAD-2 cartridge is emptied for 2 minutes under an N_2 stream, and the droplets of water from the XAD-2 cartridge are collected and added to the water fraction (F4). Record the amount of vented water by pre- and post-weighing of the XAD-2 cartridge. The secondary XAD-2 cartridge contains no water and can be extracted in this way.
- Add an appropriate amount of internal standards to the XAD2 cartridge
- Add 75 ml MeOH/1 % NH_4OH
- Top and bottom are sealed with a glass stopper and clamps
- Sonicate for 1h hour (in bucket filled with water to MeOH level)
- Replace glass stopper with connectors and flush the MeOH/1 % NH_4OH with N2 for 2 min from the XAD-2 and collect in a 500ml ml container
- Rinse XAD-2 with approximately 30mL mL of MeOH and also flush with N2 for 2 minutes, then add to the same container
- Rinse glass stoppers with a few ml MeOH
- Evaporate MeOH fraction to about 2 ml
- transfer extract to 15 ml PP tube and rinse the container 4x times with MeOH
- further evaporate to 500 µL
- Vortex regularly to prevent the components from depositing on the wall of the test tube
- +500 µl MeOH and vortex well
- Transfer to a measuring vial.

Of these, 10 µl is injected into the LC-MS.

The shelf life of preparations shall be one month when stored in the refrigerator. Preparations that have been in the refrigerator are best vortexed before placing them in the injection machine.

4.5 EXTRACTION OF WATER FRACTION (FRACTION 4)

- Determine the total weight of the water fraction from the XAD-2 cartridge, condensate and

impingers.
- The contents are transferred into a larger container, after which the original container is rinsed 4x times with MeOH (final concentration must not exceed 10 % MeOH) and added to the larger container.
- Take a subsample, e.g. 50 mL of the water fraction, and record the weight
- The remaining water fraction (max. 10 % MeOH) is divided into a few subsamples of 50ml ml for possible re-analysis.
- Add an appropriate amount of the standard solution of internal standards so that the theoretical concentration of the IS in the measuring extract is equal to that in the calibration standards.
- Vortex the whole thing
- An SPE-WAX cartridge is used for extraction. The procedure includes the following steps:
  - condition the SPE cartridge with 4 ml of MeOH/0.1 % NH_4OH solution;
  - condition the SPE cartridge with 4 ml of methanol;
  - rinse the SPE cartridge with 4 ml ultrapure water be careful not to let the sample dry;
  - transfer the subsample (50 mL) onto the SPE cartridge;
  - centrifuge the SPE cartridges so that they are dry (20 min, 2000+ rpm)
  - rinse the sample flask with 5 ml of methanol and elude the SPE cartridge with it; collect this fraction;
  - rinse the sample bottle with 5 ml MeOH/0.1 % NH_4OH solution and elute the SPE cartridge; combine this fraction with the previous one;
  - SPE extract is evaporated to 500 µL
  - Vortex regularly to prevent the components from depositing on the wall of the test tube
  - add 500 µl MeOH and vortex
  - transfer to a measuring vial;

Of these, 10 µl is injected into the LC-MS.

The shelf life of preparations shall be one month when stored in the refrigerator. Preparations that have been in the refrigerator are best vortexed before placing them in the injection machine.

4.6 Extraction of cyclohexane rinse from fraction (Fraction 6)

- If desired, the cyclohexane rinse from fraction can be divided into two equal parts and one part can be stored for re-analysis.
- Add internal standards
- Evaporate to about 2 ml
- Transfer the samples to 15 ml PP tubes and rinse the container 4x times with MeOH
- Further evaporate to a few droplets
- Vortex regularly to prevent the components from depositing on the wall of the test tube
- Add MeOH to 1 ml
- Transfer to a measuring vial.

Of these, 10 µl is injected into the LC-MS.

The shelf life of preparations shall be one month when stored in the refrigerator. Preparations that have been in the refrigerator are best vortexed before placing them in the injection machine.

4.7 LC-MS/MS MEASUREMENT

The measurement occurs in accordance with WAC/IV/A/025.

5 REPORTING

The reporting consists of the measured mass concentrations per analytical fraction (ng/fraction) for the field samples and field blank (< 10 %) and the resulting relative breakthrough (< 30 %). The emission concentration ($C_{emission}$) of each component (reputed over the different analytical fractions) is then calculated in ng/Nm³. This is calculated from the sum concentration of the samples (analysed fractions), the flow rate sampled and prevailing O_2 concentration in the stack.

The samples flow rate () is calculated from the gas counter sampling data, temperature and pressure meter as follows:

$$V_{samp} = \frac{(V_{end} - V_{start}) * 273.15 * P_{gas}}{(273.15 + T_{qas}) * 1013.25}$$

with the samples volume (Nm³dr), and the start and end volume on the gas counter (m³), the absolute pressure (mbar) on the gas counter and the temperature on the gas counter (°C) respectively. Furthermore, 273.15 is the reference temperature (Kelvin) and 1013.25 is the reference pressure (mbar).

The quantified sample concentrations (ng/fraction) are then charged over the different analytical fractions according to the lower-bound method (< DL = 0) and finally normalised for the sampled flow rate (V_{samp} in Nm³) and the oxygen content (Of_2) to arrive at the emission concentration (in ng/Nm³):

$$C_{emission} = \frac{C_{mon}(21 - O_{2\,REF})}{V_{samp}(273.151 - O_{2\,stacks})}$$

with the normalised emission concentration (ng/Nm³ dr), the aggregated sample concentration (ng) using the lower-bound method (< DL = 0), $_{2_}$ the reference oxygen content (11 %) and $_{2_h}$ the oxygen content in the stack (%).

6 QUALITY INSPECTION

6.1 SAMPLING QUALITY INSPECTION

6.1.1 SAMPLE BLANKS

Due to the risk of contamination, strict quality inspection is provided for in this LUC method. It follows the OTM-45 quality procedure, although eases the blank use depending on the use of glassware. Three sampling blanks are considered:

1. **Medium blank (S1-S5 in Appendix 1):** A blank filter, rinse from liquid (alkaline methanol and cyclohexane), blank XAD-2 and ultrapure water (five samples) are supplied to the laboratory in HDPE containers. Nothing has been done in the field with this blank, only a transfer to the sampling container. The purpose of the blank medium is to determine whether used media or other interferences are introduced into the sample by transport, storage and field environment.
2. **Field blank (S6-S10 in Appendix 1):** The setup as a whole is built up, filter housing heated, provided with a leak test and rinsed just for sampling with alkaline methanol. Recovery and aggregation of the field samples follows the same procedure as described in Section 3.5, with the exception of cylohexane rinsings and the secondary XAD-2 cartridge. The resulting

analytical fractions (6) are supplied to the laboratory in an HDPE container:
S6: Rinse from fluid of the probe and the filter
holder S7: Filter
S8: XAD-2 (primary only)
S9: Ultrapure water from the impingers
S10: Rinse from fluid of the condenser, condensate bottle and impingers
The measured field blank concentrations in each fraction shall be reported to interpret the analytical results.

3. **Re-rinse from blank (S18-S19 in Appendix 1):** After sampling and (3x) rinse from with alkaline methanol (methanol/1 % NH_4OH) and cyclohexane, the sampling train is rinsed again 3x times with alkaline methanol and 3x times with cyclohexane. The rinses are combined per solvent into 1 composite sample and delivered to the laboratory in HDPE containers (1 methanol (S18) and 1 cyclohexane (S19)). The purpose of the re-rinse from blank is to check whether all PFAS were sufficiently captured in the field sample (sampling + three times rinsing).

The different sampling blanks are collected for each campaign (maximum three consecutive measurement days at the same measurement location/company*) or measurement depending on the use of glassware (diagram):

- If for each measurement **the same glassware** is used only one field blank should be collected per campaign, and a re-rinse blank for each measurement.
- If for each measurement **new glassware** is used, a field blank is required for each measurement and one re-rinse blank per measurement campaign.

In order to avoid unnecessary analysis of medium blanks (four samples), analysis is only required when field blank results are higher than the 10 % of the field sample. When a new stack is sampled at the same location/company, the use of new glassware is required.

*Campagne = max. 3 opeenvolgende meetdagen op eenzelfde meetlocatie

**Nieuwe Schoorsteen = nieuw glaswerk

ZELFDE GLASWERK
VELDBLANCO 1x/campagne*
NASPOELBLANCO 1x/meting
VELDBLANCO > 10% veldstaal
MEDIUM BLANCO
NIEUW GLASWERK**
VELDBLANCO 1x/meting
NASPOELBLANCO 1x/campagne*
VELDBLANCO > 10% veldstaal
MEDIUM BLANCO
*Campagne = max. 3 opeenvolgende meetdagen op eenzelfde meetlocatie

SAME GLASSWARE
FIELD BLANK 1x/campaign*
RE-RINSE BLANK 1x/measurement
FIELD BLANK > 10 % field sample
MEDIUM BLANK
NEW GLASSWARE**
FIELD BLANK 1x/measurement
RE-RINSE FROM BLANK 1x/campaign*
FIELD BLANK > 10 % field sample
MEDIUM BLANK
*Campaign = max. three consecutive measurement days at the same measurement

location
**Nieuwe Schoorsteen glaswerk
**New stack glassware

6.1.2 SAMPLING STANDARDS (SS)

For each stack measurement, isotope-marked sampling standards (SS; Appendix 2) are added to the filter (^{13}C2-PFUnDA, ^{13}C2-PFDoDA) and the primary XAD-2 (^{13}C8-PFOA, ^{13}C8-PFOS ^{13}C3-PFBA) and to inspection the entire sampling and analysis process. In this LUC method, both of the sampling standards for >C10 (^{13}C2-PFUnDA, ^{13}C2-PFDoDA), C8 (^{13}C8-PFOA and ^{13}C8-PFOS) as well as for C4 (^{13}C3-PFBA) components are considered. The apparent recoveries of these standards must, after correction for the recovery of the internal standards (IS), demonstrate per stack measurement that recovery of the labelled standards complies with the required range of 70-130 %. Between 50-70 %, recoveries are still acceptable, although the recoveries must be flagged and reported. Below 50 %, standard recovery should be checked for the reason for this and a remeasurement is necessary.

As recovery, the rate of recovery (%) is reported throughout the sampling train as a migration of recovery standards through the train cannot be excluded.

6.1.3 RELATIVE BREAKTHROUGH

The relative breakthrough (RD; %) of PFAS through the sampling train shall be calculated per component on the basis of the mass ratio between the secondary (breakthrough) adsorbent (F5) and the sum of all fractions (F1-F6) in the sampling train using the following formula:

$$RD(\%) = \frac{F5_{massa}}{(F1¿¿mass + F2_{mass} + F3_{mass} + F4_{mass} + F5_{mass} + F6_{mass}) \times 100\%¿}$$

If the relative breakthrough is less than 30 %, the quantified mass for each PFAS component from fraction F5 (secondary XAD-2 cartridge) shall be met with the other fractions for the calculation of the total PFAS span emission. If the relative breakthrough is greater than 30 %, it is necessary to assess the reason for this and a remeasurement is necessary.

7 MATERIAL

7.1 SAMPLING

- Heated probe + filter housing
- Filter
- Uniquely marked glassware + couplings (possibly with glass spherical couplings):
  - Condenser
  - Condensate bottle (1)
  - Impingers (3)
  - XAD-2 cartridges (2), spiked with sampling standards (SS)
- Teflon tubes (if necessary)
- Drying column with silica
- Pump
- Cooling bath with pump
- Aluminium foil
- Fridge box with ice (preservation of field samples)
- Solvents: alkaline methanol (methanol/1 % NH_4Oh) and cyclohexane
- Sufficient HDPE containers (28)

8 ADDITIONS TO OR DEVIATIONS FROM OTM-45

Based on the accumulated practical experience of the reference lab with PFAS stack measurements, comparative measurements and bilateral communication with the Environmental Protection Agency (EPA) and Flemish commercial laboratories, a list of permitted deviations from the OTM-45 method (Section 9.1) and two alternative sampling trains (Section 9.2) were defined (Hofman et al., 2025b).

8.1 DEVIATIONS

An overview of the permitted deviations from this method from the OTM-45 procedure, with corresponding reasoning, is presented in Table 4.

