Ministerial Decree approving the Compendium for Sampling and Analysis (CMA)

Legal bases

This Decree is based on:
- the Decree of 23 December 2011 on sustainable management of material cycles and waste, Article 7, amended by the Decree of 25 April 2014;
- the VLAREL of 19 November 2010, Article 45, §1, paragraph 2, inserted pursuant to the Flemish Government Decree of 3 May 2019.

Procedural requirements

The following procedural requirements have been met:
- The OVAM Waste and Materials Management and Soil Management Departments submitted a proposal to amend the Compendium for Sampling and Analysis (CMA) on xxx 2025.
- the Flemish Supervisory Committee for the Processing of Personal Data issued Opinion No xxx issued on xxx 2025.
- The draft was notified to the European Commission in notification no xxx on xxx July 2025 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 was not submitted to the Legislation Section of the Council of State for an opinion as the draft is not regulatory in nature within the meaning of Article 3(1)(1) of the coordinated Laws on the Council of State, pursuant to Opinion No 77.363/1 issued on 16 January 2025.

Justification

This Decree is based on the following grounds:
- International and regional developments as well as recent research require adjustments to the existing methods and the addition of new methods.
- 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] and 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 both the laboratories, the government and the sponsors, it is appropriate that all compendia enter into force on the same date. 15 January 2026 is expected to be the joint date of entry into force. Laboratories have been informed of the new methods in the compendia and the methods can be consulted online at https://emis.vito.be/nl/erkende-laboratoria.
- The Flemish Supervisory Commission requests that personal data be processed on the basis of a justification. The processing of the personal data of the sampler is lawful on the basis of Article 6(1)(c) and (e) of the General Data Protection Regulation, in particular the legal obligation to lay down conditions for the protection of humans and the environment

against certain forms of nuisance and risks arising from classified/non-classified establishments or activities and requirements for use, as well as the detailed rules for the automatic suspension or expiry of the approvals. The processing is also necessary for the performance of a task carried out in the public interest or in the exercise of official authority conferred on the controller. The retention periods are regulated by, among others, Articles 49 and 50 of the VLAREL and the Administrative Decree of 7 December 2018.

THE FLEMISH MINISTER OF ENVIRONMENT AND AGRICULTURE DECREES:

Article 1. The Compendium for Sampling and Analysis, under the Materials Decree and the Soil Decree, abbreviated 'CMA' in Flemish, of which the table of contents included in the Annex to this Decree, is hereby adopted.

Article 2. The processing of personal data by the Flemish Environment Agency and the reference laboratory of the Flemish Region in the context of the application of the compendium referred to in Article 1 is limited to the name of the sampler and the date, time and place of the sampling. The location data is only used for the legally determined purposes.

Article 3. The ministerial decree of 30 March 2025 adopting the Compendium for Sampling and Analysis is repealed.

Article 4. This Decree enters into force on 15 January 2026.

Brussels, (date).

The Flemish Minister for Environment and Agriculture,

Jo BROUNS

Compendium for Sampling and Analysis (CMA)

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Approved for attachment to the Ministerial Decree adopting the Compendium for Sampling and Analysis.

Brussels, (date).

The Flemish Minister for Environment and Agriculture

Jo BROUNS

Groundwater

CONTENTS

1 INTRODUCTION

This procedure replaces the October 2024 procedure CMA/1/A.2 and is applicable in the context of decree-based soil surveys with the aim of:

1) the limitation of contamination plumes to obtain an overview of the quality status of the groundwater.
2) the determination of average groundwater concentrations in the context of drainage for soil rehabilitation works or at the level of cadastral working areas.

This procedure is not applicable in the case of drainage for construction and/or infrastructure works.

Groundwater surveys are a critical part of soil surveys. They can examine whether soil contamination has reached and spread through the saturated zone. This kind of study requires groundwater sampling. Fieldworkers must obtain these samples from wells that contain groundwater or places where groundwater comes to the surface (springs). Monitoring wells enable groundwater surveys at any location. The importance of proper groundwater sampling cannot be overstated.

First and foremost, it requires selection of the right location (soil layer), and maximum accuracy when installing the filter for sampling. Special precautions are required to ensure that the water sample at the selected location is representative and to prevent changes in contaminant levels in the sample during sampling. This document details these precautions.
Proper installation of monitoring wells also depends on selection of the right drilling method (technique + diameter, as well as application of the selected drilling technique). The chapters below summarise the following:

- the factors affecting monitoring well water quality;
- sampling equipment permitted for groundwater sampling;
- sampling guidelines and pure product sampling procedure;
- pumped groundwater disposal guidelines.

The guidelines included in CMA procedure CMA/1/A.2 do not rule out the application of alternative investigation techniques (AIT) (see standard procedure for descriptive soil studies). However, alternative techniques (i.e. measurements and uses other than monitoring well installation and sampling for analysis) must not replace conventional surveys, and must therefore always be combined with borehole/monitoring well installation and soil/groundwater sampling. In the soil rehabilitation report, the soil rehabilitation expert shall include a description of the alternative test technique used, a summary of the principle and the manner in which the results of the AIT were interpreted. In addition, it is necessary to describe the investigation techniques by which the results of the AIT were validated.

2 SAFETY MEASURES

Everyone should be aware that the sampling of groundwater in which pollutants may be present may present significant hazards and (health) risks. Groundwater sampling must be carried out in accordance with the Code on Wellbeing at work.

3 INSTALLATION OF BOREHOLES

For borehole installation before monitoring well installation (borehole installation guidelines, method for determining the drilling programme, list of available drilling techniques, guidelines on technical implementation of boreholes and reporting method), please see the standards given in procedure CMA/1/A.1 (Solid part of the soil).

4 INSTALLATION OF MONITORING WELLS

4.1 GENERAL

A monitoring well consists of a plastic or stainless steel pipe, typically with perforated bottom section. This part, the filter, allows groundwater to flow into the pipe. The non-perforated part of the monitoring well is known as the 'blind pipe' or 'riser'.
A distinction may be drawn between a permanent and a temporary monitoring well:

- A permanent monitoring well may be described as a monitoring well installed to enable long-term monitoring (up to multiple years).
- A temporary stand pipe piezometer is a stand pipe piezometer that is only present on the terrain for a limited time, usually only a few hours. Installation of monitoring wells of this kind involves mechanical installation of stainless steel filters. These filters are part of the drilling equipment.

The method of installing a monitoring well can significantly affect the results of the groundwater survey. The filter depth and length are especially vital and must always be appropriate to the survey objective, the lithological structure of the subsoil and the nature and expected distribution of the contamination, both horizontally and vertically.

Factors that may significantly affect groundwater survey results include:

- the sampling itself;
- subsequent application of pollutants spread along the borehole wall due to smearing during drilling;
- permeation of pollutants through the monitoring well material;
- leakage due to improper installation and application of bentonite plugs (with simple wells, but also and particularly with nested/multilevel wells), possibly resulting in short-circuits (leakage through borehole, filter frame or riser, preferential extraction from permeable layers, etc.).

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To minimize these issues, monitoring well installation must give due consideration to the following:

- the geohydrological preconditions/soil structure at the survey site;
- The presence of impermeable and less permeable layers that must be resealed. knowledge of the exact depth is essential here;
- the borehole installation method (drilling, rotation, percussion, vibration);
- the technical finishing of the monitoring well: couplings, sand/silt trap, finishing at ground level and at the sealing and/or less permeable layers;
- the monitoring well material used (evaluated based on the prevailing soil conditions and current or expected contamination);
- failure to properly remove the drilling water used during monitoring well installation;
- the method for flushing the monitoring well after installation.

The optimal filter depth is highly dependent on the circumstances. A distinction is drawn here between:

- intersecting monitoring wells, where the monitoring well filter intersects the groundwater table;
- non-intersecting monitoring wells, where the filter is located entirely within the saturated part of the aquifer.

4.2 INTERSECTING MONITORING WELLS

To detect any floating layer/pure product, the filter must always intersect the groundwater table (Figure 1). For these monitoring wells, the filter installation must specifically take into account the observations from the field tests during borehole installation, with the local geological structure and the natural fluctuations in the groundwater table. The filter must always have a water column of at least 50% of the filter length, including at the minimum groundwater level. At high groundwater levels, at least 10% of the filter must also remain 'free', to enable floating layer measurements during periods with high groundwater levels.

At the time of entry into force of this CMA procedure, intersecting monitoring wells are only still permitted for the detection of floating layers. They are not permitted for sampling for chemical analysis. In cases of deviations, the expert must always justify the monitoring well structure. It is however still permitted to use intersecting monitoring wells installed before 18 January 2012 for groundwater sampling/analysis, provided that the groundwater inflow is in accordance with the local soil structure. Account must also be taken of the guidelines contained in Paragraph 8.

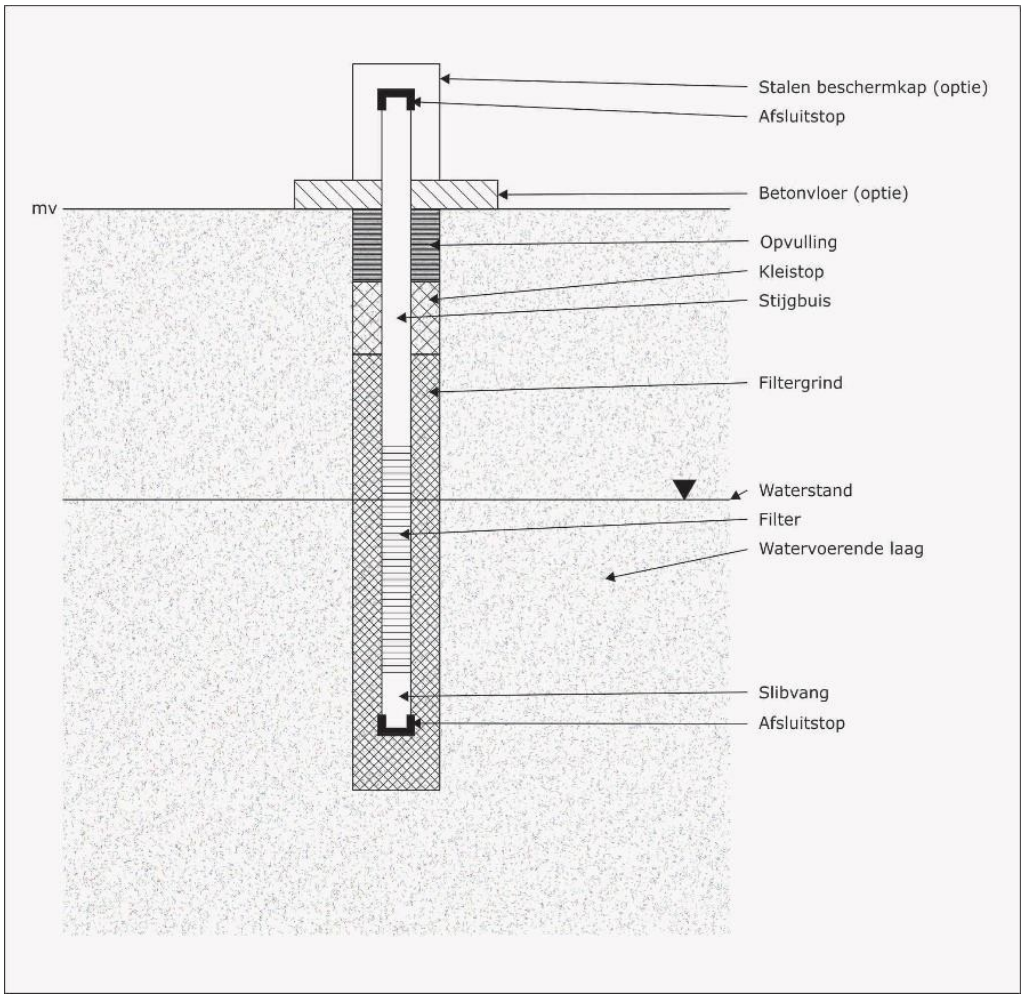


Figure 1 – Cross-section of a borehole with intersecting monitoring well (not to scale)

Stalen beschermkap (optie)	Sample protective cover (optional)
Afsluitstop	Sealing plug
Betonvloer (optie)	Concrete floor (optional)
Opvulling	Filling
Kleistop	Clay stopper
Stijgbuis	Riser pipe
Filtergrind	Filter gravel
Waterstand	Water level
Filter	Filter
Watervoerende laag	Aquifer
Slibvang	Sludge trap
Afsluitstop	Sealing plug

4.3 NON-INTERSECTING MONITORING WELLS ('FULLY SATURATED' FILTER)

For monitoring wells with a filter located entirely within the saturated zone (Figure 2), determine the filter depth and length based on the geological characteristics of the site and the nature of the contamination. The standard filter length is 1 to 2 m (see § 4.4.3.2). Monitoring wells in completely sealed aquifers (Figure 3) must have a gap of 1 m between the top of the filter and the aquifer 'roof'. The expert determines the monitoring well structure.

Selection of the monitoring well depth and filter length is critical. Thus, filter installation requires the utmost of care.

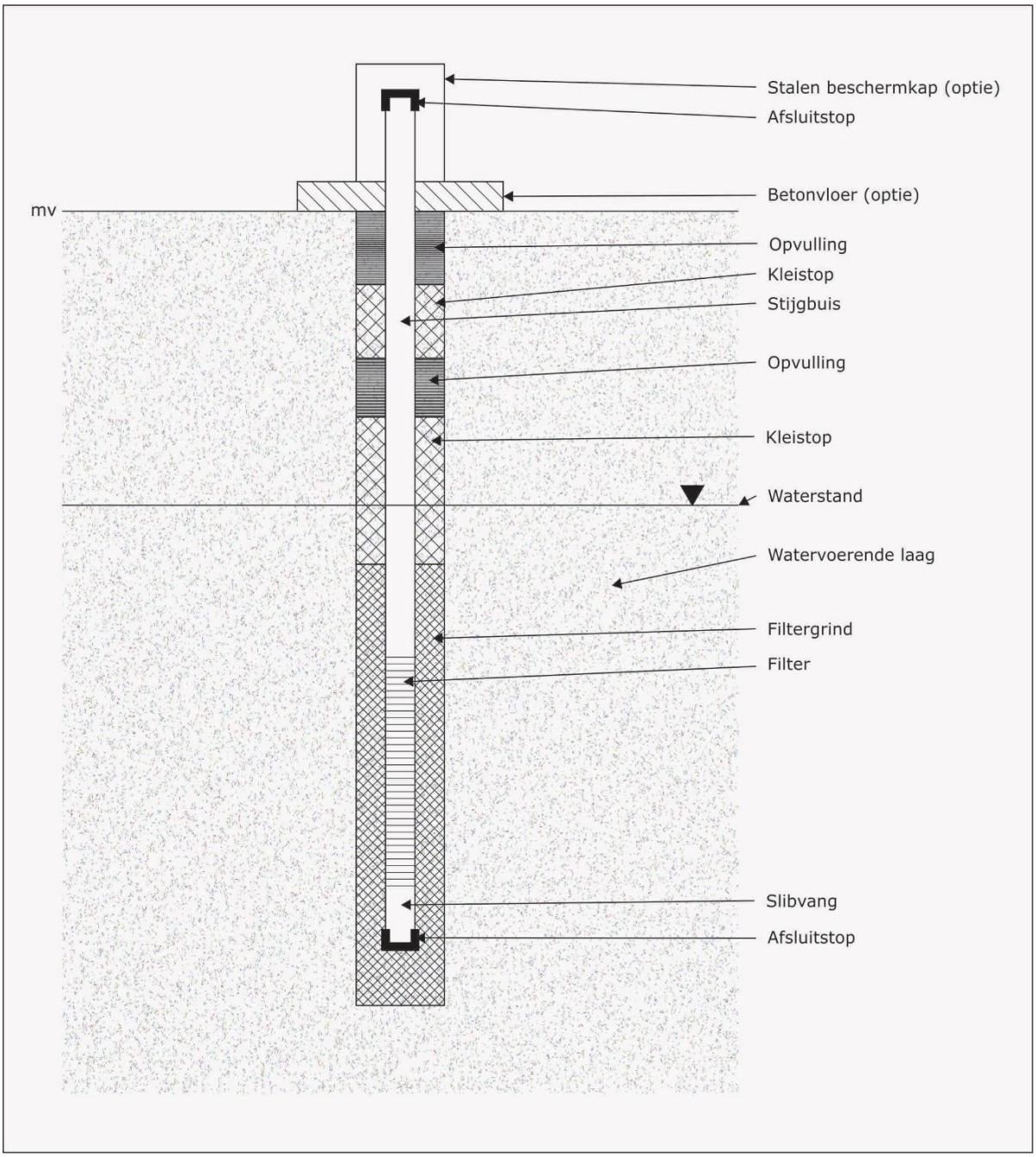


Figure 2 – Cross-section of a borehole with non-intersecting monitoring well (not to scale)

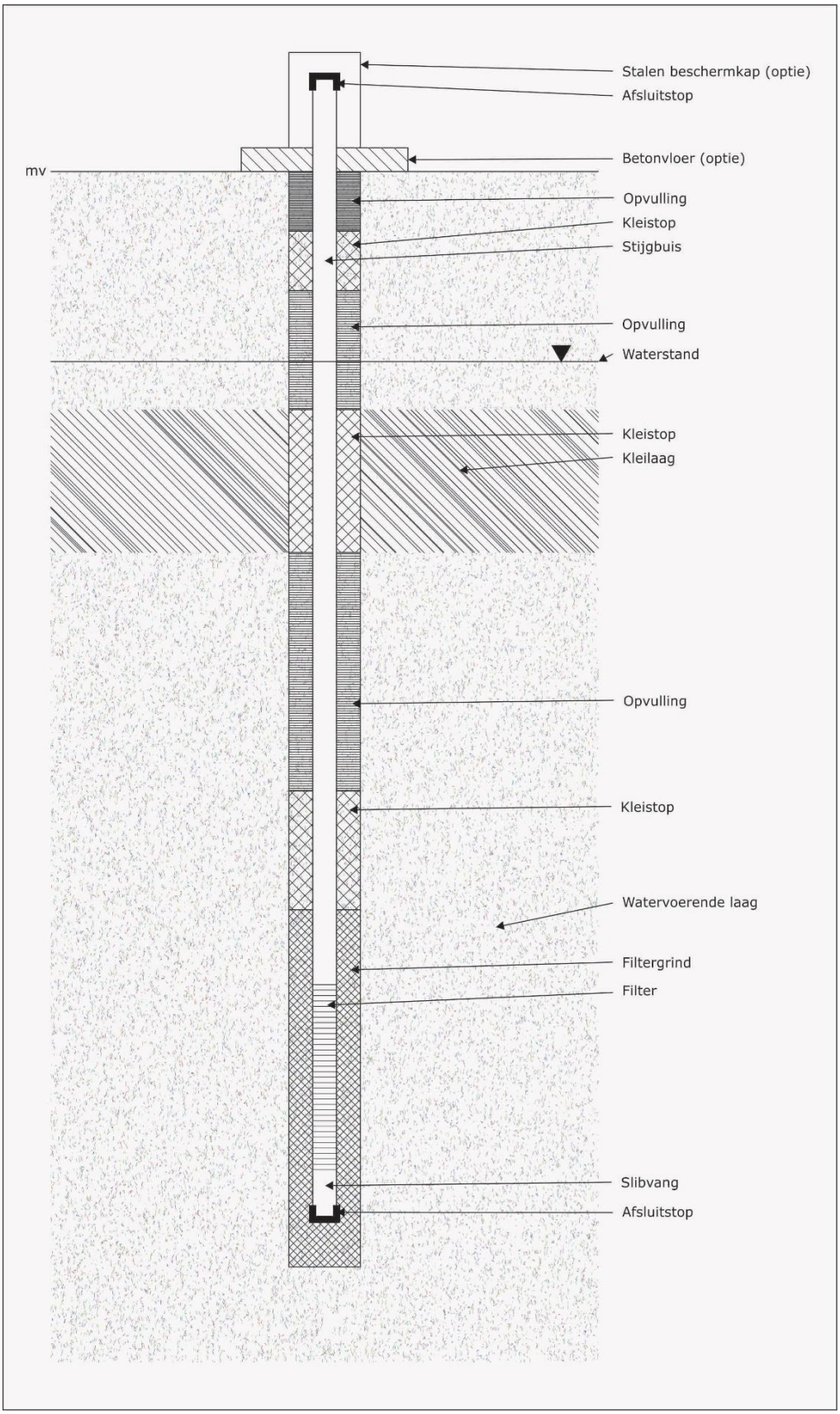


Figure 3 – Cross-section of a borehole with a monitoring well whose filter is located under a poorly permeable layer (not to scale)

4.4 Minimum requirements for monitoring well installation

4.4.1 Monitoring well characteristics

4.4.1.1 Monitoring well diameter

The minimum monitoring well diameter depends on the project objectives and the geological preconditions. Monitoring wells require a minimum pipe diameter to ensure smooth lowering of measuring equipment and level probes. It must also enable sampling. The diameter of the monitoring well is proportionate to the size of the pumps and possibly also the size of the recording equipment to be used during sampling.

When installing a monitoring well, the borehole diameter must at least be large enough to leave an annular space of at least 1.5 cm around the monitoring well after installation of the monitoring well material in the borehole. For this, please also refer to the standards in procedure CMA1/A.1 (Solid part of the soil, § 6.4).

4.4.1.2 Selection of materials

- The monitoring well material must be selected with the utmost of care. Selection of the monitoring well material must consider factors such as the nature and degree of the expected or known contamination. The monitoring well material may affect sample quality. For a list of the effects of a selection of different materials, please see Table 1. Steel monitoring wells must be made from stainless steel.
- The filter elements and risers (blind pipes) must be interconnected with threaded connections. In addition to threaded connections, PVC monitoring wells may also use socket connections.
- Bonded connections and tape are not permitted.

4.4.2 Avoiding cross-contamination

- During placement of monitoring wells, contact between the monitoring well material and drilled contaminated soil or contaminated matter (objects, pavements, etc.) is avoided.
- Smeared monitoring well material cannot be used. The monitoring well must be free from contamination.

4.4.3 Filter characteristics

4.4.3.1 General

- Filter gauze may be installed around the filter frame. This is not necessary, however. In any case, the monitoring well design must reflect the choice to use or omit filter gauze.
- For monitoring wells used for longer periods of time, it is best to install a silt trap of at least 0.5 m. The expert determines the need for and dimensions of any silt trap, taking into account the local soil structure and the monitoring well type.

- Prepack filter pipes are required if using direct push techniques (DPT), unless the requirement of having a minimum annular space of 1.5 cm around the monitoring well (see § 4.4.1.1) can be satisfied.

4.4.3.2 FILTER FRAME

- It is never permitted to install a monitoring well filter in two or more aquifers at the same time.
- As standard, monitoring wells must feature filters with a length of 1 to 2m m when installed in the top 10 m of the first aquifer. Deviations from this are possible in exceptional and justified cases, in consultation with the OVAM.
- After consultation with the OVAM, if the installer opts for a monitoring well with a filter segment longer than 2 m, it must take the following into account:
  - The possible occurrence of vertical flow along the filter interval may result in the mixing of different qualities of groundwater;
  - Vertical flow may even occur in homogeneous aquifers with slight head differences! In this way, surface contamination may infiltrate deeper parts of the aquifer;
  - The longer the filter frame (with respect to the relative thickness of the aquifer), the more difficult it is to correlate analytic concentrations at different depths;
  - mixing in the filter frame makes it impossible to determine maximum or minimum pollutant levels;
  - monitoring wells with filter frames longer than 2 m are not permitted when testing for the presence of contamination with VOCls.
- To detect any floating layers/pure product, the filters must intersect the groundwater table (see § 4.2).

Groundwater sampling under the Soil Decree requires monitoring wells with non-intersecting filters (see § 4.3).

Table 1 - Non-exhaustive list of monitoring well materials and their properties

Material	Advantages	Cons
PVC	- lightweight - high resistance to abrasion, electrochemical and galvanic corrosion - applicable with acids, oxidising media, salts, alkalis, oils, fuels and volatile aromatics - highly resistant to weak bases, alcohols, aliphatic hydrocarbons and oils - resistant to strong mineral acids, concentrated oxidants and strong bases - readily available - price	- weaker, more flexible and more temperature-sensitive than metallic materials - low resistance to (lightweight) ketones, aldehydes, amines, chlorinated alkenes and alkanes and aromatic hydrocarbons (the critical concentrations are unknown.) - can adsorb components from groundwater (tetrachloroethylene, lead, certain chlorinated ethanes, bromoform) - may react with components from groundwater - leaching of lead, cadmium and certain additives possible
HDPE High-density polyethylene	- lightweight - higher chemical resistance than PVC - highly resistant to mineral acids - resistant to highly resistant to bases, alcohols, ketones and esters - resistant to oils	- weaker, more flexible and more temperature-sensitive than metallic materials - may react with components in the groundwater

Material	Advantages	Cons
	- reasonably resistant to oxidants, aliphatic and aromatic hydrocarbons - no traces of heavy metals, ink, lubricating wax, plasticisers - price	- possible leaching of certain components - possible zinc adsorption - possible permeation/diffusion of volatile aromatics and volatile chlorinated solvents - difficult to process mechanically because it melts easily and is difficult to cut
Teflon	- lightweight - highly impact-resistant - temperature-resistant - exceptional resistance - insoluble in all organic substances except in a few rare fluorinated solvents - minimal adsorption and absorption - inert for metals	- low tension - subject to rapid wear compared to other plastics; - possible lead and cadmium adsorption - possible volatile component adsorption - price - not suitable for PFAS analyses
PVDF (polyvinylidene fluoride) (Kynar = brand name)	- more tension and more wear-resistant than Teflon - resistant to most chemicals - cheaper than Teflon	- often not available immediately - low chemical resistance to ketones and acetone - price - not suitable for PFAS analyses
Stainless steel	- very strong within a wide temperature range - highly resistant to corrosion - possible leaching and sorption of certain heavy metals - readily available	- heavier than plastics - may release metals in highly acidic environments - may act as a catalyst in organic reactions - price

4.4.4 MONITORING WELL FINISHING

4.4.4.1 MONITORING WELL SEALING

- It is necessary to seal a monitoring well on the bottom with a cap or other method, to prevent soil material flowing in. This seal must be made from either the same material as the monitoring well or another inert material.
- To limit the influence from above on water quality in the monitoring well, also install a cap on the top of the monitoring well (for finishing, see also Figures 1 to 3). Because the monitoring well must maintain atmospheric pressure inside for the groundwater to flow freely, the cap must feature a small opening.
- In cases of possible seepage, or flooding on the monitoring well site, use a sealed cap and install a waterproof surface box or protective tube.

4.4.4.2 FILLING OF ANNULAR SPACE BETWEEN FILTER AND BOREHOLE WALL

- Always pour calcined, screened and calibrated filter sand/gravel around the filter.
- Always pour the pack around the filter frame up to above the top of the filter frame (at least 0.25 m and at most 0.5 m).
- If using a silt trap, pour the pack starting from 10 cm below the filter frame.
- The particle size of the filter sand or gravel used depends on the soil type in which the filter is installed and the filter pipe perforation.

4.4.4.3 FILLING OF ANNULAR SPACE BETWEEN FILTER AND BOREHOLE WALL

- Annular space between monitoring well and borehole wall:

The annular space between the blind pipe of the monitoring well and the borehole wall may be filled with the original soil material unless this is contaminated or pure product is present. In those cases, it is necessary to dispose of the material according to the applicable regulations and fill the borehole with non-contaminated material with properties similar to the extracted sediment or install a bentonite plug over the entire length.

- Annular space under monitoring well

Fill in the annular space created when drilling deeper than the depth of the planned monitoring well if pollutants are considered likely to spread to deeper parts of the aquifer. The backfill must be bentonite or grout. This process must however take into account the swelling capacity of the bentonite, to prevent clogging of the filter frame of the future monitoring well from below.

4.4.5 INSTALLING BENTONITE PLUG (FIGURES 1 TO 3)

4.4.5.1 GENERAL GUIDANCE

- The bentonite plug must not leach contamination.
- The time interval between installation of the bentonite plug and its maximum swell cannot exceed 24 hours.
- Determine the depth and thickness of the installed bentonite plugs as accurately as possible. When using measuring tape or extension rods, bear in mind that collapse of the borehole may result in incorrect measurement values.
- Selection of sealing materials must take the following into account:
  - the technical specifications of the materials to be used: both swelling capacity and swelling speed will determine whether a particular material type is suitable for use.
  - The ambient conditions in which the sealing material will be used: these conditions may influence the effective swelling capacity and swelling speed (e.g. in highly saline conditions, clay tends to contract rather than expand).

4.4.5.2 BENTONITE PLUG LOCATIONS

Install bentonite plugs at the following locations:

- Just below ground level, unsaturated zone:

After monitoring well installation, install a bentonite plug (grout mixture or bentonite), just below ground level (depth: 0.25 down to a minimum of 0.50 m b.g.l.), to help prevent contamination from infiltrating water. The installer must however avoid a situation where the swelling of the grout mixture/bentonite fills up the surface box over time. When using bentonite, use a limited amount of water (quality comparable to drinking water) during installation, to allow the bentonite to swell.

- Sealing layers:

When drilling through sealing layers, install a bentonite plug at each sealing layer. Each individual bentonite plug must be at least as thick as the sealing layer (see Figure 3). At sealing layers, install bentonite plugs by applying liquid grout to the bottom.

- On top of poured filter gravel:

Always install a bentonite plug that is at least 1 long above the filter gravel located around the filter.

- Pure product zones:

When drilling through pure product zones, install a bentonite plug at these zones. Each individual

bentonite plug must be at least as thick as the pure product zone. At pure product zones, install bentonite plugs by applying liquid grout to the bottom.

4.4.6 DETERMINING DEPTHS OF FILTER GRAVEL AND BENTONITE PLUGS

When finishing monitoring wells, it is necessary to measure the depths of the filter gravel, the installed bentonite plugs and the filler material (such as with a measuring tape or extension rods). The field report must indicate both the bottom and top of each filler type.

4.4.7 NESTED MONITORING WELLS

- Wherever possible, avoid installing more than one filter pipe in the same borehole (known as 'nested wells'). If this approach is necessary however, do not install more than two nested wells in the same borehole, use bentonite plugs that are at least 2 long above the filters and follow the requirements set out above.
- If installing nested monitoring wells, install the individual wells separately in the borehole, to guarantee their finishing according to the requirements of the procedure above. Do not interconnect the different monitoring wells. The borehole diameter shall also be of sufficient size to ensure good quality finish.
- Install seals at the relevant places in the soil profile (see above) by applying liquid grout from below. It is not permitted to use bentonite in powder, pellet or granulate form for this.
- It is not permitted to use the hollow auger drilling technique.
- Nested monitoring wells are not permitted in core zones.
- Verify the effectiveness of the installed clay plugs by conducting a pump test on the monitoring wells in the borehole. After pumping the groundwater out of one of the monitoring wells, monitor the groundwater level in all monitoring wells in the borehole. Conduct this pump test after the installed sealing materials have cured. In the event of leakage flows in nested monitoring wells, decommission all monitoring wells in the borehole where a leakage flow has been detected. With regard to the procedure to be followed, reference is made to Paragraph 11.
- Include the pump test report in the field report on the monitoring well installation.

4.4.8 MONITORING WELL DEVELOPMENT

The term 'monitoring well development' covers all operations intended to restore the natural hydraulic properties of sediments after installation of boreholes and their finishing as monitoring wells. Monitoring well development includes at least flushing the well after installation (see §4.4.9). For the guidelines on purging monitoring wells (i.e refreshing the groundwater volume at the filter section) immediately before groundwater sampling, please see §8.5. If drilling water is used, the drilling water used must be of good quality (minimum drinking water quality). Further guidelines on the use of drilling water are included in CMA procedure CMA/1/A.1, § 6.5).

4.4.9 FLUSHING AFTER MONITORING WELL INSTALLATION

- It is necessary to flush monitoring wells immediately after installation, unless the soil contamination requires a different procedure. When drilling through material such as an impermeable layer or a pure product zone, the RSRE may decide to wait a few days after monitoring well installation before flushing the monitoring well, to leave adequate time for the installed clay plug to swell. After this comes the time interval between monitoring well installation and sampling (see § 8.10).
- Flushing includes more than just draining the installed monitoring well at a high flow rate:

- Gradual flushing, by moving the sampling hose up and down, is preferred;
- minimum 5x the water column (volume of water present in filter section and blind tube);
- Flushing: the flushing water must be free of silt and sand before the completion of flushing.

- When installing a monitoring well, drilling water may only be used if technically necessary due to terrain conditions (see CMA/1/A.1: Fixed part of the soil, § 5.2.2 and §6.5). Only water of drinking water quality may be used. Then, during flushing, the volume of water pumped must be increased by five times the volume of drilling water used.
- It is recommended to use a high pump flow rate for this, preferably in an alternating manner (extract water, then drop it back into the monitoring well).
- Nested monitoring wells: At least 1 week must pass between nested monitoring well installation and flushing. After this comes the time interval between monitoring well installation and sampling (see § 8.10).
- After flushing, observe a stabilisation period of at least 1 week before groundwater sampling.

4.5 Temporary monitoring wells

Temporary filters, installed with probing techniques, (known as 'temporary monitoring wells') offer the following benefits:

- Groundwater samples can be taken at different depths;
- rapid sampling. Groundwater samples can be taken immediately after installation of the temporary monitoring well;
- minimal disturbance of the soil;
- groundwater sampling possible for analyses for both volatile and non-volatile compounds.

If used, this technique must meet the following requirements:

- the soil structure must be known before monitoring well installation, for installation of the filter at the correct depth and in the correct soil layer;
- check for the presence and depth of any sealing clay layers;
- it is not permitted to drill through sealing layers when installing temporary monitoring wells;
- the exact depth of the filter must be known;
- this method is only permitted for determining groundwater quality, not for measuring groundwater levels or conducting long-term monitoring of any kind;
- Avoid creating preferential dispersal routes. for this, fill in the probe hole from below based on the lithological structure and degree of contamination (see CMA/1/A.1: Soil, § 6.8 – Borehole finishing - grouting).
- sampling is carried out after purging according to the guidelines for low-flow sampling (see §8.5.1).
- Temporary monitoring well finishing: see CMA/1/A.1 § 6.9.

4.6 Multi-level wells (MLW)

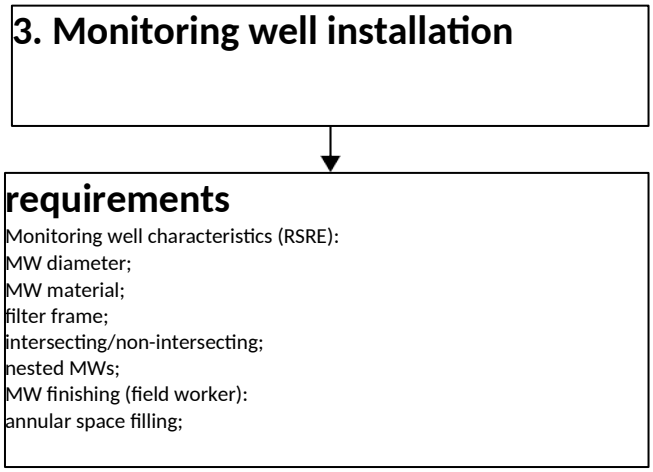
Multi-level groundwater wells are used to take groundwater samples at defined depths in the same monitoring well. MLWs may be compared to nested monitoring wells, but with characteristically shorter filter elements. However, MLW installation only requires a single borehole. Various types of MLWs are distinguished. They may be used to:

- obtain a detailed picture of the vertical distribution of the contamination;
- reduce the number of boreholes/monitoring wells to be installed.

It should be noted that both the drilling company and the expert require more knowledge to install measuring systems of this kind because it is critical to install the filters, bentonite plugs and seals at the correct depths. In addition, this measurement setup is not suitable for detection of floating layers, and the short filters prevent detection of any contamination present. If the MLW installer drills through sealing layers, it must install seals at these layers by applying liquid grout (from below).

Multi-level wells always require a properly detailed justification in the survey strategy. The report (see procedure CMA/1/A.1: Soil, § 9 Reporting) must also include a detailed description of the installed MLW.

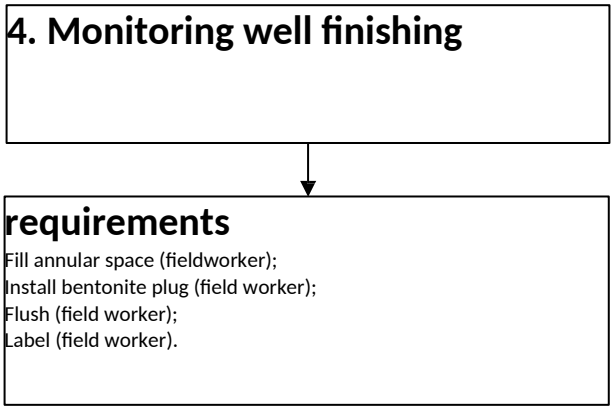
4.7 DIAGRAM



5 BOREHOLE/MONITORING WELL FINISHING

After monitoring well installation, the installer must give due attention to finishing the well. The measures below are required.

- Be sure to repair any ground subsidence and fill in any collapsed cavities along the monitoring well. This also applies when installing a temporary monitoring well.
- Immediately after installation, the installer applies unique and weatherproof identification (label, sticker, plastic plate, etc.) to the monitoring wells onsite, including at least the following details:
  - monitoring well number;
  - monitoring well installation date;
  - filter frame;
  - groundwater inflow (good, moderate, poor).
- When finishing monitoring wells on sites susceptible to damage, it is best to install a clearly visible protective tube or surface box (possibly in a concrete base). It is permitted to install a seal on the cap of the monitoring well or a surface box/protective cover with lock if the monitoring well is in a public space or easily accessible location. This may prevent intentional or unintentional contamination of the monitoring well from above.
- Use a waterproof utility hole cover to seal a monitoring well that passes through waterproof surfacing. When installing a temporary monitoring well, repair the waterproof surfacing. In addition, ensure that the surfacing at the penetration is waterproof (inner finishing of surface box is waterproof). For this, the installer may add water-repellent additives to the cement. These additives must not cause pollutant leaching.



6 FACTORS AFFECTING MONITORING WELL WATER QUALITY

6.1 SITE-SPECIFIC FACTORS

During and after monitoring well installation, the installer must take measures to prevent the inflow of surface water and other liquids (due to uneven ground, poor site selection, monitoring well finishing that does not meet the preconditions for the high-risk site, etc.).

6.2 GEOHYDROLOGICAL FACTORS

In general, the 'in-situ' composition of the groundwater depends on:
- The medium through which the water flows (mineralogy/deposits);
- The mix and origin of the different groundwaters;
- The amount of time the water spends in the different lithological units;
- The presence and spread of any contamination.

Aquifers are home to various processes that can significantly alter the physico-chemical composition of groundwater, such as:
- Biochemical processes;
- Complexation;
- Acid-base reactions;
- Redox processes;
- Precipitation-solution reactions;
- Adsorption and desorption processes.

The aforementioned processes that can determine the 'in-situ' composition may be influenced by the selected drilling technique, its application, the monitoring well structure, the sampling method and water sample preservation.

6.3 INFLUENCE OF DISSOLVED GASES

Groundwater may undergo changes when taken out of its 'in-situ' condition and exposed to atmospheric conditions with temperatures, pressures and O_2 and CO_2 levels that differ from those in the aquifer.

Groundwater contains dissolved gases, due to infiltration or chemical or biochemical gas production below the water table, as well as mineral and organic substances. In addition, it also exhibits bacterial activity. It is known that HCO_{3^-} is the predominant anion in groundwater.

In practice, the influence of dissolved gases is mainly relevant to the geochemical characterisation of groundwater. Because it is of minor relevance when sampling for a soil contamination study, it only requires limited consideration.

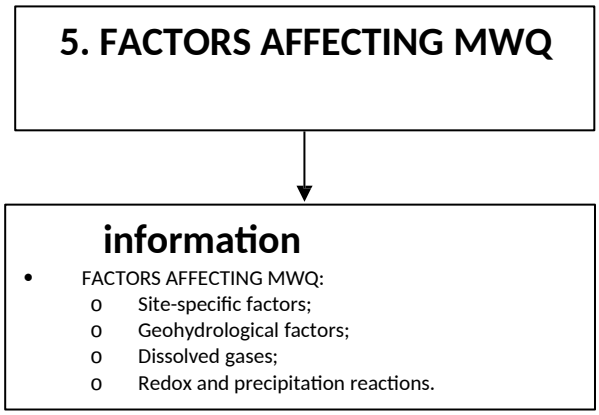
6.4 INFLUENCE OF REDOX AND PRECIPITATION REACTIONS

After a certain depth, groundwater is in a reducing state. If these groundwater samples are exposed to the air, they will undergo various reactions, such as:
- Oxidation of organic components;
- Oxidation of sulfides to sulfate;
- Oxidation of Fe^{2+} to Fe^{3+} and precipitation as $Fe(OH)_3$;
- oxidation of NH^{4+} to NO_3^{2-}.

In most cases, these redox processes will affect conductivity. Complexation, for instance, binds Mn and Fe ions to a non-oxidisable form (see also gley phenomena in soil profiles).

In these cases, precipitations of Fe and Mn oxides cause other problems for sampling. Adsorption or coprecipitation with Fe hydroxides may result in extraction of heavy metals, orthophosphate or SiO_2 from the aqueous solution, for instance.

6.5 DIAGRAM



7 SAMPLING EQUIPMENT

7.1 GENERAL

First, select pumping equipment that can meet the physical requirements such as well depth, diameter and groundwater level, with minimal impact on the physico-chemical composition of the groundwater.

As a necessary condition to minimise changes to the physico-chemical characteristics of the groundwater, the sampling process must not let any oxygen in. In these cases, it is not permitted to use airlift systems, especially because localised high pressure in the riser may cause permanent damage and leaks.

Contamination from sampling equipment is probably the main drawback of monitoring well sampling. The pump housing must be made from stainless steel or Teflon or another inert material, to minimise chemical reactions with the groundwater. In addition, it must be possible to clean or replace all parts of the sampling equipment that come into contact with groundwater from different monitoring wells. Otherwise, it is not permitted to use the equipment in question to sample groundwater for soil surveys under the decree.

Personal safety always takes precedence. It is prohibited to conduct active odour observations (smelling) and to extract groundwater by mouth.

7.2 PUMPING EQUIPMENT

Different pumping methods may be possible for groundwater sampling. Below is an overview of the most common pumping techniques.

7.2.1 WATER SAMPLING TECHNIQUES BY SUCTION

7.2.1.1 SAMPLING WITH A VACUUM PUMP

The groundwater is sucked up by creating an underpressure that causes degassing. In addition, contact with air is possible so oxidation can occur. When sampling for a soil survey under the decree, it is not permitted to use this method. This method is permitted for flushing monitoring wells after installation, but it is unsuitable for measuring field parameters, and thus also for purging before groundwater sampling (see § 8.5).

This vacuum effect occurs in all systems of above-ground pumps as long as the intake line is not filled completely. Therefore, intake lines must be completely filled before starting the actual sampling and taking the field measurements.

7.2.1.2 PERISTALTIC OR ROLLER PUMP

The hose pump can be a manually or electrically powered. The hose in the pump is covered in “silicone”. The suction side of this hose is connected to a PE hose inserted in the monitoring well. A brief underpressure prevents contact with the air, to ensure sample quality. Because the hoses are easy to replace, the risk of cross-contamination is low with proper use. One of the most common pumps is the electronic peristaltic pump with built-in battery and a connection for other pumps, a level sensor and a timer.

Due to its relatively low flow rate however (on the order of litres per minute), this pump cannot be used to flush deeper monitoring wells after installation. Purging is still possible. When using a peristaltic pump, bear in mind the maximum pump head, which is around 7 m.

7.2.1.3 ABOVE-GROUND CENTRIFUGAL PUMP

Above-ground centrifugal pumps powered by combustion engines may be used to ‘flush’ monitoring wells with larger diameters. These are available for pumping from ducts with diameters from 45 mm. The flow rate is adjustable. It is not permitted to use these centrifugal pumps for sampling. This is due to the turbulence from the high-speed vanes. After removal of this pump, a different pump is used to take the actual groundwater sample.

The maximum pump head for a centrifugal pump is 6 m.

7.2.2 WATER SAMPLING TECHNIQUES BY PUMPING UP WATER

7.2.2.1 BELLOWS PUMP

Depending on the depth, the maximum flow rate varies between 2.5 and 4 l/min. The maximum sampling depth is approx. 60 m. It also allows for inline filtration and the required measurements. The pump operates on air pressure, without contact between the air and the water sample. This is

achieved by a teflon bellow that is mounted in the pump housing. It is a pulsating pump. In the first phase, the water is sucked in by contraction of the bellows. During the second phase, the water is pressured upwards by expanding the bellows. The bellows pump is equipped with the necessary check valves in such a way that no return flow can occur.

Since certain parts of the (difficult to clean) pump casing come into contact with the groundwater from different level indicators, cross-contamination can occur. Thus, this pump type is not permitted for monitoring well sampling in soil surveys under the decree. If the monitoring well is not too large, it is possible to use this pump to flush it after installation.

However, it is important to pay attention to cross-contamination as you are working continuously with the same hoses.

7.2.2.2 FOOT VALVE PUMPS

Foot valve pumps can be hand-powered or mechanical. These pumps come in different diameters and are made from stainless steel. The field worker lowers the pump to a sampling hose into the monitoring well, and a pulsing motion forces groundwater upwards along the well.

Because these pumps do not come into contact with water, they are suitable for flushing after monitoring well installation, for purging before groundwater sampling and for the groundwater sampling itself. Only the foot valve comes into contact with the groundwater, so it must be cleaned and/or replaced regularly.

Mechanical foot valve pumps are highly suited for deep groundwater sampling.

7.2.2.3 SUBMERSIBLE PUMP

Submersible pumps are typically made from stainless steel and Teflon. The following characteristics make this pump highly suited for flushing after monitoring well installation, for purging before groundwater sampling and for groundwater sampling from deep monitoring wells:

- the flow rate is finely adjustable;
- Wide range for extraction flow rate: both low and high extraction flow rates possible;
- Thanks to a special internal structure, submersible pumps do not cause much turbulence, unlike above-ground centrifugal pumps;
- does not suck in air;
- groundwater is sucked in at the bottom of the pump.

The diameters of monitoring wells from which the submersible pump can sample depend on its type. MP1 pumps, for instance, can only be used for monitoring wells with an inner diameter of at least 50 mm.

Due to suspension of the pump in the groundwater, be mindful of potential cross-contamination. If the pump has come into contact with pure product or contaminated groundwater, clean it thoroughly before further use for flushing, purging and/or groundwater sampling.

7.2.2.4 12 SUBMERSIBLE PUMPS (DC PUMPS)

To prevent cross-contamination, 12V submersible pumps may only be used once, so they are less suited for water sampling. Their reasonably high flow rate however makes them well suited for monitoring well flushing. Usually DC pumps are used in combination with the electronic peristaltic pump.

7.2.3 OTHER METHODS

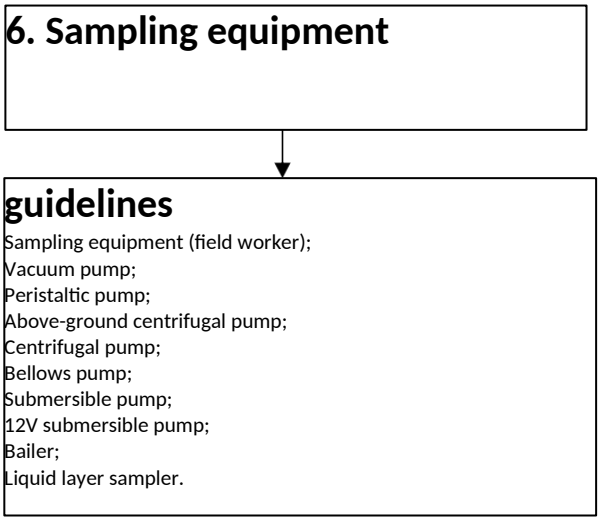
7.2.3.1 BALL VALVE SAMPLER OR BAILER

This is not a true pump, but rather a passive sampling system (known as a grab sampler) typically made from Teflon or stainless steel. This method enables the sampling of limited volumes. It is not permitted to use a bailer sampler for volatile component analysis.

7.2.3.2 LIQUID LAYER SAMPLER

A liquid layer sampler is not a pump, but rather a passive sampling system (known as a 'grab sampler'), consisting of an open Teflon tube that can be closed on the bottom. This sealing is done by means of a locking system at the bottom of the tube that is operated by a rod or cable. This system makes it possible to sample at specific depths. This method is suitable for sampling from floating and sunken layers.

7.3 DIAGRAM



8 GROUNDWATER SAMPLING GUIDELINES

8.1 GENERAL

It is best to sample groundwater from monitoring wells whose characteristics are well known.

If using existing monitoring wells whose structure is unknown, the soil survey report must clearly indicate this. Decision-making for the soil survey must exercise the necessary caution when using the analytic data from these monitoring wells.

The following aspects are important for groundwater sampling from monitoring wells:

- purpose of sampling (see § 1):
- Groundwater level measurement;
- Monitoring well characteristic measurement;
- Groundwater exchange;
- Onsite measurements;
- Filtration;
- Monitoring wells with a low groundwater inflow;
- Floating layer measurement;
- Sampling time interval;
- Volume of groundwater pumped before sampling;
- Sampling rate (avoiding turbulence/degassing for VOC analysis);
- order of filling of containers (see §8.11.2).

8.2 SAMPLING FROM EXISTING MONITORING WELLS

- The following details must be known when using existing monitoring wells (where applicable, find these in reports available from past soil surveys):
  - Exact location: for resampling, check the location indicated on the drawing and adjust as needed;
  - Aquifer from which the monitoring well draws groundwater;
  - Filter interval depth and length;
  - Monitoring well material;
  - Depth and thickness of the filter pack and the bentonite plug(s) used;
  - Monitoring well installation method;
  - Estimation of the inflow (good/medium/poor) of the monitoring well yield;
  - Most recent monitoring well sampling date.
- If resampling from existing monitoring wells, verify the usability of the monitoring well:
  - Has silting occurred?
  - Are the blind pipes still intact? If not and influence from ground level is possible (e.g. inflow of surface water), it is no longer permitted to sample from the monitoring well.
  - Does the blind pipe exhibit visible damage?
  - Is the finishing in accordance with the structure of the high-risk site being surveyed (waterproof or not)? If not, adapt the finishing to the structure of the high-risk site.

- Is the finishing still intact? If not, it is only permitted to resample from the monitoring well if the damage to the finishing has not had a potential impact on groundwater quality.
- Is there any inflow of contaminated water or other contaminated matter? If so, new sampling from the monitoring well is not permitted.
- Are the monitoring well characteristics known (filter depth, filter pack, bentonite plug depth)? If the technical data of the monitoring well are not known, a CMA-compliant sampling of groundwater is not possible.

- If a monitoring well has not been sampled for at least 2 years, flush it applying the same guidelines as with newly installed monitoring wells (see § 4.4.9). In these cases, wait at least 7 days after flushing before resampling (including purging).

8.3 MEASURING OF GROUNDWATER LEVELS AND MONITORING WELLS CHARACTERISTICS

8.3.1 GENERAL

Before flushing the monitoring wells and proceeding to groundwater sampling, it is first necessary to measure the groundwater level and some monitoring well characteristics:

- Groundwater level measurement: depth of the groundwater table with respect to a fixed point (top of monitoring well/ground level);
- Measurement of monitoring well depth with respect to fixed point, i.e. top of monitoring well/ground level (if no floating or sunken layers present). It is only permitted to take this measurement before purging or after actual groundwater sampling;
- Measurement and recording of any deviations, such as:
  - Change in monitoring well height (due to damage);
  - Contamination of surface box interior;
  - Contamination of monitoring well material;
  - Inflow of bentonite from the surface box;
  - Missing cap or surface box;
  - Presence and condition of any sampling hoses from past sampling: Naturally it is not permitted to reuse these sampling hoses. They must be disposed of (see § 8.11.1);
- Any floating layer/pure product present (see § 9.3.1).

The ‘fixed point’ serving as a reference for the level measurements is usually the edge of the monitoring well (depending on the topographic measurements, this may vary from site to site). It is necessary to apply the same method for the measurement of each site. It is best to perform levelling in m TAW (Belgian national reference level), to enable proper interpretation of water levels (especially if the survey covers different sites).

8.3.2 MEASUREMENT OF GROUNDWATER LEVEL

The groundwater level may be measured using the following:

- Level sensor: the benefit of a level sensor is that in addition to measuring the groundwater level, it can also check the monitoring well depth;
- Divers: instruments for automatic measurement of groundwater levels and groundwater temperatures. The pressure sensor and temperature sensor are located in a hermetically sealed stainless steel or ceramic housing, along with the data logger and battery. The Diver is easy to lower into the monitoring well suspended from a steel wire. The Diver can then automatically

measure the groundwater level and groundwater temperature and record these data in its internal memory. Use of Divers over longer periods of time requires corrections for air pressure fluctuations. This in turn requires concurrent measurements with a Baro-Diver.

8.4 SELECTION OF SAMPLING EQUIPMENT

§ 7.2 (Pumping equipment) summarises the available sampling equipment. Table 2 shows the suitability of the described sampling equipment based on the factors listed below:

- Flushing:
  - Groundwater level;
  - Inner diameter of monitoring well;
  - Filter depth;
  - Groundwater inflow rate
- Groundwater sampling including purging:
  - See 'flushing' factors;
  - Any turbulence;
  - Risk of cross-contamination;
  - Analysis parameters.

Table 2. Non-exhaustive list and application options for available sampling equipment

Pumping equipment	Purging		Sampling								Finishing/dimensions
	Poorly permeable layer	Highly permeable layer	Poorly permeable layer (other than	Highly permeable layer	Poorly permeable layer	Suitability for inline filtration	Risk of contamination	Maximum groundwater level (m	Suitability for analysis for Cl-VOCs	Pure product	Minimum required diameter (mm)
Bellows pump	+*	+	-	-	-	-	+	60	-	-	25
Bailer sampler (bailer)	-*	+	+	+	-	+/-	+/-	90	-	-	25
Above ground centrifugal pump	+	+	-	-	-	-	-	6	-	-	45
Underwater centrifugal pump	-*	+	-	+	+	+	-	80	+	-	50
Pulse hose (ball valve pump)	-*	+	-	+	+	+/-	-	40	+	-	25
Peristaltic pump	-*	+	+	+	+	+	+	7	+	-	25
Vacuum pump	+*	+	-	-	-	-	-	8	-	-	25
Liquid layer sampler	N/A	N/A	+	+	-	-	-	25	-	+	35

* depending on the depth of the groundwater and the diameter of the monitoring well
'+': suitable
'+/-': less suitable
'-': not permitted for soil surveys under the decree

8.5 PURGING OF MONITORING WELLS

This process assumes that the monitoring well has already been flushed after installation (taking into account the use of drilling water). If flushing was not done after installation, please see §4.4.9.

8.5.1 LOW FLOW[1]

In the case of groundwater sampling by low-flow sampling (LFS) (see §8.11.3) it must be purged at a constant low flow rate (100 to 500 ml/min) in order to prevent the release of soil particles from the layer to be sampled. Purging must not exceed the natural recharge rate of the aquifer being sampled. This requires monitoring of the water level during purging to ensure it does not exceed the prescribed maximum level drops. If the prescribed maximum level drops are exceeded (depending on the monitoring well type), the field worker must record this in the groundwater sampling field report as a deviation from the procedure. If the groundwater level drops into the filter, the field worker must note that the sample was aerated.

Fieldworkers must purge intersecting monitoring wells at a flow rate of 100 l/min and a maximum level drop of 10 cm. The criteria referred to in § 4.2 (i.e. water column in filter must be at least 50 % of the filter length, including at the lowest groundwater level; at high groundwater level, at least 10 % of the filter must be 'free') continue to apply. In addition, the inflow (at the filter) must be in accordance with the local soil structure, and the LFS principles strictly apply. It is recommended to use packers.

It is permitted to purge deep monitoring wells (> 15 m b.g.l.) at a flow rate of over 0.5 l/min, provided that the groundwater level drop in the monitoring well does not exceed 50 cm.

Purging may be terminated for low-flow sampling if at least the volumes calculated using the formula below[2] have been pumped and the following conditions have been met:

- at least the electrical conductivity value has stabilised (see §8.7) and 5 times the volume of the filter section of the monitoring well has been pumped out; or;
- electrical conductivity and dissolved oxygen have stabilised; or;
- the turbidity is less than or equal to 10 FNU (Formazin Nephelometric Units). If determining the turbidity, take the field measurements before starting the actual sampling (i.e. when monitoring the field parameters). Analysis in a lab is not acceptable.

If the borehole diameter is not known, then in order to have a CMA-compliant sampling, a safety margin of 100% must be taken into account with regard to the volume to be pumped (Table 3).

[1] EPA and other US guidelines use the term "micropurging".
[2] Excel table for performing the calculation for known borehole diameters: see Minimale afpompvolumes voor emis_vs2.xlsx

The formula for the minimum volume to be pumped is given by:

$$V\,(l) = \square * (((r_{bg})^2 - (r_{pb})^2) * \square_{fg} + (r_{pb})^2) * (\square P + 2) * (1 + marge)$$

With:

V = Volume in l

D_{bg} = borehole diameter in dm, unknown borehole diameter is set to 1 dm

D_{pb} = monitoring well diameter in dm

ρ_{fg} = filter gravel porosity, fixed value 0.35 imposed

δP = stable level drop in dm

margin = elevation outside filter gravel = 0.25 (25%) for known borehole diameter and 1 (100%) for unknown borehole diameter

With V = at least 5*the volume of the flow cell + hoses

Table 3 - Minimum volumes to be pumped out with unknown monitoring well diameter and groundwater level drop

Borehole diameter **100** (mm)						Flow cell volume **0.5** (l)				
Filter gravel porosity 35 %										
Influence zone 2 (dm)						Elevation outside filter gravel 25 %				
Monitoring well diameter (mm)	Level drop (cm)									
	1	2	3	4	5	10	20	30	40	50
32	2.5 L	2.5 L	2.5 L	2.5 L	2.5	L 2.5 L	2.5 L	2.5 L	2.5 L	2.9 L
50	2.5 L	2.5 L	2.5 L	2.5 L	2.5	L 2.5 L	2.5 L	2.5 L	3.0 L	3.5 L
63	2.5 L	2.5 L	2.5 L	2.5 L	2.5	L 2.5 L	2.5 L	3.0 L	3.6 L	4.2 L

If the above criteria cannot be met, it is necessary to change out at least 5 times the monitoring well volume.

Before actual 'purging', the field worker should check for floating layers. Purging may pump out floating layers that flow in slowly. It is possible to check for floating layers with instruments such as bailer samplers, liquid layer samplers (with transparent reservoirs) and floating layer detectors. The presence of a floating or sunken layer implies that the analytical study of groundwater samples from the monitoring well offers little added value for the survey. Do not conduct analyses in these cases, as the results would not be useful in determining groundwater quality.

During purging, shortly before the start of actual sampling, switch to a pump flow rate in accordance with the pump flow rate during sampling. (sampling flow rate of 0.5l l/min or lower – depending on the actual field inflow, the type of parameter for which the sampling is carried out and the type of monitoring well in which the sampling is carried out). Do not switch the pump off between purging and actual sampling.

Performance of water exchange requires knowledge of geohydrological conditions and monitoring well volume and structure, to prevent overpumping and preferential extraction. Pumping dry (emptying) the monitoring well should be avoided. The extraction pumping flow rate must be set so the filter section of the monitoring well is never exposed to air (except for intersecting monitoring wells).

Record the groundwater level before the start of purging as well as after sampling.

8.5.2 VOLUME-BASED SAMPLING

- Apply an extraction flow rate at which the pumping flow rate is set so that:
  - no turbulence occurs.
  - the filter portion of the monitoring well tube is never exposed to air.
  - dry pumping (evacuation) of the monitoring well is avoided.
- Add a minimum of 5x the water column in the monitoring well (volume of water present in filter section and blind tube) by the following formula:

$$V = (\pi * r^2 * h)/1000$$

with π = 3.14; r = monitoring well radius (= outer diameter of monitoring well/2; in cm)), h = height of the water column present in the monitoring well (in cm).

Table 4 summarises respectively the volume of 1x water column per linear metre and 5x water column per linear metre instead of different outer diameters.

Table 4 – Calculated volume of water for 1x water column and 5x water column per 1 m filter tube length (filter and blind tube)

Outer diameter of monitoring well	V = 1x water column for 1 m monitoring well length (filter or blind tube)	V = 5x water column for 1 m monitoring well length (filter or blind tube)
mm	L	L
32	0.804	4.020
50	1.960	9.820
63	3.120	15.600

- Discontinue the pre-flushing after stabilisation of the field parameters (e.g. EC, pH, O_2, Temperature; specific guidelines are included in § 8.7)
- Shortly before proceeding with groundwater sampling, the pump flow rate should be reduced to the anticipated sampling rate.
- Pre-flushing using the **volume-based method** is **not permitted**:
  - if floating layers or other observable impurities are raised as a result of pumping the water column. In this case, the pre-flushing
    using the volume-based method must be terminated and the monitoring well may no longer be used for groundwater sampling. A recommendation for decommissioning should be made by the eBSD (see §11).
  - when the filter portion is exposed to the air.
  - in the case of sliding monitoring wells (see § 8.9).
  -

8.6 DISPOSAL OF PUMPED GROUNDWATER

8.6.1 GENERAL

- The soil rehabilitation expert is responsible for proper disposal of the groundwater that is pumped out. The way in which the pumped groundwater is removed must be recorded in the field report of the groundwater sampling (see also § 10).
- Regardless of the situation with regard to contamination, it is not permitted to discharge pumped groundwater into monitoring wells.

8.6.2 DISCHARGE INTO SEWAGE OR ON UNPAVED TERRAIN

Discharge into the sewer or on unpaved land is only permitted if the following conditions are met:
- Sensory observations do not indicate potential presence of groundwater contamination (such as colour, odour, very high EC, deviating pH values, high PID measurements);
- absence of pure product/film on water level;
- analytical data from previous studies have not demonstrated significantly increased concentrations;
- the sewage system into which the discharge takes place, has sufficient storage and drainage capacity to handle the discharged groundwater without causing problems;
- in the case of separate sewage systems, discharge into the drainage of rainwater is only permitted if the requirements for discharge into surface water are met.

8.6.3 DISCHARGE INTO SURFACE WATER

Discharges into surface water must meet the conditions in the section above. In addition, the general water quality must meet the local discharge standards for surface water.

8.6.4 DISCHARGE INTO WASTE WATER TREATMENT PLANT (WZI)

If the groundwater sampling site features a water treatment plant, it is permitted to discharge the pumped groundwater into this WTP, under the following conditions:
- the owner, user, operator of the site agrees to this;
- the composition and size-order of groundwater concentrations are known;
- the technical specifications of the WZI are known;
- The WTP is suitable for treatment of the pollutants present in the groundwater: for instance, it is not permitted to discharge Cl-VOCs into a hydrocarbon separator (HCS);
- The WTP receiving the discharge has adequate capacity to handle the discharged groundwater.

8.6.5 DISPOSAL IN BARRELS FOR PROCESSING ACCORDING TO CURRENT REGULATIONS

It is always required to collect pure product and extremely contaminated groundwater (i.e. based on known concentrations from past surveys or on sensory observations) in vessels and transport them to a recognised processing company.

8.7 MEASUREMENTS DURING PURGING

If conditions permit (sufficient inflow), at each groundwater sampling, the field parameters pH, conductivity (EC) and temperature (T) must be measured. The recording of the oxide reduction potential (ORP) and the oxygen content (O_2)is normally carried out at a later stage of a project (BBO, BSP, BSW). In specific cases, it may also be desirable to measure additional parameters using ion-selective electrodes (e.g. H_2CO_3, HCO_{3^-}, CO_{32^-}, Fe^{2+}), prior to groundwater sampling, are desirable.

Field parameter measurements must take the following into account:

- Flow-through cell (Figure 4)
  Field measurements (electrode determination of pH, Ec, T, O_2, ORP) cannot be made in buckets, jars, etc., with the groundwater flowing into the container from above, as this is prone to outside influence. A flow-through cell must be used to take these measurements.

  The flow-through cell (Figure 4) is intended to improve in-line measurements of field parameters. It consists of a transparent cylinder through which water flows from bottom to top at a constant rate. The water touching the electrodes has not been in contact with air which may be present at the top of the flow-through cell.

  The same pump used for the actual sampling must also be used to pump the groundwater up when taking these measurements. Remove the flow-through cell when proceeding to groundwater sampling.

  The flow-through cell shall have a sufficient volume to allow a representative measurement but shall not be too large to avoid long turnaround times, i.e. the smaller the volume can be measured under LF flow rates. Note that the volume of the flow cell is appropriate for the installed flow rate.

Figure 4 – Examples of a flow-through cell

- Calibration
  Before measurement, calibrate the different electrodes using a calibration liquid or solution:
  - See procedure CMA/2/I/A.1 (Determination of pH);
  - See procedure CMA/2/I/A.2 (Conductivity);
  - See procedure CMA/7/A.5 (Redox potential).

  Also inspect the electrodes regularly for chemical scaling, and clean and replace them as needed.

- Gas bubbles
  During measurement of redox potential and oxygen content, be mindful of the formation of gas bubbles near the electrode in the flow-through cell. If gas bubbles are present, adjust the flow rate of the pump.
- Redox potential conversion
  Convert the redox potential measured in the field (Em) to the redox potential with respect to the standard platinum/hydrogen electrode (Eh) (see CMA/7/A.5). The reference value for this conversion (Eref) depends on both the temperature and the redox electrode type.
- Stabilisation of parameters measured
  It is only permitted to take a groundwater sample after stabilisation of the parameters measured. It is necessary to monitor and log measurement values to demonstrate stabilisation through the time series. This must show both the measurement value of the field parameter and the time span between the measurements. A minimum of 5 measurement values shall be displayed, where the last 3 measured values of the series of measurements shall meet the stabilisation criteria. If groundwater sampling is carried out in the event of non-stabilisation of parameters and/or the occurrence of gas bubbles, this should be included in the field work report and the final soil study report as a deviation from the CMA procedure. In addition, it is also necessary provide an explanation for this.

  Stability of the field parameters shall be achieved when the previous measurement:
  - deviates from the measured conductivity value (EC) by no more than 1% for measurement values > 500 µS/cm, or deviates by no more than 5 µS/cm for measurement values ≤ 500 µS/cm;
  - deviates by no more than 0.1 pH units for pH;
  - by no more than 0.2 mg/l (for values <2 mg/l) or 0.4 mg/l (for values > 2 mg/l) for dissolved oxygen (O_2);
  - by no more than 0.2 °C for temperature (T).

  Between 2 measurements, at least the volume of the flow cell with the suction hoses shall be pumped through.
- Poor inflow
  In cases of poor groundwater inflow, air bubbles are unavoidable. This disrupts the measurements to such an extent that they become unreliable, and thus also unusable. To minimise air bubbles, it is necessary to reduce the pump flow rate. For this, use hoses with smaller inner diameters. For field parameter measurement, it should be noted that this may result in a faster apparent stabilisation of field parameters.
- Level drops (ΔP)
  During purging, use level measurements to monitor how the groundwater table responds to the pump flow rate. Strive for a level drop stabilisation that meets the maximum permitted level drop (see § 8.5).

  The effective realised level drop should be recorded with a series of measurements. If the indicated criteria cannot be met, record the actual level drop (after stabilisation) as a deviation from the regulations.

Before the start of sampling, always ensure an adequate water column between the PE hose inlet and the stabilised groundwater table level. If (with non-intersecting/deep monitoring wells) the groundwater table level stabilises within the filter frame, then contrary to the regulations, the field report must include the following statement:
'poor inflow (Q = 100 ml/min) – sample aerated: actual level drop (Δ P = …), height of remaining water column between hose inlet and stabilised level (Δ H = …).' with indication of the Δ P and Δ H values measured onsite.

8.8 PROCEDURE FOR GROUNDWATER SAMPLE FILTRATION

- Parameter-specific guidelines: See CMA/1/C.

- For each parameter, if available, the CMA procedure regarding analysis should be consulted. After consultation with the lab, filtration in the field may or may not be required.
- If, in agreement with the lab, filtration is carried out in the field, this should preferably be done under slight overpressure. Any additional filtration carried out on the field and/or filtration different from the above guidelines must be recorded as a deviation in the field report of the groundwater sampling carried out (see § 10).

8.9 MONITORING WELLS WITH 'POOR' GROUNDWATER INFLOW

As there are many loamy and clayey soils in Flanders, it is not always possible to install monitoring wells from which large amounts of groundwater can be withdrawn. In addition, soil structure is not always the reason why a monitoring well 'doesn't give enough water': a whole range of other causes are possible. The expert must always check the cause of a malfunctioning monitoring well. In the reporting on the fieldwork carried out (see CMA/1/A.1 – Fixed part of Earth): to be indicated whether or not it is easy to take steel out of the installed monitoring wells or whether there is a poor inflow of groundwater.

A monitoring well has a poor inflow when the level drop (ΔP) is greater than 50 cm at a pump flow rate of 100 mL/min. In these cases: check the cause of the poor inflow.

8.9.1 POORLY POSITIONED MONITORING WELLS

Poor inflow at monitoring wells where the geological characteristics make a "normal" sampling possible (i.e. if the permeability K is high enough) may be caused by:

- a poorly positioned monitoring well, for example, due to inappropriate positioning of the filter (in clay layer, too shallow, wrong drilling technique used, not used in installation,....), and/or;
- a blockage of the filter caused by the bentonite stop placed above the filter gravel, and/or;

Groundwater sampling shall not be carried out in the case of poorly positioned monitoring wells. Analytical results obtained by sampling these monitoring wells are not accepted within the soil survey.

The eBSD must 1) advise to decommission poorly placed monitoring wells or 2) proceed with decommissioning (see guidelines §11).

8.9.2 POOR INFLOW DUE TO CLOGGING

Cloggin of the monitoring may be caused by sedimentation of fine-grained soil particles or biofouling, or chemical precipitation such as iron or lime deposits.

Redevelopment of the low-yield monitoring well due to clogging may be considered (see OVAM report on monitoring well placement and well development). If redevelopment of the poorly performing monitoring well does not result in improved groundwater inflow, it should be decommissioned after all (see §11).

8.9.3 POOR INFLOW DUE TO LOCAL SOIL STRUCTURE

If the evaluation shows that the poor influx originates in local soil construction, the following guidelines should be observed:

- Groundwater sampling shall only be carried out by low-flow sampling.
- Use small diameter sampling hoses (4x6mm mm). This enables lower pump flow rates, to minimise impact on the field measurements and the groundwater samples obtained.

8.9.4 GROUNDWATER SAMPLING WHEN DRAINING THE MONITORING WELL DURING LOW-FLOW PURGING

In the case of monitoring wells, which, as a result of site-specific soil construction, are characterised by poor groundwater influx and where low-flow steelworks the filter of the monitoring well during purging, only groundwater sampling can be carried out when following the following directives:

- LFS is permitted after the following conditions are met:
  - at least 50% of the water column has been restored;
  - the water column at the start of LFS is not intersecting with the monitoring well filter;
  - the waiting time of 72u hours is not exceeded.
- Start sampling according to the target parameters for analysis and not according to the order set out in § 8.11.3. If in the interest of the study the determination of volatile components and heavy metals is necessary, the sampling for volatile components should be carried out first, followed by the sampling for heavy metals and then the other parameters.
- Field parameters are not determined, including sampling for analysis in the lab
- Groundwater sampling after dry-pumping of monitoring wells is considered as a deviation from the guidelines included in the CMA procedure. The procedure followed should be clearly described in the field report.

If the groundwater sampling directives are not complied with when the filter is emptied, groundwater sampling must be carried out via passive samplers (see Directive CMA/1/A.3) or the installation of drilling with soil steelworks (see Directive CMA/1/A.2) at the depth interval at which filtering was provided.

8.10 PERIOD BETWEEN MONITORING WELL INSTALLATION AND SAMPLING

Installing a level indicator causes disturbance of the geochemical condition of the subsoil. Immediately after monitoring well installation, the composition may change due to dissolution, complexation, etc. Also, installed bentonite plugs take 24 hours to swell completely. For this reason, always wait at least 1 week between monitoring well installation and sampling, unless soil conditions require a different approach. In the case of deviations from the above, the soil survey report must clearly state that the sampling procedure was not followed. The soil survey report must also include the necessary arguments.

This rule does not apply to what are known as ‘temporary’ monitoring wells (see §4.5), which enable immediate sampling after filter installation.

8.11 GROUNDWATER SAMPLING

8.11.1 GENERAL GUIDANCE

- Wherever possible, when taking consecutive groundwater samples, work from the least polluted to the most polluted locations.
- Replace the sampling hose before each sample. It is not permitted to leave sampling hoses in monitoring wells. This also applies for reuse in the same monitoring well as well as use of the same sampling hose for flushing after installation, or purging, or sampling of different monitoring wells.
- Sampling hoses (both PE and silicone hoses) must be as short as possible.
- Use a new pair of disposable gloves for each groundwater sampling operation.
- Groundwater sampling must be done at a uniform constant laminar flow rate.
- Fill the containers completely and bubble-free in a single motion. The container must not contain any air bubbles. It is permitted to top up containers that are found to contain air bubbles after sampling, unless analysing for volatile compounds (see § 8.11.4).
- In the case of overfilling of containers with preservatives, it is necessary to take a new sample (with a new container).
- After each sampling operation, clean or replace the part of the sampling equipment that came into contact with the groundwater. If certain parts come into contact with groundwater and cannot be cleaned or replaced, it is not permitted to use that pump type to sample groundwater for a soil survey under the decree. Depending on the characteristics of the technique, it may be usable for flushing after installation (see also Table 2).
  - Immediately after sampling, prevent the sample increasing in temperature. The samples taken must be kept refrigerated on site in a refrigerated box (with frozen refrigerating elements) or in a refrigeration unit, awaiting and during transport to the storage place for the samples or the laboratory.

8.11.2 CONTAINERS

- The containers must be immediately labeled with ID information (project number, monitoring well number, sampling date, etc.). Procedure CMA/1/B provides guidelines on the containers to be used and preservation of the groundwater samples taken.
- With regard to preservation, it is recommended to work with pre-preserved containers. Addition of preservatives in the field is difficult and less precise.
- It is not permitted to use containers with expired preservatives.
- It is not permitted to use containers made from materials other than those listed in procedure CMA/1/B.

8.11.3 GROUNDWATER SAMPLING

For the purposes of groundwater sampling, a distinction is made between low-flow sampling and volume-based sampling.