Table 4 Overview of additions or deviations from the OTM-45 method

OTM-45	LUC
SAMPLING	
A heated tube between filter holder and condenser	No need for heating and therefore no requirement
Condensate sent through the primary XAD-2 cartridge	variants of sampling (cooled lance, OTM-45 variant)
analytical fractions (4): • filter + rinse from probe and filter housing • rinse from filter housing – condenser + primary XAD-2	analytical fractions (6): • filter + rinse from probe and filter housing • primary XAD-2 • rinse from filter housing - condenser + rinse from of condensate bottle • condensate + water impingers + rinse from of impingers
• condensate + rinse from condensate bottle + water impingers + rinse from of impingers • secondary XAD-2	• secondary XAD-2 • cyclohexane rinsing
Medium blanks collected at each measurement campaign and field and rinse blanks at each measurement.	Based on the observed blank concentrations easing the frequencies of field or re-rinse blanks based on glassware use.
XAD-2 cartridges are cooled	XAD-2 cartridges must only be cooled if the temperature requirements (<20 °C) are not met.
The OTM-45 method provides one condensate bottle and three additional washing bottles/impingers.	minimum 1 condensate bottle and two washing bottles/impingers in the sample take-train
concentration of ammonia in methanol is 5 %	ammonia concentration in methanol can be reduced from 5 % to 1 %
Filter quality inspection (three filters per batch) including extraction and reprocessing. (Section 7.1.1.1)	Check already included in the procedure/medium blank
Adsorbent (XAD2) quality inspection including extraction and reprocessing (Section 7.1.3)	Check already included in the procedure/medium blank
Minimum sampling of 3 Nm^3 dr flue gas (Section 8.1.2.1)	Sometimes difficult due to perceived back pressure in the sampling train: min. 3u measurement - -> ~2 Nm^3 dr
Note colour (state) of silicators in field report (Section 8.2.10)	Not required to report
Re-rinse blank (Sample Train Field Blank STFB) is again made up of full used sampling train with new media.	Re-rinse blank consists of only rinse from to check the sampling train
Temperature of the filter (120 °C) is measured in the gas stream	Temperature can also be measured in the filter housing.
The 1e, 3e and 4e impinger are of the Greenburg-Smith type with customised (longer) tip. Only the 2e impinger is of the Greenburg-Smith type.	All impingers are of the standard type

Recovery of filter in glass Petri dish sealed with HDPE tape or resealable PE bag.	Filter is recovered in a Petri dish packed in aluminium foil
Rinse aluminium foil three with 5 % ammonium hydroxide in methanol and store in cleaned Petri dish or gauze jar.	After initial laboratory tests, this procedure is no longer applied.
Recovery of XAD-2 in original container, HDPE container with PP or PE lid or in glass container sealed with glass lids.	Recovery of XAD-2 in glass container or HDPE pot
Clean glass wool by immersing it three times in 5 % ammonium hydroxide in methanol, drying it in an oven at 110 °C and storing it in a glass jar rinsed with 5 % ammonium hydroxide in methanol with a PE or PP liner lid.	Glass wool is rinsed with methanol and dried with N_2 at room temperature
Ammonium hydroxide, 5 % in methanol rinse from liquid	After initial laboratory tests, the percentage of ammonia can be reduced to 1 %
Glassware washing procedure: Hot water + soap – tap water – deionised water – acetone, dichloromethane, methanol – drying at 300°	New glassware: Glassware is rinsed with warm water and methanol and ignited at ± 150 °C for at least three hours Glassware used: Successive 1 hour sonication with methanol, 1 hour sonication with cyclohexane (fully submerged), and at least 3u hours of firing at 450 °C.
Seal glassware with glass lids or cleaned aluminium foil	Glassware is stored in sealed container
Re-rinse glassware for use with 5 % ammonium hydroxide in methanol	only rinsed with methanol
Filter temperature is maintained at 120 °C or 10 °C above stack temperature to prevent condensation on the filter.	Filter housing is kept at 120 ± 14 °C by default
Use a brush while rinse from the probe to collect any deposited material.	Single rinse from is applied (No contamination of rinse blanks observed)
REPROCESSING/ANALYSIS	
Seven samples sampled are aggregated to four analytical fractions.	7 samples are aggregated to 5 analytical fractions following moist XAD-2 and traceability throughout the train.
Recovery standards for C8 PFOA and PFOS (13C8-PFOA, 13C8-PFOS)	In addition to standard recoveries for C8 PFOA and PFOS (13C8-PFOA, 13C8-PFOS), also a spike of a C4 component (13C3-PFBA)
Shake 18u hours for extraction.	After initial laboratory tests: One hour of sonication as an alternative (+ shake up four times)
Dilution with water (or methanol) in case of evaporation	Dilution with methanol in case of evaporation
Analysis of the PFAS components (50) described in OTM-45	Analysis of the PFAS components carried out via LC-MS/MS analysis in accordance with WAC/IV/A/025. This methodology was validated for 52 components.
QC/REPORTING	
If the relative breakthrough is greater than 10 %, the fraction of the 2nd XAD-2 is included and reported in the sum of the other fractions.	The 2de XAD-2 cartridge concentration is always added to other fractions. Only if > 30 %: must be checked for the reason, and a remeasurement may be necessary.

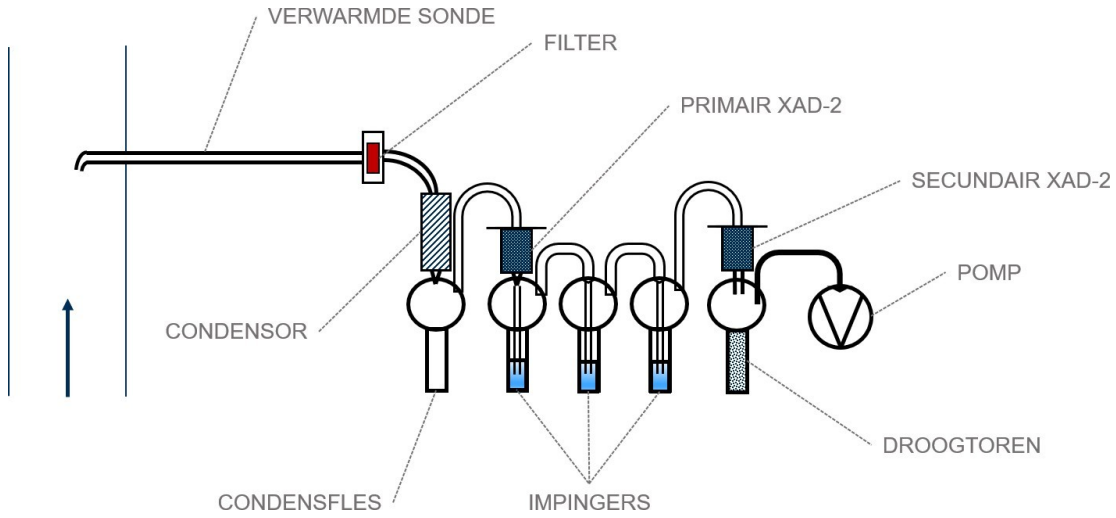
8.2 SAMPLING VARIANTS

8.2.1 OTM-45 VARIANT SETUP

This sampling train includes the same measurement principle, components and treatment as the OTM-45 method (Section 2.1) but captures the condensate in a condensate bottle before the gas stream is passed over the primary XAD-2 cartridge (see diagram below in Figure 2). After the primary XAD-2 cartridge, three impingers and a secondary XAD-2 cartridge (breakthrough) are switched in. The OTM-45 variant setup includes:

- a heated probe with quartz fibre filter
- a condenser
- an empty condensate bottle to absorb the condensate
- a primary XAD2 cartridge (start)
- a series of 3 impingers filled with ultrapure water

- a secondary XAD2 cartridge (end)
- a drying tower (silica or equivalent desiccant) with pump



VERWARMDE SONDE	HEATED PROBE
FILTER	FILTER
PRIMAIR XAD-2	PRIMARY XAD-2
SECUNDAIR XAD-2	SECONDARY XAD-2
POMP	PUMP
DROOGTOREN	DRYING TOWER
IMPINGERS	IMPINGERS
CONDENSFLESSEN	CONDENSATE BOTTLES
CONDENSOR	CONDENSER

Figure 2 Schematic representation of the OTM-45 variant setup

The sampling train consists mainly of borosilicate or quartz glass (probe, impingers, filter holder) and although Teflon (PTFE) should be avoided as much as possible (risk of contamination), it may be used in connectors, O-rings and/or coatings.

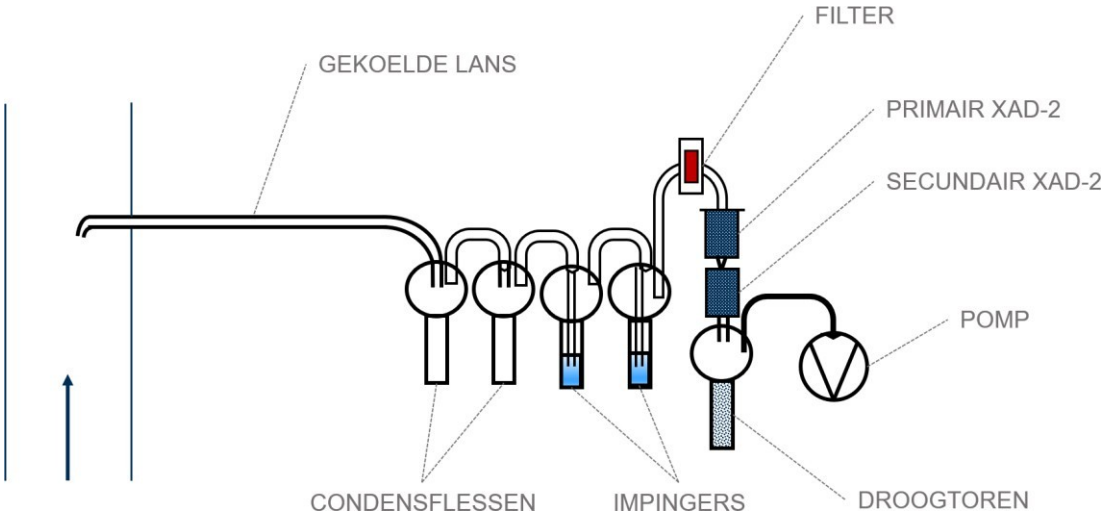
Recovery of the samples takes place in existing cartridges (e.g. XAD2 cartridges), polypropylene (PP) or polyethylene (HDPE) bottles and results in seven different samples (Section 3.5).

8.2.2 COOLED LANCE SET-UP

This sampling train provides isokinetic sampling by means of a cooled lance, after which two condensate bottles capture the condensate (see diagram below Figure 3). The resulting dry flue gases then pass over a glass wool filter, a primary XAD-2 cartridge, two impingers and a secondary XAD-2 cartridge. Any deposition of particulate PFAS in the condensate is taken into account by an additional filtration after which the residue is added to the filter language and the filtrate to the condensate. The cooled lance setup includes:

- a cooled lance
- two condensate bottles to absorb the condensate
- two impingers filled with ultrapure water
- a glass wool filter (or on top of XAD2)
- a primary XAD-2 cartridge
- a secondary XAD-2 cartridge

- a drying tower (silica or equivalent desiccant) with pump



GEKOELDE LANS	COOLED LANCE
FILTER	FILTER
PRIMAIR XAD-2	PRIMARY XAD-2
SECUNDAIR XAD-2	SECONDARY XAD-2
POMP	PUMP
DROOGTOREN	DRYING TOWER
IMPINGERS	IMPINGERS
CONDENSFLESSEN	CONDENSATE BOTTLES

Figure 3 Schematic representation of the cooled lance setup

The sampling train consists mainly of borosilicate or quartz glass (probe, impingers, filter holder) and although Teflon (PTFE) should be avoided as much as possible (risk of contamination), it may be used in connectors, O-rings and/or coatings. Recovery of the samples takes place in existing cartridges (e.g. XAD2 cartridges), polypropylene (PP) or polyethylene (HDPE) bottles and results in seven different samples (Section 3.5).

9 REFERENCES

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10 APPENDIX

Appendix 1: Samples for recovery in a PFAS emission measurement, including medium blank, field blank and re-rinse blank.

S1	MEDIUM BLANK	blank filter
S2		blank rinse from liquid methanol
S3		blank rinse from liquid cyclohexane
S4		blank XAD2
S5		blank water
S6	FIELD BLANK	Rinse from nozzle + probe + filter front (MeOH) – field blank
S7		filter - field blank
S8		XAD2-1+spike - field blank
S9		impinger water - field blank
S10		Rinse from filter back + condenser + condensate bottle + impinger (MeOH) - field blank
S11		Rinse from nozzle + probe + filter holder + condenser + condensate bottle (Cyclohex) - field blank
S12	SAMPLE	Rinse from nozzle + probe + filter front (MeOH)
S13		Rinse from nozzle + probe + condenser + condensate bottle (Cyclohex)
S14		Filter
S15		rinse from filter back + condenser + condensate bottle + impingers (MeOH)
S16		Primary XAD2 (SS spike)
S17		Condensate + Impinger 1-2-3 water
S18		Secondary XAD2
S19	NASPOE LBLANC O	Re-rinse from probe + condenser + condensate bottle + impingers (MeoH)
S20		Re-rinse from probe + condenser (CycloHex)

Appendix 2: Used isotope enriched PFAS as sampling standards (SS)

		COMPONENT	
SS	SS1	Perfluoro-n-(13C8)octanoic acid (M8PFOA)	$^{13}C_8$-PFOA
	SS2	Sodium perfluoro-1-[13C8]octanesulfonate (M8PFOS)	$^{13}C_8$-PFOS
	SS3	Perfluoro-n-(2.3.4-13C3) butanoic acid (M3PFBA)	$^{13}C_3$-PFBA
	SS4	Perfluoro-n-[1,2-13C2]-undecanoic acid (M2PFUdA)	$^{13}C_2$-PFUnDA
	SS5	Perfluoro-n-[1,2-13C2]-dodecanoic acid (M2PFDoA)	$^{13}C_2$-PFDoDA

Notice:
Variants of some isotope-tagged compounds are also available (e.g. $^{13}C_4$-PFBA, $^{13}C_4$-PFOA,$^{13}C_8$-PFOS, $^{13}C_9$- PFUnDA, $^{13}C_{12}$-PFDoDA,...). These variants may also be used instead of the above, as long as the sampling standards differ from the internal standards used (WAC/IV/A/025).

Performing emission measurements on open biofilters

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1 SCOPE

A number of articles in Vlarem II, Vlarem III and the accompanying BAT conclusions for certain sectors mention biofilters as a possible reduction technique for the removal of odour and volatile organic compounds, among others. Most biofilters are open-ended and do not have a ducted emission point, so the current compendium methods are usually not applicable to determining the emissions. This makes it difficult to assess the (proper) functioning of biofilters.

In this context, the present compendium method and methodology were developed for carrying out emission measurements on this type of sources.

The methodology established is independent of the parameter to be measured (e.g. total KWS concentration,
odour concentration, etc.).

Cases not included in the compendium method are:

- the sampling and analysis methods for determining the different parameters: depending on the parameter(s) to be measured, the appropriate compendium methods or standards are to be used, e.g. LUC/III/003 'Determination of the content of gaseous or total NH_3 in a gas channel' for determining the ammonia concentration, LUC/II/001 'Sampling for flue gases and analysis of CO, CO_2, SO_2, NOx, O_2 and TOC with monitors' for determining the total hydrocarbons concentration, NBN-EN 13725 'Stationary source emissions - Determination of odour concentration by dynamic olfactometry and odour emission rate' for determining the odour concentration, etc.
- the measurement time, the period during which the measurements are to be carried out, the number of measurements to be carried out, etc.

In parallel, a Code of Good Practice for the inspection and monitoring of the functioning of biofilters was drawn up, consisting of a minimum set of requirements for systematic control measurements (see document 'Code of Good Practice for the monitoring of the functioning of biofilters').

2 FUNCTIONAL DESCRIPTION OF BIOFILTRATION

In biofiltration, the air flow to be purified is passed through a fixed filter bed, which is made of biological material, e.g. compost, tree bark, root wood, peat, etc. The filter material is the carrier of a thin water film in which micro-organisms live. The pollutants in the air flow are retained by adsorption and absorption on the filter material, and then broken down by the microorganisms present. The filter material provides the nutrients that are necessary. The decomposition products of the conversion are water, carbon dioxide, sulphate, nitrate, etc. The nitrates and sulphates formed are discharged as salts dissolved in water.

The filter material naturally contains a sufficient number of different types of microorganisms for easily degradable substances. For less easily degradable substances, the filter can be inoculated with special cultures to speed up initiation of the filter. Careful follow-up is crucial to maintain the correct operating parameters.

Biofilters are operated in such a way that there is no net increase in the microorganisms (biomass). This is possible because microorganisms need energy to survive and they obtain this energy from the (accumulated) biomass. The addition of minerals and nutrients may be desirable if the filter material releases too few nutrients.

Depending on the nature of the air flows to be treated, they may need to be pretreated. Dust filters and gas scrubbers are often placed in front of the biofilter to clean the air flow (i.e. remove dust or high concentrations of acidifying components such as ammonia, amines, sulphur components,

chlorides, etc. that can poison the biofilter), but also to humidify it and bring it to the right temperature.

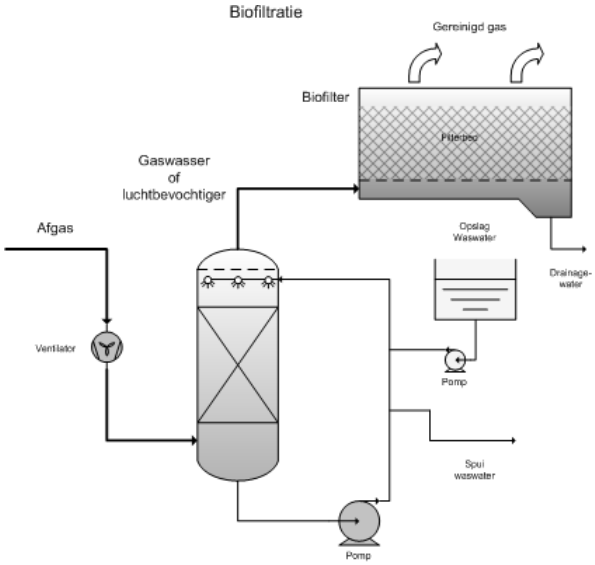


Figure 1: Schematic representation of a biofilter (emis.vito.be)

There are various types of biofilters:

- horizontal or vertical design: most biofilters are horizontal (see Figure 2). Emission takes place via a large horizontal surface. Vertical biofilters (see Figure 3) take up less space, but are more difficult to maintain (blockages, etc.), check and measure.

Figure 2: Examples of horizontal biofilters

Figure 3: Example of a vertical biofilter

- open or closed design: biofilters can be constructed as open or closed. Open biofilters are

usually 1 to 1.5 m deep and are generally larger than closed biofilters. Open biofilters are exposed to rain, sun, frost, etc. This can cause plant growth, resulting in a biofilter that is too wet in case of heavy rain or too dry in case of sun. A closed biofilter is more shielded from the external weather influences and can therefore be better controlled. The disadvantage of the closed design is that access is limited, which makes monitoring more difficult (e.g. visual inspection, etc.). A closed biofilter has one or more emission points and can therefore be considered a point source rather than a surface source. on these kinds of sources is not described in this procedure, but in other compendium methods (e.g. LUC/0/001 'Measurement site in the gas channel' and LUC/0/005 'Essential quality requirements for emission measurements').

Figure 4: Open biofilter (left) and closed biofilter (right)

3 PRINCIPLE

The emission determination consists, on the one hand, of a flow rate measurement and, on the other hand, of a concentration measurement. The flow rate measurement is preferably performed at the inlet tube(s) to the biofilter. To carry out the concentration measurements, (part of) the biofilter is covered. Under the cover, a quantity of air flows to an opening provided in the cover or to a fitted stack. The concentration measurement is to be carried out at this spot.

There are three ways to create the cover:

- fully cover the biofilter (preferred method, if possible)
- partially cover parts of the biofilter surfaces
- using a sampling hood

The method is based on the German standard 'VDI 3880 – Olfactometry – Static sampling' and the European standard for olfactometry NBN EN 13725:2022 'Air quality – Determination of odour concentration by dynamic olfactometry'. The various covering methods were tested on a horizontal biofilter for VOC removal and/or a vertical biofilter for odour removal (see Annex 1 and Annex 2).

4 METHODOLOGY

4.1 PRIOR SITE VISIT

A prior site visit to the biofilter is recommended. The main purpose of this site visit is to select the appropriate covering method and to check whether the biofilter is easily accessible and whether a ladder or other equipment is required to ensure accessibility. The characteristics of the biofilter are recorded: dimensions, type of biofilter material, supplier of the installation, visual inspection of the surface. During this prior visit, the appropriate arrangements for the actual measurements can also be made with the operator. It is recommended to clarify the visit with photographs.

4.1.1 VISUAL INSPECTION

Before measurements are taken, a visual inspection of the surface of the biofilter must always be carried out. This can be done for the first time during a prior site visit (see 4.1).

The visual inspection of the surface of the biofilter allows any abnormalities that could lead to reduced performance of the biofilter to be identified. Examples include:

- subsidence of the filling material or incomplete filling, which can cause holes in the biofilter material and preferential air flow (see, for example, Figure 5)
- the presence of dry areas that indicate a locally poorly functioning humidification
- the presence of blockages or visible salt formation in the biofilter material
- the presence of leaks at the edges of the biofilter
- the presence of weeds on the biofilter surface (see Figure 5)
- any decomposition/degradation/rotting of the biofilter material, etc.

These deviations will result in uneven air distribution, whereby part of the air to be treated shall be emitted into the atmosphere less purified or unpurified. Moreover, since the air is looking for the path of least resistance, the emission along openings/holes shall be even greater, so that this effect shall be even more pronounced.

If any deviations are found during the visual inspection, they should be reported to the operator. These should preferably be rectified by the operator before the measurements are taken.

Figure 5: Presence of holes in the biofilter material (left) and appearance of vegetation on the biofilter (right)

4.2 HOMOGENEITY CHECK

Before proceeding with the measurements, depending on the chosen covering method or in function of the choice of covering method, a homogeneity check of the emitting surface can be carried out. Since it is preferable to take into account as large a part of the surface as possible during the actual measurement, the homogeneity check is recommended but not mandatory.

The check can be carried out using:

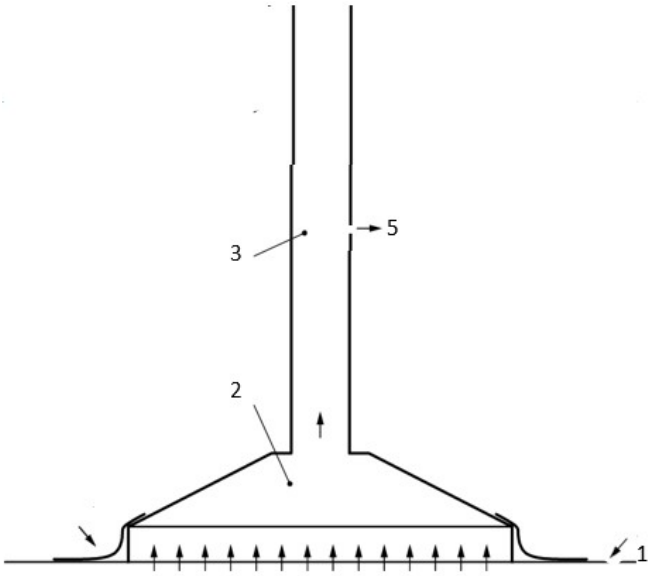
- flow rate measurements
- concentration measurements for certain chemicals (e.g. TOC with portable FID, etc.). It is not possible to perform odour measurements to determine homogeneity.

4.2.1 HOMOGENEITY CHECK BY FLOW RATE MEASUREMENTS

The homogeneity check may be carried out by performing velocity/flow rate measurements at different locations on the biofilter surface. For this purpose, a cone- or pyramid-shaped sampling hood with a surface area of at least 1 m² is used on which an attachment is placed for converting the outgoing air flow into a more ducted emission (see Figure 6). The length of the attachment must be at least 6 times the diameter. A measurement aperture must be placed in the attachment in such a way that both upstream and downstream of this aperture the length of the tube/piece of the attachment is equal to at least three times the diameter. A velocity measurement is carried out at this measuring opening using a calibrated vane anemometer or hot-wire anemometer. When carrying out the measurement, wind impact must be avoided, for example by using an attachment that is of sufficient length.

The gas velocity in the tube must be high enough to be measured with a calibrated vane anemometer (from 0.5 m/s) or a hot-wire anemometer (from 0.3 m/s). However, to prevent the build-up of back pressure under the sampling hood and the occurrence of leaks, the velocity in the attachment must not exceed 3 m/s.

To ensure a good seal at the biofilter surface and to prevent leaks, the sampling hood must be fitted with flaps.



1: biofilter surface
2: sampling hood
3: attachment
4: flap to prevent leaks
5: measuring aperture for speed measurement

Figure 6: Schematic representation of sampling hood (based on VDI3880)

To perform the homogeneity check, the biofilter is divided into a number of cells of equal size that are evenly distributed across the biofilter surface (see Figure 7). Within each cell, the sampling hood is placed in one location for the flow rate measurements. The sampling hood is positioned in such a way that the corners and edges of the biofilter are also taken into account.
The minimum number of cells to be considered in relation to the total biofilter surface area is shown in Table 1. As a guideline, a corner or edge measurement should be carried out in at least 25 % of the cells.