8.11.3.1 LOW FLOW SAMPLING[3]

Micropurging or low flow sampling is the standard method in soil decree-based studies and should be used for the determination of groundwater quality and for the containment of pollution plumes.

For low-flow sampling, groundwater is abstracted at a very low flow rate (100 ml/min – <500 ml/min) at the filter section of the monitoring well (at the depth at which a sample is to be taken). The overlying water column is not “replenished” in this way. If there has been sufficient flushing at the level of the filter section (see §8.5.1) a sample may then be taken. It may be handy to use packers here, to reduce the volume of water to be removed during flushing.

The procedure for low-flow groundwater sampling is as follows (mandatory):

- Start sampling without stopping the pump after purging.
- During purging and sampling, do not allow the water level to fall into the filter section, unless sampling from an existing intersecting monitoring well.
- When sampling, the intake opening should be at least 0.5 m above the bottom of the filter (depending on the presence and size of sludge trap and the height of the groundwater column present in the monitoring well).
- The sampling is carried out at the same or a lower rate than the purging (see §8.5.1):
    - General: the target sampling flow rate is 100 - 200 ml/min, depending on the parameters to be analysed. Flow rates <100 ml/min are considered deviations from the low-flow sampling procedure and should be reported accordingly. Sampling rates <50 ml/min are not allowed.
    - Sampling flow rate for intersecting monitoring wells: 100 ml/min
    - Sampling for volatile compounds: apply a sampling flow rate of 100 - 200 ml/min (mandatory).

[3] EPA and other US guidelines use the term "micropurging".

- When filling the receptacles, turbulence and air impact must always be avoided by:
  - use suitable sampling equipment: see Table 2
  - pumping at an adequately low flow rate (see above);
  - keeping the container upright;
  - inserting the PE hose to the bottom of container;
  - minimising contact between PE hose and liquid in the container (max. 0.5 cm);
  - bringing the PE hose up with the rising liquid level;
  - after filling the container with preservative remove the part of the PE hose which has been in contact with preservative (i.e. cut off) before filling a subsequent container;

- Fill the containers in the following order:
  1. containers without preservative;
  2. containers with solid preservatives (e.g. ascorbic acid);
  3. containers with liquid preservatives.

  Avoid overfilling the containers with preservative.

8.11.3.2 VOLUME-BASED SAMPLING

High flow sampling should only be used for the determination of average groundwater concentrations in relation to malting studies and at cadastral working areas.

- The sampling starts without stopping the pump after purging, and the same requirements must be met as during purging (see § 8.5.2)
- When sampling, the intake opening should be at least 0.5 m above the bottom of the filter (depending on the presence and size of the sludge trap and the height of the groundwater column present in the monitoring well).
- When filling the receptacles, turbulence and air impact must always be avoided by:
  - pumping with an adequately low flow rate (cf. flow rate low flow sampling);
  - keeping the container upright;
  - inserting the PE hose to the bottom of container;
  - minimising contact between PE hose and liquid in the container (max. 0.5 cm);
  - bringing the PE hose up with the rising liquid level;
  - after filling the container with preservative, removing the part of the PE hose which has been in contact with preservative (i.e. trimming) before filling a subsequent container;
- Fill the containers in the following order:
  1. containers without preservative;
  2. containers with solid preservatives (e.g. ascorbic acid);
  3. containers with liquid preservatives.

  Avoid overfilling the containers with preservative.

8.11.4 SAMPLING OF GROUNDWATER CONTAMINATED WITH VOLATILE COMPOUNDS - HEAVY METALS

See guidelines included in CMA/1/C.

8.11.5 PFAS SAMPLING GUIDELINES

See guidelines included in CMA/1/C.

8.12 SUMMARY OF LOW-FLOW SAMPLING CRITERIA

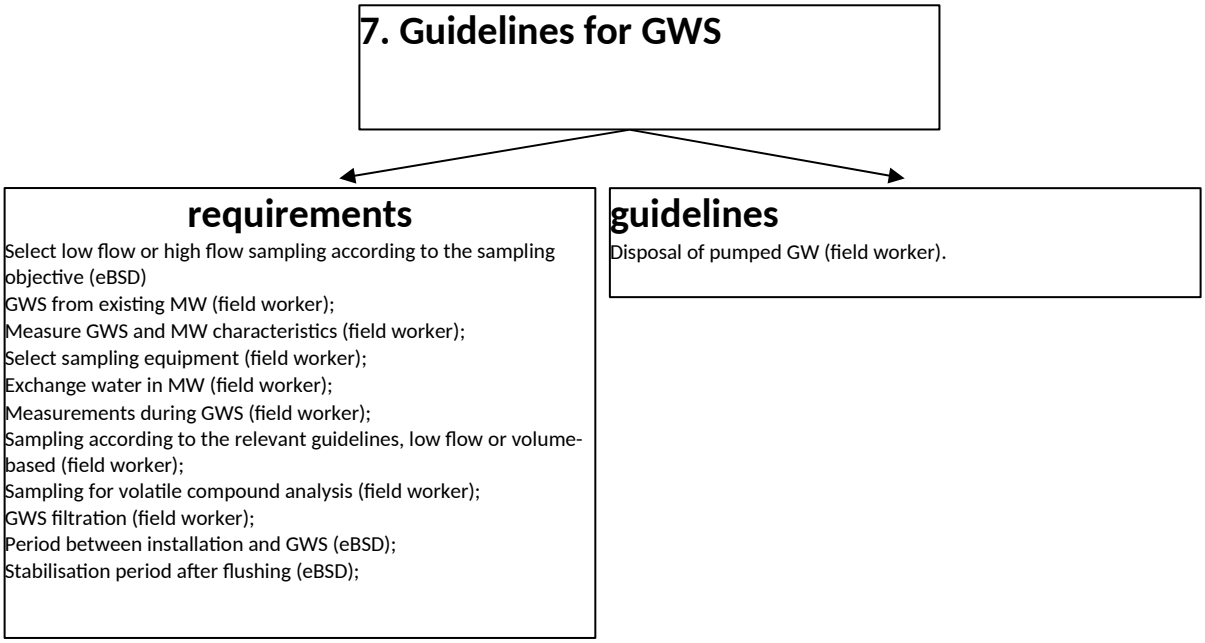
- Constant low flow rate: 100 to 500 ml/min.
- Do not exceed the natural recharge rate of the aquifer being sampled.
- Water level must not fall below the top of the filter section.
- Check for any floating layers before the exchange.
- Summary criteria: see Table 5

Table 5 - Summary of low-flow sampling criteria*

	Freatic (shallow) monitoring wells	
Filter frame	Intersecting	Non-intersecting
Purging flow rate	<200 ml/min	<500 ml/min
Sampling flow rate for volatile compounds	<100 ml/min	<200 ml/min
Sampling flow rate for non-volatile compounds	<100 ml/min	<500 ml/min
Maximum level drop	10 cm	50 cm

* flow rates <100 ml/min are considered deviations from the low-flow sampling procedure and should be so reported. Sampling rates <50 ml/min are not allowed.

8.13 DIAGRAM



9 MEASUREMENT OF FLOATING AND SUNKEN LAYERS

9.1 FLOATING LAYERS

If the monitoring well contains a floating layer, do not take groundwater samples. Groundwater samples from filters that contain a floating layer are not usable in determining groundwater quality.

'Floating layer' means: the presence of pure product in the groundwater table. This normally involves products that are less dense than water, causing them to float on the groundwater. It is however necessary to clearly distinguish between 'pure' product and a floating layer.

9.2 DEFINITIONS

***Pure product**: (liquid) contamination occurring in the soil as a separate phase. whether or not mobile. The term 'pure product' is closely related to the term '**retention capacity**' of soil. The pure product is mobile (i.e. it does not stay in the same place, but rather spreads with the groundwater, for instance) if it exceeds the retention capacity of its soil.*

***Floating layer**: pure product (of low water solubility) occurring at the groundwater level (at the groundwater table and in the water capillary zone) and giving rise to a pure product layer. In this case, the pure product is mobile. If it does not form a liquid layer, the pure product is capillary.*

***Retention capacity:** the retention capacity of a soil is defined as the average volume of pure product that the soil can hold per unit volume.*

9.3 PURE PRODUCT VERSUS FLOATING LAYER

The difference between 'pure product' and 'floating layer' is in whether or not the product is mobile at groundwater level. Thus, this also depends on seasonal effects, for instance, which may result in the formation of floating layers due to the rising groundwater table.

Figure 5 below shows the occurrence of a pure product zone of oil components around groundwater level.

From top to bottom:

- The water-unsaturated zone (air in soil pores);
- the oil-contaminated zone (oil/air in the pores; "competition" between air and oil);
- the oil-contaminated zone (oil/water in the pores; "competition" between water and oil);
- the water-saturated zone (water in the pores).

In normal (mineral) soils, the adhesion forces between the water and soil particles are greater than the adhesion forces between oil and the soil particles, which are in turn greater than the adhesion forces between air and the soil particles. This gives rise to capillary effects, where the oil spreads into the small pores of the dry part of the soil (the oil 'wets' the soil particles), forcing the air out (to the large pores). In the wet soil zone, water and oil compete for the pores, with water now 'favouring' the contact surface with the soil particles. In a rising water table in an oil-contaminated layer of soil, the water will force the oil out of the small pores, with the oil partially captured in the pores. This creates a 'smear zone' (the zone where the oil is 'smeared', as it were) around the groundwater level due to the rising

and falling groundwater.

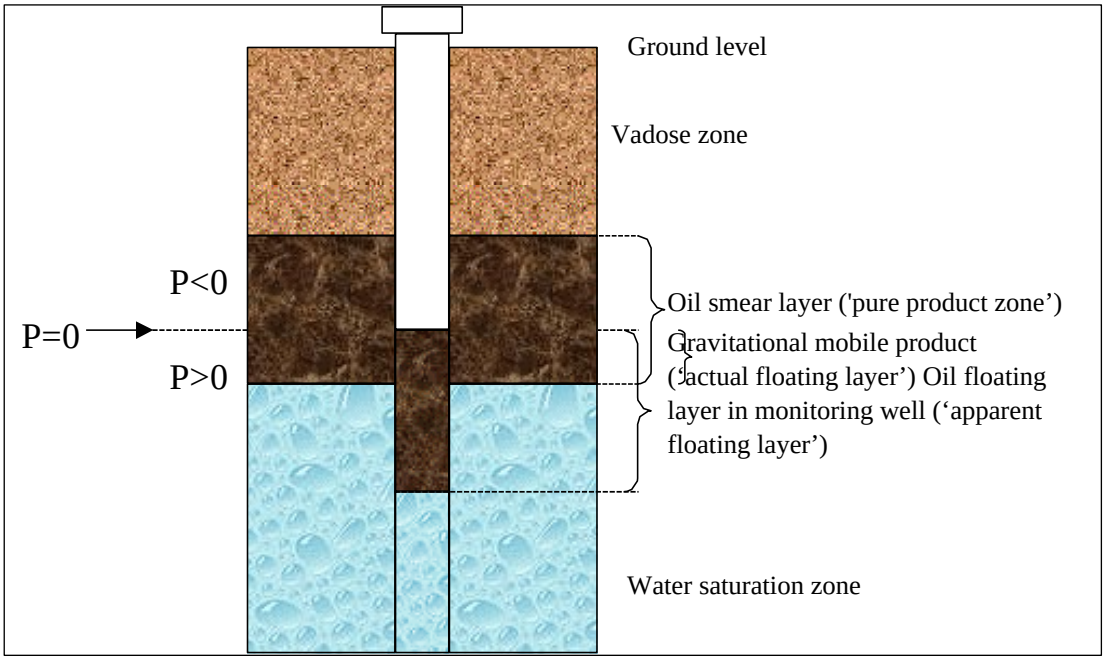


Figure 5 - Diagram of how a pure product zone (oil) occurs around the groundwater level and the terms 'apparent' and 'actual' floating layer (P = oil - capillary pressure, P = 0: oil level)

A floating layer may occur in an intersecting monitoring well installed in the spread zone. However, this does not mean that the soil contains an actual (mobile) floating layer. This depends on the capillary pressure in the pores in the oil-contaminated zone. An 'actual floating layer' only occurs if an oil layer forms (definition: horizontal plane where the oil capillary pressure P is equal to zero). Below the oil level, the pressure is positive (compare with the concept of hydrostatic pressure); above the oil level the pressure is negative.

The the soil does not contain an oil layer, capillary oil is only present if the oil flows into a monitoring well by a means other than gravity.

Monitoring wells installed to check for floating layers must always feature an intersecting filter.

9.3.1 MEASUREMENT OF FLOATING LAYERS

The fieldworker may measure floating layers using a liquid layer sampler (with transparent reservoir) or a special probe for measuring floating layers. The floating layer being measured must be 'in balance'. This means the oil pressure is in balance with the pressure of the product in the soil. The thickness of a floating layer in a monitoring well that was recently pumped out will change over time and is therefore not in balance.

The thickness of floating layers found on the same site, measured in monitoring wells, can vary widely, in both time and location, due to the heterogeneity of the subsoil as well as groundwater level fluctuations. These latter factors can have a major impact due to seasonal variations. It is therefore also advisable to measure the floating layer thickness multiple times.

If a floating layer is not found on a site strongly suspected of containing one, it is also advisable to take multiple measurements more than 1 week after monitoring well installation.

The presence of a floating layer (pure product) suppresses the water column in the monitoring well. However, a fieldworker that knows the density of the product making up the floating layer and has measured the thickness of the floating layer can also calculate the actual water level (§ 9.3.1.1).

The thickness of the floating layer consisting of LNAPL (light non-aqueous phase liquids) in a monitoring well is typically greater than the thickness of the floating layer in the surrounding aquifer. The literature indicates a factor of between 2 to 10 times. The thickness measured in the monitoring well is also called the apparent thickness of the floating layer.

Depending on the nature of the pure product however, the thickness of the floating layer may also be less than that of the zone above which the product occurs in the water layer. This may occur, for instance, with water soluble substances such as benzene. So the rule of thumb that 'the thickness of the floating layer in the monitoring well is 2 to 10 times the thickness in the aquifer' does not always apply.

Given the possible variations in floating layer thickness in the monitoring well, it is rarely possible to calculate the 'precise' thickness of the zone in the soil based on the floating layer thickness. Only an estimate is possible. This is only possible if a series of field and soil characteristics are known. Given the complexity of the problem, a simple method is not yet available to determine this.

9.3.1.1 WATER LEVEL CONVERSION

Calculate the water level in a monitoring well with a floating layer as follows:

$$h_c = h_m + (H_o * (\rho_o / \rho_w))$$

where:
- h_c = corrected water level(*) (m);
- h_m = measured water level – floating layer dividing plane(*) (m);
- H_o = floating layer thickness (m);
- ρ_o = density of floating layer product (kg/m^3);
- ρ_w = density of water (kg/m^3);
- (*)Measure all levels with respect to a fixed reference level, such as m TAW.

9.3.1.2 THICKNESS OF PURE PRODUCT ZONE

The literature contains information on conversion of the thickness measured to the corresponding thickness in the aquifer (EPA, Fetter, 1999, de Marsily, 1981). Always exercise the necessary caution when applying this method. In addition, it is advisable to use calculation methods based on empirical equations.

The most reliable method for determining the thickness of the zone containing the pure product is to take undisturbed samples. It can then be tested by an oil detection test and/or by adding oil-red to

test presence of pure product.

9.3.2 SUNKEN LAYERS

If the monitoring well contains a sunken layer, do not take groundwater samples. Groundwater samples from filters that contain a sunken layer are not usable in determining groundwater quality. It is however permitted to take a sample of the sunken layer (pure product) for analysis. A special probe may also be used to detect and measure the thickness of any sunken layers. Given the nature of the products that form sunken layers (denser than water), it is not possible to perform a conversion to determine the 'thickness' of the zone where the sunken layer is found in the aquifer.

9.3.3 PURE PRODUCT SAMPLING

If sufficient pure product is found in a monitoring well (thickness > ~1cm cm), a liquid layer sampler (see § 7.2.3.2) or peristaltic pump may be used to sample it.

9.4 IMPLEMENTATION OF OIL RECOVERY TESTS (ORT)

9.4.1 GENERAL GUIDANCE

Oil recovery tests are carried out on cutting monitoring well pipes to determine the gravitational mobility of a floating layer on site.

- Ensure that the monitoring wells are correctly installed with a filter cutting the groundwater table to allow sampling of the pure product (see § 4.4).
- Oil recovery tests may not be carried out until 7 days after the installation of the monitoring well.
- When carrying out an oil recovery test on existing monitoring wells, check that the monitoring wells are in good condition and check the technical data of the monitoring well (depth, inner diameter, drilling diameter, filter length knowledge, etc.; see also §8.2)

9.4.2 CARRYING OUT OIL RECOVERY TEST

- Prior to the oil recovery test, determine the thickness of the floating layer present in the monitoring well filter and the ground water level by means of a floating layer thickness meter:
  - Lower the probe slowly into the monitoring well, keeping it as vertical as possible.
  - The probe will give a signal (such as beep or light) as soon as it reaches the oil pure product layer. Record the depth at which this occurs.
  - Allow the probe to drop further until it reaches the water layer. Again, the meter will give a signal. Record this depth as well.
  - The thickness of the floating layer (oil) is the difference between these two depths.

- Start the oil recovery test: Pump the pure product present in the monitoring well with intersecting filter completely through skimming. When pumping, avoid mixing the oil and groundwater.
- Collect the pumped floating layer in a graduated cup;
  - Determine the pumped-up volume
  - Describe the pure product: colour, passive odour perception (weak, moderate, strong), approximate viscosity (low, medium, high)
- Then measure the recovery of the floating layer through multiple times.
- Repeat have ORT at least 1x after stagnation of floating layer thickness (Figure 6).
  Please note that re-forming a floating layer (floating layer repair) does not mean that it is mobile pure product. This requires comparing the volume of pure product pumped with the calculated volume of pure product present in the monitoring well filter and in the filter deposit (see next bullet).

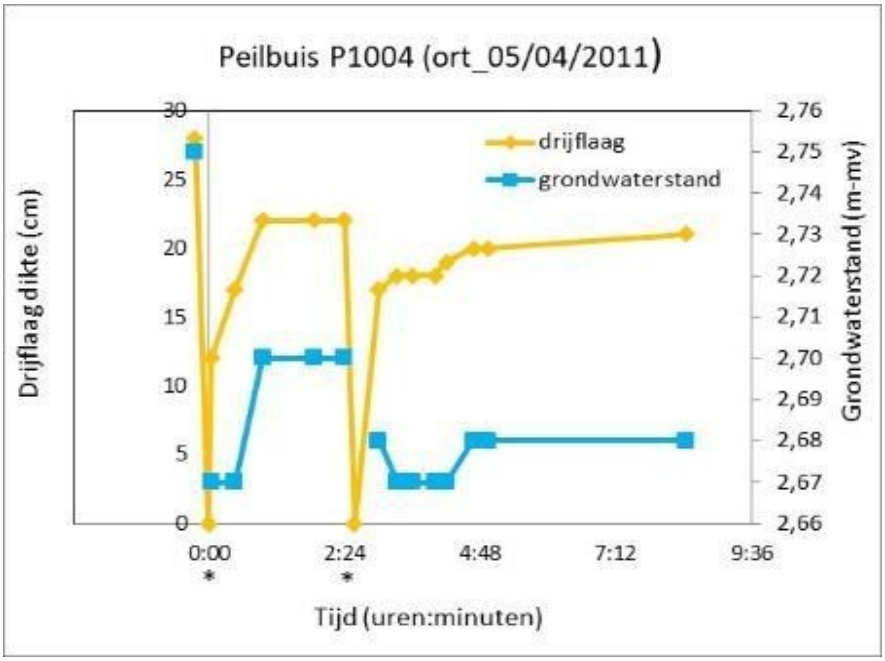


Figure 6 - Example of follow-up of oil recovery test showing the thickness of the floating layer, the groundwater level and the time (*) of pumping

Peilbuis P1004 (ort _05/04/2011	Monitoring well P1004 (ort _05/04/2011
Drijflaagdikte (cm	Floating layer thickness (cm
Tijd (uren:minuten)	Time (hours: minutes)
Grondwaterstand (m-mv)	Groundwater level (m-mv)
drijflaag	floating flayer
grondwaterstand	Ground water level

- Compare the volume of pumped pure product with the calculated volume of pure product present in filter and filter packing according to the following formulae:
  - Volume LNAPL in filter (in L): $V_f = D_o * \pi * R_i^{\ 2} * 10^{-3}$
  - Volume LNAPL in filter deposit (in L): $V_{fo} = [D_o * \pi * R_{bore2} - V_f] * \rho_{filter\ gravel} * 10^{-3}$
  - Volume of LNAPL in filter and filter packing (in L) $V_{tot} = V_f + V_{fo}$
    With D_o = thickness of floating layer at time 0 (in cm), $R_{i\ monitoring\ well}$ = internal radius of monitoring well (in cm), R_{bore} = radius of bore (in cm) and $\rho_{filter\ gravel}$ = porosity of filter gravel (Figure 7)

If the inflated volume clearly exceeds the calculated volume, there is an inflow of pure product

and thus the presence of a mobile floating layer.

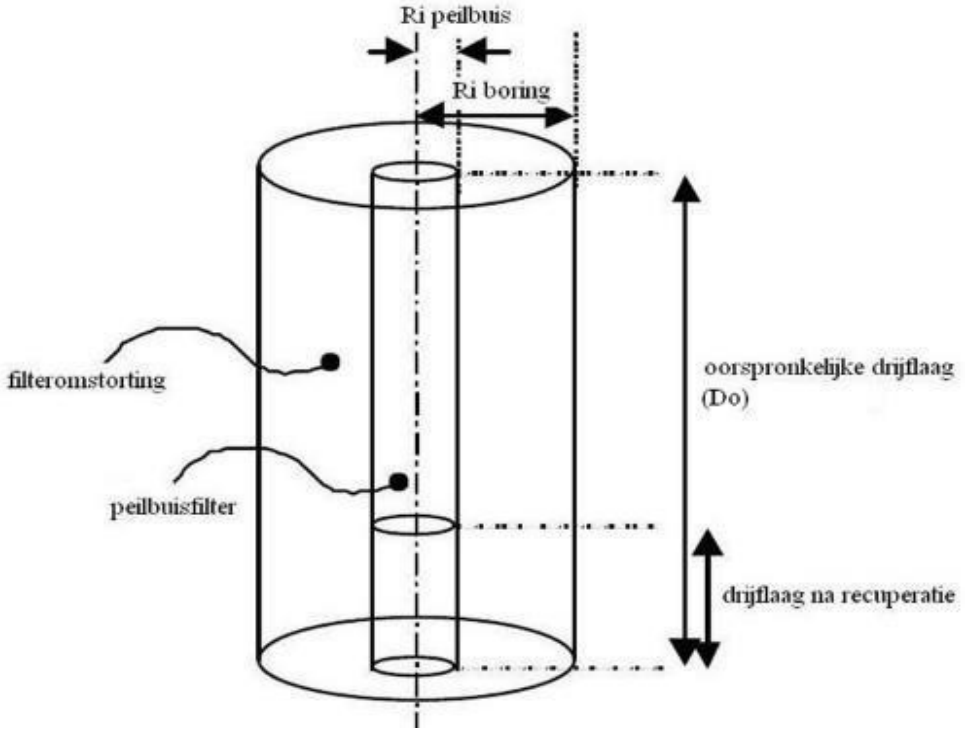
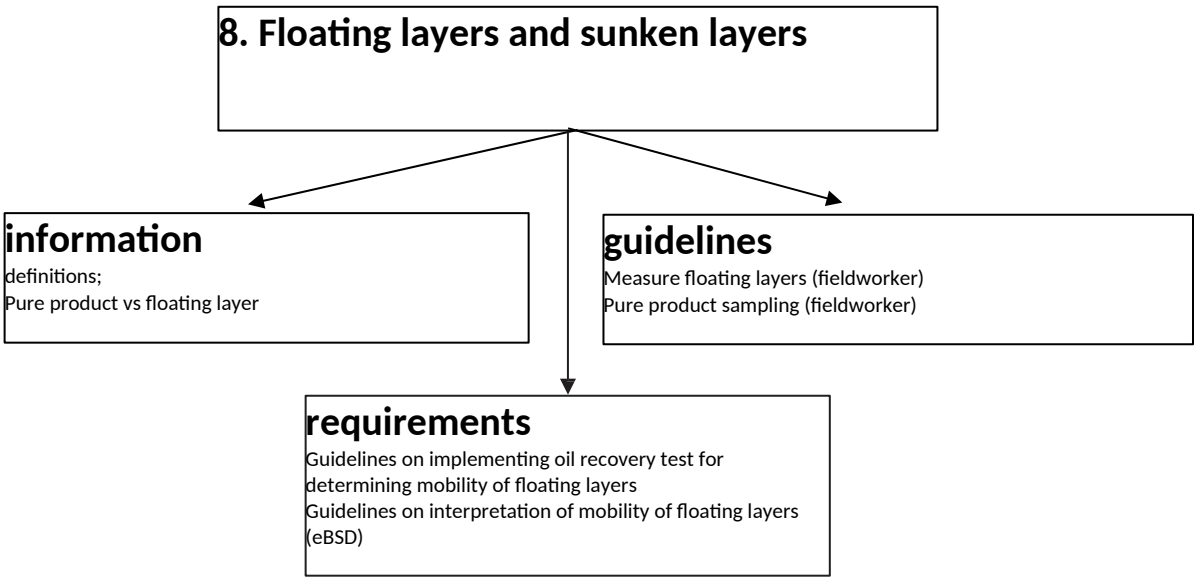


Figure 7 – Diagram of LNAPL in monitoring well, before and after pumping

Ri peilbuis	Ri monitoring well
Ri boring	Ri drilling
filteromstorting	filter deposit
peilbuisfilter	monitoring well filter
oorspronkelijke drijflaag (Do)	original floating layer (Do)
drijflaag na recuperatioe	floating layer after recovery

An example of a reporting template for the follow-up of an ORT is provided in Annex A.

9.5 DIAGRAM



10 REPORT

The fieldworker must draw up an initial description of the samples (sensory observations) immediately onsite and include it in the final soil survey reporting.

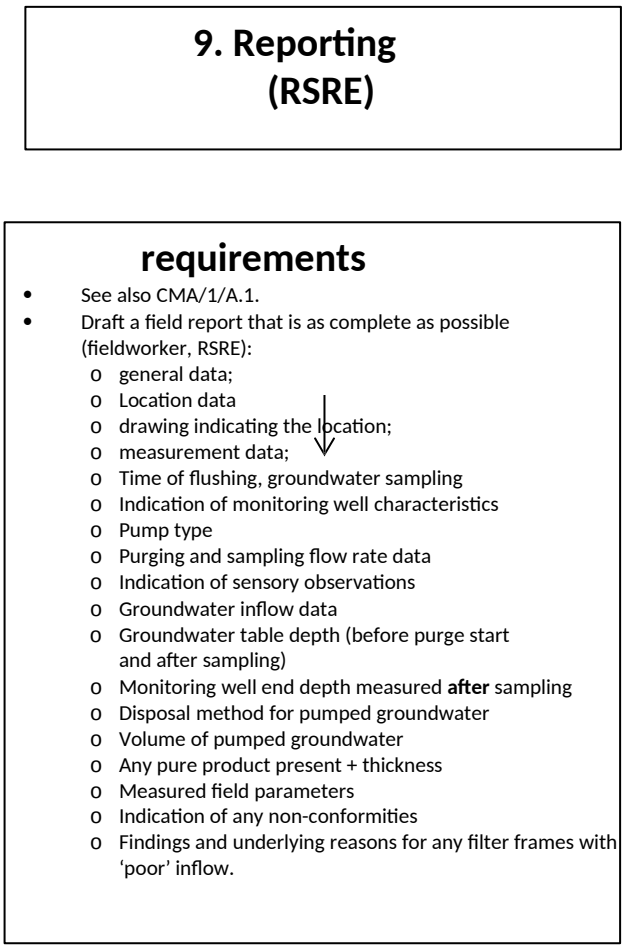
The field observation log must include the following details for each monitoring well:

- general data.
  - Groundwater sampling date
  - Survey site identification
  - Fieldworker identification
- monitoring well data:
  - Monitoring well number and filter depth
  - Monitoring well type (permanent/temporary, intersecting/non-intersecting, nested, MLW)
  - Pump types (for flushing, purging and sampling)
  - Presence of a sand/silt trap
  - description of the condition/usefulness of the monitoring well to be sampled (see § 8.2; § 8.3);
- sampling technical data
  - Environmental factors: because local environmental factors may affect the results obtained, it is appropriate to record them in the field report.
    This means: in full sun or in the shade, etc.
  - Depth of groundwater table before the start of purging and after sampling
  - Monitoring well end depth (measured before purging or after sampling)
  - Depth and thickness of any pure product present
  - Purge start and end times (including intermittent pumping intervals)
  - Flow rates for both purging and sampling
  - time-bound monitoring series:
    - measured values field measurements (pH, ORP, T; if applicable also O_2 content, conductivity, turbidity);
    - readings of drop in the measurements;
    - volume inflated at each of the measurement values field parameters/Δ P.
  - pumped ground water
    - Total volume
    - method of disposal;
    - organoleptic observations (colour, odour, limpidity, ...);
  - Total sampling duration
  - Any non-conformities with the requirements set out in the present procedure as well as other deviations identified onsite
    - if level drop criteria are not respected (Δ P: see § 8.7);
    - flow rate deviations LFS
    - missing monitoring well technical data
    - Presence of sampling hoses before sampling (including their condition);
    - Potential silting;
    - Surface damage on the monitoring well/surface box and potential consequences on results
    - Labelling intact?
    - groundwater sampling after filter drainage with purging
    - ...
  - In cases of poor inflow:
    - Evaluation of the cause

- purging time if this has not been done at the same time as the sampling (groundwater sampling in case of drainage of the monitoring well during low-flow purging);

- if applicable, volume of drilling water used in monitoring well siting and whether it has already been removed at installation (see § 4.4.9).
  Information should be included in the fieldwork assignment.
- in the case of sampling at poorly inflow filter settings (existing and/or newly installed monitoring wells), reference is made to §8.9 (Monitoring wells with a 'poor' groundwater inflow). The soil survey report must also include the corresponding findings and underlying reasons.

10.1 DIAGRAM



11 DECOMMISSIONING OF MONITORING WELLS

It is required to fill monitoring wells that are no longer in use or that must be decommissioned, for whatever reason.

The decision on decommissioning rests with:
- the eBSD in case of newly installed monitoring wells in an ongoing research phase;
- the owner of the monitoring well when sampling through existing monitoring wells.

Monitoring wells must be decommissioned in the following cases:
- Silting: first, it is permitted to attempt to flush the monitoring well and restore its flow. If the degree of silting precludes restoration of monitoring well flow, it is necessary to decommission the monitoring well.
- Damage to the blind pipe below ground level
- Monitoring well clogging due to improper use of clay plugs
- Influences from ground level that render representative sampling impossible
- Leakage, such as due to improper clay plug installation (both in nested monitoring wells and single monitoring wells)
- Inadequate groundwater inflow compared to the inflow expected based on lithology

Procedure for monitoring well decommissioning:
- If possible, completely remove the monitoring well. Next, fill the borehole according to the contamination and the structure of the subsoil:
  - If required by the contamination or soil structure or in cases of doubt regarding the presence of sealing layers, fill the borehole completely with swelling clay to prevent further spread of the contamination due to improper filling. Application of liquid grout from below is mandatory in this case (see CMA procedure CMA/1/A.1 – Solid part of the earth; §6.9 – Finishing of borehole/grouting).
  - If the penetrated layers are made up of highly permeable sediments (sand), it is permitted to fill the borehole filter gravel.
- If it is not possible to remove the monitoring well:
  - Cut the monitoring well off at at least 10 cm below ground level.
  - Next, fill the monitoring well (= the pipe itself) with grout, clay (bentonite) or another impermeable inert material (K < 10 to 8 m/s). For monitoring wells up to 8 deep, fill over the entire length of the monitoring well. For deeper monitoring wells, fill at least 8 m, with the filter section starting at least 4 m below the top of the filling. In addition, uncontaminated soil material may be used as filling material.
- At ground level, finish the filled well according to the use of the site.

12 ANNEX A: EXAMPLE OF REPORTING ON ORT IMPLEMENTATION

CMA/1/A.2 – Oil recovery test
Sample Field Report

General

Siting	
Date of implementation	
Performer (code or initials/company)	
Monitoring well no.	
Filter depth (m-mv)	
Monitoring well depth (m-mv)	
Inside diameter of monitoring well (mm)	
Borehole diameter (cm)	

Measurements prior to the execution of the ORT

Time	
Groundwater level (m-mv)	
LNAPL thickness (mm)	

Oil recovery test details

ORT	Time	Method	Volume of oil removed (L)	Volume of water removed (L)	Notes
1.					
2.					
……					

Visual properties of oil

ORT	Colour	Odour (weak, moderate, strong)	Viscosity (low, medium, high)	Other
1.				
2.				
….				

Follow-up recovery LNAPL after ORT

ORT	Time	GW position m-mv	Oil thickness cm	Recovery Hours – minutes	Anmerkungen
1					

2					
.....					

Calculated volumes LNAPL

V_{filter} (ml)	
$V_{filter\ packing}$ (ml)	
V_{total} (ml)	

Comparison of pumped LNAPL volume versus calculated volume

☐ $V_{pumped} < V_{calculated}$ → no LNAPL inflow, surface layer is not mobile
☐ $V_{pumped} = / \approx V_{calculated}$ → no LNAPL inflow, surface layer is not mobile
☐ $V_{pumped} >> V_{calculated}$ → LNAPL inflow, surface layer is mobile

Figures/Photos

Add figure showing ORT (cf Figure 6)
Add photo/photos pumped LNAPL

Sampling techniques for solid materials

CONTENTS

1 INTRODUCTION

This methodology replaces existing procedure CMA/1/A.15 of June 2024.

This procedure covers sampling techniques that can be used for solid waste and raw materials[1]. This includes the following sample types from the analysis packages for recognition as a laboratory for waste: construction and demolition waste, ash and slag, shredder waste, compost and wood waste.

For solid materials used in a specific application (e.g. a (sub)foundation or hardening layer, a line track with stoney slag or ash ballast in train tracks), the application is considered a static batch in itself, and in most cases these sampling techniques can be used

The same sampling techniques can often also be used for semi-solid paste sample types, e.g. for (dewatered) sewage sludges, filter cakes from various sludge fractions.

Sampling technique selection must be based on the guidelines and decision trees in CMA/1/A.14. The latter also gives further instructions on batch division, grab and sample size, guidelines for analysis of specific parameters, packaging, transport and logging.

CMA/1/A.18 gives the instructions for any on-site sample pretreatment.

2 EQUIPMENT AND MATERIALS

Below is a list of some commonly used sampling equipment and tools. Annex A provides further details, including on their operation. Annex B lists the possible applications in specific sampling scenarios.

2.1. Device that enables logging of spatial coordinates with an accuracy better than 5 m (e.g. GPS logger or camera with built-in GPS function) (optional)
2.2. Scoop*, with opening at least three times larger than the maximum particle size in the batch (D95)
2.3. Gouge auger* with auger body length of at least 60 cm
2.4. Soil auger* or soil auger set: Edelman auger, Riverside auger, etc. (other types possible)
2.5. Plunge borer, sampling lance, sampling tube (optional)
2.6. Flap gouge auger, peat sampler (optional)
2.7. Rake/pitchfork/hoe (optional)
2.8. Sampling beaker/cone/probe (optional)
2.9. Wheel loader, bulldozer, excavator with shovel*
2.10. Saw, auger, e.g. wood auger* and/or saw* (different types/options depending on material type)
2.11. Scissors/knife*
2.12. Any supplies needed for automated sampling of conveyor belts

[1] VLAREMA art. 1.2.1 §2 35° defines raw materials as by-products or materials that have reached the end of the waste phase as per Articles 36, 37 or 39 of the Materials Decree

2.13. Any supplies needed for manual sampling of a (stationary) conveyor belt
2.14. Supplies needed to take a spot sample for volatile parameter analysis (e.g. stainless steel sampling set with sampling canisters with a diameter of 35 mm and caps with stainless steel inserts, small auger set with 28 stainless steel auger rings and caps)*
2.15. 'Suspect asbestos-containing' and 'asbestos-containing' warning stickers

It is also permitted to use other equivalent equipment.
* basic equipment[2]

3 TECHNIQUES

3.1 PRECAUTIONS FOR THE PRESENCE OF ASBESTOS-CONTAINING AND SUSPECTED ASBESTOS-CONTAINING MATERIALS

During sampling, in cases of an **undeniable suspicion of the presence of asbestos-containing and/or suspected asbestos-containing materials[3] in the field sample or in the batch in question** (e.g. by means of visual detection in this sample and/or special asbestos inspections as per CMA/1/A.19 and/or CMA/1/A.21, etc.), **also label the sample(s) intended for chemical parameter analysis with an asbestos warning sticker[4].** In this way, the analytical laboratory can perform an alternative sample pre-treatment procedure in the chemical analyses.

If friable asbestos-containing or suspected asbestos-containing materials (e.g. residues of asbestos-containing insulation materials, asbestos rope, separate (fine) fibres) are also detected, first contact both the client and the relevant analysis laboratory to discuss the need to examine the chemical analyses, to enable additional precautions during sample analysis.

3.2 SAMPLING OF STATIC BATCHES

3.2.1 VA 1A: STOCKPILE SAMPLING IN PARTIAL BATCHES WITH BULLDOZER/WHEEL LOADER

The use of a wheel loader, excavator or bulldozer with loading shovel makes it possible to separate from large bulk batches or spread materials in a specific application (e.g. a (sub)foundation or hardening layer, a line track with stone slag or ash ballast at train tracks) , **smaller sub-batches**.

These partial batches are far easier to access and sample than the large batch, especially with manual sampling techniques.

[2]Ensure that basic equipment is available to every authorised sampling officer, for use according to the planned sampling.

[3] Annex A to CMA/1/A.19 provides guidelines on the visual properties of suspect asbestos-containing building materials. In practice, this inspection is conducted on fibrous materials. Asbestos recognition training is required at least to sample for packages MA.3, MA.7.1 and MA7.2.

[4] Always apply an asbestos warning sticker on materials with an increased or real risk of contamination with asbestos-containing and/or suspect asbestos-containing materials, such as recycled granulates of concrete and/or masonry and/or mixed rubble.

One condition here is that the wheel loader must take **material from both the surface and the core of the batch**.

The number of sub-batches to be taken from one batch (max. 1000 m^3) depends on the volume of the batch. The table below lists the (minimum) number of partial batches by batch size.

Batch size	Minimum number of partial batches
< 500 m^3	4
500 – 750 m^3	6
750 – 1000 m^3	8

The sampling officer is always free to increase this number.

The bulldozer approach is always preferred because it balances representativeness and practicality (labour intensity).
To avoid dust formation, this method is not permitted for dry and fine-grained materials (< 4 mm) or powders (< 1 mm). In this case, sample by boring through the batch (see VA2, §0).

For compost sampling, please refer to the **derived technique, VA1b** (§ 0).

Procedure:

1. Request assistance from a wheel loader with driver and give the driver clear instructions on the operations to be performed.
2. Use a wheel loader with shovel to take one or more shovel loads from the defined batch at **four (for batches < 500 m^3), six (for batches of 500 to 750 m^3) or eight (for batches of 750 to 1000 m^3) different locations**. The material from one location constitutes one partial batch.

The size of a partial batch depends on the size of the shovel used, but must be at least 1 m^3. If using a smaller excavator (shovel < 1 m^3), take multiple shovel loads for each partial batch (so each partial batch is at least 1 m^3).

Ensure that the partial batch sampling locations are distributed evenly across (the accessible part of) the batch, and take the same number of shovel loads from the bulk (core of the batch) and from the surface of the heap. Indicate locations potentially inaccessible for this sampling on a drawing or photo.

Note 1:
The sampling officer is always free to increase the number of locations or the number of shovel loads per location. Always indicate the reasons for any reduction in the number on the sampling form.

Note 2:
If the ratio of batch volume to shovel capacity is less than 10/1 (i.e. the loader can transport the full batch in no more than 10 shovel loads), it is permitted to sample by taking manual grabs along the surface (see 3.2.6). Record this as a deviation, indicating the reason: 'batch/shovel load ratio < 10'.

Note 3:
The taking of the (4,6,8) wheel bearing trays may also be staggered during the construction or movement of a stock pile (e.g. in a bunker, storage area, etc.), allowing them to be spread proportionally over the volume of the (moved) batch.

Note 4:
alternatively, for materials in a spread application or layer (e.g. a (sub)foundation or hardening layer, a line path with stoney slag or ash ballast in train tracks), a through holes/slots can be used as an alternative cf. CMA/1/A.20 §5.2.4 b) uitgevoerd worden met behulp van een sleuvengraver of graafmachine. The excavated material is further treated as in point 3 forf this technique (VA1b).

3. Have the material of one partial batch dumped onto a 'clean' subsurface, to prevent mixing and contamination of the sampled material with the subsoil.
4. Next, use the shovel to spread the partial batch into a thin layer of up to 40 cm.
5. Using the manual method (3.2.6), take **4 grabs from the partial batch**. Calculate the theoretical amount of field sample using the number of sub-batches, the number of handles per sub-batch (min. 4) and the contents of the chosen equipment (scoop). Compare this calculated quantity to the minimum field sample requirements. Increase the number of grabs per partial batch, or choose a larger scoop if necessary to meet the minimum field sample requirements.
6. Distribute the grabs as evenly as possible over the surface of the partial batch (in a systematic sampling patter, see Figure 1) and wherever possible, take them through (half) the thickness of the partial batch (at least 20 cm). Depending on the material being sampled (particle size), take the grabs with equipment that meets the requirements of CMA/1/A.14. Grabs shall not be taken from the sides of the sub-batches.

Note 5:

In this case, it is not necessary to scoop off the outer layer (0 to 50 cm) as per § 3.2.6(5) because the wheel loader has already homogenised the partial batch.

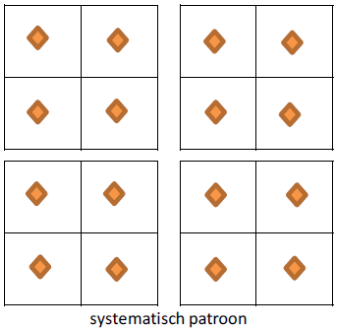


Figure 1. Systematic sampling pattern for four partial batches

7. Repeat points 3 to. 7voor for the other partial batches from the stockpile (min. 4). Depending on the number of partial batches, this technique calls for at least 16 or more grabs from the whole batch.
8. Collect and combine the grabs to create the field sample.
9. If applicable, conduct separate sampling for volatile parameters as per § 0.

3.2.2 VA 1B: SAMPLING COMPOST BY WEDGE-SHAPED INCISION WITH BULLDOZER/WHEEL LOADER

It is preferable to use a wheel loader/bulldozer for compost sampling as well. It is not the partial batch that is sampled here, but rather the steep wall in the original batch after removal of one (or more) shovel load(s). Use the (estimated) batch volume to determine the number of incisions in batch (see table under 3.2.1).

Note 6: If the walls of the wedge are unstable, transfer to the method via sub-batches with wheel loader (VA1a, see 3.2.1). First, the wheel loader removes the outer layer, through the fungus layer, in places where it shall take a shovel load from the batch. A shovel with material from a wedge/incision, away from the stock pile, is then placed on a clean substrate. This shovel load is further sampled as a partial batch (see 3.2.1). Indicate the reasons for the deviating sampling technique ('wedge/incision wall stability not guaranteed').

Method

1. The wheel loader with shovel makes a wedge-shaped incision in the batch at 4 **(for batches < 500 m³), 6 (for batches of 500 to 750 m³) or 8 (for batches of 750 to 1000 m³)** different places. Distribute the incision locations evenly in the batch.

Indicate locations potentially inaccessible for this sampling on a drawing or photo.

2. Using the manual method (§ 3.2.6), take four grabs from side of the incision. In this process, be sure to avoid the (outer) fungus layer of the batch (50 cm). If necessary, use a small hand fork or rake to break up the material from the (compacted) side walls of the incision.

Distribute the grabs as evenly as possible over the free (accessible) side of the wedge-shaped incision.

3. Repeat point 2 for the other wedge-shaped incisions from the stockpile (at least 4). Depending on the number of partial batches, take at least 16 or more grabs.
4. Collect and combine the grabs to create the field sample. Compare this quantity to the minimum requirements for the field sample. Bear in mind that a field sample of compost must be at least 20 litres (min. 12 kg). If necessary, the number of handles should be increased if the minimum requirements are not met; in this case, the same number of handles should be taken from each indentation.

3.2.3 VA 2: SAMPLING BY BORING THROUGH STOCKPILE[5]

Method

1. Sampling equipment of the type (soil) auger, gouge auger, stab tube, stab lance is used to take **2 borings** through the stock pile. The sampling officer may opt for horizontal, vertical or a combination of horizontal/vertical boreholes. Horizontal boreholes must pass through the **centre of the stockpile**. Preferably, they are perpendicular to each other. The borehole height is between approx. 30% and 70% of the batch height. Always be certain not to sample the bottom 30 cm of the stockpile (segregation affect too great). Make vertical boreholes downwards, **perpendicular to the ground**. At least one of the two boreholes must pass through the highest point of the batch.

A combination of horizontal and vertical boreholes is permitted. These combinations must meet the above conditions and the boreholes must intersect in (roughly) the centre of the batch.

Note 7:
Vertical boreholes involve stepping/climbing on a batch, something that is not always possible for safety reasons, such as with unstable stockpiles with round stones that may roll off, soft solids (e.g. shredder fluff) or pastes (e.g. sewage sludge, etc.) into which the sampling officer may sink, sharp materials or parts that may cause injury (e.g. wood waste, certain shredder residues, certain bottom ashes), etc.
Some examples of materials that allow for vertical drilling would be: stable piles of sand-like or soil-like waste (decontaminated sweepings, certain sands made from screened construction and demolition waste, etc.)

2. Push or screw the auger or tube into the material in the selected direction. Once the device is completely full, remove it from the batch and empty it ('grab').

Repeat this operation (if necessary by attaching extensions) until the borehole goes all the way through the batch (horizontal) or reaches the ground (vertical). If it is not possible to reach the full depth or diameter, the borehole must at least reach a length equal to the sum of length, width and height of the batch, but distributed over multiple, evenly spaced shallower boreholes of at least 100 cm. These boreholes may also be diagonal, instead of vertical and/or horizontal. If it is not possible to reach the centre of the batch or 50% of its depth, indicate this as a deviation.

3. Repeat point 2 for the second borehole.
4. Combine the grabs of the two boreholes to make the field sample. Compare this quantity to the minimum requirements for the field sample. Make additional borehole(s) (repeat point 2) if necessary to meet the minimum requirements.
5. If applicable, conduct separate sampling for volatile parameters as per § 0.

[5] This method is not suitable for materials that were used in limited layer thickness in a specific application (e.g. a (sub)foundation or hardening layer, a line path with rock impact or ash ballast for train tracks) because this would result in a too limited (i.e. 2) number of drill bits with limited depth so that sampling is insufficiently representative of the sampled layer.

3.2.4 VA2B: ALTERNATIVE DRILLING TECHNIQUE FOR STOCKPILES

It is preferable to use this technique with stockpiles with a large surface area and limited stacking height. The technique is also suitable for the sampling of materials in a spread application or layer (e.g. a (sub)foundation or hardening layer, a line path with rock impact or ash ballast on train tracks).

The advantage of this technique over (only) vertical boreholes as per. 0, that the spatial distribution along the length of the stockpile or of a batch spread in limited layer thickness is taken into account.

This technique may also offer a solution for stockpiles that are not safely accessible for vertical drilling.

Alternative method

1. Choose an auger that is suitable for boring (diagonally) downwards or diagonally through the waste to be sampled, e.g. soil auger (various types: Edelman augers of different sizes, gouge auger, Riverside auger, stony soil auger, etc.). Use any necessary extension(s).

2. Calculate the total length of the boreholes: 1.5 x the longest side (length, width or height) of the batch.

Divide this boring length across multiple sampling points (boreholes) distributed along the surface of the batch and decreasing in depth.

The minimum borehole length is 1 metre, but if possible, drill from human height (approx. 1.5 m or higher, up to 2.5 m if it is safe to step onto the base of the batch) to the ground. Calculate the number of sampling points (= total drilling length / length of borehole); if necessary, carry out a test drilling to test out the possibilities.

Note 8:
The number of drillings is determined for materials that were spread in limited layer thickness in a specific application (e.g. a (sub-) foundation or hardening layer, a line path with crushing or shaft ballast in the case of train injection) not on the basis of the longest side of the application, but on the basis of e.g. the volume of the material in the application (area x thickness) according to § 3.2.6 (VA4) points 1 and 2. The depth of the drilling shall be documented and preferably equal to the entire layer thickness of the application.

3. Push or screw the auger into the waste batch. Once the device is completely full, remove it from the batch and empty it ('subsample').

If necessary, repeat this operation in the same borehole (sampling point) until reaching a drilling length of at least 1 metre (but preferably until reaching the ground). Use extensions to reach the full depth or diameter of the batch. These boreholes may also be diagonal, instead of vertical and/or horizontal.

4. Repeat point 3 at the other sampling points. Ensure even distribution of the sampling points around the circumference of the batch (see also Figure 2).

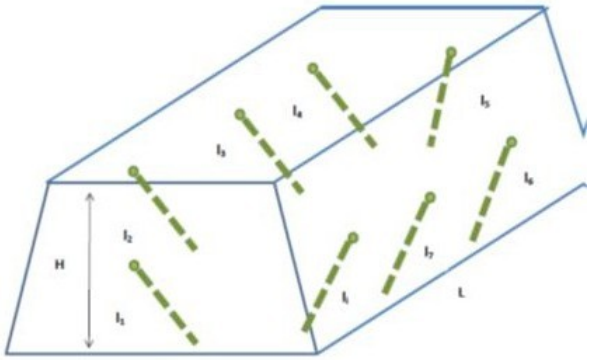


Figure 2: Example of distribution of shallow borings along the perimeter of the batch:
$l_1+l_2+l_3+l_4+l_5+l_6+l_7+l_i \geq 1.5$ x longest side (L or B or H)

5. Combine the subsamples from the drilling length of the (shallow) boreholes to make the field sample.
6. Combine the grabs of the two boreholes to make the field sample. Compare this quantity to the minimum requirements for the field sample. Make additional boreholes (repeat point 3) if necessary to meet the minimum requirements.
7. If applicable, conduct separate sampling for volatile parameters as per § 3.4.

If it is not possible to reach the centre or 50% of the diameter of the batch (for horizontal drilling), or the ground (for vertical or diagonal downward drilling), indicate this as a deviation.

3.2.5 VA 3: Sampling (of container loads) by drilling through the batch vertically[5]

Method

1. Make vertical boreholes down to the bottom of the container at two locations distributed over the surface area. For this, use sampling equipment suitable for downward boring and selected for the material being sampled (particle size, type). Some examples here would be: type of soil auger, peat sampling officer (soft pastes), flap gouge auger (powders and fine-grained solids), silo auger, etc.

Note 7:
Containers, trucks, etc., are often only accessible from the top. It is preferable to sample these batches after they are unloaded, so they are spread out (on an inert subsurface) and accessible. This is strongly recommended, especially in cases of doubts regarding batch homogeneity (container, truck, etc.). After unloading, sample the batch as a stockpile.

Note 8:
It is not necessary to walk or stand on the load itself.

2. Push or screw the drill or tube into the material vertically. Once the device is completely full, pull it out of the batch and empty it.
3. Repeat this operation (if necessary using extensions) until reaching the bottom of the batch. If less than ¾ of the total height can be drilled, several shallower boreholes (min. 100 cm) with a total length that is at least the depth of two full boreholes.

Note 9:
If drilling cannot reach ½ of the total height, revert to the method of taking grabs from the surface (§ 3.2.6). In this case, the number of grabs is based on the batch size according to the requirements of the manual method, and must be at least 10. Always document this decision and the underlying reasons on the sampling form and sampling report.

4. Points 2 to. 3 are repeated at another sampling location, distributed evenly over the (accessible) surface area of the container.
5. The grabs of the 2 boreholes merged into a field sample. Compare this quantity to the minimum requirements for the field sample. Perform (an) additional drilling(s) (repeat point 2 t.e.m. 3) if the minimum requirements are not met.
6. If applicable, conduct separate sampling for volatile parameters as per § 3.4.

3.2.6 VA 4: SAMPLING BY MANUALLY TAKING GRABS ALONG THE SURFACE

This method is permitted only on condition that the material is inaccessible or not (fully) penetrated and sampling techniques VA 1 (Paragraphs 2.1 and 0), VA 2 (§ 0) or VA 3 (§ 3.2.5) are not applicable. Log and document the cause or difficulties on the sampling form.

Method:

1. Determine the size of the batch to be sampled, expressed in volume (m^3).
2. Take a number of grabs at different places in the batch (stockpile or spread out layer).

The minimum number of grabs (n) depends on the batch size (expressed as batch volume V_{batch} in m^3) and is calculated as follows:

$$n = 10 + \frac{1}{2} \times \left(\frac{V_{batch}}{20} \right)$$

At each location, take one grab of the agreed grab size (see CMA/1/A.14). Depending on the material being sampled (particle size), take the grabs with equipment (scoop, gouge auger, sampling tube, hand scoop, etc.) that meets the requirements of CMA/1/A.14.

Note 11:
The maximum batch size is 1000 m^3. This means the (minimum) number of grabs per sample is 35. More grabs are always permitted.

3. Calculate the theoretical field sample quantity based on the calculated number of grabs and the capacity of the selected sampling equipment. Increase the number of grabs, select higher-capacity sampling equipment or use a combination of these two measures, and recalculate the theoretical field sample quantity.
4. Distribute the number of sampling locations for the grabs evenly over the surface of the batch.
   In the case of a conical or pyramid shaped stock pile, where it is fully accessible, it shall be sampled in **3 layers** according to the following **proportions**: 6/10 grabs from the bottom layer, 3/10 grabs from the middle layer and 1/10 grabs from the top layer (see Figure 1).

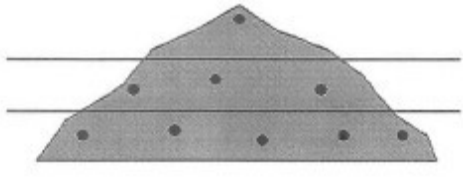


Figure 3. Distribution of sampling locations in a stockpile (side view)

If taking more than 10 grabs (depending on V_{batch}), distribute them in the same ratios as the above diagram.

In the case of a stock pile where the circumstances do not allow the upper part of the stock pile to be entered, the **number of grabs to be taken is spread over the accessible batch surface of the stock pile, usually between 0 and 150 cm in height**. In this case, indicate the accessible sampling height on the sampling form and report. This requires proper attention to the spatial distribution of the grabs. This must be uniform, both horizontally and vertically. It is preferable to use a zigzag pattern (see Figure 4).

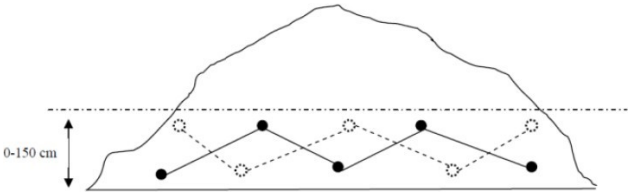


Figure 4. Zigzag distribution of sampling points in a stockpile (side view)

5. Never scoop grabs directly from the surface of the batch (outside). The material on the outside may differ from the bulk, due to the effects of moisture, sunlight and air, and is not typically representative of the batch. Before taking a grab, always scoop off the surface layer (approx. 25 cm) first, to access the underlying material. It is allowed to use non-permitted sampling tools to scoop off the outer layer (e.g. rake, flat shovel, etc.). If using a gouge auger, it is not necessary to scoop off the surface layer (minimum auger body length of. 60 cm). If using an Edelman auger, pre-drill at least once.

Note 12:
For compost, it is vital to avoid taking any material from the outer layer of 0 to 50 cm (fungus layer)!

6. Remove the excess material on top of the sampling equipment (e.g. wipe off with spatula) so all grabs are the same size.
7. Repeat point 5 for the other grabs from the stockpile.
8. Combine the grabs to make the field sample. Compare this quantity to the minimum requirements for the field sample. Take additional grabs (repeat point 5) if necessary to meet the minimum requirements.
9. If applicable, conduct separate sampling for volatile parameters as per § 3.4.

3.3 SAMPLING OF DYNAMIC MATERIAL FLOWS AND BATCHES

Representative sampling of material flows is only permitted if the sampling timeframe is representative of the sampled material.

3.3.1 VA 5: AUTOMATED SAMPLING OF A DEPOSIT FLOW

Some examples of a deposit flow would be a material flow at a transfer point (e.g. at the end of a conveyor belt) and a vertical material flow from a storage unit (outflow at the bottom of a supply silo, bunker).

Method:

1. Determine the size or volume of the batch to be sampled, expressed in volume (m^3).
2. Determine the time interval within which sampling must be performed (see also batch division in CMA/1/A.14)
3. Take a number of grabs from the deposit flow at different times. The minimum number of grabs (n) depends on the batch size (expressed as batch volume Vbatch in m^3) and is calculated as follows:

$$n = 5 + \frac{1}{4} x \left(\frac{V_{batch}}{20} \right)$$

Note 13:
The maximum batch size is 1000 m^3. This means the (minimum) number of grabs per sample is 18.

4. Calculate the theoretical field sample quantity based on the calculated number of grabs and the capacity of the selected sampling equipment. Increase the number of grabs, select higher-capacity sampling equipment or use a combination of these two measures, and recalculate the theoretical field sample quantity.

Deposit streams are typically sampled by means of valves or automatic material collection at the transfer point. The **set number of handles (min. 5)** is taken automatically and distributed evenly within the selected time interval. The grab sizes and dimensions of the valves or material collectors must also meet the requirements in CMA/1/A.14.

A material collector is a kind of receptacle that runs **back and forth through the material stream at a constant speed**. The path of the receptacle runs perpendicular to that of the material stream. It is vital to sample **the full width of the material stream** (i.e. not just along its edge). The receptacle dimensions must also be tailored to the material type (opening must be at least three times the size of the material units). Be sure that system removes the receptacle in the same direction as the stream inflow.

5. This system repeats this operation automatically at preset times.
6. Combine the grabs to make the field sample. Compare this quantity to the minimum requirements for the field sample. Take additional grabs (repeat point 5) if necessary to meet the minimum requirements.

Note 14:
A manual sampling of a deposit stream (tray manually moving through the stream) is not recommended as a sampling method. If automatic sampling equipment is not available at the transfer point, select a different sampling scenario (e.g. from the stockpile).

7. If applicable, conduct separate sampling for volatile parameters as per § 3.4.

3.3.2 VA 6: AUTOMATED SAMPLING OF A CONVEYOR BELT

Method:

1. Determine the size or scope of the batch to be sampled (belt load), expressed in volume (m^3).
2. Determine the time interval within which sampling must be performed (see also batch division in CMA/1/A.14)
3. Take a number of grabs from the conveyor belt at different times. Distribute these grabs evenly over the selected time interval. The number of grabs (n) depends on the batch size (expressed as batch volume V_{batch} in m^3) and is calculated as follows:

$$n = 5 + \frac{1}{4} x \left(\frac{V_{batch}}{20} \right)$$

Note 15:

The maximum batch size is 1000 m^3. This means the (minimum) number of grabs per sample is 18.

4. Calculate the theoretical field sample quantity based on the calculated number of grabs and the capacity of the selected sampling equipment. Increase the number of grabs, select higher-capacity sampling equipment or use a combination of these two measures, and recalculate the theoretical field sample quantity.
5. The sampling equipment consists of an automated (e.g. magnet-controlled) receptacle, scoop or material ejector, that scoops material from the moving belt in a motion perpendicular to the conveyor belt (= grab). The material collector moves over the conveyor belt at a constant speed. Ensure sampling over the full width of the conveyor belt. The capacity of the sampling equipment must be tailored to the material particle size (scoop opening) and the maximum belt load (edge height). Only use calibrated equipment.
6. The system automatically repeats the procedure at a preset number of times.
7. Combine the grabs to make the field sample. Compare this quantity to the minimum requirements for the field sample. Take additional grabs (repeat point 5) if necessary to meet the minimum requirements.

Note 16:
The advantage of this automated equipment is that it does not require production stoppage.

8. If applicable, conduct separate sampling for volatile parameters as per § 3.4.

3.3.3 VA 7: MANUAL SAMPLING OF A (STATIONARY) CONVEYOR BELT

Method:

1. Determine the size or scope of the batch to be sampled (belt load), expressed in volume (m^3).
2. Determine the time interval within which sampling must be performed (see also batch division in CMA/1/A.14)
3. Take a number of grabs from the conveyor belt at different times. Distribute these grabs evenly over the selected time interval. The number of grabs (n) depends on the batch size (expressed as batch volume V_{batch} in m^3) and is calculated as follows:

$$n = 5 + \frac{1}{4} x \left(\frac{V_{batch}}{20} \right)$$

Note 17:
The maximum batch size is 1000 m^3. This means the (minimum) number of grabs per sample is 18.

4. Calculate the theoretical field sample quantity based on the calculated number of grabs and the capacity of the selected sampling equipment. Increase the number of grabs, select higher-capacity sampling equipment or use a combination of these two measures, and recalculate the theoretical field sample quantity.
5. Stop the conveyor belt.
6. Take a scoop (with dimensions tailored to the material particle size).
7. Use the scoop to take the amount of material needed for one grab from a selected (fixed) sampling point. It is vital for the grab to take material from the entire width of the conveyor belt.
8. Restart the conveyor belt.
9. Repeat points 5 to 8 at the designated times to take the next grabs.
10. Combine the grabs to make the field sample. Compare this quantity to the minimum requirements for the field sample. Take additional grab(s) (repeat points 5 to 8) if necessary to meet the minimum requirements.

Note 18:
Another method involves placing what is known as a 'sampling frame' (Figure A.9) along the width of the belt. Moving the sampling frame back and forth over the belt (a few centimetres) makes a visible separation in the material being sampled. Ensure that material does not fall from the belt in this process. Next, remove all the material located in the frame. After sampling, remove the frame.

11. If applicable, conduct separate sampling for volatile parameters as per § 3.4.

3.3.4 VA 8: MANUAL SAMPLING OF A (STATIONARY) SCREW

Method:

1. Determine the size or volume of the batch to be sampled, expressed in volume (m^3).
2. Determine the time interval within which sampling must be performed (see also batch division in CMA/1/A.14)
3. Take a number of grabs from the screw at different times. Distribute these grabs evenly over the selected time interval. The number of grabs (n) depends on the batch size (expressed as batch volume V_{batch} in m^3) and is calculated as follows:

$$n = 5 + \frac{1}{4} x \left(\frac{V_{batch}}{20} \right)$$

Note 19: The maximum batch size is 1000 m^3. This means the (minimum) number of grabs per sample is 18.

4. Calculate the theoretical field sample quantity based on the calculated number of grabs and the capacity of the selected sampling equipment. Increase the number of grabs, select higher-capacity sampling equipment or use a combination of these two measures, and recalculate the theoretical field sample quantity.
5. Stop the screw.
6. Use a scoop or hand scoop to take the amount of material needed for the minimum grab size (CMA/1/A.14).
7. Restart the screw.
8. Repeat points 5 to 7 at the following times to take the next grabs.
9. Combine the grabs to make a field sample. Compare this quantity to the minimum requirements for the field sample. Take additional grabs (repeat point 3) if necessary to meet the minimum requirements.
10. If applicable, conduct separate sampling for volatile parameters as per § 3.4.

3.4

3.4 SOLID MATERIALS: SAMPLING WITH RESPECT TO VOLATILE PARAMETERS

To determine volatile parameters (VOCs), extensive sampling may result in significant losses, such as due to prolonged contact with the air when taking multiple grabs, disruptions during grab homogenisation and division into laboratory sample(s), volatilisation and diffusion due to unsuitable packaging, etc.
Therefore, for these parameters, it is recommended to conduct a **separate sampling operation**, consisting of a bore sample or manual sampling with a limited number of grabs and without sample pretreatment, to minimise contact with air. Thus, even for solids and pastes, this already constitutes a deviation from the principle of a composite sample made up of multiple grabs.

The market offers specialised sampling equipment for volatile parameters (e.g. soil sampling canister: see Annex A.11, small plunge borer set: see Annex A.12) or for cases of restrictions on use.
If specialised equipment is not available, separate manual (spot) sampling may also offer a solution.

The guidelines below apply to sampling for volatile parameters.

- So far as possible, use sampling equipment and containers made from 'inert' materials: stainless steel, glass, Teflon.
- Transfer the grab or spot sample directly to the sampling container.
- A spot sample for volatile parameters often consists of a single grab. Indicate this on the sampling form and sampling report. However, if it is possible to take multiple grabs, pour them (directly) into the sampling container in layers.

Note 20:
Preferably, the sample transfer form gives the instructions for taking a test portion in the laboratory by drilling through the whole sampling container.

- Do not perform any on-site mixing, homogenisation or division operations. If more than one laboratory sample is required, conduct a separate sampling operation for each laboratory sample.
- Completely fill the sampling container (zero headspace).

Note 21:
Some materials may cause gas development, e.g. sewage sludge; for such materials, the packaging may only be filled to ¾.

- Refrigerate samples and transport them for handover to the analysis laboratory as quickly as possible.

Method:

1. Select suitable sampling equipment to sample for volatile parameters, based on the waste types to be sampled: special plunge borer sets with stainless steel sampling canisters (D95 ≤ 20 mm) or rings (D95 ≤ 10 mm) or stainless steel or aluminium scoop in combination with glass container with Teflon insert in the lid (D95 > 21 mm).

2. Select a location for the spot sample: typically based on a worst-case scenario. Document the spot sample location. If preparing multiple laboratory samples (e.g. analysis and counter-analysis), take spot samples right next to each other.

3. Scoop or drill surface debris (min. 50 cm) with a hand scoop or an Edelman auger.

4. Slowly press the sampling canister, ring or glass container into the waste to be sampled, with a slight back and forth motion, until it is completely full. In cases of very firm solid waste, it may be helpful to use a mallet (with plastic head) to drive the plunge borer set with sampling canister or ring into the batch.

Once the sampler body is completely full, carefully remove the entire device. Use a knife or spatula to cut the bottom of the core to the length of the sampling canister, ring or glass container. Immediately seal the contents using the special sealing devices. Do not leave any free space between the sample and the sealing device or lid. If necessary, indicate top and bottom on the auger ring/sampling canister.

If the sampling equipment cannot be pressed directly into the batch of waste, a glass container (min. 250 ml) filled with a (small) stainless steel hand scoop. Scoop the waste directly into the glass sampling container. If necessary, close the sampling container between two scoops to minimise contact with air. Ensure that the glass container is completely full, use a spatula or knife to remove the excess material and do not leave any free space between the sample and lid (push material down if necessary). Before closing, wipe the threaded edges of the sampling container off with moistened absorbent paper, to ensure a proper seal.

5. Immediately refrigerate the laboratory sample for volatile parameters in the cool box.

3.5 SOLID MATERIALS: SHAPED MATERIALS OR MATERIALS WITH PIECE OR PARTICLE SIZE (D95) > 100 MM

This covers both materials shaped in a particular production process (e.g. shaped concrete or masonry products) and waste or materials that are a by-product of a particular production process (e.g. large (pieces of) slags in a metallurgical process). In both cases however, the dimension(s) of these materials are greater than 100 mm, in the majority of the different particles or material components.

Note 22:
Aside from fragments of up to 10 mm, a residual fraction of shredder residue with product designation '0 to 150 mm' also contains a great deal of fine material, and does not fall under this category.

Wherever possible, the sampling techniques in § 3 apply, with the difference that the grab size and/or sample quantity and equipment may vary.

One initial option is to select large material components (e.g. pieces of slag, boards, stones, beams, etc.) **individually and whole**. In this case, in principle, each grab consists of one complete material component. This option is available for both manual sampling (technique VA4, § 3.2.6) and sampling from conveyor belts.

The disadvantage is that this method may result in very large grabs and samples. Thus, in principle, proper homogenisation or distribution requires reducing the particle size (crushing) of the material. With these procedures, proper on-site implementation is no longer considered possible (lack of reduction equipment, such as crushers, grinders). Therefore, package the grabs (possibly each gab separately, if too large) and deliver them to the analysis laboratory with instructions for homogenisation and/or division on the sample transfer form.

For bulky material components (greater than 10 cm, such as beams, pallets and boards, as well as bulky lumps of paste), it sometimes makes more sense to take a **cross-section** (Figure 2-1) and/or **borehole** (Figure 2-2) from the material component (using an auger, saw, knife, etc.). The minimum volume of the cross-section is 500 ml (reference value).

Another option is to take **auger filings or scrape off pieces of material** with an auger, file, etc., (Figure 2-3). The defined minimum volume is 200 ml (reference value).

However, the guidelines below also apply.

- It is preferable to take a full perforation hole or cross-section directly through the component and perpendicular to the surface. If this does not appear possible, take the borehole or cross-section to half the depth. In the analysis, other ratios of boring/sawing depth to total diameter may result in overestimation or underestimation of contamination levels from coatings, paint, etc. (e.g. preservatives) with respect to the overall batch;
- Along the longitudinal axis, position the borehole or cross-section at half the length, or at least 30 cm from the end of the material component. To account for uneven features, maintain a margin of 10 cm for the borehole/cross-section;
- the minimum bore diameter is 5 mm. The minimum width of the cross-section shall be 50 mm.

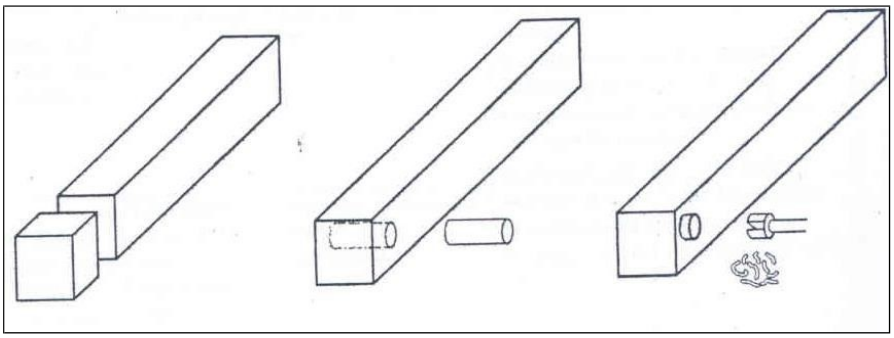


Figure 5. Cross-section (left), borehole (centre) and auger filing (right) from large material components

For shaped materials that are part of a production process, it is also possible to sample the **(fresh) mixture of the raw materials** instead of the shaped product itself (e.g. 'wet' concrete mixture instead of cured concrete). This process also uses the same techniques covered in the procedure and/or CMA/1/A.17. It is also necessary to complete sampling before the mixture cures.

ANNEX A
EXAMPLES OF EQUIPMENT AND TOOLS FOR SAMPLING SOLID WASTE

A.1 Wheel loader/shovel loader, excavator, etc.

A wheel loader or similar large rolling stock fitted with a shovel is highly useful for sampling large stockpiles of bulk material (e.g. excavated soil, rubble aggregate, bottom ash, etc.). Use of these kinds of vehicles is the preferred method for sampling static stockpiles > 20m^3. The shovel is used to remove at least four partial batches, each at least 1 m^3 in size, from the batch being sampled. Further sample these partial batches manually as static heaps.

The vehicle may also use the shovel to take grabs from the batch being sampled (see also. VA 4 'manual method', but on a larger scale). However, due to the typically large the shovel capacity (1 to 5 m^3), on-site sample pretreatment to make a laboratory sample from a field sample would be too time-consuming.
Materials containing very large components (e.g. > 300 mm) may nevertheless require grabs of this size.

A.2 Scoop

The scoop is the simplest and most universal sampling tool. Scoops come in various sizes and designs, depending on the type and appearance of the material being sampled. Plastic laboratory scoops (e.g. PP) are typically used, due to their corrosion resistance and low price. For materials and waste containing organic solvents, etc., stainless steel is preferred.

A sampling scoop has raised edges to prevent material spilling over its sides. A regular shovel or spade is not suitable for sampling granular materials. Some of the material on top shall spill out of the shovel, rendering the grab unrepresentative of the batch.

Scoops can be used to sample coarse-grained materials and waste (> 10 mm), such as rubble aggregate, wood chips, bottom ash, metallurgical slag, etc.

The disadvantage of a scoop is its limited sampling depth. With low-cohesion granular materials, penetration is limited to surface material. Large and wide scoops in particular have difficulty penetrating heaps of coarse-grained materials (e.g. rubble aggregate, wood chips, etc.).

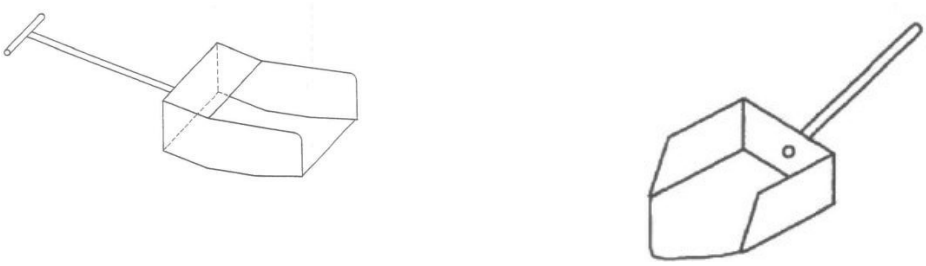
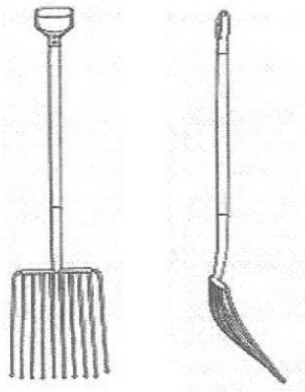


Figure 6. Sampling scoop for granular materials (left) and soft fibrous materials ('fluff') (right)

A.3 Rake/pitchfork

A rake or pitchfork may be used to excavate sampling locations below the surface of piles of (compacted) fibrous materials (manure, plant waste, compost). As pitchforks could possibly lose small material components through their tines, exercise due care during actual sampling (taking grabs). Preferably, the spacing between rake or pitchfork tines should be less than or equal to the shortest fibres in the batch. A rake/pitchfork is in fact excellently suited for dislodging or digging out deeper sampling locations (e.g. digging out the fungus layer on compost).



A.4 Soil boring, gouge auger

A gouge auger consists of a samples cylindrical tube that is almost split in half lengthwise (60/40). On the bottom, it features a sharp cutting edge to facilitate penetration. Depending on the material type and structure (particle size), they come in diameters of 20 to 60 mm and in different lengths.