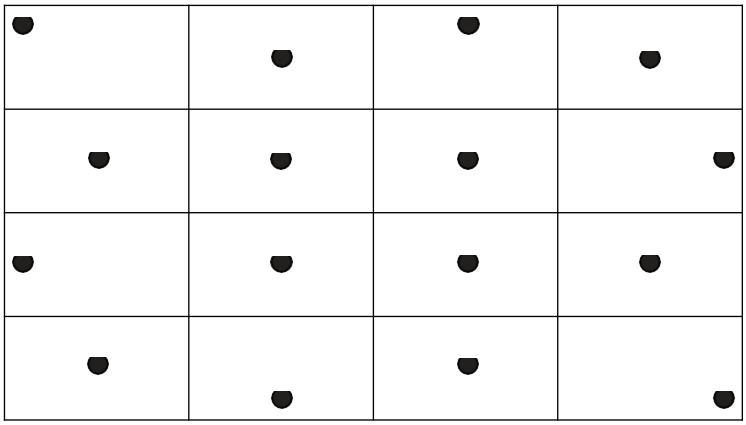


Figure 7: Example distribution of measurement locations for flow rate determination across the biofilter surface (points on the edge/corner must be taken into account)

Table 1: Minimum number of measuring points as a function of biofilter surface area

Surface of the biofilter	Min. number of measuring points for the flow rate determination
< 100 m²	10
100-2000 m²	9 + 1 per 100 m²
> 2000 m²	30

Any differences in the measured velocity or flow rate provide information about whether or not the biofilter is homogeneous. If the ratio between the maximum measured velocity/maximum flow rate and the minimum measured velocity/minimum flow rate is greater than a factor of two, the air flow is considered to be a non-homogeneous emitting source.

4.2.2 HOMOGENEITY CHECK BY CONCENTRATION MEASUREMENTS

An alternative method for assessing homogeneity is to carry out concentration measurements at several points on the biofilter surface. This can only be done if measuring equipment is available that can accurately determine the concentrations in the air leaving the biofilter. An example of this is the use of a portable FID or PID on a biofilter for the removal of VOCs.

The measurements are carried out as described in Section 4.2.1, i.e. using a sampling hood placed at various locations on the biofilter surface. The minimum number of measuring locations is shown in Table 1. The concentration measurement is taken at the height of the measuring opening in the attachment. The measurement is started when the air under the sampling hood has been refreshed at least five times.

The differences in the measured concentrations provide information about whether or not the biofilter is homogeneous. When the relative standard deviation of the measured concentrations is greater than 30 %, the biofilter must be considered a non-homogeneous emitting source.

4.3 DETERMINATION OF THE FLOW

The flow rate of the air flow to be treated is measured at the inlet tube(s) of the biofilter, if technically feasible. The measurement must be carried out in accordance with the compendium method ‘Measurement of gas velocity and volumetric flow rate in a gas channel’ (LUC/0/004). It is assumed that the flow rate of the outgoing air flow is equal to the flow rate of the incoming air flow.

If it is not technically possible to measure the flow rate at the inlet tube(s) of the biofilter, the flow rate is determined on the basis of the fan characteristics of the fan(s) present.

A third option is to carry out flow measurements at different points on the biofilter surface using the method described in point 4.2.1.

In this way, the volumetric flow rate representative of the surface area of the sampling hood at the relevant location is determined for each measuring point. The average volumetric flow rate is then calculated from the different measuring points. For the total volumetric flow rate, the calculated average volumetric flow rate is multiplied by the total surface area of the biofilter divided by the surface area of the sampling hood.

Since the velocity measurement in the sampling hood attachment can be influenced by the hood, the flow rate determined using this method is usually an underestimate and this technique can only be permitted if the other two methods are not technically feasible.

4.4 CONCENTRATION MEASUREMENT

The concentration measurement is performed by completely or partially covering the biofilter and measuring the concentration at an aperture/stack in the cover. The following methods can be used for the cover (listed in order of priority):

- fully cover the biofilter (preferred method, if possible)
- partially cover parts of the biofilter surfaces
- using a sampling hood

4.4.1 FULLY COVERING THE BIOFILTER

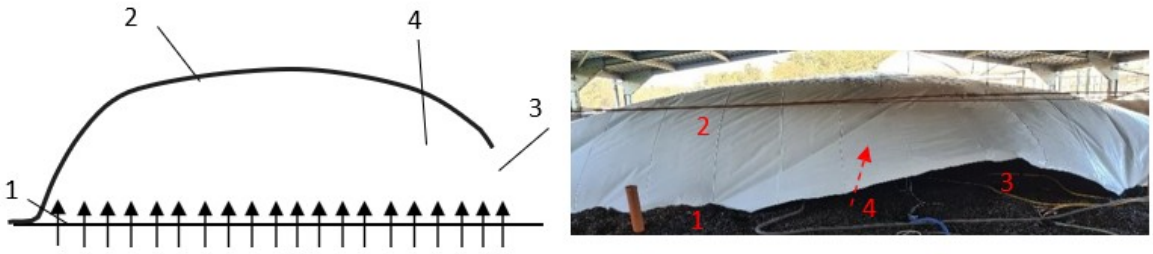
If technically and practically possible, the entire biofilter should be covered with an inert (and, when odour measurements are being carried out, odour-free/low-odour) tarpaulin (see Figure 8).

Points to consider when covering:

- the covered surface must be representative of the total surface area (the edges must also be covered)
- leaks at the edges of the cover must be prevented, e.g. by using sandbags or planks or by covering the edges of the cover with biofilter material.
- pressure build-up under the covered surface must be avoided as much as possible. To this end, one of the sides of the tarpaulin is left open so that air can flow out through this side. Wind impact is avoided by placing the open side of the tarpaulin on the downwind side of the biofilter.

The concentration measurement or sampling of the biofilter air takes place under the cover in a level area in front of the outlet opening (= the open side). In this way, a concentration measurement is carried out that is representative of the entire biofilter.

The concentration measurement is carried out in accordance with a compendium or standard method applicable to the parameter in question.



1: biofilter surface
2: tarpaulin
3: open side tarpaulin
4: location of sampling/concentration measurement

Figure 8: Concentration measurement biofilter with a tarpaulin

4.4.2 PARTIALLY COVERING THE BIOFILTER

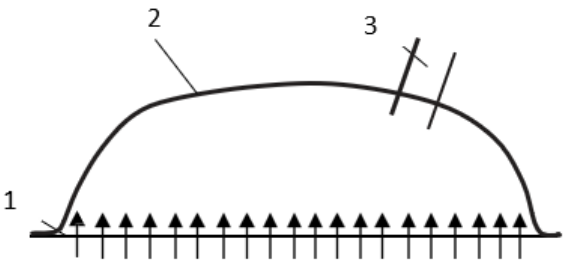
If it is not possible to cover the entire biofilter (e.g. if the surface area is too large or wind speeds are too high), it is sufficient to cover as much of the surface area as possible. When using this method, the homogeneity of the biofilter is checked in a first step as described in Section 4.2.

For homogeneous biofilters, at least 25 % of the biofilter surface must be covered with a tarpaulin (see Section 4.4.1) or a tent (see Figure 9). This can be done with a single cover or with several (consecutive) covers. At each covering, the concentration measurement is carried out in accordance with a compendium or standard method applicable to the parameter in question. When using a tarpaulin, the measurement is taken under the tarpaulin in a level area in front of the outlet opening (= the open side; see Figure 8). When a tent is used, the concentration measurement is taken at the height of a small stack.

In the case of non-homogeneous biofilters, the biofilter surface is divided into homogeneous sub-surfaces. For each sub-surface, at least 25 % of the surface is covered by means of a tarpaulin or a tent. At each covering, the concentration measurement is carried out in accordance with a compendium or standard method applicable to the parameter in question.

When partially covering the biofilter with a tent or tarpaulin, the same points should be taken into account as when completely covering the biofilter:

- the covered surface must be representative of the total surface area (in some cases, the edges must also be covered)
- leaks at the edges of the cover or tent must be prevented by, for example, using sandbags or planks or covering the edges of the cover with biofilter material.
- pressure build-up under the covered surface must be avoided as much as possible. When using a tarpaulin, this is done by leaving the downwind side of the tarpaulin open; when using a tent, this is done by using a small stack with a sufficiently large diameter.

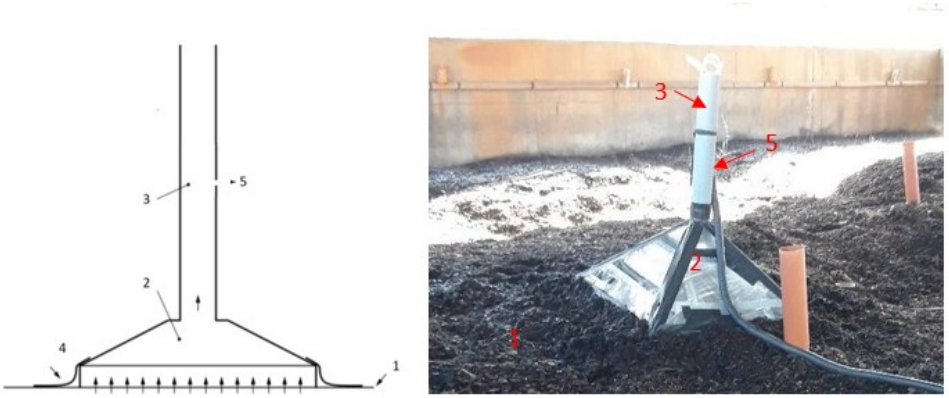


1: biofilter surface
2: tent
3: drain tube/stack = sampling/concentration measurement location

Figure 9: Concentration measurement at a biofilter using a tent

4.4.3 USE OF SAMPLING HOOD

In the third method, the measurement/sampling is carried out using a pyramid- or cylinder-shaped sampling hood with a surface area of at least 1 m², on which an attachment is placed to convert the outgoing gas stream into a more conductive emission. The sampling or concentration measurement is carried out at the opening in the attachment in accordance with a compendium or standard method applicable to the parameter in question.



1: biofilter surface
2: sampling hood
3: attachment
4: flap to prevent leaks
5: measuring aperture for speed measurement

Figure 10: Concentration measurement at a biofilter using a sampling hood

Even when a sampling hood is used, the homogeneity of the biofilter is checked in a first step as described in point 4.2.

For homogeneous biofilters, the concentration measurement is carried out as described in point 4.2.1: the biofilter is divided into a number of cells of equal size that are evenly distributed over the biofilter surface (see Figure 7). During the measurement, the sampling hood is placed successively on the different cells/sub-areas. Within each cell, the sampling hood is placed in one location for the performance of the measurements or the taking of a sample. The sampling hood is placed in such a way that both the corners and the edges of the biofilter are taken into account. The minimum number of cells to be considered in relation to the total biofilter surface area is shown in Table 1.

In the case of non-homogeneous biofilters, the biofilter surface is divided into homogeneous sub-surfaces. The method described above for homogeneous biofilters is applied to each sub-area.

When using a sampling hood, the following points should be taken into account:

- the covered surface must be representative of the total surface area (in some cases, the edges must also be covered)
- leaks at the edges of the sampling hood must be prevented by digging in the edges of the hood or providing flaps on which, for example, biofilter material or sandbags can be placed.
- when moving the sampling hood, the sampling/measurement is interrupted. After moving the sampling hood, the sampling/measurement is resumed after the air under the hood has been refreshed at least five times.

The sampling/measurement duration per sub-area is determined based on the measurement to be performed and is not part of this compendium method

Given that the sampled area is considerably smaller than in the previous methods, this method is only permitted if the two methods mentioned above cannot be applied for technical reasons.

5 EQUIPMENT

5.1 MATERIALS TO USE

All parts of the equipment that come into contact with the gases, including seals and flexible connectors, must be inert and impermeable and have a smooth surface. The materials used for odour measurements must also be odour-free. Suitable materials include PTFE (polytetrafluoroethylene), FEP (tetrafluoroethylene hexafluoropropylene copolymer), PET (polyethylene terephthalate, Nalofan) and (coated) stainless steel.

5.2 MATERIAL FOR SAMPLING

5.2.1 SAMPLING HOOD

The sampling hood is made of one of the materials listed in point 5.1. Aluminium may also be used as a material. If necessary, the inside of the pyramid may be covered with an odour-free film.

5.2.2 TARPAULIN

When covering the surface, a light, non-permeable or low-permeability, odour-free or low-odour plastic film that does not tear easily must be used. Agricultural film is a possible material for this purpose.

Note: the materials required for performing the flow and concentration measurements are described in the applicable compendium methods or standards and are not included here.

6 CALCULATIONS

The emission concentration and the mass flow rate are determined as follows:

6.1 EMISSION CONCENTRATION

For homogeneous biofilters, the concentration is determined as the average of the measured concentrations.

For non-homogeneous biofilters, a flow-weighted average concentration is calculated.

6.2 MASS FLOW RATE

When a complete cover is used, the mass flow rate is calculated as the product of the total volumetric flow rate (determined in accordance with 4.3) and the average concentration is measured during the measuring period.

When a partial cover or sampling hood is used, the mass flow rate is calculated as the product of the total volumetric flow rate (determined in accordance with 4.3) and the average of the measured concentrations at the different covers
/sampling hoods.

Volume flow rates and concentrations are expressed under normal conditions, but these may be

different depending on the parameter to be determined. For odour, the concentrations and flow rates are expressed, for example, at 20 °C and 1013 mbar.

7 FOCUS POINTS

- The determination of the flow rate is always done (if possible) at the level of the inlet tube(s) of the biofilter.
- The sampling method is chosen according to the homogeneity and accessibility of the biofilter. The entire biofilter should preferably be covered. If this is not technically or practically possible, a sufficiently large surface area must be covered.
- When covering, always cover the edges of the biofilter as well.
- When covering, the edges of the tarpaulin, tent or sampling hood must always be sealed.
- A description of the measurement method used must be included in the report. Clarification through photographs is recommended.
- Deviations from this compendium procedure and the reasons for this need to be substantiated and included in the measurement report.

8 LITERATURE AND REFERENCES

Compendium for air sampling, measurement and analysis (LUC,) Method LUC/0/001, measurement site in the gas channel

Compendium for air sampling, measurement and analysis (LUC), Method LUC/0/004, measurement of gas velocity and volumetric flow rate in a gas channel

Compendium for air sampling, measurement and analysis [LUC], Method LUC/0/005, Essential quality requirements for emission measurements

Compendium for sampling, measurement and analysis of air (LUC), Method LUC/II/001, Sampling for flue gases and analysis of CO, CO_2, SO_2, NOx, O_2 and TOC with monitors

Compendium for sampling, measurement and analysis of air (LUC), Method LUC/III/003, Determination of gaseous or total NH_3 content in a gas channel

NBN EN13725:2022 – Stationary source emissions - Determination of odour concentration by dynamic olfactometry and odour emission rate

NTA 9065:2012 en
Air quality - Odour measurements - Odour measurement and calculation

VDI 3880: 2011
Olfactometry – Static Sampling

Code of Good Practice Code of Good Practice for monitoring the operation of biofilters (May 2025)

ANNEX 1 MEASUREMENTS ON A HORIZONTAL BIOFILTER FOR VOC REMOVAL

1. SITUATION

In order to examine the applicability of the various covering methods and to determine whether there are significant differences between the implementation of different measurement strategies, measurements were carried out on an open horizontal biofilter used to remove VOCs in a company that produces paint and varnish, among other products. The measurements took place in March 2022.

2. DESCRIPTION OF BIOFILTER

2.1 BIOFILTER

An overview of the biofilter is shown in Figure 11. The covered biofilter has a surface area of ± 400m m^2 and is loaded with a flow rate of ± 30000 m^3/h. The operator installed seven measuring points on the biofilter, which are used to carry out control measurements by a recognised laboratory. The measuring points consist of plastic tubes with a diameter of ± 15 cm and a height of ± 50 cm, installed vertically in the biofilter (to a depth of 10 to 20 cm below the biofilter surface). The tubes mainly serve to prevent wind from influencing the measurements.

Figure 11 : Horizontal biofilter for VOC removal

The location of the measuring points used by the company is shown in Figure 12 (schematic view) and Figure 13 (photo biofilter surface).

Figure 12 : Overview of the biofilter with measuring points 1 to 7 (left: schematic representation; right: photo; numbers = numbering of measuring points by VITO)

As a result of several wooden beams supporting the biofilter material breaking, the filling material of the biofilter had sunken in a number of places. The largest subsidence occurred at measuring point 4, between measuring points 2 and 6, near measuring points 1 and 5 and behind measuring point 7. The photos below show the approximate location of the subsidence.

Figure 13: Subsidence at measuring point 4 (left) and between measuring points 1, 2, 5 and 6 (right)

Figure 14: Subsidence behind measuring point 7

2.2 Input airflow

The air to be treated is fed in via a single tube and distributed across a number of channels at the bottom of the biofilter. The channels contain fixed baffles that ensure that the air flow is evenly distributed across each channel.

As mentioned above, due to the breaking of a number of wooden beams supporting the bark, a number of subsidence areas were present in the biofilter during the measurements. This may have resulted in an uneven distribution of the inflows and therefore also of the outflows.

3. Measurement method

The measurements were carried out using FID analysers (flame ionisation detectors), which enable continuous monitoring of the total hydrocarbon content (THC) at the inlet and at various measuring locations on the biofilter.

The hydrocarbon monitors are JUM type FID 3-300 with a measuring range of 0 to 10 000 ppm. A mixture of H_2 and He in a ratio of 40/60 is used as burner gas. The FIDs are calibrated on site with a calibration gas with a concentration of ± 90 ppm propane in nitrogen. The control gas has a concentration of ± 30 ppm propane in nitrogen.

The measurements are carried out in accordance with the compendium procedure LUC/0/005 – LUC/II/001 and according to VITO procedure MIM-LU-007: 'Determination of the gaseous emission of the total hydrocarbon content'. The flue gas is drawn into the monitor through a Teflon tube heated to 180 °C. This setup is shown schematically in Figure 15.

Verwarmde leiding

FID-monitor

Data-logger

PC

Heated tube

FID monitor

Data logger

PC

Figure 15: Schematic representation of the setup for determining the hydrocarbons concentration

Three FIDs were used simultaneously:

- FIDin: continuously installed at the inlet
- FID1: usually used as a fixed measuring point in different situations
- FID2: mainly used for scanning at different points

The FIDs used for measurement at the outlet of the biofilter were located at the bottom of the biofilter. Two heated tubes of the same length were used to carry out the measurements at the various points on the biofilter surface, ensuring that there was no difference in response time between the two measuring systems.

4. MEASUREMENTS CARRIED OUT

During three measurement days, the practical feasibility of the various methods was investigated and it was determined whether there were significant differences between the implementation of different measurement or covering methods. The following measurement strategies were used:

- point measurement at the current measurement locations (not part of the compendium method, but carried out by the operator)
- measurement using a sampling hood/cap with a surface area of 1m^3
- sampling using a sampling tent with a surface area of 25m m^2
- sampling by partially covering the biofilter, approximately one third to one half of the surface area

The biofilter was not completely covered.
The following measurements were carried out on 8, 9 and 10 March 2022:

- 8 March: exploratory measurement to obtain an initial idea of the order of magnitude of the concentrations, etc.
- 9 March:
  - determination of the influence of covering part of the biofilter with a tarpaulin on the concentrations at the measuring points that are not covered.
  - comparison of the concentration at the outlet/opening of the tarpaulin with the concentrations at various locations under the tarpaulin
- 10 March:
  - determination of the influence of covering part of the biofilter with a tarpaulin on the concentrations at the measuring points that are not covered (= repeat test from the previous day).
  - comparison of the concentration at the outlet/opening of the tarpaulin with the concentrations under the tarpaulin (= repeat of the previous day's test).
  - determination of possible differences in the results of a point measurement, a

measurement with a sampling hood/sampling cap (1m m²) and a measurement with a 5m m x 5m m sampling tent.

4.1 MEASUREMENT DAY 1 – 08/03/2022 – EXPLORATORY MEASUREMENTS

During the first measurement day, exploratory measurements were carried out with two FIDs (FID1 and FID2) at the various points specified by the operator (1 to 6) in order to obtain an idea of the concentrations in the various zones on the biofilter.

If the response factor of both FIDs is not the same, different concentrations may be measured with both systems when the same gas is presented. For this reason, measurements were taken simultaneously with both FIDs at point 1 at the start of the measurements. Based on the difference in measured concentrations between the two FIDs, a small correction was made to the measured values of FID1.

To get an idea of the fluctuations in the total KWS concentration at the inlet, this concentration was continuously monitored during the measurements with a third FID analyser (FIDin).

The results of the exploratory measurements are shown in Table 2 and Figure 16.

Table 2: Overview of exploratory measurement results

Measuring period	Measuring point		Result [ppm]		
	FID1	FID2	FID1	FID2	FIDin
14u03 - 14u08	1	1	116	116	130
14u10 - 14u25	2	2	123	118	110
14u28 - 14u40	3	4	51	55	113
14u43 - 14u51	5	6	70	102	128
14u53 - 15u03	1	1	98	98	119

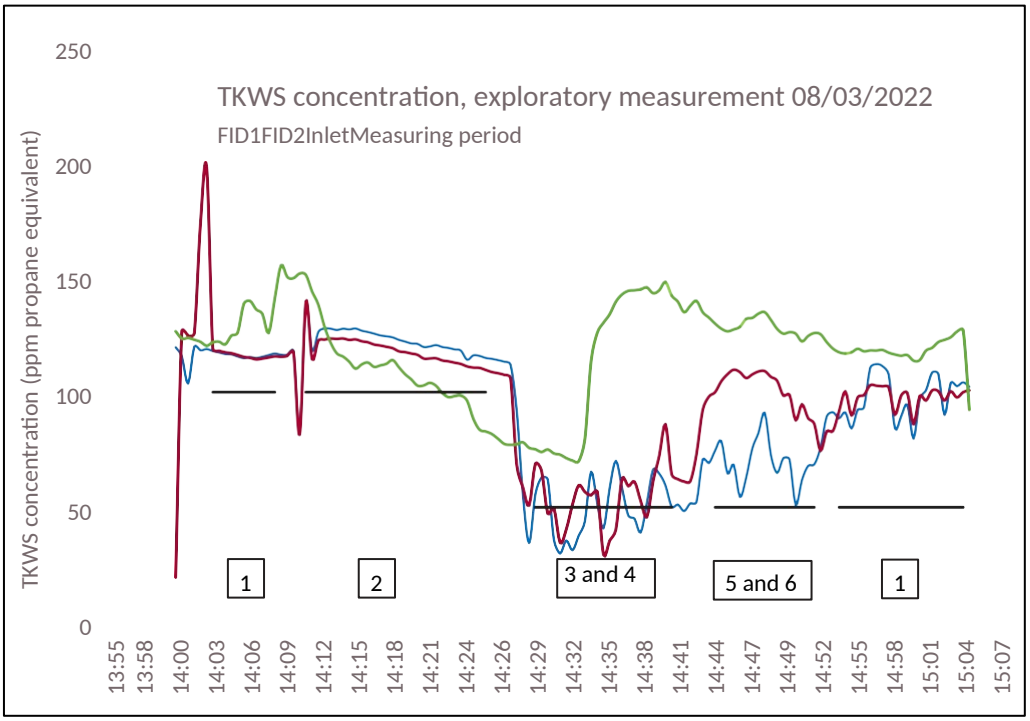


Figure 16: Total hydrocarbons concentrations during exploratory measurements on 8/3/2022

The measurement results show that significantly lower concentrations were measured at measurement locations 3, 4 and 5 than at measurement locations 1, 2 and 6, while the average concentration of the incoming air flow over the measuring periods considered was relatively stable. This is probably due to the biofilter not being homogeneous as a result of the presence of

subsidence (see point 2 'Description of biofilter').

4.2 MEASUREMENT DAY 2 – 09/03/2022

The following matters were examined during the second day of measurement:

- determination of the influence of covering part of the biofilter with a tarpaulin on the concentrations at the measuring points that are not covered.
- comparison of the concentration at the outlet/opening of the tarpaulin with the concentrations under the tarpaulin

To determine the response factor of the FIDs, FID1 and FID2 were measured simultaneously at measuring point 5. A correction was made to FID1 to account for the small deviation between the two FIDs.

4.2.1 EFFECT OF COVERING THE BIOFILTER ON THE TKWS CONCENTRATION IN INDIVIDUAL MEASURING POINTS

Since covering (part of) the biofilter may affect its performance (and therefore the concentrations at the outlet of the biofilter), an initial test was carried out to determine whether covering part of the biofilter causes concentration changes at the uncovered measuring points. In this test, measurements were first taken at two fixed points using FID1 and FID2 without covering any part of the biofilter. Measurements were then taken at the same points with part (± one third) of the biofilter covered. The test was carried out for three different situations.

Situation 1: FID1 and FID2 fixed at points 6 and 2 – measurement without covering and with partial covering of (1/3) of the biofilter (zone 3-4)

In an initial test, the TKWS concentration was measured at measuring points 2 and 6 for 20 minutes. Next, a cover was constructed over the biofilter at measuring points 3 and 4, and the effect of this on the TKWS concentrations at measuring points 2 and 6, which were not located under the cover, was examined. A schematic representation of the measuring points and the location of the covered zone are shown in Figure 17. The results of the measurements are shown in Table 3 and Figure 18.