Gouge augers are mainly used to take (minimally) disturbed samples in moderately cohesive soils and ground. They are however also highly useful for less cohesive materials, such as granular waste and powders (e.g. screened sand, fine ash). In this case, do not use the gouge vertically (as for boring in soil), but rather horizontally (see also use of a scoop to sample stockpiles). Hold the gouge perpendicular to the material surface of the stockpile. Concurrently push and screw the gouge to force it into the material. If necessary, turn the gouge one rotation on its longitudinal axis to dislodge the material column. Next, carefully pull the gouge out of the material (with the opening facing up!).

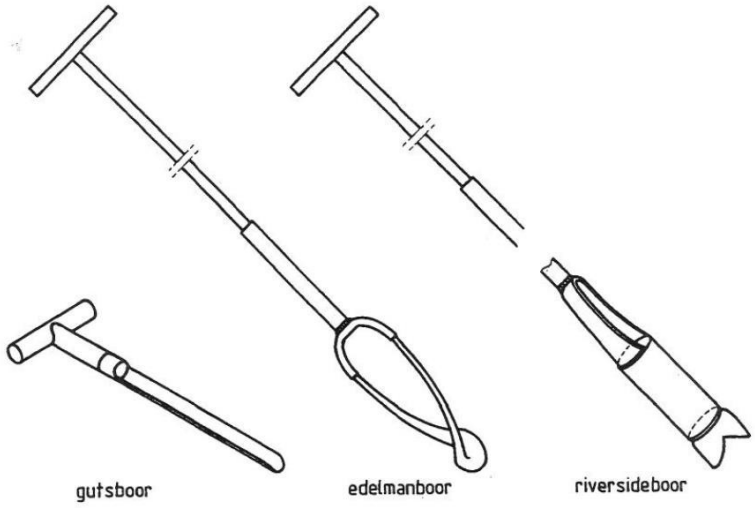
It is possible to attach extensions to the gouge auger to bore holes in stages through fine-grained material, up to depths of 5 to 10 m.

The main advantage of the gouge auger (just as with the plunge borer/sampling lance) over the scoop is its greater penetration depth.

In principle, it is not permitted to use the gouge to sample materials and waste in containers or trucks (these require vertical sampling).

The Edelman auger is a soil auger that is suitable for sampling low-cohesion substances (sandy and clayey materials).

The Riverside auger is used to drill into layers of hard, rigid soil and to sample granular or gravely material.



gust boor
edelman boor
riverside boor

gouge auger
Edelman auger
Riverside auger

A.5 Plunge borer, sampling lance, sampling tube

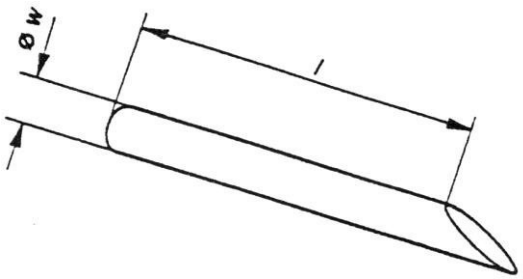
These terms cover numerous sampling devices with the common feature that the working part of the auger consists of a hollow cylindrical or concentric tube with a sharp opening at the front to facilitate penetration. They may also have a sampling container or bag a mounted on the back, to collect the material.

Some types consist of two (concentric) hollow tubes that can be opened, with the inner tube featuring perforations.

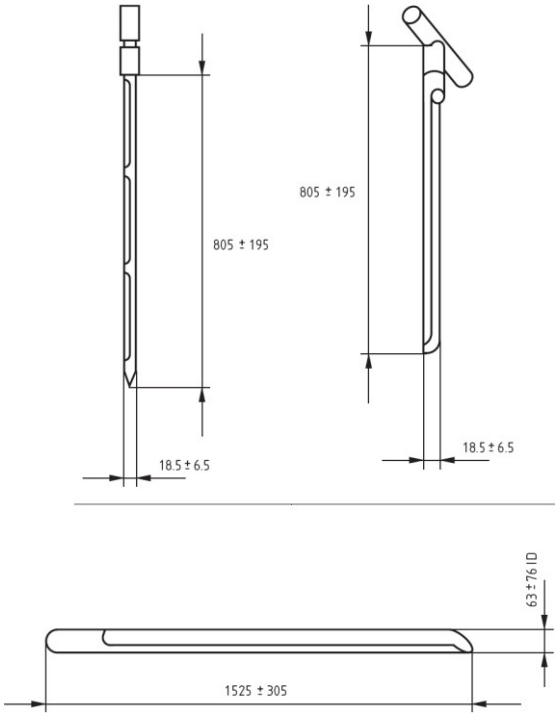
Push the plunge borer or sampling tube/lance into the material at an angle between 30° and 75°. If applicable, turn the outer tube to open the opening or perforations. Giving the tube a shake can help fill it. Close the tube (if applicable), and carefully pull it out of the material.

A plunge borer is excellently suited for sampling dry granular materials and powders. This type is mainly used in the food and pharmaceutical sectors to sample materials in bags, including big bags. These samplers come in different diameters (10 to 50 mm) and lengths (20 to 120 cm), depending on the material and the type of testing. It is easy to ‘customise’ a plunge borer/sampling lance to the material being sampled using a plastic tube cut at an angle (e.g. sewer pipe).

Especially for lighter materials with larger particle sizes (such as wood chips), this is an easy and budget-friendly solution.



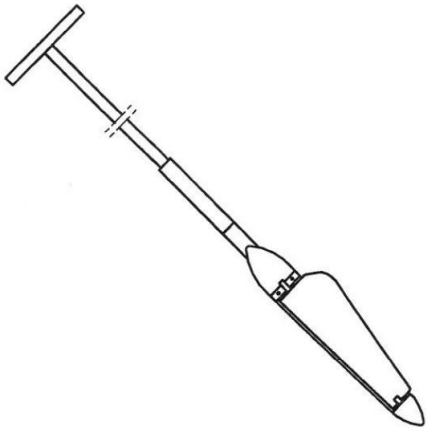
One advantage of the plunge borer/sampling lance over a scoop is greater penetration depth (as with the gouge auger).

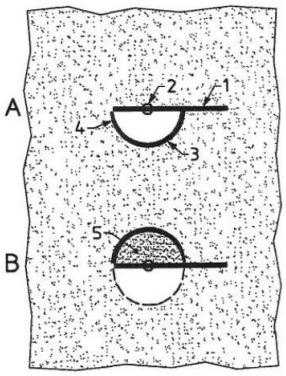


A.6 Flap gouge auger, peat sampler

It is possible to use a flap gauge auger (50 ml capacity) or peat sampling officer (500 ml capacity) to take non-cohesive material in bulk (powder, granulate, etc.), both dry and wet. The slim, robust design facilitates penetration. These devices can also be used to take samples at specific depths.

They are suitable for horizontal and vertical sampling of small granular materials, powders or dusts (up to 3 mm for the flap gouge) and pastes, including at greater depths (e.g. in trucks, barrels, big bags).

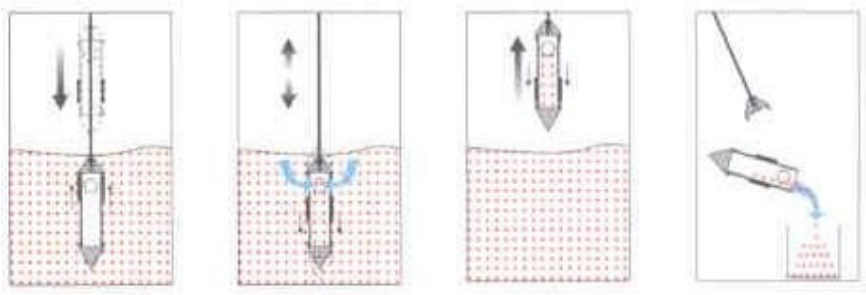




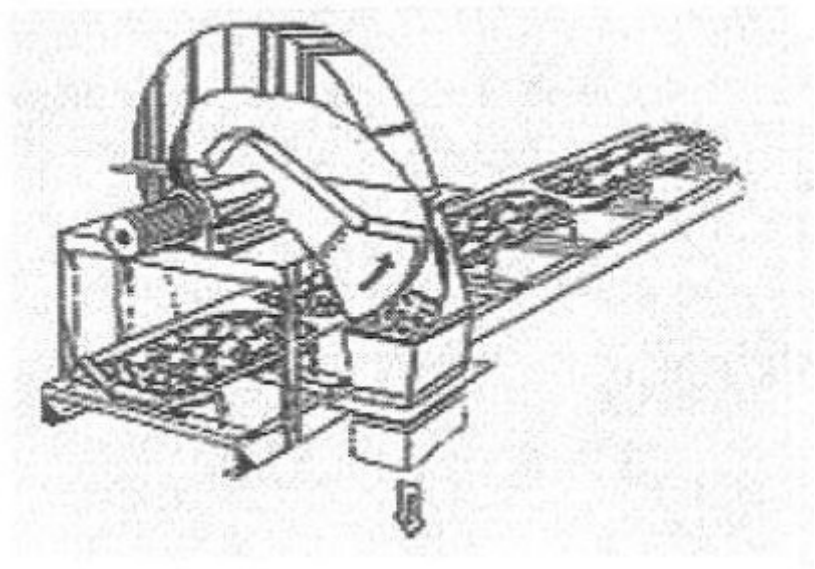
A.7 Sampling probe, silo auger

The ‘sampling probe’ consists of a (more or less) conical or cylindrical beaker (probe) with a point, with a metal bar attached at top. The metal rod can be used to push (or screw and push) the probe into the material.

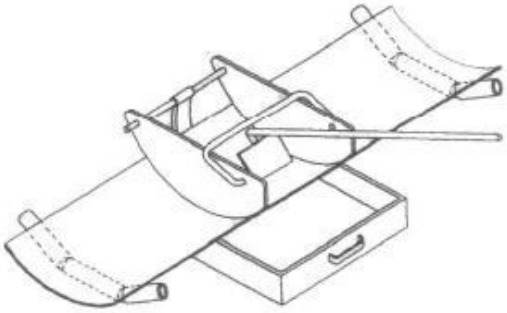
These devices are highly suited for vertical sampling of fine granular materials, powders or dusts, including at greater depths (e.g. in trucks). In some models, the cone (or probe) can close. They also come in different beaker capacities (5 to 250 ml).



A.8 Automated sampling of a conveyor belt



A.9 Manual sampling of a (stationary) conveyor belt



A.10 Saw, auger, etc.

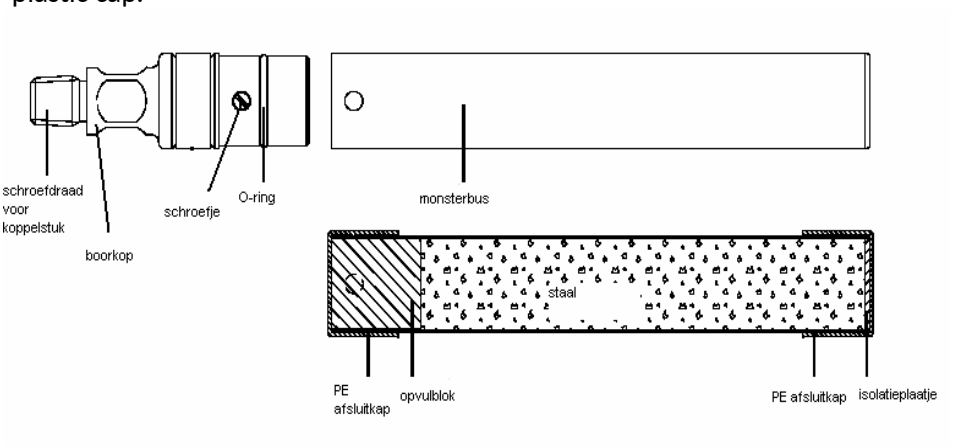
Figure 7: (hand) saw

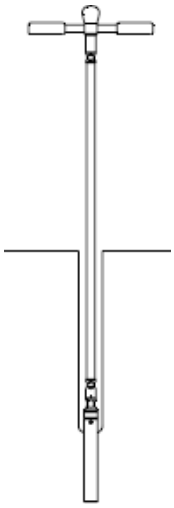
Figure 8: forstner drill (wood)

A.11 Sampling canister to determine volatile parameters in materials with particle sizes up to approx. 20 mm.

The sampling canister is a small plunge borer (35 mm in diameter) in which the (soil) sample does not come into contact with plastics. It minimises contact with the air (zero headspace) and greatly limits potential diffusion. This makes the device particularly suited for both sampling and transporting cohesive soil-like and sand-like samples (possibly also semisolid and/or dewatered sludges) for volatile parameter testing. Non-cohesive materials or dry powders are not cohesive enough for (vertical) sampling with this plunge borer.

The plunge borer (set) consists of a stainless steel hollow sampling canister to which extensions with grabs are mounted. After sampling, the same canister can be used to transport the sample. For this, it comes with suitable stainless steel isolation plates to place between the sample and the plastic cap.

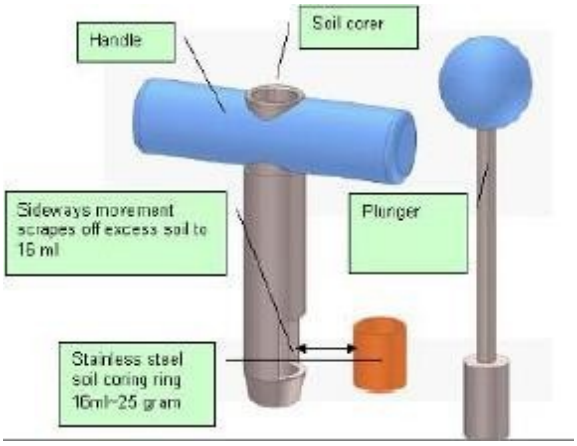




schroefdraad voor koppelstuk	threading for connector
boorkop	auger head
schroefje	screw
O-ring	O-ring
monsterbus	sampling canister
PE Afsluitkap	PE cap
opvulblok	filling block
isolatieplaatje	isolation plate

- Gently moves the grip back and forth to slowly push the sampling canister into the material being sampled until the entire canister is in the material. This method enables surface samples of approx. 30 cm. To sample deeper, it is permitted to first use an Edelman auger to drill out the material to the desired depth. In cases of very firm solid material, it may be helpful to use a mallet (with plastic head) to drive the sampling canister into the soil.
- Once the sampling canister is completely full, carefully remove the whole unit (by twisting it back and forth while pulling it upwards).
- Use a knife or spatula to cut the bottom of the core to the length of the sampling canister. Place a stainless steel isolation plate on this, and seal the bottom airtight with a polyethylene cap. Next, remove the sampling canister from the core sampling officer with extension. Fill the open space at the top of the canister with a stainless steel filling block. Also seal the top of the sampling canister with a polyethylene cap. The stainless steel isolation plate and filling block prevent contact between the sample and the plastic cap.
- If taking multiple laboratory samples, use the same procedure to take one or more new, separate sample(s) (as close as possible to the previous sampling location). Each grab from sampling canister is itself a laboratory sample, and is therefore packaged separately.

A.12 (small) Core sampling officer to determine volatile parameters in materials with particle sizes up to 10 mm



ANNEX B
POTENTIAL APPLICATIONS OF SAMPLING EQUIPMENT

B.1 Static batches (stockpile)

		Wheel loader/shovel loader, excavator, etc.	(Sampling) scoop, hand scoop (raised edge)	Pitchfork/rake[6]	Gouge auger	Soil augers: Edelman, Riverside types	Plunge borer, sampling lance/tube	Flap gouge auger, peat sampler	Sampling probe, silo auger
2.1	VA 1a: sampling by partial batch with bulldozer/wheel loader + manual grabs	++	++	+	+/-	+/-	-	-	-
0	VA 1b: COMPOST: Wedge-shaped incision with bulldozer/wheel loader	++	++	++	-	+/-	-	-	-
0	VA 2: Sampling by boring through batch (horizontal or vertical)	N/A	-	-	+/-	+/-	+/-	+/-	-
3.2.5	VA 3: Sampling of container (cargo) by drilling through vertically	N/A	-	-	+/-	+/-	+/-	+/-	+
3.2.6	VA 4: Sampling by manually taking grabs along the surface	N/A	++	-	++	+/-	+	-	-

++ highly suitable
+ suitable
- not suitable
N/A not applicable

6 Preferably, the spacing between rake or pitchfork tines should be less than or equal to the shortest fibres in the batch.

B.2 Material streams

See §**Error! Reference source not found.**

Sampling techniques for liquids

CONTENTS

1 INTRODUCTION

This method replaces the procedure CMA/1/A.16 of May 2023.
The sampling of liquid and semi-liquid waste and materials[1] poses a special challenge to collecting a representative sample. The term 'liquid waste' covers an array of waste types, often stored in a wide variety of ways. These are typically not pure chemicals, but rather suspensions or liquid mixtures of varying densities, viscosities and compositions.

This method describes sampling scenarios for liquids in:
- small storage units up to 20 litres (e.g. bottles, jerry cans);
- barrels, drums and small tanks up to 2 m high;
- vertical and horizontal cylindrical tanks over 2 m high.

For sampling of liquid materials and liquids in reservoirs, lagoons, etc., please refer to CMA/1/A.17.
This procedure does not expressly include sampling from pipelines, large storage units such as land tanks and (ocean-going) ships (with capacities exceeding 20 m^3) or non-cylindrical tanks (oval, etc.). The samples techniques detailed below can however be used for these applications.

Sampling technique selection must be based on the guidelines and decision trees in CMA/1/A.14. The latter also gives further instructions on batch division, grab and sample size, guidelines for analysis of specific parameters, packaging, transport and logging.

The procedure also provides for a visual inspection of the liquid to be sampled. This is intended to identify different phases in the batch (solid-liquid, liquid-liquid) and the medium (aqueous, non-aqueous). The sampling techniques do not distinguish between different mediums (e.g. oil, water); but they do in terms of sample pretreatment.
If sampling a non-aqueous medium or aqueous heterogeneous or layered liquids, follow the on-site sample pretreatment instructions as per CMA/1/A.18.
If sampling an aqueous, homogeneous, non-layered medium, on-site sample pretreatment and preservation must meet the guidelines (sample volume, preservative) in the WAC (Water Analysis Compendium).

[1] VLAREMA, Article 1.2.1 § 2.35° defines raw materials as by-products or materials that have reached the end of the waste phase as per Articles 36, 37 or 39 of the Materials Decree

2 EQUIPMENT - SUPPLIES

Below is a list of some commonly used sampling equipment and tools. Annex A provides further details, including on their operation. Annex B lists the possible applications for specific sampling scenarios.

2.1 Plunging siphon (see annexA.1)*
2.2 Liquid layer sampler * (rod- and/or cable-operated, see Annex A.2)
2.3 Ball valve sampler (see Annex A.3, optional)
2.4 Multisampler (see Annex A.4, optional, or as an alternative to 2.1)
2.5 Sampling bottle or jug* (see Annex A.5)
2.6 Shell stick with cup* (see Annex A.9)
2.7 Vacuum pump, hose pump or peristaltic pump (see Annex A.6 , A.7)
2.8 Side tube type sampling device or "side tube device" (see Annex A.8, example and use: see BAM/Part 3/01 "Liquid manure - sampling")
2.9 Measuring tape or ruler*
2.10 Dipstick or level sensor*
2.11 Stirring rod or dipper*
2.12 Non-sparking drum spanner*
2.13 Water finding paste or test strips for water detection*
2.14 (Step)ladder or sampling platform (if necessary)

It is also permitted to use other equivalent equipment.
* basic equipment[2]

3 PREPARATION FOR SAMPLING

The section below offers tips/guidelines to ensure proper sampling of liquid waste.

- Barrels, drums, etc., are used for a wide array of (chemical) products, which may pose various health and/or safety hazards, such as contact with heated, toxic or aggressive substances, explosions, pressurised packaging, etc. Attempt to identify the substance in advance, possibly based on the labelling.
- The contents of unlabelled and/or unknown barrels or tanks are always treated as hazardous waste. Barrels are often reused (e.g. to store liquid waste). For this reason, even labelled barrels should be treated with due caution. Always assume that the contents a barrel may differ from that indicated on the label!
- If possible, set the container being sampled upright, so the opening with cap is on top. Let the contents settle for 20 to 30 minutes. It is sometimes possible to sample a cylindrical barrel that is lying on its side in that position. In this case, be sure to brace the sides with chocks.

[2] Ensure that basic equipment is available to every authorised sampling officer, for use according to the planned sampling.

- Treat rusty or dented barrels with extreme caution. They may break easily if moved (e.g. set upright).
- Be mindful of static electricity (especially when unloading tanks/tankers). Special cable connections are often available to earth vehicles at loading or unloading points. It is possible to earth metal barrels by wrapping an iron belt or wire around them and letting one end hang on the ground. Always open barrels with a special non-sparking drum spanner (typically made of copper).
- In addition, always open and sample barrels and tanks upwind, to minimise contact with any vapours. Always open barrels, drums (unscrew cap) or tanks (tank cover) slowly, for gradual release of any pressure/vacuum. Be careful of any pressurisation of the tank cover, or of the tank cover falling opening by its hinges.
- Ensure that all supplies are cleaned in advance and that they are ready to hand. Clean the environment around the sampling opening, drain line or tap before the start of sampling.

4 TECHNIQUES

Sampling of liquid materials always starts with a visual inspection, identification of the liquid (aqueous/non-aqueous) and determination of the liquid level.

4.1 VISUAL INSPECTION

Conduct a thorough visual inspection to describe the batch being sampled and determine the sampling procedure or preservation method to apply in the situation at hand (homogeneous, heterogeneous or layered liquid).

First check the contents for the presence of a **surface layer**.

- Record the thickness and type of the surface layer on the sampling form. Also add this information to the sampling report.
- Carefully remove the surface layer before continuing with the visual inspection.
- If desired, it is permitted to analyse the surface layer separately.

Next, check the storage unit contents for the presence of **a bottom layer**. This refers to (solid) sediment on the bottom of the storage unit.

- Determine the thickness of the bottom layer and if possible, its nature. Record these on the sampling form. Also add this information to the sampling report.
- Do not include solid bottom layers in the batch being sampled. Use the thickness of the bottom layer and the total liquid level to calculate the percentage of the bottom layer in the overall volume. If applicable, it is permitted to sample the bottom layer separately.
- If the bottom layer is dispersible, mix it under the liquid if possible (stir thoroughly with stirring rod) and then sample it.

Comment 1:
This kind of homogenisation is rarely optimal and is only possible in small containers (< 20l l). In other cases, it is best to sample the bottom layer separately.

For further visual assessment of liquids, take a liquid column over its entire height using a transparent sampling tube (plunging siphon, bailer sampler or liquid layer sampler) (14.5.1). Differences in colour and/or viscosity enable detection of any **heterogeneity or stratification (layer formation)** in the liquid. The sampling tube contents may also be considered an (initial) (laboratory) sample.

- Use a measuring tape or ruler to measure any layers. The individual layer thicknesses and total liquid height can be used to express the volume of each layer as a percentage. Always record these percentages by volume for any layers on the sampling form as well as the sampling report.
- If the layers are subsequently sampled separately (0), this enables future calculation of the average concentration in the storage unit, based on the volumes and concentrations of the different layers. In this case, dispose of the visual inspection sample properly.

Another method for determining the heterogeneity or stratification of the liquid is to take a surface and bottom sample (0) and mix the two thoroughly. After letting the container rest for few minutes, inspect the liquid. Differences in colour or viscosity usually indicate heterogeneity or stratification. This method may be used to sample deeper tanks or storage units that cannot be sampled over their entire liquid height. It is not permitted to use the (composite) sample for the visual inspection as a (laboratory) sample. Instead, dispose of it properly or return it the batch after sampling.

4.2 IDENTIFICATION OF THE LIQUID

In a subsequent stage, the sampling officer may proceed to (partial) identification of the liquid and/or any bottom or surface layers. It is permitted use what is known as ‘water finding paste’ and ‘oil finding paste’ to determine **whether the liquid and/or liquid layers are aqueous or organic (non-aqueous)**.
Apply a thin layer of paste on a dipstick or measuring tape. It will change colour as soon as it comes into contact with water (water finding paste) or an organic medium (oil finding paste).
Water finding paste is highly suited for detecting water layers at the bottom of a barrel or tank. Hydrocarbons (oil, solvents) do not affect the paste. The paste treats inorganic acids and bases as aqueous matrices. The original colour of the paste is yellowish green. In contact with water, it turns reddish.
Other liquid identification products are also available on the market (test strips, etc.).

> Comment 2:
> This identification technique is mainly intended for transparent liquids and may not be useful for silts, sludges and (coloured) viscous suspensions.

4.3 DETERMINATION OF LIQUID HEIGHT

It is vital to determine the total liquid height when sampling at specific depths. It is preferable to measure the liquid height using a dipstick or a special measuring tape to which a weight is attached. The boundary between the wet and dry parts of the stick indicates the liquid height. It is also possible to measure this with a separate measuring tape or ruler. If necessary, smear a thin layer of ‘gauging paste’ at the level of the liquid height on the tape or ruler, for better indication of the liquid height. To avoid errors, it is preferable to conduct the liquid height measurement twice.

4.4 VL1: SAMPLING OF LIQUIDS FROM SMALL STORAGE UNITS (< 20 LITRES)

The term 'small storage units' refers to bottles, cans, plastic containers, jugs or barrels with a capacity of about 20 litres, which are still easy to grip manually.

Method:

1. Use a transparent plunging siphon or sampling device with transparent sampling tube to conduct a preliminary check for stratification in the container.
2. If visual stratification is not observed, gently shake the container or stir it thoroughly with a stirring rod to properly homogenise the liquid. When opening the container, be careful of any excess pressure, as this may release when opened!
3. Pour a quantity of liquid into a 1-litre sampling container. If it is necessary to fill multiple sampling containers (for a spare or counter sample), pour smaller quantities into the sampling containers in turns.
4. To sample layered liquids in small containers, use a plunging siphon or other device with (transparent) sampling tube (liquid layer sampler, multisampler) to take a liquid column over the full depth of the container. The laboratory shall grip any separation of the layers at a later stage. Enter the instructions for this on the sample transfer form. Alternatively, if the layers are clearly distinguishable, it is permitted to take samples from the middle of each layer (e.g. with a plunging siphon, see 0).

Comment 3:
If the storage unit capacity ≤ 5 litres, a 500 (laboratory) sample shall suffice.

4.5 SAMPLING OF LIQUIDS FROM A BARREL, DRUM OR TANK (H < 2 M)

Barrels typically feature three small openings: a filling opening, a vent and an opening in the side wall for drainage. Sampling is usually possible from one of these openings.

4.5.1 VL2: GENERAL PROCEDURE FOR SAMPLING LIQUIDS IN BARRELS, DRUMS OR TANKS (H < 2 M)

To sample liquids in barrels or drums, take a liquid column over the full liquid height in the barrel or drum. For heterogeneous and layered liquids as well, this method also offers sufficient guarantees that the sample shall be representative of the full contents of a small liquid storage unit (diameter or horizontal dimension(s) ≤ 5 m).
Numerous specialised sampling devices are currently available, such as liquid layer samplers, plunging siphons and bailer samplers, in various sizes and designs. The only requirement is that the device is long enough to contain the total liquid height in the storage unit and that the device does not have a narrowed inflow opening (may exert a localised suction effect). If the device (sampling tube) is shorter than the liquid height, it is permitted to lower the device multiple times to cover the full liquid height. In small containers, do this from top to bottom, to avoid disturbing the lower layers.

> Comment 4:
> The repeated lowering of a sampling device (liquid layer sampler, sampling tube, etc.) at the same location is also an alternative sampling method for large tanks (see 0). The effect of the repetition sequence (top to bottom, or bottom to top) is negligible in large tanks.

1. For sampling, very slowly lower the (opened) apparatus (see ANNEX B) through the liquid so that the liquid level in the storage unit and in the tube remains the same during insertion. The downward movement fills the device up to the liquid level in the storage unit. If the length of the device is adequate for the liquid height, lower the (opened) device roughly to the bottom. In any case, the device shall stop when its top is just above the liquid level.

   > Comment 5:
   > A preliminary test, before the actual sampling, to practice the appropriate speed for inserting the tube in the given situation is recommended. Insertion is sufficiently slow if the liquid height in the tube after collection is equal to the liquid height in the liquid unit.

2. Seal the filled tube and then carefully lift it out.
3. Always transfer all device contents to a sampling container.
4. If it was not possible to sample the full liquid height in the container, continue sampling the remaining portion of the liquid height with the same device.
5. If one ‘liquid column’ does not provide enough liquid, repeat the procedure over the same sampling path. Always transfer the full contents of the device to the container.
   If the sampling container overflows after too many repetitions, the sample is no longer representative. In this case, it is necessary to perform the sampling operation again.
6. If multiple laboratory samples are required (spare sample, counter sample), conduct separate sampling operations for these. Under no circumstances is it permitted to distribute the contents of one device over multiple containers (risk of non-representative distribution with heterogeneous and/or layered liquids).

7. With larger tanks/liquid storage units, be sure to take horizontal heterogeneity into account. Repeat points 1 to 5 in at least three locations (liquid storage unit with diameter or horizontal dimension > 5 and ≤ 20 m) or five locations (liquid storage unit > 20 m), evenly distributed around the diameter of the tank/liquid storage unit. Document and report any horizontal deviations from or limitations (e.g. due to accessibility, etc.) of this sampling strategy (i.e. the number of sampling locations due to the size of the liquid storage unit). Combine the grabs from each sampling location into a field sample of 5 to 10 litres (which may be used to prepare multiple laboratory samples). If collecting fewer samples, take additional grabs (equal number of grabs from each sampling location).

Comment 6:
For stirred or mixed liquid storage units with aqueous or thin liquid materials, it is also permitted to take an 80% ('top') sample instead of a full liquid column. See also the instructions and conditions in Note 8, § 0.

4.5.2 VL 3: Sampling of layered liquids in barrels, drums or small tanks (h <2 m)

Alternatively, it is permitted to sample clearly distinguishable layers by taking a sample from the centre of each layer. In this case, properly identify the different liquid layers in advance (see 4.1).

Method:

1. Calculate the liquid height of the centre of each layer (see 4.3) and mark it in advance on the sampling device (or its rod or cable).
2. Also estimate the volume of each layer, to enable subsequent calculation of the average concentration in the storage unit.
3. Start with the top layer, to minimise disturbance of the lower layers. Lower the sampling device to the desired depth and take a quantity of liquid from that particular depth.
4. The device (see ANNEX B) is collected quietly. If one operation does not provide enough liquid, repeat the procedure at that particular depth.
5. Repeat points 4 to 5 for the different layers
6. Each layer is considered a separate sampling operation; the sample for each layer is collected separately in a sampling container. The description on the sampling container must include information on the percentage by volume of each layer.
7. Due to the complex volumetric composition, the laboratory shall grip selection of the composite sample. Indicate the instructions for selecting a composite sample on the sample transfer form. It is also permitted to analyse the layers separately. In this case, the volume percentages by layer provide adequate information to calculate an average composition in a later stage.
8. If multiple laboratory samples are required (spare sample, counter sample), conduct separate sampling operations for each layer. Under no circumstances is it permitted to distribute the contents of one device over multiple containers (risk of non-representative distribution with heterogeneous and/or layered liquids).
9. With larger liquid storage units, be sure to take horizontal heterogeneity into account. Repeat points 3 to 6 in at least three locations (liquid storage unit with diameter or dimension > 5 and ≤ 20 m) or five locations (liquid storage unit with diameter/dimension > 20 m), evenly distributed around the diameter of the tank/liquid storage unit. Document and report any horizontal deviations from or limitations (e.g. due to accessibility, etc.) of this sampling strategy (i.e. the number of sampling locations due to the size of the liquid storage unit). Combine the grabs from each sampling location for each layer.

A rod-operated liquid layer sampler, plunging siphon or pump is excellently suited for this sampling operation. A multisampler is also suitable (see Annex A).

4.6 SAMPLING OF LIQUIDS FROM TANKS (H > 2 M)

Tanks usually feature probe holes and access hatches. Due to the larger capacity and liquid height in tanks (> 2 m), it is not possible to use certain compact and user-friendly samplers (e.g. plunging siphon, liquid layer sampler).
Tanks are usually cylindrical in shape – vertical cylindrical (e.g. small land tanks) or horizontal cylindrical – and can hold up to around 20 m^3. 'Horizontal cylindrical tanks' refers to round containers lying on the ground, such as tankers, railway tank wagons or similar horizontal stationary tanks. It is also best to sample railway tank wagons, which may hold to 60 m^3, using the sampling procedures below for horizontal tanks of up to 20 m^3.

> Comment 7:
> NB: The proposed selection of the composite sample only applies for horizontal cylindrical tanks, not for other tanker shapes.

Tankers are usually subdivided into multiple compartments or chambers. Some compartments may be empty and others filled, without the total loading capacity of the tanker dropping below 80%. Compartmentalisation allows the tanker to transport different substances at the same time. Sample each compartment separately. It is not permitted to mix samples from different compartments. For the transport of chemicals, there are usually three compartments; for the transport of hydrocarbons, more compartments are often used (7 or 8). To prevent sloshing, tankers are often fitted with what are known as 'anti-sloshing floors' or 'baffles'.

4.6.1 VL 4: SAMPLING OF LIQUIDS FROM LARGE VERTICAL TANKS AND LIQUID STORAGE UNITS

It is preferable to sample tanks (or each tank compartment) and large liquid storage units by taking multiple samples spaced equidistantly from each other and evenly distributed over the total liquid height.

Method:

1. First determine the total liquid height in the tank (see 4.3).
2. Take at least three grabs (top, middle and bottom grabs). For straight-walled tanks and vertical cylindrical tanks, take the samples at 20%, 50% and 80% of the total liquid height of the tank. If taking more grabs, also space them equidistantly from each other and distribute them evenly over the total liquid height.
3. It is best to mark the different sampling levels on the cable of the device beforehand. Always start by sampling the top layer, to minimise disturbance of the lower layers.
4. Next, lower the sampling device (see ANNEX B) to 80% of the total liquid height ('top') and take a quantity of liquid at that particular depth.
5. Carefully pull the device out and collect the liquid in a sampling container.
6. Repeat points 4 to 5 for sampling levels at 50% (middle) and 20% (bottom) of the total liquid height. Document and report any vertical deviations from or limitations of this sampling strategy (i.e. deviation from one or more of the prescribed sampling levels).

   Comment 8:
   It is also permitted to use any taps installed at the relevant heights. For practical implementation, see 4.6.4.

   Comment 9:
   For stirred or mixed tanks/liquid storage units with aqueous or thin liquid materials, it typically suffices to take the 80% ('top') sample, provided that stirring/mixing is observable at the time of sampling and is also documented on the sampling report. In this case, it is also permitted to take the 80% sample as a 'scoop sample' with dipper. This method is not permitted for thick or paste-like liquids.

7. It is permitted to combine the grabs of the top/middle/bottom samples onsite into a composite sample, provided that the top, middle and bottom samples all contain the same volume of liquid and only complete grabs are selected.
8. If multiple laboratory samples are required (spare sample, counter sample), conduct separate sampling operations for top, middle and bottom samples. Under no circumstances is it permitted to distribute the contents of one device over multiple containers (risk of non-representative distribution with heterogeneous and/or layered liquids).
9. With larger tanks/liquid storage units, be sure to take horizontal heterogeneity into account. Repeat points 2 to 7 in at least three locations (liquid storage unit with diameter or horizontal dimension > 5 and ≤ 20 m) or five locations (liquid storage unit with diameter or horizontal dimension > 20 m), evenly distributed around the diameter of the tank/liquid storage unit. Document and report any horizontal deviations from or limitations (e.g. due to accessibility, etc.) of this sampling strategy. Combine the grabs from each sampling location into a field sample of 5 to 10 litres (which may be used to prepare multiple laboratory samples). If collecting fewer samples, take additional grabs, distributed evenly across all sampling levels and locations.

4.6.2 VL 4 B: ALL-LEVEL SAMPLE OF LIQUIDS FROM TANKS

It is preferable to use this alternative method for 0 and 0 if stratification is visible or may be assumed (e.g. multiple products present). This technique offers the advantage of sampling all intermediate layers. However, using this technique with horizontal cylindrical tanks results in deviations in the (quantitative) composition of the sample (even though all possible layers and products are represented in the sample).

Application of this technique does require some skill and experience, so only sampling officers with the relevant experience are permitted to use this technique (demonstrated with a training file).

1. First determine the total liquid height in the tank (see 4.3).
2. Use a sampling can or bottle (cage) with rope to take at least one grab over the entire liquid height, from bottom to top (all-level sample).
3. Then lower the sealed sampling device to the bottom.
4. Pull the cap off the bottle and carefully pull the device up, at an even pace, so the retrieval operation fills the bottle about 85% full.

   Note 10:
   If the bottle fills completely (100%), the technique was not performed properly and the grab must be repeated.

5. Repeat points 2 to 4. if insufficient volume has been retrieved for a single laboratory sample.
6. If multiple laboratory samples are required (spare sample, counter sample), conduct one or more additional, separate sampling operation(s) for these. Under no circumstances is it permitted to distribute the contents of one device over multiple containers (risk of non-representative distribution with heterogeneous and/or layered liquids).
7. With larger liquid storage units, be sure to take horizontal heterogeneity into account. Repeat in at least three locations (liquid storage unit with diameter or dimension > 5 and ≤ 20 m) or five locations (liquid storage unit with diameter/dimension > 20 m), evenly distributed around the diameter of the tank/liquid storage unit. Document and report any horizontal deviations from or limitations (e.g. due to accessibility, etc.) of this sampling strategy. Combine the grabs from each sampling level and location into a field sample of 5 to 10 litres (which may be used to prepare multiple laboratory samples). If collecting fewer samples, take additional grabs, distributed evenly across the sampling locations.

4.6.3 VL5: SAMPLING OF LIQUIDS IN LARGE HORIZONTAL CYLINDRICAL TANKS

With horizontal cylindrical tanks, the sampling level depends on the fill level of the tank. In this case, take the samples as per Table 1.

Note 11:
These sampling levels only apply for cylindrical tanks. Other tank or tanker shapes are subject to different selection rules, which are not included in this procedure.

Table 1: Sampling level based on fill level of horizontal cylindrical tanks (< 20 m³)

Tank fill level (% filled)	Sampling level % height from bottom			Composite sample selection (%)		
	top	middle	bottom	top	middle	Human activity buildings
100	80	50	20	30	40	30
90	75	50	20	30	40	30
80	70	50	20	20	50	30
70	-	50	20	-	60	40
60	-	50	20	-	50	50
50	-	40	20	-	40	60
40	-	-	20	-	-	100
30	-	-	15	-	-	100
20	-	-	10	-	-	100
10	-	-	5	-	-	100

Method:

1. So first determine the liquid height and total height of the tank (see 4.3). Based on this, determine the fill level (liquid height/total height * 100).
2. It is best to mark the different sampling levels on the cable of the device beforehand. Always start by sampling the top layer, to minimise disturbance of the lower layers.
3. Then lower the sampling device (see ANNEX B) bags to the desired depth and take an amount of liquid from that particular depth.
4. Carefully pull the device out and collect the liquid in a sampling container.
5. Repeat points 2 to 4 for the middle and bottom samples.
6. For each sampling level, **collect the grabs separately in a sampling container.** Top, middle and bottom samples from horizontal cylindrical tanks are always regarded as separate sampling operations. The sampling containers always bear the inscription ‘top’, ‘middle’ or ‘bottom’ and, where applicable, the ratio for composite sample selection.
7. Due to the more complex volumetric composition of the composite sample, the laboratory shall grip mixing of the top, middle and bottom samples. Indicate the instructions
   for selecting a composite sample (Table 1) on the sample transfer form.
   It is also permitted to analyse the layers separately. In this case, the selection percentages provide adequate information to calculate an average composition in a later stage.
8. If multiple laboratory samples are required (spare sample, counter sample), conduct separate sampling operations for top, middle and bottom samples. Under no circumstances is it permitted to distribute the contents of one device over multiple

containers (risk of non-representative distribution with heterogeneous and/or layered liquids).

4.6.4 VL6: SAMPLING OF LIQUIDS DURING PUMPING OR USING A TAP 15

If it is not possible to sample the storage unit (barrel, drum, tank) from above, an alternative sampling method during pumping or drainage may offer a solution. **Clearly describe/document the (in)accessibility of the storage unit on the sampling form (proof required).**
The drawback of this sampling method is the safety risks associated with opening/closing the taps and valves. Moreover, it is hardly possible to identify stratification or heterogeneities with this method.

Method:

1. Thoroughly rinse the drain pipe/valve/tap in advance. For this, let a quantity of flushing liquid slowly stream out of the storage unit. The minimum flushing volume is three times the dead volume of the sampling tap and sampling line circuit up to the storage unit, up to a maximum of 50 litres. Collect the flushing liquid in a waste bottle and close the tap. Afterwards, dispose of the rinsed liquid properly.

   Note 10:
   If flushing of the minimum volume (10 l) takes more than 1 minute at a fully open tap, flushing may be stopped after at least 3 times the dead volume of the tap and pipe volume to this tap has been flushed (e.g. for taps with very low flow).

2. For the actual sampling, reopen the tap and ensure that the liquid flows out of the tank at a moderate and constant rate.
3. It is possible to obtain a representative sample by sampling during pumping or drainage of the entire contents of the storage unit. This process may be manual or automatic, and may involve use of a sampling device of the side tube type (‘side tube device’). After 20% of the amount of liquid to pump, take a sample, and collect the liquid from each sample separately in a sampling container.
4. Repeat points 2 and 3 after 50% and 80% of pumped liquid.
5. It is allowed to combine the samples after 20%, 50% and 80% of pumping into a composite sample on site, provided that these samples all contain the same volume of liquid and only complete samples are selected.
6. If multiple laboratory samples are required (spare sample, counter sample), fill multiple sampling containers after having completed 20%, 50% and 80% of pumping or collect a larger composite sample quantity at 20%, 50% and 80% of pumping and then take a representative subsample after homogenisation with a dipper (see CMA 1/A.18).

If the liquid may be assumed to be homogeneous based on the visual inspection or process description (presence of stirring systems), it is not necessary to the pump the contents. In this case, sampling is done using the drain pipe/valve/tap for sampling as per points 1 to 2, as described above. 2, as described above. It is also necessary to document and report the visual observation at the time of sampling and any situation or process descriptions that support the choice.

Note 11:
If the liquid in the tank shall not or cannot be pumped and/or the contents cannot be confirmed to be homogeneous or are confirmed not to be homogeneous, sample from the tap in a single operation (i.e. without repeating at 20%, 50% or 80% of pumping) after the flushing procedure. Describe this operation as a deviation from this method, indicating the circumstances (process description, visual identification of homogeneity and operation of mixer/stirrer/pumping).

ANNEX A
EXAMPLES OF EQUIPMENT AND TOOLS FOR SAMPLING LIQUIDS

A.1 Plunging siphon

The simplest version of a plunging siphon is nothing more than a (transparent) tube made from glass, Teflon or another (chemically resistant and transparent) material. Once lowered in the liquid, the tube fills. There are also versions with narrowed inflow opening (of the pipette type) or with narrowing cones for liquids with different viscosity.

A plunging siphon (without narrowing) can be used to take almost undisturbed samples from drums and barrels in a very simple way. The plunging siphon is highly suited for layered liquids. The drawback of this device is its limited sampling volume.

Depth sampling

- Slowly lower the open siphon into the liquid.
- During lowering, the siphon fills with liquid.
- When the siphon reaches the bottom, cover its top with your thumb. Then carefully lift sealed siphon up.
- Wipe off the outside of the siphon.
- Transfer the full contents of the siphon to a container.

Spot sampling (depth-specific)

- Seal the siphon at the top (with your thumb).
- Lower the sealed siphon into the liquid down to the desired depth (e.g. top sample, or centre of a specific layer). At this depth, release the opening at the top. The siphon then fills with liquid from that particular depth. After closing the top, the siphon may be retrieved again.
- If necessary, clean the outside of the tube with adsorbent paper.
- To collect the sample, place the bottom of the siphon into a sampling container. The siphon drains from the bottom when the top is opened again.

A.2 Liquid layer sampler

The liquid layer sampler consists of an open Teflon tube sealable on the bottom with a stopper. It is possible to operate this sealing system using a rod or a cable.

A.2.1 Rod-operated liquid layer sampler

The rod-operated liquid layer sampler is suitable for sampling barrels and shallow liquid containers, to demonstrate stratification and measure thicknesses of floating layers. This can be used with normal, viscous and paste-like liquids. The device is made up of a stainless steel rod with a stopper at the bottom. A teflon sampling tube fits over the rod and cap. The advantage of this device is that it can also sample viscous liquids. A cable-operated model is recommended for sampling at greater depths.

Sampling over the entire depth

- Slowly lower the sampling tube with (bottom) cap opened into the storage unit. The liquid streams into the bottom of the tube and the downward movement lets the liquid stream freely 'through' the tube (NB: adjust speed to liquid viscosity).
- On reaching the bottom of the storage unit, press the tube into place onto the bottom stopper.
- The filled liquid layer sampler can now be carefully retrieved.
- If necessary, clean the outside of the tube with absorbent paper.
- To collect the sample, place the bottom of the tube into a sampling container. Pull the tube off of the bottom stopper so the liquid can flow into the sampling bottle.

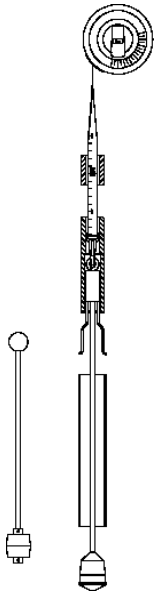
Sampling at specific depths

- First press the tube into place onto the rod with bottom stopper.
- Lower the sealed tube into the liquid down to the desired depth (e.g. centre of a layer). At this depth, pull the tube up to detach it from the stopper. The tube fills with liquid from that particular depth.
- After closing the tube, the filled liquid layer sampler can be retrieved.
- If necessary, clean the outside of the tube with absorbent paper.
- To collect the sample, place the bottom of the tube into a sampling container. Pull the tube off of the bottom stopper so the liquid can flow into the sampling bottle.

A.2.2 Cable-operated liquid layer sampler

The main difference between this sampler and the rod-operated variant is that a clip holds the (teflon) sampling tube in place at the top. A teflon-coated steel wire attached to the centre rod may be used to move the device to the desired depth. Once at the desired depth, lower down a 'messenger'. This falls onto a 'receiver', which causes the clip to close. This removes the sampling tube from the clip and drops it onto the bottom stopper. The sample is now sealed inside, and the filled device can be pulled up.

This type is used to sample deeper tanks, monitoring wells and other containers at any depth. The cable-operated variant is less suitable for thick viscous liquids.



- Use the cable to lower the liquid layer sampler to the desired height. The liquid streams into the bottom of the sampling tube and the downward movement lets the liquid stream freely 'through' the tube (NB: adjust speed to liquid viscosity).
- The messenger is dropped at the desired height. This releases the clip from the tube, and the tube falls onto the bottom stopper. The sample is now sealed inside.
- Carefully lift the liquid layer sampler out.
- If necessary, clean the outside of the tube with absorbent paper.
- To collect the sample, place the bottom of the tube into a sampling container. Pull the tube off of the bottom stopper so the liquid can flow into the sampling bottle.

A.3 Bailer sampler

The bailer sampler is the most universal type of bailer. This device consists of a stainless steel, transparent plastic or Teflon sampling tube, with a ball at bottom that closes the tube when the sample is pulled up.

These also come in variants with the top open, to promote proper stream of contents during lowering. In other designs, the top is half closed, to minimise mixing of liquid (layers) during retrieval. They often feature a cable mounted at top, to lower the device to the desired height in the liquid. An outlet adapter can be used to drain the bailer sampler without turbulence or aeration.

The bailer valve sampler is available in different lengths and diameters, with or without cable. Models with cable are similar to an immersion sampler.

Bailer samplers are excellently suited for sampling still waters or other liquids at any desired depth. Due to their low weight and the resistance of the ball, bailer samplers are less suitable for viscous fluids. In addition, it cannot take undisturbed samples.

- Slowly lower the bailer sampler into the liquid. The downward movement causes the liquid to stream into the bottom of the tube, and pushes the ball up. (NB: adjust speed to liquid viscosity.)
- During retrieval, the ball closes the tube at the bottom, sealing the sample inside.
- If necessary, clean the outside of the tube with adsorbent paper.
- To collect the sample, place the bottom of the tube into a sampling container. Mount the provided outlet adapter to the bottom of the device so the liquid can stream into the sampling bottle.

If the length of the device is adequate for the sampling height, it is also possible to sample the complete depth (see plunging siphon).

A.4 Multisampler

The multisampler consists of a stainless steel or transparent acrylic sampling canister and features a rubber piston and a stainless steel piston rod. This device can be used for two purposes. The 5-diameter canister can be fitted with either a cutting shoe to sample hard material, or a ball valve to sample liquid and/or viscous waste. If applicable, it is possible to operate the piston rod with a cable, enabling sampling at (great) depths. The multisampler cannot sample aggressive waste.

Sampling at specific depths

- Move the multisampler to the desired height in the liquid.
- For sampling at a specific depth, hold the sampling tube stationary in the liquid. Thus, only pull up on the piston. Pulling the piston rod up creates a vacuum in the tube, which facilitates sampling. This also prevents sample loss. With a connecting rod, it is possible to use the device at greater depths (e.g. 10 m).

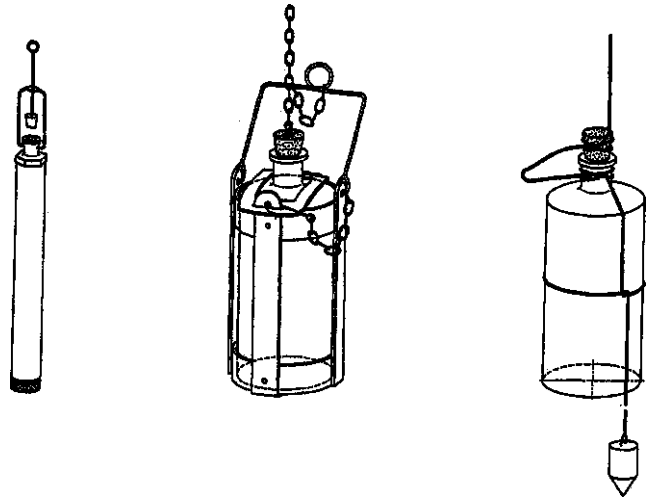
Sampling over the entire depth

- Put the multisampler into the top of the liquid.
- Hold piston rod stationary with respect to the material being sampled. Thus, only push the tube downwards, taking in a liquid column. Once the tube is completely full, carefully lift the device (tube with piston rod) out.

A.5 Sampling bottle or can

This device consists of a holder with glass or plastic bottle with stopper, possibly with zinc lead, and a retrieval cable. A sampling bottle can be used to take depth-specific samples (e.g. top/middle/bottom samples) in tanks.

The disadvantage of the sampling bottle is that it requires a large sampling opening. The sampling bottle is less suitable for viscous liquids. The advantage of this method is that the bottle can also serve as the sampling container for the laboratory sample.



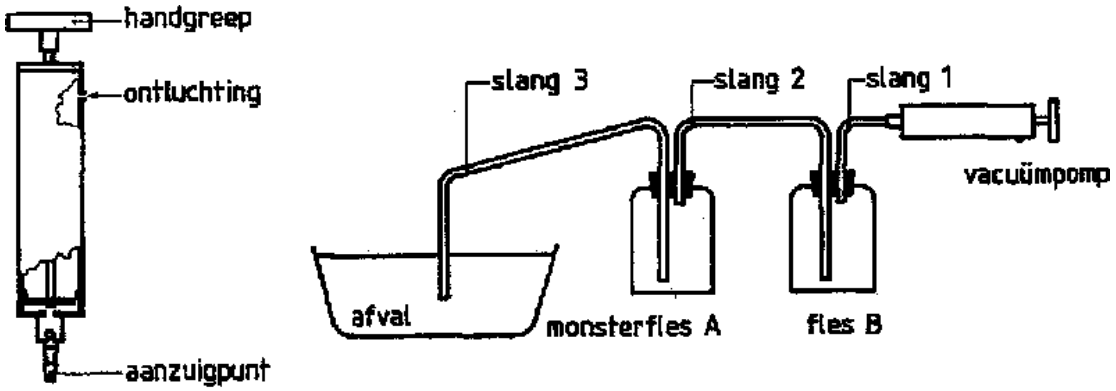
- Use the cable to carefully lower the closed sampling bottle into the liquid to the desired depth.
- A sharp tug on the cord ‘uncorks’ the bottle so it can fill with liquid.
- Once the bottle is full, carefully lift it out.

Depending on the speed at which the cap is removed and at which the bottle is retrieved during filling, it may also be possible to take an all-level sample (this requires a certain degree of experience and skill!)

A.6 Vacuum pump

The vacuum pump can be used to take spot samples of sufficiently liquid waste. The vacuum pump is not suitable for sampling aggressive substances (strong acids and bases). It is difficult to use with highly viscous fluids. This pump is less suitable for sampling volatile materials, due to loss of volatile components. In fact, it is not recommended to use a vacuum pump to sample liquids whose composition is completely unknown. Use of the vacuum pump is limited by the maximum head when extracting approx. 7 metres. Electric and manual vacuum pumps (piston type) are available. The electric variant is recommended if a sustained vacuum is required, e.g. to collect larger volumes of liquid.

Vacuum pumps are used in a setup with one or more filter flasks between the pump and the liquid. This setup prevents the pump itself coming into contact with the liquid waste. This must be strictly avoided, as it would compromise the inside of the pump. The pump creates a vacuum in the system to draw the liquid from the storage unit through a hose connection (suction hose) and into the sampling bottle.



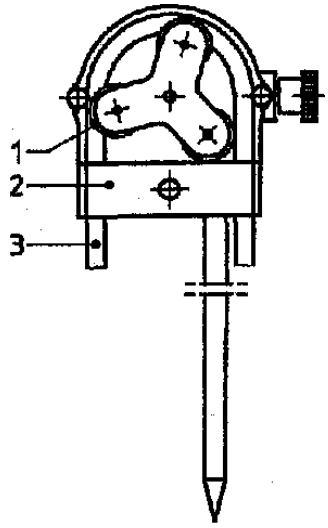
handgreep	handle
ontluchting	vent
aanzuigpunt	intake point
afval	waste
slang	hose
monsterfles	sample bottle
vacuümpomp	vacuum pump

- Connect the vacuum pump to the filter flask with a hose. A second suction hose connects the container to the liquid being sampled and the filter flask (this hose runs through the filter flask stopper almost to the bottom of the flask).
- Place the end of the suction hose at the desired depth in the liquid (e.g. the centre of a layer).
- Start the pump so the liquid streams through the suction hose to the filter flask. The filter flask fills with liquid.

A.7 Peristaltic or roller pump

The principle of a peristaltic pump is based on peristaltic movement in a hose. Pump head rollers squeeze the hose and push the liquid along. Peristaltic pumps come in manual and automatic variants. Peristaltic pumps are suitable for spot sampling liquids that are not too viscous. They are less suitable for sampling volatile substances, though they lose less than vacuum pumps.

One benefit of the peristaltic pump is that the liquid only comes into contact with the hose, which limits maintenance and extends its service life. Selection of hose material is also vital for proper pump functioning, and thus also proper sampling. The hoses are simple to replace, to prevent contamination from previous sampling operations.



1 aandrukrollen
2 aandrukbeugel
3 pompslang

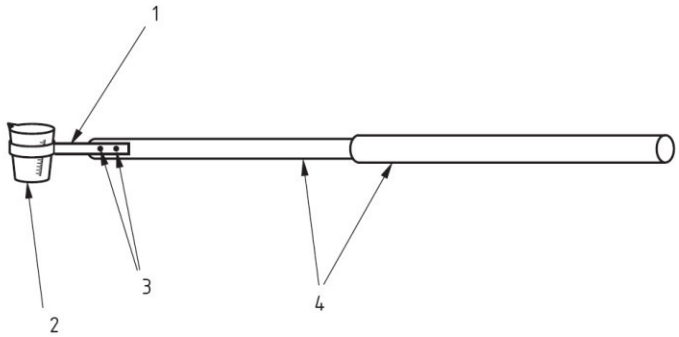
aandrukrollen	pressure rollers
aandrukbeugel	pressure clamp
pompslag	pump stroke

- Place the end of the suction hose at the desired depth in the liquid (e.g. the centre of a layer). Place the other end of the hose in a sampling container.
- As soon as the pump starts, it creates a slight vacuum to pump the liquid into the sampling container.

A.8 Side tube device

See §4 "sampling during transport" of BAM/Part 3/01 "Liquid manure - sampling".

A.9 Dipper with beaker



Key

1 = Varigrip
2 = Beaker 150 ml to 600 ml
3 = Bolt Holes
4 = Telescoping Aluminium Pole 2,5 m to 4,5 m (8 to 15ft)

ANNEX B
POTENTIAL APPLICATIONS OF SAMPLING EQUIPMENT

		Plunging siphon	Bailer sampler (long model, with sampling tube)	Bailer sampler (short model, bailer type)	Liquid layer sampler, rod-operated	Liquid layer sampler, cable-operated	Multisampler	Vacuum pump	Peristaltic or roller pump	Sampling bottle or can	Dipper with beaker	Side tube device
4.4	VL1: Liquids in small storage units	++	-	-	-	-	-	-	-	-	-	N/A
4.5.1	VL2: General procedure for sampling liquids in barrels or drums	+[3]/-[4]	+	-	++	-	++	++	++	-	-	N/A
0	VL3: Sampling of layered liquids in drums or drums	++	-	-	++	-	+	++	++	-	-	N/A
0	VL4: Sampling of liquids in large vertical tanks or liquid storage units	-	-	++	-	++	++	-	+	++	+[5]	N/A
4.6.2	VL4 b: all-level sample of liquids in large vertical tanks	-	-	-	-	-	-	-	+	++	-	N/A
0	VL5: Sampling in large horizontal cylindrical tanks	-	-	++	-	++	++	-	+	++	-	N/A
4.6.4	VL6: Sampling of liquids during pumping or using a tap	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	++

++ highly suitable
+ suitable
- not suitable
N/A not applicable

[3] Only suitable for plunging siphon with uniform diameter (without narrowed inflow opening)
[4] Not suitable for plunging siphon with narrow inflow opening
[5] See note 9

On-site sample pretreatment

CONTENTS

1 INTRODUCTION

This method replaces the version of June 2024.

This procedure describes onsite sample pretreatment of solid, liquid and pasty raw materials[1], conducted after the actual sampling but still considered part of the sampling process.

This method does not apply to special on-site sample pretreatment for asbestos analysis. For this, please refer to the guidelines in the relevant sampling procedures (CMA/1/A.19, CMA/1/A.20).

The various options for on-site sample pretreatment are only intended for:

- selection of a field sample (if applicable);
- reduction of the sample quantity from the field sample to the laboratory sample, and any subsamples taken (counter analysis, etc.) from the field sample;

to facilitate the transfer, storage, preservation and transport of samples to the analysis laboratory.

Changes in material integrity (e.g. particle size reduction)[2] and/or subsampling for collection of test portions from solid materials result in the loss of fine particles or in cases of solids and pastes (e.g. sewage sludge), may cause significant sampling errors or deviations in representativeness. Therefore, *only the analysis laboratory* should perform the aforementioned operations on solids and pastes.

Subsampling of pastes, such as wet (sewage) sludges, in smaller test portions is permitted as an alternative onsite, if using a powerful mechanical mixer.

For guidelines on sample pretreatment in the laboratory, please refer to CMA/5/A. If it appears that changes in material integrity are necessary and must be performed onsite, detail the operations performed and equipment used on the sampling form and copy these to the sampling report.

2 EQUIPMENT AND MATERIALS

The sampling officer must have the necessary equipment and tools or have access to a mobile laboratory. To guarantee the condition and integrity of the sample(s) taken, it must be possible to shield on-site sample pretreatment from rain and wind to minimise losses (volatilisation of moisture and/or volatile components, fine particles) and/or cross-contamination.

[1] VLAREMA art. 1.2.1 §2 35° defines raw materials as by-products or materials that have reached the end of the waste phase as per Articles 36, 37 or 39 of the Materials Decree

[2] One exception to this is the sampling of shaped materials. For these materials, taking grabs often requires taking a cross-section, borehole or filing.

2.1 Sturdy tarpaulin or tray (e.g. PE) of suitable dimensions to contain the sample quantity*
2.2 Mixing vessels, 10 to 50 litres (for sludges)*
2.3 Mixing barrel or bucket, sealable with lid (for homogenisation of powders)
2.4 Bucket of 20 l to homogenise liquids*
2.5 Scoop or hand scoop for quartering and/or filling of sampling containers*
2.6 Shovel/spade*
2.7 Quartering divider (optional)
2.8 (Static) splitter* with at least eight chutes of custom dimensions (3 x D_{95})
2.9 Wheel loader, bulldozer or excavator with shovel (optional, in combination with 5.3.3)
2.10 Spring scale or other scales with a measurement accuracy of better than 1% (optional)
2.11 Stirring rod(s), suitable for homogenisation of liquid and viscous materials*
2.12 Powerful mechanical mixer (of the cement mixer type) with mixing paddle and drive (subsampling of wet sludges; optional)
2.13 Dipper, suitable for subsampling sludges (optional, only in combination with 2.12)
2.14 Pipette or plunging siphon*

For figures: see Annex ABasic equipment[3]

3 GUIDELINES FOR LABORATORY SAMPLE SIZE

3.1 SOLIDS AND PASTES

Depending on the particle size of the sampled material, it may be necessary to reduce the size (quantity) of the field sample taken to a laboratory sample (Table 1).

In addition, the minimum size of the laboratory sample also depends on the planned analyses. As a general rule, (completely) filled sampling containers with a capacity of 1 litre shall suffice for standard analyses.
Certain analysis methods (e.g. leaching tests, determination of non-stony and organic contaminants, etc.) require larger quantities (at least 2.5 kg ds or sample container 3 litres).

If the field sample is too small for the specified laboratory sample size, it is possible to make the field sample larger by increasing the number of grabs and/or the grab size.

For practical reasons, the sample quantity delivered to the laboratory is limited to 10 or 20 (compost) litres.

Determination of volatile parameters requires a separate sampling* operation and a separate container of at least 200 ml.

[3] Ensure that basic equipment is available to every authorised sampling officer, for use according to the planned sampling.

Table 1. Guidelines for field and laboratory sample sizes

<table>
<tr><th>Granular size D_{95}</th><th>Field sample size[4]</th><th>Minimum laboratory sample size</th></tr>
<tr><td rowspan="5">0-10 mm</td><td>- Variable: grab size x number of grabs[5] (approx. 1* to 6 litres)[6]
- Min. 40 kg to determine floaters, sinkers and glass</td><td>- Standard: 1 litre
- 3 litres if leaching tests[7] need to be carried out
- 10 litres if determination of floaters, sinkers and glass is required
- separate sample (sampling tube or glass container at least 200 ml) for volatile organic components (if applicable)</td></tr>
<tr><td colspan="2">Solid waste (wet sewage sludge)</td></tr>
<tr><td>5 or 10 litres*</td><td>- Standard: a 5-litre bucket
- 5 to 10 litres[8] if leaching tests[7],
- smaller volume only permitted with adequate use of mechanical mixer and careful subsampling with dipper.
- separate sample (sampling tube or glass container at least 200 ml) for volatile organic components (if applicable)</td></tr>
<tr><td colspan="2">Compost</td></tr>
<tr><td colspan="2">10 litres[9] (min. 6 kg) / 20 litres (or min. 12 kg)</td></tr>
</table>

[4] see also CMA/1/A.13 § 6
[5] grab size x number of grabs according to CMA/1/A.15 (solid), CMA1/A.17 (sludges)
[6] depending on method and equipment used!
[7] CMA/2/II/A.9.1 (Building material percolation test), CMA/2/II/A.9.5 (Landfill percolation test), CMA/2/II/A.12 (Single-step landfill batch leaching test)
[8] corresponding to a minimum of 2.5 kg dry matter
[9] if only parameters of analysis packages A.2.1 and/or A.2.2 are to be analysed

11–40 mm	- Variable: grab size x number of grabs[5] (approx. 1* to 80 litres)[6] - Min. 80 kg to determine floaters, sinkers and glass	- ***Standard: 3 litres*** - 10 litres if determination of floaters, sinkers and glass is required in construction materials of particle sizes of up to 20 mm, 20 litres if determination of floaters, sinkers and glass is required in construction materials of particle sizes of up to 40 mm, - separate sample (sampling tube or glass container at least 200 ml) for volatile organic components (if applicable)
	Compost	
	min. 20 litres, > 21 mm variable: grab size x number of grabs[5] (up to 80 litres)[6]	10 litres[9] (min. 6 kg) / 20 litres (min. 12 kg)
	Soil improver	
	Variable: grab size x number of grabs[5] (approx. 1* to 80 litres)[6]	- ***At least 5 litres*** - If determining volatile organic parameters, provide a separate glass container of at least 1000 ml
41-100 mm	- Variable: grab size x number of grabs[5] (approx. 25 to 600 litres) - For determination of floaters, sinkers and glass: see CMA/2/II/A.22 (Table 1)	- ***Standard: 5 litres*** - For compost: 10 litres[9] (min. 6 kg) / 20 litres (min. 12 kg) - 30 litres if determination of floaters, sinkers and glass is required
> 101 mm	Variable: grab size x number of grabs[5] (approx. 100 to 600 litres)	- **Standard: 10 litres).** - For compost: 10 litres[9] (min. 6 kg) / 20 litres (min. 12 kg)

*field sample = laboratory sample

3.2 LIQUID WASTE

3.2.1 NON-AQUEOUS LIQUIDS

As a standard, the sampler delivers a laboratory sample of 1 litre.

The quantity may be less than 1 litre, if applicable. One condition here is that the laboratory must receive sufficient material for analysis. Always document any deviations from the standard quantity on the sampling form.

3.2.2 AQUEOUS LIQUIDS

The Water Analysis Compendium (WAC) gives the guidelines for sample quantities for the different analysis parameters.
In practice, it is often necessary to take a quantity that is much larger than 1 litre.
Dividing into separate sub-samples for the various analysis parameters is a common practice in the sample pre-treatment of water samples. During sampling, this involves dividing the water sample over several sub-samples for the parameters to be analysed - with or without preservative cf. WAC/I/A/010 - divided.

For specific liquid streams of soil improvers with a dry matter content < 2%, the following applies:

- effluents, effluent stream, biological treatment effluent, wash water, chaste water: divide into separate subsamples for the various analytical parameters with corresponding preservation cf. WAC/I/A/010 or BAM/part 3/01-C;
- other streams (e.g. concentrate) are not distributed onsite among sub-samples for the different parameters, but are delivered to the lab as a whole (quantity of field sample collected according to CMA/1/A.16).

Comment 1:
Depending on the nature of the sample (e.g. samples with high organic carbon), a violent reaction may occur when acidifying. For safety reasons, it may then be decided not to carry out on-site conservation based on expert judgement (visual assessment). In case of doubt, the acidification is left to the laboratory.

4 PREPARATION

- Select a suitable location to perform sample pretreatment: a flat, hard subsurface shielded from rain and wind and large enough to process the sample on all sides. Alternatively, the mobile cleanroom may feature a mixing and quartering table or space of adequate size.
- For sample pretreatment of solids: use a plastic tray or tarpaulin to avoid contaminating the subsurface.
- For liquid materials, it is preferable to use absorbent paper or plastic, to prevent spillage.
- Make sure that all equipment and tools are cleaned between uses.

5 SAMPLE PRETREATMENT FOR SOLID MATERIALS (OTHER THAN FOR ASBESTOS ANALYSIS)

5.1 GENERAL

Various sample pretreatment scenarios are possible:

1. The required number of grabs taken together (=field sample) is a quantity that is *equal to* the prescribed quantities for the laboratory sample[10] (Table 1).

Combine the grabs into a field sample, which is also the laboratory sample. Mixing/homogenisation and other sample pretreatment steps are not necessary.

2. The prescribed number of holds together (= field sample) constitutes an amount *smaller than* the minimum size of the laboratory sample[10] (Table1).

Increase the number of grabs and/or grab size until the field sample quantity meets the minimum laboratory sample size. Combine the grabs into a field sample, which is also the laboratory sample. Mixing/homogenisation and other sample pretreatment steps are not necessary.

3. The required number of grabs taken together (=field sample) is a quantity that is *greater than* the prescribed quantities for the laboratory sample[10] (Table 1).
   a. Since the values in Table 1 are minimum sample sizes, it is permitted to take a larger laboratory sample.

For practical and economic reasons (storage), the laboratory sample size is limited to 10 or 20 (compost) litres. Deviations from this rule are only permitted in consultation with the relevant laboratory.[11]

b. Mix the field sample as per § 5.2 and reduce the sample size as per § 5.3 until the laboratory sample meets the size indicated in Table 1.

c. Alternatively, it is also permitted to reduce the amount of material in the individual grabs evenly as per § 5.3 and then select a laboratory sample of the desired size from the reduced grabs (all with the same apportionment factor) and homogenise it as per § 5.2. However, this method does not entail that the grab may be less than that indicated in CMA/1/A.14.

4. It is necessary to *prepare more than one laboratory sample from the same field sample* (e.g. sample and counter sample, spare sample).

Mix the sample as per § 5.2 and divide it into the required number of specimens as per § 5.4.

The resources, conditions or heterogeneity of the sampled material do not permit *proper on-site sample pretreatment* (e.g. large bulky material components).

Package each grab separately and deliver them as is to the analysis laboratory, along with instructions for selecting the final laboratory sample.

[10] Only if a single laboratory sample specimen is required. If multiple laboratory sample specimens are required, scenario 4 applies.

[11] In the case of wet paste sludge, smaller volumes of samples for labo may be allowed, provided that proper use of a mechanical mixer and careful partial sampling (§ 7.2).

5. The analysis laboratory can preform the necessary pretreatments with special equipment and techniques (overhead mixer, roller bed, Turbula, particle size reduction, etc., see CMA/5/A.7).

6. Volatile parameters must be determined. This requires a separate sample (CMA/1/A.15, § 3.4). Package the additional field sample for volatile parameters (often consisting of a single grab) immediately without handling it, to minimise contact with air.

As a general rule in all of the above scenarios, sample pretreatment does *not* involve removal of materials foreign to the matrix. The analysis laboratory performs, records (nature, percentage by weight of removed components) and reports on this operation as part of sample pretreatment for analysis (CMA/5/A1-A.9).

Comment 2:
Examples of matrix foreign materials include, for example: metals, glass, wood, textiles in recycled debris aggregates from concrete, masonry and/or asphalt rubble; branches, twigs, plant residues; plastics in wood waste, metals in shredder fluff, pieces of furnace bricks in pellet ash from combustion, residue from mould in moulding sand, etc

It is only permitted to remove material during sampling if it would interfere with further sample pretreatment (5.2, 5.3, 5.4) and if the sampling officer records its type and quantity (m%) on the sampling form. This information must also appear on the sampling report.

5.2 MIXING - HOMOGENISATION OF SOLID MATERIALS

General procedure

Spread the grabs out on an inert subsurface (tarpaulin or flat tray). Always be sure to mix equal amounts from the grabs. In practice, this is done by combining the grabs in their entirely (unless using the scenario in § 5.1, 3c).

Comment 3:
Because weighing grabs on site is cumbersome and impractical, equal volumes are preferred. Equal grab volumes are only possible with reproducible sampling equipment (e.g. scoop with raised edges that is filled completely or to the same level each time).

1. To mix the material, us a scoop, hand scoop, shovel or spade to move the outer sides of the material towards the centre. The dimensions of the scoop, shovel or spade must require at least 20 repetitions of this operation to move the entire (composite) sample.
2. Flatten out the resulting pile and then spread it out again.
3. Points 2 to 3 is repeated 3 times.

Another method consists in scooping the material from one pile to another three times. Place each scoop on the top of the new pile.

Note 4: dry powders and/or dusts (< 1 mm)
Mixing dry powders and/or dusts (e.g. fly ash, wood dust) on site often results in a great deal of dust formation and loss, especially when unshielded or outdoors. In addition, these materials may contain harmful amounts of contaminants that disperse easily during mixing operations. Homogenisation of these materials on site should be avoided as much as possible. Simply package the field sample, as is, to create the laboratory sample.

5.3 SAMPLE SIZE REDUCTION

The current techniques that are easy to apply on site are (static) splitting and quartering. The 'long pile' method is used for large quantities. These techniques cut the sample quantity in half with each application. Each (halved) sample, in its entirety, should be considered a laboratory sample. If the sample quantity is still too large for the planned laboratory sample quantity, repeat the reduction step one or more times.

Although mechanical sample dividers (rotary dividers, rotating tube dividers, etc.) produce better results (less deviation), these sampling guidelines do not cover them in detail. They require a power supply and an amount of space that are often not available in a mobile cleanroom. The use of these devices is of course permitted. Also indicate the technique used on the sampling form.

Comment 5:
It is not permitted to reduce the quantity of a field sample consisting of powders and dusts (< 1 mm) on site, to prevent dust formation (see also Note 4). Due to the fine particle size, the equipment and grabs are generally small enough to preclude the need for further reduction (scenario 1 or 2).

5.3.1 STATIC SAMPLE SPLITTER

A static splitter consists of a container featuring multiple partitions at the bottom, each ending in a different collecting bin ('splitting' or 'riffling'). The slits have a pre-set slit width (minimum) return the maximum particle size2 provided that the free passage of the material is ensured). The number of chutes typically ranges from 6 to 12.

Splitters can only be used with relatively dry materials that can fall freely through the chutes. Static splitters can be used for sample quantities of up to around 50 litres, provided that it features specially selected collecting bins and chutes.

Method

1. Check that the chute width of the divider is at least three times greater than the maximum particle size (D_{95}). Also check that the collecting bins of the divider can accommodate the sample quantity.
2. Distribute the full quantity of the material evenly across the splitter, so it falls through the chutes into two collecting bins. This produces two subsamples of equal size.
3. After completion, check that the divider chutes are empty. If they are not, tap the divider to dislodge any remnants from the chutes.
4. One part is removed; the other is further considered.
5. Repeat points 2 to 4 until the desired quantity of laboratory sample is obtained.

Comment 6:
Rotary splitters are also available for dry fine-grained materials. These are fed using an automatic vibratory feeder. These devices provide more accurate division, but are not suitable for use in the field (possibly in a mobile or process laboratory onsite).

5.3.2 QUARTERING

This manual method is permitted for reducing sample quantities of up to 250 litres down to as little as approx. 0.5 litres.