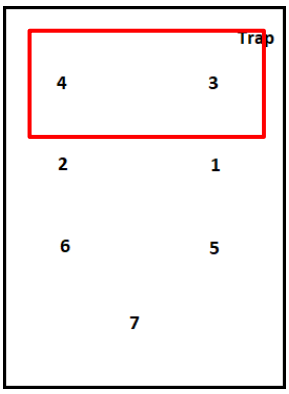


Figure 17: Overview of the measuring points and the covered zone 3-4

Table 3: Average TKWS concentrations in measuring points 2 and 6 with and without covering zone 3-4

Measuring period	Measuring point			TKWS concentration [ppm]		
	FID1	FID2	Covered zone	FID1	FID2	FIDin
9:35-9:55	6	2	no	55	80	114
9:56-10:14	6	2	3-44	59	79	110

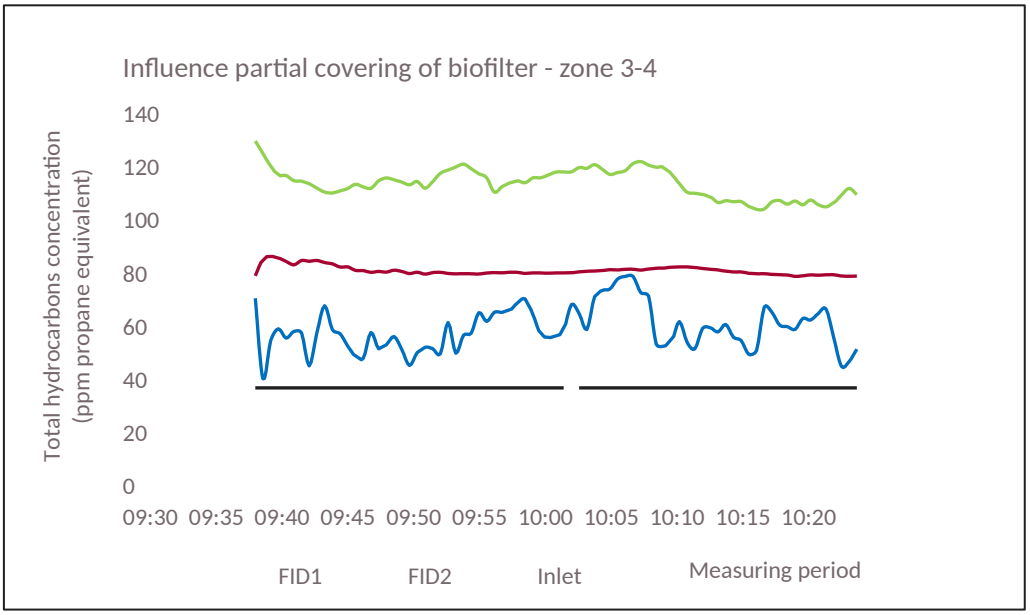


Figure 18: TKWS concentration profile in points 2 and 6 with and without covering of zone 3-4

The results show that covering the zone around measuring points 3 and 4 does not cause a significant change in the TKWS concentration at the uncovered measuring points.

Notice: During the measurements, the measurement signals from FID2 (measuring point 2) were more stable than those from FID1 (measuring point 6). No clear explanation for this was identified. Possible causes include wind impact or other effects such as inhomogeneity.

Situation 2: FID1 and FID2 fixed at points 2 and 1 – measurement without covering and with partial covering of (1/3) of the biofilter (zone 1-2-5-6)

The above test was repeated for measuring points 1 and 2, with these measuring points located under the cover during the covering process (see Figure 19). The results of the measurements are shown in Table 4 and Figure 20.

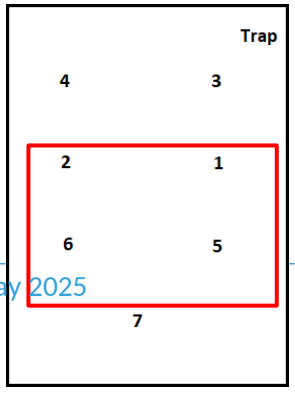


Figure 19: Overview of the measuring points and the covered zone 1-2-5-6

Table 4: Average TKWS concentrations in measuring points 2 and 6 with and without covering zone 1-2-5-6

Measuring period	Measuring point			TKWS concentration [ppm]		
	FID1	FID2	Covered zone	FID1	FID2	FIDin
10:52-11:02	2	1	no	91	92	132
11:05-11:15	2	1	1-2-5-6	87	100	135

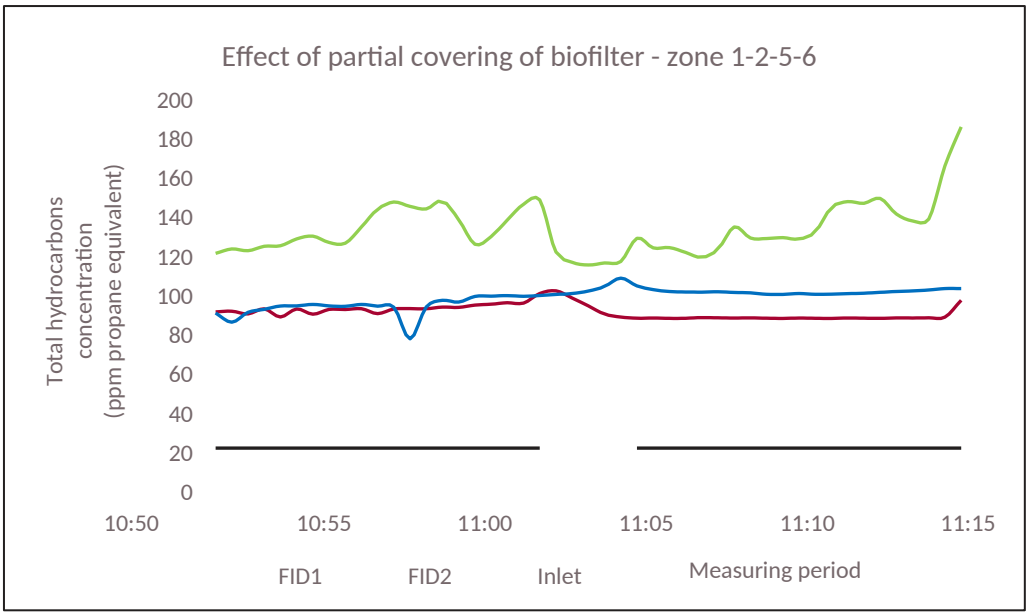


Figure 20: TKWS concentration profile in points 1 and 2 with and without covering of zone 1-2-5-6

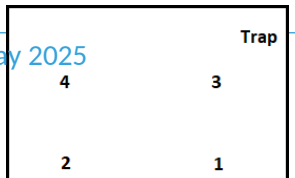
The results show that the TKWS concentration was virtually the same at both measuring points after covering. No direct explanation was identified for the increased deviation between the measured values after covering the zone. Covering part of the biofilter can therefore have an impact on the TKWS concentrations measured at specific points. It is recommended to measure at the outlet of the cover because the air flow shall be more homogeneous there (see paragraph 4.2.2).

Situation 3: FID1 and FID2 fixed at points 5 and 6 – measurement without covering and with partial covering of (1/3) of the biofilter (zone 5-6-7)

Finally, the test was repeated again for measuring points 5 and 6, with these measuring points also located under the cover of zone 5-6-7 during covering (see Figure 21). The results of the measurements are shown in Table 5 and Figure 22.

Table 5: Average TKWS concentrations in measuring points 5 and 6 with and without covering zone 5-6-7

Measuring period	Measuring point			TKWS concentration [ppm]		
	FID1	FID2	Covered zone	FID1	FID2	FIDin
11:53-12:03	5	6	no	75	83	116
12:05-12:15	5	6	5-6-7	77	93	112



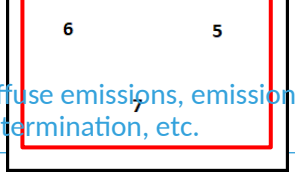


Figure 21: Overview of the measuring points and the covered zone 5-6-7

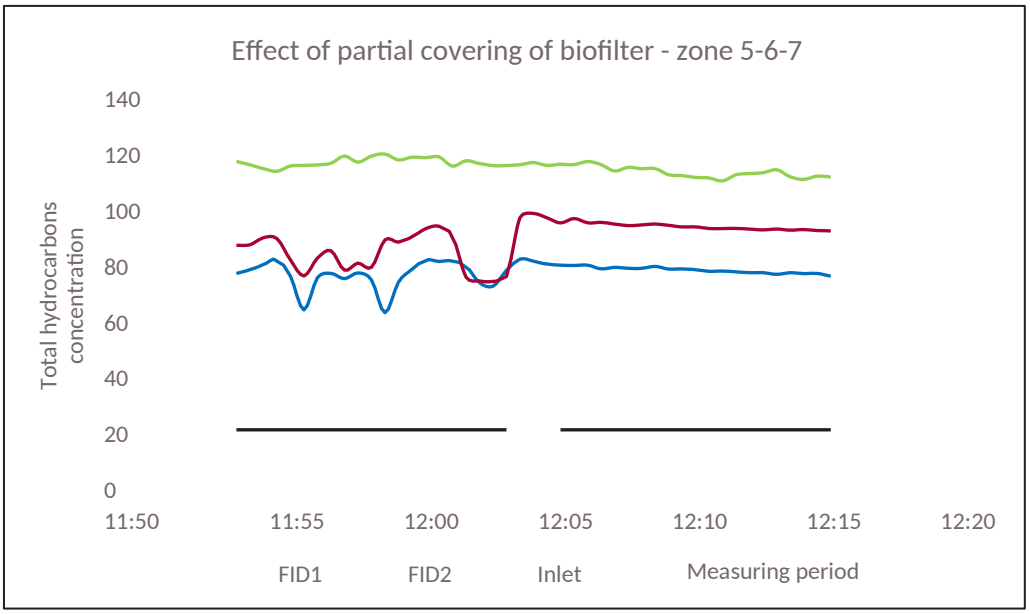


Figure 22: TKWS concentration profile in points 5 and 6 with and without covering of zone 5-6-7

This measurement also shows that after covering, the difference in TKWS concentration between the two measuring points is greater than before covering, and that covering part of the biofilter can therefore influence the TKWS concentrations at the covered measuring points.

4.2.2 Comparison of concentration at the 'outlet' of the tarpaulin with the concentrations under the tarpaulin

When using a tarpaulin to cover part of the biofilter, the measured concentration at one point at the 'outlet' of the covered zone (= the opening in the tarpaulin) is checked to see if it is comparable to the concentration at three different points under the tarpaulin. This test was carried out three times, each time covering a different part of the biofilter.

<u>Situation 1: Measuring points zone 3-4 covered</u>

The zone of measuring points 3-4 was covered (see Figure 23 and Figure 24), FID1 was set up at the outlet/opening of the tarpaulin (± 30 cm above the biofilter surface). With FID2, measurements were taken at 3 different points, left, middle and right under the tarpaulin (± 30 cm above the biofilter surface).

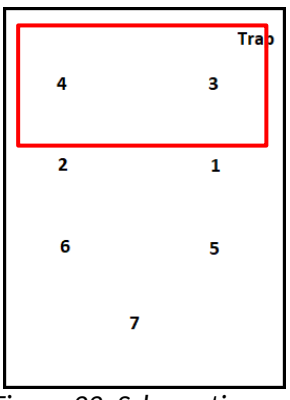


Figure 23: Schematic overview covered zone 3-4

Figure 24: Partial covering of the biofilter (zone 3-4) with a tarpaulin

The results of the measurements are shown in Table 6 and Figure 25. The figure shows the concentration profile of the measurement under the tarpaulin and at the outlet of the tarpaulin. The black lines in the figure indicate the periods during which measurements were taken on the left, centre and right under the tarpaulin.