Method

1. Homogenise the sample quantity as per 5.2 to create a conical pile.
2. Flatten the pile out and spread the material into a circular layer.
3. Use the scoop to divide the circle into four equal quarters (this is quite simple with a 'quartering divider').
4. Discard two diagonally opposite quarters. Be sure to remove all the material (including the fine material).
5. Combine and homogenise the two remaining quarters (5.2).
6. Repeat points 2 to 5 until the desired sample quantity is obtained. Transfer the entirely of the two remaining quarters into a single sampling container.

5.3.3 LONG HEAP METHOD

This method is useful for dividing sample quantities of over 50 litres. If the sample quantity is greater than approx. 250 litres, it is recommended to use large rolling stock (wheel loader) to move the pile.

Method

1. Homogenise the sample quantity as per 5.2, creating a conical pile.
2. Shape the conical pile into a long pile by taking a scoop from the bottom of the pile and setting it just to the left of the pile. Take a second scoop from another point in the pile and set it next to the pile directly opposite from the first scoop. Place a third scoop from the pile right next to the first one, a fourth scoop next to the second one, and so on. Repeat, alternating between setting scoops to the left and right, to spread the pile out in a long line.
3. The method for halving the pile is as follows: leave one scoop of material at one end in the long pile. Scoop up and discard the amount of material directly next to the first scoop. Leave the third scoop next to it in the long pile, but discard the fourth one. Continue this procedure from one side of the line to the other, discarding every other scoop (the even ones) until the pile is divided in half. The material placed in a line must be taken with a shovel having a flat bottom and straight edges.
4. Repeat points 2 to 3 until the desired sample quantity is obtained.

5.4 DIVISION

The splitter and the quartering method may also be used as a division technique if two or more specimens of a sample are required as spare sample(s) or any counter samples. The two subsamples are assumed to have the same composition and quantity.

The two techniques produce two subsamples of equal size.

If an odd number of subsamples is required, apply a variant of the quartering method. Instead of dividing the circle into four parts, divide into more equal parts, and take two opposite parts together for each subsample.

Comment 7:

It is not recommended to divide powders and dusts (< 1 mm) on site, due to dust formation (see also Note 3 and Note 4). If multiple specimens of the laboratory sample are required, take separate grabs during sampling to obtain multiple field samples from the same sampling operation. This method is only permitted for fine powders and dusts < 1 mm because the distribution (heterogeneity) of powders is more similar to (viscous) liquids than coarse-grained solid materials.

In practice, this means taking the required multiples of every grab at the same sampling location. Take these grabs for the different field samples close together at the same sampling location (height, depth), using the same equipment and method. Package each composite sample, which constitutes a separate field sample, as a laboratory sample without homogenisation or sample quantity reduction.

6 SAMPLE PRETREATMENT FOR LIQUIDS

6.1 GENERAL

Minimise on-site sample pretreatment of liquids. In particular for non-aqueous liquids and aqueous heterogeneous or layered liquids, on-site resources are limited and manual techniques are inadequate for proper homogenisation and subsampling. The prescribed quantities for liquid field samples are also less than with solid materials. The responsibility for mixing, homogenisation and division (subsampling) should fall to the analysis laboratory wherever possible.

The guidelines and points for attention below apply.

- Unlike sampling for solid materials, liquid sampling may consist of a single grab (if only one specimen of the sample is needed).
  Naturally it is permitted to take multiple grabs, such as if a single operation (grab) does not provide enough material for a laboratory sample of 1 litre.

- Conduct separate sampling operations (grab(s)) to obtain multiple specimens of a non-aqueous sample.
  For aqueous liquids, it is permitted to prepare an composite sample (field sample) from different grabs, homogenised and divided as per 0 and 6.3, respectively.

- If determining volatile parameters, it is preferable to take a separate sample (grab) and package it and deliver it to the laboratory as soon as possible.

- As a general rule for liquids, always transfer the full contents of the sampling device to a sampling container, even in cases of multiple grabs per sampling container. Thus, it is not permitted to divide a single grab across multiple sampling containers, without homogenisation. In addition, it is not permitted to overfill the sampling container. This means a proper estimate of sampling device capacity is critical!

- When sampling at specific depths, take the same number of grabs each height, to keep the composition of the composite sample representative of the batch.

- It is preferable for the laboratory to mix the top/middle/bottom subsamples into a composite sample in horizontal cylindrical tanks, but for practical considerations, this is also permitted on the sampling site itself. Indicate the sample selection instructions on the sampling form.

6.2 MIXING - HOMOGENISATION

6.2.1 NON-AQUEOUS LIQUIDS

In principle, it is not permitted to homogenise non-aqueous liquids on site. This is because the (laboratory) sample always consists of one or more complete grab(s), and it is necessary to prepare multiple specimens of the laboratory sample using separate (alternating) sampling operations (grabs).
Deliver the field sample(s), consisting of one or more grabs, to the laboratory without any pretreatment. The laboratory will perform thorough homogenisation anyway for division into test portions for the requested analyses (CMA/5/A.2 and CMA/5/A.6).

6.2.2 AQUEOUS LIQUIDS

It is easy to mix homogeneous liquid samples by shaking or stirring with a stirrer or dipper. Perform stirring or shaking operations both horizontally and vertically.

In practice, liquid samples often also contain solid particles, separate phases or layers that are (highly) susceptible to gravity and/or that separate easily. For this reason, thorough homogenisation, including between two subsamples, is vital.

Deliver heterogeneous liquids (e.g. suspended particles) or ***solid-liquid mixtures*** that are difficult to homogenise manually to the analysis laboratory for mixing and division with specialised equipment (overhead mixer, roller bed, Turbula, mixer, etc.). Enter the instructions for this on the sampling form.

It is not permitted to mix or divide layered liquid samples on site. Package each individual grab of these samples and deliver these to the analysis laboratory, along with analysis instructions on the sampling form.

6.3 SAMPLE SIZE REDUCTION - DIVISION

6.3.1 NON-AQUEOUS LIQUIDS

For non-aqueous samples, a single grab or multiple of a grab is always a single laboratory sample.

Use separate (alternating) grabs to prepare multiple specimens of the sample (see on the sampling of liquids, CMA/1/A.16).

Non-aqueous liquids are generally not preserved on site. Typically, a single laboratory sample is provided for all analysis parameters.
For volatile parameters, deliver a separate laboratory sample from a separate sampling operation (grab) to the laboratory.

6.3.2 AQUEOUS LIQUIDS

It is necessary to preserve aqueous samples on site and thus also divide them on site into subsamples for the different analysis parameters.

Immediately after homogenisation, take necessary subsamples with a pipette, dipper or plunging siphon and transfer them to one or more sampling containers. Always stir or homogenise again between each subsample.

Guidance on the method of preservation and volume according to the analytical parameter is given in WAC/I/A/010. The general points for attention below apply when filling the containers after subsampling.

- Do not overfill sampling containers containing a preserved sample, or fill them over other containers, to avoid cross-contamination with the preservative.
- Prevent bubbles during sampling. For this reason, it is best to maintain a low flow rate, to prevent turbulence.
- Fill sampling containers completely, unless explicitly indicated otherwise for sample preservation.

For specific liquid streams of soil improvers with a dry matter content < 2%, the following applies:

- effluents, effluent stream, biological treatment effluent, wash water, chaste water: divide into separate subsamples for the various analytical parameters with corresponding preservation cf. WAC/I/A/010 or BAM/part 3/01-C;
- other streams (e.g. concentrate) are not distributed onsite among sub-samples for the different parameters, but are delivered to the lab as a whole (quantity of field sample collected according to CMA/1/A.16).

7 SAMPLE PRETREATMENT FOR PASTES

7.1 MIXING - HOMOGENISATION

In general, malleable pastes (e.g. sewage sludge, clay-like materials) are pretreated as with solid materials. For homogenisation, they are manually worked and kneaded into a homogeneous paste.

Of course, it is only necessary to perform these mixing operations if the composite sample > 10 litres or if preparing multiple specimens of the laboratory sample, scenarios 3 and 4 as per § 5.1, respectively.

For ***wet viscous and/or sludge-like materials*** (e.g. non-dewatered sewage sludge, other sludges and slurries), it is preferable not to perform any on-site homogenisation or division step (see also 7.2), and instead to leave this to the laboratory.

7.2 SAMPLE SIZE REDUCTION - DIVISION

After homogenisation (7.1), reduce and/or divide cohesive, malleable pastes as with solid materials as per 5.3 and 5.4.

It is preferable not to divide heavy viscous sludges on site. In this case, deliver the field sample to the laboratory without on-site pretreatment (up to 10 litres).
If necessary, partial sampling may be carried out on site, but only after careful homogenisation with a powerful mechanical mixer (cement mixer type). Transferring the material into one or more sample containers is done with a sample spoon or ladle. To reduce the risk of separation, use the dipper to homogenise before each scoop. It is not permitted to fill multiple sampling containers in a single operation or with the contents of a single dipper at the same time. Always transfer the full contents of one dipper to one sampling container[12] (see also under 'liquids', 6.1). This must not cause the sampling container to overflow.

[12] This means that the capacity of the dipper must be less than or equal to the capacity of the sampling container. Avoid pouring out a portion of the contents of the dipper, as this may cause separation or segregation of the sludge.

ANNEX A

EXAMPLES OF SAMPLE PRETREATMENT TECHNIQUES AND EQUIPMENT

A.1 Static sample splitter

A (static) sample splitter always has an even number of chutes, with a minimum of six. To avoid clogging, the chutes should be at least three times larger than the maximum particle size.

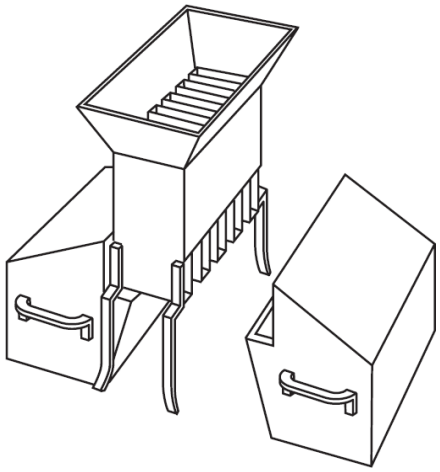


Figure 1. Sample splitter (source: EN 932-1)

A.2 Quartering

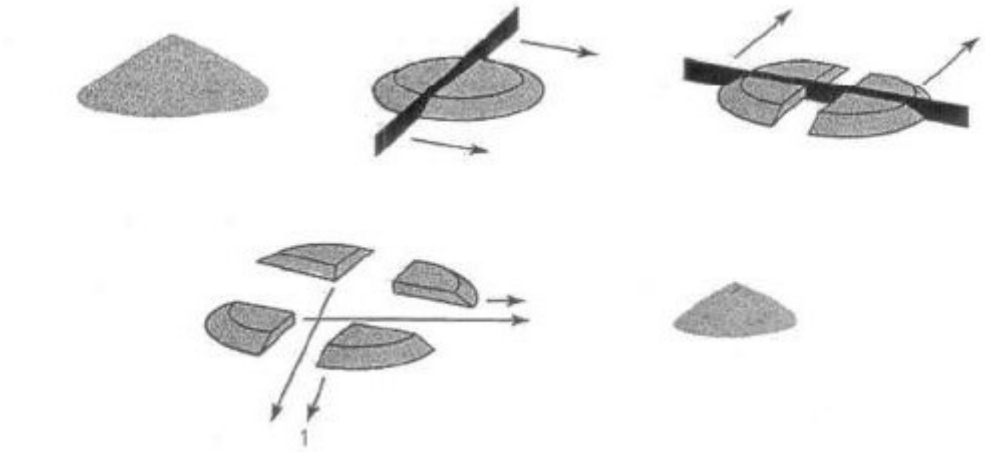


Figure 2. Quartering technique (source: CEN/TS 14780:2005)

A.3

A.4 Mechanical dividers: rotary splitter

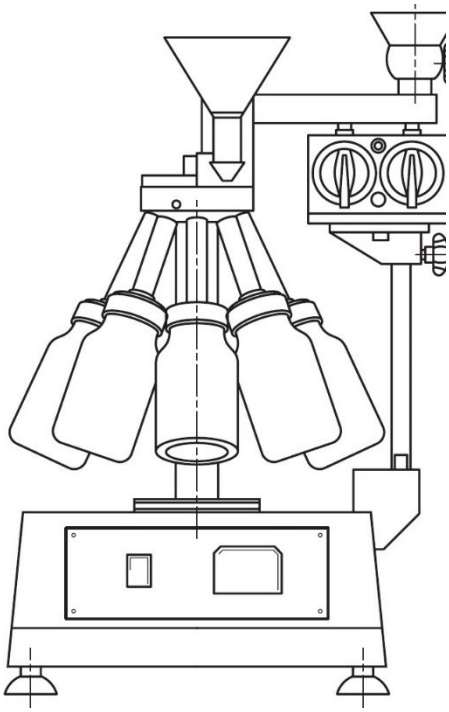


Figure 3. Rotary splitter for fine-grained solid materials
(source: EN 932-1)

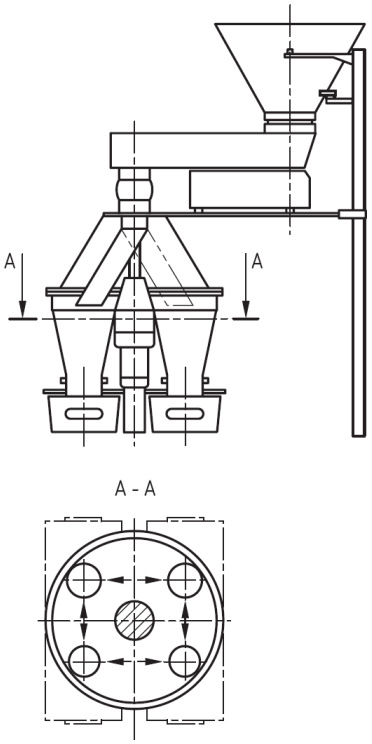


Figure 4. Rotary splitter for coarse-grained solid materials
(source: CEN/TR 15310-3:2006)

Parameter-specific guidelines

CONTENTS

1 OBJECTIVE AND SCOPE

This procedure is new.

The method describes the specific guidelines to be followed when sampling various matrices including soil, water bottom, semi-solid soil materials, non-semi-solid soil materials, groundwater, surface water, drinking water and solid, pasty or liquid waste streams for the analysis for specific parameters.

2 PFAS

When sampling is carried out in the context of PFAS analyses, the following guidelines shall be taken into account:

- In sampling, only materials that do not release/leach PFAS may be used.
- Any direct contact with the sample must be avoided. This means that the sample must be taken with a cleaned tool or using gloves that can be shown not to leach PFAS.
- Cleaning of material:
  - only with water of drinking water quality;
  - When applying the cleaning product, only use PFAS-free detergents, and rinse thoroughly afterwards.
- The use of cooling elements shall be permitted under the following conditions:
  - The freeze blocks do not exhibit any leaks;
  - Only hard freeze blocks are permitted, as flexible ones pose a higher risk of cracking.
- No restriction shall be imposed on rain, safety and other clothing to be worn if the following conditions are met:
  - Avoid direct contact with the sample;
  - Do not allow rainwater to run off the clothing and into the sample;
- When laying out and homogenising/quartering samples, it is only permitted to use HDPE plastic ducts or plastic liners if the filling of the containers does not damage them. If this cannot be guaranteed, lay the soil samples out on clean, untreated jute (bags).

In addition, these additional guidelines also apply to the following matrices:

- (Soil) water: no filtration of samples on the site.

3 VOLATILE (ORGANIC) PARAMETERS

To determine volatile parameters (VOCs), extensive sampling may result in significant losses, such as due to prolonged contact with the air when taking multiple grabs, disruptions during grab homogenisation and division into laboratory sample(s), volatilisation and diffusion due to unsuitable packaging, etc.

When samples are taken as part of analyses for volatile (organic) parameters, the following general guidelines should be taken into account:

- During sampling, the container must be completely filled without headspace and this in one movement.
- Analyses for volatile (organic) parameters may not be carried out on mixed samples.
- A spot sample for volatile parameters often consists of a single grab. Indicate this on the sampling form and sampling report.
- However, if it is possible to take multiple grabs, pour them (directly) into the sampling container in layers.
- Do not perform any on-site mixing, homogenisation or division operations. If more than one laboratory sample is required, conduct a separate sampling operation for each laboratory sample.
- Refrigerate samples and transport them for handover to the analysis laboratory as quickly as possible.

In addition, these additional guidelines also apply to the following matrices:

- Soil:
  - Samples should be taken undisturbed using liners or canisters.
  - Sample cans using the Foil Sampler Set should not be used in the context of analyses for volatile (organic) parameters.
- Semi-solid soil materials:
  - Samples should be taken undisturbed using liners or canisters.
  - Sample cans using the Foil Sampler Set should not be used in the context of analyses for volatile (organic) parameters.
  - Because evaporation of volatile parameters may have already occurred from the top layer of a batch, you should remove this top layer first. For this, you scoop or drill to a depth of at least 25 cm and remove that material before taking the sample.
  - In case of sampling with a bulldozer, wheel loader,... take the spot sample as best as possible undisturbed, at the place where the wheel loader, bulldozer,... took away the material for a sub-batch. Take the undisturbed samples immediately after scooping them up with the wheel loader.
  - If you need to perform duplicate sampling (sample and sample for counteranalysis), take both undisturbed samples as close to each other as possible.
- Groundwater:
  - Sampling should not be carried out with the ball valve sampler.
  - Sampling must not be carried out from stand pipe piezometers with a intersecting filter, except in the case of intersecting stand pipe piezometers installed before 18 January 2012.
  - Turbulence and bubbling in the sample hoses must always be avoided. If turbulence or bubble formation occurs and the sampling rate can no longer be reduced (see guidelines CMA/1/A.2), this should be mentioned in the field report.
  - Set an adequately low sampling flow rate and and do not let it exceed 0.2 L/min.
  - Samples should not be filtered.
  - If air bubbles are still present in the container after sampling, this container must not be refilled but a new container must be filled.
- Surface water, drinking water, waste water:
  - Samples should not be filtered.
  - If air bubbles are still present in the container after sampling, this container must not be refilled but a new container must be filled.
- Water bottom, non-semisolid soil materials:

- Sampling is done by taking a separate spot sample.
- Sampling consists of a single grab, filling the container as quickly as possible, in a single operation, without headspace.
- Sample cans using the Foil Sampler Set should not be used in the context of analyses for volatile (organic) parameters.
- Record the spot sampling location and depth on the sampling form.

- Liquid materials:
  - For sampling, the vacuum pump is less suitable, due to the loss of volatile parameters.

4 HEAVY METALS

When samples are taken as part of analyses for heavy metals, these guidelines should be taken into account for the following matrices:

- Groundwater:
  - Sampling should not be carried out with the ball valve sampler.
  - Sampling must not be carried out from stand pipe piezometers with a intersecting filter, except in the case of intersecting stand pipe piezometers installed before 18 January 2012.
  - Turbulence and bubbling in the sample hoses must always be avoided. If turbulence or bubble formation occurs and the sampling rate can no longer be reduced (see guidelines CMA/1/A.2), this should be mentioned in the field report.
  - Set an adequately low sampling flow rate and and do not let it exceed 0.2 L/min.
  - Filtration should be done immediately in the field.

5 CYANIDES

Appropriate precautions should be taken during sampling to prevent decomposition of complex cyanides under the influence of light. The lab sample is therefore delivered in a light-proof container or stored in a dark environment (e.g. store in a cooler or wrap with aluminium foil).

In addition, these additional guidelines also apply to the following matrices:

- Groundwater: individual groundwater sample in the field filtered over 0.45 µm membrane filter for total and free CN.

Methods for determining anions

This procedure replaces procedure CMA/2/I/C of May 2024.
For guidelines on the preservation and treatment of water samples, please refer to CMA/1/B.

MATRIX: GROUNDWATER

The following analytical methods can be used to determine anions in groundwater:

Chloride (a)	• NBN EN ISO 10304-1:2009 Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007) (CMA/2/I/C.3) en ISO 10304-1:2007/Cor 1:2010 • NBN EN ISO 10304-4:1999 Water quality - Determination of dissolved anions using liquid chromatography of ions. Part 4: Determination of chlorate, chloride and chlorite in water with low contamination (ISO 10304-4:1997) • ISO 9297:1989 Water quality – Determination of chloride – Silver nitrate titration with chromate indicator (Mohr's method) • NBN EN ISO 15682:2001 Water quality – Determination of chloride by flow analysis (CFA and FIA) and photometric or potentiometric detection (ISO 15682: 2000) • NBN EN ISO 15923-1:2024 Water quality – Determinations of ions by a discrete analysis system and spectrophotometric detection – Part 1: Ammonium, chloride, nitrate, nitrite, orthophosphate, silicate and sulfate (ISO 15923-1:2013) (CMA/2/I/C.8)
Fluoride	• NBN EN ISO 10304-1:2009 Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007) (CMA/2/I/C.3) en ISO 10304-1:2007/Cor 1:2010 • ISO 10359-1:1992 Water quality – Determination of fluoride – Part 1: Electrochemical probe method for potable and light polluted water (b) (CMA/2/I/C.1.1) • NEN 6589:2005 Water - Potentimetric determination of total inorganic fluoride content with stream systems (FIA and CFA) • CMA/2/I/C.1.2 Spectrophotometric determination of dissolved fluoride with a stream analysis system
Nitrate	• NBN EN ISO 10304-1:2009 Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007) (CMA/2/I/C.3) en ISO 10304-1:2007/Cor 1:2010 • NBN EN ISO 13395:1996 Water quality – Determination of nitrite nitrogen and nitrate nitrogen and the sum of both by flow analyses (CFA en FIA) and spectrometric detection (ISO 13395:1996) (CMA/2/I/C.6) • ISO 7890-3:1988 Water quality – Determination of nitrate – Part 3: Spectrometric method using sulfosalicylic acid • NBN EN ISO 15923-1:2024 Water quality – Determinations of ions by a discrete analysis system and spectrophotometric detection – Part 1: Ammonium, chloride, nitrate, nitrite, orthophosphate, silicate and sulfate (ISO 15923-1:2013) (CMA/2/I/C.8

Nitrite	• NBN EN ISO 10304-1:2009 Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007) (CMA/2/I/C.3) en ISO 10304-1:2007/Cor 1:2010 • NBN EN ISO 13395:1996 Water quality – Determination of nitrite nitrogen and nitrate nitrogen and the sum of both by flow analyses (CFA en FIA) and spectrometric detection (ISO 13395:1996) (CMA/2/I/C.6) • NBN EN 26777:1993 Water quality – Determination of nitrite – Molecular absorption spectrometric method (ISO 6777:1984) • NBN EN ISO 15923-1:2024 Water quality – Determinations of ions by a discrete analysis system and spectrophotometric detection – Part 1: Ammonium, chloride, nitrate, nitrite, orthophosphate, silicate and sulfate (ISO 15923-1:2013) (CMA/2/I/C.8)
Sulphate	• NBN EN ISO 10304-1:2009 Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007) (CMA/2/I/C.3) en ISO 10304-1:2007/Cor 1:2010 • ISO 22743:2006 Water quality - Determination of sulfates - Method by continuous flow analysis (CFA) • NBN EN ISO 15923-1:2024 Water quality – Determinations of ions by a discrete analysis system and spectrophotometric detection – Part 1: Ammonium, chloride, nitrate, nitrite, orthophosphate, silicate and sulfate (ISO 15923-1:2013) (CMA/2/I/C.8
Total cyanide	• NBN EN ISO 14403-2:2012 Water quality – Determination of total cyanide and free cyanide by continuous flow analysis (ISO 14403-2:2012) (CMA/2/I/C.2.2) • ISO 6703-1:1984 Water quality – Determination of cyanide – Part 1: Determination of total cyanide (CMA/2/I/C.2.1)
Free cyanide	• NBN EN ISO 14403-2:2012 Water quality – Determination of total cyanide and free cyanide by continuous flow analysis (ISO 14403-2:2012) (CMA/2/I/C.2.3)
Chromium(VI)	• NBN EN ISO 10304-3:1997 Water quality - Determination of dissolved anions using liquid chromatography of ions. Part 3: Determination of chromate, iodide, sulfite, thiocyanate and thiosulfate (ISO 10304-3:1997) • EPA 218.6:1996 Determination of dissolved hexavalent chromium in drinking water, groundwater and industrial wastewater effluents by ion chromatography (CMA/2/I/C.7) • EPA 218.7:2011 Determination of hexavalent chromium in drinking water by ion chromatography with post-column derivatization and UV-Visible detection (CMA/2/I/C.7) • EPA 7199:1996 Determination of hexavalent chromium in drinking water, groundwater and industrial waste water effluents by ion chromatography (CMA/2/I/C.7) • ISO 11083:1994 Water quality – Determination of chromium(VI) – Spectrometric method using 1,5 diphenylcarbazide (c)

(a) For the determination of chloride, a total halogen content (chloride, bromide, iodide) shall always be determined when using the titrimetric method and the stream analysis method. Ion chromatography, on the other hand, is capable of selectively measuring chloride.

(b) For the determination of fluoride with ion-selective electrode according to ISO 10359-1, the use of the buffer as described in DIN 38405-D4 is recommended. The validation data

included in ISO 10359-1 are also determined on the basis of this buffer.

(c) When using the direct method without IC separation according to ISO 11083:1994, various interferences are possible. In the case of oxidising and/or reducing constituents, the pre-treatment procedure described in the standard should be followed.

MATRIX: ELUATES

The following methods of analysis can be used to determine anions in eluates:

Bromide	• NBN EN ISO 10304-1:2009 Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007) (CMA/2/I/C.3) en ISO 10304-1:2007/Cor 1:2010
Chloride (a)	• NBN EN ISO 10304-1:2009 Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007) (CMA/2/I/C.3) en ISO 10304-1:2007/Cor 1:2010 • NBN EN ISO 10304-4:1999 Water quality - Determination of dissolved anions using liquid chromatography of ions. Part 4: Determination of chlorate, chloride and chlorite in water with low contamination (ISO 10304-4:1997) • ISO 9297:1989 Water quality – Determination of chloride – Silver nitrate titration with chromate indicator (Mohr's method) • NBN EN ISO 15682:2001 Water quality – Determination of chloride by flow analysis (CFA and FIA) and photometric or potentiometric detection (ISO 15682: 2000) • NBN EN ISO 15923-1:2024 Water quality – Determinations of ions by a discrete analysis system and spectrophotometric detection – Part 1: Ammonium, chloride, nitrate, nitrite, orthophosphate, silicate and sulfate (ISO 15923-1:2013) (CMA/2/I/C.8)
Fluoride	• NBN EN ISO 10304-1:2009 Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007) (CMA/2/I/C.3) en ISO 10304-1:2007/Cor 1:2010 • ISO 10359-1:1992 Water quality – Determination of fluoride – Part 1: Electrochemical probe method for potable and light polluted water (b) (CMA/2/I/C.1.1) • NEN 6589:2005 Water - Potentimetric determination of total inorganic fluoride content with stream systems (FIA and CFA) • CMA/2/I/C.1.2 Spectrophotometric determination of dissolved fluoride with a stream analysis system
Nitrite	• NBN EN ISO 10304-1:2009 Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (CMA/2/I/C.3) and ISO 10304-1:2007/Cor 1:2010 • NBN EN ISO 13395:1996 Water quality – Determination of nitrite nitrogen and nitrate nitrogen and the sum of both by flow analyses (CFA and FIA) and spectrometric detection (CMA/2/I/C.6) • NBN EN 26777:1993 Water quality – Determination of nitrite – Molecular absorption spectrometric method (ISO 6777:1984) • NBN ISO 15923-1:2023 Water quality – Determinations of ions by a discrete analysis system and spectrophotometric detection – Part 1: Ammonium, chloride, nitrate, nitrite, orthophosphate, silicate and sulfate (ISO 15923- 1:2013) (CMA/2/I/C.8)
Sulphate	• BN EN ISO 10304-1:2009 Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (ISO 10304-1:2007) (CMA/2/I/C.3) en ISO 10304-1:2007/Cor 1:2010

	• ISO 22743:2006 Water quality - Determination of sulfates - Method by continuous flow analysis (CFA) • NBN EN ISO 15923-1:2024 Water quality – Determinations of ions by a discrete analysis system and spectrophotometric detection – Part 1: Ammonium, chloride, nitrate, nitrite, orthophosphate, silicate and sulfate (ISO 15923-1:2013) (CMA/2/I/C.8)
Total cyanide	• NBN EN ISO 14403-2:2012 Water quality – Determination of total cyanide and free cyanide by continuous flow analysis (ISO 14403-2:2012) (CMA/2/I/C.2.2) • ISO 6703-1:1984 Water quality – Determination of cyanide – Part 1: Determination of total cyanide (CMA/2/I/C.2.1)
Chromium (VI)	• NBN EN ISO 10304-3:1997 Water quality - Determination of dissolved anions using liquid chromatography of ions. Part 3: Determination of chromate, iodide, sulfite, thiocyanate and thiosulfate (ISO 10304-3:1997) • EPA 218.6:1996 Determination of dissolved hexavalent chromium in drinking water, groundwater and industrial wastewater effluents by ion chromatography (CMA/2/I/C.7) • EPA 218.7:2011 Determination of hexavalent chromium in drinking water by ion chromatography with post-column derivatization and UV-Visible detection (CMA/2/I/C.7) • EPA 7199:1996 Determination of hexavalent chromium in drinking water, groundwater and industrial waste water effluents by ion chromatography (CMA/2/I/C.7) • ISO 11083:1994 Water quality – Determination of chromium(VI) – Spectrometric method using 1,5 diphenylcarbazide (c)

(a) For the determination of chloride, a total halogen content (chloride, bromide, iodide) shall always be determined when using the titrimetric method and the stream analysis method. Ion chromatography, on the other hand, is capable of selectively measuring chloride.

(b) For the determination of fluoride with ion-selective electrode according to ISO 10359-1, the use of the buffer as described in DIN 38405-D4 is recommended. The validation data included in ISO 10359-1 are also determined on the basis of this buffer.

(c) When using the direct method without IC separation according to ISO 11083:1994, various interferences are possible. In the case of oxidising and/or reducing constituents, the pre-treatment procedure described in the standard should be followed.

Selected parameters by discrete analysis systems - ammonium, nitrate, nitrite, chloride, orthophosphate and sulfate

1 OBJECTIVE AND SCOPE

This procedure replaces procedure CMA/2/I/C.8 of May 2024. This procedure describes methods for the automatic conduct of spectrophotometric and turbidimetric analyses using a discrete analytical system for the determination of ammonium, chloride, nitrate, nitrite, orthophosphate and sulphate. The method is applicable to drinking water, groundwater, surface water, waste water and eluates.

The procedure described in NBN/ISO 15923-1:2024 applies subject to the following additions:

- § 5 Other reagents and concentrations may be used if adequate for this application.
- >§ 7 For the preservation and treatment of the samples, reference is made to CMA/1/B.
- Annex B v. H: H: deviations from carrying out the methods described are allowed as long as the procedure is based on the same principle as an existing EN or ISO standard and as long as the required performance characteristics are met;
- Paragraphs 8.1 and 8.2: Additional quality control for the determination of parameters ammonium, nitrate and nitrite in eluates and leaching. When analysing these samples, at least one of the following quality checks must be carried out:
  - Analysis of the sample with a minimum of 1 grading, the bias vs the theoretical value may not exceed 10%
  - At least two measurements of the same sample, the dilution factor of which differs by at least a factor of 2, resulting in two measurement results within the measuring range which differ no more than 10 % from each other

Notice: When determining the ammonium content, false negative results may occur at high concentrations. The quality controls stated above are intended to overcome this.

Notice: Ammonium and nitrate may be determined in extracts from samples which may be used in or as fertilisers or soil improvers. These samples should normally be heavily diluted to eliminate matrix interference.

2 REFERENCES

- NBN EN ISO 15923-1:2024 Water quality -- Determination of selected parameters by discrete analysis systems -- Part 1: Ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection (ISO 15923-1:2013)(CMA/2/I/C.8)
- NEN 6604:2007 Water - Determination of the content of ammonium, nitrate, nitrite, chloride, orthophosphate, sulphate and silicate with discrete analysis system and spectrophotometric detection.
  - C. Vanhoof, A. Cluyts, E. Poelmans, W. Wouters en K. Tirez, *Evaluatie discrete analyser voor de analyse van uitlogingen/destructies van afvalmonsters*, Vito rapport 2012/MANT/R/03, https://esites.vito.be/sites/reflabos/onderzoeksrapporten/Online%20documenten/2011_Discrete_analyser_OVAM_final.pdf.

Clay content (Robinson/Köhn pipette method)

1 PRINCIPLE

This procedure replaces the procedure CMA/2/A.6 of May 2024.
Determine the clay content by conducting a partial texture analysis. Texture analysis involves separating the mineral soil into particle size fractions, specifically sand, loam and clay, as well as determining the proportions of the fractions. Conduct the analysis on the fine soil (< 2 mm), after separation of coarse elements. To obtain proper dispersion of the clay fraction, remove all cementing materials such as organic matter, carbonates and dissolved salts.
Separate the fine fractions (loam and clay) from the sand by wet screening with a 50µm or 63µm sieve. Determine the clay content using a Robinson/Köhn pipette after dispersing the colloidal fraction with a dispersant. The pipetting time and depth (10 cm) are derived from Stokes' law.

The procedure described in ISO 11277:2020 and ISO 11277:2020/Amd 1:2024 applies subject to the following additions/amendments.

2 SUPPLEMENTS TO ISO 11277:2020

- § 7 Sample pretreatment: Sample preservation is described in CMA/1/By, and sample pre-treatment in CMA/5/B.4. The analysis is performed on the sample dried at max. 40°C and sieve over a sieve with mesh size 2 mm.
- §9.2.2. Insertion depth: The uptake of clay is based on the relationship between the sedimentation rate and the size of the particles and may occur at a varying insertion depth (10 ±0.5 cm) at a predetermined time and a specified temperature or at a fixed insertion depth (10 ±0.1 cm) at a varying time and a specified temperature (see Table 3 in the ISO standard).
- §9.2.10 Drying oven set at a temperature of 105 °C ± 5 °C
- §9.6 Removal of organic matter (obligatory)
  - Washing until conductivity < 40 mS/m is optional. Perform a washing step after removing the carbonates, i.e. before the dispersion step.
  - After removing the organic matter, the above liquid should not be removed before removing the carbonates.
- §9.7 Removal of salts and gypsum is combined with § 9.8 Removal of carbonates.
- §9.8 Removal of carbonates (if present)
  - The presence of carbonates can be tested by adding a few drops of HCl to a small amount of the sieved sample.
  - Wash until conductivity < 40 mS/m.
- §9.9 Removal of iron oxide: not applicable
- §9.11 Wet screening at 63 µm: screening at 50 µm is also permitted.

Notice: In the context of 'soil cleanlinability', after sieving over 63 µm, the mass fraction shall be immediately determined relative to the fraction <2 mm.

3 REFERENCE

- ISO 11277:2020: Soil quality – Determination of particle size distribution in mineral soil material – Method by sieving and sedimentation
- ISO 11277:2020/Amd 1:2024 Soil quality — Determination of particle size distribution in mineral soil material — Method by sieving and sedimentation — Amendment 1

Cyanides in soil, water soil and solid waste

1 APPLICATION

This procedure is new.
This procedure describes the determination of free and total cyanides in soil, water soil and solid waste using continuous stream analysis method, after alkaline extraction in 1M NaOH.

> Notice: The procedure for the determination of cyanides in solid materials was previously included in CMA/2/I/C.2.2 and CMA/2/I/C.2.3. Without adjustments to the method, a division was made between solid and aqueous matrices. CMA/2/II/A.25 describes the determination of free and total cyanides in soil, sediment and solid waste. CMA/2/I/C.2.2 and CMA/2/I/C.2.3 describes the determination of free and total cyanides in water and eluates.

For the determination of free cyanide in soil and solid/paste waste, a reporting limit of 1 mg/kg ds shall be used.

The non-chlorooxideable cyanides are calculated from the difference between total cyanides and free cyanides. If the free cyanides <1 mg/kg ds, they are equated to 0 (lower bound approach) in the difference calculation.

Chloroxydeable cyanides are assimilated to free cyanides.

2 SAMPLE PRETREATMENT

CMA/1/B covers sample preservation and CMA/5/B.1 covers sample pretreatment.

3 PROCEDURE

3.1 EXTRACTION PROCEDURE

Weigh, to the nearest 0.1 g, 10 g of the original homogenised sample in a suitable container. Add 100 ml of NaOH 1M, shielded from light and shake for 16 hours.
Filter through a black band filter and store in dark glass bottles.

> Comment 1: In the case of highly wet samples, more sample may be taken with a maximum of 10 g of dry matter.

> Comment 2: As an alternative to filtration, a centrifugation step may be carried out.

3.2 ANALYTICAL DETERMINATION OF FREE AND TOTAL CYANIDES IN THE EXTRACT

For the analytical measurement, the extract is diluted 10 times with ultra pure water. Additional dilutions are performed with 0.01M NaOH.

The analytical determination of total cyanides in the extract is described in CMA/2/I/C.2.2.

The analytical determination of free cyanides in the extract is described in CMA/2/I/C.2.3.

3.3 CALCULATION OF FREE AND TOTAL CYANIDE CONCENTRATION IN THE SAMPLE

Calculate the concentrations of free and/or total cyanides in the dried sample, expressed in mg/kg ds, as follows:

$$C_{CN} = C_x \, x \, f_1 \, x \, f_2 \, x \, \frac{V + \left(\frac{100 - DS \, x \, m}{100} \right)}{1000 \, x \, m} \, x \, \frac{100}{DS}$$

By which:

C_{CN} cyanide concentration of the dried sample in mg/kg ds C_x cyanide concentration in the diluted extract, µg/l
f_1 initial dilution factor (=10)
f_2 additional dilution factor
V volume of the NaOH extraction solution, in ml (= 100 ml)
DS dry matter content of the sample expressed in % by weight (determined according to CMA/2/II/A.1)
m mass of original sample, expressed in grams

4 REFERENCE

- NBN EN ISO 14403-2:2012 Water quality – Determination of total cyanide and free cyanide by continuous flow analysis (ISO 14403-2:2012)
- NBN EN ISO 17380: 2013 Soil quality – Determination of total cyanide and easily liberatable cyanide – Continuous-flow analysis method

Leaching of per- and polyfluoroalkyl compounds (PFAS) from soil, soil materials, water-bearing layer and granulates with the single shake test

CONTENTS

1 OBJECTIVE AND SCOPE

This procedure is new and contains guidelines for determining the leachability of PFAS using a single shake test with a liquid-to-solids ratio (L/S) of 10 L/kg ds.
The method applies to field humid 1) soil samples and semisolid batches of soil material, 2) water soils and 3) granulates, incl. strained sand in self-monitoring under the unitary regulation for recycled granulates (Unified Regulation (EHR) recycled granulates)
The test material has a grain size less than or equal to 4 mm (with or without reduction). The result of this determination is the release of components with representation of the concentration of PFAS in the eluate (in ng/L) and/or the leached amount of the component relative to the total mass of the analytical sample (in µg/kg ds) determined according to the experimental test conditions, described in the sections below.

2 PRINCIPLE

The test material, with a particle size less than or equal to 4 mm (with or without particle size reduction) is placed in contact with the leaching solution (see further §5) for 24 hours and shaken. The method is based on the assumption that an equilibrium (or near equilibrium) state is reached between the liquid and solid phase during this test period. After 24 hours, the solid residue is separated by centrifugation. The concentrations of the leached components in the centrifuged supernatant (eluate) are determined according to the methods described in WAC/IV/A/025 (Determination of per- and polyfluoroalkyl compounds (PFAS) in water with LC-MS/MS). After the test, the leaching conditions are recorded by measuring the pH, conductivity and temperature of the eluate (redox potential is optional).

3 REMARKS

As regards the determination of the leachability of soil containing PFASs and soil materials, it should be borne in mind that during the entire process of the shake test (including sample preparation and analysis), there is an increased risk of release of PFAS from materials-containing PFAS materials, as well as adsorption of mainly long-chain PFAS to materials and containers. The following guidelines should therefore be observed:

- During the shake test, the number of containers and their transfer must be kept to the minimum. The more containers are handled, the greater the chance of adsorption and the lower the recovery of the long chains.
- In addition to the use of blanks (see § 7.3), it is also necessary to pre-test the possible release of PFAS from materials used during the conduct of the shake-up test[1] for the presence and/or possible release of PFAS.

[1] To the extent possible, it should be verified (with the supplier by certificate/analysis) whether the material used in the shake test may contain PFAS.

- Furthermore, any direct contact with both the solid fraction and the eluate, even when wearing gloves, should be avoided.
- Cleaning of material must only be done with demineralised water or ultra pure water. When applying the cleaning product, only use PFAS-free detergents, and rinse thoroughly afterwards.

4 EQUIPMENT AND MATERIALS

4.1 Shake containers with a capacity of 250 ml and eluate containers: Lockable HDPE, PP or borosilicate glasses, discharge containers with a capacity of 250 ml. The glass bottles must have a closure with PP or HDPE insert.
4.2 Overhead mixer (active at 5 revolutions/min) or roller dynamometer which induces a speed of the leaching containers of 10 revolutions/min. Centrifuge at a frequency of 3,000 – 4,000 rcf.
4.3 (Analytical) balance with a measurement accuracy better than ± 0,1 g
4.4 pH meter with a measurement accuracy better than + 0.05 pH units
4.5 Conductivity meter with a measurement accuracy of better than 5 µS/cm
4.6 Redox potential meter (optional)
4.7 Measuring cylinders for volume determination, with a measurement accuracy of 1 %
4.8 Thermometers for ambient temperature and temperature of the eluates

5 LEACHING FLUID

- For soil, soil materials and water bottom: $CaCl_2$ solution (0.01 M)

  Comment 1: Each batch of concentrated $CaCl_2$ should be analysed for the presence of PFAS components (target analysis according to WAC/IV/A/025) prior to the preparation of $CaCl_2$ (0.01M).

- For granulates incl. sieve sand: Ultra pure water: demineralised water, distilled water or water of the same purity (5 < pH < 7), with a conductivity not exceeding 5 µS/cm.

6 STEEL TREATMENT

6.1 SAMPLE PREPARATION

For steel preparation, see CMA/2/II/A.19 §6.2.

6.2 DETERMINATION OF DRY MATTER CONTENT (%) AND RELATIVE MOISTURE CONTENT (%)

The specimen obtained after 6.1 shall not be further dried.
The dry matter content dm of the specimen must be determined on a separate subsample dried at (105 + 5)°C according to CMA 2/II/A.1 (dry residue).
The relative moisture content rh (%) is the ratio between the mass of water present in the material and the mass of the corresponding dried material. The relative moisture content is taken into account when determining the L/S ratio. The calculation of the relative moisture content rh, expressed in %, is as follows:

(1)

$$relative\ humidity\ rv = \frac{m_b - m_c}{\ldots} x\ 100(\%)$$

with
m_a = mass of the empty container in g
m_b = mass of the container with wet sample, in g
m_c = mass of the container with dried sample, in g

6.3 ANALYSIS PORTION

For the single shake test to be carried out in singular, an analysis portion shall be taken from the test specimen obtained after §6.2 with a wet mass in accordance with 0.020 kg ± 0.005 kg of dry matter, with known dry matter content and relative moisture content rh (§6.1), and whose particle size is less than 4 mm.

7 PROCEDURE

7.1 PERFORMANCE OF THE SINGLE SHAKE TEST

The single shake test is performed at room temperature (20 ± 5 °C).

7.1.1 PREPARATION

According to formula (2), weigh a quantity of wet analytical sample m_w equal to 0.020 kg ± 0.005 kg dry matter (m_D). The analytical sample is obtained by dividing or quartering.

(2)

$$m_w = \frac{m\,D}{DW} x\ 100$$

where:
ds = dry matter content (%)
m_D = mass of required sample quantity expressed as dry matter (kg dm) m_w = corresponding mass of the wet sample to be weighed (kg)

Transfer this test sample quantitatively into a 250 ml container.

> Notice: The volume of 250 ml was chosen in combination with the mass of 20 g ds to minimise the free space (amount of air) in the bottle above the leaching liquid. Any deviations from this must be reported. However, one should always try to keep the free space in the bottle limited.

7.1.2 LEACHING STEP

According to formula (3), add a quantity of leaching liquid L so that a liquid-to-solid ratio (L/S) equal to 10 l/kg ds ± 2% is achieved during extraction. Record the volume of leaching solution added and the corresponding L/S ratio. Ensure that good mixing of solids and liquids is achieved.

> Comment 2: Proper mixing prior to mechanical shaking is essential to ensure that the solids are homogeneously distributed throughout the leaching solution and no clumps are present. Premixing should be done manually using circular swivelling movements with the container.

(3)

$$L = 10 - \left(\frac{rv}{100} \right) x\, m$$

where:
L = volume of leaching liquid (l)
m_D = mass of dry matter of the sample (kg)
rv = relative humidity (%)

Immediately place the sealed bottle in a shaker (4.2). Record the start time. Shake for 24 hours ± 0.5 hours at a shaking speed equal to 5 revolutions/min (overhead mixer) or 10 revolutions/min (roller dynamometer). This should avoid sedimentation of the solid at the bottom of the bottle during extraction. Record the temperature during leaching.

7.1.3 LIQUID-SOLID SEPARATION

Stop the shaker after 24 hours ± 0.5 hours. Note the end time of shaking.

Immediately transfer the recipient into a centrifuge. Then centrifuge for 2x15 min at at least 3 000 rcf.
Decant the clear eluate immediately into appropriate containers (cf. guidelines CMA/1/B preservation and storage) for 1) determination of pH and conductivity and 2) delivery to the laboratory for further analysis. The recipients for delivery to the laboratory must be completely filled up.

Comment 3: If there is a floating layer or floating material on top of the clear eluate, the clear eluate must be transferred by pipette into the containers that will be delivered to the lab. This additional action should be included in the report.

Comment 4: If no analysis can be carried out due to turbidity, this should be explicitly reported. No additional filtration or ultracentrifugation may be carried out.

7.2 ANALYSIS:

Immediately measure the pH (± 0.1 pH unit) and temperature of the eluate (see §7.1.3) according to method CMA/2/I/A.1. Record the pH value and temperature.

Measure the conductivity (± 1%) of the eluate according to method CMA/2/I/A.2.

Determine the concentration of the components to be followed in the eluate in accordance with the methods described in the procedure WAC/IV/A/025 (Determination of per- and polyfluoroalkyl compounds (PFAS) in water with LC-MS/MS).

7.3 BLANK TEST

A procedure blank must be included for each batch of shake tests carried out. A volume of leaching fluid of 250 ml is subjected to the full test method, including the eluate analysis and the determination of conductivity, but with the exception of the screening, reduction and distribution step.

For none of the PFAS components analysed, the concentration in the eluate of the blank should be greater than or equal to the reporting limit. The results of the blank test shall not be deducted from the results of the leaching test. If violations of the reporting limits are observed for the blanks, the cause of these violations should be identified and the need for re-running the shake test should be ascertained.

8 CALCULATIONS

The analysis of the eluate gives the concentration of the PFAS components in the eluate, in ng/L. Leached quantities are calculated using the following formula 4:

(4)

$$E_{es} = c\ x\left(\frac{L}{m\ D} + \frac{rv}{100}\right)$$

where:

E_{es} = the leached amount of a component in the single shake test at
L/S = 10 (in µg/kg dry matter)
c = the concentration of that component in the eluate (µg/l), allow for possible dilution at higher conductivities
L = the volume of leaching fluid added (l)
rv = the relative moisture content (in % of dry mass, as calculated by Equation 2)
m_D = the mass of the analytical sample, expressed in dry matter (kg ds)

The concentration c referred to in formula (4) is the concentration originally present in the eluate converted to µg/l.

9 QUALITY CONTROL

- In the event of a change of materials, a new evaluation should be carried out regarding the possible presence/release of PFAS (see § 3)
- Water quality in accordance with the requirements of § 5.
- Temperature of the laboratory as per requirement in §7.
- For each measurement series (= measurement day), a procedure blank according to 7.3 is performed.

10 REPORT

The report must contain at least the information below:

- Reference to relevant CMA procedure. If the single shake test for the determination of PFAS leaching has not been carried out in full compliance, all deviations should be justified otherwise reference should not be made; Note 5: Problems with clarity should also be reported and if possible illustrated by photographs.
- Date of implementation of the test;
- Liquid-to-solid ratio in the test;
- the pH of the eluate;
- Conductivity of the eluate;

- Temperature of the eluate;
- Analysed components in the eluate and corresponding determination limits;
- Measured concentrations of the components examined in the eluate at a maximum of 3 significant figures.
- Include additional information:
  - Indicate whether an overhead mixer or a roller bench was used.
  - Indicated leaching liquid sieve sand: ultrapure or demineralised water.

Sulphur and halogens

1 OBJECTIVE AND SCOPE

This procedure replaces the procedure CMA/2/B.2 of May 2024.
Sulfur and halogens (fluorine, chlorine, bromine and iodine) can be found in waste in various forms. Oxygen combustion can be used to render the waste and dissolve the elements to be determined.

For the determination of halogens and sulphur, the following European standards shall apply:

- NBN EN 14582: 2016 Characterisation of waste – halogen and sulfur content – Oxygen combustion in closed systems and determination methods
- NBN EN 17813: 2023 Environmental solid matrices – Determination of halogens and sulfur by oxidative pyrohydrolytic combustion followed by ion chromatography

In addition, the following methods may be used for specific matrices:

- S determination of solids: digestion according to CMA/2/II/A.3 and analytical determination using ICP- AES according to CMA/2/I/B.1
- S determination in wood and shredder polymer: 10 g, sacrifice, 450 °C, 6 hours, CMA/2/II/A.3 digestion and analytical determination with ICP-AES according to CMA/2/I/B.1

For the determination of F in difficult combustible materials (e.g. fly ash), hydropyrolysis according to CMA/2/II/B.1 or oxidative pyrohydrolytic combustion followed by ion chromatography according to NBN EN 17183 should be applied. This applies to package A.5.1 *Landfills*.

The determination of sulphur and halogens in oil following NBN EN 14582 is described in CMA/III/D.

2 NBN EN 14582: 2016

NBN EN 14582 describes a combustion method for the determination of halogenated and sulphur content in materials by combustion in a closed system with oxygen (calorimetric bomb), and the subsequent analysis of the combustion product using different analytical techniques.

This method is applicable to both solid and liquid waste. Only the organic and inorganically bound sulphur and halogens that are converted by combustion to fluoride, chloride, bromide, iodide and sulfate, and absorb or dissolve in the aqueous solution can be determined. The determination of the halogens and sulphur present can be carried out using various analytical techniques such as:

- CMA/2/I/B.1 Metals with inductively coupled plasma - atomic spectrometry (ICP-AES)
- CMA/2/I/C Methods for the determination of anions

The detection limit depends on the analytical technique used.

The procedure described in NBN EN 14582:2016 applies subject to the following additions:

- §8: Sample storage and pre-treatment of the test portion:
  - The preservation and treatment of samples is described in CMA/1/B.
  - A description of the sample pre-treatment can be found in CMA/5/A.1 to A.9, and in function of the matrix, the monter pretreatment is described in CMA/5/B.1 t.e.m. CMA/5/B.7.

3 NBN EN 17813: 2023

NBN EN 17813 describes oxidative pyrohydrolytic combustion followed by ion chromatography.
The homogenised sample is burned under oxidative conditions. For the determination of fluorine, combustion is carried out under pyrohydrolytic conditions. The combustion gases are absorbed in an aqueous solution. For the determination of sulphur, the absorption solution contains an oxidising agent to ensure full omzetting to sulphate. Changes in the volume of the absorption solution are included in the concentration calculations. The anions (bromide, chloride, fluoride and sulphate) are separated by ion chromatography and detected by a conductivity detector.

The procedure described in NBN EN 17813:2023 applies subject to the following additions:

- §8: Sample storage and pre-treatment of the test portion:
    - The preservation and treatment of samples is described in CMA/1/B.
    - A description of the sample pre-treatment can be found in CMA/5/A.1 to A.9, and in function of the matrix, the monter pretreatment is described in CMA/5/B.1 t.e.m. CMA/5/B.7.

4 REFERENCE

- NBN EN 14582:2016 Characterization of waste - Halogen and sulfur content - Oxygen combustion in closed systems and determination methods.
- NBN EN 17813:2023 Environmental solid matrices – Determination of halogens and sulfur by oxidative pyrohydrolytic combustion followed by ion chromatography.
- C. Vanhoof, Groep AN, V. Corthouts en K. Tirez, *Bepaling van halogenen en zwavel in vaste stoffen*, VITO rapport 2004/MIM/R/07, January 2004, https://esites.vito.be/sites/reflabos/onderzoeksrapporten/Online%20documenten/rapport_halogenen_2004.pdf

Sulphur and halogens

1 OBJECTIVE AND SCOPE

This procedure replaces the procedure CMA/2/III/C of October 2018.
Oxygen combustion can be used to render oil samples and dissolve the elements to be determined.
For the determination of halogens and sulphur, the following European standards shall apply:

- NBN EN 14582: 2016 Characterisation of waste – halogen and sulfur content – Oxygen combustion in closed systems and determination methods
- NBN EN 17813: 2023 Environmental solid matrices – Determination of halogens and sulfur by oxidative pyrohydrolytic combustion followed by ion chromatography

2 PRINCIPLE

NBN EN 14582 describes a combustion method for the determination of halogenated and sulphur content in materials by combustion in a closed system with oxygen (calorimetric bomb), and the subsequent analysis of the combustion product using different analytical techniques.

NBN EN 17813 describes oxidative pyrohydrolytic combustion followed by ion chromatography. The homogenised sample is burned under oxidative conditions. For the determination of fluorine, combustion is carried out under pyrohydrolytic conditions. The combustion gases are absorbed in an aqueous solution. For the determination of sulphur, the absorption solution contains an oxidising agent to ensure full omzetting to sulphate. Changes in the volume of the absorption solution are included in the concentration calculations. The anions (bromide, chloride, fluoride and sulphate) are separated by ion chromatography and detected by a conductivity detector.

3 REMARKS

Oil samples can have high viscosity and are difficult to mix in some cases. Sub-sampling should be carried out after thorough mixing of the laboratory sample. Viscous samples can be heated at 40 °C in an ultrasonic bath. After homogenisation, a subsample should be taken immediately for analysis.

4 ANALYSIS PROCESS

The samples are deconstructed and analysed as described in the compendium method CMA/2/II/B.2.

5 REFERENCE

- NBN EN 14582: 2016 Characterization of waste – Halogen and sulfur content – Oxygen combustion in closed systems and determination methods.
- NBN EN 17813:2023 Environmental solid matrices – Determination of halogens and sulfur by oxidative pyrohydrolytic combustion followed by ion chromatography
- C. Vanhoof, Groep AN, V. Corthouts en K. Tirez, *Bepaling van halogenen en zwavel in vaste stoffen*, VITO rapport 2004/MIM/R/07, January 2004, https://esites.vito.be/sites/reflabos/onderzoeksrapporten/Online%20documenten/rapport_halogenen_2004.pdf
- CMA/2/II/B.2 Determination of sulphur and halogens by oxygen combustion in closed bomb.

Ammonium nitrogen and nitrate nitrogen

This procedure replaces procedure CMA/2/IV/7 of June 2024 .

1 SCOPE

The ammonium and nitrate nitrogen content is determined in various extracts (CMA/2/IV/6) and/or rendering solutions (CMA/2/IV/4).

Notice: Liquid samples with a dry matter content of < 2 % are treated as waste water. The methods for the determination of ammonium N and nitraat-N are described in the Compendium for water sampling, measurement and analysis WAC/III/E and WAC/III/C respectively.

The following analytical methods may be used for the determination of ammonium and nitrate in extracts and rendering solutions:

Ammonium	• SO 7150-1:1984 Water quality – Determination of ammonium – Part 1: Manual spectrometric method (CMA/2/I/E.1) • ISO 11732:2005 Water quality – Determination of ammonium nitrogen – Method by flow analysis (CFA and FIA) and spectrometric detection (CMA/2/I/E.2) • ISO 5664: 1984 Water quality – Determination of ammonium- Distillation and titration method (CMA/2/I/E.3) • ISO 14911:1998 Water quality – Determination of dissolved Li^+, Na^+, NH4+, K^+, Mn^{2+}, Ca^{2+}, Mg^{2+}, Sr^{2+} and Ba^{2+} using ion chromatography – Method for water and waste water (CMA/2/I/E.4) • NBN EN ISO 15923-1:2024 Water quality -- Determination of selected parameters by discrete analysis systems -- Part 1: Ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection (ISO 15923-1:2013)(CMA/2/I/C.8) • ISO 10304-1:2007 Water quality - Determination of dissolved anions by liquid chromatography of ions - Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate (CMA/2/I/C.3) • ISO 13395:1996 Water quality – Determination of nitrite nitrogen and nitrate nitrogen and the sum of both by flow analyses (CFA and FIA) and spectrometric detection (CMA/2/I/C.6)
Nitrate	• SO 7890-3: 1988 Water quality – Determination of nitrate – Part 3: Spectrometric method using sulfosalicylic acid • NBN EN ISO 15923-1:2024 Water quality -- Determination of selected parameters by discrete analysis systems -- Part 1: Ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection (ISO 15923-1:2013)(CMA/2/I/C.8

Notice: The nitrate content may be considered as the total oxidised nitrogen (TON) content.

For the determination of ammonium in solid and paste soil improvers, including compost, the following checks shall be taken into account:

- Procedure for blanks: during drying of the sample with tartaric acid, a blank procedure is taken from each batch (25 grams of white sand + 30 ml tartaric acid (c (C4H6O6) = 0.445 mol/l)). The concentration of ammonia N in fresh material, expressed as kg N/1000 kg, shall be calculated as follows:

$$C N = \frac{C i \, x \, V ext}{m} \, x \, D$$

where:

C_N concentration of ammonia N in fresh material expressed as kg N/1000 kg

C_i concentration of ammonium in the extract after blank correction, in mg N/l

m mass of sample extracted, in g (i.e. 5 g, see CMA/2/IV/6 paragraph 5.7)

V_{ext} volume of extraction solvent, in l (i.e. 0.05 ml, see CMA/2/IV/6 point 5.7)

D Dry factor determined according to CMA/5/B.1

As a guide, ammonium content for the blank procedure is < 0.1 kg N/1000 kg. (see CMA/5/B.1 § 4.1.1)

- Blank KCl solution: this is the extraction solution (see CMA/2/IV/6)
- QAQC 1STE line inspection sample for Approval Package A.2.1 Fertiliser/Soil improver – Inorganic parameters (see CMA/6/D)

2 CALCULATIONS

2.1 NITRATE IN AQUEOUS EXTRACT FROM SOLID AND PASTE SAMPLES (INCLUDING COMPOST)

The result is expressed as nitrogen concentration C_N (mg/l) in fresh material using the following formula.

$$C N = \frac{C i \, x \, V ext}{V sample}$$

with

C_N concentration of nitrate-N in fresh material expressed as mg NO_3— N/l

C_i nitrate-N concentration in the extract after blank correctionm in mg N/l

V_{sample} volume of sample extracted, in ml (i.e. 50 ml, see CMA/2/IV/6 section 5.1)

V_{ext}volume of extraction solvent, in ml (i.e. 250 ml, see CMA/2/IV/6 section 5.1)

2.2 AMMONIUM-N 1M KCL EXTRACT FROM SOLID AND PASTE SAMPLES (INCLUDING COMPOST)

The result is expressed as nitrogen concentration C_N (mg/l) in fresh material using the following formula.

$$C N = \frac{C i \, x \, V ext}{m} \, x \, VD \, VM \, x \, D$$

where:

C_N concentration of ammonia N in fresh material expressed as mg NH_4— N/l

C_i concentration of ammonium-N in the extract after blank correction, in mg N/l

m mass of sample extracted, in g (i.e. 5 g, see CMA/2/IV/6 paragraph 5.7)

V_{EXT} volume of extraction solvent, in ml (i.e. 50 ml, see CMA/2/IV/6 point 5.7)

VD_{VM} volume density based on fresh material, in kg/l (CMA/2/IV/24)

D Dry factor determined according to CMA/5/B.1

2.3 AMMONIUM AND NITRATE IN AQUEOUS EXTRACT OF LIQUID AND AQUEOUS PASTE SAMPLES

The result is expressed as nitrogen concentration C_N (mg/l) in fresh material using the following formula.

$CN = \frac{Ci \times V_{ext}}{m_{ust}} \times VD_{VM}$ where:

C_N concentration of ammonia N or nitraat-N in fresh material, expressed as mg NH_4— N/l or mg NO_3— N/l

C_i concentration of ammonia N or nitrate-N in the extract, in mg N/l

must mass of sample extracted, in g (see CMA/2/IV/6 paragraph 5.8)

V_{ext} volume of extraction solvent, in ml (see CMA/2/IV/6 paragraph 5.8)

VD_{VM} gravimetric bulk density based on fresh material, in kg/l (CMA/2/IV/24)

3 NITRIC NITROGEN TO AMMONIACAL NITROGEN RATIO

The ratio is calculated by dividing the nitrate nitrogen content by the ammoniacal nitrogen content.

Germinable seeds

1 OBJECTIVE AND SCOPE

This procedure replaces procedure CMA/2/IV/10 of September 2011.
This procedure applies to all matrix types except liquid samples with a dry matter content of < 2%.

CMA/1/B covers sample preservation and CMA/5/B.1 covers sample pretreatment.

2 SOLID AND PASTELIKE (SEMISOLID) SAMPLES

500 ml of fresh analytical material is mixed with 2000 ml of peat, moistened and placed in a flat dish as a 2-3 cm layer. This scale is placed in a room where the relative humidity is close to 100%, at an illuminance of at least 2000 Lux for 12 to 16 hours a day and at a temperature of 18 - 30°C without exposure to direct sunlight.

For 14 days, the emergence of any germinating seeds is monitored.

3 PASTY (NON-STICKY) AND LIQUID SAMPLES

Plastic trays of approximately 20 cm by 50 cm (preferably fitted with an appropriate lid) are used for the research on germinable seeds.
A paper is spread on the bottom of the tray to prevent the water from draining too quickly.
Above, a layer of approximately 5 cm of coarse sand is spread.
500 ml of fresh analytical material to be examined is then applied to the sand layer.
The scale is placed in a room at an illumination strength of at least 2000 Lux for 12 to 16 hours per day and at a temperature of 18-30 °C without exposure to direct sunlight. Additional humidification may be necessary during the test.

For 14 days, the emergence of any germinating seeds is monitored.

4 CONTROL SAMPLES

The following checks are provided for:

- As a blank inspection, the same test is run without the addition of the analytical sample. This check is carried out with each batch/batch.
- As a (positive) inspection, at least 10 cress seeds are placed on top of the inspection sample and germination must exceed 90%. This check is carried out weekly.

5 REFERENCES

- Practical study on the agronomic added value of fermented manure over unfermented manure, M. Melai, T. Verstraten and P. van de Weijer, 's-Hertogenbosch, April 2004 (www.novem.nl).

Per- and polyfluoroalkyl compounds (PFAS) in soil and sediment

CONTENTS

1 OBJECTIVE AND SCOPE

This procedure replaces procedure CMA/3/D of January 2025 and describes the quantitative determination of per- and polyfluoroalkyl compounds in soil and sediment using liquid chromatography. The method can also be used for the determination of PFAS in inorganic waste. For the determination of PFAS in groundwater, see WAC/IV/A/025.
The compounds are widely used for the water and dirt repellent of textiles (including carpets), leather, paper and cardboard and in fire extinguishers. These compounds are harmful with long-term exposure, are persistent and bioaccumulate. The main of these compounds are perfluoral acids (e.g. PFOA), perfluoralkane sulphonic acids (e.g. PFOS) and amides and fluortelomer compounds. The latter compounds are also considered precursors for the first.

This method describes the extraction and analysis of the following compounds: Table 1: quantitative PFAS

PFAS	*Abbreviation*	*CAS no.*
perfluoro-n-butanoic acid	PFBA	375-22-4
perfluoro-n-pentanic acid	PFPeA	2706-90-3
perfluoro-n-hexanoic acid	PFHxA	307-24-4
perfluoro-n-heptanoic acid	PFHpA	375-85-9
perfluoro-n-octanic acid	PFOA	335-67-1
perfluoro-n-nonanoic acid	PFNA	375-95-1
perfluoro-n-decanoic acid	PFDA	335-76-2
perfluoro-n-undecanoic acid	PFUnDA	2058-94-8
perfluoro-n-dodecanoic acid	PFDoDA	307-55-1
perfluoro-n-tridecanoic acid	PFTrDA	72629-94-8
perfluoro-n-tetradecanoic acid	PFTeDA	376-06-7
perfluoro-n-hexadecanoic acid	PFHxDA	67905-19-5
perfluoro-n-butanesulphonic acid	PFBS	375-73-5
Perfluoro-n-pentanesulphonic acid	PFPeS	2706-91-4
perfluorohexane sulphonic acid	PFHxS	355-46-4
perfluoroheptane sulphonic acid	PFHpS	375-92-8
perfluoro-n-octanesulphonic acid	PFOS	1763-23-1
perfluoro-n-nonanesulphonic acid	PFNS	68259-12-1
perfluoro-n-decanansulfonic acid	PFDS	335-77-3
perfluoro-n-dodecanesulfonic acid	PFDoDS	79780-39-5
perfluorooctane sulphonamide	PFOSA	754-91-6
N-methylperfluoro-n-octan sulphonamide	MePFOSA	31506-32-8
N-ethylperfluoro-n-octan sulphonamide	EtPFOSA	4151-50-2
N-methylperfluoro-n-octane sulphonamidoacetic acid	MePFOSAA	2355-31-9
N-ethylperfluoro-n-octan sulphonamidoacetic acid	EtPFOSAA	2991-50-6
4:2 fluortelomer sulphonic acid	4:2 FTS	757124-72-4
6:2 fluortelomer sulphonic acid	6:2 FTS	27619-97-2
8:2 fluortelomer sulphonic acid	8:2 FTS	39108-34-4
8:2 fluorotelomer phosphate diester	8:2 diPAP	678-41-1

10:2 fluortelomer sulphonic acid	10:2 FTS	120226-60-0
perfluoro-2-propoxypropanoic acid	HFPO-DA	13252-13-6
4,8-dioxa-3H-perfluorononanoic acid	DONA	919005-14-4
Perfluoro-4-ethylcyclohexane sulphonic acid	PFECHS	646-83-3
perfluoro-n-butane sulphonamide	PFBSA	30334-69-1
N-methyl perfluoro-n-butane sulphonamide	MePFBSA	68298-12-4
perfluoro-n-hexane sulphonamide	PFHxSA	41997-13-1
Total linear and branched PFOA	PFOA total	-
Total linear and branched PFOS	PFOS total	-
Total linear and branched PFOSA	PFOSA total	-
Total linear and branched MePFOSA	MePFOSA total	-
Total linear and branched EtPFOSA	EtPFOSA total	-
Total linear and branched PFHxS	PFHxS total	-

At the same time, the following compounds can also be determined. However, less reliable levels are obtained due to insufficient recovery, loss due to adsorption or possible interference. In this case, the measured levels can only be reported as indicative.

Table 2: indicative PFAS

PFAS	*Abbreviation*	*CAS no.*
perfluoro-n-octadecanoic acid	PFODA	16517-11-6
6:2 fluorotelomer phosphate diester	6:2 diPAP	57677-95-9
6:2/8:2 fluortelomer phosphate diester	6:2/8:2 diPAP	943913-15-3
N-methylperfluoro-n-butane sulphonylamide acetic acid	MePFBSAA	159381-10-9
perfluoro-n-undecanesulfonic acid	PFUnDS	749786-16-1
perfluoro-n-tridecanesulfonic acid	PFTrDS	791563-89-8

Notes:

- For the determination of per- and polyfluoroakyl compounds in groundwater, see the Water Analysis Compendium procedure WAC/IV/A/025.
- Most PFAS only occur in the linear form; the available standards are also linear. A number of PFAS (PFOA, PFOS, and PFOSA, MePFOSA, EtPFOSA and PFHxS) can also be found. In this procedure, both linear and branched forms shall be determined for will determine for PFOA, PFOS, PFOSA, MePFOSA, EtPFOSA and PFHxS. The total linear and branched forms shall be reported as, respectively. PFOA Total, PFOS Total, PFOSA Total, MePFOSA Total, EtPFOSA Total and PFHxS Total respectively.
- In the analysis of activated carbon, PFDoDS, MePFBSA, EtPFOSA, 8:2 diPAP, HFPO-DA, PFECHS and DONA are indicative PFAS instead of quantitative; 6:2 diPAP and 6:2/8:2 diPAP are quantitative PFAS instead of indicative.

2 PRINCIPLE

Known amounts of isotopic fluorine compounds are added to the dried samples. The samples are then extracted after which the extract is further purified. The extract is absorbed in a known volume mobile phase and analysed with liquid chromatography with tandem mass spectrometric detection. The content of the different PFAS is calculated using the internal standard method.

3 EQUIPMENT AND MATERIALS

3.1 Analytical balance with a read-out accuracy of 0.1 mg
3.2 Single-pan balance with a read-out accuracy of 0.01 g
3.3 Agitator
3.4 Vortex mixer
3.5 Centrifuge device
3.6 Unit for evaporation under nitrogen stream with adjustable stream rate
3.7 Polypropylene centrifuge tubes e.g. 50 ml and 15 ml
3.8 Active carbon SPE cartridges (e.g. ENVI-Carb)
3.9 SPE cartridges with a weak anion exchange phase: e.g. OASIS WAX 6cc cart, 150mg mg. Other cartridges can also be used if validated
3.10 Fibreglass filters (e.g. Pall Glass Acrodisc 1 µm)
3.11 Measuring vials of 1.5 ml
3.12 LC-MS system consisting of:
- A HPLC or UPLC liquid chromatograph with injection machine, liquid pump, thermostated column and degassing unit.

Note: in order to reduce the system blank, a *isolator* column, placed between LC pump and injector, is highly recommended

- A tandem quadrupole mass spectrometer with electrospray ionisation chamber

Remark: Alternatively, an ion trap or a high resolution accurate mass (time-of-flight (TOF) or Fourier Transform) mass spectrometer can be used

-A datastation for the setting of the instrumental settings, the data acquisition and the data analysis

3.13 HPLC or UPLC column, e.g. Waters Acquity UPLC BEH C18 1.7µm column, 2.1x100 mm

Notice:
2 PFECHS isomers occur (cis and trans), which are not separated on a C18 column. However, there are column phases at which they are separated. In both cases, PFECHS means the total of the 2 isomers.

3.14 Ultrasonic bath
3.15 SPE vacuum or pressure unit

4 REAGENTS AND STANDARDS

4.1 Methanol, acetonitrile: residue quality
4.2 Water: ultrapure (Milli-Q)
4.3 Ammonium acetate p.a.
4.4 Formic acid p.a.
4.5 Sodium hydroxide p.a
4.6 Extraction solvent: the extraction can be carried out with methanol, acetonitrile or a mixture of methanol and acetonitrile. The extraction solvent may be alkaline, e.g. 0.05 M sodium hydroxide. Dissolve 2 g of sodium hydroxide in 1 litres of methanol, acetonitrile or the mixture of methanol and acetonitrile.
4.7 Acetate buffer solution: dissolve 0.286 ml of acetic acid in 200 ml of ultra-pure water (solution 1). Dissolve 0.097 g of ammonium acetate in 50 ml of ultrapure water (solution 2). Merge solution 1 and solution 2, final volume is 250 ml

4.8 Stock calibration solutions of Native PFAS in methanol: monocomponent or mixing stock solutions, purchased or self-made from the pure substances

4.9 Stock inspection standard of Native PFAS: this is an independent mixing standard in methanol

4.10 Standard solution of isotope enriched PFAS (internal standards): this is purchased or produced as a standard blending solution and diluted to a concentration of e.g. 400 µg/l. The following isotopic PFAS shall be used as a minimum:

perfluoro-n-[1,2,3,4-13C1]-butanoic acid	$^{13}C_4$-PFBA
perfluoro-N-[13C5]-pentanoic acid	$^{13}C_5$-PFPeA
perfluoro-n-[1,2-13C2]-hexanoic acid	$^{13}C_2$-PFHxA
perfluoro -n-[1,2,3,4-13C4]-octanic acid	$^{13}C_4$-PFOA
perfluoro-n-[1,2,3,4,5-13C5]-nonanoic acid	$^{13}C_5$-PFNA
perfluoro-n-[1,2-13C2]-decanoic acid	$^{13}C_2$-PFDA
Perfluoro-n-[1,2-13C2]-undecanoic acid	$^{13}C_2$-PUnDA
Perfluoro-n-[1,2-13C2]-dodecanoic acid	$^{13}C_2$-PFDoDA
Perfluoro-n-[1,2-13C2]-tetradecanoic acid	$^{13}C_2$-PFTeDA
perfluoro-n-[1,2-13C2]-hexadecanoic acid	$^{13}C_2$-PFHxDA
perfluoro-n-hexane[18O2]sulphonic acid	$^{18}O_2$-PFHxS
perfluoro-n-[1,2,3,4-13C4]-octane sulphonate	$^{13}C_4$-PFOS
sodium-1H,1H,2H,2H-perfluoro-1-[1,2-13C2]-n-octane sulphonate	$^{13}C_2$-6:2FTS
perfluor-1-[13C8]-n-octane sulphonamide	$^{13}C_8$-PFOSA
N-methyl-d3-perfluoro-n-octane sulphonamide	D_3-MePFOSA
N-methyl-d3-perfluoro-n-octane sulphonamidoacetic acid	D_3-MePFOSAA
sodium bis(1H,1H,2H,2H-[1,2-13C2]perfluordecyl)phosphate	$^{13}C_2$-8:2 diPAP
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-13C3-propanoic acid	$^{13}C_3$-HFPO-DA

Notes:

- From some isotope marked connections, variants are also available (e.g. $^{13}C_3$-PFHxS, $^{13}C_8$, etc.). These variants may also be used instead of the above.
- For the quantification of PFDoDS and 10:2 FTS, $_{13}$C-PFOS and $_{13}$C-10:2 FTS should be used as an internal standard, respectively Depressed variants are also authorised, e.g. D4-10: 2 FTS instead of $_{13}$C-10: 2 FTS.
- $^{13}C_3$-HFPO-DA may not be stable in acetonitrile and solutions may be better generated in methanol

4.11 Calibration standards: from the stock calibration solutions of native PFAS (4.7) and the standard isotope-rich PFAS (4.9) solution, make a series of dilutions in 1/1 methanol/water with varying concentrations of native PFAS, running from e.g. 0.1 to 100 µg/l and constant isotope-rich PFAS concentrations of approx. 4 µg/l; these solutions are produced anew for each measurement series

4.12 QC standards: based on the stock inspection standard (4.8) QC standards are produced in 1/1 methanol/water at one or more concentration levels

5 SAMPLE STORAGE AND PRE-TREATMENT

For sample storage, reference is made to CMA/1/B.
For sample pretreatment, reference is made to CMA/5/B.