Table 6: Average TKWS concentrations when covering zone 3-4 with a tarpaulin

Measuring period	Measuring point			TKWS concentration [ppm]		
	FID1	FID2	Covered zone	FID1	FID2	FIDin
10:16-10:26	outlet/ opening of tarpaulin	left side under tarpaulin	3-4	84	92	109
10:27-10:37		centre under tarpaulin	3-4	105	111	156
10:39-10:49		right side under tarpaulin	3-4	109	106	137
			Average:	99	103	134

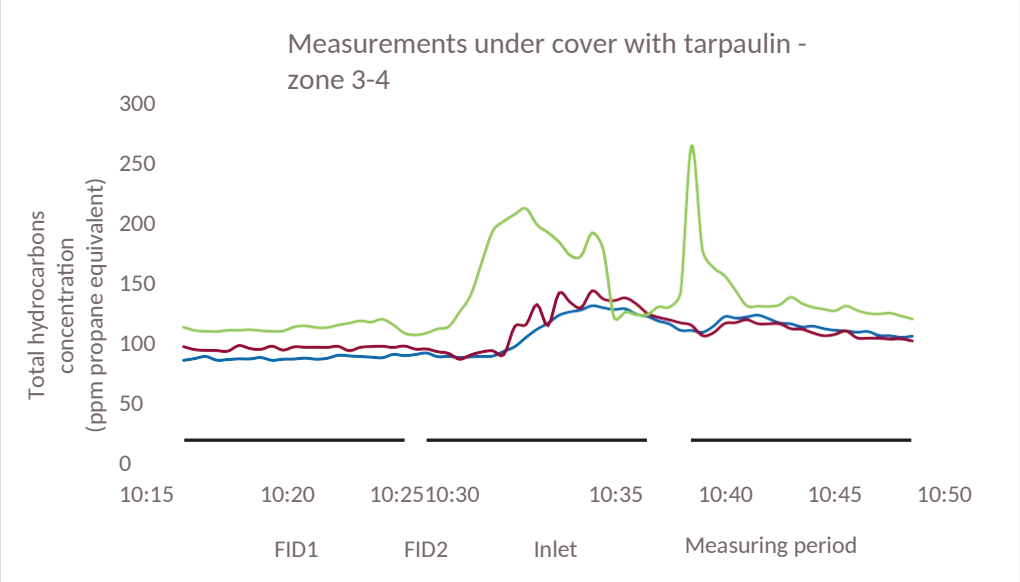


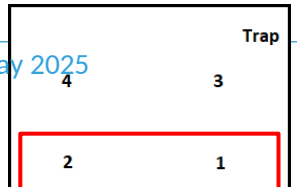
Figure 25: TKWS concentration profile at different locations under covered zone 3-4

The results show that the TKWS concentrations measured under the tarpaulin differ slightly from those measured at the opening in the tarpaulin (outlet). Slightly more fluctuations were measured under the tarpaulin (FID2) than at the outlet/opening in the tarpaulin. Since the air emitted from the covered biofilter surface is better mixed at the outlet and it is also more practical to take measurements at this location than under the tarpaulin, it is recommended that, when using a tarpaulin, the concentration measurements be taken at the opening/outlet of the tarpaulin.

Note: the measurement results show that the concentration profile at the biofilter surface (FID1 and FID2) follows the concentration profile at the inlet, but with a delay of 3 to 4 minutes.

<u>Situation 2: Measuring points zone 1-2-5-6 covered</u>

The zone of measuring points 1-2-5-6 was covered (see Figure and Figure 26), FID1 was set up at the 'outlet' of the tarpaulin (± 30 cm above the biofilter surface). With FID2, measurements were taken at 3 different points, left, middle and right under the tarpaulin (± 30 cm above the biofilter surface).



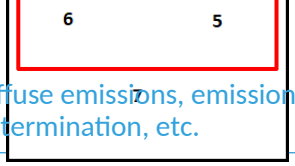


Figure 26: Schematic overview covered zone 1-2-5-6

The results of the measurements are shown in Table 7 and Figure 27.

Table 7: Average TKWS concentrations when covering zone 1-2-5-6 with a tarpaulin

Measuring period	Measuring point			TKWS concentration [ppm]		
	FID1	FID2	Covered zone	FID1	FID2	FIDin
11:16-11:25	outlet/ opening of tarpaulin	left side under tarpaulin	1-2-5-6	121	114	157
11:26-11:36		centre under tarpaulin	1-2-5-6	113	111	135
11:37-11:47		right side under tarpaulin	1-2-5-6	101	104	122
			Average:	111	110	138

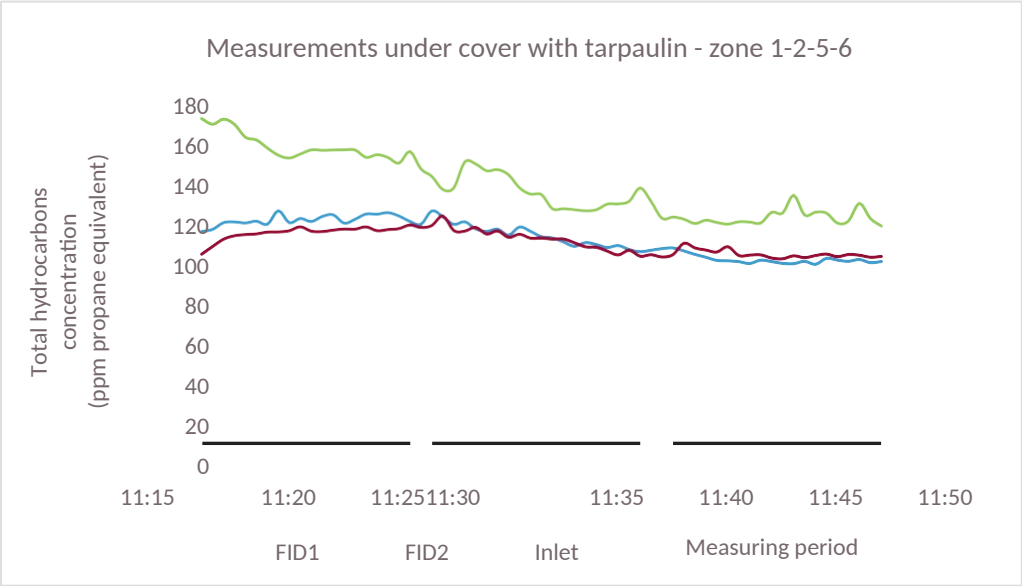


Figure 27: TKWS concentration profile at different locations under covered zone 1-2-5-6

The results show that, also with these measurements, the TKWS concentrations measured under the tarpaulin sometimes differ slightly from those measured at the opening in the tarpaulin (outlet).

Situation 3: Measuring points zone 5-6-7 covered

The zone of measuring points 5-6-7 was covered (see Figure 28), FID1 was set up at the 'outlet' of the tarpaulin (± 30 cm above the biofilter surface). With FID2, measurements were taken at 3 different points, left, middle and right under the tarpaulin (± 30 cm above the biofilter surface).

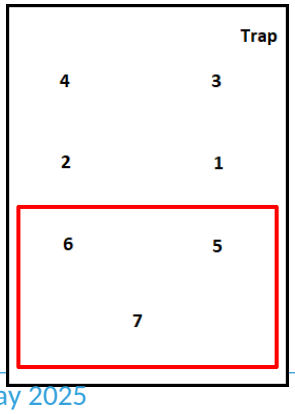


Figure 28: Schematic overview covered zone 5-6-7

The results of the measurements are shown in Table 8 and Figure 29.

Table 8: Average TKWS concentrations when covering zone 5-6-7 with a tarpaulin

Measuring period	Measuring point			TKWS concentration [ppm]		
	FID1	FID2	Covered zone	FID1	FID2	FIDin
12:16-12:26	outlet/ opening of tarpaulin	left side under tarpaulin	5-6-7	92	100	116
12:27-12:37		centre under tarpaulin	5-6-7	101	107	132
12:38-12:48		right side under tarpaulin	5-6-7	98	102	115
			Average:	97	103	121

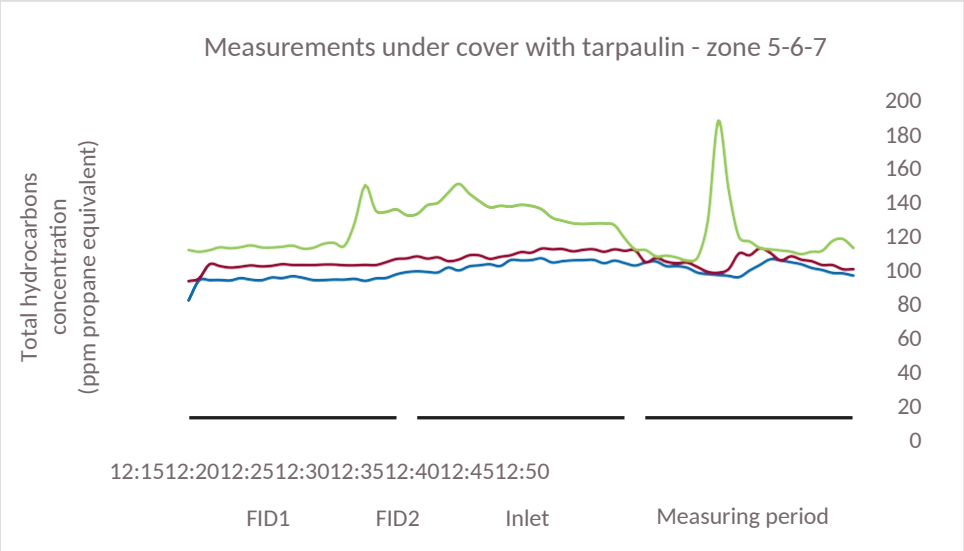


Figure 29: TKWS concentration profile at different locations under covered zone 5-6-7

The results show that the TKWS concentrations measured under the tarpaulin were always slightly higher than those measured at the opening in the tarpaulin (outlet). The deviation of 5 % may be partly due to wind impact (in addition to the uncertainty on the FID measurement, etc.).

4.3 MEASUREMENT DAY 3 – 10/03/2022

The following matters were examined during the third day of measurement:

- determination of the influence of covering part of the biofilter with a tarpaulin on the concentrations at the measuring points that are not covered (= repeat test from measurement day 2).
- comparison of the concentration at the outlet/opening of the tarpaulin with the concentrations under the tarpaulin (= repeat of measurement day 2's test).
- determination of possible differences in the results of a point measurement, a measurement with a sampling cap (1 m²) and a measurement with a 5 m x 5 m sampling tent.

At the start of the measurements, the three FIDs were measured simultaneously at the incoming airflow. Due to the very small differences in measured values, no correction factor was applied.

4.3.1 Effect of covering part of the biofilter on the TKWS concentration in individual measuring points

This test was a repeat of the tests carried out on measurement day 2, which examined whether covering part of the biofilter caused concentration changes at the uncovered measuring points (see paragraph 4.2.1). During the test, the TKWS concentration was first measured at measuring points 5 and 6 for 20 minutes. Next, a tarpaulin cover was constructed over the biofilter at measuring points 5, 6 and 7, and the effect of this on the TKWS concentrations at measuring points 5 and 6, which were also located under the cover, was examined.

A schematic representation of the measuring points and the location of the covered zone are shown in Figure 30. The results of the measurements are shown in Table 9 and Figure 31.

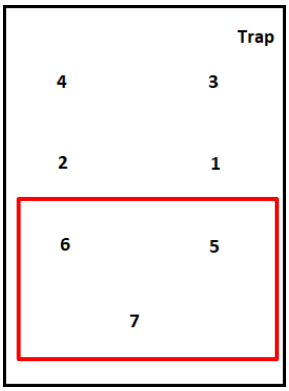


Figure 30: Overview of the measuring points and the covered zone 5-6-7

Table 9: Average TKWS concentrations in measuring points 5 and 6 with and without covering zone 5-6-7 (measurement day 3)

Measuring period	Measuring point			TKWS concentration [ppm]		
	FID1	FID2	Covered zone	FID1	FID2	FIDin
9:40-9:50	5	6	no	104	107	157
9:52-10:07	5	6	5-6-7	113	128	162

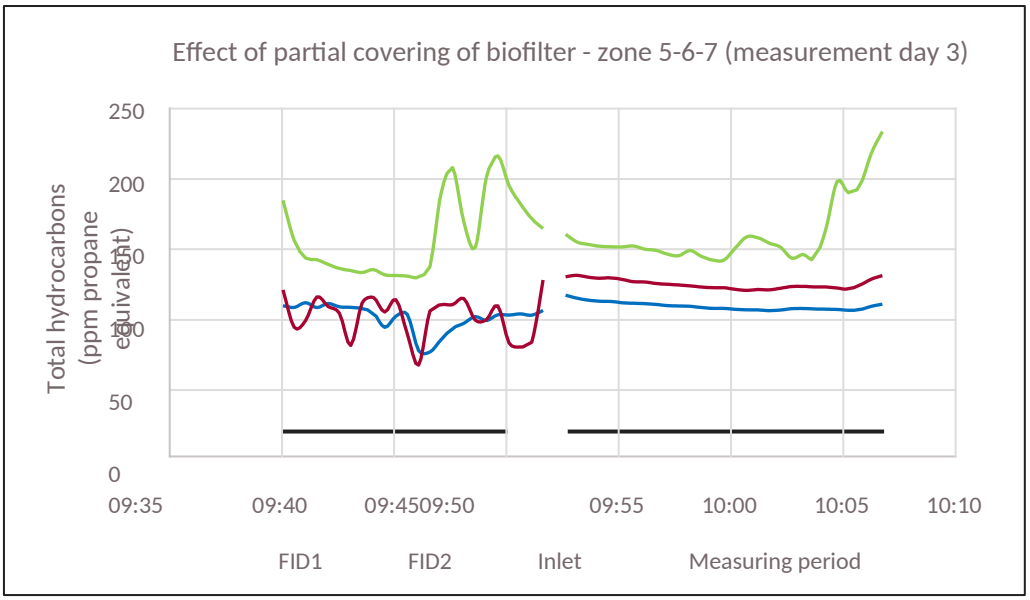


Figure 31: TKWS concentration profile in points 5 and 6 with and without covering of zone 5-6-7 (measurement day 3)

As was the case on measurement day 2, the difference in TKWS concentration between the two measuring points is greater after covering than before covering. This confirms the conclusion that covering part of the biofilter can affect TKWS emissions at specific emission points.