Notes:
- Contact with Teflon or other fluorinated polymers should be avoided
- Before extraction, the samples are dried in air at 40 °C (overnight or longer if necessary, e.g. for highly wet steel), freeze-drying or chemical drying. The sample should then be reduced to 1 mm (for a steel intake of 2 g or more) or to 0.5 mm (for a steel intake of less than 2 g).

6 ANALYSIS PROCEDURE

6.1 EXTRACTION

Soil, sediment and inorganic waste except activated carbon

- Weigh 1 to 5 g of dried and homogenised sample in a polypropylene (PP) centrifuge tube
- Add the appropriate amount of the internal standard solution (4.9) so that the concentration of the internal standards in the final extract is equal to those of the calibration standards. Allow the internal standards to act for about 15 min before adding the extraction fluid to the sample.
- Add at least 5 ml methanol per gram of steel intake, shake vigorously or vortex the sample and shake for 60 min on a shaking device.
- Allow the solid phase to settle, whether or not by centrifugation, and remove the supernatant.

Notice:
Depending on the matrix, lower extraction efficiencies are sometimes obtained for MePFOSAA, EtPFOSAA and diPAPS; in that case, better extraction efficiencies can be obtained when using alkaline methanol (e.g. 50 mM NaOH) as extraction solvent.

Activated carbon

- Weigh 2 to 3 g of dried and homogenised steel in a PP tube.
- Add the internal standard solution and allow to operate for 15 minutes before adding the extraction solvent to the steel.
- Add 12 ml of acetonitrile to the steel, shake vigorously or forge the steel and sonicate the mixture for 1 hours.
- Decant the acetonitrile into a new pre-rinsed PP tube.
- Add 12 ml of hexane to the sample and sonicate for 1 hours.
- Add the hexane extract to the acetonitrile extract.
- Rinse the activated carbon with 4 ml of alkaline methanol and combine the methanol with the hexane/acetonitrile extract.
- Evaporate the collected fractions under a stream of nitrogen to almost dry.
- Add 10 ml of blank water and shake vigorously or vortex.
- Analyse like a water sample according to WAC/IV/A/025 (SPE extraction).

6.2 PURIFIYING THE EXTRACT

- The extract (6.1, excluding extract of activated carbon) is purified by applying it over an activated carbon cartridge (3.8), which was previously rinsed with 10 ml of acetonitrile. The eluate is collected in a PP centrifuge tube. The cartridge is rinsed with twice 2.5 ml acetonitrile.

  Comment
  Alternatively, the column is eluated with two times 2.5 ml of methanol. In this case, the column should be blown dry before elution.

- If necessary, the purified extract is evaporated under a nitrogen stream at 40-65 °C; it must be avoided that the extract evaporates dry (possibly add a keeper, e.g. 50 µl DMF);

  Comment
  When extracted with alkaline methanol, crystal formation may occur during evaporation. This can be avoided by acidifying the eluation of the carbon column. In this case, the eluation contain e.g. 0.05 M of acetic acid.

- Dilute the extract if desired to with ultra pure water and/or methanol. The calibration standards measurement solutions are produced in the same solvent mixture as the sample measurement extract; this measurement solution is filtered over a fibreglass filter or centrifuged only if necessary for the analysis.

6.3 LC-MS/MS ANALYSIS

Remark: 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.

6.3.1 LC CONDITIONS

A UPLC analysis is done e.g. on a UPLC BEH C18 column, 1.7µm, 2.1 x 100 mm, with gradient clearance. Typical UPLC settings are:

- mobile phase:
  A = Water + 5 % MeOH and 2 mM ammonium acetate B= MeOH + 2 mM ammonium acetate
- flow rate: 0.3 ml/min
- column temperature: 40 °C
- injection volume: 10 µl
- gradient:

Time	A%	B%
min	%	%
0.00	70	30
0.50	70	30
25.00	10	90
27.00	10	90
28.00	1	99
30.00	70	30

The LC analysis can also be done on an HPLC installation, on a C18 column with gradient clearance. Typical HPLC settings are:

- mobile phase:
  A= methanol
  B= water + 2 mM ammonium acetate
- flow rate: 1 ml/min.
- column temperature: 35°C
- injection volume: 100 µl
- gradient:

Time	**A**	**B**
min	**%**	**%**
0	50	50
15	100	0
16	100	0
17	50	50
23	50	50

Detection of FOSAA, 6:2 PAP and 8:2 PAP requires an alkaline mobile phase; an example of UPLC settings is given below:

- mobile phase:
  A = water + 2 mM ammonium acetate + NH3 (pH 10.5)
  B = ACN/MeOH 1/1 + 5 mM methylpiperidine
  C = Water/MeOH/ACN/isopropanol 25/25/25/25
- flow rate: 0.3 ml/min
- column temperature: 40 °C
- injection volume: 10 µl
- gradient:

Time	A	B	C
	(%)	(%)	(%)
0.0	90	10	0
0.5	90	10	0
5	55	45	0
10	55	45	0
20	0	95	5
23	0	95	5
25	90	10	0

6.3.2 MS CONDITIONS

All recordings are performed with Multiple Reaction Monitoring (MRM), with ionisation via electrospray in negative mode (ES-).

Typical settings for the MS acquisition are given below for a Waters Xevo TQ-S:

Ion Mode:	ES-
Capillary Voltage:	1kV
Cone Voltage:	component dependent
Source Offset:	30V
Desolvation Temperature:	450°C

Source Temperature:	150°C
Desolvation:	800L/Hr
Cone:	150L/Hr
Nebuliser:	7Bar
Ion Energy1:	0.9
Ion Energy2 :	1.1
Collision gas flow:	0.20ml/min
Collision energy:	component dependent

The following ion transitions are recorded. At the same time, typical UPLC retention times are indicated. These can shift depending on the used column. The table also shows which isotope marked internal standard can be used for the quantification of the Native compound. In order to avoid as much as possible problems of low recovery or dispersion of results (due to sorption to container or injector and/or matrix duress/amplification), efforts should be made to use as many equivalent internal standards as possible.

Table 3: ion transitions of the PFAS

Compound	**Parent (m/z)**	**Daughter (m/z)**	**(Q/q)**	**Cone (V)**	**Collision (V)**	**IS**	**Rt NH_4Ac (min)**	**Rt NH_3 (min)**
PFBA	213	169	Q	30	8	13C-PFBA	2.01	3.91
PFPeA	263	219	Q	30	8	13C-PFPeA	4.78	5.96
PFHxA	313	119 269	q Q	40 40	20 8	13C-PFHxA	8.72	7.24
PFHpA	363	169 319	q Q	40 40	20 11	13C-PFHxA	12.21	8.75
PFOA	413	169 369	q Q	40 40	17 11	13C-PFOA	14.94	11.81
PFNA	463	169 419	q Q	40 40	17 11	13C-PFNA	17.13	14.48
PFDA	513	219 469	q Q	40 40	20 11	13C-PFDA	18.95	15.69
PFUnDA	563	169 519	q Q	40 40	23 11	13C-PFUnDA	20.5	16.57
PFDoDA	613	319 569	q Q	40 4	20 11	13C-PFDoDA	21.84	17.27

				0				
PFTrDA	663	319 619	q Q	40 40	23 14	13C-PFDoDA	22.97	17.87
PFTeDA	713	319 669	q Q	40 40	20 14	13C-PFTeDA	23.96	18.4
PFHxDA	813	219 769	q Q	40 40	32 14	13C-PFHxDA	25.56	19.24
PFODA	913	219 869	q Q	50 50	29 17	13C-PFHxDA	26.79	19.89
PFBS	299	80 99	Q q	50 50	41 41	13C-PFHxS	5.76	6.84

PFPeS	349	80 99	Q q	50 50	32 30	18O2-PFHxS	9.53	8.09
PFHxS	399	80 99	Q q	50 50	38 32	18O2-PFHxS	12.7	10.4
PFHpS	449	80 99	Q q	50 50	41 36	13C-PFHxS	15.23	13.8
PFOS	499	80 99	Q q	60 60	50 40	13C-PFOS	17.29	15.26
PFNS	549	80 99	Q q	60 60	46 45	13C-PFOS	19.04	16.2
PFDS	599	80 99	Q q	65 65	50 50	13C-PFOS	20.53	16.94
PFDoDS	699	80 99	Q q	65 65	49 47	13C-PFOS	22.95	18.11
4:2 FTS	327	80.5 306.9	q Q	40 40	26 20	13C 6:2 FTS	8.37	7
6:2 FTS	427	80.7 406.9	q Q	40 40	28 30	13C 6:2 FTS	14.8	10.83
8:2 FTS	527	80.7 506.9	q Q	40 40	32 34	13C 6:2 FTS	18.92	15.44
10:2 FTS	627	80.7 606.9	q Q	40 40	37 30	13C 10:2 FTS	21.87	17.14
PFOSA	498	78 169	Q q	50 50	29 32	13C-PFOSA	19.99	14.67
MePFOSA	512	169 219	Q q	40 40	25 22	D3-MePFOSA	22.64	15.84
EtPFOSA	526	169 219	Q q	50 50	25 25	D3-MePFOSA	23.49	16.69

PFOSAA	556	419 498	q Q	86 86	26 28	D3-MePFOSAA	-	9.61
MePFOSAA	570	419 483	Q q	40 40	19 15	D3-MePFOSAA	19.7	16.05
EtPFOSAA	584	419 526	Q q	40 40	20 20	D3-MePFOSAA	20.44	16.45
6:2 PAP	443 443	79 97	q Q	28 28	46 18	13C 8:2 PAP	-	6.5
8:2 PAP	543	79 97	q Q	32 32	58 16	13C 8:2 PAP	-	8.62
6:2 diPAP	789	97 443	Q q	40 40	31 19	13C 8:2 diPAP	23.75	18.19
6:2/8:2 diPAP	889	97 443	Q q	40 40	30 20	13C 8:2 diPAP	25.18	19
8:2 diPAP	989	97 543	Q q	50 50	30 25	13C 8:2 diPAP	26.3	19.63
HFPO-DA	285	185 169	Q q	7 7	18 14	13C HFPO-DA	9.7	7.64
ADONA	376.97	84.95	q	8	26	13C HFPO-DA	12.54	9.31

		250.96	Q	8	12			
PFECHS	461	99 381	q Q	40 40	24 24	13C-PFOA	14.84	13.47
PFBSA	298	78 64	q Q	50 50	29 32	13C-PFOSA		
MePFBSA	312	219 65	q Q	40 40	25 22	13C-PFOSA		
MePFBSAA	370	283 312	q Q	86 86	26 28	13C-PFOSA		
PFHxSA	398	78 119	q Q	50 50	29 32	13C-PFOSA		
13C-PFBA	217	172	IS	30	8		2.01	3.9
13C-PFPeA	268	223	IS	30	8		4.77	5.96
13C-PFHxA	315	270	IS	40	11		8.72	7.24
13C-PFOA	417	372	IS	40	8		14.95	11.8
13C-PFNA	468	423	IS	40	11		17.13	14.47
13C-PFDA	515	470	IS	50	11		18.95	15.69
13C-PFUnDA	565	520	IS	50	14		20.5	16.57
13C-PFDoDA	615	570	IS	50	14		21.84	17.27
13C-PFTeDA	715	670	IS	50	14		23.96	18.4
13C-PFHxDA	815	770	IS	50	14		25.56	19.25
18O2-PFHxS	403	84	IS	50	40		12.7	10.41
13C-PFOS	503	80	IS	60	40		17.28	15.26
13C 6:2 FTS	429	409	IS	10	24		14.8	10.82
13C-PFOSA	506	78	IS	50	40		19.99	14.68
D3-MePFOSA	515	169	IS	50	40		22.62	15.82
D3-MePFOSAA	573	419	IS	40	20		19.69	16.03
13C 8:2 PAP	545	97	IS	32	16			8.62
13C 8:2 diPAP	993	97	IS	25	20		23.75	18.19
13C HFPO-DA	287	119	IS	10	18		9.7	7.64

Q: Transition for quantification of the component
q: Transition to confirm (qualification) of the quantification transition

6.3.3 IDENTIFICATION AND INTEGRATION

The presence of native fluorine compounds in the samples is confirmed on the basis of the criteria for retention times and ion ratios as specified in CMA/6/D.

The identification of the isotope enriched compounds is also based on the characteristic m/z and

retention time.

The identified peaks are integrated using the equipment software and manually verified.

6.3.4 CALIBRATION

Calibration can take place in a number of different ways, see CMA/6/D. For the quality requirements to be met by the calibration, see also CMA/6/D.

7 CALCULATIONS

For the sample extracts, the transitions are recorded in the same way as described above for the standard solutions. Based on the sample integration values and the calibration straight line/curve determined for the calibration standard, the contents of the different compounds in the sample shall be calculated. For soil, sediment and inorganic waste, the result shall be expressed in µg/kg ds. The dry matter content should therefore be determined at 105 °C in accordance with CMA/2/II/A.1.

Notes:
- If the upper limit of the working area is exceeded:
    - the extract shall be diluted and remeasured at a maximum of 10 times;
    - if the recovery of the internal standards exceeds 70 %, it may be diluted more than 10 times with extra addition of internal standard;
    - alternatively, the sample is reprocessed with less sample intake and/or more extraction solvent.

- For the reporting of the 'sum of measured quantitative PFAS', the 'lower bound' sum of the quantitative PFAS is calculated (see Table 1).
- For the reporting of the 'sum of measured indicative PFAS', the 'lower bound' sum made from the indicative PFAS (see Table 2).
- For the reporting of the 'sum of measured indicative PFAS', the 'lower bound' sum of quantitative PFAS (see Table 1) and indicative PFAS (see Table 2) is calculated.

Notes:
- 'lower bound' means that all individual PFAS concentrations below the reporting limit requirement (CMA/6/A) are equated to 0 when making the sum.
- If all individual quantitative PFAS concentrations are lower than the reporting limit requirements in CMA/6/A, then for the 'sum of measured quantitative PFAS', '< the highest reporting limit of an individual quantitative PFAS' shall be reported.
- If all individual indicative PFAS concentrations are lower than the reporting limit requirements in CMA/6/A then for the 'sum of measured indicative PFAS', ' < the highest reporting limit of an individual indicative PFAS' shall be reported.
- If all individual PFAS concentrations are lower than the reporting limit requirements in CMA/6/A, then for the "sum of measured PFAS" "< the highest reporting limit requirement of an individual PFAS" is reported.
- If the concentration of one or more PFAS parameters exceeds the reporting limits used by the laboratory (which should not exceed the reporting limit requirements in CMA/6/A) then for the 'sum of measured PFAS', the sum of these concentrations shall be reported regardless of whether this sum is greater or lower than the highest reporting limit of an individual PFAS.

- For PFOS, PFOA, PFOSA, MePFOSA, EtPFOSA and PFHxS, the technical mixtures consist of both linear and branched isomers. The standards, on the other hand, are purely linear forms. Quantify, pending appropriate standards and clear regulatory/international agreements, both linear and branched forms using the MRM transition and RRF value of the linear form.
- For PFOA, PFOS, PFOSA, MePFOSA, EtPFOSA and PFHxS, both linear and branched forms are determined. The total of linear forms and branched forms shall be reported, as PFOA Total, PFOS Total, PFOSA Total, MePFOSA Total, EtPFOSA Total and PFHxS Total respectively.
- If the branched PFAS are to be reported separately, they are calculated as the difference of the result for the linear and branched forms and the result of the linear form.
- For groundwater, the sum of the measured PFAS is made as described above for soil. The classification into quantitative and indicative parameters as set out in WAC/IV/A/025 shall be applied. For the branched forms PFAS, the sum of linear and branched forms is included in the sum of the measured quantitative PFAS. For the determination of PFAS in groundwater, see WAC/VI/A/001.

8 QUALITY CONTROL

For the quality requirements for calibration, procedure blank, sensitivity inspection, inspection sample, drift inspection and independent inspection standard, reference is made to CMA/6/D.

Notice:
- For the procedure blank, a blank soil (possibly pre-rinsed with methanol) should be taken as a sample. The procedure blank will complete the pretreatment and reprocessing process, including the drying and homogenisation of the sample.
- In the case of an analysis series with both soil and sediment samples, two inspection samples should be included (a soil and a sediment).

8.1 RECOVERY OF THE ISOTOPE-MARKED FLUORINE COMPOUNDS

For each sample, the recovery of isotope-observed internal standards is determined, i.e. the experimentally recovered quantity of each of the standards added at the beginning of the analysis. This is achieved by comparing the area of the isotope enriched compound obtained for the sample (A_{is}(sample)) compared to the surface area expected for a calibration standard (A_{is}(calibration standard)), where there is approximately the same concentration of native compound as measured in the sample preparation (this to take into account the suppression of the isotope-marked connection signal by the cohesive native compound). The recovery is given by:

$$R\% = A_{is}(sample) * 100/A_{is}(calibration\ standard)$$

Recovery efficiency depends on sorption phenomena, signal suppression/reinforcement by matrix components and extraction/clean up efficiency. For responsible quantification, the recovery efficiency of the ^{13}C-marked fluorine compounds shall be between 30 % minimum and 200 % maximal. If, where appropriate (e.g. if the sample is no longer available or if re-analysis is not relevant for the use of the result), results are reported where the criterion for recovery is not met, this should be reported as a comment on the report. For the analysis of activated carbon steel, the recovery of the internal standards shall be at least 10 %.

9 PERFORMANCE CHARACTERISTICS

For the performance characteristics, see CMA Part 6.

10

11 REFERENCES

- DIN 38414-14 Deutsche Einheitsverfahren zur Wasser-, Abwasser- und Schlammuntersuchung - Schlamm und Sedimente (Gruppe S) - Teil 14: Bestimmung ausgewählter polyfluorierter Verbindungen (PFC) in Schlamm, Kompost und Boden - Verfahren mittels Hochleistungs-Flüssigkeitschromatographie und massenspektrometrischer Detektion (HPLC-MS/MS) (S 14)
- ISO 21675:2019: Water quality — Determination of perfluoroalkyl and polyfluoroalkyl substances (PFAS) in water — Method using solid phase extraction and liquid chromatography- tandem mass spectrometry (LC-MS/MS)
- C.R. Powley, S.W. George, T.W. Ryan, R.C. Buck, Matrix Effect-Free Analytical Methods for Determination of Perfluorinated Carboxylic Acids in Environmental Matrixes, Analytical Chemistry 2005, 77 (19)
- Dutch Technical Arrangement (NTA): Analysis of PFAS in soil, water bottom and sludge, with HPLC and mass spectrometry.

Mineral oil with GC/FID

CONTENTS

1 OBJECTIVE AND SCOPE

This procedure replaces procedure CMA/3/R.1 of June 2024.
This method describes the quantitative determination of mineral oil content in soil, sediment, sludge, solid waste and water using gas chromatography.
This method does not apply to the determination of mineral oil in wastes used in or as fertilisers or soil improvers such as compost, digestates, related input and output streams from the treatment of biological waste, sewage sludge. This is the CMA/3/W method.

Mineral oil refers to compounds extractable with a hexane/acetone mixture from solid matrices or with hexane from water, which are not adsorbing to florisil and which are gas chromatographable at retention times between the retention times of n-decane and n-tetracontane (on an apolar column).
Note: oil contaminants where a volatile fraction is present are insufficiently recovered using the following method. Similarly, aromatic and weak polar compounds, whether or not constituents of oil products, are not found quantitatively.

2 PRINCIPLE

Water samples are extracted with hexane.
Solid samples and sludge samples with sufficient dry weight content will first be mixed with diatomaceous earth or sodium sulphate (Na_2SO_4) as desiccant and subsequently subjected to a PLE extraction ('pressurised liquid extraction') or Soxhlet extraction with an n-hexane/acetone mixture. If PLE extraction is practically difficult to implement or allows insufficient sampling, a Soxhlet extraction shall be carried out.

Notes:

- For soil samples, extraction by microwave is also permitted subject to a declaration of equivalence by OVAM.
- miniaturisation and automation of sample preparation for the determination of mineral oil in groundwater is permitted subject to a declaration of equivalence by OVAM.

The extracts are purified with florisil to remove the polar compounds.
The purified extracts shall be evaporated if necessary, and injected into a gas chromatograph using a non-discriminatory injection system equipped with an apolar column and a flame ionisation detector. The total area of the chromatogram located between the retention times of n-decane and n-tetracontane shall be integrated and compared to the surface area for a calibration standard of diesel and engine oil. The internal standard method is used, adding a known amount of n-tetracontane after extraction.

3 EQUIPMENT AND MATERIALS

3.1 analytical balance with a reading accuracy of 0.1 mg
3.2 Single-pan balance with a read-out accuracy of 0.01 g
3.3 pestle and mortar (porcelain)
3.4 in case of PLE extraction: PLE apparatus suitable for a sample intake of approximately 10 g and capable of operating at a minimum pressure of 40 bar at a temperature of 100 °C
3.5 in the case of Soxhlet extraction: Soxhlet device and electric heating jacket with temperature control
3.6 usual laboratory glassware, cleaned and rinsed with acetone according to the usual procedure, then hexane or petroleum ether
3.7 calibrated dot tubes
3.8 100 ml and 250 ml Erlenmeyers
3.9 separating funnels from 1000 to 2000 ml
3.10 unit for vapour under nitrogen stream with adjustable stream rate or a Rotavapor
3.11 gas chromatograph for capillary columns, equipped with a non-discriminatory injection system (on-column or cold PTV injector), a flame ionisation detector and a data station equipped with the necessary control and data processing software; the gas chromatograph may be equipped with a large-volume injector
3.12 capillary columns with apolar phase (polydimethylsiloxane or poly-95 %-dimethyl-5 %-diphenylsiloxane), from 10 to 25 m in length, with a diameter of 0.10 to 0.32 mm and a film thickness of 0.1 to 0.25 µm
3.13 note: a column with sufficient resolution must be used to characterise oil contamination with GC/MS (see 6.3.5). in that case, the gas chromatographic separation rate (valley/lowest peak height) of phytane and n-octadecane shall be less than 30 %.
3.14 10 µl syringe

4 REAGENTS AND NORMS

4.1 N-hexane, acetone: residue analysis quality

note: instead of n-hexane, n-heptane, isohexane, cyclohexane or petroleum ether (40°-60 °C) can also be used

4.2 sodium sulphate (Na_2SO_4): granular and anhydrous; an opened package is poured into a dish and kept at 130°C in a drying oven
4.3 diatomaceous earth (celite, kieselguhr, etc.): grain size 0.01-0.04 mm
4.4 sea sand: acid-cleaned and annealed
4.5 hydrochloric acid
4.6 magnesium sulphate ($MgSO_4$)
4.7 NMI calibration mixture of mineral oils, consisting of a diesel oil and an engine oil, as available on the market (NMI VSL-01)
4.8 calibration mixture of even n-alkanes, running from C10 to C40
4.9 n-decane (C10)
4.10 N-Tetracontane (C40)
4.11 4-cholesten-3-one
4.12 tetraline
4.13 florisil, grain size 0.15 mm to 0.25 mm (60-100 mesh), heated at 140 °C for 16 hours and then stored in a desiccator

Calibration and test solutions (in case of large-volume injection, the concentrations of the following solutions should be adjusted according to the injection volume)

4.14 calibration work solutions: these contain NMI oil in hexane at concentrations of 50 to 10000 µg/ml, each solution contains 100 µg/ml n-C40 as internal standard

4.15 inspection-working solution for the verification of calibration straightness: contains 5000 µg/ml NMI oil in hexane and 100 µg/ml n-C40 as internal standard

4.16 working solution of even n-alkanes in hexane, each at a concentration of 10 µg/ml, for recording retention times

4.17 internal standard solution of 1000 1000 µg/ml n-decane and 1 000 µg/ml n-tetracontane in hexane, the solution is prepared from the pure products, the solution is added to the samples after extraction and serves to check the proper conduct of the analysis and to mark the starting and end points of the integration, the solution is stored at room temperature and is sonicated before use to dissolve C40 crystallised if necessary

4.18 doping solution of 40000 µg/ml NMI oil in acetone, for the determination of recovery (at higher concentrations there is a risk of decomposition in the refrigerator)

4.19 test solution to determine the quality of florisil, containing 50 µg/ml tetraline, 50 µg/ml decane and 200 µg/ml 4-cholesten-3-one in hexane

5 SAMPLE STORAGE AND PRE-TREATMENT

For sample preservation and storage, reference is made to CMA/1/B.
For sample pretreatment, reference is made to CMA/5/B.

No quantitative determination shall be made of water samples with a visible float layer. The identity of the floating layer shall be investigated. In the report, the presence of a floating layer is reported together with the identity.

Groundwater samples (usually sampled as part of soil research) are shaken upon arrival in the lab and the samples are left to rest for at least 4 hours so that the particles can sink. The samples are then carefully decanted. Not more than half of the top water layer is decanted to minimise the number of particles in operation. All other types of water samples are generally not decanted in advance!

For solid samples with low water content such as bottom samples, a PLE extraction with hexane/acetone is used as standard. If PLE extraction is practically difficult to implement or allows insufficient sampling, a regular Soxhlet extraction with hexane/acetone shall be used. For samples with a water content exceeding 30% (e.g. water table after decantation, sewage sludge, etc.), single PLE is not an appropriate extraction technique: the water is not retained under the PLE conditions due to the desiccant and the extraction is too low. In this case, the extraction shall be carried out with soxhlet. In the case of dredging and water bottom, the sample may be concentrated by drying to the air at 40 °C before carrying out the PLE extraction. Soil samples with a water content of less than 40 % can also be extracted with double PLE extraction.

Shredder materials containing plastic components can cause problems during hot extraction with acetone/hexane; shredder materials containing plastic are therefore always extracted with hexane alone.

Notice:
For soil samples, extraction by microwave is also permitted subject to a declaration of equivalence by OVAM.

6 ANALYSIS PROCEDURE

6.1 EXTRACTION

6.1.1 SOIL, SLUDGE AND SOLID WASTE

Method of PLE extraction:

- weigh in a mortar a quantity (e.g. 10 g) of the homogeneous sample to the nearest 0.01 g
- weigh at least an equivalent quantity of diatomaceous earth, to the nearest 0.01 g, mixed with the sample in the mortar until a dry mass is obtained
- place a cellulose filter in the extraction cell and then weigh into the extraction cell, depending on the expected degree of contamination of the sample, a quantity of the sample mixed with diatomaceous earth, up to the nearest 0.01 g;
- close the top of the extraction cell hand-sealed with a 'cap'
- run the PLE extraction using the basic settings below:

PRESSURE	70-100 bar
TEMPERATURE	> 100 °C
SOLVENT	acetone 50 %
	n-hexane 50 %

Note: depending on the PLE device used, additional parameters such as extraction duration, etc. These should be optimised for maximum extraction of the mineral oil. The following table gives the specific settings for the Dionex ASE-200:

EXTRACTION CYCLES	1
HEAT	5 min
STATIC	5 min
FLUSH%	60 vol.
PURGE	150 sec
CYCLES	1

- transfer the extract to a separating funnel and rinse with 10 ml hexane, add an amount of the internal standard solution so that the concentration of C40 in the final extract shall be approximately 100 mg/l (in the case of large-volume injection, the amount should be adjusted according to the injection volume)

Note: alternatively, the internal standard may also be added to the sample for extraction (i.e. in the extraction cell).

- remove the acetone by shaking the extract for a few minutes with 500 ml of water containing 40 g of $MgSO_4$, possibly adding an additional amount of hexane to facilitate separation
- dry the extract with Na_2SO_4
- ling, if no evaporation step is provided, the hexane extract to known volume (e.g. 50 ml) or determine the exact weight

Method of double PLE extraction:

- weigh in a mortar a quantity (e.g. 10 g) of the homogeneous sample to the nearest 0.01 g;
- weigh a quantity of drying agent (diatomaceous earth or sodium sulphate), to the nearest 0.01 g; mix this with the sample in the mortar until a dry mass is obtained;
- place a cellulose filter in the extraction cell and then weigh into the extraction cell, depending on the expected degree of contamination of the sample, a quantity of the sample mixed with diatomaceous soil, to the nearest 0.01 g (if necessary, the sample shall be divided into 2 cells); top the extraction cell up with sea sand;
- close the top of the extraction cell manually with a 'cap';
- perform a first extraction with acetone, with the typical PLE settings below;

HEAT	5 min	PRESSURE	70-100 bar
STATIC	5 min	TEMPERATURE	> 100 °C
FLUSH%	60 vol.	SOL # 1	Acetone 100 %
PURGE	150 sec	SOL # 2	- %
CYCLES	1	SOL #3	- %

- perform a second extraction with acetone/hexane, with the following typical PLE settings;

HEAT	5 min	PRESSURE	70-100 bar
STATIC	5 min:	TEMPERATURE	> 100 °C
FLUSH%	60 vol.	SOL # 1	Acetone/Hexane 50/50
PURGE	150 sec	SOL # 2	- %
CYCLES	1	SOL #3	- %

- Merge both extracts together and homogenise
- transfer the extract to a separating funnel and rinse with 10 ml hexane, add an amount of the internal standard solution so that the concentration of C40 in the final extract will be approximately 100 mg/l (in the case of large-volume injection, the amount should be adjusted according to the injection volume)

Note: alternatively, the internal standard may also be added to the sample for extraction (i.e. in the extraction cell).

- remove the acetone by shaking the extract for a few minutes with 500 ml of water containing 40 g of MgSO4, possibly adding an additional amount of hexane to facilitate separation
- dry the extract with Na2SO4
- dilute, if no evaporation step is provided, the hexane extract to known volume or determine the exact weight

Method of Soxhlet Extraction:

- before extraction of a sample, rinse the soxhlet setup (including the extraction sleeve and glass wool plug to be used) by refluxing acetone/hexane (50/50) for at least half an hour; then empty the setup and dry the extraction sleeve and glass wool plug in a drying oven;
- in the case of a sludge sample or similar free wet sample, first carry out chemical drying of the sample by weighing in a mortar a quantity (approximately 10 g) of the homogeneous sample to the nearest 0.01 g, weighing at least an equivalent amount of sodium sulphate or diatomaceous soil to the nearest 0.01 g, and mixed with the sample in the mortar until a dry mass is obtained
- weigh 1-30 g of the sample (if necessary, pre-mixed with drying agent) depending on the expected degree of contamination, to the nearest 0.01 g, apply into the extraction sleeve of the pre-cleaned Soxhlet setup, and close with a pre-cleaned glass wool plug
- extract with a known amount of acetone for approximately three hours;
- add n-hexane to the round-bottom flask (as much as acetone was added)
- extract with the n-hexane/acetone mixture for approximately 16 hours;
- transfer the extract to a separating funnel and rinse 3 times with 5 ml hexane, add an amount of the internal standard solution so that the concentration of C40 in the final extract shall be approximately 100 mg/l (in the case of large-volume injection, the amount should be adjusted according to the injection volume)

  Note: alternatively, the internal standard may also be added to the sample for extraction (i.e. in the extraction sleeve).

- remove the acetone by shaking the extract for a few minutes with 500 ml of water containing 40 g of MgSO4, possibly adding an additional amount of hexane to facilitate separation
- dry the extract with Na2SO4
- dilute, if no evaporation step is provided, the hexane extract to known volume or determine the exact weight

Working method for shredder materials:

- dry the shredder material in an oven at 40 °C
- before extraction of a sample, rinse the soxhlet apparatus (including the extraction sleeve and glass wool plug to be used) by refluxing with hexane for at least half an hour; then empty the apparatus and dry the extraction sleeve and glass wool plug in a drying oven
- weigh about 10g of the dried sample into the Soxhlet sleeve
- extract with n-hexane for 16 hours
- transfer the extract to a separating funnel and rinse 3 times with 5 ml hexane, add an amount of the internal standard solution so that the concentration of C40 in the final extract shall be approximately 100 mg/l (in the case of large-volume injection, the amount should be adjusted according to the injection volume)

  Note: alternatively, the internal standard may also be added to the sample for extraction (i.e. in the extraction sleeve).

- remove the acetone by shaking the extract for a few minutes with 500 ml of water containing 40 g of $MgSO_4$, possibly adding an additional amount of hexane to facilitate separation
- dry the extract with Na2SO4
- dilute, if no evaporation step is provided, the hexane extract to known volume or determine the exact weight

6.1.2 WATER

- weigh the sample flask to the nearest 0.1 g
- transfer the entire contents of the sample bottle to a suitable separating funnel
- add 80 g $MgSO_4$ per 1000 ml of water and, if not already done, apply to pH 2 with hydrochloric acid
- rinse the sample bottle with at least 20 ml hexane and transfer the rinse fluid to the separating funnel
- shake the whole vigorously for around 30 min
- Allow the phases to separate: in case of emulsion formation, separation can be stimulated by centrifugation, freezing, salting or ultrasonic treatment
- leave the organic phase over a fiberglass filter with Na_2SO_4 and catch in an Erlenmeyer
- repeat the rinsing and extraction step at least once with 20 ml hexane
- rinse the separating funnel and filter with 5 ml hexane and add to the extract, add an amount of the internal standard solution so that the concentration of C40 in the final extract shall be approximately 100 mg/l (in case of large-volume injection, the amount should be adjusted according to the injection volume)
- dilute, if no evaporation step is provided, the collected hexane fractions to known volume (e.g. 50 ml) or determine the exact weight of the extract
- weigh the empty sample flask and determine the weight and volume of the original contents

6.2 PURIFICATION WITH FLORISIL

6.2.1 TESTING FLORISIL QUALITY

The purification efficiency of the florisil shall be checked at least monthly. The check should also be carried out when a new batch of florisil is used.
1.5 g florisil is added to 5 ml of the test solution of decane, tetraline and 4-cholesten-3-one. The whole is shaken for 10 minutes and the solution is examined gas chromatographically. The recoveries of the different compounds are determined by comparing the surfaces of the chromatogram peaks obtained for the treated and untreated test solution:

$$T = (A_b/A_{nb}) * 100$$

with
T = the recovery in %
A_b = the peak area of the component for the treated solution
A_{nb} = the peak area of the component for the untreated solution

The recovery of the compounds must meet the following criteria:

tetraline	>50%
4-cholesten-3-one	< 3%
decane	> 90%

Notice: If necessary, the recovery of tetraline can be increased by adding water to the florisil (e.g. 3-10 %).

6.2.2 PURIFYING EXTRACTS AND CONCENTRATION

- add an appropriate amount of florisil to the whole extract or to a part of the extract:
  for water-sample extracts, 2 g florisil per 500 ml water intake is used for extracts of soil and solid waste 3 g florisil per intake of 10 g of wet sample
- shake the whole thing for 10 min
- if no evaporation step is provided, a part of the purified extract, after settling the florisil and, if necessary, filtering, is transferred to a GC injection bottle. If a evaporation step is provided, the florisil is separated by centrifugation or filtration; the purified whole is evaporated to a known volume, e.g. 1 ml

Notice:

- one florisil purification step shall be carried out regardless of the degree of contamination of the extract, and irrespective of the possible occurrence of a different oil pattern
- in order to obtain sufficient detectability, it may be necessary, depending on the expected degree of contamination, to vapour extracts of water samples, this evaporation step leads to losses of any volatile fraction present, these losses can be avoided by large-volume injection with a suitable type of injector

6.3 GC-FID ANALYSE

6.3.1 GAS CHROMATOGRAPH SETTINGS

Adjust the gas chromatograph in such a way that:
- for the working solution of n-alkanes, all alkanes are separated to the baseline;
- for a calibration standard of NMI oil, the ratio of the total area of C20 to C40 relative to that of C10 to C20 is between 1.25 and 1.40 (the middle of the C20 peak is taken as an integration limit).

Typical GC settings are shown below:

column specifications:	DB-5ms, 10 m x 0.25 mm x 0.25 µm, apolar front column, 1.5 m x 0.53 mm carrier gas and flow: helium, 1.0 ml/min
injection mode:	on column or other, if the above criteria are met
injection volume:	1 µl
GC-progr. (hexane):	60 °C, 5 min, 25 °C/min to 320 °C, 6 min (total duration 22 min) FID
temperature:	325 °C

6.3.2 MEASUREMENT

Register a chromatogram of the ‘column bleed’ by injection of extraction solvent, whether or not evaporated in accordance with the method used for the samples.
Inject a blank procedure (a blank water sample or a quantity of diatomaceous earth or After$_2SO_4$ which undergoes the complete reprocessing procedure according to the procedure for samples), the calibration standard(s) and the samples. Record the chromatograms and subtract the 'column bleeding'.

When integrating the chromatograms, the total peak area shall be integrated from n-decane to n-tetracontane, excluding the aforementioned alkanes. Integration shall be started immediately after the peak of n-decane (or corresponding at the retention time) at the signal level before or immediately after the solvent peak. The integration is terminated immediately before the peak of n-tetracontane (or corresponding at the retention time) at the same signal level. Chromatograms for the NMI oil and a real sample are attached.
In addition, n-tetracontane is integrated separately. In case a contamination is present in the sample extracts which continues after C40, the integration takes place up to the base of the chromatogram peak (and thus not to the straight-through baseline).

Notes:

- an elevated baseline for tetracontane indicates heavy oil contamination; this should be reported on the analysis report;
- if the chromatographic peak of the internal standard (n-C40) is disrupted so that the peak area of the IS cannot be determined, external quantification may be applied; this should be reported on the analysis report
- The recovery of the internal standard (calculated in relation to the average of the calibration standards) shall be between 50 % and 150 %. If the recovery of the internal standard is below 50 % but higher than 20 %, the results may be reported provided a comment on the analysis report (except if the result is less than the reporting limit, then the comment on the report is not necessary). If the recovery of the internal standard is less than 20 %, no quantitative result shall be reported.
- if a signal is observed greater than the highest concentration of the calibration sequence or of the linear range (see below), the solution should be diluted.

6.3.3 CALIBRATION

Initial calibration
The initial calibration shall be performed at least once a month. Inject the calibration work solutions into increasing concentration. These solutions contain a variable concentration of NMI oil (from 50 to 10000 µg/ml or other depending on the linear range and scope) and a constant concentration (approximately 100 mg/l) of internal standard C40. In case of large-volume injection, the concentrations are adjusted according to the volume injected.
Integrate the chromatograms after correction for the 'column bleeding'. The surface ratio (fraction C10-C40 to the internal standard n-C40) shall be plotted according to the concentration ratio of the NMI oil and n-C40. If the linearity criterion (see below) is met, the mean relative response factor (RRF) shall be calculated. Alternatively, the calibration can be performed using the slope of the origin forced calibration straight line, provided that the origin is within the 95 % confidence curves.

Daily calibration
Check the validity of the initial calibration every 15 samples by injecting the NMI oil inspection working solution. Based on the observed surfaces of the fraction C10-C40 and the internal standard C40, calculate the content of the working solution using the mean RRF or the slope of the calibration right determined at the initial calibration.

Compare the content calculated with the content ingested. If the deviation is less than 10 %, the initial calibration shall be considered valid and the calculations may be made using the initially determined RRF or calibration right. Otherwise, the calibration rate shall be re-established as described above.

6.3.4 QUALITATIVE DESCRIPTION OF THE IMPURITY

The obtained chromatogram is split into fractions: it integrates from C10 to C12, from C12 to C20, from C20 to C30 and from C30 to C40, integrating from immediately after peak to immediately after peak, with the exception of C40 where the integration stops immediately before the peak.
On the basis of the areas obtained, absolute contents can be calculated for the individual fractions (see 7.2).

The report will include:

- the total content in µg/l or mg/kg dm
- the contents of the individual fractions in µg/l or mg/kg of dm:
  >C10-C12
  >C12-C20
  >C20-C30
  >C30-<C40

Notes:
- optionally, the percentage contributions of the different political groups can also be mentioned
- if a fraction >C40 is present in the chromatogram, this should be indicated in the report.

7 CALCULATIONS

7.1 RELATIVE RESPONSE FACTOR

The internal standard method is based on the determination of a relative response factor (RRF). As mentioned above, in practice a calibration series is used. For each calibration work solution, the RRF shall be calculated:

$$RRF = \frac{A_{rivm} \cdot C_{is}}{C_{rivm} \cdot A_{is}}$$ with

RRF	=	relative response factor
A_{rivm}	=	peak area of the C10-C40 fraction in the calibration standard
C_{rivm}	=	concentration of NMI oil in calibration working solution (µg/ml)
A_{is}	=	peak area of the internal standard n-C40
C_{is}	=	concentration of the internal standard in the calibration work solution (µg/ml)

Within the linear area (see 8.1), the average RRF is calculated:

$$¿\, RRF \geq \frac{RRF\, i}{n}$$

with

< RRF	=	the mean relative response factor
RRFi	=	obtain the relative response factor for calibration working solution i
n	=	the number of calibration working solutions

Note: alternatively, the average RRF can be assimilated to the slope of the origin forced calibration duty, provided that the origin is within 95 % confidence curves.

7.2 MINERAL OIL CONTENT IN THE SAMPLES

The mineral oil content is given by the following formula: For water:

$$C = \frac{A \cdot G\, is}{A\, is \cdot <RRF> . V}$$

with

C	=	mineral oil content in µg/l
A	=	the peak area of the fraction C10-C40 in the sample
Ais	=	the peak area of the internal standard N-C40 in the sample
Gis	=	the amount of internal standard added to the sample (µg)
V	=	volume of extracted sample (l)
<RRF	=	the mean relative response factor

For soil and sediment:

$$C = \frac{A \cdot G\, is}{A\, is \cdot <RRF> . G}$$

with

C	=	mineral oil content in mg/kg dm
A	=	the peak area of the fraction C10-C40 in the sample
Ais	=	the peak area of the internal standard N-C40 in the sample
Gis	=	the amount of internal standard added to the sample (µg)
G	=	weight of sample extracted, calculated by dry weight (g dm)
<RRF>	=	the mean relative response factor

Note: for the calculation of the contents of the fractions >C10-C12, >C12-C20, >C20-C30 and C30–<C40, the surface percentage of each fraction is multiplied by the total mineral oil content. For the fractions, the reporting limit at convention is set at 25 µg/l for groundwater, 12.5 mg/kg dm for soil and 25 mg/kg dm for waste and construction material. The total mineral oil content (C10-C40) is considered as a separate fraction, calculated as indicated above and thus not obtained by summation of the sub-fractions. If the total mineral oil content is below the reporting limit, no sub-fractions shall be reported.

8 QUALITY CONTROL

For the quality requirements related to calibration, blank procedure, internal standard recovery, sensitivity inspection, inspection language and inspection standard, reference is made to CMA/6/D.

8.1 LINEARITY

The methodology for determining linearity is described in CMA Part 6.

If the peak area for a particular component in an injected sample preparation is higher than the highest area obtained in the most recent linearity test or calibration straight setting, a remeasurement shall be made on the original preparation after dilution.

8.2 GAS CHROMATOGRAPHIC CHARACTERISTICS

As mentioned under 6.3.1, a regular check on the separation of alkanes and non-discriminatory behaviour of the injector shall be carried out.

9 PERFORMANCE CHARACTERISTICS

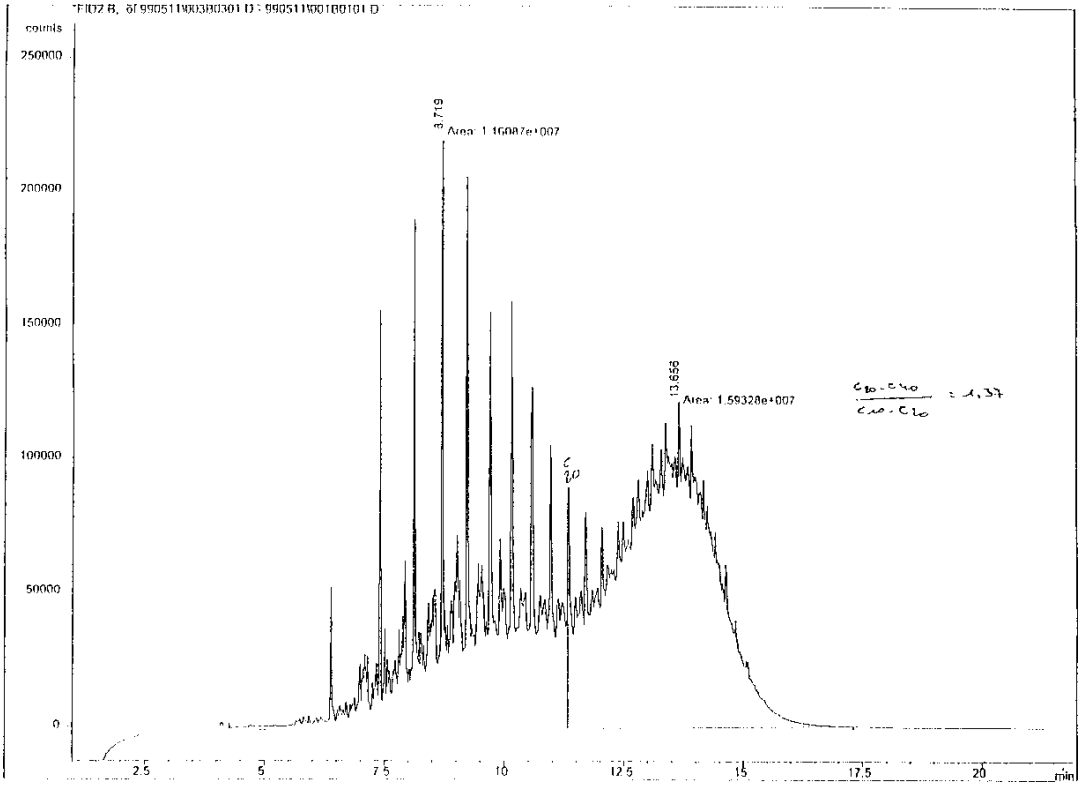
For the performance characteristics, see CMA Part 6.

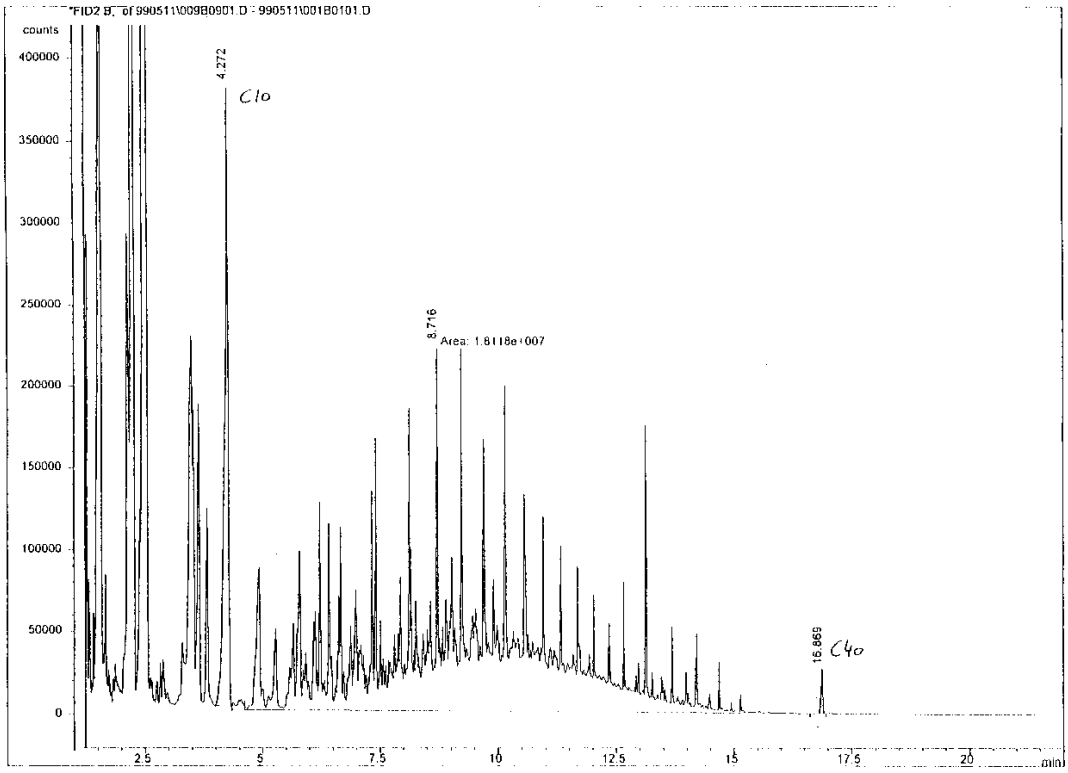
10 REFERENCES

- NEN 5733, Soil, Determining mineral oil content in soil and water soil with gas chromatography, 1997.
- NVN 6678, Water, Determining mineral oil content with gas chromatography, 1997.
- EN ISO 9377-4: Water Quality: Determining hydrocarbon oil index – part 4: Method using solvent extraction and gas chromatography, 1999.
- EN 14039: Characterisation of waste – Determining hydrocarbon content in the range C10 – C40 by gas chromatography, 2004.

ANNEX A

GC-FID chromatograms for NMI oil (A) and petrol and diesel pollution (B)





Soil

1 OBJECTIVE AND SCOPE

This procedure replaces procedure CMA/5/B.4 of March 2023.
This method describes the pretreatment of soil samples and is applicable for the determination of parameters described in point 3 'Homogenisation of the laboratory sample'. The method follows the sampling procedure CMA/1/A.1.
This method is also applicable to the consolidated part of water bottom (the solid part of water bottom). The method follows the sampling procedure CMA/1/A.4.

2 GENERAL COMMENTS

The preparation of the test specimens from the laboratory sample, received at the laboratory, has been carried out in a sequence of operations in such a way that the smallest quantities, prescribed in the analytical methods, are representative of the laboratory sample.

The general location, definitions, summary scheme and references are explained in procedure CMA/5/A.1. The various sample pretreatments are explained in separate procedures: homogenising (CMA/5/A.2), phase separation (CMA/5/A.3), drying (CMA/5/A.4), reducing particle size (CMA/5/A.5) and reducing sample size and part sampling (CMA/5/A.6). The CMA/5/A.7 procedure describes the devices and techniques that can be used for the successive operations. Some practical examples are developed in CMA/5/A.8 based on detailed diagrams.

During the various analytical steps, attention should be paid to the risk of contamination, especially in the determination of heavy metals. In order to reduce the general risk of contamination, work should be carried out in a dust-free atmosphere with extremely clean equipment and carefully washed glassware.

Soil samples may contain asbestos-suspected materials. The necessary precautions should be taken in this respect, such as extraction of the preparation equipment (with suitable filter and extraction rate of at least 0.5 m/s), personal protective measures, etc.

3 HOMOGENISATION OF THE LABORATORY SAMPLE

If the laboratory sample is not provided with the minimum required sample quantity (depending on the type of soil examination), this shall be stated in the analysis report.

Mixing samples shall be produced according to the instructions of the soil remediation expert. The latter is responsible for having only mixed samples taken from samples of the same geology and contamination degree. If the laboratory detects any discrepancies, this shall be recorded as a disclaimer on the report. There is no limitation in the number of samples to be merged.
Mixing samples can be produced in two possible ways:

1. Preferred method (retention of individual samples): Each individual pot is homogenised, then partial samples are taken proportionally to compose the composite sample;
2. All pots are poured out together, homogenised and a partial sample is taken.

The choice of method used is the responsibility of the soil remediation expert. The minimum quantity of the final composite sample shall be 400 g.

3.1 SOIL SURVEYS WITHIN THE SCOPE OF EXPLORATORY SOIL SURVEY (ESS), DESCRIPTIVE SOIL SURVEY (DSS) EN SOIL REMEDIATION PROJECTS (SRP)

For soil studies in the framework of ESS, DSS and SRP, a minimum sample of 375 ml (completely filling glass jar with a capacity of 375 ml!) must be supplied to the laboratory.

The laboratory sample shall be taken from the original container, spread and converted manually. Material extraneous to soil (CMA/2/II/A.11) and material suspect of containing asbestos removed. If, after visual assessment, particles larger than 4 mm are present, the sample shall be strained over a 4 mm mesh sieve. The sieve residue must be removed. The following test specimens/portions may apply:

(1) Dry residue, TOC, heavy metals
(2) Clay, pH
(3) Cyanide
(4) Mineral oil, PAH, OCP and PCB

Note 2: if cyanides are to be determined, the sample must be shielded from light.

Figure 1 provides an overview of the sample pretreatment steps to arrive at the final analysis portions.

For a VOC determination, the samples must be supplied in sealed liners or bushings, or suspended in methanol. If the standard analysis package (SAP) and the VOC are to be determined on the same sample, the sample should be taken in a sealed stopper or liner. The laboratory shall remove the test portion for the VOC test from the sampling canister/liner. Other parameters can be determined on the remaining quantity.

3.2 EXCAVATED SOIL FOR USE AS SOIL

For analyses of excavated soil for use as soil, the laboratory sample shall be pretreated in the same way as in soil studies carried out in the framework of ESS, DDS and SRP.

A minimum sample quantity of 375 ml (full filling of 375 ml glass jar!) must be provided to the laboratory. The same test specimens/portions may apply as for ESS, DDS and SRP.

Figure 1 provides an overview of the sample pretreatment steps to arrive at the final analytical portions.

For a VOC determination, the samples must be supplied in sealed liners or bushings, or suspended in methanol.

3.3 SOIL EXCAVATED FOR CONSTRUCTION SOIL USE OR IN SHAPE-RETAINING PRODUCT

If excavated soil needs to be assessed for construction soil use or in form-solid product, the total concentrations should be determined at the same soil fraction as for use as soil (i.e. soil fraction < 4 mm). A minimum sample quantity of 375 ml (full filling of 375 ml glass jar!) must be provided to the laboratory. Figure 1 provides an overview of the sample pretreatment steps to arrive at the final analytical portions.

For a VOC determination, the samples must be supplied in sealed liners or bushings, or suspended in methanol.

A sample of at least 2 litres (e.g. plastic container) must be supplied to carry out the shaking test of excavated soil for construction soil use or in a form-solid product. Conduct the leaching test on a separately pretreated sample, because the screening residue (> 4 mm) is removed from the soil sample for determination of the total concentration, regardless of the percentage by mass of this residue.

For analysis for leachability (shaking test cf. CMA/2/II/A.19) the complete sample must therefore be processed including waste > 4 mm. If a shaking test is required and the sample is too wet to sieve, it may be dried but at a temperature of less than or equal to 40 °C. In the presence of material suspected of containing asbestos, appropriate precautions shall be taken.
After straining using a sieve with mesh size 4 mm, the sieve residue shall be sorted out and the nature and mass of the fractions shall be described in the analysis report. Organic materials (plants/roots naturally present, twigs, twigs) and/or material suspected of containing asbestos are first removed from the sample and are not part of the test portion. Non-breakable material (e.g. metallic particles such as nuts, bolts, scrap) is removed and its weight and nature is recorded in the analysis report. The reduction of the sieve residue is only necessary if the sieve residue has a mass greater than 5 % (in relation to the original sample). The method of reduction (e.g. a jaw crusher) will also be documented and reported. Grinding is not permitted.

As a result of breaking and sieving, contamination of the material may occur in such a way that it affects the leaching of key components, e.g. Co and W in jaw crushers equipped in tungsten carbide, or Cr, Ni, Mo and V from stainless sample breakers or sieves.

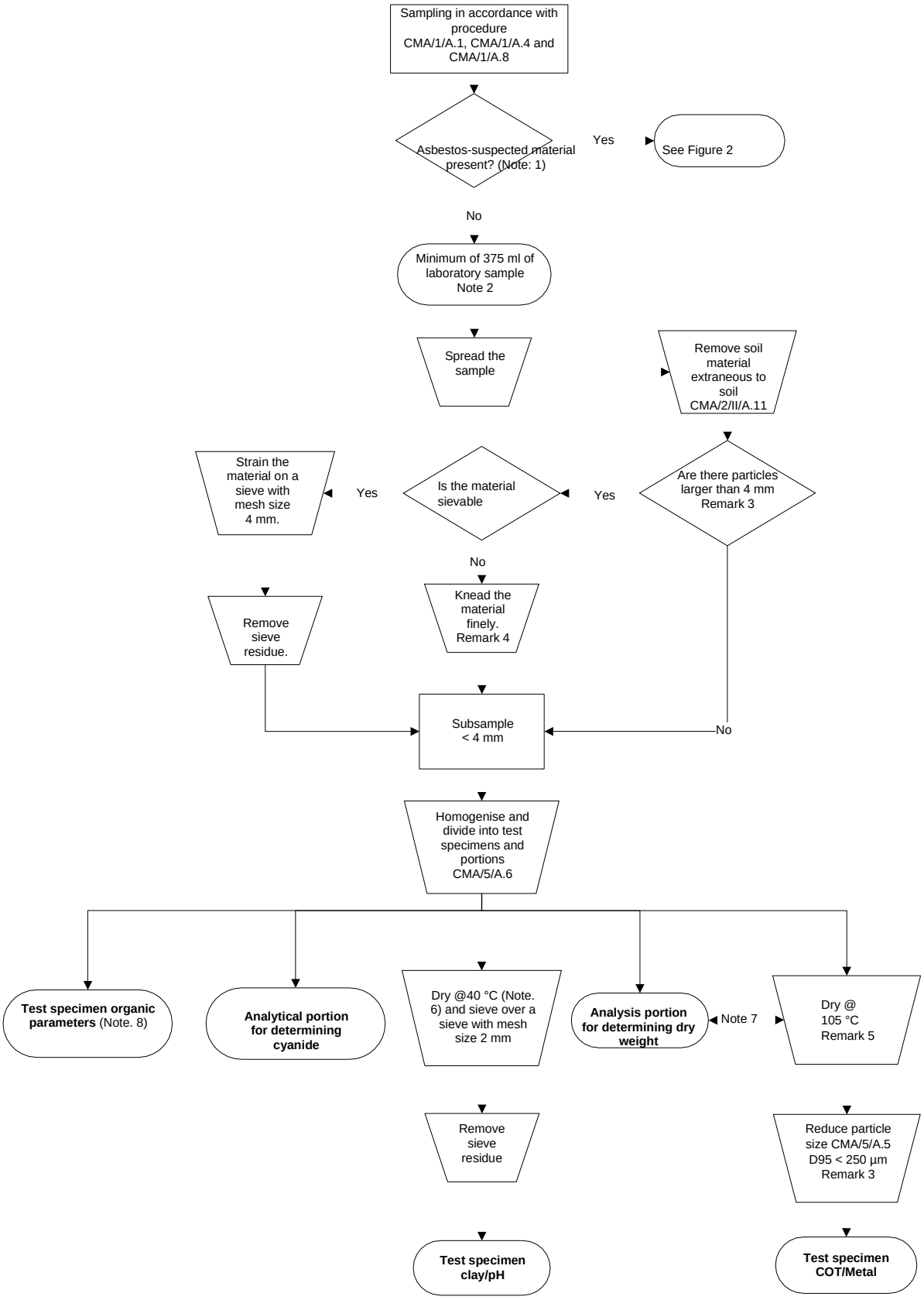


Figure 1: flowchart sample pretreatment Soil

Notes on Figure 1:

- Note 1: presence of asbestos sticker or visually detectable or prior-knowledge sample
- Note 2: a jar with volume 375 ml should be completely filled
- Note 3: the particle size distribution shall be determined visually.
- Note 4: specifically for clay samples, particles > 4 mm present are removed
- Note 5: other forms of drying as described in CMA are also permitted (e.g. utilisation)
- Note 6: the pH determination can also be carried out on an air-dried soil sample
- Note 7: If the sample is dried at 105 °C, this specimen may also be used for determining dry matter.
- Note 8: for the determination of PFAS, the sample should be reduced in a ball mill, knife mill or cryogenic grinding, and then sieved down to <0.5 mm (for a sample intake of <2 g) or <1 mm (for a sample intake of 2 to 10 g).

Figure 2 provides an overview of the sample pretreatment steps to arrive at the final analysis portion for the shake test.

Notes on Figure 2:

- Note 1: presence of asbestos sticker or visually detectable
- Note 2: a jar with volume 375 ml should be completely filled
- Note 3: The necessary precautions should be taken in this respect, such as extraction of the preparation equipment (with suitable filter and extraction rate of at least 0.5 m/s), personal protective measures, etc. The sample must not be pre-dried. Moistening the sample through atomisation is a possible option to minimise the spread of asbestos.
- Note 4: the particle size distribution shall be determined visually.
- Note 5: specifically for clay samples, particles > 4 mm present shall be removed
- Note 6: other forms of drying as described in CMA are also permitted (e.g. lyofilisation)
- Note 7: the pH determination can also be carried out on an air-dried soil sample
- Note 8: When the sample is dried, the drying oven shall be provided with the necessary extraction with an appropriate filter and an extraction rate of at least 0.5 m/s.
- Note 9: If the specimen is dried at 105 °C, it may also be used for determining dry matter.
- Note 10: duplicate analyses are required because the sample is not reduced to <250 µm and is therefore less homogeneous.
- Note 11: for the determination of PFAS, the sample should be reduced in a ball mill, knife mill or cryogenic grinding, and then sieved down to <0.5 mm (for a sample intake of <2 g) or <1 mm (for a sample intake of 2 to 10 g).

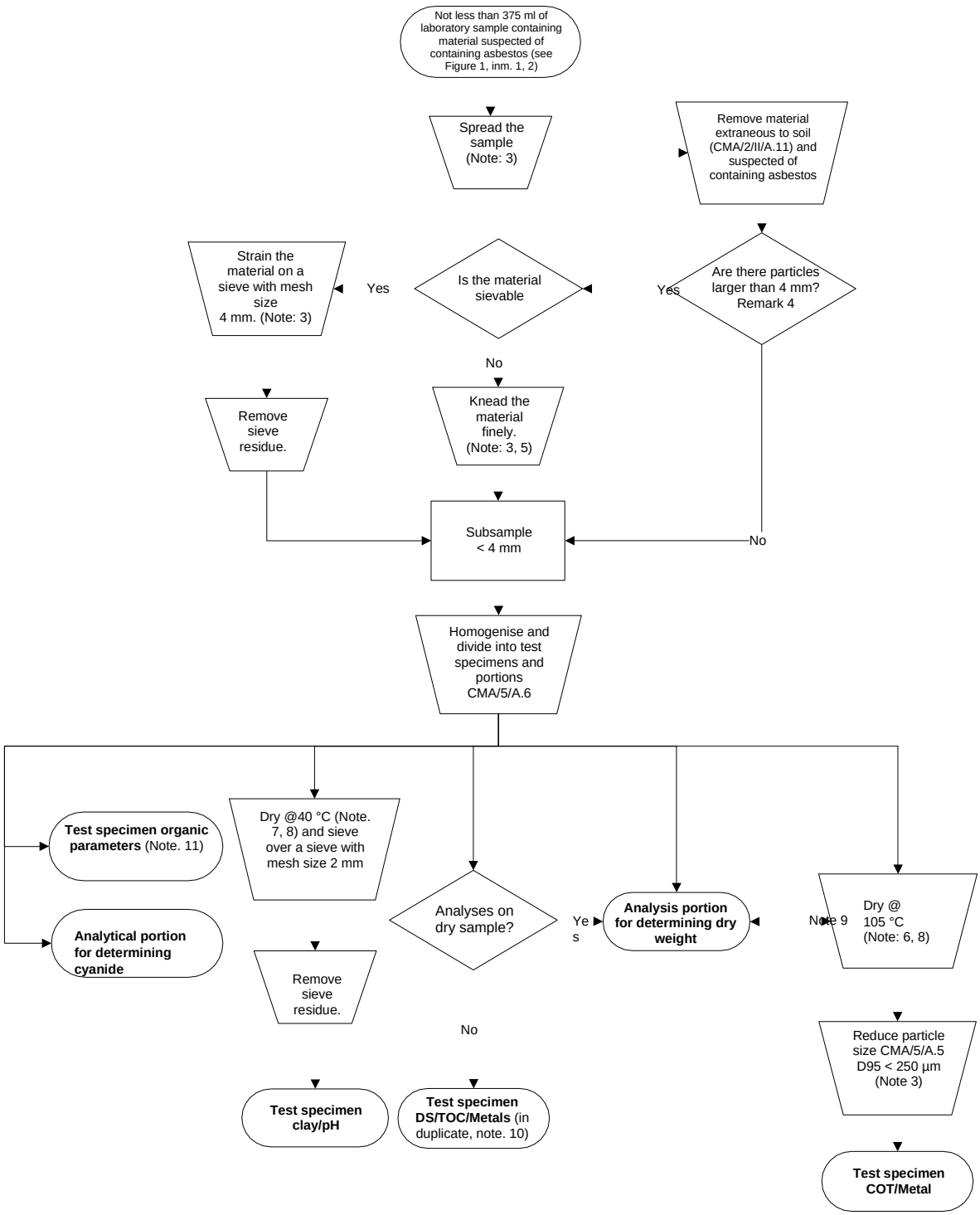


Figure 2: flowchart sample pretreatment of Soil with material suspected of containing asbestos

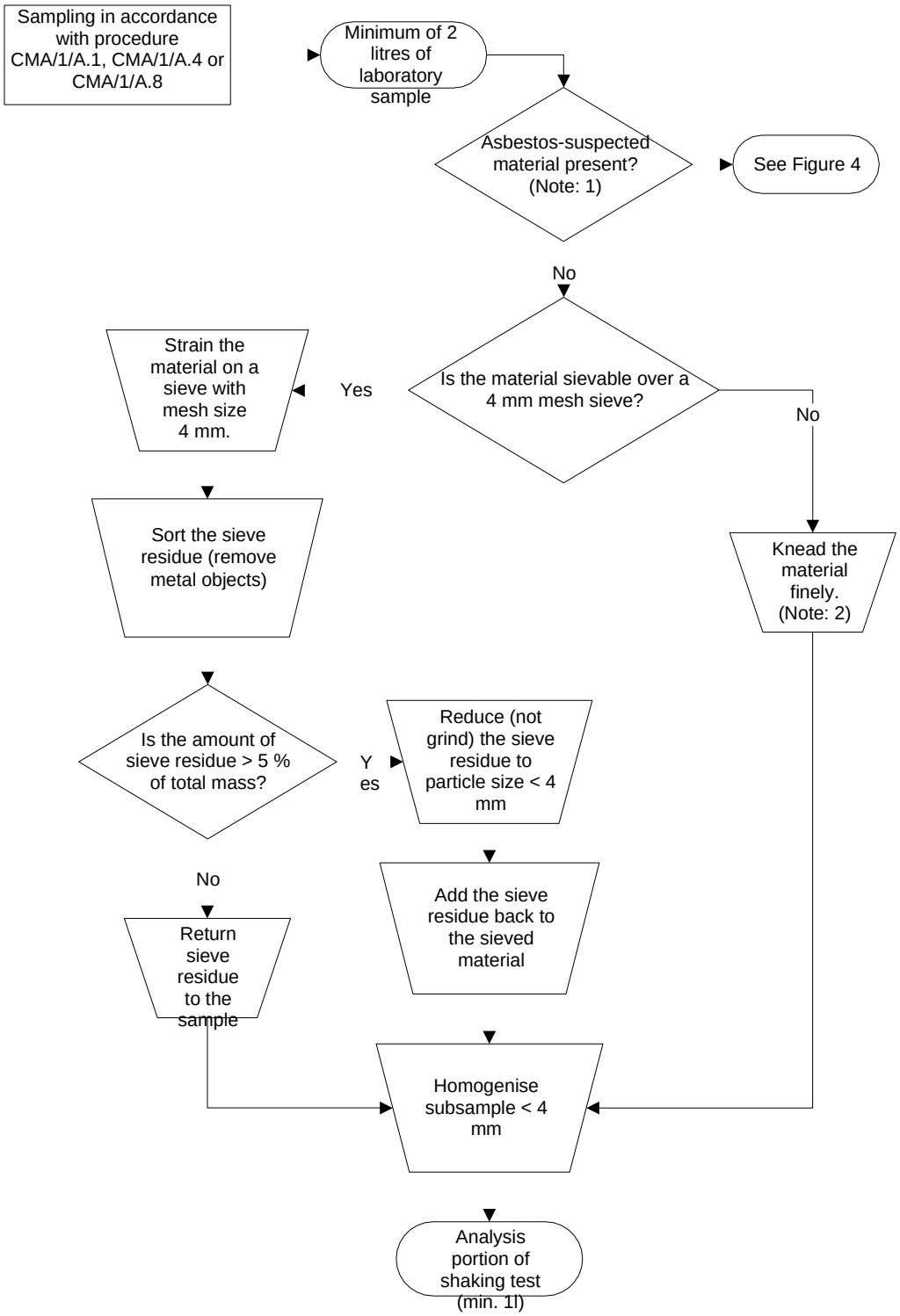


Figure 3: flowchart for sample pretreatment Excavated soil for constructional soil use for the performance of the shake test

Notes on Figure 3:

- Note 1: presence of asbestos sticker or visually detectable or prior-knowledge sample
- Note 2: specifically for clay samples, particles > 4 mm present are removed

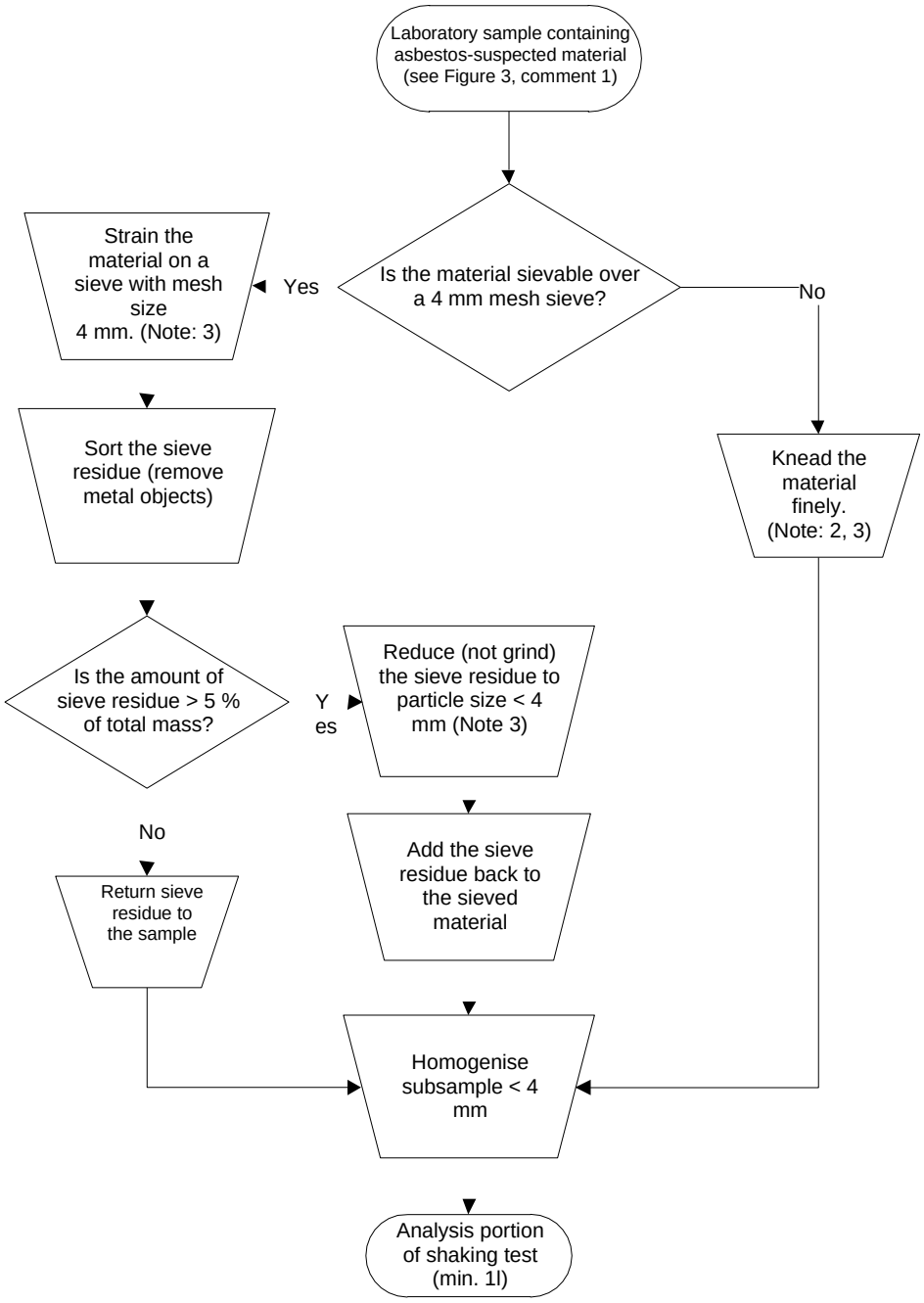


Figure 4: flowchart sample pretreatment Excavated soil with material suspected of containing asbestos for constructional soil use for performing the shake test

Notes on Figure 4:

- Note 1: presence of asbestos sticker or visually detectable
- Note 2: specifically for clay samples, particles > 4 mm present are removed
- Note 3: The necessary precautions should be taken in this respect, such as extraction of the preparation equipment (with suitable filter and extraction rate of at least 0.5 m/s), personal protective measures, etc. The sample must not be pre-dried. Moistening the sample through atomisation is a possible option to minimise the spread of asbestos.

4 PREPARING ANALYTICIAL SAMPLE

The various preparations of the specimen are described in Figures 1 to 4. For a description of the analysis, see the CMAs on inorganic (CMA/2/II/A.1-21) and organic analytical methods (CMA/3/A-V).

Shredder

1 OBJECTIVE AND SCOPE

This procedure replaces CMA/5/B.5 of July 2021.
This method describes the pretreatment of shredder samples, the sampling of which was carried out in accordance with CMA/1/A.15.

2 GENERAL COMMENTS

The preparation of test specimens from the laboratory sample has been carried out in a sequence of operations in such a way that the smallest quantities, prescribed in the analytical methods, are representative of the final sample.

The general location, definitions, summary scheme and references are explained in procedure CMA/5/A.1. The various sample pretreatments are explained in separate procedures: homogenising (CMA/5/A.2), phase separation (CMA/5/A.3), drying (CMA/5/A.4), reducing particle size (CMA/5/A.5) and reducing sample size and part sampling (CMA/5/A.6). The CMA/5/A.7 procedure describes the devices and techniques that can be used for the successive operations. CMA/5/A.8 develops some practical examples based on detailed schemes and CMA/5/A.9 describes the minimum sample size for heterogeneous wastes.

During the various analytical steps, attention should be paid to the risk of contamination, especially in the determination of heavy metals. In order to reduce the general risk of contamination, work should be carried out in a dust-free atmosphere with extremely clean equipment and carefully washed glassware.

Due to the possible diversity of shredder material, each pretreatment carried out in the analysis report must be described accurately. Especially when the standard procedure is deviated from (see Figure 1).

3 HOMOGENISATION OF THE LABORATORY SAMPLE

Pre-drying of the laboratory sample is allowed at a maximum temperature of 40 °C. However, the moisture content should be taken into account. The sample shall be inspected visually at the start of sample pretreatment. If there is hard material such as metal or stones, these must be removed by sorting. After sorting, the various political groups are described in the report in terms of nature and mass.

4 PREPARING THE TEST SPECIMEN

Figure 1 shows in a flowchart which pretreatments are to be carried out in order to produce representative test specimens and portions for subsequent analysis packages (cf. CMA/6/A):

- Burn package A.4
- Insert package A.5

These packages contain the following relevant analyses:

- Shaking test and column test;
- Loss of ignition and dry residue;
- COT/Caloric value/Cl,F, S and metals;
- PACs/PCBs/EOX/mineral oil.

For a description of the analysis, see the CMAs on inorganic (CMA/2/II/A.1-21) and organic analytical methods (CMA/3/A-V) Notes on Figure 1:

Note 1: pre-drying at a max. of 40 °C is permitted. Take moisture content into account. If oil pollution is detected on the basis of odour and/or vision, a 200 ml partial sample should be taken and treated with an appropriate extraction solvent. The extract is stored as a sample.

Note 2: the nature and mass of the sorted fractions must always be reported.

Note 3: estimation is done by visual assessment.

Note 4: examine after sieving

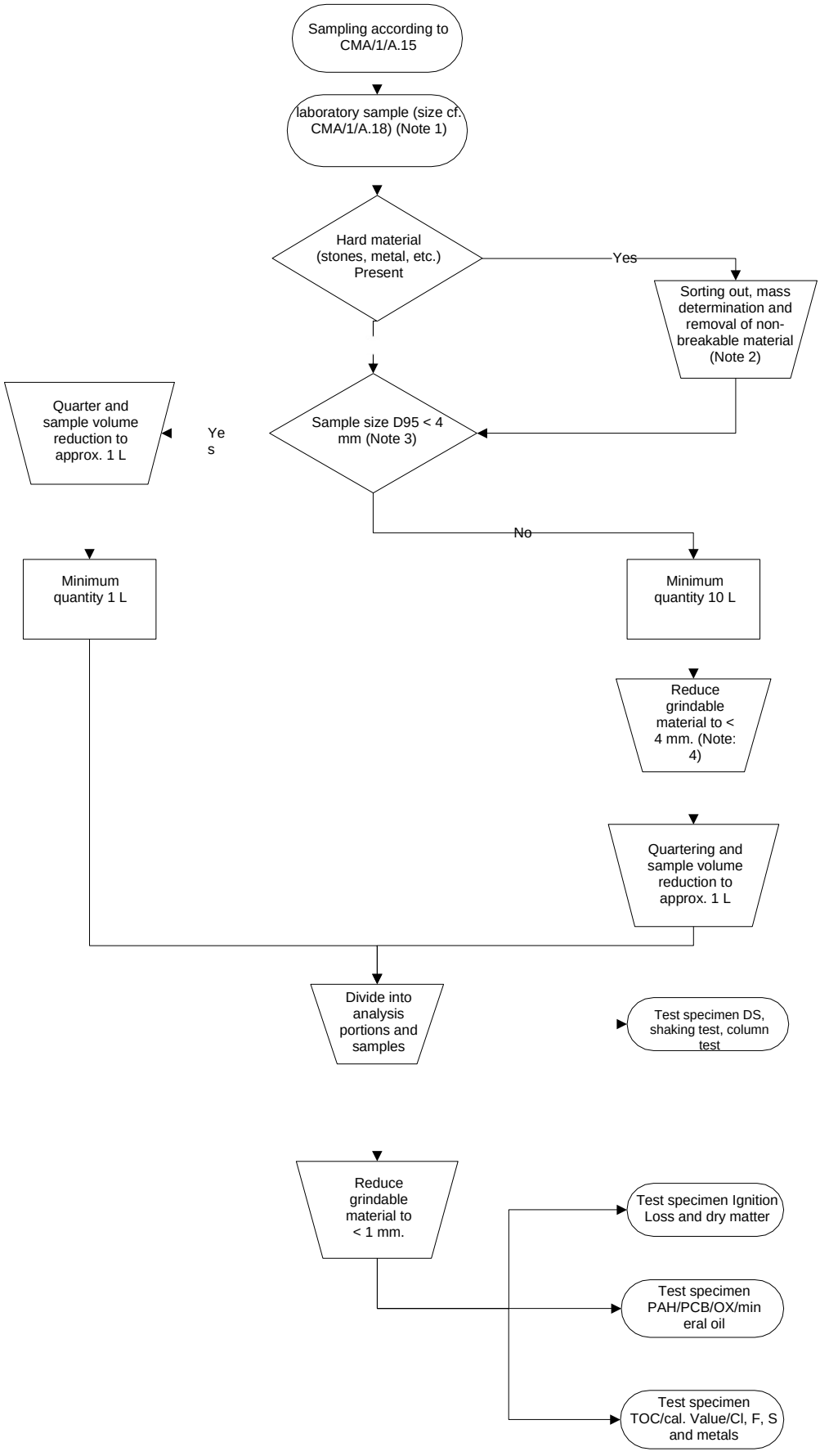


Figure 1 Flowchart sample pretreatment for shredder

5 VARIETIES OF SHREDDER

The following photographs illustrate the physical differences between shredder samples of various origins.

- Fine fluff

- Coarse

- Coarse

The photographs indicate that with certain samples, the pre-sorting and removal of inert non-reduced material can take a considerable time, while others are delivered fairly homogeneously. It is extremely important to describe different fractions properly in terms of nature and mass.

Granulates/ashes and slags

1 OBJECTIVE AND SCOPE

This procedure replaces the procedure of June 2024. This method describes the pretreatment of granulates/ashes and slags the sampling of which was carried out in accordance with CMA/1/A.15 and CMA/1/A.18.

2 GENERAL COMMENTS

If determining **VOCs and solvents (aspecific)** is required, a separate laboratory sample shall be provided.

The preparation of test specimens from the laboratory sample has been carried out in a sequence of operations in such a way that the smallest quantities, prescribed in the analytical methods, are representative of the final sample.

The general location, definitions, summary scheme and references are explained in procedure CMA/5/A.1. The various sample pretreatments are explained in separate procedures, namely homogenising (CMA/5/A.2), drying (CMA/5/A.4), reducing particle size (CMA/5/A.5) and reducing sample size and subsampling (CMA/5/A.6). The CMA/5/A.7 procedure describes the devices and techniques that can be used for the successive operations. CMA/5/A.8 develops some practical examples based on detailed schemes and CMA/5/A.9 describes the minimum sample size for heterogeneous wastes.

During the various analytical steps, attention should be paid to the risk of contamination, especially in the determination of heavy metals. In order to reduce the general risk of contamination, work should be carried out in a dust-free atmosphere with extremely clean equipment and carefully washed glassware.

These streams may contain asbestos-suspected materials. In addition, recycled granules of concrete and/or masonry and/or debris must always be treated as material suspected of containing asbestos Appropriate precautions must be taken for this purpose, such as extracting the preparation equipment (with suitable filter and extraction rate of at least 0.5 m/s), personal protective measures, etc.

Due to the possible diversity of the samples, each pretreatment carried out in the analysis report must be accurately described. Particularly when the standard procedure is deviated from (see Figure 1 and Figure 2).

3 HOMOGENISATION OF THE LABORATORY SAMPLE

If the sample does not contain material suspected of containing asbestos (absence of asbestos sticker or visually detectable or prior knowledge sample) and if the parameter cyanide is not to be determined, pre-drying of the laboratory sample is allowed at a maximum temperature of 40 °C. The moisture content should be taken into account. The sample shall be inspected visually at the start of sample pretreatment. If there is material that cannot be broken with the jaw crusher such as: metal, wood, organic materials, etc..., this must be removed by sorting. After sorting, the various political groups are described in the report in terms of nature and mass.

4 PREPARING THE TEST SPECIMEN

In Figure 1, a flowchart presents which pretreatments are to be carried out in order to produce representative test specimens and portions for subsequent analysis packages (cf. CMA/6/A) having as relevant parameters:

- Package A.3 use as building material
    - Dry matter
    - TOC, metals
    - Cyanide: free cyanide, non-chlorine oxidisable cyanides
    - Column test (metals and anions)
    - BTEXS, alkanes, PAH (16), mineral oil, PCB (7)
    - PFAS
- Burn package A.4
    - Dry matter and ignition loss
    - TOC
- Insert package A.5
    - Dry matter and ignition loss
    - Mineral oil, EOX, solvents (aspecific)
    - TOC
    - Single shake test and column test
    - Metals
    - Fluoride
    - Free cyanides
    - BTEXS
    - PAH (16)
    - PCB (7)

Notice: For the parameters solvents (aspecific), alkanes, MAK, max. availability and diffusion test, separate laboratory samples are provided (see § 2).

In the presence of material suspected of containing asbestos, the various pretreatment steps are described in Figure 2.

For a description of carrying out the analyses, reference is made to the relevant CMA methods.

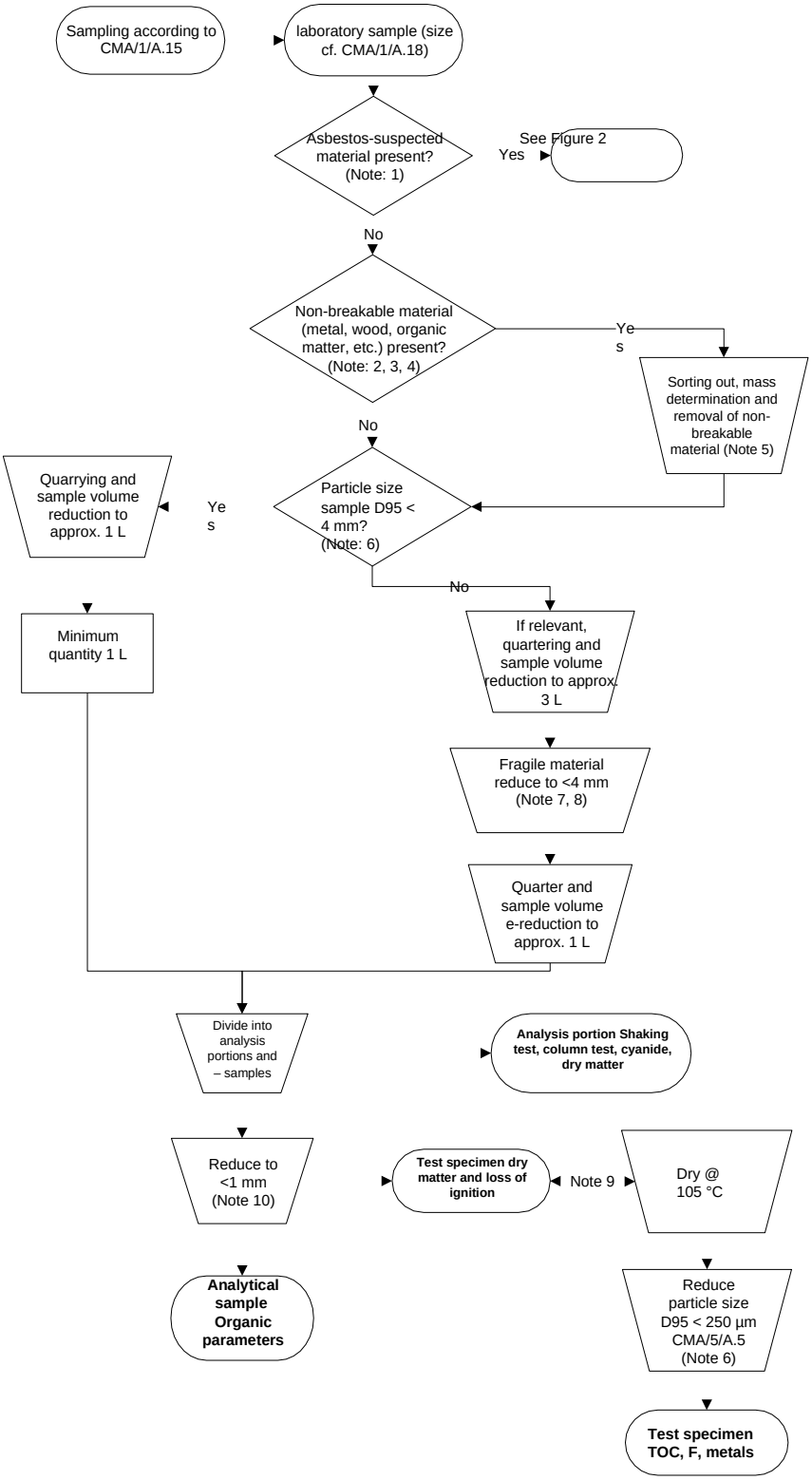


Figure 1 Flowchart sample pretreatment for granulates/ashes and slags

Notes on Figure 1:

- Note 1: presence of asbestos sticker or visually detectable or prior knowledge sample.
- Note 2: pre-drying at max. 40 °C is allowed, if no cyanide determination is required. Take moisture content into account.
- Note 3: If cyanide should be determined and pre-dried at max. 40°C is necessary for further reduction of the sample, this should be stated on the report.
- Note 4: The following shall be understood: material that cannot be broken with the jaw crusher.
- Note 5: the nature and mass of the sorted-out fractions must always be reported.

- Note 6: estimation is performed by visual assessment.
- Note 7: For the performance of the shake test and the column test, the grain size may affect the result. The material may only be reduced with a jaw crusher and should under no circumstances be broken too finely.
- Note 8: examination after sieving. For the purpose of this method, at least 95 % (mass) of the test sample must have a particle size less than 4 mm. For this purpose, the material (laboratory sample) is sieved and the sieve residue is weighed. If the sieve residue is less than 5 % (mass), the sieve residue shall be added back to the sieve pass-through and homogenised. If the coarser material (sieve residue) is more than 5 % (mass), the entire coarser fraction shall be reduced.
- Note 9: If the sample is dried at 105 °C, this specimen may also be used for determining dry matter.
- Note 10: for the analysis of PFAS, the sample should be reduced in a ball mill, knife mill or cryogenic grinding to <0.5 mm (for a sample intake of <2 g) or <1 mm (for a sample intake of 2 to 10 g).

Notes on Figure 2:

- Note 2: The following shall be understood: material that cannot be broken with the jaw crusher.
- Note 3: The nature and mass of the sorted fractions must always be reported.
- Note 4: The necessary precautions should be taken in this respect, such as extraction of the preparation equipment (with suitable filter and extraction rate of at least 0.5 m/s), personal protective measures, etc. The sample must not be pre-dried. Moistening the sample through atomisation is a possible option to minimise the spread of asbestos.
- Note 5: estimation is done by visual assessment.
- Note 6: For the performance of the shake test and the column test, the grain size may affect the result. The material may only be reduced with a jaw crusher and should under no circumstances be broken too finely.
- Note 7: examination after sieving. For the purpose of this method, at least 95 % (mass) of the test sample must have a particle size less than 4 mm. For this purpose, the material (laboratory sample) is sieved and the sieve residue is weighed. If the sieve residue is less than 5 % (mass), the sieve residue shall be added back to the sieve pass-through and homogenised. If the coarser material (sieve residue) is more than 5 % (mass), the entire coarser fraction shall be reduced.
- Note 8: If the specimen is dried at 105 °C, this specimen may also be used for determining dry matter.
- Note 9: use closed grinding systems only and always open using extraction (with suitable filter and extraction rate of at least 0.5 m/s), personal
  protective measures, ...
- Note 10: When the sample is dried, the drying oven shall be provided with the necessary extraction with an appropriate filter and an extraction rate of at least 0.5 m/s.
- Note 11: duplicate analyses are required because the sample is not reduced to <250 µm and is therefore less homogeneous.
- Note 12: for the analysis of PFAS, the sample should be reduced in a ball mill, knife mill or cryogenic grinding to <0.5 mm (for a sample intake of <2 g) or <1 mm (for a sample intake of 2 to 10 g).

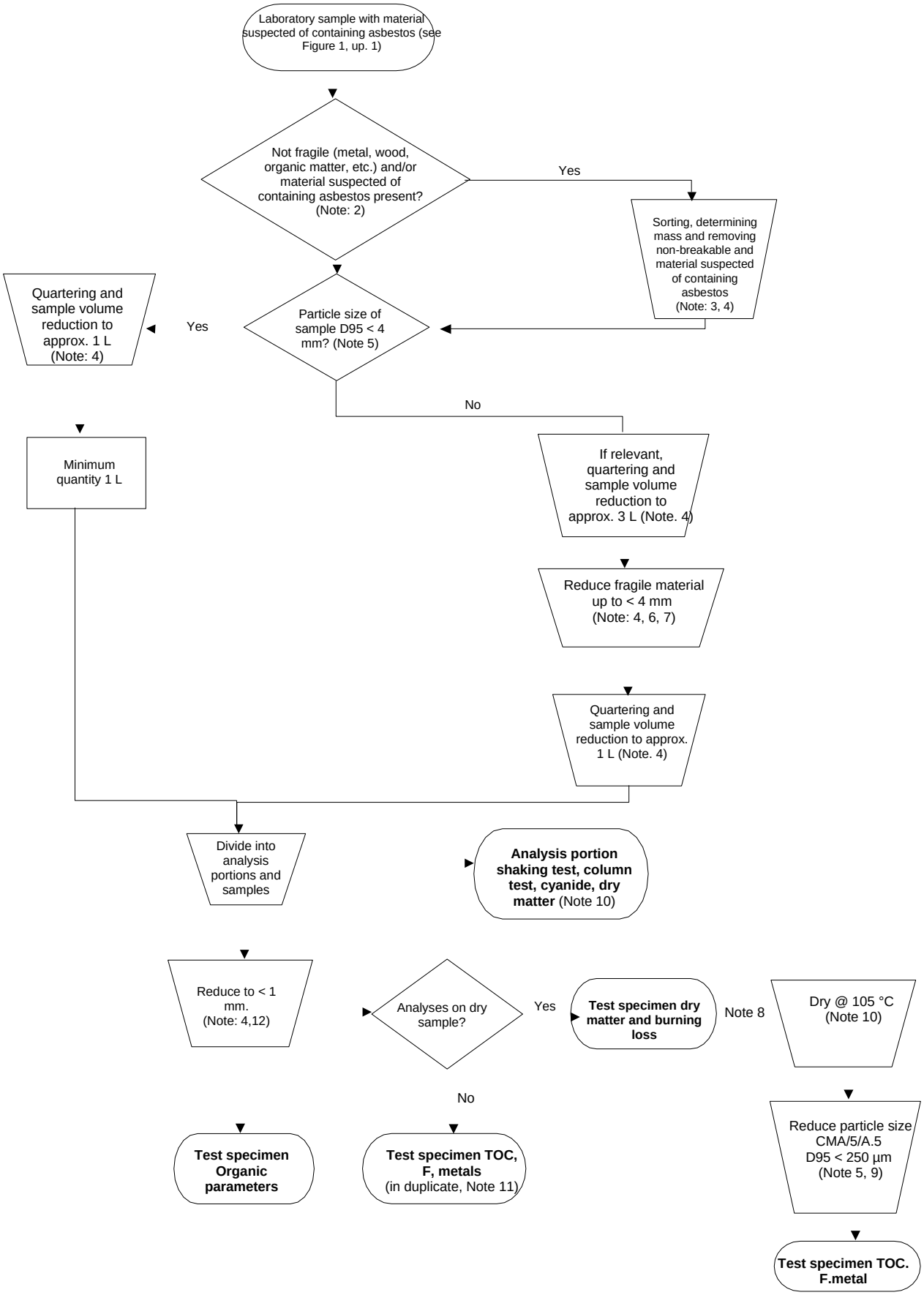


Figure 2 Flowchart sample preparation for granulates/ashes and slags with material suspected of containing asbestos

5 EXAMPLES

Figure 3 Left: Concrete rubble (0-20 mm); Right: sieve residue concrete rubble larger than 4 mm

Figure 4 Left: Crushed concrete debris < 4 mm; Right: crushed concrete debris < 1 mm

Performance Characteristics

CONTENTS

1 OBJECTIVE AND SCOPE

This procedure replaces procedure CMA/6/A of June 2024.
The scope has been extended to cover all methods of analysis. The methodological equivalence approach is no longer included, since a statistical approach to equivalence entails a number of limitations in terms of matrices, concentration ranges, etc. Expert opinion is decisive for judging the equivalence of analytical methods.

Analysis results are only approximations to the true value. The extent to which analysis results may differ shall be quantified with performance characteristics. They reflect the performance of an analytical method under different circumstances. The social importance of measurements makes it necessary to have sufficient insight into the possibilities and limitations of the analytical methods used.

The validation of an analytical method is defined as demonstrating that the (described) analytical method is appropriate for the intended application. This implies the determination of the relevant performance characteristics of the analytical method and the assessment of suitability for the intended purpose. This is worded as follows in ISO 8402: 'confirmation by examination and provision of objective evidence that the particular requirements for a specified intended use are fulfilled'. It should be noted that in current practice, there is not always clarity regarding external requirements (from regulations, contracts, etc.) and their status.

Methods shall be validated whenever necessary to verify that the performance characteristics are suitable for application in the event of a specific analytical problem. This is the case, for example, for new analytical methods developed for a particular purpose, but also for analysis methods already validated that are being adapted or extended, which, according to quality inspection, change over time, which are used in another laboratory or by other analysts or with other equipment, etc. The performance characteristics obtained are therefore valid only for the laboratory concerned, and the performance conditions (analysis method, etc.) should hereby be laid down. The extent of a validation/revalidation depends on the degree of change. Eurachem and the national accreditation bodies such as Belac argue that a minimum validation is always appropriate, even if a standardised analytical method is applied.

With regard to the parameters to be validated, a distinction can be made between qualitative analytical methods, high-level quantitative analysis methods and low concentration quantitative analytical methods. The majority of environmental analysis methods belong to the latter category and, consequently, this procedure also focuses mainly on this type of method. In general, a validation such as this should always pay maximum attention to accuracy and precision and, where relevant, to the limit of detection.

A separate report shall be made of each validation study, which will comply with predefined rules in terms of structure, content, assessment, approval and archiving.

This procedure only deals with the intra-laboratory validation of analytical methods for physical and chemical univariate quantities. For determining the performance characteristics for a group of laboratories, procedures are provided, inter alia, in ISO 5725; the provision of such performance characteristics requires inter-laboratory studies.

2 TERMS AND DEFINITIONS

2.1 TRUENESS

The correctness of an analytical method is the degree of correspondence between the average of a set of measurement values and the actual value or the actual assumed value of the quantity to be determined (cf. ISO 3534-1). The usual measure of accuracy is bias b, which therefore corresponds to the (positive or negative) systematic error[1].

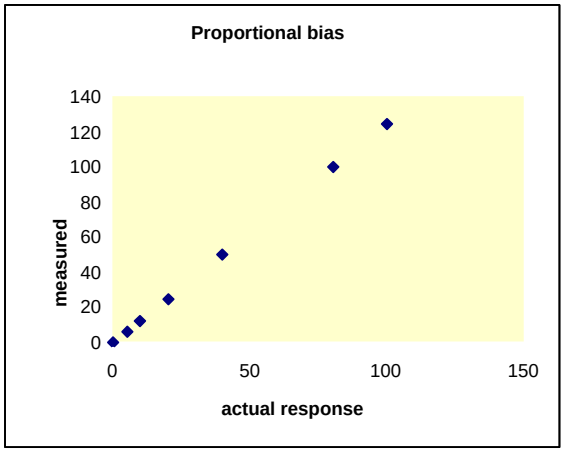
The reference of correctness to the term accuracy, precision or a derivative of this is discouraged by Eurachem. In their most usual sense, these terms include a combination of 'random' components and the 'bias' component, so they are more consistent with the term measurement uncertainty.

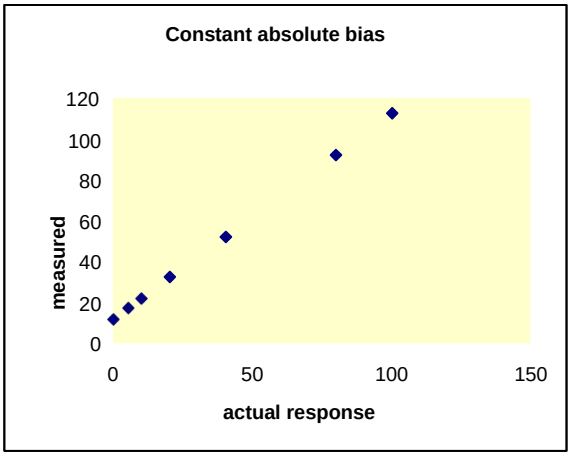
With the term recovery T, the fraction of the component found during analysis is recovered, after the addition of a known amount of component to the sample under defined conditions. This document considers recovery experiments as one of the possible methods of determining the correctness of an analytical method and does not systematically distinguish between the terms accuracy and recovery.

In the event that a (reference) method is imposed for the measurement of a specified quantity, accuracy may also be interpreted as the degree of correspondence between the laboratory's measurement value and the actual assumed value of the quantity to be determined with the same method (usually following an interlaboratory comparison). In this case, bias is said to be relative compared with a method average. The choice of bias to be reported should be clear from the context of the analysis request and, if necessary, agreed with the customer.

A further division can be made between two cases of bias, namely constant absolute bias and proportional bias. In the case of proportional bias, the relative deviation at each concentration level is constant and expressed as a percentage. In the case of absolute constant bias, the deviation at each concentration level, expressed in absolute terms, is the same. The graphs below illustrate both cases of trueness effects relative to the ideal case (diagonal). For higher contents, the proportional part of the trueness usually dominates the constant section. Proportional trueness effects play a minor role for contents close to the limit of detection.

[1] In principle, the systematic deviation is an average that is the result of an infinite number of measurements of the same measurement quantity, performed under repeatability conditions, less the true value of the measurement quantity (cf. NPR 2814).





2.2 PRECISION

The precision of an analytical method is the degree of dispersion in the analysis results obtained by carrying out the analytical method a repeated number of times under defined conditions on the same sample (cf. ISO 3534-1). The usual degree of precision is the standard deviation or coefficient of variation (relative standard deviation) of the test results, i.e. precision is expressed as imprecision. The conditions are normally referred to as repeatability and reproducibility.

2.3 REPEATABILITY

Repeatability refers to precision obtained when all the relevant measurements are carried out by the same analyst, using the same measuring equipment, as close as possible to each other (cf. ISO 3534-1). The usual measure is the repeatability standard deviation s_r or the repeatability coefficient CV_r.

2.4 REPRODUCIBILITY

Reproducibility refers to precision obtained by performing all relevant measurements under variable conditions, i.e. in different laboratory rooms, by different analysts, with different apparatus and batches of reagents/standards, at different times at larger intervals (cf. ISO 3534-1). Any ruling on reproducibility must therefore indicate which conditions have been changed.

This procedure focuses only on reproducibility within a given laboratory (shortly intra-reproducibility), and as a minimum requirement for this, the factor of time is varied, i.e. that the relevant analyses are carried out on different days and in different analytical series (time-dependent intermediate precision). The usual degree is the intra-reproducibility standard deviation s_R or the intra-reproducibility coefficient of variation CV_R.

2.5 EXPANDED UNCERTAINTY

The expanded uncertainty U is defined as the half the length of an interval within which the true value is expected to lie, at a certain level of confidence. In ISO GUM, this is worded as follows: 'Quantity Defining an interval about a result of a measurement that may be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand'.

In principle, the measurement uncertainty should include all factors affecting the result, combined according to established procedures. Relevant factors are:

- the sample retention in the laboratory;
- initial sample pretreatment (e.g. homogenisation, drying, subsampling, etc.);
- sample preparation (e.g. extraction, extraction, purification, etc.);
- the actual measurement of the preparation (e.g. calibration, interference, etc.) and
- the calculation of the analysis result (e.g. Corrections, etc.).

In the context of this procedure, it has been assumed that the measurement uncertainty is limited to the actual laboratory event (i.e. the five steps mentioned above) and that factors such as the sampling itself (e.g. representativeness, etc.) and the transport of the sample to the laboratory (e.g. preservation, etc.) are not necessarily included in the measurement uncertainty.

The performance characteristics that determine the measurement uncertainty – or at least part of it – are the intra-reproducibility of the analytical method (in terms of accidental deviation) and the correctness of the analytical method (as regards the systematic deviation). By including a factor (coverage factor) in the accidental deviation, the probability that the true value lies in the interval is sufficiently large.

Note that measurement uncertainty is not a performance reference, but a reference of a measurement value. Measurement uncertainty is also examined in a separate section for this reason (see CMA/6/B).

2.6 SELECTIVITY AND SPECIFICITY

The selectivity of an analytical method is the dependence/independence of a quantity other than the measurement quantity, i.e. the extent to which it can distinguish the component to be determined from other components in a mixture or matrix (cf. IUPAC, draft NEN 7777). In a sufficiently selective analysis, the determination conditions shall be selected in such a way that contributions from other components are eliminated or fall within the margin of uncertainty. These potential contributions concern both interferences (due to quantities that cause a signal themselves) and matrix effects (due to quantities that change the signal of the measurement quantity).

An analytical method is only specified if it responds to the component to be determined (cf. AOAC, IUPAC). Specificity can therefore be considered the ultimate selectivity. The use of this term is discouraged by IUPAC.

2.7 LIMIT OF DETECTION

The limit of detection LD (also called detection limit) is the smallest amount of substance or concentration of the component in the sample that can be demonstrated by a certain (and reasonable) statistical probability by the analytical method, i.e. the presence of which can still be established with a certain certainty/uncertainty (cf. AOAC, IUPAC). It is therefore a qualitative

criterion.

In the usual operational definition, the probability aspect is taken into account by equating the detection limit to 3 times the standard deviation at this level (cf. IUPAC). The limit of detection is thus the value of the measurement quantity where the coefficient of variation at convention is 33 %. For more background information, please refer to draft NEN 7777 and the references it contains.

2.8 LIMIT OF QUANTIFICATION

The limit of quantification, LOQ, is defined as the smallest amount of substance or lowest concentration of the component in the sample that can be quantified with a certain (and reasonable) precision and trueness by the analytical method, i.e. the measurement value of which can still be determined with a certain certainty/uncertainty. Unlike the limit of detection, the limit of determination is a quantitative criterion.

Pragmatically, the limit of determination for the purposes of this procedure is equal to 6 times the standard deviation in units of the measurement quantity. Consequently, the limit of determination is that of the measurement quantity where the coefficient of variation is 17 %.

2.9 LINEARITY

The linearity of an analytical method is the characteristic that, within defined limits, there is a linear relationship between the response and the amount (concentration) of the component to be determined. The linear area is the corresponding working area.

If linearity is not met, the operating range shall be narrowed or switched to another (e.g. quadratic) function between response and amount (concentration) of the component to be determined.

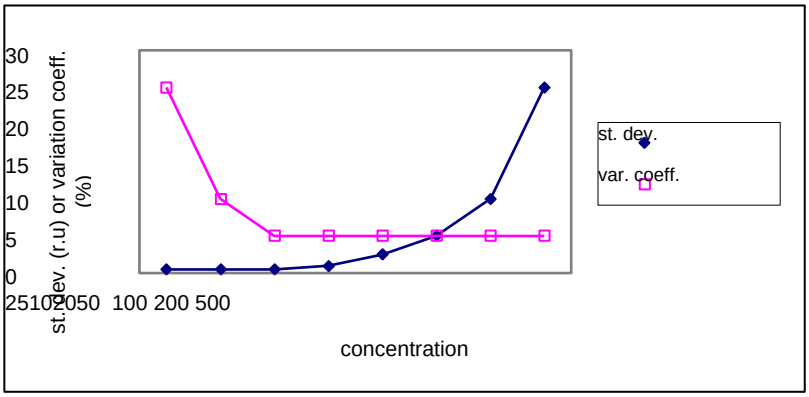
The concept of model deviation generally refers to the 'lack of fit' of the
the relationship between measurement magnitude and response.

2.10 RANGE, MEASURING RANGE, WORKING RANGE

As an extrapolation of the IUPAC definition which is instrument-oriented, the working range of an analytical method is defined as the interval between the smallest and the largest amount (concentration) of the component to be determined for which the analytical method has been validated, i.e. within which the performance characteristics meet defined requirements.

It follows that a working range is usually predetermined at the beginning of the validation study, taking into account e.g. device data, expected levels in samples, applicable regulations, etc. In an optimal working range, current concentrations can be measured easily, and critical (cf. regulations) concentrations are not entirely at the extremes of the working range.

A working range is called homogeneous if the standard deviation is constant over the entire working range (homoscedasticity or homogeneity of variances). In practice, this is rarely the case, except for small working areas; usually, in the upper part of the working area, the relative standard deviation is constant (coefficient of variation) and, in other words, the standard deviation increases proportionally with concentration.



r.u.: unit response

2.11 RUGGEDNESS, ROBUSTNESS

Robustness of a method means the analysis result's lack of sensitivity to minor variations in implementation, conditions and quality of waste as they may occur in practice (cf. AOAC, draft NEN 7777).

2.12 ANALYSIS:

In the context of validation experiments, the concept of analysis, as further stated in the text, amounts to carrying out as faithfully as possible the complete set of steps that a routine sample undergoes in the laboratory, from the usual sample pretreatment (subsampling, etc.) to the measurement and calculation. In this way, performance characteristics and measurement uncertainty encompass the entire laboratory activity and correctly characterise the laboratory's method of analysis or measurement values.
As regards sample pretreatment, the following situations typically occur:

- in routine, the laboratory sample as a whole undergoes a certain sample pretreatment (homogenisation, drying, etc.) before a portion of analysis is taken: in this case, validation experiments shall be based on a new portion of the sample initially pretreated for each determination and that portion will continue to follow in full the method used for routine samples;
- in routine, a subsample shall be taken from the laboratory sample in a specified manner, which then undergoes further pretreatment (such as drying) or is used directly in the sample preparation: in this case, validation experiments shall be based on a new subsample obtained in the usual manner from the laboratory sample for each determination and will follow in full the method used for routine samples.

For the determination of parameters for which the whole sample is to be treated or that require immediate analysis (e.g. suspended matter, biological oxygen consumption, volatile organic compounds, etc.), it may be necessary to provide - in the context of validation experiments - several subsamples during sampling.

3 GENERAL VALIDATION PLAN

In general, validation studies follow the following steps (cf. draft NEN 7777):

- determine the performance characteristics to be determined based on the purpose and status of the analytical method;
- determine the scope (matrices and working area) for which the analytical method is to be validated, and how many sub-areas should be practically split;
- verify that external requirements apply to the performance characteristics – partly or otherwise – and, if so, what form they take;
- identify the samples needed for the validation study;
- carry out the validation study;
- assess the defined performance compared to any external requirements, or (in the absence of external quantitative requirements) directly in relation to the intended use;
- report the results in a validation report.

3.1 SELECTING PERFORMANCE CHARACTERISTICS TO BE DETERMINED

What performance characteristics should be determined depends mainly on the status of the analytical method that one wishes to perform.

A complete validation is required for entirely new analytical methods (i.e. fully developed by the laboratory itself and not previously validated). This always includes the following performance characteristics:

- working area;
- trueness
- intra-reproducibility;
- selectivity;
- robustness.

where relevant, it will further include:

- limit of detection and quantitation;
- repeatability;
- linearity, or more general model deviation.

In most cases, however, laboratories will prefer to adopt the reference methods. These are methods of analysis laid down by law or recommended by the competent authority, laid down, for example, in a compendium or in international standards. If such an analytical method is adopted without modification, it will in principle be sufficient to demonstrate that the essential performance characteristics are consistent with the documented and/or intended use. The validation to be carried out will include at least:

- trueness
- intra-reproducibility.

and, where relevant, also:

- working area;
- limit of detection and quantitation;
- repeatability;
- selectivity.

A modified analytical method, i.e. an analytical method that has already been validated but in which a change has been made, requires a revalidation aimed at those performance characteristics that can reasonably be affected by the change.

3.2 DETERMINING THE SCOPE ANY SUBAREAS WITH THE VALIDATION

Performance characteristics only have meaning for unambiguously defined analytical objects and analysis results. If the analysis procedure does not provide sufficient clarity, a clear interpretation should be drawn up and included in the validation report prior to validation. This applies, for example, to the number of underlying measurements of an analysis result (one or average) and to any corrections applied (for extraction standards, procedure blank, etc.). The intended scope of application should also be specified in advance, i.e. which matrices (including possible interferences) and which work area (possibly per matrix).

The starting point is that in validation, similar matrices are combined as much as possible in a single scope. In some cases, it may be desirable to distinguish and validate different sub-areas (both matrix and working area) separately. The main consideration here is prior knowledge of, or doubt about, the effects of certain interferences.

In certain cases, validation in the 'most difficult' matrix is sufficient. This applies only to the extent to which there is an unambiguous gradation and, if necessary, should be considered for each performance feature. For example, high matrix-loaded waste water can be used to validate a method for domestic and industrial waste water, leachate water from landfills, eluates and the like, and the precision obtained from such samples may be used as a “worst case” value for the relevant concentration range in other water types such as groundwater, surface water and drinking water.

Overall, for the matrices to be considered separately, the following rule of thumb is laid down for the validation of analytical methods for solid/pastelike and liquid environmental matrices:

liquids:
- water and aqueous solutions: a distinction is made between:
  - drinking water
  - waste water,
    in addition, specific performance characteristics in surface and/or groundwater (in relation to soil remediation) should be examined, provided that not covered by the already validated matrices, additional research on specific interferences is also necessary where applicable (e.g. seawater, swimming pool water and thin digestate fraction)
- organic liquids (waste oil, etc.)

solid/pastelike substances: the following four main matrices are distinguished:
- solid waste (construction and demolition waste, ash and slag, shredder waste, wood waste, compost, etc.)
- pastelike wastes (cleaning sludge, dredging and depopulation species, animal fat waste, thick digestate fraction, etc.)
- soil

It is recommended that the method be fully validated at least for the solid and pastelike wastes for one representative sample type from the normal sample supply. An additional verification of the method is required on at least 5 actual samples, from the normal sample range of different origins/nature, e.g. replicate analyses, standard addition, use of internal standards, re-extraction, etc.

Various analysis packages have been drawn up by the Minister, for the OVAM's accreditation of laboratories. These analysis packages are based on legislation (VLAREA, VLAREBO and VLAREM) and not on the relevant main matrices or parameters. Agreements relating to the necessary method validation for each analysis package for the main matrix/matrices and associated sample types can be found in Annex A.

3.3 DEALING WITH EXTERNAL REQUIREMENTS

Because the value of a performance feature is obtained by measurements, this is associated with an uncertainty. This is particularly important when assessing compliance with externally imposed absolute limit values. In this case, it must be demonstrated that the performance reference with a reliability of at least 95 % meets the requirement, i.e. that the numerical value of the performance reference plus (or minus) the uncertainty is less (or greater) than the upper limit (or lower limit).

Traditionally, however, external requirements are generally considered to be an estimated limit value and can be limited to a direct comparison of the numerical value of the performance reference with the limit value.

As a rule, any outliers in the collected analysis results are only removed if the cause is known and does not reflect the practical situation (e.g. incorrect preparation of a synthetic sample, and technically defective measuring device).

3.4 CHOICE OF SAMPLES FOR VALIDATION STUDY

As a general rule, the samples must be representative of the scope (or sub-area) as far as possible. The collection of validation samples should therefore include the most common matrices, based on an understanding of the relative shares of the different samples in the sample stream of the laboratory. If necessary, the sample selection may be focused on the "most difficult" matrix, taking into account the limitations described in point 3.2 .

In principle, intra-reproducibility, repeatability, selectivity and robustness shall be determined on practical samples or samples similar to them as much as possible (e.g. practical samples added).

For validation of the limit of detection and determination, in order of preference, a practical sample or representative reference material with content near the expected limit of detection, a blank practical sample to which the components to be determined were adopted to a level close to the expected limit of detection, or a blank practical sample shall be used.

To validate correctness, in order of preference but always taking into account the similarity to practical samples:

- certified reference material and participation in inter-laboratory testing with traceable reference values, i.e. values related to a suitable (preferably international) measurement standard;
- advected field samples whose actual value is based on the gravimetric/volumetric added amount, these can be either self-created or part of a 'proficiency testing'; it should be noted that addition experiments can produce overly optimistic results because the added part of the component is not incorporated into the sample in the same way as the original part;
- circular samples with a consensus value (e.g. mean value from proficiency testing schemes) using different/equal methods;
- non-certified reference material and/or practical samples with a value independent of the system to be validated, e.g. determined by another method the bias of which is known.

For determining bias with respect to a method average, a certified reference material or a circular sample with a consensus value (e.g. mean ring test value) may be used, provided that only measurement results obtained by the same method are used for the calculation of the certified value or consensus value. This consensus value may differ from the actual value.

For evaluating linearity, the same matrix as for calibration (this is usually standard solution, but also sample for some techniques) is used.

4 GENERAL PROCEDURE FOR DETERMINING THE INDIVIDUAL PERFORMANCE CHARACTERISTICS

4.1 TRUENESS

Two classical methods are discussed below, namely multiple analysis of a reference material (with known true value) and execution of recovery experiments on a selected sample.

If the trueness is validated at only one concentration level, this should preferably close to the regulatory critical value, in the area between the typical values for practical samples and this critical value. When studying at more than one level of concentration (which is otherwise recommended by Eurachem), a factor of 10 is selected as a minimum.

An alternative method is also described, i.e. evaluation of recovery and/or deviation from the reference value for different samples. Not only can the recovery of additions on different samples and/or different concentration levels be used, but also results obtained through participation in proficiency testing schemes (interlaboratory testing), comparative results compared to a reference method. This method is particularly appropriate if there is doubt about the impact of various matrices within the scope (or, where appropriate, sub-area) on the performance characteristics' trueness and/or selectivity (matrix effect).

In addition to information on accuracy, each of the classical methods provides simultaneous information on the intra-reproducibility of the analytical method.

4.1.1 MULTIPLE ANALYSIS OF A REFERENCE MATERIAL

If a sample with a true value or consensus value is available for the scope (or, where appropriate, a sub-area), perform at least 5 analyses (cf. 2.12), under intra-reproducibility conditions (on different days, reflecting typical laboratory conditions in terms of performing analysts, calibration, etc.). Calculate the average of the measurement values obtained. The bias is given by:

$$b\,c\,(rel\,) = \frac{X\,avg\,. - C\,ref}{C\,ref}\,x\,100\,\%$$

(in case of a proportional bias as %),

or,

$b_{c(abs)} = X_{gem} - C_{ref}$

(in case of a proportional bias),

where:

$b_{c(rel)}$ percentage bias (at a value c of the measurement quantity)
$b_{c(abs)}$ absolute bias (at a value c of the measurement quantity)
X_{GSM} mean value of the measurement
C_{ref} true value of the reference material

The trueness (expressed in %) is then given by ($100 + b_{c(rel)}$).

The situation of a constant absolute bias can occur in a low concentration range, with constant unknown interference.

If multiple analyses such as these have been carried out on different reference materials within a given scope, the trueness is preferably calculated by averaging out over the waste materials analysed. The sense of deviation (+ or -) should be taken into account and, where appropriate, a distinction should be made between the situations' proportional or constant absolute bias.

4.1.2 RECOVERY EXPERIMENTS ON A SELECTED SAMPLE

If no suitable sample with a true value or consensus value is available and the component can be added to the sample in a representative way, a recovery experiment is the most appropriate approach. One disadvantage is that certain systematic deviations, for example caused by interference, cannot be identified.

Take at least 10 subsamples for this purpose (see Point 2.12) and add to half a known amount of the component as representative as possible, increasing the measurement quantity by Δc. Guidelines on the method of addition are given in Annex B.

For each sample, determine the recovery from the analysis result for the sample without and the sample adding the component. As far as possible, perform both measurements under the same conditions, i.e. in the same series of analysis. Perform at least five such pairs of analyses under intra-reproducibility conditions (i.e. each pair on a different day, reflecting typical laboratory conditions in terms of performing analysts and calibration).

$$Ph\Delta c,i = \frac{Xc+\Delta c,i - Xc,i}{\Delta c} x\,100\,\%$$

Calculate the recovery (expressed in %) for each of the experiments if:

where:

$T_{\Delta c,i}$	of recovery in the i-d experiment (when adding Δc of the measurement quantity)
$X_{c\Delta c,i}$	i-the analysis result for value c with addition Δc of the measurement quantity
$X_{c,i}$	i-the analysis result for value c of the measurement quantity
Δc	added value of the measurement quantity

Calculate the mean value T, from the recoveries thus obtained, which is a measure for the trueness, expressed in %. The percentage bias can be calculated from $b_{c\ rel} = T$ -100.

4.1.3 Evaluation of recovery and/or development of the reference value for different samples

In terms of additions, this method applies the same boundary conditions and limitations as described in point 4.1.2.

Add a known quantity of the component, as representative as possible, to a subsample of a sample previously analysed, increasing the measurement quantity by Δc. Analyse this sample with addition in the same measurement series, as far as possible under the same conditions as the original sample. Perform a total of 5-10 such recovery analyses on different samples and concentration levels, while ensuring that they represent the best possible practical situation as a whole. Perform the various analyses on samples with additive under intra-reproducibility conditions (on different days, reflecting typical laboratory conditions in terms of performing analysts, calibration,...).

For each of the at least 5 experiments, calculate the recovery (expressed in %) from the analysis result for the sample without and the sample with addition of the component (for formula see point 4.1.2). Calculate the mean value from the recoveries thus obtained. The percentage bias can be calculated from

$b_{c,rel} = T$ - 100

In addition to recoveries obtained through an additive experiment, recoveries can also be derived from participation in proficiency testing schemes, from comparison of the method to a reference method, etc. The main principle here remains the use of different samples, with matrix and concentration levels covering as the scope far as possible.

4.2 REPEATABILITY AND INTRA-REPRODUCIBILITY

The general procedure for determining the performance characteristics repeatability and intra-reproducibility is the same, with the exception of the analytical conditions in the experiments (cf. point 2.3 and 2.4). For intra-reproducibility, the factor of time is additionally varied as a minimum (repeat analyses conducted on different days) and, where relevant, other sources of variation (such as analyst, device and calibration). The aim is to achieve the best possible reflection of the practical situation.

There are two classical methods, namely repeated analysis of the same sample and duplicate analysis of different samples (see point 4.2.1 and 4.2.2). The first approach has the disadvantage that the information in a strict sense is limited to that single sample. On the other hand, the latter approach is, in principle, subject to the restrictive condition that either the standard deviation or the coefficient of variation should not depend on the value of the measurement quantity, which often also leads to a small measuring range.

If, according to the former, precision is validated at only one concentration level, this should preferably be close to the regulatory critical value, in the range between the typical values for practical samples and this critical value. For studies at more than one concentration level (which is also recommended for repeatability studies by Eurachem), a factor of 10 is selected as a minimum.

In practice, an analytical method, after putting into use, will usually also be followed based on a relevant inspection sample, so that the results of this first-line check can also be used as input for periodic re-evaluation of intra-reproducibility.

4.2.1 MULTIPLE ANALYSIS OF THE SAME SAMPLE

Perform at least 5 analyses (cf. point 2.12) on a sample selected according to the recommendations in point 3.4, where appropriate by sub-area under repeatability conditions and intra-reproducibility conditions respectively. Calculate from this the repeatability standard deviation s_r, and the intra-reproducibility standard deviation s_R, respectively, as:

$$s=\sqrt{\sum_{i=1}^{n}\left(\frac{\left(xi-x^{2}\right)}{n-1}\right)}$$ s = standard deviation, in unit of analysis result

n = number of analyses, n ≥ 5

xi = i-th analysis result

x = mean of n analysis results

In the absence of an appropriate sample, a practical sample to which an appropriate quantity of the component has been added may be used, provided that the component added is included in the sample as far as possible in the same way as the component of the practical sample. For guidelines concerning addition, reference is made to Annex B.

4.2.2 DUPLICATE ANALYSIS OF DIFFERENT SAMPLES

Select the size of the area/sub-area so that it can reasonably be assumed that either the standard deviation or the coefficient of variation of the analysis results do not depend on the value of the measurement quantity. In most cases, the spread tends to be well above the proportionality-detection limit, and the coefficient of variation may be assumed consistently in this part of the working On the other hand, near the limit of detection, the standard deviation can often be assumed consistently (cf. point 2.10).

Use samples according to the recommendations in point 3.4. Analyse (cf point 2.12), where appropriate by sub-area, at least 5 different samples in duplicate. In the repeatability study, both analyses of a pair should be performed under repeatability conditions. However, the different samples should preferably be analysed on different days. In the intra-reproducibility study, both analyses of a pair should not be performed on the same day and the whole should be spread over at least as many days as the number of sample pairs.

If for the relevant range the standard deviation can be assumed independently of the value of the measurement quantity (homogeneity of variances), then the n standard deviations from duplicates may be aggregated and s_r, or s_R, can be calculated as (design NEN 7777: 2001; AP 04 SG: 1998):

$$s = \sqrt{\sum_{i=1}^{n} \frac{(x_{i1} - x_{i2})^2}{2n}}$$

s = standard deviation, in unit of analysis result
n = number of samples analysed in duplicate, n ≥ 5
x_{i1} = first analysis result of a duplicate analysis on sample i
x_{i2} = second analysis result of a duplicate analysis on sample i

If, for the relevant measurement range, the coefficient of variation can be assumed to be independent of the value of the measurement quantity, then the n coefficients of variation may be combined from duplicates and the coefficient of variation CV_r, or CV_R, can be calculated from the normalised differences as (design NEN 7777: 2001; AP 04 SG: 1998):

$$CV = \sqrt{\sum_{i=1}^{n} \frac{(x_{i1} - x_{i2})^2}{2n}} \, x \, 100\,\%$$

CV = coefficient of variation, in %
n = number of samples analysed in duplicate, n ≥ 5
x_{i1} = first analysis result of a duplicate analysis on sample i
x_{i2} = second analysis result of a duplicate analysis on sample i

4.3 SELECTIVITY AND ROBUSTNESS

Individual selectivity tests are only useful in those cases where the influence is not sufficiently covered by another performance reference such as accuracy. Experiments in this area are often at the stage of development of an analytical method rather than at the validation stage.

To examine the effect of a certain influence magnitude, select a practical sample if possible, according to the recommendations in point 3.4. Add an increasing amount of influence to different subsamples, as representative as possible and resulting in minimal dilution. Analyse the samples thus obtained under repeatability conditions and determine the change in the analysis result due to a change in the value of the influence magnitude. Depending on prior knowledge, it may be necessary to carry out the above test in the lower and upper section of the working range.

In case there is no prior knowledge on interferences and reference materials are not available, selectivity can be examined as follows:

- analysing practical samples using the appropriate analytical method and with another independent method of analysis or measurement (other separation and/or detection principle);
- analysing successive, ever larger, dilutions of practical samples (provided that the component to be determined is present in sufficient concentration to be measured in dilution).

Robustness is implicitly included in intra-reproducibility, and will not usually require specific validation. The ratio between intra-reproducibility and repeatability can be used as a measure of (the lack of) robustness. This ratio should ideally be 1, although in practice ratios of 1.5 to 2 are not unusual.

Any specific experiments aimed at changing the analysis result due to a change in the execution conditions may be organised in a similar way as described above for selectivity research. In most cases, the first-line quality inspection programme shall be designed in such a way that the most critical variables, namely those with the greatest impact on trueness and precision, are explicitly inspectionled.

4.4 LIMIT OF DETECTION AND DETERMINATION LIMIT

In this procedure, the limit of detection (LOD) is operationalised as 3 times the low-level standard deviation (cf. point 2.7) and this under intra-reproducibility conditions.

Two approaches are described below, i.e. multiple analysis of a practice sample with levels close to the limit of detection and replicate analysis of different practice samples with levels close to the detection limit.

The former is the most traditional, but has the drawback that the uncertainty resulting from the presence of interferences in samples, different matrix effects and the like is not covered. Therefore, the actual limit of detection may be higher under practical conditions. This restriction is somewhat mitigated by the second method.

The term ‘content near the limit of detection’ (or ‘low content’) refers to a level that preferably lies in the range between approximately 1 and 5 times the limit of detection, and which does not exceed 10 times the limit of detection. If a parameter group contains a large number of components (e.g. >10), it may be practically difficult to find a sample that is ideal for each component. In such a case, some components of the above restriction may deviate in terms of concentration range or the limit of detection may be extrapolated from similar components.

4.4.1 MULTIPLE ANALYSIS OF A PRACTICE SAMPLE WITH LOW CONTENT

For this purpose, use a sample according to the recommendations in point 3.4. Analyse the sample strictly according to the usual analytical procedure (see Item 2.12). Re-analyse the sample after one or more days and continue until at least 5 analysis results have been obtained. Carry out the tests in such a way that the whole reflects the practical situation (in terms of performing analysts, environmental conditions, sequence number, etc.).

Calculate the standard deviation s_R from the analysis results and from this the limit of detection $AG_R = 3\ s_R$.
Increase the value of the limit of detection by the average value of the procedure blank if it is measurable and a correction for the procedure blank is not part of the usual analytical procedure.

If several practical samples with low content were analysed several times, the highest of the obtained values shall be used as the final measure for the limit of detection and determination limit.

4.4.2 DUPLICATE ANALYSIS OF DIFFERENT PRACTICAL SAMPLES WITH LOW CONTENT

Use samples according to the recommendations in point 3.4. Analyse (point 2.12) at least 5 different samples in duplicate, do not perform both analyses of a pair on the same day and spread over at least as many days as the number of sample pairs.
Calculate sR as (cf. point 4.2.2):

$$s_R = \sqrt{\sum_{i=1}^{n} \frac{(x_{i1} - x_{i2})^2}{2n}}$$

s_R = intra-reproducibility standard deviation, in unit of analysis result
n = number of samples analysed in duplicate, n ≥ 5
x_{i1} = first analysis result of a duplicate analysis on sample i
x_{i2} = second analysis result of a duplicate analysis on sample i

Calculate the limit of detection $AG_R = 3\ s_R$.

Increase the value of the limit of detection by the average value of the procedure blank if a correction for the procedure blank is not part of the usual analytical procedure.

4.5 LINEARITY - MODEL DEVIATION

When determining the paragraph, or more generally the 'lack-of-fit' model deviation, the experimental deviation from the calibration model shall be quantified. This often concerns only the instrumental part of the analysis. In practice, the experimental measurement values of a calibration can be used.

Define the calibration model using at least 6 different concentration levels, spread over the entire operating range. In the validation study, it is recommended to measure each concentration level in triplicate once in order to assess how the variance of repeat measurements varies with concentration.

Perform an appropriate regression analysis on the results obtained. If there is a proportional relationship between the variance of repeat measurements and the concentration, a weighted regression should be applied. The mere use of a correlation coefficient to determine linearity is not recommended because it is a measure of correlation, not for linearity. Of course, the restrained calibration model should also be used in applying the analytical method in practice.

Calculate the model deviation for a value c of the measurement quantity if:

$\delta_{c,\,model} = X_{c,\,exp} - X_{c,\,model}$

where:
$X_{c,\,exp}$ experimental measurement value or, where applicable, the mean value of the repetition measurements at a value c of the measurement quantity
$X_{c,\,model}$ calculated measurement value obtained according to the calibration model for a value c of the measurement quantity

Graphically plot the model deviations according to the measurement quantity and verify whether these are randomly distributed, systematic trends point to non-linearity. Large percentage deviations are not necessarily an indication of a lack of linearity, but deserve special attention because they can lead to poor intra-reproducibility and high measurement uncertainty. Alternatively, the response factors $RF_c = X_c / c$ (or relative response factors, when using an internal standard) can be plotted and tested against a specified requirement. In most cases, such a requirement takes into account the nature of the method of determination.

A second option is to evaluate the calibration data using a linear model and a quadratic model (ISO 8466-1, 1990). The residual standard deviation s_{y1} and s_{y2} is obtained from both models. The difference of variants DS^2 is calculated as follows:

$$DS^2 = (N-2)\,s_{y1}^2 - (N-3)\,s_{y2}^2$$

where:
N the number of concentration levels ($N \geq 6$)
s_{y1}^2 residual standard deviation in linear model
s_{y2}^2 residual standard deviation at square model number of degrees of freedom f is 1.

Then DM^2 and the variance of the quadratic calibration function shall be subjected to an F test to determine whether significant differences exist. The test value, F_{er}, is obtained from:

$$F\,ber = \frac{DS^2}{s_{y2}^2}$$

If $Fber \leq Ftable$, then the quadratic function does not lead to a significantly better model, in other words the calibration function is linear.
If $Fber \leq Ftable$, the operating range is reduced to obtain a linear calibration function, or the measured values of samples are evaluated using the quadratic function.

A third possibility, although less fundamental, is the calculation of a quadratic function, after which a t-test is used to assess whether the coefficient of the quadratic term deviates significantly from 0.

When establishing a non-linear relationship, it is recommended to assess the relevance of the difference between a result obtained with a linear function and a quadratic function. If this is small compared to the measurement uncertainty, consideration may be given to continuing with the linear equation and offsetting the corresponding error in the overall measurement uncertainty.

For practical elaboration, see the examples in CMA/6/C.

4.6 WORKING RANGE

The lower limit of the working range of a quantitative method of analysis is in principle at or above the limit of quantitation and within the range for which linearity was demonstrated.

As a rule of thumb (target value), for the purposes of this procedure, the reporting limit, the value below which a component is reported as not quantifiable ('<') will not exceed one fifth of the test value or standard value for the measured samples unless not feasible according to current state of the art[2]. This applies both in cases where the limit of quantitation acts as a reporting limit and in the case of systematic use of a reporting limit higher than the limit of quantitation.

If the point of intersection of the calibration line with the Y axis is not significantly different from zero, the operating range may be extended below the concentration level of the lowest calibration standard and to the limit of determination. In the other case, it is recommended to consider the lowest calibration standard as the lower limit of the operating range.

In certain cases, it may be appropriate to also report values between the detection limit and the limit of determination, as there may be information in such values as soon as a number of results is mediated. Such a method should be clear from the analysis report, for example by indicating the measurement values together with the limit of determination. With regard to the rule of thumb described in the first sub paragraph, this does not affect the limit of determination to be assessed against the criterion.

In most cases, the working area can be extended above the linear area by dilution of the sample or by less sample processing. Limiting factors for the upper limit are possible, depending on the method of analysis, the occurrence of memory effects, the capacity of certain preparatory steps,...

5 REQUIREMENTS FOR VALIDATION OF ANALYSIS PACKAGES FOR WASTE SUBSTANCES AND SOIL

Before considering the minimum requirements, the general framework of recognition is outlined. The principle of the recognitions is completely different from that of accreditation. However, ISO 17025 is, on the one hand the basis for a laboratory's accreditation, but at the same time the standard should also be applied for approval as a laboratory by OVAM.

The 'Recognition and Laboratories' instrument was introduced by the Flemish Government for carrying out analyses on waste, among other things, and it determines the conditions and procedure for approval. VLARELbis lists the scope of approval laboratories for the analysis of waste and soil. Several Flemish environmental legislation mentions specific standards for certain parameters depending on the intended purpose, either as waste (landfilling, combustion, or as raw material (soil improver, building material, soil) thermally treated animal waste, check for hazard properties) or as soil (remediation, use as soil, structural land use). The above-mentioned standard frameworks thus group different parameters that are reflected in analysis packages. Annex A contains the complete list of packages for which laboratories may be approved, established by VLARELbis.

[2] Where applicable, this shall be clearly indicated in the relevant CMA procedure and/or in a report from OVAM or the reference laboratory.

For waste, there are many different sample types meaning that validate the parameters of the standards frameworks in all waste types is work not yet commenced. OVAM decided to retain a very limited number of relevant sample types per main matrix. Depending on environmental legislation, 1 or more main matrices may be offered for each analysis package with 1 or more submatrices or sample types within each master matrix. (Cf. Annex B).

For a number of laboratories, the sample types listed will not correspond to their usual sample offer. However, from the point of view of sample supply for recognition application and quality follow-up (3rd line control), it is not possible, with the available financial means, to further split the approval for analysis packages into specific sample types while continuing to comply with the legal provisions (annual supply and inspection). For the reasons above, no further limitation of the number of main matrices per analysis package is allowed.

Due to the aforementioned cross-compliance, certain parameter(s) in the analysis packages are only relevant for specific main matrix or sample type. It is therefore logical that the performance characteristics should be derived only for the relevant sample types. In addition, the method of rendering or analysis may still differ depending on the sample type. OVAM clarified the requirements for method validation and the analytical method to be followed for certain parameters in sample types. Further additions were also subsequently provided. All further refinements for each analysis package are grouped in Annex B.

Validation is conducted per main matrix where all relevant submatrices are taken as sample type. Preferably, for a given analysis package for each main matrix, the laboratory will select from the listed sample types at least 5 samples of different nature/origin corresponding to the actual sample offer.
On these 5 real samples, intrareproducibility is determined by replicate analyses (with separate pretreatment). If the above selection is not possible, the laboratory must select at least 2 actual samples of different nature/origin from the listed sample types. The analysis shall be carried out on each sample in at least 5-fold for determining intrareproducibility.
The limit of detection shall be determined by analogy, but with samples having an appropriate content.
If the method of analysis for a submatrix is different from the analytical method for the other submatrices or if the performance characteristics for parameter(s) in a submatrix differ from those in the other submatrices, a separate validation for the submatrix is necessary.

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ANNEX A PACKAGES FOR WHICH LABORATORIES CAN BE ACCREDITED

MA Sampling of waste and other materials and sample pretreatment on site
MA.2. use as fertiliser/soil improver
MA.3 use as building solids
MA.4 combustion
MA.5 dumping
MA.6 finished products in the processing of animal waste
MA.7 asbestos
MA.7.1 asbestos in heaps MA.7.2 asbestos in layers

A.2 use as fertiliser/soil improver

A.2.1 use as fertiliser/soil improver – inorganic parameters:

acidity, dry/moisture, organic matter, total nitrogen, diphosphorus pentoxide, nitric nitrogen and ammoniacal nitrogen, conductivity

metals (total concentrations): arsenic, cadmium, chromium, copper, mercury, lead, nickel and zinc

A.2.2 use as fertiliser/soil improver – organic parameters:

hydrochlorocarbons: sum of 1,2,3,5-tetrachlorobenzene and 1,2,4,5-tetrachlorobenzene, 1,2,3,4-tetrachlorobenzene, pentachlorobenzene and hexachlorobenzene

polycyclic aromatic hydrocarbons (PAH): naphthalene, benzo(a)pyrene, phenanthrene, fluoranthene, benzo(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(ghi)perylene, indeno(1,2,3-cd)pyrene, acenaphthene, acenaphthylene, anthracene, dibenzo(a,h)anthracene, fluorene, pyrene

mineral oil: fraction C10-C20 and fraction C20-C40

polychlorinated biphenyls (PCB): PCB 28, PCB 52, PCB 101, PCB 118, PCB 138, PCB 153, PCB 180

A.2.3 use as fertiliser/soil improver – specific parameters:

Pebbles, larger than 5 mm
degree of contamination (glass, metal, plastics) greater than 2 mm germination seeds
degree of maturity
stability with closed respirometer

A.2.4 PFAS in fertiliser/soil improver

This package is an extension of the full A.2.2. perfluoro-nbutanic acid (PFBA) package.
perfluoro-n-pentanoic acid (PFPeA)
perfluoro-n-hexanoic acid (PFHxA)
perfluoro-n-heptanoic acid (PFHpA)
perfluoro-n-octanoic acid (PFOA)
perfluoro-n-nonanoic acid (PFNA)
perfluoro-n-decanoic acid (PFDA)
perfluoro-n-undecanoic acid (PFUnDA)
perfluoro-n-dodecanoic acid (PFDoDA)

perfluoro-n-tridecanoic acid (PFTrDA)
perfluoro-n-tetradecanoic acid (PFTeDA)
perfluoro-n-hexadecanoic acid (PFHxDA)
perfluorobutane-n-sulphonic acid (PFBS)
perfluoropentane-n-sulphonic acid (PFPeS)
perfluorohexane-sulphonic acid (PFHxS)
perfluoroheptane-n-sulphonic acid (PFHpS)
perfluorooctane-n-sulphonic acid (PFOS)
perfluorononane-n-sulphonic acid (PFNS)
perfluorooctane sulphonamide (PFOSA)
N-methylperfluoro-n-octane sulphonamide (MePFOSA)
4:2 fluorotelomer sulphonic acid (4:2 FTS)
6:2 fluorotelomer sulphonic acid (6:2 FTS)
8:2 fluorotelomer sulphonic acid (8:2 FTS)
4.8-dioxa-3H-perfluornonanoic acid (DONA)
perfluoro-4-ethylcyclohexane sulphonic acid (PFECHS)
perfluoro-n-hexane sulphonamide (PFHxSA)
perfluoro-n-octadecanoic acid (PFODA)
perfluorooctanesulphonic acid (PFDS)
perfluorododecane sulphonic acid (PFDoDS)
10:2 fluorotelomer sulphonic acid (10:2 FTS)
perfluoro-2-propoxypropanoic acid (HFPO-DA)
6:2 fluorotelomer phosphate diester (6:2 diPAP)
8:2 fluorotelomer phosphate diester (8:2 diPAP)
6:2/8:2 fluorotelomer phosphate diester (6:2/8:2 diPAP)
perfluoro-n-butane sulphonamide (PFBSA)
N-methylperfluoro-n-butane sulphonamide (MePFBSA)
N-methylperfluor-n-butanesulphonylamide acetic acid (MePFBSAA)
N-ethylperfluoro-n-octane sulphonamide (EtPFOSA)
N-methylperfluoro-noctane sulphonamidoacetic acid (MePFOSAA)
N-ethylperfluor-n-octane sulphonamidoacetic acid (EtPFOSAA)
perfluoroundecane sulphonic acid (PFUnDS)
perfluoro-n-trisdecane sulphonic acid (PFTrDS)';

A.3 use as a building material

A.3.1 use as non-formed building material (leaching) dry residue metals (total concentration and leachable fraction by column test): arsenic, cadmium, chromium, copper, mercury, lead, nickel and zinc

BTEXS:
benzene, toluene, ethyl benzene, sum xylenes and styrene

Alkanes:
hexane, heptan and octane

polycyclic aromatic hydrocarbons (PAH):
naphthalene, benzo(a)pyrene, phenanthrene, fluorantee, benzo(a)anthracene, chrysene, benzo(b)fluorinated, benzo(k)fluorinated, benzo(hi)perylene, indeno(1,2,3-cd)pyrene
mineral oil
polychlorinated biphenyls (PCB):
PCB 28, PCB 52, PCB 101, PCB 118, PCB 138, PCB 153, PCB 180

A.3.2 use as shaped building material (extinguishing)
this package is an extension of package non-shaped building material A.3.1

metals (leachable fraction with maximum availability test and via stand test): arsenic, cadmium, chromium, copper, mercury, lead, nickel and zinc

pH, sulphate, chlorides and calcium (via leaching in the maximum availability test and in the stand test)
conductivity (through leaching in the position test)

A.3.3 physical impurities:

floating contaminants, non-floating contaminants and glass

A.3.4 use as a building material

dry residue

metals (total concentrations and leachable fraction using the column test): arsenic, cadmium, chromium, copper, mercury, lead, nickel and zinc

metals (leachable fraction by column test): antimony, barium, molybdenum, vanadium, cobalt, selenium, tin

anions (leachable fraction via column test): bromide, chloride, fluoride and sulphate

BTEXS: benzene, toluene, ethylbenzene, sum xylenes and styrene

alkanes: hexane, heptane and octane

polycyclic aromatic hydrocarbons (PAHs): naphthalene, benzo(a)pyrene, phenanthrene, fluoranthene, benzo(a)anthracene, chrysene, benzo(k)fluoranthene, benzo(k)fluoranthene, benzo(ghi)perylene, indeno(1,2,3-cd)pyrene, acenaphthene, acenaphthylene, anthracene, dibenzo(a,h)anthracene, fluorene and pyrene

mineral oil

polychlorinated biphenyls (PCB): PCB 28, PCB 52, PCB 101, PCB 118, PCB 138, PCB 153, PCB 180

cyanides: free cyanide, non-chlorine oxidisable cyanides';

A.3.5 PFAS in construction material

This package is an extension of the full package A.3.1 or A.3.4.
perfluoro-n-butanoic acid (PFBA)
perfluoro-n-pentanoic acid (PFPeA)
perfluoro-n-hexanoic acid (PFHxA)
perfluoro-n-heptanoic acid (PFHpA)
perfluoro-n-octanoic acid (PFOA)
perfluoro-n-nonanoic acid (PFNA)
perfluoro-n-decanoic acid (PFDA)
perfluoro-n-undecanoic acid (PFUnDA)
perfluoro-n-dodecanoic acid (PFDoDA)
perfluoro-n-tridecanoic acid (PFTrDA)
perfluoro-n-tetradecanoic acid (PFTeDA)
perfluoro-n-hexadecanoic acid (PFHxDA)
perfluorobutane-n-sulphonic acid (PFBS)
perfluoro-n-pentan sulphonic acid (PFPeS)
perfluorohexane-sulphonic acid (PFHxS)
perfluoro-n-heptane sulphonic acid (PFHpS) perfluoro-n-octane sulphonic acid
PFOS: perfluoro-n-nonane sulphonic acid;
perfluoro-1-decane sulphonic acid (PFDs)
perfluorooctane sulphonamide (PFOSA)
N-methylperfluoroctan sulphonamide (MePFOSA)
N-ethyl perfluorooctane sulphonamide (EtPFOSA)
N-methyl perfluorooctane sulphonamidoacetic acid (MePFOSAA)
N-ethyl perfluorooctane sulphonamidoacetic acid (EtPFOSAA)
4:2 fluorotelomer sulphonic acid (4:2 FTS)
6:2 fluorotelomer sulphonic acid (6:2 FTS)
8:2 fluorotelomer sulphonic acid (8:2 FTS)
8:2 fluorotelomer phosphate diester (8:2 diPAP)
perfluoro-2-propoxypropanoic acid (HFPO-DA)
4.8-dioxa-3H-perfluornonanoic acid (DONA)
perfluoro-4-ethylcyclohexane sulphonic acid (PFECHS)
perfluoro-n-butane sulphonamide (PFBSA)
N-methylperfluoro-n-butane sulphonamide (MePFBSA)
perfluoro-n-hexane sulphonamide (PFHxSA)
perfluoro-n-octadecanoic acid (PFODA)
perfluorododecane sulphonic acid (PFDoDS)
6:2 fluorotelomer phosphate diester (6:2 diPAP)
6:2/8:2 fluorotelomer phosphate diester (6:2/8:2 diPAP)
10:2 fluorotelomer sulphonic acid (10:2 FTS)
N-methylperfluor-n-butanesulphonylamide acetic acid (MePFBSAA)

A.4 incinerating:

dry residue, flash point, loss of ignition, total organic carbon (TOC), calorific value, pentachlorophenol (PCP), benzo(a)pyrene, chlorides, fluorides, sulphur, extractable organohalogen compounds (EOX)

metals (total concentrations): cadmium, thallium, mercury, antimony, arsenic, lead, chromium, cobalt, copper, manganese, nickel, vanadium and tin

polychlorinated biphenyls (PCB): PCB 28, PCB 52, PCB 101, PCB 138, PCB 153, PCB 180

A.5 dumping

A.5.1 dumping – general parameters:

dry residue, mineral oil with GC-FID, IR extractable apolar hydrocarbons, loss on ignition, total organic carbon (TOC), total solvents (non-specific), total extractable organo halogen compounds (EOX), steadness (shear stress)

metals (total concentration): arsenic, thallium, mercury, cadmium, beryllium, barium, lead, chromium, copper, nickel, zinc, molybdenum, antimony and selenium

Free cyanides Fluorides

1-step shake test with eluate determination of: pH, arsenic, barium, lead, cadmium, total chromium VI, copper, nickel, mercury, zinc, molybdenum, antimony, selenium, fluoride, cyanide (total), ammonium, nitrite, chloride, sulphate, total dissolved solids (TDS), dissolved organic carbon (DOC), phenol index

A.5.2 dumping – specific organic parameters:

monocyclic aromatic hydrocarbons (BTEXS): benzene, toluene, ethyl benzene, sum xylenes, styrene

polycyclic aromatic hydrocarbons (PAH): naphthalene, benzo(a)pyrene, phenanthrene, fluoranthene, benzo(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(ghi)perylene, indeno(1,2,3-cd)pyrene, acenaphthene, acenaphthylene, anthracene, dibenzo(a,h)anthracene, fluorene, pyrene

polychlorinated biphenyls (PCB): PCB 28, PCB 52, PCB 101, PCB 118, PCB 138, PCB 153, PCB 180

A.6 microbiological provisions on the final products of processed animal waste:

Salmonella
Enterobacteriaceae
Clostridium perfringens

A.7 asbestos

B.1 soil – solid part

clay
organic material (TOC)
metals (total concentration):
arsenic, cadmium, chromium, copper, mercury, lead, nickel, zinc

cyanides:
free cyanides, non-chlorooxidable cyanides

monocyclic aromatic hydrocarbons:
benzene, toluene, ethyl benzene, som xylenes, styrene
1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, 1,3,5-trimethylbenzene

alkanes:
hexane, heptan and octane

hydrochlorocarbons:
dichloromethane, trichloromethane, tetrachloromethane, vinyl chloride, 1,1-dichloroethane, 1,2-dichloroethane, cis+trans-1,2-dichloroethylene, 1,1,1-trichloroethane, 1,1,2-trichloroethane, trichloroethylene, tetrachloroethylene, monochlorobenzene, 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, sum trichlorobenzenes, sum tetrachlorobenzenes, pentachlorobenzene and hexachlorobenzene

chlorphenols:
1- chlorophenol, 2,4-dichlorophenol, 2,4,5-trichlorophenol, 2,4,6-trichlorophenol, 2,3,4,6-tetrachlorophenol, pentachlorophenol

methyl tertiary butyl ether

mineral oil

polycyclic aromatic hydrocarbons (PAH):
naphthalene, acenaphthene, acenaphthene, fluorene, phenanthrene, anthracene, fluorantee, pyrene, benzo(a) anthracene, chrysene, benzo(b)fluorinated, benzo(k)fluorinated, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, dibenzo(a,h) anthracene, benzo(hi)perylene

pH (KCl)

B.4 asbestos in soil

This package is not an enlargement package.