4.3.2 Comparison of the concentration at the outlet/opening of the tarpaulin with the concentrations under the tarpaulin

This test was also a repeat of a test carried out on measurement day 2, in which the concentrations measured at different locations (left, centre and right) under a tarpaulin were compared with the concentration at the outlet/opening of the tarpaulin (see paragraph 4.2.2). The test was carried out in the zone of measuring points 5, 6 and 7, with FID1 always sampling at the outlet/opening of the tarpaulin and the measurements being taken on the left, centre and right under the tarpaulin with FID2 (± 30 cm above the biofilter surface). The results of the measurements are shown in Table 10 and Figure 32.

Table 10: Average TKWS concentrations when covering zone 5-6-7 with a tarpaulin (measurement day 3)

Measuring period	**Measuring location**			**TKWS concentration [ppm]**		
	FID1	**FID2**	**Covered zone**	**FID1**	**FID2**	**FIDin**
10:08-10:18	outlet/ opening of tarpaulin	left side under tarpaulin	5-6-7	142	149	180
10:20-10:30		centre under tarpaulin	5-6-7	149	137	229
10:31-10:40		right side under tarpaulin	5-6-7	164	166	246
			Average:	152	151	218

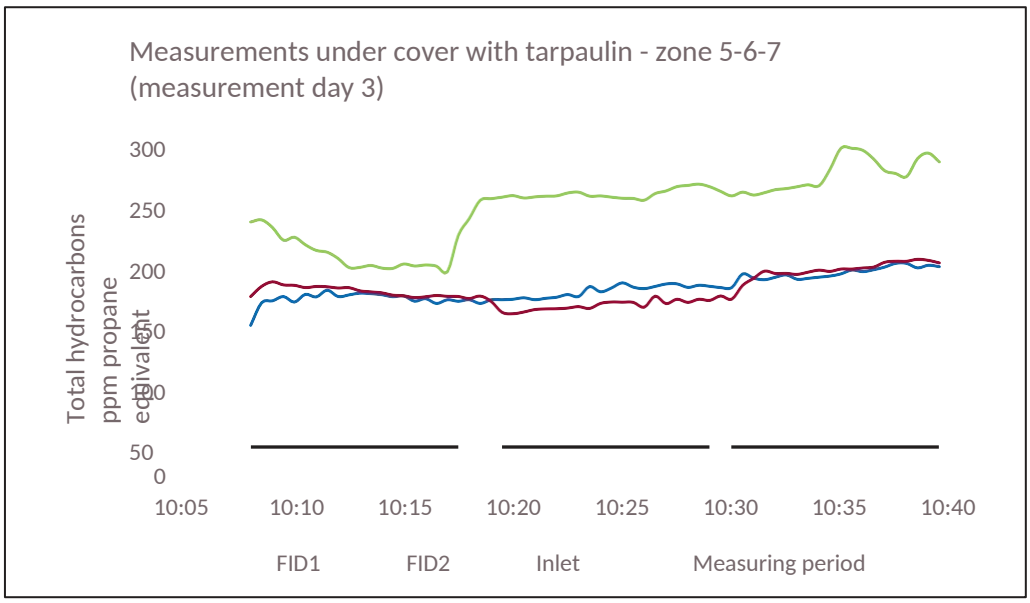


Figure 32: TKWS concentration profile at different locations under covered zone 5-6-7 (measurement day 3)

Once again, the results show that the TKWS concentrations measured under the tarpaulin sometimes differ slightly from those measured at the opening in the tarpaulin (outlet). As mentioned above, it is recommended to perform the measurements at the outlet/opening in the tarpaulin.

Note: the results also show that the removal efficiency of the biofilter is higher when the inlet concentrations are high.

4.3.3 Comparison of point measurement, measurement with sampling hood and measurement with tent

In a final test, it was examined whether differences in concentrations are observed when different sampling or covering methods are used. Three sampling methods were used for this purpose.

- point measurement in the existing tubes
- measurement with a cap/hood of 1m³
- measurement with a tent of 5 m by 5 m

The pictures below show the different measurement methods:

Figure 33: Point measurement in existing tube + sampling cap

Figure 34: Sampling tent

Figure 35: Three different sampling methods (from left to right: tent, point/tube, cap/hood)

The measurements were carried out at the level of measuring point 1. A point measurement was started, after which the sampling hood/sampling cap and the tent were placed over this measuring location. It was measured for a period of 10 minutes each time. The results of the measurements are shown in Table 11 and Figure 36.

Table 11: Average TKWS concentrations when applying different sampling /covering methods in measuring point 1

Measuring period	Measuring location		TKWS concentration [ppm]		
	FID1	FID2	FID1	FID2	FIDin
13:13-13:23	1	1	91	91	127
13:24-13:34	1	cap measurement (at point 1)	116	143	184
13:36-13:46	1	tent measurement (at the height of measuring point 1)	117	135	164

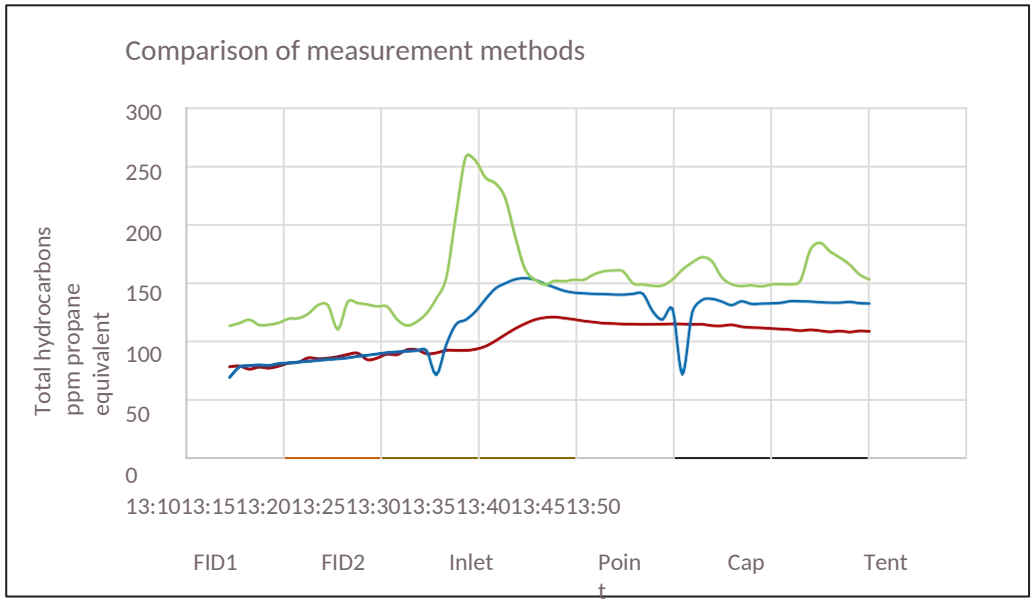


Figure 36: TKWS concentration profile comparison measurement methods measuring point 1

5. CONCLUSIONS

During the measurements, the practical applicability of the various covering methods was examined and the results of different measurement methods were compared. The occurrence of subsidence in the biofilter made it considerably more difficult to interpret the measurements

taken.

The results of the measurements clearly show the following conclusions:

- the three covering methods included in the compendium (hood/cap, tent, tarpaulin) can be applied in practice
- when performing point measurements, local effects can cause large differences in concentrations. This method is therefore not permitted as a measurement method in the compendium.
- The use of a sampling hood/sampling cap (1 m^2) is easy to apply in practice. However, since the hood/cap often has to be moved in order to determine the emissions from the biofilter, this method can be quite cumbersome. Since the sampled surface area is smaller than when using a tent or tarpaulin, the use of a sampling hood is not the preferred method, but is only permitted when it is not possible to cover a larger part of the biofilter.
- It is also easy to use a sampling tent. The advantage of this method is that a larger surface area is covered, which means that when used on non-homogeneous surfaces, the concentration measurement shall be more representative.
- the use of a tarpaulin may require more work than the use of a tent or a cap (especially in windy conditions), but it is also practical. Since this covering method samples the largest surface area, it is the most representative and is therefore the preferred method. To avoid wind impact, the tarpaulin must be positioned so that the opening is on the downwind side. Sampling is carried out at the height of the outlet/opening in the tarpaulin.

ANNEX 2 ODOUR MEASUREMENTS ON A VERTICAL BIOFILTER FOR ODOUR REMOVAL

1. SITUATION

In order to examine the practical feasibility of using a partial cover on a vertical biofilter, odour measurements were carried out on a vertical biofilter in a manure processing plant. The plant had four vertical biofilters installed side by side, each consisting of a perforated tube (diameter = 4 m; height = 12 m) filled with bark. The odour-containing air was fed into the top of the biofilters (see figure below).

On one side of the biofilters there is a concrete wall (3 to 4 m high), which makes the bottom of the biofilters inaccessible on this side. On the other side of the biofilters there is a metal structure and a staircase, which also makes this side of the four biofilters difficult to access. The 'side' of the first and last biofilters can be accessed with a cherry picker for carrying out measurements.

Figure 37: Biofilter situation

Figure 38: Top side of biofilter

2. MEASUREMENTS CARRIED OUT

On 23 June 2021, odour samples were taken at the last biofilter in the row. The measurements were carried out using a sampling cap (surface area 1 m²) placed against the wall of the biofilter. To ensure that the sampling cap fitted snugly against the biofilter wall, flaps were attached to the sides of the box.

A schematic representation of the measurement setup is shown in the figure below. During the measurement, samples were taken at different locations/heights (see figure). An initial measurement was taken for 30 minutes at measurement location 1. This was then repeated at measurement locations 2 to 4. Finally, a measurement was performed in which a mixing sample was sampled by placing the sampling cap successively at each location for 7.5 minutes each time. This complete sampling series was carried out twice. At each measurement location, the flow rate in the tube of the sampling hood/sampling cap was additionally measured with a vane anemometer.

Figure 39: Location of odour sampling situation

3. RESULTS

An overview of the measured odour concentrations is provided in the table below.

Table 12: Overview of odour concentrations in vertical biofilter

Measuring point	Sampling hour	Odour sampling	Odour concentration [ou_E/m^3]	Tube cap velocity [m/s]
1	8u30-9u20	21062301	4340	3,1
2	10u10-10u40	21062302	3444	2,9
3	10u43-11u13	21062303	4871	3,7
4	11u15-11u45	21062304	10935	2,3
4-3-2-1	11u46-12u16	21062305	10321	
1	12u18-12u48	21062306	13777	2,6

2	12u49-13u19	21062307	9742	2,5
3	13u20-13u50	21062308	11585	2,5
4	13u51-14u21	21062309	8192	0,8
4-3-2-1	14u22-14u52	21062310	8679	

Because high ammonia concentrations were observed during the first sampling at measuring point 1 (ammonia concentration with Dräger tube > 100 ppm in the tube of the cap) sampling was temporarily stopped after measuring point 1 and only restarted at measuring point 2 when lower ammonia concentrations were observed.

The results show that the odour concentration during the measurements varied from 3444 ou_E/m^3 to 13777 ou_E/m^3. These variations may be caused by variations in the odour concentration of the incoming air (sampling at different times), by the occurrence of inhomogeneity in the biofilter, etc.

The homogeneity of the biofilter cannot be assessed on the basis of the results of the odour analyses. As described in the compendium method, this must be done on the basis of flow rate measurements or concentration measurements for a specific chemical substance. The results of the velocity measurements carried out in the sampling cap tube show that the velocity varied from 0.8 m/s to 3.1 m/s and that the biofilter was not homogeneous.

4. CONCLUSION

Based on the measurements carried out, it appears that it is practically possible to take odour samples from a vertical biofilter with the help of a sampling hood/sampling cap.

As with horizontal biofilters, the representativeness of the covered surface shall be greater if a larger proportion of the biofilter surface/biofilter wall is covered.

However, the applicability of this will depend on the construction of the biofilter (vertical wall or cylinder, etc.) and the accessibility of the biofilter wall.