B.5 water bed

dry residue
clay
organic material (TOC)

metals (total concentration):
arsenic, cadmium, chromium, copper, mercury, lead, nickel, zinc

cyanides:
free cyanides, non-chlorooxidable cyanides

monocyclic aromatic hydrocarbons (BTEXS): benzene, toluene, ethyl benzene, sum xylenes, styrene

alkanes:
hexane, heptan and octane

mineral oil

organochloropesticides (OCP):
Aldrin, dieldrin, chlordane (α and γ isomer), DDT, DDE, DDD, hexachlorocyclohexane (α-, β- and γ-isomers), endosulphan (α, β and sulphate)

polychlorinated biphenyls (PCB):
PCB 28, PCB 52, PCB 101, PCB 118, PCB 138, PCB 153, PCB 180

polycyclic aromatic hydrocarbons (PAH):
naphthalene, acenaphthene, acenaphthene, fluorene, phenanthrene, anthracene, fluorantee, pyrene, benzo(a) anthracene, chrysene, benzo(b)fluorinated, benzo(k)fluorinated, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, dibenzo(a,h) anthracene, benzo(hi)perylene

pH (KCl)

B.6 use of soil materials

This package is an extension of the full package B.1 or the full package B.5.

polychlorinated biphenyls (PCB):
PCB 28, PCB 52, PCB 101, PCB 118, PCB 138, PCB 153, PCB 180

stones
foreign materials

shaking test with eluate determination of:
arsenic, cadmium, chromium, copper, mercury, lead, nickel, zinc, pH and conductivity

B.7 dumping of soil materials

This package is an extension of the full package B.1 or the full package B.5.

extractable apolar hydrocarbons with IR ignition loss
total solvents (aspecific)
total extractable organic halogen compounds
stab resistance (shear stress)

1-step shaking test (CMA/2/II/A.12) with eluate determination of:
pH, arsenic, barium, lead, cadmium, total chromium, chromium VI, copper, nickel, mercury, zinc, molybdenum, antimony, selenium, fluoride, cyanide, ammonium, nitrite, chloride, sulphate, total dissolved solids (TDS), dissolved organic carbon (DOC), phenol index

B.8 PFAS in soil and water soil

This package is an extension of the full package.
B.1 or the full package
B.5. perfluoro-n-butanoic acid (PFBA);
perfluoro-n-pentanoic acid (PFPeA)
perfluoro-n-hexanoic acid (PFHxA)
perfluoro-n-heptanoic acid (PFHpA)
perfluoro-n-octanoic acid (PFOA)
perfluoro-n-nonanoic acid (PFNA)
perfluoro-n-decanoic acid (PFDA)
perfluoro-n-undecanoic acid (PFUnDA)
perfluoro-n-dodecanoic acid (PFDoDA)

perfluoro-n-tridecanoic acid (PFTrDA)
perfluoro-n-tetradecanoic acid (PFTeDA)
perfluoro-n-hexadecanoic acid (PFHxDA)
perfluoro-n-butane sulphonic acid (PFBS)
perfluoro-n-pentan sulphonic acid (PFPeS)
perfluoro-n-hexane sulphonic acid (PFHxS)
perfluoro-n-heptane sulphonic acid (PFHpS)
perfluoro-n-octane sulphonic acid (PFOS)
perfluoro-n-nonanesulphonic acid (PFNS)
perfluoro-1-decanansulphonic acid (PFDs)
perfluoro-1-octan sulphonamide (PFOSA)
N-methylperfluoroctane sulphonamide (MePFOSA)
N-ethyl perfluorooctane sulphonamide (EtPFOSA)
N-methyl perfluorooctane sulphonamidoacetic acid (MePFOSAA)
N-ethyl perfluorooctane sulphonamidoacetic acid (EtPFOSAA)
4:2 fluorotelomer sulphonic acid (4:2 FTS)
6:2 fluorotelomer sulphonic acid (6:2 FTS);
8:2 fluorotelomer sulphonic acid (8:2 FTS);
8:2 fluorotelomer phosphate diester (8:2 diPAP), hexafluoropropyleneoxide dimeric acid (HFPO-DA);
4.8-dioxa-3H-perfluornonanoic acid (ADONA)
perfluoro-4-ethylcyclohexane sulphonic acid (PFECHS)

G1 groundwater

metals (total concentration):
arsenic, cadmium, chromium, copper, mercury, lead, nickel, zinc chromium VI

cyanides:
total cyanides

monocyclic aromatic hydrocarbons:
benzene, toluene, ethyl benzene, som xylenes, styrene
1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, 1,3,5-trimethylbenzene

hydrochlorocarbons:
dichloromethane, trichloromethane, tetrachloromethane, vinyl chloride, 1,1-dichloroethane, 1,2-dichloroethane, cis+trans-1,2-dichloroethylene, 1,1,1-trichloroethane, 1,1,2-trichloroethane, trichloroethylene, tetrachloroethylene, monochlorobenzene, 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, sum trichlorobenzenes, sum tetrachlorobenzenes, pentachlorobenzene and hexachlorobenzene

chlorphenols:
2-chlorophenol, 2,4-dichlorophenol, 2,4,5-trichlorophenol, 2,4,6-trichlorophenol, 2,3,4,6-tetrachlorophenol, pentachlorophenol

methyl tertiary butyl ether

mineral oil

polycyclic aromatic hydrocarbons (PAH):
naphthalene, acenaphthene, acenaphthene, fluorene, phenanthrene, anthracene, fluorantee, pyrene, benzo(a) anthracene, chrysene, benzo(b)fluorinated, benzo(k)fluorinated, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, dibenzo(a,h) anthracene, benzo(hi)perylene

organochloropesticides (OCP):
aldrin, dieldrin, chlordane (cis + trans), DDT, DDE, DDD, hexachlorocyclohexane (α, β and γ- isomer), endosulfan (α, β and sulphate)

G.2 PFAS in groundwater

This package is an extension to the full G.1. perfluor-n-butaanzuur (PFBA) package;
perfluoro-n-pentanoic acid (PFPeA)
perfluoro-n-hexanoic acid (PFHxA)
perfluoro-n-heptanoic acid (PFHpA)
perfluoro-n-octanoic acid (PFOA)
perfluoro-n-nonanoic acid (PFNA)
perfluoro-n-decanoic acid (PFDA)
perfluoro-n-undecanoic acid (PFUnDA)
perfluoro-n-dodecanoic acid (PFDoDA)
perfluoro-n-tetradecanoic acid (PFTeDA)
perfluoro-n-hexadecanoic acid (PFHxDA)
perfluoro-n-butane sulphonic acid (PFBS)
perfluoro-n-pentan sulphonic acid (PFPeS)
perfluoro-n-hexane sulphonic acid (PFHxS)
perfluoro-n-heptane sulphonic acid (PFHpS)
perfluoro-n-octane sulphonic acid (PFOS)
perfluoro-n-nonanesulphonic acid (PFNS)
perfluoro-1-decanansulphonic acid (PFDs)
perfluoro-1-octan sulphonamide (PFOSA)
N-methylperfluoroctan sulphonamide (MePFOSA)
N-ethyl perfluorooctane sulphonamide (EtPFOSA)
N-methyl perfluorooctane sulphonamidoacetic acid (MePFOSAA)
N-ethyl perfluorooctane sulphonamidoacetic acid (EtPFOSAA)
4:2 fluorotelomer sulphonic acid (4:2 FTS);
6:2 fluorotelomer sulphonic acid (6:2 FTS);
8:2 fluorotelomer sulphonic acid (8:2 FTS)
8:2 fluortelomer phosphate diester (8:2 diPAP)
Hexafluorpropylene Oxide Dimeric Acid (HFPO-DA)
4.8-dioxa-3H-perfluornonanoic acid (ADONA)
perfluoro-4-ethylcyclohexane sulphonic acid (PFECHS)

ANNEX B METHOD VALIDATION OF ANALYSIS PACKAGE WITH MAIN MATRIX/MATRICES AND ASSOCIATED SAMPLE TYPES

B.1 General overview per package

PACKAGE	MAIN MATRIX	SAMPLE TYPES
A.2.use as fertiliser/soil improver	liquid substances	raw digestate, thin fraction digestate
	pastelike materials	purification sludge, thick fraction digestate
	solid materials	compost, dried digestate
A.3 use as a building material	Fixed/paste substances	construction and demolition waste, ash and slag; sludges from natural stone processing(3)
A.4 incinerating	oil	waste oil
	pastelike materials	sewage sludge
	solid materials	wood waste, ash and slag, shredder waste
A.5dumping	pastelike materials	sewage sludge
	solid materials	construction and demolition waste, shredder waste, ash and slag
A.6 finished products in the processing of animal waste	pastelike materials	animal fat
	solid materials	meat-and-bone meal
A.7 asbestos	solid materials	granulates
B.1 soil – solid part	soil	soil
B.4asbestos in soil	soil	soil
B.5 water bed	water bed	sediment(1)
B.6 use of soil materials	solid soil materials	excavated soil, solid part of the water bed
	pasty soil materials(2)	sediment(1)
B.7 dumping of soil materials	solid soil materials	excavated soil
	pasty soil materials(2)	sediment(1)
G.1 groundwater	water	groundwater

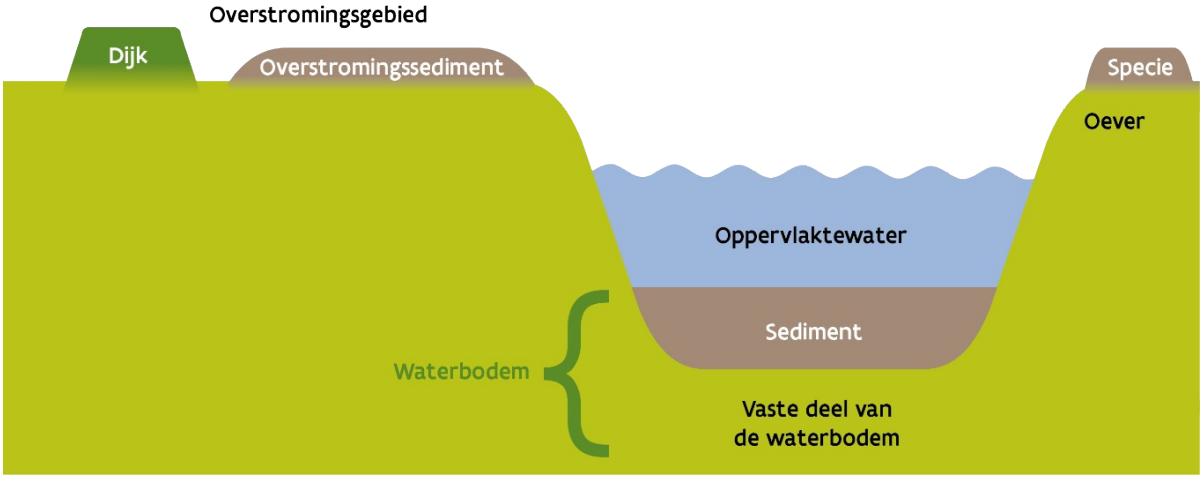
(1) See Figure 1
(2) Ground mill and bentonite sludge belong to the pastelike soil materials
(3) Activated carbon is not a construction material and does not need to be validated

For the validation of materials for use as fertiliser/soil improver, many other relevant sample types are possible in the 3 main matrices, namely:

- for solids – organic-organic waste (OBA) with manure;
- for pastelike substances – OBA mix and various organic-biological wastes
- for liquid substances – effluents after biology, injection stream.

Validation will be carried out as a minimum for the sample types listed in the table above.

The indication ‘ash and slag’ as sample type under the main matrix ‘solids’ in the analytical packages A.3, A.4 and A.5 refers to only one sample type because of the high similarity. Figure 1



Overstromingsgebied	Flood plain
Dijk	Dike
Overstromingssediment	Floodplain sediment
Specie	Dredgings
Oppervlaktewater	Surface water
Oever	Bank
Vaste deel van de waterbodem	Solid part of the water bed
Waterbodem	Water bed

B.2 Package A.2

The materials that can be used as fertilisers/soil improvers (see list in Flemish legislation) are very diverse in terms of physical nature (liquid, pastelike to solid) and composition (content of organic matter, nutrients). The compost analysis package (package 5) is integrated when preparing the package, given that compost is also used as a fertiliser/soil improver. The A.2 package is further broken down into 3 sub-packages due to the very different equipment and expertise for conducting the tests.

The CMA procedures for the determination of the inorganic parameters in compost have been extended to the matrix ‘Manure/soil improver’. All methods are adapted and applicable for the determination of the relevant parameters in materials that can be used as fertilisers or soil improvers.

B.2.1 Package A.2.1

Acidity, dry residue (dry weight), organic matter and conductivity are relevant parameters to assess the soil-improving properties of the material. Total nitrogen, nitrate and ammoniacal nitrogen are important parameters for use as fertilisers but also for the assessment of composting and fermentation processes. The parameter diphosphorus pentoxide will certainly become more relevant in the context of phosphate saturation of agricultural land.

In the Flemish regulations for the use of materials as fertiliser/soil improver (VLAREMA), the heavy metals are, among other things, normised while the parameters for the positive properties as soil improver/fertiliser are not directly normised. The General Regulations of Certification for Gft and Green Composts or for the final material of the biological treatment of organic-organic wastes (OBA) provide for certain properties. These General Regulations are mandatory in the application of the Flemish regulations. In order to perform a proper assessment of all potential substances, the correct determination is also necessary for the latter parameter group.

As indicated in CMA/5/B.1, the following parameters can be measured on the different main matrices.

PARAMETER	MAIN MATRIX		
	Liquid	Pastelike	Solid
Moisture/dry residue	X	X	X
Organic matter	X	X	X
pH, electrical conductivity, ammonia N, nitraat-N, total N	X	X	X
Total content of elements (heavy metals incl. Hg, P, Ca, K, Mg)	X	X	X

For materials that can be used as fertiliser/soil improver, the rendering must be carried out in accordance with CMA/2/IV/6 (§5.3, §5.5 and §5.6) with the exception of liquid samples with a dry weight content < 2 %. These are treated as waste water and discharged in accordance with WAC/III/B/002.

B.2.2 Package A.2.2

This package contains all norm parameters for use as fertiliser or soil improver as listed in VLAREMA 6 and in force until 4 March 2018. VLAREMA 6 (Belgian Official Gazette 23 February 2018) amended the criteria for raw materials. These criteria are set out in Annex 2.3.1.A., Annex 2.3.1.B. and Annex 2.3.1.C. Compliance of the fertiliser or soil improver with the normisation must be demonstrated by 4 March 2019 at the latest.

B.2.3 Package A.2.3

The parameters, except stability with closed respirometer, were originally only measured on compost samples. The different treatment techniques on waste streams create very diverse materials for which the determination of some parameters is not relevant or less relevant. In such a situation, validation is therefore not necessary. As indicated in CMA/5/B.1, the following parameters can be measured on the different main matrices.

PARAMETER	MAIN MATRIX			SAMPLE TYPE
	Liquid	Pastelike	Solid	
Stones > 5 mm, Pollution > 2 mm and Repirometric test germ strength seeds	X	X	X	Except for samples with dry weight content <2 %
Degree of ripeness			X	Only applicable for compost (Green, GFT and OBA compost)

B.2.4 Package A.2.4

This is a new package for the determination of PFAS in the context of test values for use as fertiliser/soil improvers.

B.3 Package A.3

B.3.1 Package A.3.1

Package A.3.1 was repealed from VLAREMA 9 and cancelling the existing approvals.

B.3.2 Package A.3.2

Package A.3.2 was repealed from VLAREMA 9 and cancelling the existing approvals.
The constructed construction substances are no longer tested with the diffusion test and maximum availability test.

B.3.3 Package A.3.3

Physical indiscriminate package.

B.3.4 Package A.3.4

This is a new package replacing package A.3.1. with a number of additional parameters in the leaching fraction via the column test:
metals: antimony, barium, molybdenum, vanadium, cobalt, selenium, tin and anions: bromide, chloride, fluoride and sulphate.

B.3.5 Package A.3.5

This is a new package (PFAS in construction) as an extension of the full package A.3.1 or A.3.4

B.4 Package A.4

Due to the wide variety of possible sample types and the fact that not all parameters are to be measured on this sample, the differentiation of parameters to the relevant main matrices and submatrices is necessary. The previous analysis package 4.2.8 has been removed so that the PCB must also be validated.

The parameter 'flame point' should only be analysed in the matrix 'oil' so that validation can be limited to the sample type of oil. The solvent parameter (aspecific) is not a relevant parameter for assessing the end-of-waste status for the oil matrix and therefore no longer needs to be validated. The parameters ‘ignition loss and TOC’ are also not relevant parameters for the matrix ‘oil’ so that they should not be validated on the oil sample type.

Ignition loss and TOC are important parameters for the characterisation of combustion residues (Vlarem II norms). Both parameters are also relevant in sewage sludge to verify the presence of at least 50 % flammable substances. However, for wood waste, these 2 parameters are not relevant from an environmental point of view.

The parameter 'benzo(a)pyrene' will be measured cf. VLAREM II only to verify that wood waste complies with the guide values for uncontaminated treated wood waste. In the matrix ‘solid dust’, the parameter B(a)p must therefore only be validated in the sample type ‘wood waste’. For the parameter pentachlorophenol (PCP) a maximum level can be set out in the environmental permit of a co-combustion plant for hazardous waste. Since this parameter is currently not included in any environmental permit for co-combustion and there is no validated CMA method for PCP determination in materials other than wood waste, the PCP parameter should, for the time being,

only be validated in the sample type 'wood waste'
(cfr guide values for wood waste).

Dry residue is a relevant parameter for all sample types (water content is relevant for matrix oil).

The parameters (calorific value, F, Cl, S, metals and EOX) are specifically aimed at checking the input for combustion for operation and the frequency of measurement of the emission parameters. The sample type 'axis and slag' can be deleted. For the calorific value, the determination of the limit of detection is not relevant since the test value is at least 5 MJ/kg.

The combustion of PCB-containing waste is prohibited in cement kilns and since oil and shredder waste are high-calorific waste, laboratories approved for combustion should be able to correctly determine whether or not these wastes are PCB-containing. Shredder waste and oil are therefore relevant matrices for PCB validation.
In the first instance, the combustion of waste with PCB can be described as a disposal operation. The minimum kiln temperature is determined by the 1 % norm for total halogenated compounds containing PCB, among others. Wastes containing more than 1 % in total of halogenated compounds must be incinerated at a minimum of 1100 °C. The other wastes may be incinerated at a minimum temperature of 850 °C.

More important when assessing waste incinerated is Council Directive 96/59/EC of 16 September 1996 on the disposal of polychlorinated fiphenyls and polychoorterphenyls (PCs/PCTs) and its framework of norms. The total PCB content is defined as five times the sum of the levels of PCB-28, PCB-52, PCB-101, PCB-138, PCB-153 and PCB-180 (6 congeners). Waste is PCB-containing if the aforementioned total PCB content exceeds the limit value of 50 ppm, as laid down in the European Directive 96/59/EC and the Flemish legislation in force.

The PCB analysis will serve to designate a waste as persistent or not. For using reprocessed waste oils as a fuel (end of waste criterion), the norm for total PCB content is 5 ppm.

Package A.4				
PARAMETER	MAIN MATRIX			SAMPLE TYPES
	oil	pastelike	solid	
Dry residue	X	X	X	see B.1(1)
Flash point	X			Oil
Loss on ignition		X	X	Sewage sludge, ash and slag, shredder waste
TOC		X	X	Sewage sludge, ash and slag, shredder waste
Benzo(a)pyrene, PCP			X	wood waste
Calorific value	X	X	X	Oil, Sewerage sludge, wood waste, shredder waste
Chlorides Fluorides Sulphur	X	X	X	Oil, Sewerage sludge, wood waste, shredder waste

Package A.4				
Metals	X	X	X	Oil, Sewerage sludge, wood waste, shredder waste
EOX	X	X	X	Oil, Sewerage sludge, shredder waste
PCB	X		X	Oil, shredder waste

(1) For oil: determination of water content

B.5 Package A.5

The stability parameter in the analysis package A.5 should not be validated in the main matrix 'solids' since the determination of solid resistance is only relevant on pastelike materials.

PCB is a norm parameter for landfills for inert materials. For package A.5.2, the PCB validation will be carried out on all relevant solid sample types.

The PAK was extended from 10 to 16 PAK.

B.6 Package A.6

The bacteriological parameters in the final products of processed animal by-products must be validated on a liquid fraction and a solid fraction, respectively, of animal fat and meat meal.

B.7 Package A.7

The validation of asbestos is done on recycled granules or on hardening/funding material.

B.8 Package B.1, B.4, B.5, B.6, B.7 and B.8

Soil materials originate from the soil and, depending on how and where these materials are extracted from the soil, such as excavated soil, dredging and depopulation species, ground mash or bentonite sludge. The soil materials are similar in nature and composition. However, the dry weight of the soil materials may vary greatly depending on the place of origin. The soil materials have a similar environmental impact when used, except for the water aspect and any auxiliary substances in drilling.
Since 1 April 2019, there is a single use framework for all soil materials in the Vlarebo. The different analysis packages apply according to the nature, origin and use of the soil materials.
The water content in sediment (drying and depopulation) or bentonite sludge/ground mill is generally much higher than in soil (excavated or otherwise). This difference can significantly affect the performance features of organic parameters. Validation is therefore also done on the 2 main matrices.
The new package B.8 (PFAS in soil and water soil) is an extension of the full B.5 package

ANNEX C GUIDELINES RELATING TO ADDITION

C.1 GENERAL GUIDELINES FOR ADDITION

The addition of the component is performed proportionally. Optimally, between 50 % and 200 % of the quantity present in the original sample is added.

The concentration difference should be sufficiently large in relation to the intended measurement uncertainty, without compromising the similarity with practical samples.

The addition should be carried out in such a way that minimum dilution of the sample (maintenance of matrix) occurs. The dilution should preferably not exceed 1 % (m/m, v/v of v/m).

The addition should be carried out in such a way that the measurement uncertainty on the sample and the adjoined sample remains comparable (using sufficiently reliable norm solution, etc.).

C.2 SPECIFIC GUIDELINES FOR ADDING DIFFERENT MATRICES FOR ORGANIC PARAMETERS

C.2.1 Add to solid materials

Moderately volatile compounds

Depending on the size of the material to be used for the determination, a volume (about one-tenth of the sample volume) of a polar solvent (methanol, 2-propanol, acetone, etc.) with a known amount of components to be determined should be added. Do not apply this quantity in one place, but distribute it over the material. Homogenise the sample and allow the solvent to evaporate to the air for a sufficient period of time (e.g. one night). Homogenise the sample again.

Volatile compounds

Place a quantity of material in a container and add the amount of solvent required for extraction/disclosure. Then add a known, small amount (< 1 % v/v) of a solution with components. This solution should be made with the same solvent as that used for extraction/disclosure. Seal the container, homogenise and allow for a sufficient period of time (at least 2 hours). Homogenise the sample again.

C.2.2 Adding to liquids

Aqueous solutions

If the components dissolve sufficiently well in water (e.g. phenols, polar pesticides, etc.), a small volume (< 0.1 % v/v) is placed in the sample with a known amount of the substances to be determined. These substances should be dissolved in a water-mixable solvent (methanol, 2-propanol, acetone, etc.). Homogenise the sample.

In the case of components which do not dissolve properly in water, in the case of a liquid/liquid extraction, first transfer the sample to a separating funnel and then carry out the addition (< 0.1 % v/v). Once again, the components are incorporated in a water-mixable solvent. Rinse the sample containers with the solvent to be used for extraction.

Another option is to add an additive to keep the apolar components in the matrix (e.g.: 2-propanol, detergents, etc.). In this case, one can work as indicated for polar compounds.

Oil

To a known amount of material, add a small amount (< 0.1 % v/v) of solution containing the components to be determined. Use a solvent that is well mixed with the oil to be investigated (2-propanol, acetone, hexane, etc.). Homogenise the sample.

ANNEX D MINIMAL REQUIREMENTS IN RELATION TO REPORTING LIMITS

The accredited laboratories and VITO derived performance characteristics for the parameters covered by the accreditation under ISO 17025. These were collected and processed by VITO. In processing, it was assumed that these data also include sample pretreatment. In deriving the performance requirements, evaluation was made of what is required by the legislation and what is analytically feasible. The results were discussed in the working groups on inorganic and organic parameters.

If the analysis fails to meet the minimum reporting limit, adjustments to the analysis process should ensure that the minimum reporting limit is reached. These adjustments will be mentioned in the analytical report. If the minimum reporting limit cannot be met despite the adjustments, the increased reporting limit will be reported together with the adjustments made.

D.1 Package A.2.1 Use as fertiliser/soil improver — compost sample type

Parameters	**Units**	**VGF KDS**[1]	**GFT norm**	**Green CDB**[1]	**Green norm**	Reporting limit
Moisture	weight % on fresh weight	< 50 (<45)	< 55 (<50)	< 50 (<45)	< 55 (50)	-
pH	on fresh weight		6,5-9,5		6,5-9,5	-
Organic matter	weight % on fresh weight	>16 (>18)	> 14 (>16)	>16 (>18)	> 14 (>16)	-
Total nitrogen	%w/w N (fresh weight)					-
Ammoniacal nitrogen	mg NO4-N/l compost by fresh weight					20
Nitrate nitrogen	mg NO3-N/l compost by fresh weight					10
Conductivity* at 25 °C **(in 1/5 dilution)	µs/cm (fresh weight)					-
Heavy metal						
Arsenic	mg/kg DM	15	20	15	20	2
Cadmium	mg/kg DM	1.5	2	1.5	2	0.5
Copper	mg/kg DM	90	150	90	150	15
Lead	mg/kg DM	120	150	120	150	25
Chromium	mg/kg DM	70	70	70	70	15
Nickel	mg/kg DM	20	30	20	30	4
Zinc	mg/kg DM	300	400	300	400	50
Mercury	mg/kg DM	1	1	1	1	0.2
Germinable seeds	number/l (fresh weight)	1	1	1	1	absent
Stones > 5 mm	weight % on fresh weight on fresh weight	< 2	< 4	< 2	< 4	< 0,05
Impurities > 2 mm	weight % on fresh weight on fresh weight					< 0,05
Impurities > 2 mm	w/w % (dry weight)	< 0,5	< 0,5	< 0,5	< 0,5	< 0,1
Phytotoxicity	%	-	-	-	-	< 1
T max	°C	< 40	< 45	< 30	< 40	-

[1] QO: quality objective requirement for VLACO label

D.2 Package A.2.1 Use as fertiliser/soil improver — inorganic VLAREMA parameters

	Raw materials > 2 % dry weight (DM)			Raw materials < 2 % dry weight per litre (l)		
	Unit	Vlarema Norm Flemish Government Decree 22/12/2017 Annex 2.3.1.A	Reporting limit	Unit	Vlarema Norm Flemish Government Decree 22/12/2017 Annex 2.3.1.B	Reporting limit
Arsenic	mg/kg DM	20	2	mg/l	0.4	0.04
Cadmium	mg/kg DM	6	0,5	mg/l	0.12	0.01
Chromium	mg/kg DM	150	15	mg/l	3	0.3
Copper	mg/kg DM	800	15	mg/l	16	0.3
Lead	mg/kg DM	300	25	mg/l	6	0.5
Nickel	mg/kg DM	100	4	mg/l	2	0.08
Zinc	mg/kg DM	1500	50	mg/l	30	1
Mercury	mg/kg DM	1	0.2	mg/l	0.02	0.01

D.3 Package A.2.2 Use as fertiliser/soil improver — organic parameters

1) Samples with more than 2 % dry weight (DM)

	Unit	Norm value Vlarema Flemish Government Decree 22/12/2017 2.3.1.A	1/5 norm	1/2 norm	Reporting limit[(1)]
Higher chlorobenzenes					
sum of 1,2,3,5-tetrachlorobenzene and 1,2,4,5-tetrachlorobenzene	mg/kg DM	4	0.8	2	0.4
1,2,3,4-tetrachlorobenzene	mg/kg DM	2	0.4	1	0.2
pentachlorobenzene	mg/kg DM	1.5	0.3	0.75	0.15
hexachlorobenzene	mg/kg DM	0.5	0.1	0.25	0.05
Polycyclic aromatic hydrocarbons					
acenaphthene	mg/kg DM	10	2	5	1
acenaphthylene	mg/kg DM	10	2	5	1
anthracene	mg/kg DM	5	1	2.5	0.5
naphthalene	mg/kg DM	3	0.6	1.5	0.3
benzo(a)pyrene	mg/kg DM	3	0.6	1.5	0.3
phenanthrene	mg/kg DM	10	2	5	1
fluoranthene	mg/kg DM	10	2	5	1
benzo(a)anthracene	mg/kg DM	3	0.6	1.5	0.3
chrysene	mg/kg DM	3	0.6	1.5	0.3
benzo(b)fluoranthene	mg/kg DM	10	2	5	1
benzo(k)fluoranthene	mg/kg DM	5	1	2.5	0.5
benzo(ghi)perylene	mg/kg DM	5	1	2.5	0.5

	Unit	Norm value Vlarema Flemish Government Decree 22/12/2017 Annex 2.3.1.A	1/5 norm	1/2 norm	Reporting limit(1)
Indeno(1,2,3-cd)pyrene	mg/kg DM	5	1	2.5	0.5
dibenzo(a,h)anthracene	mg/kg DM	5	1	2.5	0.5
fluorene	mg/kg DM	10	2	5	1
pyrene	mg/kg DM	3	0.6	1.5	0.3
Mineral oil					
Group C10-C20 Fraction C20-C40	mg/kg DM mg/kg DM	560 5600	112 1120	280 2800	100 1000
Polychlorinated biphenyls (sum of 7 PCBs)					
Sum of 7 PCBs	mg/kg DM	0.6	0.12	0.3	0.2
PCB 28	mg/kg DM				(0,025)
PCB 52	mg/kg DM				(0,025)
PCB 101	mg/kg DM				(0,025)
PCB 118	mg/kg DM				(0,025)
PCB 153	mg/kg DM				(0,025)
PCB 138	mg/kg DM				(0,025)
PCB 180	mg/kg DM				(0,025)

(1) the reporting limits of the individual PCBs are test values

2) Samples with less than 2 % dry weight per litre

	Unit	Norm value Vlarema Flemish Government Decree 22/12/2017 Annex 2.3.1.A	1/5 norm	1/2 norm	Reporting limit(1)
Higher chlorobenzenes					
sum of 1,2,3,5-tetrachlorobenzene and 1,2,4,5 — tetrachlorobenzene	µg/L	80	16	40	10
1,2,3,4-tetrachlorobenzene	µg/L	40	8	16	5
pentachlorobenzene	µg/L	30	6	15	5
hexachlorobenzene	µg/L	10	2	4	2
Polycyclic aromatic hydrocarbons					
acenaphthene	µg/L	200	40	100	40
acenaphthylene	µg/L	200	40	100	40
anthracene	µg/L	100	20	50	20
naphthalene	µg/L	60	12	30	10
benzo(a)pyrene	µg/L	60	12	30	10
phenanthrene	µg/L	200	40	100	40
fluoranthene	µg/L	200	40	100	40
benzo(a)anthracene	µg/L	60	12	30	10
chrysene	µg/L	60	12	30	10
benzo(b)fluoranthene	µg/L	200	40	100	40
benzo(k)fluoranthene	µg/L	100	20	50	20
benzo(ghi)perylene	µg/L	100	20	50	20

indeno(1,2,3-cd)pyrene	µg/L	100	20	50	20

	Unit	Norm value Vlarema Flemish Government Decree 22/12/2017 Annex 2.3.1.A	1/5 norm	1/2 norm	Reporting limit[1]
dibenzo(a,h)anthracene	µg/L	100	20	50	20
fluorene	µg/L	200	40	100	40
pyrene	µg/L	60	12	30	10
Mineral oil					
Group C10-C20	mg/l	11.2	2.2	5.6	5
Fraction C20-C40	mg/l	112	22.4	56	20
Polychlorinated biphenyls (sum of 7 PCBs)					
Sum of 7 PCBs	µg/L	12	2.4	6	4
PCB 28	µg/L				(0,5)
PCB 52	µg/L				(0,5)
PCB 101	µg/L				(0,5)
PCB 118	µg/L				(0,5)
PCB 153	µg/L				(0,5)
PCB 138	µg/L				(0,5)
PCB 180	µg/L				(0,5)

[1] the reporting limits of the individual PCBs are test values

D.4 Package A.2.4 PFAS in fertiliser/soil improver

1) Samples with more than 2 % dry weight (DM)

Perfluoroalkyl and polyfluoroalkyl compounds (PFAS)				Reporting limit
perfluoro-n-butanoic acid (PFBA)	µg/kg DW			3
perfluoro-n-pentanoic acid (PFPeA)	µg/kg DW			3
perfluoro-n-hexanoic acid (PFHxA)	µg/kg DW			2
perfluoro-n-heptanoic acid (PFHpA)	µg/kg DW			2
perfluoro-n-octanoic acid (PFOA)	µg/kg DW			2
perfluoro-n-nonanoic acid (PFNA)	µg/kg DW			1
perfluoro-n-decanoic acid (PFDA)	µg/kg DW			1
perfluoro-n-undecanoic acid (PFUnDA)	µg/kg DW			1
perfluoro-n-dodecanoic acid (PFDoDA)	µg/kg DW			1
perfluoro-n-tridecanoic acid (PFTrDA)	µg/kg DW			1
perfluoro-n-tetradecanoic acid (PFTeDA)	µg/kg DW			1
perfluoro-n-hexadecanoic acid (PFHxDA)	µg/kg DW			2
perfluorobutane-n-sulphonic acid (PFBS)	µg/kg DW			1
perfluoropentane-n-sulphonic acid	µg/kg			1

(PFPeS)	DW			
perfluorohexane-sulphonic acid (PFHxS)	µg/kg DW			**1**
perfluoroheptane-n-sulphonic acid (PFHpS)	µg/kg DW			**1**
perfluorooctane-n-sulphonic acid (PFOS)	µg/kg DW			**3**
perfluorononane-n-sulphonic acid (PFNS)	µg/kg DW			**1**
4:2 fluorotelomer sulphonic acid (4:2 FTS)	µg/kg DW			**1**
6:2 fluorotelomer sulphonic acid (6:2 FTS)	µg/kg DW			**3**

Perfluoroalkyl and polyfluoroalkyl compounds (PFAS)				**Reporting limit**
8:2 fluorotelomer sulphonic acid (8:2 FTS)	µg/kg DW			**1**
perfluorooctane sulphonamide (PFOSA)	µg/kg DW			**1**
N-methylperfluoroctan sulphonamide (MePFOSA)	µg/kg DW			**1**
4.8-dioxa-3H-perfluornonanoic acid (DONA)	µg/kg DW			**1**
perfluoro-4-ethylcyclohexane sulphonic acid (PFECHS)	µg/kg DW			**1**
perfluoro-n-hexane sulphonamide (PFHxSA)	µg/kg DW			**3**
perfluoro-n-octadecanoic acid (PFODA)	µg/kg DW			**4**
perfluorooctanesulphonic acid (PFDS)	µg/kg DW			**4**
perfluorododecane sulphonic acid (PFDoDS)	µg/kg DW			**4**
10:2 fluorotelomer sulphonic acid (10:2 FTS)	µg/kg DW			**4**
perfluoro-2-propoxypropanoic acid (HFPO-DA)	µg/kg DW			**4**
6:2 fluorotelomer phosphate diester (6:2 diPAP)	µg/kg DW			**4**
8:2 fluorotelomer phosphate diester (8:2 diPAP)	µg/kg DW			**4**
6:2/8:2 fluorotelomer phosphate diester (6:2 8:2 diPAP)	µg/kg DW			**4**
perfluoro-n-butane sulphonamide (PFBSA)	µg/kg DW			**4**
N-methylperfluoro-n-butane sulphonamide (MePFBSA)	µg/kg DW			**4**
N-methyl perfluoro-n-butane sulphonamide acetate (MePFBSAA)	µg/kg DW			**4**
N-ethyl perfluorooctane sulphonamide (EtPFOSA)	µg/kg DW			**4**
N-methyl perfluorooctane sulphonamidoacetic acid (MePFOSAA)	µg/kg DW			**4**
N-ethyl perfluorooctane sulphonamidoacetic acid (EtPFOSAA)	µg/kg DW			**4**
perfluoroundecane sulphonic acid (PFUnDS)	µg/kg DW			**4**
perfluorotridecane sulphonic acid (PFTrDS)	µg/kg DW			**4**
perfluorooctanoic acid (total of linear and branched) (PFOAtotal)	µg/kg DW			**2**
perfluorooctane sulphonic acid (total of linear and branched) (PFOS total)	µg/kg DW			**3**
perfluorooctane sulphonic acid (total of linear and branched) (PFOSA total)	µg/kg DW			**1**
N-methylperfluorooctane sulphonamide (total linear and branched) (MePFOSAtotal)	µg/kg DW			**1**
N-ethylperfluorooctane sulphonamide (total linear and branched)	µg/kg DW			**3**

(EtPFOSAtotal)				
perfluorohexane sulphonic acid (total of linear and branched) (PFHxStotal)	µg/kg DW			**1**

2)

3) Samples containing less than 2 % dry matter (ds)

Perfluoroalkyl and polyfluoroalkyl compounds (PFAS)				**Reporting limit**
perfluoro-n-butanoic acid (PFBA)	ng/l			**100**
perfluoro-n-pentanoic acid (PFPeA)	ng/l			**100**
perfluoro-n-hexanoic acid (PFHxA)	ng/l			**100**
perfluoro-n-heptanoic acid (PFHpA)	ng/l			**100**
perfluoro-n-octanoic acid (PFOA)	ng/l			**100**
perfluoro-n-nonanoic acid (PFNA)	ng/l			**100**
perfluoro-n-decanoic acid (PFDA)	ng/l			**100**
perfluoro-n-undecanoic acid (PFUnDA)	ng/l			**100**
perfluoro-n-dodecanoic acid (PFDoDA)	ng/l			**100**
perfluoro-n-tridecanoic acid (PFTrDA)	ng/l			**100**
perfluoro-n-tetradecanoic acid (PFTeDA)	ng/l			**100**
perfluoro-n-hexadecanoic acid (PFHxDA)	ng/l			**100**
perfluorobutane-n-sulphonic acid (PFBS)	ng/l			**100**
perfluoropentane-n-sulphonic acid (PFPeS)	ng/l			**100**
perfluorohexane-sulphonic acid (PFHxS)	ng/l			**100**
perfluoroheptane-n-sulphonic acid (PFHpS)	ng/l			**100**
perfluorooctane-n-sulphonic acid (PFOS)	ng/l			**100**
perfluorononane-n-sulphonic acid (PFNS)	ng/l			**100**
4:2 fluorotelomer sulphonic acid (4:2 FTS)	ng/l			**100**
6:2 fluorotelomer sulphonic acid (6:2 FTS)	ng/l			**100**
8:2 fluorotelomer sulphonic acid (8:2 FTS)	ng/l			**100**
perfluorooctane sulphonamide (PFOSA)	ng/l			**100**
N-methylperfluoroctan sulphonamide (MePFOSA)	ng/l			**100**
4.8-dioxa-3H-perfluornonanoic acid (DONA)	ng/l			**100**
perfluoro-4-ethylcyclohexane sulphonic acid (PFECHS)	ng/l			**100**
perfluoro-n-hexane sulphonamide (PFHxSA)	ng/l			**100**
perfluoro-n-octadecanoic acid (PFODA)	ng/l			**100**
perfluorooctanesulphonic acid (PFDS)	ng/l			**100**
perfluorododecane sulphonic acid (PFDoDS)	ng/l			**100**
10:2 fluorotelomer sulphonic acid (10:2 FTS)	ng/l			**100**
perfluoro-2-propoxypropanoic acid (HFPO-DA)	ng/l			**100**
6:2 fluorotelomer phosphate diester (6:2 diPAP)	ng/l			**100**
8:2 fluorotelomer phosphate diester (8:2 diPAP)	ng/l			**100**
6:2/8:2 fluorotelomer phosphate diester (6:2 8:2 diPAP)	ng/l			**100**
perfluoro-n-butane sulphonamide (PFBSA)	ng/l			**100**
N-methylperfluoro-n-butane sulphonamide (MePFBSA)	ng/l			**100**
N-methyl perfluoro-n-butane sulphonamide acetate (MePFBSAA)	ng/l			**100**

Perfluoroalkyl and polyfluoroalkyl compounds (PFAS)				**Reporting limit**
N-ethyl perfluorooctane sulphonamide (EtPFOSA)	ng/l			**100**
N-methyl perfluorooctane sulphonamidoacetic acid (MePFOSAA)	ng/l			**100**
N-ethyl perfluorooctane sulphonamidoacetic acid (EtPFOSAA)	ng/l			**100**
perfluoroundecane sulphonic acid (PFUnDS)	ng/l			**100**
perfluorotridecane sulphonic acid (PFTrDS)	ng/l			**100**
perfluorooctanoic acid (total of linear and branched) (PFOAtotal)	ng/l			**100**
perfluorooctane sulphonic acid (total of linear and branched) (PFOS total)	ng/l			**100**
perfluorooctane sulphonic acid (total of linear and branched) (PFOSA total)	ng/l			**100**
N-methylperfluorooctane sulphonamide (total linear and branched) (MePFOSAtotal)	ng/l			**100**
N-ethylperfluorooctane sulphonamide (total linear and branched) (EtPFOSAtotal)	ng/l			**100**
perfluorohexane sulphonic acid (total of linear and branched) (PFHxStotal)	ng/l			**100**

D.5 Package A.3.4 Use as a building material — paste and solid

The reporting limits apply to the analysis of waste for use as a building material in accordance with Article 2.3.2.1 (1) of the Vlarema:
Taking into account the applicable conditions for the use of construction substances, the following compositional criteria must be met as a minimum in order to consider the intended raw materials to be used as construction materials:

1° the maximum total concentrations of organic compounds set out in Annex VI to the Flemish Government Decree of 14 December 2007 laying down the Flemish Regulation on soil remediation and soil protection are not exceeded;

2° the maximum total concentrations of metals, listed in Annex VI of the VLAREBO decree of 14 December 2007, are mandatory values for the unmixed use as unformed building material and for the mixed use in unformed building material with compressive strength less than 6.0 N/mm2;

3° the maximum leachability values of the metals arsenic, cadmium, chromium, copper, mercury, lead, nickel and zinc, listed in Annex 2.3.2.B, shall not be exceeded during the intended use in or as a building material. The leachability values of barium, bromide, chloride, fluoride and sulphate listed in Annex 2.3.2.B are guideline values.

In ash from waste incineration and slag from metallurgy, the maximum leachability values of the metals antimony, molybdenum, vanadium, cobalt, selenium and tin, listed in Annex 2.3.2.B, are not exceeded when intended to be used in or as building materials;' '7° recycled aggregates and physicochemically treated recycled aggregates are exempt from the mandatory value for the total metal concentration. If the total concentrations for each heavy metal is lower than the value for free use of excavated soil listed in Annex V of the VLAREBO, recycled granules and physico-chemically treated granules shall be exempted from the determination of leaching of heavy metals.

	Unit	**Norm value VLAREBO Annex VI**[1]	1/5 norm VLAREBO Annex VI	Reporting limit[2]
HEAVY METALS AND METALLOIDS				
Arsenic	mg/kg DM	267	53	10
Cadmium	mg/kg DM	30	6	0,5
Chromium	mg/kg DM	880	176	20
Copper	mg/kg DM	500	100	10
Mercury	mg/kg DM	11	2.2	0.1
Lead	mg/kg DM	1250	250	20
Nickel	mg/kg DM	530	106	10
Zinc	mg/kg DM	1250	250	20
Monocyclic aromatic hydrocarbons				
benzene	mg/kg DM	0.5	0.1	0.2
toluene	mg/kg DM	15	3	0.2
ethylbenzene	mg/kg DM	5	1	0.2
xylenes (sum)	mg/kg DM	15	3	0.2
styrene	mg/kg DM	10	2	0.2
Polycyclic aromatic hydrocarbons				
naphthalene	mg/kg DM	6	1.2	0.5
benzo(a)pyrene	mg/kg DM	7.2	1.4	0.2
phenanthrene	mg/kg DM	30	6	0.2
fluoranthene	mg/kg DM	30	6	0.5
benzo(a)anthracene	mg/kg DM	30	6	0.1
chrysene	mg/kg DM	20	4	0.3
benzo(b)fluoranthene	mg/kg DM	4.4	0.9	0.5
benzo(k)fluoranthene	mg/kg DM	10	2	0.5
benzo(ghi)perylene	mg/kg DM	10	2	0.2
indeno(1,2,3-cd)pyrene	mg/kg DM	15	3	0.2
acenaphthene	mg/kg DM	6.2	1.2	0.5
acenaphthylene	mg/kg DM	1.2	0.24	0.2
anthracene	mg/kg DM	10	2	0.5
dibenzo(a,h)anthracene	mg/kg DM	3.2	0.64	0.5
fluorene	mg/kg DM	20	4	0.5
pyrene	mg/kg DM	46	9.2	0.5
Alkanes				
hexane	mg/kg DM	1.2	0.25	0.5
heptane	mg/kg DM	20	4	1
octane	mg/kg DM	60	12	1
Mineral oil	***mg/kg DM***	***1000***	***200***	***100***
Polychlorinated biphenyls (sum of 7 PCBs)				
Sum of 7 PCBs	mg/kg DM	0.5	0.1	0.1
PCB 28	mg/kg DM			(0,015)
PCB 52	mg/kg DM			(0,015)
PCB 101	mg/kg DM			(0,015)
PCB 118	mg/kg DM			(0,015)
PCB 153	mg/kg DM			(0,015)
PCB 138	mg/kg DM			(0,015)
PCB 180	mg/kg DM			(0,015)
Cyanides				
Free cyanides	mg/kg DM	5	1	1
Non-chlorinated oxidisable cyanides	mg/kg DM	12	2.4	1

[1] Vlarebo Annex VI construction soil use

[2] the reporting limits of the individual PCBs are test values

Metal leachability (column test CMA/2/II/A.9.1 at L/S=10 l/kg DM)						
	Norm value VLAREMA Annex 2.3.2.B. mg/kg DM	Norm µg/l	1/5 norm µg/l	1/2 norm µg/l	RL µg/L	RL mg/kg DM
Arsenic	0,8[(1)]	80	16	40	15	0.15
Cadmium	0,03[(1)]	3	0.6	1.5	1.5	0.015
Chromium	2,6[(1)]	260	52	130	10	0.1
Copper	0,8[(1)]	80	16	40	10	0.1
Mercury	0,02[(1)]	2	0.4	1	0.5	0.005
Lead	1,3[(1)]	130	26	65	20	0.2
Nickel	0,75[(1)]	75	15	37.5	10	0.1
Zinc	2,8[(1)]	280	56	140	50	0.5
Antimony	1[(1)(2)]	100	20	50	20	0.2
Molybdenum	55[(1)(2)]	5500	1100	2750	20	0.2
Vanadium	2.5[(1)(2)]	250	50	125	20	0.2
Cobalt	0.5[(1)(2)]	50	10	25	10	0.1
Selenium	2[(1)(2)]	200	40	100	40	0.4
Tin	1[(1)(2)]	100	20	50	20	0.2
Barium	20[(3)]	2000	400	1000	400	4
Bromide	20[(3)]	2000	400	1000	400	4
Fluoride	55[(3)]	5500	1100	2750	1000	10
Chloride	1000[(3)]	100000	20000	50000	20000	200
Sulphate	2200[(3)]	220000	44000	110000	20000	200

[(1)] compulsory value
[(2)] only in ash from waste incineration and in slag from metallurgy
[(3)] guide values

Metals (total concentration)			
Parameter	**Unit**	**Norm**	
		Values for free use of excavated soil Cf. Annex V Vlarebo	**Reporting limit**
Arsenic	mg/kg DM	35	10
Cadmium	mg/kg DM	1.2	0.5
Chromium	mg/kg DM	91	20
Copper	mg/kg DM	72	10
Mercury	mg/kg DM	1.7	0.1
Lead	mg/kg DM	120	20
Nickel	mg/kg DM	48	10
Zinc	mg/kg DM	200	20

Article 2.3.2.1. §2 Notwithstanding section 1, the materials listed in Appendix 2.2, Part 2, in particular asphalt granulates, recycled bituminous granulates and asphaltic sands, need not satisfy the total concentration for the mineral oil parameter, and for polycyclic aromatic hydrocarbons, the total concentrations stated in Annex 2.3.2.A shall apply.

The PAH spray test is used to determine compliance with the overall concentrations mentioned above. If the PAH-spray test shows a yellow discoloration, it is considered that the above norms are not met. In case of unclear discoloration, an infrared spectroscopy confirmatory test may be carried out. If the infrared spectroscopy shows clear peaks, it shall be considered that the aforementioned norms have not been met. Qualitative testing may be done with infrared spectroscopy without a prior PAH spray test. In case of doubt, a counter-test using chemical analysis for PAH using GC-MS shall be undertaken to determine whether the norms in Appendix 2.3.2.A have been exceeded. The unified regulations for recycled granulates specifies the testing method and conformity inspection method for the PAH spray test.'.

Polycyclic aromatic hydrocarbons			
Parameter	**Unit**	**Norm**	
		Normw.VLAREMA Annex 2.3.2.A	**Reporting limit**
benzo(a)anthracene	mg/kg DM	35	0.1
benzo(a)pyrene	mg/kg DM	8.5	0.2
benzo(ghi)perylene	mg/kg DM	35	0.2
benzo(b)fluoranthene	mg/kg DM	55	0.5
benzo(k)fluoranthene	mg/kg DM	55	0.5
chrysene	mg/kg DM	400	0.3
phenanthrene	mg/kg DM	30	0.2
fluoranthene	mg/kg DM	40	0.5
indeno(1,2,3-cd)pyrene	mg/kg DM	35	0.2
naphthalene	mg/kg DM	20	0.5

D.6 Package A.3.3 Physical contaminants

Parameters	**Units**	**VLAREMA art 2.3.2.1**	Reporting limit
floating contaminants (fraction 4-63 mm) FL	cm^3/kg DW	5.0	1.0
non-floating contaminants (fraction 4-63 mm) X	% (m/m)	1.0	0.2
Unbound glass (fraction 4-63 mm) Rg	% (m/m)	2.0	0.2

D.7 Package A.4 Burning — oil, pastelike and solid

Parameter	**Unit**	**Norm or limit value for oil**[(1)]	RL oil	RL paste /shredder	RL ash/slags	**Norm wood**	RL wood
Water content	m/m %	0.5	0.4	not relevant	not relevant		not relevant
Sediment content	m/m%	0.1	0.05	N/A	N/A		N/A
Flash point	°C		40 °C	not relevant	not relevant		N/A
Loss on ignition	m/m % DM		N/A	1 %	1 %		N/A
TOC	m/m% C DM		N/A				

Parameter	Unit	Norm or limit value for oil[1]	RL oil	RL paste /shredder	RL ash/slags	Norm wood	RL wood
EOX	mg/kg DM		200	200			N/A
Pentachlorophenol (PCP)	mg/kg DM		N/A	N/A	N/A	3	0.6
Benzo(a)pyrene	mg/kg DM		N/A	N/A	N/A	0.5	0.1
Calorific value	mg/kg DM	-	not relevant	not relevant	N/A		not relevant
Chlorides	mg/kg DM	200	50	150	N/A	600	150
Fluorides	mg/kg DM	-		40	N/A	30	10
Sulphur	mg/kg DM	10000[2]		100	N/A		100
Elements (total concentrations)							
Cadmium	mg/kg DM	[4]	1	5			
Thallium	mg/kg DM	-	1	20			
Mercury	mg/kg DM	1	0.5	0.5			
Antimony	mg/kg DM	-	1	20			
Arsenic	mg/kg DM	[4]	1	20		2	1
Lead	mg/kg DM	[4]	1	20		90	10
Chromium	mg/kg DM	[4]	1	20		30	5
Cobalt	mg/kg DM	[4]	1	20			
Copper	mg/kg DM	[4]	1	20		20	5
Manganese	mg/kg DM	[4]	1	20			
Nickel	mg/kg DM	[4]	1	20			
Vanadium	mg/kg DM	-	1	20			
Tin	mg/kg DM	[4]	1	20			
Zinc	mg/kg DM	15[3]	1				
Calcium	mg/kg DM	30[3]	10				
Phosphorus	mg/kg DM	15[3]	7.5				

N/A: not applicable

[1] VLAREMA Annex 2.3.3. Composition conditions maximum pollutant contents for reprocessed waste oil and reprocessed fuel residues

[2] * or lower if the S content in the applicable product norm is lower than 1 % (m/m)

(3) If the combination of Ca and Zn or Ca and P exceeds the specified maximum levels, the waste oil is not considered reprocessed.

(4) (As, Cd, Cr, Co, Cu, Mn, Pb, Ni, Sn) < 25 mg/kg. For the calculation of the sum of these elements, the content per element below the reporting limit is set to 0 (lower bound approach).

Parameter	**Unit**	Limit PCB-containing	1/5 Limit	Reporting limit(1)
Total PCB(2)	mg/kg DM	50	10	10
Sum of the 6 marker PCBs	mg/kg DM	10	2	2
PCB 28	mg/kg DM			(0,3)
PCB 52	mg/kg DM			(0,3)
PCB 101	mg/kg DM			(0,3)
PCB 138	mg/kg DM			(0,3)
PCB 153	mg/kg DM			(0,3)
PCB 180	mg/kg DM			(0,3)

(1) the reporting limits for individual marker PCBs are approximate

(2) the total PCB content is defined as five times the sum of the levels of PCB-28, PCB-52, PCB-101, PCB-138, PCB-153 and PCB-180 (6 congeners). When calculating the sum, individual PCB levels below the reporting limit will be equal to 0.

Parameter (in oil matrix)	Unit	VLAREMA norm value Annex 2.3.3[3]	1/5 Limit	Reporting limit[1]
Total PCB[2]	mg/kg DM	5	1	1
PCB 28	mg/kg DM			(0,1)
PCB 52	mg/kg DM			(0,1)
PCB 101	mg/kg DM			(0,1)
PCB 138	mg/kg DM			(0,1)
PCB 153	mg/kg DM			(0,1)
PCB 180	mg/kg DM			(0,1)

[1] the reporting limits for individual marker PCBs are approximate
[2] the total PCB content is defined as five times the sum of the levels of PCB-28, PCB-52, PCB-101, PCB-138, PCB-153 and PCB-180 (6 congeners). When calculating the sum, individual PCB levels below the reporting limit will be equal to 0.
[3] VLAREMA Annex 2.3.3. Composition conditions maximum pollutant contents for reprocessed waste oil and reprocessed fuel residues

D.8 Package A.5.1 landfills — pastelike and solid

Parameter	Unit	Norm value	1/5 norm	1/2 norm	Reporting limit
Dry residue	m/m%				not relevant
Mineral oil with GC-FID	mg/kg DM	500	100	250	100
TCE extractable (apolar) substances with IR	m/m % DM	2	0.4	1	0.1
Solvents (non-specific)	%	1	0.2	0.5	0.2
EOX	mg Cl/kg DM	1000	200	500	200
Loss on ignition	m/m % DM	10%	2	5	2
Total Organic Carbon (TOC)	m/m% C DM	3			1
Shear strength	kN/m²	10	2	5	2

1-step shaking test (CMA/2/II/A.12) with determination in eluate of:						
Parameter	**Unit**	**Norm value**	**1/5 norm**	**1/2 norm**	Reporting limit	**mg/kg DM at L/S 10**
pH	-				not relevant	not relevant
Arsenic	µg/L	50	10	25	15	0.15
Barium	µg/L	2000	400	1000	400	4
Lead	µg/L	50	10	25	20	0.2
Cadmium	µg/L	4	0,8	2	1,5	0.015
Total chromium	µg/L	50	10	25	10	0.1
Chromium VI	µg/L	500	100	250	100	1
Copper	µg/L	200	40	100	40	0.4
Nickel	µg/L	40	8	20	10	0.1
Mercury	µg/L	1	0.2	0.5	0.5	0.005
Zinc	µg/L	400	80	200	50	0.5
Molybdenum	µg/L	50	10	25	15	0.15
Antimony	µg/L	6	1.2	3	3	0.03
Selenium	µg/L	10	2	5	5	0.05
Fluoride	mg/l	1	0.2	0.5	0.2	2
Cyanide	mg/l	1	0.2	0.5	0.2	2

1-step shaking test (CMA/2/II/A.12) with determination in eluate of:						
Parameter	**Unit**	**Norm value**	**1/5 norm**	**1/2 norm**	Reporting limit	**mg/kg DM at L/S 10**
Chloride	mg/l	80	16	40	20	200
Sulphate	mg/l	100	20	50	20	200
Ammonium	mg/l	1000	200	500	200	2000
Nitrite	mg/l	30	6	3	6	60
Total dissolved solids (TDS)	mg/l	400	80	200	100	1000
Dissolved organic carbon (DOC)	mg/l	50	10	25	10	100
Phenol index	µg/L	100	20	50	20	0.2 200

D.9 Package A.5.2 landfills — pastelike and solid

Parameter	**Unit**	**Norm value**[(1)]	**1/5 norm**	**1/2 norm**	Reporting limit
Monocyclic aromatic hydrocarbons					
Sum BTEX	mg/kg ds	6	1.2	3	1
Benzene	mg/kg ds	0.5	0.1	0.25	0.2
Toluene**	mg/kg ds	6	1.2	3	0.2
Ethylbenzene	mg/kg ds	5	1	2.5	0.2
Xylenes (sum)	mg/kg ds	6	1.2	3	0.2
Styrene	mg/kg ds	1.5	0.3	0.75	0.2
Polycyclische aromatische koolwaterstoffen					
Naphthalene	mg/kg ds	6	1.2	3	0.5
Benzo(a)pyrene	mg/kg ds	7.2	1.4	3.6	0.2
Phenanthrene	mg/kg ds	30	6	15	0.2
Fluoranthene	mg/kg ds	30	6	15	0.5
Benzo(a)anthracene	mg/kg ds	30	6	15	0.1
Chrysene	mg/kg ds	20	4	10	0.3
Benzo(b)fluoranthene	mg/kg ds	4.4	0.9	2.2	0.5
Benzo(k)fluoranthene	mg/kg ds	10	2	5	0.5
Benzo(ghi)perylene	mg/kg ds	10	2	5	0.2
Indeno(1,2,3-cd)pyrene	mg/kg ds	15	2	7.5	0.2
Acenaphthene	mg/kg ds	6.2	1.2	3.1	0.5
Acenaphthylene	mg/kg ds	1.2	0.24	0.6	0.2
Anthracene	mg/kg ds	10	2	5	0.5
Aminobenzoic(a,h)anthraces	mg/kg ds	3.2	0.64	1.6	0.5
Fluorene	mg/kg ds	20	4	10	0.5
Pyrene	mg/kg ds	46	9.2	23	0.5

(1) inert and non-hazardous waste

Parameter	**Unit**	Norm value	1/5 Limit value	Reporting limit[(1)]
Total PCB (sum of the 7)	mg/kg DM	1	0.2	0.2
PCB 28	mg/kg DM			(0,025)
PCB 52	mg/kg DM			(0,025)
PCB 101	mg/kg DM			(0,025)
PCB 118	mg/kg DM			(0.025)

PCB 138 PCB 153	mg/kg DM mg/kg DM			(0,025) (0,025)
PCB 180	mg/kg DM			(0,025)

(1) the reporting limits for individual marker PCBs are approximate

D.10 Package G.1 Groundwater and B.1 Soil

GROUNDWATER	Unit	BSN norm[1]	Guide value[2]	1/5 SN	1/5 GV	1/2 GV	Reporting limit
Metals							
Arsenic	µg/L	20	12	4	2.4	6	10
Cadmium	µg/L	5	3	1	0.6	1.5	1.5
Chromium	µg/L	50	30	10	6	15	10
Copper	µg/L	100	60	20	12	30	20
Mercury	µg/L	1	0.6	0.2	0.12	0.3	0.2
Lead	µg/L	20	12	4	2.4	6	10
Nickel	µg/L	40	24	8	4.8	12	10
Zinc	µg/L	500	300	100	60	150	50
Chromium (VI)	µg/L						15
Petroleum hydrocarbons							
benzene	µg/L	10	2	2	0,4	1	1
toluene	µg/L	700	20	140	4	10	4
ethylbenzene	µg/L	300	20	60	4	10	4
m+p-xylene	µg/L						2
o-xylene	µg/L						1
xylenes (sum)	µg/L	500	20	100	4	10	
styrene	µg/L	20	10	4	2	5	2
MTBE	µg/L	300	20	60	4	10	4
Mineral oil	µg/L	500	300	100	60	150	100
Chlorinated solvents							
dichloromethane	µg/L	20	5	4	1	2.5	2.5
tetrachloromethane	µg/L	2	1.2	0.4	0.2	0.6	0.6
tetrachloroethene	µg/L	40	5	8	1	2.5	1
trichloroethylene	µg/L	70	5	14	1	2.5	1
1,1,1-trichloroethane	µg/L	500	5	100	1	2.5	1
1,1,2-trichloroethane	µg/L	12	5	2.4	1	2.5	1
1,1-dichloroethane	µg/L	330	5	66	1	2.5	1
cis-1,2-dichloroethylene	µg/L						1.2
trans-1,2-dichloroethane	µg/L						1.2
1,2-dichloroethylene (sum)	µg/L		5		1	2.5	
1,2-dichloroethane	µg/L	30	5	6	1	2.5	1
vinyl chloride	µg/L	5	2	1	0.4	1	1
trichloromethane	µg/L	200	5	40	1	2.5	1
monochlorobenzene	µg/L	300	5	60	1	2.5	1
1,2-dichlorobenzene	µg/L	1000	5	200	1	2.5	1
1,3-dichlorobenzene	µg/L	1000	5	200	1	2.5	1
1,4-dichlorobenzene	µg/L	300	5	60	1	2.5	1

GROUNDWATER	Unit	BSN norm[1]	Guide value[2]	1/5 BSN	1/5 GV	1/2 GV	Reporting limit
Moderately volatile chlorobenzenes							
1,2,3-trichlorobenzene[3]	µg/L	20	5	4	1	2,5	1
1,2,4-trichlorobenzene[3]	µg/L	20	5	4	1	2,5	1
1,3,5-trichlorobenzene[3]	µg/L	20	5	4	1	2,5	1
trichlorobenzenes (sum)[3]	µg/L						
1,2,3,5+1,2,4,5-tetrachlorobenzene	µg/L	18	10	3.6	2	5	2
1,2,3,4-tetrachlorobenzene	µg/L	9	5	1.8	1	2.5	1
tetrachlorobenzenes (sum)	µg/L						
pentachlorobenzene	µg/L	2.4	1.4	0.5	0.3	0.7	0.3
hexachlorobenzene	µg/L	1	0.6	0.2	0.1	0.3	0.1
Polycyclic aromatic hydrocarbons							
naphthalene	µg/L	60	20	12	4	10	2
benzo(a)pyrene	µg/L	0.7	0.4	0.14	0.1	0.2	0.1
phenanthrene	µg/L	120	20	24	4	10	1
fluoranthene	µg/L	4	2	0.8	0.4	1	0.2
benzo(a)anthracene	µg/L	7	2	1.4	0.4	1	0.2
chrysene	µg/L	1.5	0.9	0.3	0.2	0.5	0.1
benzo(b)fluoranthene	µg/L	1.2	0.7	0.24	0.1	0.4	0.1
benzo(k)fluoranthene	µg/L	0.76	0.4	0.15	0.1	0.2	0.1
benzo(ghi)perylene	µg/L	0.26	0.1	0.05	0.02	0.05	0.05
indeno(1,2,3-cd)pyrene	µg/L	0.1	0.06	0.2	0.01	0.03	0.03
anthracene	µg/L	75	20	15	4	10	0.5
fluorene	µg/L	120	20	24	4	10	0.5
dibenzo(a,h)anthracene	µg/L	0.5	0.3	0.1	0.06	0.15	0.06
acenaphthene	µg/L	180	20	36	4	10	0.5
acenaphthylene	µg/L	70	20	14	4	10	0.5
pyrene	µg/L	90	20	18	4	10	0.5
Cyanides							
Cyanide total	µg/L	70	40	14	8	20	10
Organochlorine pesticides							
aldrin	µg/L						0.01
dieldrin	µg/L						0.01
aldrin+dieldrin	µg/L	0.03	0.02	0.006	0.004	0.01	
cis-chlordane	µg/L						0.03
trans-chlordane	µg/L						0.03
chlordane (cis+trans)	µg/L	0.2	0.12	0.04	0.024	0.06	
o,p'-DDD	µg/L						0.04
p,p'-DDD	µg/L						0.04
o,p'-DDE	µg/L						0.04
p,p'-DDE	µg/L						0.04
o,p'-DDT	µg/L						0.04
p,p'-DDT	µg/L						0.04
DDD+DDE+DDT	µg/L	2	1.2	0.4	0.24	0.6	

GROUNDWATER	Unit	BSN norm[1]	Guide value[2]	1/5 BSN	1/5 GV	1/2 GV	Reporting limit
lindane (α-isomer)	µg/L	0.06	0.03	0.01	0.006	0.015	0.02
lindane (β-isomer)	µg/L	0.2	0.12	0.04	0.024	0.06	0.025
lindane (γ-isomer)	µg/L	2	1.2	0.4	0.24	0.6	0.25
α-endosulfan	µg/L						0.07
β-endosulfan	µg/L						0.07
endosulfansulphate	µg/L						0.07
endosulphan(α+β+sulphate)	µg/L	1.8	1	0.36	0.2	0.5	
Trimethylbenzenes							
1,2,3-trimethylbenzene	µg/L	150	-	30	-	-	10
1,2,4-trimethylbenzene	µg/L	150	-	30	-	-	10
1,3,5-trimethylbenzene	µg/L	150	-	30	-	-	10
Chlorophenols							
2,4,6-trichlorophenol	µg/L	200	-	40	-	-	1
pentachlorophenol	µg/L	9	-	1.8	-	-	1
2-chlorophenol	µg/L	15	-	3	-	-	1
2.4-dichlorophenol	µg/L	9	-	1.8	-	-	1
2,4,5-trichlorophenol	µg/L	300	-	60	-	-	1
2,3,4,6-tetrachlorophenol	µg/L	90	-	18	-	-	1

[1] BSN: soil remediation norm, Annex IV Vlarebo 2008: Soil Remediation Norms

[2] Guide value Annex II Vlarebo 2008: Guide values for soil quality

[3] According to CMA/3/I

SOIL		BSN norm[1]	Guide value.[2]/ Free use[3]	1/5 BSN	1/5 GV	1/2 GV	Reporting limit
Clay	%	(2%)	(2%)				2%
Organic material							
Org. mat via TOC determination (= 1.72 x TOC content) (TOC)	m/m% ds (m/m% C ds)	(1%) (0.58%)	(1%) (0.58%)				0.35 % (0,2%)
Metals							
Arsenic	mg/kg DM	58	35	11.6	7	17.5	10
Cadmium	mg/kg DM	2	1.2	0.4	0.24	0.6	0.5
Chromium	mg/kg DM	130	91	26	18.2	45.5	20
Copper	mg/kg DM	120	72	24	14.4	36	10
Mercury	mg/kg DM	2.9	1.7	0.58	0.34	0.85	0.3
Lead	mg/kg DM	200	120	40	24	60	20
Nickel	mg/kg DM	93	48	18.6	9.6	24	10
Zinc	mg/kg DM	333	200	66.6	40	100	20
Petroleum hydrocarbons							
benzene	mg/kg DM	0.5	0.3	0.1	0.06	0.15	0.05
toluene	mg/kg DM	4	1.6	0.8	0.32	0.8	0.3
ethylbenzene	mg/kg DM	2	0.8	0.4	0.16	0.4	0.15
m+p-xylene	mg/kg DM						0.1
o-xylene	mg/kg DM						0.05
xylenes (sum)	mg/kg DM	3	1.2	0.6	0.24	0.6	
styrene	mg/kg DM	0.8	0.32	0.16	0.064	0.16	0.05
hexane	mg/kg DM	1.5	0.6	0.3	0.12	0.3	0.1
heptane	mg/kg DM	25	10	5	2	5	0.5
octane	mg/kg DM	75	30	15	6	15	0.5
MTBE	mg/kg DM	2	1	0.4	0.2	0.5	0.2
Mineral oil	mg/kg DM	1000	300	200	60	150	50
Chlorinated solvents							
dichloromethane	mg/kg DM	0.13	0.05	0.026	0.01	0.025	0.025
tetrachloromethane	mg/kg DM	0.1	0.04	0.02	0.008	0.02	0.02
tetrachloroethene	mg/kg DM	0.7	0.28	0.14	0.056	0.14	0.05
trichloroethylene	mg/kg DM	0.65	0.26	0.13	0.052	0.13	0.05
1,1,1-trichloroethane	mg/kg DM	10	4	2	0.8	2	0.5
1,1,2-trichloroethane	mg/kg DM	0.2	0.08	0.04	0.016	0.04	0.04
1,1-dichloroethane	mg/kg DM	2	0.08	0.4	0.016	0.04	0.04
cis-1,2-dichloroethylene	mg/kg DM						0.04
trans-1,2-dichloroethane	mg/kg DM						0.04
1,2-dichloroethylene (sum)	mg/kg DM	0.4	0.16	0.08	0.032	0.08	
1,2-dichloroethane	mg/kg DM	0.1	0.06	0.02	0.012	0.03	0.03
vinyl chloride	mg/kg DM	0.1	0.06	0.02	0.012	0.03	0.03
trichloromethane	mg/kg DM	0.1	0.06	0.02	0.012	0.03	0.03
monochlorobenzene	mg/kg DM	2.5	1	0.5	0.2	0.5	0.2
1,2-dichlorobenzene	mg/kg DM	35	14	7	2.8	7	0.5
1,3-dichlorobenzene	mg/kg DM	40	16	8	3.2	8	0.5
1,4-dichlorobenzene	mg/kg DM	4	1,6	0.8	0.32	0.8	0.3

SOIL		BSN norm[1]	Guide value.[2]/ Free use[3]	1/5 BSN	1/5 GV	1/2 GV	Reporting limit
Moderately volatile chlorobenzenes							
1,2,3-trichlorobenzene	mg/kg DM		0.2		0.04	0.1	0.04
1,2,4-trichlorobenzene	mg/kg DM		0.2		0.04	0.1	0.04
1,3,5-trichlorobenzene	mg/kg DM		0.2		0.04	0.1	0.04
trichlorobenzenes (sum)	mg/kg DM	0.5		0.1			
1,2,3,5+1,2,4,5-tetrachlorobenzene	mg/kg DM		0.08		0.016	0.04	0.04
1,2,3,4-tetrachlorobenzene	mg/kg DM		0.04			0.02	0.02
tetrachlorobenzenes (sum)	mg/kg DM	0.1		0.02			
pentachlorobenzene	mg/kg DM	0.5	0.2	0.1	0.04	0.1	0.04
hexachlorobenzene	mg/kg DM	0.1	0.06	0.02	0.012	0.03	0.03
Polycyclic aromatic hydrocarbons							
naphthalene	mg/kg DM	1.5	0.3	0.3	0.06	0.15	0.15
benzo(a)pyrene	mg/kg DM	0.5	0.3	0.1	0.06	0.15	0.06
phenanthrene	mg/kg DM	60	15	12	3	7.5	0.2
fluoranthene	mg/kg DM	20	2	4	0.4	1	0.2
benzo(a)anthracene	mg/kg DM	5	3.9	1	0.78	2	0.2
chrysene	mg/kg DM	10	2.5	2	0.5	1.25	0.2
benzo(b)fluoranthene	mg/kg DM	2	1.1	0.4	0.22	0.55	0.1
benzo(k)fluoranthene	mg/kg DM	1	0.6	0.2	0.12	0.3	0.1
benzo(ghi)perylene	mg/kg DM	160	0.3	32	0.06	0.15	0.15
indeno(1,2,3-cd)pyrene	mg/kg DM	1	0.7	0.2	0.14	0.35	0.1
anthracene	mg/kg DM	3	2.4	0.6	0.5	1.2	0.1
fluorene	mg/kg DM	45	9.5	9	1.9	4.7	0.1
dibenzo(a,h)anthracene	mg/kg DM	0.5	0.3	0.1	0.06	0.15	0.06
acenaphthene	mg/kg DM	9	3.1	1.8	0.62	1.5	0.1
acenaphthylene	mg/kg DM	1	0.6	0.2	0.12	0.3	0.1
pyrene	mg/kg DM	125	21	25	4	10	0.2

Cyanides							
Cyanide total	mg/kg DM						
Cyanide-free	mg/kg DM	5	3	1	0.6	1.5	1
Cyanide-non-chl.ox. (Total CN-free CN)	mg/kg DM	5	3	1	0.6	1.5	1
Trimethylbenzenes							
1,2,3-trimethylbenzene	mg/kg DM	0.81	-	0.16	-	-	0.1
1,2,4-trimethylbenzene	mg/kg DM	1.3	-	0.26	-	-	0.1
1,3,5-trimethylbenzene	mg/kg DM	0.61	-	0.12	-	-	0.1
Chlorophenols							
2,4,6-trichlorophenol	mg/kg DM	0.64	-	0.13	-	-	0.1
pentachlorophenol	mg/kg DM	0.25	-	0.05	-	-	0.1
2-chlorophenol	mg/kg DM	3.93	-	0.79	-	-	0.1
2.4-dichlorophenol	mg/kg DM	0.67	-	0.13	-	-	0.1
2,4,5-trichlorophenol	mg/kg DM	24	-	4.8	-	-	0.1
2,3,4,6-tetrachlorophenol	mg/kg DM	1.79	-	0.36	-	-	0.1

(1) BSN: soil-remediation norm destination type 1, Annex IV Vlarebo 2008: Soil Remediation Norms

(2) Annex II: VLAREBO 2008: Guide values for soil quality

(3) Annex V Vlarebo 2008: Values for free use of soil materials

D.11 Package A.7 Asbestos

The reporting limit will be 2 mg/kg DM if no asbestos is found and 20 mg/kg DM if a small amount (< 2 mg/kg) of friable asbestos is detected.

D.12 Package B.4 Asbestos in soil

The reporting limit will be 2 mg/kg DM if no asbestos is found and 20 mg/kg DM if a small amount (< 2 mg/kg) of friable asbestos is detected.

D.13 Package B.5 Water bed

WATER BED		**BSN norm**[1]	**Guide value.**[2]**/ Free use**[3]	1/5 BSN	1/5 GV	1/2 GV	Reporting limit
Clay	%	(2%)	(2%)				2%
Organic material							
Org.mat.- TOC	%	(1%)	(1%)				1%
Metals							
Arsenic	mg/kg DM	58	35	11.6	7	17.5	10
Cadmium	mg/kg DM	2	1.2	0.4	0.24	0.6	0.5
Chromium	mg/kg DM	130	91	26	18.2	45.5	20
Copper	mg/kg DM	120	72	24	14.4	36	10
Mercury	mg/kg DM	2,9	1.7	0.58	0.34	0.85	0.3
Lead	mg/kg DM	200	120	40	24	60	20
Nickel	mg/kg DM	93	48	18.6	9.6	24	10
Zinc	mg/kg DM	333	200	66.6	40	100	20
Petroleum hydrocarbons							
benzene	mg/kg DM	0.5	0.3	0.1	0.06	0.15	0.05
toluene	mg/kg DM	4	1.6	0.8	0.32	0.8	0.3
ethylbenzene	mg/kg DM	2	0.8	0.4	0.16	0.4	0.15
m+p-xylene	mg/kg DM						0.1
o-xylene	mg/kg DM						0.05
xylenes (sum)	mg/kg DM	3	1.2	0.6	0.24	0.6	
styrene	mg/kg DM	0.8	0.32	0.16	0.064	0.16	0.05
hexane	mg/kg DM	1.5	0.6	0.3	0.12	0.3	0.1
heptane	mg/kg DM	25	10	5	2	5	0.5
octane	mg/kg DM	75	30	15	6	15	0.5
Mineral oil	mg/kg DM	1000	300	200	60	150	50
Polycyclic aromatic hydrocarbons							
naphthalene	mg/kg DM	1.5	0.3	0.3	0.06	0.15	0.15
benzo(a)pyrene	mg/kg DM	0.5	0.3	0.1	0.06	0.15	0.06
phenanthrene	mg/kg DM	60	15	12	3	7.5	0.2
fluoranthene	mg/kg DM	20	2	4	0.4	1	0.2
benzo(a)anthracene	mg/kg DM	5	3.9	1	0.78	2	0.2

WATER BED		BSN norm[1]	Guide value.[2]/ Free use[3]	1/5 BSN	1/5 GV	1/2 GV	Reporting limit
chrysene	mg/kg DM	10	2.5	2	0.5	1.25	0.2
benzo(b)fluoranthene	mg/kg DM	2	1.1	0.4	0.22	0.55	0.1
benzo(k)fluoranthene	mg/kg DM	1	0.6	0.2	0.12	0.3	0.1
benzo(ghi)perylene	mg/kg DM	160	0.3	32	0.06	0.15	0.15
indeno(1,2,3-cd)pyrene	mg/kg DM	1	0.7	0.2	0.14	0.35	0.1
anthracene	mg/kg DM	3	2.4	0.6	0.5	1.2	0.1
fluorene	mg/kg DM	45	9.5	9	1.9	4.7	0.1
dibenzo(a,h)anthracene	mg/kg DM	0.5	0.3	0.1	0.06	0.15	0.06
acenaphthene	mg/kg DM	9	3.1	1.8	0.62	1.5	0.1
acenaphthylene	mg/kg DM	1	0.6	0.2	0.12	0.3	0.1
pyrene	mg/kg DM	125	21	25	4	10	0.2
Cyanides							
Cyanide total	mg/kg DM						
Cyanide-free	mg/kg DM	5	3	1	0.6	1.5	1
Cyanide-non-chl.ox. (Total CN-free CN)	mg/kg DM	5	3	1	0.6	1.5	1
Polychlorinated biphenyls (PCB)[4]							
Sum of 7 congeners	mg/kg DM		0.033		0.007	0.017	0.015
PCB 28	mg/kg DM						(0,002)
PCB 52	mg/kg DM						(0,002)
PCB 101	mg/kg DM						(0,002)
PCB 118	mg/kg DM						(0,002)
PCB 153	mg/kg DM						(0,002)
PCB 138	mg/kg DM						(0,002)
PCB 180	mg/kg DM						(0,002)
Organochlorine pesticides							
alpha-HCH	mg/kg DM		0.05				0.01
gamma-HCH	mg/kg DM		0.05				0.01
beta-HCH	mg/kg DM		0.05				0.01
o,p'-DDE	mg/kg DM		0.05				0.01
p,p'-DDE	mg/kg DM		0.05				0.01
o,p'-DDD	mg/kg DM		0.05				0.01
o,p'-DDT	mg/kg DM		0.05				0.01
p,p'-DDD	mg/kg DM		0.05				0.01
p,p'-DDT	mg/kg DM		0.05				0.01
gamma-chlordane	mg/kg DM		0.05				0.01
alpha-chlordane	mg/kg DM		0.05				0.01
alpha-endosulphan	mg/kg DM		0.05				0.05
beta endosulphan	mg/kg DM		0.25				0.05
endosulfansulphate	mg/kg DM		0.25				0.01
dieldrin	mg/kg DM		0.05				0.01
aldrin	mg/kg DM		0.1				0.02

(1) BSN: soil remediation norm destination type 1 Annex IV Vlarebo 2008; Soil Remediation Norms
(2) Annex II Vlarebo 2008: Guide values for soil quality
(3) Annex V Vlarebo 2008: Values for free use of soil materials
(4) the reporting limits for individual marker PCBs are approximate

D.14 Package B.6 Use of soil materials

Parameter	**Norm[1] mg/kg DM**	**Norm µg/l**	1/5 norm µg/l	1/2 norm µg/l	RG µg/l	RG mg/kg DM
Leachability values (CMA/2/II/A.19)						
Arsenic	0.3	30	6	15	10	0.1
Cadmium	0.02	2	0.4	1	0.75	0.0075
Chromium	0.1	10	2	5	5	0.05
Copper	0.6	60	12	30	10	0.1
Nickel	0.2	20	4	10	10	0.1
Lead	0.3	30	6	15	10	0.1
Zinc	1	100	20	50	20	0.2
Mercury	0.003	0.3	0.06	0.15	0.15	0.0015

(1) Annex VII Vlarebo: Leachability values for the use of soil materials for construction soil use or in a dimensionally stable product

Parameter	**Unit**	**BSN norm**	**Guide value[1]**	1/5 BSN	1/5 GV	1/2 GV	Reporting limit
Polychlorinated biphenyls (PCB)[2]							
Sum of 7 PCBs	mg/kg DM		0.033		0.007	0.017	0.015
PCB 28	mg/kg DM						(0,002)
PCB 52	mg/kg DM						(0,002)
PCB 101	mg/kg DM						(0,002)
PCB 118	mg/kg DM						(0,002)
PCB 153	mg/kg DM						(0,002)
PCB 138	mg/kg DM						(0,002)
PCB 180	mg/kg DM						(0,002)

(1) Annex II Vlarebo 2008: Guide values for soil quality
(2) The reporting limits for individual marker PCBs are approximate

D.15 Package B.7 Disposal of soil materials

Parameter	**Unit**	**Norm value**	1/5 norm	1/2 norm	Reporting limit
TCE extractable (apolar) substances with IR	m/m % DM	2	0.4	1	0.1
Loss on ignition	m/m % DM	10%			1%
Solvents (non-specific)	%	1	0.2	0.5	0.2
EOX	mg Cl/kg DM	1000	200	500	200
Shear strength	N/m2	10	2	5	2

1-step shaking test (CMA/2/II/A.12) with determination in eluate of:						
Parameter	**Unit**	**Norm value**	1/5 norm	1/2 norm	Reporting limit	**mg/kg DM at L/S 10**
pH	-				not relevant	not relevant
Arsenic	µg/L	50	10	25	15	0.15
Barium	µg/L	2000	400	1000	400	4

Lead	µg/L	50	10	25	20	0.2
Cadmium	µg/L	4	0.8	2	1.5	0.015
Total chromium	µg/L	50	10	25	10	0.15

1-step shaking test (CMA/2/II/A.12) with determination in eluate of:						
Parameter	**Unit**	**Norm value**	1/5 norm	1/2 norm	Reporting limit	**mg/kg DM at L/S 10**
Chromium VI	µg/L	500	100	250	100	1
Copper	µg/L	200	40	100	40	0.4
Nickel	µg/L	40	8	20	10	0.1
Mercury	µg/L	1	0.2	0.5	0.5	0.005
Zinc	µg/L	400	80	200	50	0.5
Molybdenum	µg/L	50	10	25	15	0.15
Antimony	µg/L	6	1.2	3	3	0.03
Selenium	µg/L	10	2	5	5	0.05
Fluoride	mg/l	1	0.2	0.5	0.2	2
Cyanide	mg/l	1	0.2	0.5	0.2	2
Chloride	mg/l	80	16	40	20	200
Sulphate	mg/l	100	20	50	20	200
Ammonium	mg/l	1000	200	500	200	2000
Nitrite	mg/l	30	6	3	6	60
Total dissolved solids (TDS)	mg/l	400	80	200	100	1000
Dissolved organic carbon (DOC)	mg/l	50	10	25	10	100 0.2
Phenol index	µg/L	100	20	50	20	1

Perfluoroalkyl and polyfluoroalkyl compounds (PFAS)				**Reporting limit**
perfluoro-n-butanoic acid (PFBA)	µg/kg DW			**0.5**
perfluoro-n-pentanoic acid (PFPeA)	µg/kg DW			**0.5**
perfluoro-n-hexanoic acid (PFHxA)	µg/kg DW			**0.5**
perfluoro-n-heptanoic acid (PFHpA)	µg/kg DW			**0.5**
perfluoro-n-octanoic acid (PFOA)	µg/kg DW			**0.5**
perfluoro-n-nonanoic acid (PFNA)	µg/kg DW			**0.5**
perfluoro-n-decanoic acid (PFDA)	µg/kg DW			**0.5**
perfluoro-n-undecanoic acid (PFUnDA)	µg/kg DW			**0.5**
perfluoro-n-dodecanoic acid (PFDoDA)	µg/kg DW			**0.5**
perfluoro-n-tridecanoic acid (PFTrDA)	µg/kg DW			**0.5**
perfluoro-n-tetradecanoic acid (PFTeDA)	µg/kg DW			**0.5**
perfluoro-n-hexadecanoic acid (PFHxDA)	µg/kg DW			**0.5**
perfluorobutane-n-sulphonic acid (PFBS)	µg/kg DW			**0.5**
perfluoropentane-n-sulphonic acid (PFPeS)	µg/kg DW			**0.5**
perfluorohexane-sulphonic acid (PFHxS)	µg/kg DW			**0.5**
perfluoroheptane-n-sulphonic acid (PFHpS)	µg/kg DW			**0.5**
perfluorooctane-n-sulphonic acid (PFOS)	µg/kg DW			**0.5**
perfluorononane-n-sulphonic acid (PFNS)	µg/kg DW			**0.5**
perfluorooctanesulphonic acid (PFDS)	µg/kg DW			**0.5**
perfluorododecane sulphonic acid (PFDoDS)	µg/kg DW			**0.5**
perfluorooctane sulphonamide (PFOSA)	µg/kg DW			**0.5**
N-methylperfluoroctan sulphonamide (MePFOSA)	µg/kg DW			**0.5**
N-ethyl perfluorooctane sulphonamide (EtPFOSA)	µg/kg DW			**0.5**
N-methyl perfluorooctane sulphonamidoacetic acid (MePFOSAA)	µg/kg DW			**0.5**

N-ethyl perfluorooctane sulphonamidoacetic acid (EtPFOSAA)	µg/kg DW			**0.5**
4:2 fluorotelomer sulphonic acid (4:2 FTS)	µg/kg DW			**0.5**
6:2 fluorotelomer sulphonic acid (6:2 FTS)	µg/kg DW			**0.5**
8:2 fluorotelomer sulphonic acid (8:2 FTS)	µg/kg DW			**0.5**
8:2 fluorotelomer phosphate diester (8:2 diPAP)	µg/kg DW			**0.5**
10:2 fluorotelomer sulphonic acid (10:2 FTS)	µg/kg DW			**0.5**
perfluoro-2-propoxypropanoic acid (HFPO-DA)	µg/kg DW			**0.5**
4.8-dioxa-3H-perfluornonanoic acid (DONA)	µg/kg DW			**0.5**
perfluoro-4-ethylcyclohexane sulphonic acid (PFECHS)	µg/kg DW			**0.5**
perfluoro-n-butane sulphonamide (PFBSA)	µg/kg DW			**0.5**
N-methylperfluoro-n-butane sulphonamide (MePFBSA)	µg/kg DW			**0.5**
perfluoro-n-hexane sulphonamide (PFHxSA)	µg/kg DW			**0.5**
perfluoro-n-octadecanoic acid (PFODA)	µg/kg DW			**4**
6:2 fluorotelomer phosphate diester (6:2 diPAP)	µg/kg DW			**4**
6:2/8:2 fluorotelomer phosphate diester (6:2 8:2 diPAP)	µg/kg DW			**4**
N-methyl perfluoro-n-butane sulphonamide acetate (MePFBSAA)	µg/kg DW			**4**
perfluoroundecane sulphonic acid (PFUnDS)	µg/kg DW			**4**
perfluorotridecane sulphonic acid (PFTrDS)	µg/kg DW			**4**
perfluorooctanoic acid (total of linear and branched) (PFOAtotal)	µg/kg DW			**0.5**
perfluorooctane sulphonic acid (total of linear and branched) (PFOS total)	µg/kg DW			**0.5**
perfluorooctane sulphonamide (total of linear and branched) (PFOSA total)	µg/kg DW			**0.5**
N-methylperfluorooctane sulphonamide (total linear and branched) (MePFOSAtotal)	µg/kg DW			**0.5**
N-ethylperfluorooctane sulphonamide (total linear and branched) (EtPFOSAtotal)	µg/kg DW			**0.5**
perfluorohexane sulphonic acid (total of linear and branched) (PFHxStotal)	µg/kg DW			**0.5**

D.17 Package G.2 PFAS in groundwater

For PFAS reporting limit requirements in groundwater, reference is made to WAC procedure WAC/VI/A/001.

ANNEX E MINIMAL REQUIREMENTS WITH RELATION TO MEASUREMENT UNCERTAINTIES

The accredited laboratories and VITO have derived measurement uncertainties in the context of ISO 17025 for the analytical parameters covered by the accreditation. These were collected and processed by VITO. It has been assumed for the processing that these data also include sample pre-treatment excluding sampling. The results were discussed in the corresponding working groups.

E.1 Package A.2.1 Use as fertiliser/soil improver — Compost

Parameters	Units	U (%)
Moisture	weight % on fresh weight on fresh weight	10%
pH		10%
Organic matter	weight % on fresh weight	30%
Total nitrogen	%w/w N (fresh weight)	30%
Ammoniacal nitrogen	mg NH4-N/l compost (fresh weight)	30%
Nitrate nitrogen	mg NO3-N/l compost (fresh weight)	30%
Conductivity* at 25 °C *(in 1/5 dilution)	µs/cm (fresh weight)	30%
Heavy metal		
Arsenic	mg/kg DM	50%
Cadmium	mg/kg DM	50%
Copper	mg/kg DM	50%
Lead	mg/kg DM	50%
Chromium	mg/kg DM	50%
Nickel	mg/kg DM	50%
Zinc	mg/kg DM	50%
Mercury	mg/kg DM	50%
Germinable seeds	number/l (fresh weight)	-
Stones > 5 mm	weight % on fresh weight on fresh weight	-
Impurities* > 2 mm	weight % on fresh weight on fresh weight	-
Phytotoxicity	%	-
T max	°C	7°C(1)

(1) Absolute limit

E.2 Package A.2.1 Use as fertiliser/soil improver — sewage sludge

Parameter	U %
Acidity	10%
Dry residue	10%
Organic matter (=depletion)	30%
Nitrogen	30%
Phosphorus pentoxide	30%

E.3 Package A.2.2 Use as fertiliser/soil improver — organic parameters

Parameter	U (%)
Hydrochlorocarbons	
sum of 1,2,3,5-tetrachlorobenzene and 1,2,4,5-tetrachlorobenzene	50%
1,2,3,4-tetrachlorobenzene	50%
pentachlorobenzene	50%
hexachlorobenzene	50%
Polycyclic aromatic hydrocarbons	
Acenaphthene	50%
Acenaphthylene	50%
Anthracene	50%
Naphthalene	50%
Benzo(a)pyrene	50%
Phenanthrene	50%
Fluoranthene	50%
Benzo(a)anthracene	50%
Chrysene	50%
Benzo(b)fluoranthene	50%
Benzo(k)fluoranthene	50%
Benzo(ghi)perylene	50%
Indeno(1,2,3-cd)pyrene	50%
Dibenzo(a,h)anthracene	50%
Fluorene	50%
Pyrene	50%
Mineral oil	50%
Polychlorinated biphenyls (sum of 7 PCBs)	
PCB 28	50%
PCB 52	50%
PCB 101	50%
PCB 118	50%
PCB 153	50%
PCB 138	50%
PCB 180	50%

E.4 Package A.3.4 Use as a building material — pastelike and solid

Parameter	U %
Metals (total concentration)	
Arseni c Cadmi um Chrom ium Coppe r Mercu ry Lead Nickel Zinc	50% 50% 50% 50% 50% 50% 50% 50%

Leachability of metals
(column test CMA/2/II/A.9.1 at L/S=10 l/kg DM)

Parameter	U %
Arsenic	70 %
Cadmiu	70%
m	70%
Chromi	70%
um	-
Copper	7
Mercur	0
y	%
Lead	70%
Nickel	70%
Zinc	-
Antimo	-
ny	-
Barium	-
Molybde	-
num	-
Vanadiu	-
m	
Cobalt	
Selenium	
Tin	
Anions leachability (column test CMA/2/II/A.9.1 at L/S=10 l/kg DM)	
Bromi	-
de	-
Chlori	-
de	-
Fluori	
de	
Sulphate	

Parameter	U (%)
Monocyclic aromatic hydrocarbons (BTEXS)	
Benzene	50%
Toluene	50%
Ethylbenzene	50%
Xylenes (sum)	50%
Styrene	50%
Alkanes	
n-hexane	50%
n-heptane	50%
n-octane	50%
Polycyclic aromatic hydrocarbons	
Naphthalene	50%
Benzo(a)pyrene	50%
Phenanthrene	50%
Fluoranthene	50%
Benzo(a)anthracene	50%
Chrysene	50%
Benzo(b)fluoranthene	50%
Benzo(k)fluoranthene	50%
Benzo(ghi)perylene	50%
Indeno(1,2,3-cd)pyrene	50%

Acenaphthene	50%
Acenaphthylene	50%
Anthracene	50%
Dibenzo(a,h)anthracene	50%
Fluorene	50%

Parameter	U (%)
Pyrene	50%
Mineral oil	50%
Polychlorinated biphenyls (sum of 7 PCBs)	
PCB 28	50%
PCB 52	50%
PCB 101	50%
PCB 118	50%
PCB 153	50%
PCB 138	50%
PCB 180	50%
Cyanides	
Free cyanide	-
Non-chlorine oxideable cyanides	-

E.5 Package A.4 Burning — oil, pastelike and solid

Parameter	U (%)
Water content	10%
Flash point	10%
Loss on ignition	30%
TOC	30%
Calorific value	30%
Chlorides	30%
Fluorides	50%
Sulphur	50%
Metals (total concentration)	
Cadmium	50%
Thallium	50%
Mercury	50%
Antimony	50%
Arsenic	50%
Lead	50%
Chromium	50%
Cobalt	50%
Copper	50%
Manganese	50%
Nickel	50%
Vanadium	50%
Tin	50%
EOX	50%
pentachlorophenol	50%
benzo(a)pyrene	50%

	U (%)
Polychlorinated biphenyls (PCBs)	
Sum of 7 congeners	50%
PCB 28	50%
PCB 52	50%
PCB 101	50%
PCB 118	50%
PCB 153	50%
	U (%)
Polychlorinated biphenyls (PCBs)	
PCB 138	50%

PCB 180	50%

E.6 Package A.5.1 Landfills — pastelike and solid

Parameter	U (%)
Dry residue	10%
Mineral oil with GC-FID	50%
Solvents (non-specific)	50%
EOX	50%
Loss on ignition	30%
Total Organic Carbon (TOC)	30%
Shear strength	-
1-step shaking test (CMA/2/II/A.12) with determination in eluate of:	
pH	10%
Arsenic	50%
Barium	50%
Lead	50%
Cadmium	50%
Total chromium	50%
Chromium VI	50%
Copper	50%
Nickel	50%
Mercury	50%
Zinc	50%
Molybdenum	50%
Antimony	50%
Selenium	50%
Fluoride	50%
Cyanide	50%
Chloride	50%
Sulphate	50%
Total dissolved solids (TDS)	50%
Dissolved organic carbon (DOC)	50%

E.7 Package A.5.2 Landfills — pastelike and solid

Parameter	U (%)
Monocyclic aromatic hydrocarbons (BTEXS)	
Benzene	50%
Toluene	50%
Ethylbenzene	50%
Xylenes (sum)	50%
Styrene	50%
Polycyclic aromatic hydrocarbons	
Naphthalene	50%
Benzo(a)pyrene	50%
Phenanthrene	50%
Fluoranthene	50%
Benzo(a)anthracene	50%

Parameter	U (%)
Chrysene	50%
Benzo(b)fluoranthene	50%
Benzo(k)fluoranthene	50%
Benzo(ghi)perylene	50%
Indeno(1,2,3-cd)pyrene	50%
Acenaphthene	50%
Acenaphthylene	50%
Anthracene	50%
Dibenzoic(a,h)anthraces	50%
Fluorene	50%
Pyrene	50%
Phenol index	50%

Quality requirements for analytical methods

CONTENTS

1 OBJECTIVE AND SCOPE

This procedure replaces procedure CMA/6/D of June 2024.

An analysis of a sample will include carrying out as faithfully as possible a complete set of steps in the laboratory from the usual sample pre-treatment (subsampling, etc.) to measurement and calculation. The quality system should include each of these steps.

The quality requirements for the measurement set out in this procedure are generally applicable for the determination of various chemical parameters in groundwater, soil, waste and other materials in implementation of the Soil Decree and the Materials Decree. For the quality requirements for the determination of organic parameters, criteria have been established which apply to all procedures described in Part 3 of CMA. General guidelines for the quality requirements for the determination of inorganic parameters (CMA Part 2) were laid down in this procedure. In addition, specific/additional requirements may be prescribed in the relevant procedure for a specific inorganic parameter.

The quality requirements for bacteriological parameters set out in this procedure are applicable to derived products released from processors of animal by-products: solid fractions (bone meal, feather meal, blood meal, etc.) and liquid fractions (fat). These quality requirements apply for the procedure described under CMA Part 4.

2 QUALITY FOLLOW-UP OF SAMPLE PRE-TREATMENT

For example, the quality follow-up of sample pre-treatment may include duplicate analyses on a number of parameters and/or dry weight content. For this purpose, two sub-samples will be taken after the sample pre-treatment, which will pass the full analysis path.

3 QUALITY REQUIREMENTS FOR INORGANIC PARAMETERS (CMA PART 2)

(1) Linearity

- The calibration model (linear or quadratic) will be recorded during validation and will be periodically checked according to one of the following procedures:
    - At least six-monthly verification of the calibration model and in the case of any serious instrumental procedure; or
    - Verification of residual norm deviations in linear/quadratic regression analysis, only applicable if the entire measurement range is calibrated.

(2) Sensitivity

- At least at the start of each measurement series, the device sensitivity will be checked. The device must be and remain sufficiently sensitive to meet the reporting limit. The procedure may be completed by the laboratory.
  E.g: Ion chromatography, spectrophotometry: area of a specified parameter

(3) Calibration

- If calibration is carried out by means of a calibration line, at least 5 calibration solutions (including zero if applicable) are analysed for each measurement series and spread over the linear range. Then, linear regression is used to calculate the equation of the calibration line, at which the correlation coefficient is at least 0.995. A maximum of 1 point may be removed, but not the lowest point, and at least 4 points should be retained. Removal of the highest calibration point, resulting in a restriction of the calibration area. The deviation from each point to the curve will not exceed 10 % (except point at level ≤ 2 x reporting limit). The deviation from the point at level ≤ 2 x reporting limit may not deviate more than 25 % from the line.
- If the reporting limit is lower than ½ of the lowest concentration of the calibration solution (different from zero), an additional inspection should be performed at the level of the reporting limit. The deviation from the point at the level of the reporting limit may not deviate more than 25 % from the theoretical value.
- If any of the criteria are not met, a new calibration will be established with or without new calibration solutions.

Notice: Norm solutions are created in a similar medium (e.g. acid, H_2O, etc.) as the samples.

Notice: The permitted deviation should always be defined in relation to the measurement uncertainty. In the case of small measurement uncertainties, the permitted deviation is smaller.

- If validation has shown that there is a quadratic rather than a linear relationship between concentration and response, a quadratic curve can be used for calibration.
- A maximum of one point may be removed, but not the lowest point, and at least five points (different from the origin) should be retained. Removal of the highest calibration point, resulting in a restriction of the calibration area. The deviation from each point to the curve will not exceed 10 % (except point at level ≤ 2 x reporting limit). The deviation from the point at level ≤ 2 x reporting limit may not deviate more than 25 % from the line.
- If any of the criteria are not met, a new calibration will be established with or without new calibration solutions.

Notice: The permitted deviation should always be defined in relation to the measurement uncertainty. In the case of small measurement uncertainties, the permitted deviation is smaller.

- If possible, do not force the calibration lines through the origin.

- After calibration, the following solutions are measured and checked:
    - Procedure and/or calibration blank solution: the blank measurement value is less than half of the reporting limit or max. 10% of the measured value, depending on what is highest of the two;
    - Independent inspection norm and/or drift inspection: the measured concentration may deviate no more than 10 % from the actual value.

- The calibration curve is verified at the end of each measurement series by analysis of the drift inspection. The measured concentration may deviate no more than 10 % from the actual value.

- A procedure blank is included for each set of samples. The procedure blank runs through the full analysis cycle. The value of the blank should be monitored. Unless specified in the procedure, the blank correction is not applied.

Notice: Different containers are always used to check the procedure blank.

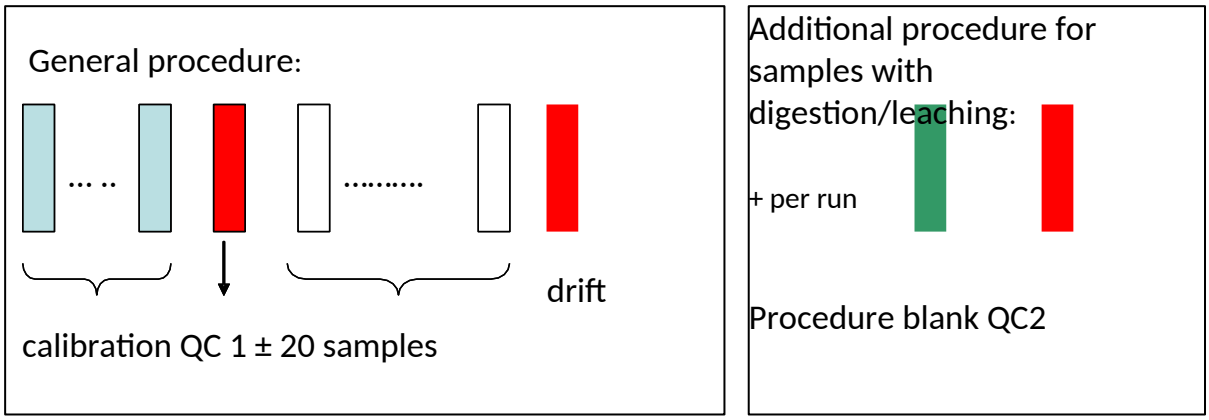
- Where daily calibration is not performed, at least the following controls are carried out:
  - Measurement of (highest) calibration norm: the measured concentration may deviate no more than 10 % from the actual value.
  - Procedure and/or calibration blank solution: the blank measurement value is less than half of the reporting limit or max. 10% of the measured value, depending on what is highest of the two;
  - Independent inspection norm and/or drift inspection: the measured concentration may deviate no more than 10 % from the actual value. In the case of a linear regression, 1 inspection norm is measured, while 2 inspection norms (e.g. concentration levels 1/3 and 2/3 of the measurement range) are measured in the case of a quadratic regression.

(4) Generally applicable procedure (including sample pretreatment) for methods using a calibration line

The following clarification of procedure is set out in the diagram below:

- QC 1: independently created inspection;
- Analysis series of ± 20 samples: this is an approximate number, the laboratories must be able to demonstrate that the frequency of execution of QA/QC is chosen in such a way as to provide sufficient quality assurance;
- Drift: calibration norm or independent norm (QC1);
- In the case of samples with destruction/leaching, a procedure blank and a QC sample (QC2), both of which have passed through the whole procedure, should be analysed for each series (run of a digestion/leaching device).

In addition, possible memory effects should be avoided.



The criteria for quality controls should be defined within the laboratory in such a way that the legally defined performance characteristics are met. The measurement value of QC1 will be within ± 10 % of the actual value.

(5)

(6) Control sample

For each set of samples, 1 inspection sample is included. This inspection sample runs through the full analysis cycle. If several series are analysed per day, 1 inspection sample per day is sufficient.
A inspection sample can be:

- a certified reference material
- an actual sample
- defined synthetic matrix
- a doped sample, with a matrix that is representative of the samples analysed in the laboratory. All representative matrices should be considered over time.

The following 1st line inspection samples are presented by VITO and must be analysed at the specified frequency:

- Accreditation package A.4 Combustion:
  - Wood waste for the determination of As, Cu, Pb, Cr, F, Cl, pentachlorophenol and benzo(a)pyrene – frequency daily (with analysis)
- Accreditation package A.3.1 Loose building material
  - Ash/slag to perform column test and determination of pH, conductivity, elements present – frequency monthly (with analysis)
- Accreditation package A.5.1 Disposal – general parameters
  - Ash/slag to perform shake test and determination of pH, conductivity, elements and anions present, TDS – frequency weekly (with analysis)
- Accreditation package A.2.1 Fertiliser/soil improver – inorganic parameters
  - Dried digestate for the determination of pH, conductivity, organic matter, total N, NH_4-N, elements present, P_2O_5 – frequency daily (with analysis). The parameters NaO, K_2O, CaO, MgO can be observed, but are not obligatory.
- Accreditation package A.2.3 Fertiliser/soil improver – specific parameters
  - Dried digestate for the determination of stability – frequency daily (with analysis).

(7) Duplicate analyses (as an alternative to inspection sample, if not available)

For each set of samples, 1 duplicate analysis is included. If several series are analysed per day, 1 duplicate analysis per day is sufficient.

4 QUALITY REQUIREMENTS FOR ORGANIC PARAMETERS (CMA PART 3)

Preliminary remark: a ‘measurement series’ lasts up to 72 hours.

(1) Linearity

The calibration model (linear, or quadratic, or bracketing) is established at the validation stage. ‘Bracketing’ means working with several successive linear sub-areas. Linearity should be checked every six months and at every severe instrumental procedure.

(2) Calibration

- If the calibration is carried out by the relative response factor, the RRF shall be determined with at least one calibration solution. The calibration solution is injected at least at the start and at the end of each measurement series, and further at least every 15 samples. The concentrations in this calibration solution are approximately in the middle of the linear range or are representative of the expected sample concentrations. The concentrations in a sample are calculated using the average RRF of the 2 calibration solutions between which the sample was injected. The RRFs of the 2 consecutive calibration solutions may not deviate by more than 10 % from their mean (except for VOC, phenols and OPP/triazines: 15%).

- if the calibration is done by means of 'bracketing', the calibration series will be injected at the start and at the end of the measurement series, and at least every 15 samples. The concentrations in a sample are calculated using the average RRF of the two injections of the two points between which the sample is included. The RRFs of the two consecutive corresponding calibration solutions will not deviate by more than 10 % from their mean (except for VOC, phenols and OPP/triazines: 15%).

- If calibration is carried out by means of a calibration straight line, at least four calibration solutions will be analysed for each measurement series with concentrations greater than zero and spread over the linear portion. The lowest concentration may not exceed 2 times the lower limit of the measuring range. Then, linear regression will be used to calculate the equation of the calibration straight line. A maximum of one point may be removed and at least four points (different from the origin) must be retained. If the lowest point is omitted, the reporting limit should be adjusted. If the highest point is omitted, the measuring range shall be restricted at the top. The deviation from each point to the curve shall not exceed 20 %. The lowest point may differ by 25 % from the straight line. If the calibration norms pass through the sample reprocessing, the lowest point may differ by 35 %.
Note: for measurement with FID (mineral oil), the calibration curve should not be set for each measurement series, but at least once a month. A calibration solution will be injected at the start and end of each measurement series, and then at least every 15 samples, to check the calibration curve. The calculated concentration may differ by up to 10 % from the real concentration.
- If the validation has shown that there is a quadratic rather than a linear relationship between concentration and response, a quadratic curve can be used for calibration.
For this purpose, a minimum of 5 calibration solutions are analysed at the start of the measurement series with concentrations spread over the measurement range. The lowest concentration may not exceed 2 times the lower limit of the measuring range. The equation of the curve is then calculated by means of quadratic curve fitting. A maximum of 1 point may be removed and at least 5 points (different from the origin) must be retained. If the lowest point is omitted, the reporting limit should be adjusted. If the highest point is omitted, the measuring range shall be restricted at the top. The deviation from each point to the curve may not exceed 10 % (15% voor PFAS). The lowest point may deviate 15 %. If the calibration norms pass through the sample reprocessing, the lowest point may differ by 25 %.

- In addition, for all calibrations except those based on RRF and 'bracketing', the drift should be checked during the measurement series. To this end, in the case of a linear calibration curve one of the calibration solutions is injected, in case of a quadratic curve, 2 calibration solutions are injected, 1 of which with a concentration located in the highest third of the curve. Drift controls are carried out at least at the end of the measurement series and further at least every 15 samples. In addition, for measurement series lasting more than 24 hours, at least one drift inspection should be carried out every 24 hours. The measured concentrations in the drift check may differ by up to 20 % (for measurement with FID maximum 10 %) from the actual concentrations.

(3) Sensitivity control

At least at the start and at the end of each measurement series, the device sensitivity shall be checked. The device must be and remain sufficiently sensitive to meet the reporting limit.

(4) Procedure blank

For each series of sample reprocessing, a procedure blank is included. If several series are reprocessed per day then 1 procedure blank is sufficient. The procedure blank passes through the complete sample reprocessing except with the analysis of PFAS where the procedure blank must also go through sample pre-treatment.
Remarks:
- a sample reprocessing series lasts up to 24 hours, meaning that the sample reprocessing of all samples is started within 24 hours. Sample reprocessing means: extraction, saponification, etc. Preparatory steps (e.g. mixing with desiccant, preservation, subsampling, etc.) are not included in the sample reprocessing.
- If within the same sample reprocessing series different matrices are processed for the same parameters but each using their own method (e.g. PLE extraction for solid samples and liquid/liquid extraction for water samples), a procedure blank should be included for each method.
The recorded chromatogram should be free from interfering peaks greater than 10 % of the peaks recorded for the sample, with the exception of sample values less than 5 times the reporting limit used, for which the interfering peaks should not exceed half of the reporting limit used.

(5) Recovery of internal standards and surrogate compounds

The recovery requirements of the internal standards and surrogate compounds depend on the parameter, see the overview in Table 1. If the recovery of the internal standard is lower than the lower limit but higher than 20 % (7 % for CMA/3/Y), the results may be reported subject to a comment on the analysis report (except if the result is less than the reporting limit, then the comment on the report is not necessary). If the recovery of the internal standard is below 20 % (7 % for CMA/3/Y), no quantitative result will be reported. If the extract was diluted with additional internal standard, this should be reported on the analysis report.
Notice:
For the analysis of PFAS in activated carbon, the recovery of the internal standard shall be between 10 % and 200 %. Thus, when determining PFAS in activated carbon, a result may be reported without comment on the report if the recovery of the internal standard is between 10 % and 200 %.

(6) Control sample (water)

For each series of sample reprocessing, 1 inspection sample is included. If several series are reprocessed per day then 1 inspection sample per day is sufficient.
A doped water sample is used as the inspection sample. All components reported in the samples should be added to the inspection sample.
The matrix that is doped can be a real sample, or blank water sample (when analysing groundwater and drinking water), or a synthetic waste water sample (when analysing waste water). All representative matrices should be considered over time.

Recovery of all components should be between 70 % and 130 %. For a minimum number of components (Table 2), the measured concentration or recovery is recorded on a control card with statistical limits. The selected components should be representative of the measured components (in terms of retention time, type of compound, etc.).

(7) Control sample (solid, paste and liquid non-aqueous matrices)

For each series of sample reprocessing, 1 inspection sample is included. If several series are reprocessed per day then 1 inspection sample per day is sufficient.
A inspection sample can be:
- a certified reference material
- an actual sample
- a doped sample, with a matrix that is representative of the samples analysed in the laboratory. All representative matrices should be considered over time. All components reported in the samples should be added to the inspection sample. Doped samples should be 'aged' (min. 4 hours) before the necessary internal standards or surrogates are added and the extraction is started.
Note: When preparing a inspection sample for VOC in solid and pasty matrices, add methanol first and then the VOC. The doped sample shall be aged at least 2 hours before adding the internal standards and performing the shake extraction.

The following 1st line inspection samples are presented by VITO and must be analysed at the specified frequency:

- Accreditation package A.2.2 use as fertiliser/soil improver:
    - Dried digestate for the determination of mineral oil, naphthalene, acenaphthene, fluorene, fenanthrene, anthracene, fluoranthene and pyrene – frequency weekly (with analysis)
- Accreditation package A.3.1 Loose building material
    - Granulate for the determination of mineral oil and 16 PAH — frequency weekly (with analysis)

For a minimum number of components (Table 2), the measured concentration or recovery is recorded on a control card with statistical limits. The selected components should be representative of the measured components (in terms of retention time, type of compound, etc.).
In addition, for a doped sample, the recovery of all components should be between 70 % and 130 % (except aldrin in CMA/3/Y: 50%-130%).

(8) Independent inspection norm

The accuracy of the calibration solutions should be checked regularly (at least every 3 measurement series). To this end, a inspection norm independent of the calibration solutions is measured after calibration. At least 1/3 of the components reported in the samples that are representative of all of the measured components (in terms of type and retention time) should be present in the inspection norm. The concentrations measured should not deviate by more than 20 % from the actual concentrations.

(9) Criteria for identification: retention time and ion ratios

Retention time

For a positive identification of a tested component in a sample, the retention time of its quantification ion will, within certain limits, correspond to that in the calibration norm. The other diagnostic ions should correspond to the quantification ion.

For a GC measurement, the deviation of the relative retention time of a component is maximum 5 seconds. Even if this criterion is not met, a component can be positively identified on the basis of additional elements: prior knowledge of the sample and/or presence of other isomers or of an isomer pattern.

For an LC measurement, the deviation of the relative retention time of a component in case of isotope dilution (i.e. the isotope-labelled variant of the component is used as internal standard) is maximum 2.5 % from the retention time in the calibration norm. If the internal standard is a different isotope-labelled compound than the native component to be measured, the deviation of the relative retention time may be maximum 5 %.

Ion ratios

For analysis with MS (full scan and SIM), HRMS and MS/MS a minimum number of diagnostic ions should be measured. This number is specified in the individual analysis procedures.
The following principles will be taken into account in the choice of diagnostic ion:

- an ion with a high mass (m/z) is preferable to a low-mass ion
- ions of equal mass (m/z) are preferred
- if possible, the molecular ion is used as a diagnostic ion
- diagnostic ions preferably have an intensity of at least 15 % compared to the largest (most intense) mass
- if isotope clusters are present, (e.g. Chlorine) it is preferable to take 2 ions from the same cluster

For positive identification, the ion ratios of a tested component in a sample should be consistent with those in the calibration norm, within certain limits. The ion ratio is defined as:

- in case of MS measurement: the peak area of the diagnostic ion divided by the peak area of the most intense diagnostic ion, times 100 %
- in the case of MS/MS: the peak area of daughter ion divided by the peak area of the most intense daughter ion, times 100 %.

The deviation from a calibration norm is defined as:

- the difference of the ion ratio in the calibration norm and the ion ratio in the sample divided by the ion ratio in the calibration norm, times 100 %.

The maximum deviation of ion ratios from those in the calibration norm is shown in Table 3.
Note: for peaks near the limit of quantification (1 to 3 times the limit of quantification), the deviation may be larger, the assessment will be based on expert judgement.

Table 1: Requirements for recovery of internal standards and surrogates

Method	Recovery requirements for internal standards and surrogates
PCB in oil (CMA/3/A)	50 %-130 % (after acetonitrile purification 30 %-100 %)
PCP and B(a)P in wood (CMA/3/V)	50%-130%
P (CMA/3/B) — measurement with MS	IS: 50 %-130 % (except D8-naphthalene: 40%-130%)
PAH (CMA/3/B) — measurement with HPLC-UV/FLUO	Surrogate: 70 %-130 % (except groundwater: 75%-125%) / Online SPE: matrix addition 16 PAK 75-125 %
PFAS (CMA/3/D)	30 % -200 % (except analysis of activated carbon: 10%-200%)
VOC (CMA/3/E)	IS 50 %-150 % (calculated in relation to the average of the calibration norms)
OCP, PCB and higher CBZ (CMA/3/I)	50 % -130 % (except isotope OCP 50 % -150 % and isotope detected trichlorobenzenes: 40%-130%)
Phenols (CMA/3/K)	50%-130%(*)(**)
Tributyltin (CMA/3/L)	IS 20-50 %/surrogate 70-120 %
Mineral oil (CMA/3/R.1)	50 %-150 % (calculated in relation to the average of the calibration norms)
Mineral oil GC/MS (CMA/3/R.2)	50 %-150 % (calculated in relation to the average of the calibration norms)
Volatile mineral oil (CMA/3/R.5)	50 %-150 % (calculated in relation to the average of the calibration norms)
PAH in SI (CMA/3/W)	30%-130%
Mineral oil in SI (CMA/3/W)	50 %-150 % (calculated in relation to the average of the calibration norms)
PCB and CBZ in SI (CMA/3/X)	50 %-130 % (13C-HCB: 20%-100%)
OCP in the water bed (CMA/3/Y)	10%-60%
Dioxins in soil (CMA/3/G)	50-130%
Multi-class methods GC-MS/MS (CMA/3/T)	50-130 % (except D8 naphthalene 40-130 % and OCP 50-150 %)

(*) D_3-2,4-dimethylphenol may break down under the influence of acids or other matrix influences. If the recovery is less than 80 %, the components may be calculated in relation to another internal standard (e.g. $^{13}C_6$-4-chlorophenol or D_5-2-chlorophenol) instead of D_3-2,4-dimethylphenol.
(**) if the method of double derivatisation is applied for water samples, no requirements are set for the recovery of the internal standard

Table 2: Minimum number of components to be recorded on control card

Method	Minimum number of components
PCB in oil (CMA/3/A)	2
PCP and B(a)P in wood (CMA/3/V)	2
Polycyclic aromatic hydrocarbons (CMA/3/B)	4
PFAS (CMA/3/D)	7 (PFBA, PFOA linear, PFHxDA, PFBS, PFOS linear, 6:2 FTS and EtFOSAA)
VOC (CMA/3/E)	10 (benzene, toluene, an isomer of xylene, isomer of trimethylbenzene, 1,1-dichloroethane, chloroform, trichloroethylene, tetrachloroethylene, monochlorobenzene and an isomer of dichlorobenzene)
Organochlorinated pesticides, polychlorinated biphenyls and higher chlorobenzenes (CMA/3/I)	3 OCP, 2 PCB, 2 CBZ
Phenols (CMA/3/K)	2 chlorophenols
Mineral oil GC/MS (CMA/3/R.1)	1 (Mineral oil)
Mineral oil GC/MS (CMA/3/R.2)	1 (Mineral oil)
Volatile mineral oil (CMA/3/R.5)	1 (Volatile mineral oil)
PAH in SI (CMA/3/W)	4
Mineral oil in SI (CMA/3/W)	1 (Mineral oil)
PCB and CBZ in SI (CMA/3/X)	2 PCB, 2 CBZ
OCP in the water bed (CMA/3/Y)	3
Dioxins in soil (CMA/3/G)	3
Multi-class methods GC-MS/MS (CMA/3/T)	4 PAH, 3 OCP, 2 PCB, 2 CBZ

Table 3: Maximum deviation of ion ratios

Ion Ratio	Max. deviation EI-GC-MS	Max. deviation CI-GC-MS, GC-MS/MS, LC-MS, LC-MS/MS
>50%	+- 10 %	+- 30 %
>20% - 50%	+- 15 %	+- 30%
10% - 20%	+- 20 %	+- 30 %
<10%	+- 50 %	+- 50 %

5 QUALITY REQUIREMENTS FOR THE BACTERIOLOGICAL PARAMETERS DETERMINED ON DERIVED PRODUCTS RELEASED BY PROCESSORS OF ANIMAL BY-PRODUCTS (CMA PART 4)

FIRST-LINE CONTROL

(1) Blank inspection of bacteriological parameters

- *Enterobacteriaceae*
- *Salmonella spp*.
- *Clostridium perfringens*

For the parameters *Enterobacteriaceae* and *Clostridium perfringens*, a blank inspection shall be carried out by placing 1 or 2 ml of sterile buffered peptone water — used to suspend the samples — into an empty petri dish with the corresponding agar (VRBG or chromogenic/(T)SC agar) respectively. These controls shall be incubated and read simultaneously with the analysis of samples under the same conditions.
For *Salmonella*, a blank (from a vial of sterile buffered peptone water) is included throughout all steps of the complete analytical method but should only be checked on one selective dish (XLD or chromogenic medium).

(2) Frequency of blank inspection

For each set of reprocessing of samples, the blanks described in 4.1.1. shall be taken into account. If several series per day are reprocessed per day, one blank inspection is sufficient, provided that all people involved regularly work with the blank inspections.

(3) Positive inspection in the case of media purchased with a quality certificate

For the purpose of the positive inspection, the quality certificate for the relevant batch of nutrient medium made available by the producer and on which the growth of relevant reference strains is evaluated may be used.
In addition, the laboratory itself should perform at least the following quantitative positive controls:

- ✓ testing one *Enterobacteriaceae* strain;
- ✓ testing one *Clostridium perfringens* strain.

For this purpose, a inspection sample (a sterile matrix as closely related as possible to animal by-products) shall be inoculated with this positive inspection strain and treated as any other unknown sample.
The results of the quantitative positive inspections carried out by the laboratory shall be recorded and evaluated using statistical control cards.
For *Salmonella spp.* a qualitative positive inspection at a level close to the limit of detection should be included.

(4) Positive inspection in case of self-prepared media

A qualitative positive inspection will be performed on each self-prepared batch of a particular nutrient medium before the batch is used.
In addition, the same minimum positive controls to be carried out as described in 4.1.3.

(5)

(6) Minimum frequency of quantitative positive controls

The minimum frequency of the positive controls described in 4.1.3. is as follows:

- if this parameter is performed daily or several times a week: test a positive inspection for each new batch of selective media, and repeat the positive inspection at least every month for a batch already tested;
- if the relevant parameter is carried out weekly: test a positive inspection on a monthly basis;
- if the parameter in question is performed monthly: test a positive inspection every two months;
- if the parameter in question is performed less frequently than monthly: every analysis day a positive inspection test.

(7) pH inspection

If the laboratory prepares nutrient media itself, it must carry out a pH inspection measurement each time a new batch of a medium is prepared. If the pH is not between the declared values of the producer, the medium should not be used (any more). For commercial media, the manufacturer's certificate suffices.

(8) Follow-up of different controls

If the results of the positive inspection samples do not meet the specified criteria, or if the blank inspection gives a positive result (>1 cve presumptive parameter/g of sample), the analytical sequence is considered not reliable. A resampling shall then be carried out. Where applicable, the impact on previously reported results should also be assessed.

Second-line inspection

A programme of second-line checks is carried out by having real positive samples analysed by two people. Every parameter should be addressed every six months. In the longer term, all relevant matrices (fat or meal/gelatin) should be tested if possible.

Third-line inspection

Instead of second-line inspection, an accredited laboratory can participate in a ring test offered by third parties, and on a matrix that is comparable to the derived products that are released by processors of animal by-products.

6 REFERENCES

- ISO/NP 21253-1 (2018): Water Quality – Multiclass methods Part 1 – Guidance for identification of target compounds by GC-MS and LC-MS
- NBN EN ISO 7218:2007/Amd 1:2013 (Corrected version 2014-04-15) Bacteriology of food and animal feeding stuffs -- General requirements and guidance for bacteriological examinations

Conditions for reporting sampling data and analysis results by an accredited laboratory

CONTENTS

1 OBJECTIVE AND SCOPE

This procedure replaces procedure CMA/6/E of May 2024.
This procedure prescribes how laboratories which are accredited in the Flemish Region according to VLAREL or still have a valid accreditation according to previous regulations, are required to report sampling data and analytical results of assignments carried out in the capacity of accredited laboratory.

The purpose of this procedure is to establish uniform reporting requirements. The conditions in this procedure are to be regarded as minimum requirements, which take precedence over any bilateral agreements between the accredited laboratory and its sponsor, unless the competent authority determines otherwise.

The scope of this procedure covers all laboratories accredited in the Flemish Region for one or more of the following disciplines: water; air; manure; animal feed; soil; waste and other materials This procedure concerns both sampling and analysis, insofar as the laboratory is accredited for them.

The ISO/IEC 17025 norm, which lays down general requirements for laboratory competence and is to be applied by accredited laboratories, already contains reporting requirements, but these are more generally formulated and not sufficiently focused on the typical problems of environmental analyses (e.g. limited shelf life of samples, etc.). Reporting requirements in the ISO/IEC 17025 norm, which are not explicitly included in this procedure, shall continue to apply, without prejudice, to accredited laboratories. This also applies to reporting instructions that apply to a specific discipline (may be a sub-domain), e.g.

- Code of Good Practice Soil Protection (see https://emis.vito.be/nl/erkende-laboratoria/bodembescherming-gop/compendium-boc - most recent version);
- Methods BAM/part n/20 for reporting (see http://www.emis.vito.be/referentielabo-vlm);
- Ministerial Decree of 19 March 2004 establishing the list of low ammonia emission stabling systems (see https://navigator.emis.vito.be/detail?woId=283&woLang=nl).

This procedure applies to both paper and electronic reporting. If an electronic file is used, it must meet all the requirements of this procedure; otherwise, a classic analysis report on paper, which meets all the requirements of this procedure, must always be sent.

Provisions displayed in square brackets [ ] are not strictly part of this procedure, but are added for clarification purposes.

2 DEFINITIONS

For the purposes of this procedure, the following definitions shall apply:

1° VLAREL: Flemish Government Decree of 19 November 2010 establishing the Flemish regulations on accreditations relating to the environment

2° fit for purpose: suitable for use in the context set out by the sponsor (e.g. specific requirements) or which may be considered to be known by an accredited laboratory (e.g. regulations in force)
3° accredited laboratory: laboratory, accredited by the Flemish Region, which carries out sampling, measurements, analyses or tests in accordance with the provisions in VLAREL
4° contractor: accredited laboratory with which the sponsor has a contractual relationship
5° final report: the analysis report that the client receives from the contractor
6° reporting limit: the value below which a component is considered non-quantifiable ('<'); this is at least the limit of quantification, unless otherwise laid down in the applicable legislation.

3 PRINCIPLES

The following basic principles were used to define the reporting conditions:
- sampling data and analytical results reported by an accredited laboratory must be "fit for purpose", i.e. suitable for use in a defined context;
- the accredited laboratory must be able to obtain these 'fit for purpose' sampling data and analytical results in the most efficient way possible.

In order to achieve this, it is essential that throughout the entire analysis process in its broadest sense — from defining the need for analyses by the sponsor to the reporting of the results by the contractor, i.e. the accredited laboratory to whom the assignment was entrusted — sufficient consultation takes place and clear agreements are made.

Both the sponsors and accredited laboratories bear part of the responsibility
for obtaining fit for purpose analytical results in an efficient manner.

An accredited laboratory may expect the sponsor, and ask the sponsor, if necessary, to:
- communicate the context of the analysis assignment (nature of the sample, purpose) to the laboratory at the earliest possible stage;
- confirm the analysis assignment in good time, i.e. at the latest at the time of the sampling (if carried out by the laboratory) or at the time of delivery of the sample to the laboratory;
- align, as far as possible, specific requirements with the requirements for the use of the accreditation to which the laboratories are subject through VLAREL (e.g. the mandatory use of the compendium method if available, etc.).

It is crucial that the sponsor indicates the nature of the sample correctly, as well as the objectives in the context of which the analysis is carried out.

It is expected from the contractor and, where appropriate, the other accredited laboratories
involved in the execution of the assignment, that they:
- assist the sponsor, if necessary, in defining a sampling and/or analysis assignment in accordance with the applicable regulations;
- have the necessary organisation/facilities to respect the shelf life of samples;

- carry out the necessary analysis work until a fit for purpose result is available, even if this requires additional measurement/analysis;
- optimise own organisation/methods with a view to minimising the use of comments on sampling and analysis reports.

The final responsibility for correctly formulating the research to be carried out and indicating the applicable regulations lies with the sponsor.

The final responsibility for the complete and correct reporting in accordance with this procedure lies with the accredited laboratory which is acting as a contractor. This may be either the laboratory that performs the sampling or the laboratory that performs the measurements/analysis (or at least part of them) depending on whom the sponsor has a contractual relationship with. In the event that the sponsor enters into a contractual relationship with two or more different contractors for a particular assignment, each of these accredited laboratories is responsible for its own part of the assignment.

The final responsibility for the reporting includes the responsibility for the necessary coordination between all accredited laboratories involved: ensuring the necessary agreements on critical deadlines in the analysis process, the availability of a sampling report, etc.

If sampling is carried out by another competent authority (e.g. inspection service; drilling company under the supervision of an approved soil remediation expert; operator if provided for in the regulations; etc.) or by the client himself, the final responsibility of the approved laboratory is limited to raising awareness of compliance with the conditions in this procedure. In such a case, the accredited laboratory will refer to its accreditation status in the analytical results, but not the sampling data.

4 REPORTING OF ASSIGNMENT DATA

4.1 DESCRIPTION OF THE SAMPLE

The description of the sample in the analytical report must be consistent with the context of the analysis assignment and with the characteristics of the sample (or at least the visually perceptible characteristics). The accredited laboratory may expect the sponsor to describe the nature of the sample correctly, as well as the objectives in the context of which the analysis is carried out. However, upon receipt of the sample, the accredited laboratory must verify that the information provided corresponds to the nature and appearance of the sample. In case of doubt as to whether the sample corresponds to the description, or if it is established that a sample cannot be analysed automatically as a normal sample of the type in the description (e.g. sample contains more phases, etc.), the accredited laboratory should contact the sponsor to clarify the inconsistencies. Agreements made must be recorded.

4.2 TESTING FRAMEWORK

As a service to the sponsor or on explicit request, an accredited laboratory can include test values in the analysis report. In this case, it should also be clearly indicated from which legislation or other document these test values come from.

4.3 SEALING SAMPLE

When samples are delivered sealed, the accredited laboratory should indicate on the (sampling or analysis) report whether the seal was intact when the samples were received. In case of a seal that is not intact, the accredited laboratory should take a photograph of it; it should then be kept with the details of the order and handed over as proof upon request by a competent authority.

5 REPORTING OF SAMPLING DATA

Note: the conditions below only apply if the sampling has been carried out by an accredited laboratory; this may be the accredited laboratory that is also responsible for the analyses, or another accredited laboratory.

5.1 MINIMUM SAMPLING DETAILS

The analysis report, or a sampling report accompanying the analysis report, shall include at least the following sampling data:

- date of sampling;
- time of sampling in the case of waste water, surface water, groundwater, air or waste and other materials;
- sampler identification code (e.g. internal identification number, initials, ,...);
- identification (preferably by GPS coordinates) or a detailed description of the location where the sample was taken, if necessary with a sketch and/or photographs attached;
- recording of the characteristics of the measurement site and, where appropriate, verification of their conformity with the compendium or the other method required in accordance with Article 45 of the VLAREL;
- in the case of sampling of a batch: a description of the sampled batch, including batch definition or mixed batch where applicable; justification must also be given for the choices made;
- reference to the sampling method, comprising at least the code of the applicable compendium or norm method and the specification of the sampling technique used;
- additional information if required by the sampling method or regulations; e.g. for waste and other materials: number of grabs taken, grab size, number and size (in litres) of laboratory samples, cf. CMA/1/A.14;
- if the laboratory is accredited for several sub-domains in the discipline in question: the sub-domain to which the sampling relates.

If a sampling report is used to report the aforementioned data, the data of which is not included in the analysis report, the analysis report must refer unambiguously to that sampling report; the analysis and sampling reports are then both submitted to the client and archived. In practice, the sampling report and the analysis report may also form part of an integrated study report, which includes e.g. a discussion of the results and/or technological advice.

If sampling has been carried out at the request of another accredited laboratory acting as a contractor, the laboratory carrying out the sampling must ensure that a sampling report containing at least the aforementioned information is made available to the other laboratory, at the latest at the time when the samples are delivered.

The contractor that carries out the sampling itself, but calls upon another accredited laboratory for part or all of the analyses, does not have to provide a complete sampling report to that laboratory, as the contractor is responsible for the final report. In such a case, it is sufficient to provide the date of sampling and, where appropriate, the sub-domain to which the sampling relates.

If a competent authority is present at the time of sampling and it records some of the minimum data itself, the sampler must have the representative of that competent authority verify that the data recorded is consistent.

The name and address of the accredited laboratory that carried out the sampling should be unambiguously deducible from the analysis report or a sampling report attached to the analysis report, and correspond to those to which the accreditation was issued.

5.2 VLAREL ACCREDITATION LOGO AND REFERENCE TO ACCREDITATION STATUS FOR SAMPLING

In accordance with Article In accordance with Article 49 of the VLAREL, an accredited laboratory shall affix the VLAREL accreditation logo to the analysis report or sampling report:

In addition, the laboratory must clearly indicate for which samples it is accredited and for which it is not. The following rules apply:
- each sampling method in an analysis or sampling report must clearly indicate, by means of a code or symbol, whether or not it falls within the scope of the laboratory's accreditation;
- the code or symbol used must be explained, e.g. in a footnote of the analysis or sampling report.

If the laboratory is accredited for several sub-domains in the relevant discipline, this should be taken into account when referring to the accreditation status.

For reference to the accreditation status for measurements, tests and analyses, see point 7.2.

5.3 USE OF SAMPLING COMMENTS

A comment on the sampling or analysis report is necessary in the following cases:

*a) if, **at the request of the client**, the sampling was carried out in a manner **not fully compliant with** the compendium or the other required method according to Article 45 of the VLAREL.*

In this case, it must be clearly indicated that the deviation is at the request of the client; it must also be specified where there has been a deviation from the compendium or the other required method according to Article. 45 of the VLAREL. If the sponsor has given reasons why they considered the adjustment necessary, this can also be stated.

b) *if*, ***due to local conditions***, *sampling* ***could not be carried out in full compliance with*** *the compendium or the other method required according to Article 45 of the VLAREL.*

In this case, it should be specified in which points were deviated from the compendium or the other required method under Art. 45 of the VLAREL and because of what local circumstances this was necessary.
An accredited laboratory is expected to clearly indicate any defects in an installation from the sponsor which make compliant sampling impossible. Remedying this is the responsibility of the sponsor.

c) *if* ***abnormalities related to the operation of the installation to be sampled*** *were reported by the client or were identified.*

In this case, it is necessary to specify which operational abnormalities are involved and whether they have been reported by the client or have been identified/confirmed by the accredited laboratory.
An accredited laboratory may not make a judgement on whether or not reported abnormalities are justified or on the impact that the abnormalities may have had on the representativeness of the sampling.

In the following case, a comment on the analysis report is necessary:

d) *if on receipt at the analytical laboratory a sample* ***does not conform to*** *the compendium or the other required method according to art. 45 of the VLAREL appears* ***in terms of preservation, container or quantity***.

In this case, it is necessary to specify the points not complying with the requirements of the compendium or the other required method according to Article 45 45 of the VLAREL.

The inclusion of comments on sampling should not lead to the omission of the reference to accreditation and therefore does not relieve the accredited laboratory from the other requirements of this procedure.

6 REPORTING ON CRITICAL DEADLINES IN THE ANALYSIS PROCESS

6.1 MINIMUM REQUIRED DATA

At least the date of receipt of the sample by the laboratory should be included in the analysis report, as well as the date of analysis.

6.2 DEALING WITH THE PRESCRIBED SHELF LIFE OF SAMPLES

For the interpretation of shelf life, the date of sampling must be considered to be day 0. If a composite sample is to be prepared by the accredited laboratory from several samples supplied, day 0 shall be the date of sampling of the oldest sample.

For aggregate samples cf. WAC/I/A/004, which are made up within a given sampling cycle (usually 24 h) via an automatic sampling device, the shelf life starts at the moment the aggregate sample is taken from the sampling device. The aggregate sample of the relevant sampling cycle shall be taken from the sampling device for further analysis no later than the next working day after this sampling cycle, regardless of whether or not further sampling cycles will take place.

Example:
- In the case of a 3-day waste water sampling campaign starting on Monday(night) 0:00h h, the aggregate sample shall be taken from the sampling device from day 1 on Tuesday for further pre-treatment, partial sampling and on-site measurements (i.e. parameters with a shelf life of 24 hours shall be analysed no later than Wednesday). The shelf life of the aggregate sample from day 1 shall start on Tuesday. The composite samples from Day 2 and Day 3 shall then be taken from the appliance on Wednesday and Thursday, respectively. The shelf life of the aggregate samples from Day 2 and Day 3 start on Wednesday and Thursday respectively.
- If the previous sampling campaign is a 5-day, the aggregate samples from days 1, 2, 3 and 4 shall be taken from the sampling device on Tuesday, Wednesday, Thursday and Friday respectively. The day 5 aggregate sample expires on Friday(night) at 23:59h. This aggregate sample is taken from the sampler on the next working day for the accredited laboratory: this can be e.g. on Saturday (if the laboratory has a weekend shift), on Monday (= next working day for a laboratory without weekend shift that is not a legal holiday), on Tuesday (= next working day for a laboratory without weekend shift if Monday is a legal holiday).

In addition, in the context of the interpretation of shelf life, the date of the start of the analysis is important. In the case of analyses requiring digestion, extraction or other preparatory action, this is normally the date on which the aforementioned action was carried out. In the other cases, it is the date on which the content was measured.

The contractor is responsible for respecting the prescribed shelf life of the samples for each parameter to be determined. In the event that the sponsor enters into a contractual relationship with two or more different contractors for a particular assignment, each of these accredited laboratories is responsible for its own part of the assignment.

[An accredited laboratory with final responsibility for reporting is expected to monitor for each parameter (group) how often the shelf life is exceeded and to use the results of this monitoring in the continuous improvement of its processes or organisation within the context of the ISO/IEC 17025 quality system]

6.3 USE OF COMMENTS ON CRITICAL DEADLINES

A comment on the analysis report is necessary in the following cases:

*a) if the **shelf life of the sample** for a particular parameter (group) was **exceeded***

In this case, it should be clearly indicated which parameter (group) is affected.

The laboratory must, upon request, inform the sponsor whether the shelf life was exceeded due to the laboratory itself, the prior process (= late delivery of the sample or the analysis assignment) or a combination of both.

b) if the **date of sampling is not known** is by the laboratory drawing up the analytical report.

In this case, it should be clearly stated that the date of sampling was not communicated to the laboratory and that therefore it cannot be guaranteed that the analyses were carried out within the prescribed shelf life.

7 REPORTING OF ANALYTICAL RESULTS AND INFORMATION RELATING TO THE METHOD USED

7.1 MINIMUM REQUIRED DATA

The analysis report should include at least the following data relating to the analysis results and methods:
- the analysis result and the unit in which it is expressed;
- a reference to the applied method of digestion, leaching and analysis;
- additional information if required by the analysis method or regulations;
- relevant deviations or explanations during the analysis.

The unit in which the final analysis result is expressed should be in accordance with the testing framework. The naming of the parameters should also be aligned as far as possible with the regulations.

The reference to the method of digestion, leaching and analysis, as included in the analysis report, should contain at least the code of the applicable compendium method or the other required method according to Article 45 of the VLAREL.

Relevant deviations or explanations during the analysis mean all additional information necessary for a correct interpretation of the analysis results by the sponsor and a possible end user. See also point 7.4 Use of comments on analysis reports.

The name and address of the accredited laboratory that carried out the analysis should be unambiguously deducible from the analysis report, and correspond to those to which the accreditation was issued.

7.2 VLAREL ACCREDITATION LOGO AND REFERENCE TO THE ACCREDITATION STATUS FOR EACH PARAMETER

In accordance with Article 49 of the VLAREL, an accredited laboratory shall affix the VLAREL accreditation logo to the analysis report:

In addition, the laboratory must clearly indicate for which measurements, tests and analyses it is accredited and for which it is not. The following rules apply:
- for each analysis result reported, it must be clearly indicated, by a code/symbol choice, whether or not it falls within the scope of the laboratory's accreditation;
- the code or symbol used must be explained, e.g. in a footnote of the analysis report.

If the laboratory is accredited for several sub-domains in the relevant discipline, this should be taken into account when referring to the accreditation status.

For the reference to the accreditation status for sampling, see point 5.2.

7.3 CALCULATION OF SUM CONTENTS OR DIFFERENCE CONTENTS

If a procedure for the calculation of sum contents or difference contents is laid down in the relevant regulations, compendium or other required method according to Article 45 45 of the VLAREL, this method should be applied. If no procedure is established, the 'lower bound' principle is applied.

7.4 USE OF COMMENTS IN ANALYSIS REPORTS

A comment on the analysis report is necessary in the following cases:

a) *if **no result** could be obtained **or** if it concerns a component that has been determined according to the compendium or the other required method according to art. 45 of the VLAREL could be determined **only approximately or semi-quantitatively**.*

   In this case, a symbol is chosen to replace or clarify the result. This symbol should be explained by a footnote or comment on the analysis report.

   A reporting limit in the result field is considered to be a result; a remark is then only necessary if a reporting limit that is too high (i.e. higher than required by legislation) is passed on (see point (c)). Even '>' values are considered to be results, but their use is permitted only if this is explicitly stated in the compendium or the other required method according to Article 45 of the VLAREL (e.g. when determining BOD in water).

   The laboratory should, upon request, make additional information, e.g. why no result could be obtained, available to the sponsor.

b) *if a **deviation in calibration or quality control** may compromise the 'fitness for purpose' of the result and this deviation **could not be remedied** by means of a remeasurement/re-analysis with additional precautions cf. the compendium or the other required method according to Article 45 of the VLAREL.*

In this case, the deviation should be clearly defined: which parameter(s) it covers, what deviates and in what direction.

The laboratory should, upon request, make additional information, such as the expected impact on the reported analysis result (suspected underestimation or overestimation, etc.) or what has been done to eliminate this deviation, available to the sponsor.

[As regards the elimination of such deviation, it is assumed that a 'reasonable' additional effort from the accredited laboratory (= 1 re-analysis) is sufficient, provided that sufficient additional technical precautions have been taken (e.g. application of an additional purification technique, use of an adapted sample quantity, etc.).

If, at the time the deviation is detected, the shelf life of the sample has already expired, re-analysis with additional precautions no longer needs to be started.]

c) *if a **higher than the regulatory or customer-required reporting limit** is passed as result.*

In this case, it should be specified that the reporting limit was increased.

The laboratory should, upon request, make additional information, such as the reason (dilution due to matrix effects that cannot be eliminated, too high blank value, etc.) or what has been done to eliminate this deviation (see point b)), available to the sponsor.

[The diluted use of samples with a view to fewer matrix effects is only acceptable if the 'fitness for purpose' of the analysis result is not compromised. This should therefore not result in an excessively high reporting limit, unless other technical possibilities for mitigating matrix effects (cf. compendium or other required method) have proved insufficient for the sample in question.

Where one or more of the range of components to be determined by a specific analytical method are present in extremely high levels in the sample, the reporting limits for the remaining components should, as a general rule, not exceed the guide or limit value for the content of these components according to the applicable regulations.]

d) *If, in carrying out the sample pretreatment, analysis or calculation of the content, **it was necessary to** deviate **from the compendium** or the other required method according to Article 45 of the VLAREL.*

In this case, the deviation should be clearly described as well as the reason for the deviation.

e) *if **sum or difference contents** are reported where at least one of the components has a measurement value below the reporting limit (<RL) **and** the **method to be applied is not specified** in the relevant regulations, compendium or other required method according to Article 45 of the VLAREL.*

In this case, the 'lower bound' principle is applied.

f) *if **more than one result for a parameter to be determined** is reported and these results differ significantly, taking into account the context of the analysis.*

In this case, the difference between the results should be explained in greater detail and the result which the laboratory considers to be the most reliable should be specified.

The inclusion of comments on shelf life (cf. point 6.3) or analyses (cf. point 7.4) should not lead to the omission of the reference to the accreditation and thus does not relieve the accredited laboratory from the other requirements of this procedure.

8 REPORTING IN THE CASE OF (PARTIAL) OUTSOURCING

If sampling or analyses, which according to VLAREL are to be carried out by an accredited laboratory, are outsourced to another accredited laboratory, the following reporting guidelines apply (for the definition of the term final report, refer to point 2):

- the sampling or analysis report of the laboratory carrying out the assignment shall be included in its entirety as an annex to the final report, i.e. final report and accompanying annexes shall always form a single entity;
- the accreditation status for the outsourced sampling or analysis must be indicated by a code or symbol of one's own choice indicating that it concerns outsourced operations; the code or symbol used must be explained, e.g. in a footnote to the final report;
- the method reference for outsourced sampling or analyses in the final report is replaced by the term 'outsourced' or by the method reference of the laboratory carrying out the assignment.

A contractor who outsources part of the assignment bears final responsibility for the whole assignment, in accordance with the provisions of the ISO/IEC 17025 norm, and is therefore liable for both the correct transfer of the samples to/from the other laboratory performing the work and for the results/data provided by that laboratory.

9 WORKFLOW AND REPORTING FOR SELF-ANALYSIS FOR RAW MATERIAL WITH AS RESOURCE CERTIFICATE

VLAREMA Article 2.3.1.3/2(2) provides that

The materials listed in Article 2.3.1.3, which are considered as raw materials, are raw materials sampled and analysed at least once a year by an approved laboratory in the discipline of waste and other materials referred to in Article 6 (5) (e) of the VLAREL of 19 November 2010, unless otherwise specified in the raw material declaration.

The sample is representative of the production over a given time interval. Compliance with the applicable criteria shall be ensured on the basis of representative sampling and analysis.
Depending on the origin, the degree of contamination and the use, resource producers or persons acting on their behalf may restrict the parameter list given in Appendices 2.3.1 and 2.3.2 in consultation with OVAM.

For the materials listed in Article 2.3.1.3, which are considered to be raw materials, the raw material producer or, by way of deviation, the person acting on his behalf, when placing the order for the sampling and analysis of the raw material, shall transmit the number of the raw material declaration to the accredited laboratory carrying out the sampling or analysis, or both, of those materials, requesting that number be included in the sampling report.

The accredited laboratory reports the start of the analysis of the raw material declaration in the Labo Loket - Analysis results of OVAM within 3 working days after sampling. With that notification, the following inspection data are compulsorily reported in the Labo Loket -
Analysis results:

1° identification of the accredited laboratory carrying out the analysis of the materials via the Lab-ID;
2° the number of the raw material declaration; 3° the date of sampling;
4° the reason for sampling via the reference list available on the OVAM website;
5° the identification of the accredited laboratory that carried out sampling, via the Lab-ID;
6° the number of the sample;
7° the sampling report in PDF format.

The Labo Loket - Analysis Results then provides the accredited laboratory with an OVAM order reference for the notified analysis of the materials considered as raw materials.'

Article 2.3.1.3/2 §3 provides that

Records of sampling and analysis of the material considered as a raw material shall be kept on an electronic medium for easy data exchange between the recognised laboratories and OVAM. The minister shall determine in a norm procedure the technical specifications to be met by the data from sampling and analysis, and the technical specifications of data exchange, as provided for in this Article. For the materials considered as raw materials, where more than one accredited laboratory carries out analyses in the context of the same analysis, the accredited laboratory designated by the contractor is responsible for reporting the results to the OVAM.

The recognised laboratories reporting the results of the analyses mentioned in section 2 to OVAM shall provide those analysis data with the OVAM order reference. This shall be done by electronic means immediately after the analysis has been carried out or immediately after receipt of the analysis results from the other recognised laboratories concerned. The exchange of data is done according to the specifications included in the norm procedure.

The raw material producer of metallurgical production processes for ferrous metals shall also provide to the OVAM annually in PDF format the analysis results demonstrating compliance with the ministerial order mentioned in Article 2.3.6.1(2).

Analytical data not covered by the reporting duties of this section shall be kept available to the regulator and OVAM by the raw material producer or the person acting on its behalf for five years.'

For commercial reasons, the report uses a unique command reference linked to the commodity declaration number.

Parties concerned:

- **Contractor**: accredited laboratory commissioned for self-analysis by the raw material producer or the person acting on their behalf and carrying out at least the sampling or part of the analysis. The above condition may be waived only if an essential device is missing for a long period of time or the only authorised person is absent, subject to OVAM's agreement.
- **Approved laboratory**: accredited for sampling, for analysis or for both activities

1) The contractor will start the assignment for self-analysis at OVAM (manually or via API) no later than 3 hours after the sampling has been carried out.
   At the start of the contract, the following information must be filled out: see legal text above - own sampling reference is optional).
   After completing the above information, the contractor receives the unique OVAM assignment reference from OVAM and is also ultimately responsible for stopping the assignment.

2) After sampling, samples are analysed by one or more accredited laboratories. Laboratories other than the contractor will only receive the unique OVAM assignment reference from the contractor with the sample(s). They therefore have no access to the number of the resource certificate to which the self-analysis relates. The identification of the raw material producer is also unknown.

3) Each laboratory accredited for analysis may subcontract parameters to other accredited laboratories indicating the unique OVAM assignment reference.

4) Each analytical laboratory is responsible for its analysis and can upload the results in XML to OVAM (manually or via API), linked to the unique OVAM assignment reference.
   If the analyses are outsourced to another accredited laboratory, the analysis results may also be passed on to the accredited laboratory that has outsourced the analysis. The latter laboratory can then either insert the analysis results in into its own XML file, or choose to upload the XML file prepared by the subcontractor in full to OVAM. Note, however, that the analysis results should include a clear indication of the laboratory that has actually carried out the analysis.

5) The contractor will check that all analysis results for the unique OVAM assignment reference are uploaded and then close the order manually or via API through its own system.

6) If a contract is not completed by the contractor after 1 month, the OVAM will send an email reminder that the contract is still open.

7) OVAM will assess the results of the self-analysis after the contract has been concluded. When the validation was carried out by OVAM, a successful tenderer can notify a new sampling. Unless the analysis is determined per volume.